# Stability
## (Biodegradability)

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## SUMMARY

EU directives require a reduction in landfill and where possible recycling of waste in the form of composted materials. Methods of test are required to indicate the stage in the biodegradation process when the product is suitable for its intended use.

Stability is a term generally used to describe a stage in the composting/biodegradation process at which the level of microbial activity has slowed and will not resurge under altered conditions. Stabilised sewage sludge is a term generally used to describe sludge that has undergone some form of digestion and will not set free malodours whilst for landfill it means the mass will only generate very low levels of gas.
With the word “stability” having different meanings for composted biowaste, sewage and landfill it is perhaps now the time to use different and meaningful terminology.

Many methods of test exist and have been reported in literature, these are reviewed with critical comment.
The need to test for potential biodegradability (short term and long term) on materials intended for landfill before and after treatment is still under review. For materials that have not been separated or treated it may become an EU requirement to test for long-term potential biodegradability. The mix of materials will influence the rate of biodegradation. It may be possible to adapt the proposed method for this purpose should it become necessary.

Methods such as near infrared reflectance (NIR) may have the potential to be able to give a much better picture than any of the existing methods but will require development.

There are many methods in existence as can be seen by any literature search however there are no International standard methods for the determination of biodegradability (stability) after aerobic composting/biodegradation of wastes and similar materials. (The Austrian ASTM method is often cited as a National Standard.)

In soils the term “stability” is more often used to describe physical characteristics and is related to particle size, organic matter and water retention. There are several tests and standards that are intended to give guidance on structural damage during cultivation, erosion, suitability for civil engineering purposes etc. and are referred to in a separate section. Standard methods exist to determine microbial soil respiration. A high rate of respiration would indicate the presence of readily biodegradable materials.

Two ISO standards determine the aerobic biodegradability of plastics; one in particular determines the biodegradability and disintegration of plastics under controlled composting conditions.

It is believed that no one method is yet available to provide all the answers to all the questions.

The proposed draft standard, which is based on several ISO methods, is believed to be able to answer most questions. Further work is required to confirm certain aspects and ruggedness of the proposed method.

# 1. INTRODUCTION

Is there a distinction between biodegradability and stability? Does the same process apply to all wastes, i.e. municipal solid waste, sewage sludge's and materials intended for landfill with or without pre-treatment?

(ISO 11266:1994) describes a method for determination the biodegradation of organic chemicals in soil under aerobic conditions. This standard defines biodegradability as follows:

a) Biodegradation:
The molecular degradation of an organic substance resulting from the complex actions of living organisms.

b) Primary biodegradation
The degradation of a substance to an extent sufficient to remove some chrematistic property of the parent molecule. In practice, this will be determined by analysis as a loss of parent compound or some specific function of the parent compound.

c) Ultimate biodegradation:
The breakdown of an organic compound to carbon dioxide, water, the oxides or mineral salts of any other elements present, and products associated with the normal metabolic processes of microorganisms.

d) Materialisation:
The complete degradation of an organic substance to inorganic products.

These term/definitions are worth remembering throughout this paper.

(Godley 2003) stated that organic compounds can be classified either as readily degradable, moderately degradable, poorly degradable or recalcitrant depending on how easily they are decomposed. He further states that most tests of biodegradability are of limited time duration and may only measure the amount of readily and moderately biodegradable organic matter.
Rates of biodegradation may be misleading. A waste composed of a small fraction of readily degradable organic material with the remainder being composed of recalcitrant organic matter may briefly give a rapid degradation rate due to decomposition of the readily degradable fraction. This may give an apparent high biodegradation rate result when most of the organic matter is poorly or non-biodegradable.
Within the biodegradation process many intermediaries are formed. A waste composed entirely of readily biodegradable material may produce an intermediary that subsequently mineralizes very slowly. In this situation the readily degradable waste would give the impression of being partially degraded, as it will not mineralize totally within the test period even though it has been completely degraded. Godley believes that a better measurement of biodegradability is the degree of decomposition. Most methods of biodegradability monitor only for either mineralization products ($CO_2$ or $CO_2 + CH_4$) or the $O_2$ consumed which is analogous to measuring mineralization.
The rate of biodegradation will be waste dependant along with the environment in which the biodegradation is taking place. Waste characterization will give an indication as to what may or may not readily biodegrade, it cannot give a definitive time scale. Waste characterization is useful when committing waste to landfill especially the potential environmental impact of the biodegradation by products. Organic matter in material going to landfill can lead to instability due to gas formation and settling. (Sloot 2003) Dissolved organic matter can mobilize both metals and organic micro pollutants with potential hazardous consequences to animal and plant life. Methods exist to determine the mineralization of organic chemicals in soils for example, (ISO 14239:1997). A similar method has been proposed (XP U 44-163) to determine potentially mineralizable carbon and nitrogen in soil improver or growing media.

In these methods soil is mixed with the test sample and the rate of respiration recorded. The draft standard does not include this step but it could be incorporated if considered beneficial to the safe disposal of waste.

## 1.1 Stability

What is meant by "Stability"? Is it a state of no change or stage in the biodegradation process? Many methods purport to determine *stability* yet none in fact determine "stability" as defined in an English dictionary.
For example: -

The Oxford English Dictionary

"Of a chemical compound or combination: capacity to resist decomposition or disruption"

or

Webster's Unabridged Dictionary of the English Language (2001)

*Chem.* resistance or the degree or resistance to change, esp. sudden change or deterioration.

Of the papers researched the greater majority refer to the respiration rate or the biological activity to define the degree or stage of decomposition.
Many workers have attempted to define "Stability", some of which are listed below: -

"The level of activity of the microbial mass" (Butler *et al* 2001)

"A stage or state of organic matter decomposition during composting which is related to the type of organic compounds remaining and the resultant biological activity in the material" (Californian Compost Quality Council 2001)

"The degree of biological decomposition that composting feedstock's have achieved" (The Composting Association 2001)

"The point at which the rate of oxygen consumption is reduced so that anaerobic or odorous conditions are not produced to the extent that they cause problems with storage and end use of the product" (Haug 1993)

"The point where readily degradable substrate is diminished so that its decomposition rate does not control the overall rate of decomposition" (McAdams and White 1996)

"The actual point reached in the biodegradation process; the degree of decomposition, that is, the extent to which the composting reaction has advanced" (Stentiford 2000)

'Stabilisation' means the reduction of the decomposition properties of Biowaste to such an extent that offensive odours are minimal and that either Respiration Activity after four days ($AT_4$) is below 10 mg $O_2$/g dm or the Dynamic Respiration Index is below 1,000 mg $O_2$/kg VS/h (CEC 2001)
"Stable, stabilized, stability – degree of biodegradation at which the rate of biological activity under conditions favourable for aerobic biodegradation has slowed and microbial respiration will not resurge under altered conditions, such as manipulation of moisture and oxygen levels or temperature". (PAS 100 2002)

Project HORIOZONTAL also uses the terms "Stability" and "Dynamic respiration Index".

Workers to mean the same thing often use the words "stability" and "maturity". (Nordtest Report 1998) "…the term maturity is interpreted in a wide sense, and also includes the term stability. An attempt to define maturity could be that it is a measure of the compost's readiness for use that is

related to the composting process. This readiness depends upon several factors, e.g. high degree of decomposition, low levels of phytotoxic compounds like ammonia and volatile organic acids." Maturity can be defined as the point at which the end product is stable and the process of rapid degradation is finished, or, a biodegraded product that can be used in horticultural situations with out any adverse effects.

Most published methods measure a rate of reaction for example the oxygen uptake, carbon dioxide evolution or heat emission, that being the case then the correct terminology should be ***"rate of biodegradation*** (stability/maturity)". Many workers consider that it is not possible to determine "*stability"* with a single test. The California Compost Quality Council suggest that the C/N ration must be <25 before any test is undertaken. They then suggest that two further test be carried out, $CO_2$ evolution, $O_2$ uptake or Dewar self-heating and one of the following $NH_4/NO_3$, $NH_4$, plant growth, or volatile organic acids.
Care must also be taken when using research results and transferring these results to other materials not used in the trials. (Adani et. al. 1995) used liquid dairy-cattle manure and rice hulls in their research, a far cry from composted bark or domestic refuse.

It is not correct to assume that just because a sample has ceased activity no further biodegradation will take place. When oxygen is depleted, as can occur in the interior of large compost piles or waste heaps the degradation process can be anaerobic. Under anaerobic conditions microorganisms cannot break down organic materials as quickly or as completely as under aerobic conditions. This can cause odour problems and the formation or partially oxidised compounds. The partially oxidised compounds generated by anaerobic microorganisms can be very toxic to plants.

Much of the work listed above relates to composted municipal solids or other materials intend for agriculture or horticulture. Few if any have them appear to have considered biodegradation as a means of reducing the amount of material going for landfill and the potential for further biodegradation in a landfill environment.

