Stability
(Biodegradability)

B J Cooper

UK – Private Consultant

Acknowledgement

This work has been carried out with financial support from the following EU Member States: UK, Germany, France, Italy, Spain, Nordic countries, Netherlands, Denmark, Austria, EU DG XI and JRC, Ispra.

CONTENTS

LIST OF TABLES

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. Many methods of test exist and have been reported in literature, these are reviewed with critical comment.
The need to test for potential biodegradability 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. It may be possible to adapt the proposed method for this purpose should it become necessary.

No standard methods exist for the determination of biodegradability (stability) after aerobic composting/biodegradation of wastes and similar materials.

In soils the term “stability” is more generally used to describe physical characteristics and is generally 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.

The proposed draft standard is based on several ISO methods. Further work is required to confirm certain aspects and ruggedness of the proposed method.

1. INTRODUCTION

Is there a distinction between biodegradability and stability?

(ISO 11266:1994) is a method for determining 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) Mineralisation:
The complete degradation of an organic substance to inorganic products.

(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.

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".

The words "stability" and "maturity" are often used by workers to mean the same thing. (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.

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 mineralisation. 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.?
Some laboratories are looking at the possibility of using NIR (near infra-red reflectance) to determine the degree of mineralisation 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.

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 microflora. Methods exist to measure abundance and activity of microflora (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) has 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.
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 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 sludges 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 sludges 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 sulphtate, 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."

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.

(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).

(Adini 2003) has proposed an oxygen uptake method that is capable of handling samples up to 50 L in volume. 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.

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 sludges and sample with dry solids of less than 5%.

The method of (Adini 2002) has interesting possibilities and should be considered for further development.

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). 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.
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 and stages biodegradation. The test may take up to 10 days to complete, a time span than may be considered to long for many operations.

2.4 Solvita Test®

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 – colorimetry 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.

3. EVALUATION OF DRAFTING A HORIZONTAL STANDARD

3.1 Sample size

The range of samples received into the laboratory will vary from finely ground material as will be found in sewage sludges 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 (2001) 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.

3.2 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. (Adini 2002) 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.
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.

3.3 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.
(Adini 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.

3.4 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?

3.5 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? Ancient tradition recommended the addition of urine to the compost heap to maintain microbial activity. (ISO 14855:1999) uses an inoculum 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 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

inoculant 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."

N-immobilization is well understood and occurs when soil microorganisms 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 be 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?

3.6 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_2C /unit VS / day	<2	2 – 8	>8
Dewar max Temp. rise °C	<10	10 – 20	>20
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 - No odours - No continued decomposition
1 – 3	Stable	- Cured compost - Limited door potential - Minimal impact on soil carbon and nitrogen dynamics
3 – 6	Moderately stable	- Uncured compost - Minimal door production - Addition to soil may result in nitrogen immobilization - High phytotoxicity potential - Not recommended for growing plants from seeds
6 –11	Unstable compost	- Very immature compost - High door and phytotoxicity potential - Not recommended for growing plants from seeds
>11	Un-stabilized material	- Extremely unstable material - Very high odour and phytotoxicity potential - Not recommended for use

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 landfilling 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.

4.3 Criteria for test methods

4.3.1 The test methods should, when ever possible be simple to operate.

4.3.2 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.)

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

4.3.4 The method must be rugged.

4.4 Method Recommendations (Rate of biodegradation)

4.4.1 A titrimetric method based on dynamic carbon dioxide evolution

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

4.5 Addition parameters,

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

4.5.1 Ammonia nitrogen and nitrate nitrogen

4.5.2 Total carbon by loss on ignition or dichromate oxidation

4.5.3 The carbon nitrogen ratio C/N

4.5.4 Carbohydrate fractionation e.g. ADF, NDF

4.6 Method Development (Rate of biodegradation)

4.6.1 The optimum moisture content

4.6.2 The optimum temperature for pre incubation

4.6.3 Time for pre-incubation

4.6.4 The optimum temperature to run the test

4.6.5 Nutrient status

4.6.6 Larger particle sizes

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

5. DRAFT 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 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 1 Barium 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* $\pm$ *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 4 If <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) = $\{[B_{vol} - S_{vol}] \times 44.2\} / 2 \times (T/24)/W$

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).

Bibliography

ISO 16072:2002 Soil quality – Laboratory methods for determination of microbial soil respiration

6. REFERENCES

Adani F., Genevini P.L. and Tambone F A New Index of Organic Matter Stability: Compost Science & Utilization (1995), Vol. 3, No. 2, 25-37

Adani F., Scaglia B., Biological Stability Determination in Compost and Waste by Dynamic Respiration Index (DiProVe Method) – Private communication (2003)

Bord Na Mona Private communication

Brinton - Compost quality Standards & Guidelines: An International View: Dec 2000

BS 1377:- Methods of tests for civil engineering purposes.

Butler T.A.; Sikora L.J.; Steinhilber P.M.; Douglass L.W.; L.J. Sikora, (2001) Compost age and sample storage effects on maturity indicators of biosolids compost. Journal of Environmental Quality (United States) 2001 VOL/ISS/PG: 30/6 (2141-2148).