Perhaps one should ask "Why test for stability and when?" Are the methods intended for research tools or quality control? Quality composted biowaste intended for horticulture will have undergone a rigorous and specified composting programme. The composted material should have been left to mature by which time there should be limited biodegradation. The material will be cool so why take a sample to a laboratory to try and measure oxygen uptake, carbon dioxide release or heat generation? If there is any concern a simple temperature check on the heap with a thermocouple/probe may be all that is necessary.
A further question one should ask is what is the intended end use of the product. Stabilised sludge and soil improvers can be applied to the land at a much earlier stage of biodegradation than composted biowaste that is intended for incorporation into growing media as it requires a much higher specification. It is essential for these composts that further biodegradation is at an acceptably low rate.
Materials intended for landfill do not require a "maturity" phase and may be incorporated as soon as the biodegradation process has reached an acceptable point.

Are the tests to be used on composting sites, if so, then surely the simplest test available should be used?

The information that is really required is knowledge of any further potential biodegradation and especially so under changed conditions. Has the biodegradation stalled due to lack of moisture, nutrients etc. Will biodegradation change at a rate to cause concern in a different environment, either anaerobic or aerobic or when mixed with other products?

## 1.2 Potential biodegradability

Knowledge of the potential biodegradability is a valuable tool, especially for material intended for landfill. As written above (Godley 2003) stated that organic compounds can be classified either as readily degradable, moderately degradable, poorly degradable or recalcitrant depending on how easily they are decomposed. The question to be answered is how to determine how readily degradable a material will be in the environment is which it will be placed.
(Sloot 2003) has suggested that water-soluble organic matter is a simple and rapid procedure giving valuable information on the potential biodegradability of the material under test.
The determination of cellulose, hemi-cellulose and lignin gives further information. (Godley, 2003) states that the cost of cellulose/lignin to be £50-80 (€70-100), this is not correct. Most animal nutrition laboratories undertake this test (acid detergent fibre – ADF) for less than £10 (€15). Hemi-cellulose (ADF-NDF)_[neutral detergent fibre] can also be determined at a similar cost.
XP U 44 162 proposes a method of carbohydrate fractionation, as a measure of estimating the biological stability, included in the method is a calculation to determine the organic matter resistant to mineralization. The method has been statistically tested on bovine manure, co-composted sewage sludge and a mixture of vegetable waste and animal manure.
All the carbohydrate fractionation methods are very well established having been used in the food and animal feed stuffs industries for very many years.
Is it essential to undertake a carbohydrate fractionation? These methods were developed to give a better understanding of the animal digestion of plants. Would old-fashioned "crude fibre" (acid digestion followed by alkali digestion) be equally effective when dealing with wastes etc.? What these methods will not determine are the new biodegradable plastics, nor do or can the methods give any guidance as to the effects of mixed wastes. A waste may be stable in its own right but when mixed with another waste further biodegradation may occur.
Methods based on homogenization with water with or without alteration of the chemical status might give a guide to the potential biodegradability, however the published papers refer to "stability" and not "potential biodegradability" hence at this point in time it is not possible to give guidance to preferred technique/s.
Some laboratories are looking at the possibility of using NIR (near infra-red reflectance) to determine the degree of mineralization of wastes. Preliminary findings (private communication) would indicate that the technique has potential but could well be waste specific. Once the spectra have been established (very costly) the actual method of test is very rapid and inexpensive. NIR is also used to determine protein (nitrogen), a useful parameter for material that is either going to landfill or agricultural use.

No methods for the determination of potential biodegradability are suggested within this paper.

## 1.3 Biowaste for landfill

Quoting directly from the EU Draft Discussion Document (CEN/TC223 N426)

**"Biodegradable waste** is defined in Article 2(m) of the Landfill Directive 1999/31/EC as "waste that is capable of undergoing anaerobic or aerobic decomposition, such as food and garden waste, and paper and paperboard". For the purposes of this Working Document, biowaste is meant to be the biodegradable fraction of municipal solid waste (MSW). Depending on local conditions, food and drink habits, climate and degree of industrialization, between 30 and 40% of MSW consists of food and garden waste, and another 20 to 30% consists of paper and cardboard waste. In total, between 60 and 70% of MSW can be considered as biodegradable waste. As the quantity of MSW generated amounts to almost 200 million tonnes, it can be assumed that between 100 and 140 million tonnes of municipal biodegradable waste is produced every year in the EU-15. On average, about 65% of MSW is sent to land filling, 20% to incineration, 10% to recycling and 5% to composting."

In the whole document the word "Stability" appears only once and that is under Section 4 (positive Aspects of sludge and biowaste to soil) – and in fact refers to the stability of the soil and not the landfill site. The word "Stabilized" appears more often usually in connection with the term "well

stabilized organic matter" or "stabilized biowaste". It would appear that "stabilized biowaste " is material that has undergone a composting process. There appears to be no universally accepted definition of the term "stabilized biowaste".

(Adani, 2003-2004) has suggested that that a scientific definition of stability could be:

Biological stability determines the extent to which readily biodegradable organic matter has decomposed (Lasaridi and Stentiford, 1996). It identifies the actual point reached in the decomposition process and represents a gradation on a recognized scale of values, which thus enable comparison of the process of decomposition (Lasaridi and Stentiford, 1996).

(Binner, 2004) has suggested "generating very low amounts of gas after land filling"

### 1.3.1 Landfill Gas Generation

The microbial decomposition processes occurring in landfill sites are complex and are not yet fully understood. Biodegradable matter in wastes undergoes two forms of decomposition involving aerobic (in the presence of oxygen) and anaerobic (absence of free oxygen) microorganism.

Aerobic decomposition primarily occurs during the land filling process and after capping until all the free oxygen is consumed. In some sites there may be ingress of oxygen allowing aerobic decomposition to continue but this is highly unusual only taking place in shallow sites.

Biodegradable waste decomposes in landfills following a long ecological cycle. The decomposition produces landfill gas (methane 64% v/v, carbon dioxide 34% v/v, nitrogen 2.4% v/v plus many minor gasses some of which are sulphinated and cause noxious smells even at very low levels) (Landfill Gas 1992) and highly polluting leachate. However, the major share of the waste remains in the landfill and the nutrients are not available for plant growth. When less organic matter is land filled, less landfill gas is produced.
The biogas production depends both by the amount of organic matter present and its quality (e.g. the ease at which it will be/can be biodegraded). Much data exists (Binner 1999), (Müller 1998), (Binner 1998), (Bidlingmaier 2002), and (Adani 2004) that correlates stability indexes with residual biogas production. This correlation is linear. Therefore landfill biogas reduction can be obtained by OM reduction or its stabilization. That is also true for leachates (BOD and COD).
(Binner, 2004) said that in his laboratory tests (incubation tests 1.0 kg dry matter, wetted to water capacity, incubated at 40°C) showed 50% -70% $CH_4$ which increases up to>80% for well pretreated MPB-Wastes. Nitrogen production cannot be measured by their system because they flush the reactor with nitrogen to remove all the oxygen.

Landfill gas, which may only be partially captured, contributes considerably to the greenhouse effect. (About 30-50 % of methane is lost in atmosphere independently by the capture.) (Adani 2003-2004)

The composition of waste affects the rate, quality, and quantity of gas generated per unit mass. Initial gas composition may be derived from the more readily degradable organic matter, while some components of the waste such as heavy metals may, at least locally, inhibit gas generation. Reduction in particle size will hasten the onset of anaerobic decomposition for the more readily degradable materials. Where large volumes of high BOD leachate are produced and removed from the sites, the resultant loss of nutrients on which landfill gas evolution relies will reduce the overall quantities of gas produced from the site.

The degree of saturation of the waste and its density depends on both the void space and absorptive capacity of the waste. The greater the waste density in a landfill the higher the theoretical yield of landfill gas per unit volume of void space. Water movement within the waste is necessary to ensure adequate nutrients for the bacteria to flourish. A moist landfill site is associated with high gas generation. Incoming household waste has average moisture content of about 25%, food and garden

waste providing the highest moisture input (Landfill gas 1992). (Adani, 2004) and (Binner, 2004) both consider this figure to be low thinking that 35% - 45% is a more realistic figure however Adani does say that the moisture content in the case of OM separate collection can be of 25 %, in the other cases it is 35-45%.
Once filled the re-circulation of leachate as practiced on some sites will maintain high moisture content and provide nutrients for the bacteria and enhance the rate of degradation.

Methanogenesis will proceed optimally between pH ranges 6.5 to 8.5 and is only inhibited when the pH value is outside this range. (Landfill Gas 1992)

(Adani, 2003-2004) disagrees with the above statement and states that methane production is more complicated. He stated that a lot of data shows that by buffering pH methane is not produced due to the presence of VFA (above all under the un-dissociated form) and other not well-known molecules. Only equilibrium between microbial populations can avoid the inhibition. Biogas production follows a scheduled phases and, without inoculums can require more than one year to start (stable methanogenesis). Tests undertaken by Adani showed that only 50-70 % dm/dm of inoculums (under stable methanogenesis e.g. 60 % CH4) allow methane production. Adani (2004) says "This is true for fresh organic matter. More stable MSW can produce immediately methane due to fact that unstable methanegenesis phases are avoided (the elimination of easily degradable fraction limited the production of high amount VFA)."

(Binner, 2004) offers the following: - " We do our laboratory test (MBP- waste) without inoculums. The gas generation starts very quickly.
Long lag-phases in our tests were shown only in case of "fresh" wastes (not or very short treated) because of acidification (pH <6). In the case of stabilized sewage sludge (stabilized by adding lime, pH> 10) no degradation started over a period of 90 days. After neutralization this material by adding phosphoric acid, an inoculation is necessary. To make neutralization and inoculation easier the tests are best carried out under aqueous conditions."
Adani (2004) commented as follows "This is correct but the same Binner (such I suggested above), reported that for fresh material no biogas can be produce in the first stage and he suggested the use of the correlation biogas vs respiration activity, in order to found correct number (See Binner and Zach, 1998). Moreover the biogas test used in German (Gare test..rif) consider an high amount of inoculum.

Household waste produces acid leachate as a consequence of rapid degradation of easily biodegradable materials. Unless other waste buffers this it may be responsible for inhibiting the onset of methane production and thus giving a false picture of potential biodegradability.

### 1.3.2 Site Assessment

When a site is considered suitable for restoration it is suggested (Landfill Gas 1992) that specialist advice be sought to assess whether gas is evolving in a quantity and concentrations that may cause a hazard. One factor that is continuously evolving is the type of waste going to landfill and thus desktop studies can have only be of limited value.

Looking at the type of waste going to landfill over the last centaury. Prior to the 1914-1918 war very little rural community waste went to landfill. Town waste consisted mainly of coal ash, tins and bottles with a small amount of food waste e.g. potato peeling and the outer part of vegetables. Most other packaging would be burnt on the open fire. From 1918 to 1939 there was little change more packaging would be thrown out and some rural areas came into the refuse collection areas, but this would add very little to the organic matter content as vegetable waste would be returned to the soil as most properties had household gardens for vegetable production. Many rural areas were not on mains sewage and this waste "night soils" would also be added to the household plot. After 1945 a period of recovery up to the early 1960's saw a society beginning to enjoy the chance to "throw away" rather than having to economise by recycling and make do and mend. From the 1970's onwards there was a massive increase in the use of throwaway nappies (diapers) causing a massive

increase in organic matter content containing a rich source of bacteria! Town houses as well as the majority of rural dwellings are now virtually all gas/ electric or oil fired thus reducing the inorganic element of the waste. Gardens were very small with no chance for vegetable production thus all organic waste was now disposed of as household waste including the grass cuttings with and without the addition of herbicides.
Supermarket sales increase as did the glossy packaging of the goods produced thus again increasing the mass of waste with high organic matter content. Waste site operators consider 40 years to be sufficient to say the site is stable i.e. no further gas production but from the above synopsis how can this be concluded when the type and quantity of waste has varied so much over the last 100 years?

### 1.3.3 Stability of biowaste in landfill sites.

As has been shown above stability will be measured in terms of gas evolution namely methane and carbon dioxide. (Adani, 2003-2004) says that this can be very time consuming (20-30 days) and as such becomes very expensive to undertake. As with all biodegradation processes the lack of gas production does not mean that the material will not undergo further degradation if the conditions change. Equally it cannot be assumed that the gas being produced is from the organic matter undergoing degradation. In certain areas near coal measures high concentrations of methane have been detected in boreholes before wastes have been deposited. There have also been examples of high concentrations of naturally occurring carbon dioxide. It is therefore essential to determine the sources of gas if a suitable gas control system is to be introduced. Marshes and natural gas pipelines are other possible sources of methane in addition to landfill gas. High concentrations of carbon dioxide occur naturally at shallow depths of up to 2 metres due to microbial activity associated with the roots of many types of vegetation. At greater depths high concentrations of carbon dioxide may arise due to the action of acidic water on limestone rocks or due to microbial activity above certain sulphide containing mineral veins. There may therefore be a wide range of background concentrations of carbon dioxide in the ground around sites that need consideration.
A further consideration is that “no gas” does not mean that the material will not undergo further biodegradation should conditions change. Taking a sample and carrying out tests such as pH, moisture and gas generation following the introduction of inoculums can only verify this.

### 1.3.4 Monitoring for landfill gas

There are many ways in which gasses can be monitored, either on site or back in the laboratory. The frequency of monitoring will depend on many factors and is outside the remit of this paper. It must be stressed however that many sites produce gas even though they have supposedly only been filled with “inert” wastes. It is therefore important not to rely on information about the waste unless the site has been exceptionally good control over what is incorporated into the landfill site.
In an ideal situation monitoring should continue until the whole of the biodegradable substrate within the wastes has been consumed and this could only be determined by examination of all of the waste within the landfill – not an option!
It is suggested (Landfill Gas 1992) that monitoring should continue until the maximum concentration of flammable gas from biodegradation within the landfill remains less than 1% by volume (20% lower explosion limit [LEL]) and the concentration of carbon dioxide from biodegradation within the landfill remains less than 1.5% by volume measured in any monitoring point within the wastes over a period of 24 months and samples being taken on at least 4 separate occasions, including two occasions when atmospheric pressure was falling and was below 1,000mb.
(Adani, 2004) has used the respiration index on large mass e.g. 15-20 kg, however (Binner, 2004) urges some caution in that the gas flow may not be homogeneous throughout the whole sample. If there are preferential channels the degradation is not the same all over the tested sample.
Care must also be taken to ensure the sample does not dry out during the testing process.

Well-trained personnel must carry out any on site testing.

### 1.3.5 Sampling

If continuous monitoring required the rate of gas going through the detector **must** be constant for the results to be meaningful. It is more common for the operator to undertaker manual sampling. The gas is either drawn across the on site detector or transferred to a bag for transportation to the laboratory. It would appear that no container is perfect. (Landfill Gas 1992)

### 1.3.6 Instrumentation.

Portable instruments are best used in reconnaissance and preliminary surveys. There are many types of portable instruments available for landfill gas monitoring. The accuracy of field instruments should be confirmed periodically by taking duplicate samples for laboratory analyses using gas chromatography or similar techniques.

#### 1.3.6.1 Catalytic oxidation detectors

These instruments detect low concentrations of flammable gasses as a percentage of the L.E.L. The instrument is calibrated with a pure gas so if more than one flammable gas is present false readings will be obtained. In addition an oxygen concentration in excess of 12% by volume is also required otherwise the instrument may not respond giving false low answers.

#### 1.3.6.2 Thermal Conductivity detectors

These instruments can measure the total concentrations of all flammable gasses and carbon dioxide in the sample by comparing the thermal conductivity of the sample against an internal electronic standard representing normal atmospheric air. The sensitivity of these instruments is poor below the L.E.L. and insufficient to provide accurate results.

#### 1.3.6.3 Combined catalytic and thermal conductivity detectors

Both catalytic and thermal detectors are incorporated in the same instrument and share a common display. The gas being analysed is fed into the detectors in a constant stream and may exceed the volume of gas available for analysis – if air is inadvertently drawn in the results are null and void.

#### 1.3.6.4 Infrared analysers

This type of analyser can be used to measure specific components in landfill gas mixtures. Specific analysers are available for methane and carbon dioxide and are capable of measuring from sub ppm to 100% V/V gas. Infrared sensors are relatively pressure sensitive and will not give accurate readings where a pressure differential exists between reference and sample cell.

#### 1.3.6.5 Gas indicator tubes

A crude but simple indication of the presence of gasses. As the title says they are “indicators” and little weight should be attached to the results.

#### 1.3.6.6 Barometers and barographs

These instruments are seldom used as they cannot differentiate between gasses and are usually used in borehole or wells.

#### 1.3.6.7 Gas Chromatography

This is the most reliable way for determining specific gasses and their concentration. The instrumentation is invariable laboratory based and requires experienced operators to set up and perform accurate analyses.

As with all measuring techniques there is continuous development and other methods and techniques may be suitable.

### 1.3.7 Conclusions

There appears little point in drawing up test methods specific for land filled sites. Two gasses are evolved (methane and carbon dioxide) which can be measured in many ways. The fact that no gasses are being evolved does not categorically state that the biodegradation process has gone to completion; it may be that conditions within the landfill have changed resulting in the temporary cessation of the biodegradation process.

The Draft Discussion Document (CEN/TC223 N426) refers to "stabilised biowaste" and from this it is assumed to mean biowaste that has undergone a "composting process". Composting is biodegradation under aerobic conditions with the evolution of carbon dioxide. The proposed method for the determination of carbon dioxide can be used in a landfill situation prior to the material going for landfill. The method can be used in aerobic or anaerobic conditions since the release of carbon dioxide indicates some form of biodegradation. Under anaerobic conditions methane is also given off and would need to be measured if the total rate of biodegradation was required.