California Compost Quality Council (2001) The CCQC Compost Maturity index.

CEC (2001) *Working document on biological treatment of biowaste, 2nd Draft.* ENV.A.2/LM 12 February 2001

Emerson W. (1967) A classification of soil aggregates based on their coherence in water. Aust. J. Soil Res., 5, 47-57

Evans T (2003): personal communication

Godley A. (2003) Estimating biodegradable municipal solid waste diversion from landfill. Phase 1 Review of tests for assessing the potential biodegradability of MSW. (Draft report Project record P1-513 Environment Agency R&D Dissemination Centre, Frankland Rd, Swindon, Wiltshire SN5 8YF UK)

Haines W. B. (1930) Studies on the physical properties of soils, J. Agric. Sci., 20 97-116

Haug, R.T. (1993) The Practical Handbook of Compost Engineering. Process Kinetics & Product Stability. Lewis Publishers, Boca Raton, FL. 717 p.

Henrik Lystad: Norstead report Assessment of compost Maturity and Ecotoxicity NT TECHN REPORT 404

Hue N.V. and Liu J. Predicting Compost Stability, Compost Science & Utilization (1995), Vol. 3, No. 2, 8-15

ISO 11274: 1988 Soil quality – Part 5: Physical methods – Section 5.5: Determination of the water retention characteristics – Laboratory methods

ISO 11277:1998 Soil Quality – Determination of particle size distribution in mineral soil material – Method by sieving and sedimentation

ISO 14239: 1997 Soil quality Part 4 Biological methods Section 4.4 Effects of pollutants on microbes; Subsection 4.4.4 laboratory incubation systems for measuring the mineralization of organic chemicals in soil under aerobic conditions

ISO 14852:1999 Determination of the ultimate biodegradability of plastic material in an aqueous medium – Method by analysis of evolved carbon dioxide

ISO 14855:1999 Determination of the ultimate biodegradability and disintegration of plastic materials under controlled composting conditions – Method by analysis of evolved carbon dioxide

ISO 16072:2002 Soil quality – Laboratory methods for determination of microbial soil respiration

ISO 17155:2002 Soil quality – Determination of abundance and activity of soil microflora using respiration curves

ISO 22476-3:2002 Geotechnical investigation and testing; Field testing; Part 3: Standard penetration test

Itävaara M, Venelampi O, Vikman M, Kapanen A – Compost Maturity – Problems Associated with testing. Microbiology of Composting Eds. Insam H, Riddech N, and Klammer S. Published by Springer 2002 ISBN 3-540-67568

de Kreij C., van Elderen C. W., Wever G., Duijuestijn R. C. M. - Extraction of Growing Media Regarding its Water Holding Capacity: Proc. Int. Synp. On Growing Media and Hydroponics; Eds Maloupa & Gerasopoulos, Acta Hort 548. ISHS 2001

McAdams, M.W.R.K. White (1996) Compost Stability Determination. Composting in the Carolinas – Proceedings of the 1996 Conference. October 23-25, Myrtle Beach, South Carolina.

Microbiology of Composting: Editors Insam H, Riddech N, Klammer S; Published by springer-Verlag, Germany 2002

Morten Carlsbaek (2003) Private correspondence

NEN 6745-1:2002 Geotechnics; Load test on foundation piles; Part 1: Static axial loading in compression

Nordtest report NT TECHN REPORT 404 (Approved 1998-10) Assessment of Compost Maturity and Ecotoxicity

PAS 100 Publicly Available Specification – PAS 100 Specification for composted materials. Published by BSI, London, UK

Project 99-PUM-3, Developing Protocols for Measuring Biosolids Stability Water Environment Research Foundation, USA

Sloot van der H.A., Comans R.N.J., Bakker F.P. (2003) Role of stable organic matter, degradable organic matter, combustible organic matter and dissolved organic matter in waste characterisation.

Soil Control Laboratory) CCQC (2001)

Stentiford, E., Lasaridi, K. (2000) Why and how to test composts for stability. Wastes Manage.

pp. 47-48 (Aug. 2000)

Stentiford, E. (2002) Biological treatment of biodegradable waste – EC-JRC Workshop 8 –10 April 2002 Brussels

The Composting Association (2001) Large-Scale Composting. A Practical Manual for the UK.

Waller 2003 Private communication

Weppen P 2002 Determining Compost maturity: Evaluation of Analytical properties Compost Science and Utilisation (2002) Vol.10 No 1, 6-15

WRAP Review of Compost Standards in Denmark Nation Specific Supplement 3: 2002

WRAP (2003) Waste and resources Action Programme (WRAP) Assessment of Options and Requirements for Stability and Maturity Testing of Composts

XP U 44-162 Organic soil improvers and growing media – biochemical fractionationning and estimation of biological stability - method of organic mater characterisation by successive solubilisations AFNOR undated

XP U 44-163 Organic soil improvers and growing media – determination of potentially mineralisable carbon and nitrogen – method of incubation under controlled conditions AFNOR undated