## 1.4 Sewage Sludge

The term "stability" when used with in the sewage sludge industry tends to mean a material that has undergone a digestion process according to a pre-determined Code of Practice. TC 308 found difficulty in defining "a stabilized sludge" and concluded that the definition in EN 1085 (Wastewater treatment - Vocabulary) was the best they could get namely :-
"Stabilization" Process whereby organic substances (dissolved or particulate) are converted to materials, which are either inorganic or very slowly degradable.
"Stabilized sludge" Sludge, which has been subjected to a stabilization process, thereby reducing its tendency to degrade below a specified level.
"Degree of Stabilization" Degree of degradation achievable by sludge stabilization measured e.g. by the reduction in organic material.
Raw sewage sludge has a dry mater between 3 and 6%. During the digestion stage the sludge is heated to between 35°C - 55°C for a period of time not less than 12 days and normally about 16-20 days. During this digestion stage some of the organic matter is converted to methane gas and carbon dioxide. The methane gas is collected and used to maintain the elevated temperature of 35°C. The end product is generally referred to as "Stabilised sludge." The biological reactivity of this "stabilised" sludge is much higher than the Austrian landfill ordinance allows. For landfill some more stabilisation (biodegradation) is necessary
Occasionally further tests are carried out to determine the possibility of potential odour nuisance. Some workers have suggested an enzymatic test but this is considered by the trade to be expensive and long-winded and of little use. A second test biological oxygen demand (BOD) is a well recognised test but again is considered to be time consuming and costly. A further simple test is to enclose the material in a sealed container and to check for offensive odours after 4 days. The smell should be "earthy"; if offensive odours are detected it is assumed that the sludge is not "stable". Whilst this is a subjective test most humans are very sensitive to sulphurous odours. (Binner, 2004) believes it is impossible to get an "earth" smell after 4 days and considers 20 days more appropriate including some aerobic biodegradation.

Two further definitions can be found in EN 12832 Characterisation of sludge's – Utilisation and disposal of sludge's – Vocabulary
"Chemically stabilized sludge" Sludge stabilized by a chemical process.

"Thermally stabilized sludge's" Sludge stabilized by a thermal process
Sludge cake is "stabilised sludge" that has been de watered usually by centrifugation to about 25-30% solids. De-watering is carried out to reduce transportation costs. It is best practice to apply sludge cake to land as soon as possible as stockpiled sludge cake can over a period of several weeks begin to undergo anaerobic digestion with the production of unacceptable odours.
Lime stabilised sludge is where calcium oxide (quick lime) is added to either raw sludge or sludge cake. When the raw sludge maintained at pH12 for a period of not less than 2 hrs virtually all the pathogens are killed including salmonella and e-coli. E-coli is reduced by a factor log 6 i.e. a figure of 1 million is reduced to 1.
If the pH is maintained at 10+ then there will be no re-growth of bacteria and hence no odours. However the lime quickly absorbs carbon dioxide from the air to from calcium carbonate (chalk) with a much lower pH and the possibility of anaerobic activity taking place. (Anne Bøen, 2004) reported that lime stabilised sludge is time limited as the pH will reduce with time, by the time pH drops the microbial activity has increased and the sludge should have been applied to the land. It is normal practice to apply lime-stabilised sludge to land as soon as possible. In oxygen rich atmospheres there should be no offensive odours. In some Countries, particularly in the northern hemispheres, sludge can only be applied during the summer months, giving rise to the practical problem of storage for several months.

(Anne Bøen, 2004) further stated that maintenance of high pH after the addition of calcium oxide is necessary, but the time the sludge is maintained at a high pH (>11) is more relevant as a measure of stability. In a Norwegian study on stability parameters in sludge (Paulsrud *et al* 2003), different amounts of calcium oxide were added. The pH rose to more than 12 with all levels of addition, but the length of time with a high pH varied. Increased addition of calcium oxide and the time with pH>11 showed a positive correlation up to a certain addition of lime.
Time with pH >11 is not relevant as an operational measuring method for sludge, but it is relevant taking into consideration when optimising the calcium oxide addition in a sludge treatment plant.

Limed cake is normally carried out in large establishments. The lower water content of the sludge means that on the addition of the lime the temperature can rise to 55°C - 60°C thus enhancing the pathogenic kill.

Dried sludge i.e. that containing >95% solids is considered very stable, as there will be no bacterial activity. If the moisture level increase to >10% then fungi will develop and if the moisture content > 20% there is every likelihood of bacterial growth and subsequent odour generation.

### 1.4.1 Conclusion

There appears to be no definitive methods to determine "stability" or stabilized sludge".
In conducting this research paper it has become apparent that within the sewage industry very little actual testing takes place once the process has been developed and is up and running. It would appear that adherence to good practice as defined in various Directives is all that is required with an occasional test. Any tests that are carried out are simple to operate i.e. organic matter by loss on ignition or dichromate oxidation. Dry matter by an equally simple oven drying process. BOD may be of interest, the method being well documented. For lime treated sludge pH could be a factor and again many methods exist.

Other HORIZONTAL project leaders are covering, pH, dry matter and organic matter.

## 1.5 Soil Stability

Soil stability as a physical parameter is related to the particle size, organic matter and water content of the soil. Knowledge of soil stability is important when considering land drainage, landslip and for structural engineering purposes.

For agricultural purposes, specifically land drainage, a number of techniques exist for assessing the structural stability of soil aggregates. These techniques fall into three groups, firstly those assessing the size and number of aggregates remaining after the application of mild slaking forces, (Emerson 1967) secondly those measuring the quantity of silt and clay dispersed in water as a result of mild applied forces (ISO 11277:1998) and thirdly those measuring the difference in water release curves between collapsed and normal soil aggregates. (Haines 1930)

The water retention characteristics of soils are covered in (ISO 11274:1998). With reference to this standard "…Care should be taken not to leave sandy soils wetting for too long because their structure may collapse. Low-density subsoil sands without the stabilizing influence of organic matter or roots are most susceptible."

Methods of tests for soils for civil engineering purposes can be found within BS 1377. Geotechnical methods of test are also available e.g. ISO 22476 (2002) and (NEN 6745-1 2002).

Soil stability as a physical parameter is considered to be outside the remit of this report.

Soil organic matter is decomposed by micro flora. Methods exist to measure abundance and activity of micro flora (ISO 17155:2002), whilst (ISO 16072:2002) offers a wide range of tests to determine microbial soil respiration. Both static and dynamic systems are included with a wide range of testing procedures. Virtually all the procedures measure carbon dioxide evolution and by deduction oxygen uptake. Care must be taken when doing direct mathematical conversions of carbon dioxide release to oxygen uptake and vice versa as the two are not necessarily the same. Oxygen will be used in the conversion of metals to metal oxides plus the formation of sulphur and nitrogen oxides. Carbon dioxide may be released if carbonates are present and may also be released in anaerobic conditions.

# 2. EXISTING METHODOLOGY

Four methods are commonly used to determine the rate of biodegradation, namely carbon dioxide release, oxygen uptake, the evolution of heat and the Solvita® test kit.

## 2.1 Carbon Dioxide Release

This method is well established and has been in use for very many years.
Carbon dioxide is released after carbon has been oxidized during the composting/biodegradation process. There is a small possibility of carbon dioxide being formed under anaerobic conditions but this should not be the case in most composting situations where aerobic conditions prevail. Mineral carbonates may also release carbon dioxide under acidic conditions a situation that is extremely unlikely to occur in a composting situation.
Two methods for the measurement of carbon dioxide are in current usage, a static system (Project 99-PUM-3, 2002) and a dynamic or flow system (ISO 14852:1999), (ISO 14855:1999), (Hue and Liu 1995) and (WRAP 2003). (ISO 16072:2002) describes both static and dynamic systems.
The static system tends to be limited to a lower sample size (25g) whilst with the dynamic system, 100g is used (WRAP 2003) and 600g in (ISO 14855:1999). Sample size is discussed below.
Proponents of the dynamic system have raised the question of oxygen depletion in a static system; to date there appears to be no experimental confirmation.

The Convenor of ISO/TC61/SC5/WG22, supplied the following:

"The reasoning why a flow system was used (*ISO 14852:1999), (ISO 14855:1999*) instead of a static system is because it's necessary to remove evolved carbon dioxide and water from test vessels by using a flow system. In addition, continued absorption of the evolved carbon dioxide in carbon dioxide traps will be ensured by supplying air at a flow rate between 50 ml/min and 100 ml/min."

The sample under test must be of sufficient moisture content to enable the biodegradation process to proceed, if the sample is considered to be too dry then water is added prior to the test being undertaken. How much water should be added and how long after the addition of water should the sample be left until the test is undertaken needs to be defined.

The temperature at which the test take place is also under debate, (ISO 16072: 2002) use 22°C, (WRAP: 2003) suggests a temperature of 25°C while (Project 99-PUM-3, 2002) recommend 35°C. It would appear to be obvious that at a higher temperature the reaction will be more vigorous but is this necessary?

How long should the test be undertaken? Increased activity after a sample has been rewetted has been noted and recorded – papers referring to this issue probably run into hundreds (Ed Stentiford private communication). Work carried out by (WRAP: 2003) would indicate that a test of up to 7 days might be necessary before a steady state is obtained.

### 2.1.1 Conclusions

The method is well established. (ISO methods exist)

A wide range of un-amended sample sizes can be taken for test.
The dynamic system is recommended.
The system is simple to operate, inexpensive to set up and does not require highly skilled personnel to undertake the tests.
The method can be adapted to electronic sensors and continuous monitoring
The addition of moisture, best temperature and duration of test need to be clarified by experimentation.

## 2.2 Oxygen uptake

There are at least two variations in the method of oxygen uptake; one is based on the sludge biological oxygen demand method and the other on the sample as received using gas sensors.

The sludge's and other aqueous wastes biological oxygen demand methods have been modified and adapted for other materials such as composts. The sample is either analysed "as received" as is the case for sludge's or a small sample is homogenized with water to give a total solids value of about 2%. (Standard Oxygen Uptake Rate [SOUR]) In some instances a buffer solution containing magnesium sulphate, ferric chloride, calcium chloride and potassium phosphate is added. A dissolved oxygen probe or a respirometer is used to record oxygen uptake.

Quoting from (Project 99-PUM-3, 2002) "Several shortcomings associated with the test protocol were noted, including issues associated with both conducting the test and calculating SOUR values. At present, the SOUR test is limited to aerobically digested sludge with a concentration of 2% solids or less which is collected in a 10°C to 30°C temperature range. In addition, testing should occur not more that 1 hr after taking the sample…Some data collected to date offer encouragement to extending the use of the SOUR test. However a further systematic study is needed before this can be recommended."

(Morten Carlsbaek 2003) says that the SOUR method is well suited for product description (i.e. compost ready for maturation versus compost ready for growth media mixtures).

The OxiTop® measuring system measures the oxygen depletion as a gas pressure drop in an enclosed vessel containing a homogenized sample of no more than 3 g volatile solids. Oxygen is released into the vessel to replace consumed oxygen. Carbon dioxide is removed by alkali. It is possible to take continuous readings. The test is normally carried out over 7 days.

(Adani, 2003) has proposed an oxygen uptake method that is capable of handling samples up from grams to 50 L) i.e. dynamic condition do not depend on the vessel size. The maximum water holding capacity of the sample is determined. The sample under test is then adjusted to 75% of this figure by the addition of water. The sample is place in a continuous flow adiabatic respirometer with the measurement of oxygen at the inlet and outlet, the change being the oxygen uptake. From the literature research it would appear that no other worker/reviewer has considered this method of standardising the addition of water. This method is used to determine the Dynamic Respiration Index.

The ASTM method (D 5975 – 96) is quite complex and requires a laboratory prepared stabilized compost inoculum. The test is carried out in a temperature controlled envioronment capable of holding at least nine composting vessels each of 2 to 5 L volume. The gasses evolved are measured by gas chromatography. As stated earlier this method is complex and it is unlikely that many laboratories would be capable of undertaking the test.

### 2.2.1 Conclusions

Henrik Lystand (Norstead report 1998) states, "The results attained by the $O_2$ uptake method provide useful information about the degree of decomposition. However, the complexity of other analyses and higher cost means that the test is mainly restricted to scientific use and in larger laboratories".
A small sample is taken and is amended which may have an influence on the data obtained. (Stentiford 2002) stated that the SOUR method was well suited as a diagnostic tool for process control and performance evaluation but less suited for product description (i.e. compost ready for maturation versus compost ready for growth media mixtures.
Moisture content of the sample is not a determining factor in the SOUR test.
No one appears to consider that a sample may have ceased working because of external factors such as drying out and that it may start to work again when re-wetted. It appears that the approach is that the sample is suspended in water so that any re-activation will automatically take place, a conclusion that has not; it would appear, to have been verified experimentally.

It is suggested that this method be used for sludge's and sample with dry solids of less than 5%.

The method of (Adani 2003) and ASTM (1996) have interesting possibilities and should be considered for further development and used in conjunction with the carbon dioxide release method.

## 2.3 Evolution of heat (Dewar)

Techniques have been developed to measure the heat evolved during the composting process. The sample is placed in a heat-retaining flask (Dewar or similar) and the heat evolved is measured over a period of up to 10 days either by a thermocouple or thermometer. The compaction of the sample into the flask has a considerable influence on the result (Weppen 2002). The position of the thermocouple or equivalent will also influence the result. Some papers suggest placing the thermocouple within the compost (Nordtest report 1998) whilst others place the thermometer in the water jacket surrounding the Dewar flask. It is to be expected that precise control on the exact positioning of the thermocouple would be required to obtain reproducible results. The upper third (Nordtest report 1998) and 2 inches [50mm] from the bottom in the (Bord na Mona method). Dr G Becker states in her dissertation that the thermometer must in the lower third within the compost, in the upper third cooling effects might happen. It is very difficult to give precise instructions for compaction of the sample into the Dewar flask. Particle size will be a considerable factor for variation on compaction in the Dewar flask and hence increasing the chances for variable results.
According to Dr Becker the moisture content is critical and that to ensure proper contact between sample and thermometer the sample must be sieved to less than 10mm.
(Brinton, 2000) 'The Dewar test is limited in the sense that it best distinguishes very immature from mature compost; it can not distinguish moderate maturity from high maturity which may be important for potting mix use of composts'.

### 2.3.1 Conclusions

It is unlikely that the Dewar flask method will give any meaningful results during the latter stages of composting and maturation.
(Nordtest report 1998) concludes that the method is cheap and simple to set up (assuming no continuous monitoring take place when costs can escalate). They go on to say 'Since the method does not reveal maturity effects that are not related to the degree of degradation, the test should not be used alone to determine compost maturity'
(Weppen 2002) found that in trials the method was not reproducible.
In view of the comments above one must conclude that a method that is limited to only certain phases in the composting process cannot be proposed as general standard. A standard method must be capable of working over a wide spectrum of samples, sample sizes and stages biodegradation. The test may take up to 10 days to complete, a time span that may be considered to long for many operations.

## 2.4 Solvita Testâ

The Solvita Test appears to be the only commercially available field test kit. Quoting from the Solvita literature “Solvita is based on a newly developed and patented gel – colorimetric technology in which respiration gases from composts are captured and accurately indicated in a colour-coded system calibrated to a wide range of known conditions.”

A sample of compost is checked for moisture content by hand squeezing, the sample should feel wet but not exude water. The sample is brought to the required moister content either by drying or the addition of water. For dry samples that have been moistened it may necessary to leave the sample for 48hr to equilibrate. The test jar is loaded with compost to a prescribed limit, followed by the two test paddles being inserted into the compost. The lid is secured and the jar left at room temperature for 4 hrs. The test paddles are removed and the colour changes matched against colour charts.

### 2.4.1 Conclusions

(Nordtest report 1998) found that a comparison of the Solvita test procedure against the Dewar flask and $CO_2$ methods were consistent at both ends of the scale, however for middle levels variation was noticeable especially to the Dewar method.

The kit is easy to use. Correct moisture content is essential for the test to work. (Nordtest report) conclude that the kit represents an interesting approach in reducing time and costs and that with some reservations they believe the kit can become a valuable tool for a composting facility.

It is unlikely that any “commercially available test kit” will become a “Standard Method.” The kit appears to have possibilities and could well be used as a field test with an appropriate laboratory based Standard Method being used to verify any questionable results

(WRAP 2003) have on occasions been unable to obtain comparable results between their carbon dioxide method and the Solvita test method. Adani et al. (2003), did not obtain good result by comparing dynamic test and Solvita, due to the impossibility to determine “a priori” lag phase for Solvita.

# 3. EVALUATION OF DRAFTING A HORIZONTAL STANDARD

## 3.1 Comparison of methods.

The four basic methods are compared in the following table.

| | Carbon Dioxide release | Oxygen uptake | Oxygen uptake<br>Adani method | Dewar<br>Evolution of heat | Solvita |
|---|---|---|---|---|---|
| Basic method | Evolved $CO_2$ is measured from basic titration to continuous electronic monitoring. | Sample is homogenised and the oxygen uptake measure by oxygen probe. | Oxygen content monitored entering and leaving the reaction flask. | Sample is placed in Dewar flask and temperature changes recorded over several days (thermometer or probe) | $CO_2$ and $NH_4$ measured within 4 hrs by change in colour on indicator strips |
| Existing Standards | ISO 14852:1999<br>ISO 14855:1999 | None | ASTM (1996) very similar | No international standards | None |
| Sample size | Normally up to 1kg but can be as large as available sample vessel (5L have been used) | 10-20 g | Up to 50 L possible | Enough sample to fill a 1L flask | Sample jar filled to specified line (10-20 g) |
| Pre-treatment | None needed but when small samples are taken (less than 100g) advisable to pass a 10-15mm screen | Sample is sieved to pass 10 mm, large particles are removed | None needed | Sample is sieved to pass 10 mm, large particles are removed | Sample is sieved to pass 10 mm, large particles are removed |
| Moisture content | Has to be adjusted to between 40 –60 %. This figure has yet to be confirmed | Not applicable as sample is homogenised in water. Final mix 2% solids. | Moisture adjusted to 70% water holding capacity | Has to be adjusted to between 40–60 %. This figure has yet to be confirmed | Hand squeeze to asses moisture content – adjust as required. |
| Stabilisation after addition of water | Test can be run as soon as water added – 4 days have been suggested but this requires confirmation | None | Test can be run immediately | Suggested 4 days | Up to 48 hrs suggested for very dry samples |
| Temperature control | An elevated stabilised temperature | An elevated stabilised temperature | No temperature control | No temperature control | |
| Time for test | Continuous – evolution of gas plotted against time 4-6 days appear to give | Can be over several days – results are continuously plotted. | Continuous – plot of oxygen consumption over 4-6 days. Typical time required 2-3 | Recommended up to 10 days – maximum temperature reached is recorded. | Actual test less than 2 hrs. |

| | | | | | |
|---|---|---|---|---|---|
| | satisfactory results | | days | | |
| Versatility (Samples) | Can be used on a wide range of sample size type including sludge's and materials going for land fill | Can be used on sludge's and fine particulate materials | Can be used on a wide range of sample size type including sludges and materials going for land fill | Can only be used on fine materials | Can only be used on fine materials |
| Versatility (Equipment) | Very easy to set up in its simplest form (air pump, flasks for samples, water bath, gas traps and burette) to full electronic monitoring with $CO_2$ sensors | Requires oxygen probe, homogeniser, water bath and electronic recording | Flow regulator and electronic monitoring required. | Very simple with just a Dewar flask and thermometer. Continuous electronic recording with a probe usual format | Commercial equipment |
| Static or dynamic process | Dynamic | Not applicable. In some cases a static system | Dynamic | Static | Static |
| Potential errors | In acid conditions it is possible to get breakdown of carbonates but under normal composting the sample is always in an alkaline situation | Sample size – very small giving rise to potential sampling errors. As the whole sample is homogenised creates an unnatural situation – more like a measurement of ultimate bio-degradability than current situation. | Loss of oxygen is not necessarily all from a biodegradation process. | Sample size and elimination of larger particles. Placement of thermometer (thermocouple) critical. Method best used in an active composting situation. | Sample size.<br>Short duration – after sample has been sieved etc. has the sample equilibrated? |
| Potential for external calibration | Yes – $CO_2$ can be released from a standard carbonate solution and trapped | Not easily – no method has been found | None identified | Probes and thermometers can be calibrated | No |

Various aspects highlighted in the above table are now discussed in greater detail.

## 3.2 Sample type and size

The range of samples received into the laboratory will vary from finely ground material as will be found in sewage sledges to very coarse materials for example composted bark. Within the UK, composters are preparing products with a sample size range from 6mm up to 65mm. For landfill the particle size could be even greater. It would be advantageous if the recommended method were to be able to cope with this wide range of particle size. Most oxygen uptake methods use fine material (i.e. will pass a 10mm sieve) the sieved sample being blended with water. The sieving removes coarse materials and the blending changes the matrix of the sample. The surface area is radically altered, a factor to be considered when "hard" or "woody" materials are being composted. The proposed standard is written for particle size up to 20 mm but can be adapted to take any particle size.
The sample weight/volume taken for test varies from 5g –20g total solids for the SOUR method. The Dewar self-heating method depends upon the size of flask being used, 1 to 2 liters appears to be the norm. The Dewar flask is filled with sieved material and gently tapped. Compaction will depend on the material being tested and fractionation that may occur with some samples especially those that vary greatly in particle size and contain a significant amount of fine material.
Carbon dioxide methods can be adjusted to accommodate almost any sample size, Adani (2003) uses up to 50l sample. It would appear that no work has been undertaken on carbon dioxide release with slurry samples, however if carbon dioxide is evolved there appears to be no reason why the proposed standard method should not work. Materials going to landfill contain a high percentage of plastics, some modern plastics are designed to be biodegradable and these must also be taken into consideration when designing a method.

## 3.3 Moisture content

Biological activity slows down in a dry environment.
The Dewar, Solvita®, carbon dioxide and some oxygen uptake methods all recommend a moist sample. Words like "visual", "feel", and "slightly damp" etc. are all very subjective.
(WRAP 2003) suggested a figure of 40-60%; in (Project 99-PUM-3 2002) the figure is 50% ± 2%. Whilst these figures may be obtainable for some compost they may be outside the range of others. It is highly unlikely that coarse woody composts will fall into this category. (Evans 2003) has suggested field capacity. (Adani 2003) recommends a figure of 75% of water holding capacity. (Itävaara et al 2002) used 80% water holding capacity.
How is a sample to be "wetted"? For normal moist samples the simple addition of water in small quantities should be satisfactory, for samples that have dried out the task is considerably harder.
Any proposed standard method should specify a reproducible procedure to obtain the required moisture level for the type of material under test.
It was suggested at CEN TC 223 (Soil Improvers and Growing Media) meeting MILAN 2003 that it may be possible to produce a reference table of acceptable moisture contents for a range of materials. Adani (2004) believe this to be impossible and quite unnecessary.
A possible method would be to determine the water content after leaching (Kreij et al., 2001). This would be determined on a sub-sample of the material under test. This will always give the water content that is most appropriate. The volume of water is gravimetrically determined by saturating the material and letting it leach out on a grid. The sample is dried in an oven at 105°C to determine the amount of water, after which the water content can be calculated.

The SOUR method is not affected by sample moisture as the test is carried out in an aqueous suspension, however as has been mentioned above the SOUR test is undertaken as soon as the sample is homogenized thus not allowing for any re-activation of a dormant composting process.
The Adani method (2003) appears to be the best method as it can be applied to all samples (excluding sewage sludge).

## 3.4 Pre-incubation

The purpose of the pre-incubation period is to ensure that the indigenous microorganisms are acclimatized to the mesophilic environment in which the test is conducted. All carbon dioxide

release and Dewar flask methods refer to incubation of (composted materials) prior to the test being undertaken. The time and temperature of this incubation is fairly constant, 20°C for 3 days (Project 99-PUM-3, 2002), (WRAP 2003) 25°C for 3 days. (WRAP 2003) found a 40% variation in results for sample that had not been incubated compared to those with a 2-day incubation period. There appears to be very little research carried out on samples that for some reason have ceased working, but are capable or restarting under favorable conditions. (Itävaara 2002) "attaining optimal water content and activating microbial degradation may take several days if the compost has been completely inactivated and dried out. This should be taken into consideration when determining stability, i.e. respiration activity of the microbes, and is connected to the availability of easily utilizable nutrients". Manufacturers of peat-based products that incorporate composted materials claim that reactivation is not an uncommon occurrence (Waller 2003). It has been suggested that the high levels of nitrogen in the commercial products has initiated the restart of the composting process. What is not clear is how mature the composted material was prior to use. Is it possible that the microorganisms are in fact reacting to the peat, which is a ready source of carbonaceous material? This is an area that will require further investigation.
(Adani 2003) appears to show that maximum activity occurs within 40-45hours after setting up the equipment. It is not known if this time scale for maximum activity is the same for all sample types. (WRAP 2003) would appear to show that the surge of activity may take place over a period of up to 5 days. Maximum activity depends on the lag-phase and that depends by many factors. That is why Adani et al., (2004) and above all ASTM (1996), indicate 4 observation days. Nevertheless typically maximum activity occurs within 24-36 hours.

## 3.5 Temperature

It is interesting to note that some methods do not define the temperature at which the test should be undertaken whilst other do. The temperature ranges from 20° C - 30° C for pre-incubation and from 25° C to 37° C for the respiration tests. (WRAP 2003) found a variation of 121% between 20° C and 25° C for the respiration test. It is highly likely that similar variations will occur between other temperatures. The (WRAP 2003) works suggests that tight control on temperature is required. For soils the temperature is much lower, 22° C. It is possible that workers have considered the natural environment of the material under test to be the appropriate temperature for test purposes. When looking for long-term stability e.g. landfill then temperature of the test may be significant – do different flora act at different temperatures? Adani (2004) said "Self-heating temperature (adiabatic approach) can solve this dilemma: under optimal condition (O2, moisture and nutrient) micro-flora will decide temperature".

## 3.6 Nutrient status

This is an area that has received very little comment or research. The topic was raised at TC223 Soil Improvers and Growing Media meeting in Milan 2003. If there is insufficient nitrogen in the initial sample should further nitrogen be added? Comments the writer has received indicate that there were ample nutrients available especially so with MSW but without analytical data can this assumption be right on every occasion and certainly not so with materials intended for landfill Ancient tradition recommended the addition of urine to the compost heap to maintain microbial activity. (ISO 14855:1999) uses inoculums of solid municipal waste compost that is between 2 – 4 months old. Admittedly the ISO method is deterring the ultimate biodegradability of plastics. Materials that have a high carbon low nitrogen status e.g. paper waste require the addition of large amounts of nitrogen to achieve complete biodegradation. If this low nitrogen situation exists in the field then a similar situation would also exist in the laboratory.
To obtain the potential respiration rate inoculants have been added, (Microbiology of Composting 2002; Soil Control Laboratory CCQC (2001)) "Before a three day incubation at 37° C, the sample receives additions of Hoagland's nutrient solution and mesophilic microbial inoculants to remove any biological limitation. This method attempts to remove nutrient and microbial limitations may

successfully overcome the limitations due to anaerobic conditions, samples from thermophilic zones or heat damage."
Adani (2004) recommends adjustment of the C:N ratio to below 35. In a landfill situation is it likely that this will occur either naturally or artificially? When composted materials that are mixed with fertilized peat's etc. there is the potential for further biodegradation.

N-immobilization is well understood and occurs when soil micro organisms are triggered to consume soluble nitrogen to assist in their metabolism of carbon, a situation that arises when immature composted material is applied to land.
Should samples are brought to a minimum nitrogen level before testing and if so in what form should the nitrogen be added and what should that figure be?

## 3.7 Interpretation

Nearly all the methods take relatively small samples, and are run over a short time scale. The results obtained can only give a snapshot of what is happening over that time scale and perhaps give an indication as to the long term "stability" of the product. What can be determined is the rate of activity at the time of testing. From this figure it may be possible to determine the time when activity will have slowed, or has slowed to such a point as to make the end product useable.
Of the numerous papers published on the topic, graphically illustrated in (Project 99-PUM-3, 2002), (WRAP 2003) and (Nordtest report 1998), only a few research papers appear to draw conclusions as to the meaning of their results.

(Hue and Liu 1995) recommended 120 mg $CO_2$/kg dry matter/hr as the cut off point for stability.

The 2nd of the EU working document on Biological Treatment of Biowaste (2001) suggested that either the Respiration Activity after four days (AT4) is below 10 mg $O_2$/g dm or the Dynamic respiration Index is below 1000mg $O_2$/kg VS/h.

Listed below are tables of interpretation from various workers.

**Table 1**

WRAP 2002 – Review of Compost Standards in Denmark

| Method | Not Ready | Fresh | Stable | Very Stable |
|---|---|---|---|---|
| Solvita | 1 | 2 – 3 | 4 – 5 | 6 – 8 |
| Self Heating (max. temp. in °C) | >60 | 60 – 40.1 | 40 – 30.1 | <30 |

**Table 2**

Brinton 2000 – Compost Quality Standards & Guidelines

| Method | Units rating | | |
|---|---|---|---|
| | Very Mature | Mature | Immature |
| Oxygen Uptake $O_2$/VS/hr | <0.5 | 0.5 – 1.5 | >1.5 |
| $CO_2$C /unit VS / day | <2 | 2 – 8 | >8 |
| Dewar max Temp. rise °C | <10 | 10 – 20 | >20 |
| **Table 2** continued | | | |
| Solvita Index value | 7 – 8 | 5 – 6 | <5 |

**Table 3**

Nordtest report 1998

| Rise in temperature (°C) | Degree of decomposition | Description |
|---|---|---|
| >40 | I | Raw refuse, very slight decomposition |
| 30-40 | II | Slight decomposition |
| 20-30 | III | Medium decomposition |
| 10-20 | IV | Good decomposition |
| 0-10 | V | Decomposition advanced or completely finished |

**Table 4**

Project 99-PUM-3, (2002) produced a table relating milligrams $CO_2$-C per gram organic matter (volatile solids) per day to define various stages of compost stability.

| Respiration rate (mg $CO_2$-C/g Volatile solids per day) | Rating | Characteristics |
|---|---|---|
| <1 | Very stable | - Well cured<br>- No odours<br>- No continued decomposition |
| 1 – 3 | Stable | - Cured compost<br>- Limited door potential<br>- Minimal impact on soil carbon and nitrogen dynamics |
| 3 – 6 | Moderately stable | - Uncured compost<br>- Minimal door production<br>- Addition to soil may result in nitrogen immobilization<br>- High phytotoxicity potential<br>- Not recommended for growing plants from seeds |
| 6 –11 | Unstable compost | - Very immature compost<br>- High door and phytotoxicity potential<br>- Not recommended for growing plants from seeds |
| >11 | Un-stabilized material | - Extremely unstable material<br>- Very high odour and phytotoxicity potential<br>- Not recommended for use |

**Table 5 Adani**

| **Biomass typology** | **PDRI** | **Self Hating test** |
|---|---|---|
| Compost (or waste) at medium stability | $\leq$ 1000 mg $O_2$ kg $VS^{-1}$ $h^{-1}$ | Between III and IV |
| Compost (or waste) stable | $\leq$ 500mg $O_2$ kg $VS^{-1}$ $h^{-1}$ | IV |

As can be seen there are differences in interpretation especially in the Dewar test results. In one paper (WRAP 2002) the maximum temperature is noted whilst (Brinton 2000) and (Nordtest 1998) record actual temperature increases.
(Brinton 2000) states a temperature increase of >20 indicate immature compost whilst Nordtest records medium to good decomposition for such a temperature change.

There is an obvious need for standardization – it is possible that the results above are in fact influenced by the actual method used and that it is not possible to correlate results from one method or worker to another.

What would be an appropriate unit for landfill purposes?

# 4. CRITICAL POINTS AND RECOMMENDATIONS

No one method is capable of giving a definitive answer to the question "Is this product stable?" The first problem to be resolved is what is meant by the term "stability". To this must be added the question should one look to the long-term potential biodegradability as a guide to the potential rate of biodegradation?
Many tests are available, some simple to operate, others requiring quite sophisticated and expensive apparatus. Some workers hold views that more than one test may be required. A problem of potential methane production from landfill sites exists. If the potential biodegradability of the material were known then perhaps remedial action could be taken prior to land filling. Addition of an inoculums and or nutrients to the sample under test within the proposed method may give an answer to this problem.

## 4.1 Terminology

The term "stability" is misleading, it is suggested that this term is no longer used. What is being measured is the **rate of biodegradation** of the material under test. **Potential biodegradability** is a separate determination requiring its own standard. It may become necessary to determine both the potential and rate of biodegradation in some situations.

## 4.2 Methods of test

There is a need for methods of test to determine the potential biodegradability and the current rate of biodegradation. The rate of biodegradation, under ideal conditions will give a very good indication as to what is happening in a composting situation. It would be advantageous if it were possible to predict the time required for the material under test to reach a specified rate of biodegradation. This may be possible knowing the organic structure of the materials and the environment in which the material will be used i.e. landfill or agricultural/horticultural use.

Obtaining data is, in some respects easy. It is essential that resources be directed to the obtaining of relevant data and to the understanding of that data once it has been obtained. Looking at carbohydrate fractions before composting and/or land filling must be related to what actually happens in real life situations. The rate of biodegradation will be dependent on many factors moisture, oxygen and nitrogen availability being three factors having considerable influence on the end result.

It is recommended that a European funded working party be set up composed of people from all aspects of the waste and recycling industries to determine and agree what is required. Any proposed method(s) must be based on scientific reasoning and not on the wishes of some researcher wanting to have his/her method as the European standard.

In an ideal world one would have at least three or more methods of test to determine the rate of biodegradation and potential biodegradability. These would include an oxygen uptake, carbon dioxide release and chemical composition of the sample. It is obviously not possible to go down this path and a technique has to be chosen. The carbon dioxide method is believed to be the simplest and cheapest to set up and run; it is also the only method that has ISO parentage.

## 4.3 Possible Alternative Procedure

It has been suggested that a simple test would be to add a solution of ammonium nitrate to a sample leave for a specified time and then determine the residual ammonia and nitrate content. If there is no loss of ammonia or nitrate the sample is "stable". To date the author of this paper has been

unable to find any references to this procedure in the literature. The method is simple and easy to undertake. It could be applied to any sample size without any amelioration of the sample. It is suggested that further work be undertaken to validate the usefulness of this method

## 4.4 Criteria for test methods

4.4.1 The test methods should be robust, repeatable, when ever possible be simple to operate.

4.4.2 The test methods should be able to accommodate a wide range of sample types (The proposed method as written and tested is not applicable to samples with more than 10% > 20 mm but it could be adapted to take much larger sample volumes.)

4.4.3 The cost of the method and apparatus used must be taken into account.

4.4.4 The method must be rugged.

4.4.5 Fresh sample should be used.

4.4.6 Where possible the sample should not sieved prior to analysis.

## 4.5 Method Recommendations (Rate of biodegradation)

4.5.1 A titrimetric method based on dynamic carbon dioxide evolution

4.5.2 If gas sensors are used then either carbon dioxide or oxygen uptake or both can be determined.

## 4.6 Additional parameters,

These parameters may be considered for both landfill and or composted materials.

4.6.1 Ammonia nitrogen and nitrate nitrogen

4.6.2 Total carbon by loss on ignition or dichromate oxidation

4.6.3 The carbon nitrogen ratio C/N

4.6.4 Carbohydrate fractionation e.g. ADF, NDF

## 4.7 Method Development (Rate of biodegradation)

4.7.1 The optimum moisture content

4.7.2 The optimum temperature for pre incubation

4.7.3 Time for pre-incubation

4.7.4 The optimum temperature to run the test

4.7.5 Nutrient status

4.7.6 Larger particle sizes

4.7.7 Suitability of the method to determine the potential biodegradability of materials going to land fill.

## 4.8 Conclusion

It is essential to have a method that will be able to confirm if a sample is "stalled" and has not reached its full potential biodegradability.

No one method would appear to be applicable to all aspects, materials and stages of the biodegradation process. Field test kits such as "Solvita" may have a role to play.

The suggested method of ammonium nitrate addition requires investigation.

Should the standard method be capable of giving an indication of the rate of biodegradation e.g. self heating, or should the standard method be versatile enough to cover the whole range eventualities?

Should the Standard method be suitable for only well equipped laboratories or should it be capable of being used in any laboratory environment. It must be remembered that the cost of analysis could have a significant impact on production costs.

NIR offers the only hope that a single method of test will be able to cover all aspects of biodegradation, without further research and funding the method will be a long time coming.

The proposed carbon dioxide release method is versatile, cost effective and simple to operate.

With so many methods of test available and in use it is highly unlikely that the specified method will find favour with all. It may become necessary to hold a meeting between all interested parties, these will include producers and users of compost in horticulture and agricultural, those involved in landfill with and without prior degradation and finally the sewage sludge industry.

# 5. DRAFT STANDARD

This draft standard is included as the basis of a possible National standard.

**NOTE 1 Where italics appear in the draft method it indicates an area that requires additional work and confirmation.**

**NOTE 2 Whilst the title of the method and body of the text states "wastes composted organic materials" it does not mean that the method may not be suitable for other forms of waste or materials that are undergoing a form of respiration.**

# A method based on carbon dioxide evolution to determine the aerobic stability of wastes and composted organic materials

**Contents** **Page**

## Safety warning

**Care should be taken when handling samples that may contain sharp fragments, chemical contaminants or possible pathogenic organisms.**

## 1. Scope and field of application

A method is described for the determination of aerobic stability in wastes and composted materials. The sample shall be obtained in accordance with HORIZONTAL STANDARD - SAMPLING (EN 00000). The procedures described herein are not necessarily applicable to or suitable for all types of waste or composted materials.

## 2. Normative references

This method incorporates by dated or undated reference, provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this method only when incorporated in it by amendment or revision. For undated references the latest edition of the publications referred to apply.

| | |
|---|---|
| ISO 5725:1994 | Precision of test methods - determination of repeatability and reproducibility for a standard test method by inter-laboratory tests. |
| EN 00000:200# | HORIZONTAL - Sampling |
| EN 13040:1999 | Soil improvers and growing media - Sample preparation for chemical and physical test, determination of dry matter content, moisture content and laboratory compacted bulk density |
| EN 13039:2000 | Soil improvers and growing medium -Determination of organic matter content and ash |
| PAS 100:2002 | Specification for composted material |

## 3. Principle

A moisture-adjusted sample is incubated at *25°C* with continuous replacement of carbon dioxide free air. Carbon dioxide evolved from the sample is collected in a sodium hydroxide solution as sodium carbonate. The collected carbonate is precipitated as barium carbonate by the addition of excess barium chloride. The concentration of carbon dioxide evolved by the sample is measured by titration of the residual sodium hydroxide with standard acid.

NOTE 1Barium carbonate is not decomposed by the action of the acid when phenolphthalein is used as an indicator, colour change occurs at pH 8.5.
NOTE2 Other methods for the detection of carbon dioxide exist and can be found in ISO 16072:2002.

## 4. Definitions

For the purpose of this standard the definitions given in PD CR 13456, EN 12579, EN13040 and PAS 100 apply.

## 5. Reagents

**5.1 General,** all reagents used shall be of recognized analytical quality. Use water of grade 2 complying with EN ISO 3696

**5.2 Saturated barium chloride solution,** c ($BaCl_2$) = dissolve an excess of $BaCl_2.2H_2O$ in 1 litre of water and filter.

**5.3 Hydrochloric acid,** *c* (HCl) = 1mol/l; purchase this solution ready prepared.

**5.4 Phenolphthalein indicator solution** *c* ($C_{20}H_{14}O_4$) = dissolve 1g of phenolphthalein in 100 ml of ethyl or isopropyl alcohol. Add 100 ml water. The indicator may be purchased ready prepared.

**5.5 Sodium hydroxide** *c* (NaOH) = 1mol/l; purchase this solution ready prepared and standardized in a collapsible airtight container. Discard when blanks turn cloudy after addition of barium chloride.

## 6 Apparatus

**6.1 Constant temperature room, incubator or water bath** capable of maintaining a temperature of *25±1° C*.

**6.2 Carbon dioxide scrubbing vessel,** 500 ml Drechsel bottle design or similar fitted with a sintered disc e.g. aquarium air diffuser.

**6.3 Carbon dioxide collecting vessel,** 100 ml Drechsel bottle design or similar fitted with a sintered disc e.g. aquarium air diffuser. A simple 150 ml test tube with rubber bung fitted with inlet and outlet tube connections is sufficient.

**6.4 Incubation vessels,** 500 ml – 1000 ml polyethylene jars with airtight screw top lids incorporating internal and external inlet and outlet tube connections.

**NOTE** Vessels of larger volume may be required when handling very coarse samples or when large samples weights/volumes are tested.

**6.5 Flexible tubing,** narrow bore plastic.

**6.6 Air pump,** small aquarium type. Ability to adjust airflow is advantageous but not essential as long as airflow of 1–2 L / hr is achieved.

**6.7 Dispensing pipette,** 50 ml capacity, grade A.

**6.8 Burette,** 50 ml capacity, grade A.

**6.9 Titration flask**, Erlenmeyer type 500 ml

**6.10 Magnetic stirrer,** optional

**6.11 Sieve,** 20 mm apertures

**6.12** **Balance,** capable of weighing 120 g with an accuracy of 0.1 g

**6.13** **Diffusers, Aquarium type or similar**

**6.14** **Flow restrictor or bleed valve,** to adjust flow. Only needed if pump is not adjustable.

# 7 Procedure

## 7.1 Apparatus

Sequentially connect together with the flexible tubing (**6.5**) the air pump (**6.6),** the carbon dioxide scrubbing vessel (**6.2),** the incubation vessel (**6.4**) and the carbon dioxide trapping vessel (**6.3**).

## 7.2 Sample preparation

**7.2.1** Prepare the test sample in accordance with EN 13040:1999, clause 8.5.

**7.2.2** Determine the total solids content in accordance with EN 13040:1999, clause 10.

**7.2.3** Determine the volatile solids in accordance with EN 13039:2000.

**7.2.4** Adjust the total solids concentration of approximately 500g sample (**7.2.1**) to between *40 % and 60 % mass/mass by the small addition of water*. Add the water gradually with mixing until the compost is visibly wet but no free liquid drains. The compost must remain friable with plenty of air porosity.

NOTE Larger quantities may be required for very coarse samples.

**7.2.5** Determine the final total solids in accordance with EN 13040:1999, clause 10.

## 7.3 Determination of carbon dioxide evolution rate.

Transfer *100 g ± 2 g* of the sample **(7.2.4)** weighed to the nearest 0.1 g to the incubation vessel **(6.4)**. Transfer approximately 250 ml of sodium hydroxide solution (**5.5**) to the carbon dioxide scrubbing vessel (**6.2)** and accurately pipette 50.0 ml of 1 M sodium hydroxide solution (**5.5**) into the carbon dioxide collecting vessel (**6.3**). Place the complete apparatus in the constant temperature environment (**6.1**). Switch on the air pump and adjust the airflow rate **(6.14)** to 1–2 L / hr. After 24 hrs wash the internal delivery tube and aerator into the collecting solution and transfer into a pre-prepared collecting tube containing a further 50 ml 0f 1M sodium hydroxide. Stopper the tube being removed to prevent absorption of atmospheric carbon dioxide. Note the times the first trap is removed and the replacement trap fitted. Repeat this process every 24 hours over a 7-day period. Do not turn off the air pump at any time or backpressure may cause NaOH to siphon back to the pump.

Transfer the contents of the carbon dioxide trapping vessel (**6.3**) into the titration flask (**6.9**) with water washing. Add 20 ml of barium chloride solution (**5.2**) to precipitate any carbon dioxide. Add two to three drops of phenolphthalein solution (**5.4**) and titrate with 1M hydrochloric acid (**5.3**) until the pink colour just changes to white (colourless in the case of blanks) with one drop of the acid.

NOTE 1 For very coarse samples much larger samples e.g. *1-2 kg* may be necessary.

NOTE 2 In the presence of strong alkali it is better to use rubber stoppers than glass stoppers.

NOTE 3 An automatic titration can also be used. Titration should be performed until pH 8.5.

NOTE 4If <5 ml hydrochloric acid has to added the test should be restarted with 50% of material.

## 7.4 Determination of blank value

An apparatus and reagent blank test shall be carried out in parallel with the determination, by the same procedure using the same quantities of all reagents but omitting the test portion.
If the apparatus has been set up correctly the titration value shall be very near to 50 ml indicating that all atmospheric carbon dioxide has been trapped in the first trapping vessel.

**NOTE** It is preferable to set up a series of parallel tests using the same pump to facilitate running replicates and blanks simultaneously with same batch of reagents.

## 8 Calculations and expression of results

The mass of carbon dioxide evolved each day is given by the following equations

**Respiration value (**mg $CO_2$ evolved per 24 h time period per gram material) = {[$B_{vol} - S_{vol}$] x 44.2} / 2 x (T/24)/W

Where
$B_{vol}$ is the volume in ml M HCl for the blank titre
$S_{vol}$ is the volume in ml M HCl for the sample titre
W is the dry weight of the material tested
T is time in between two measurements (hours)
DM is dry matter

Plot the mg $CO_2$/g DM/d against time (days)

Stability is defined as the point at which a plateau is reached. Note the average respiration value at stability over as many points as possible.

## 9 Precision

Perform the test in duplicate.
Reproducibility and repeatability data yet to be determined

## 10 Test report

The test report shall include the following information:

a) A reference to this Standard;
b) A complete identification of the sample;
c) The results of the determination of the average respiration value at stability and the graph of the plot the mg $CO_2$/g DM/d against time (days);
d) Moisture content;
e) Any details not specified in the Standard, or which are optional, as well as any other

factor, which may have affected the result).

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