Monitoring Carbon-Dioxide and Ammonia from Composts by Use of Solvita® Digital Colour Reader

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SUMMARY
The Solvita® test system was developed as a rapid field procedure for compost stability and maturity testing. It employs chemistry-gels that equilibrate with headspace CO_2 and NH_3. The tests have been found to be accurate and reliable over a wide range of composts. However, Solvita requires visual colour identification and therefore may cause subjective intervention of results, increasing operator error. The test also employs volume-basis and not weighed samples, so correlation to gravimetric data are not consistent. This paper presents development of a digitized-light approach to measuring Solvita, by employing inexpensive diode array detectors. With this DCR it is demonstrated that Solvita attains high responsiveness, greater accuracy, precision and eliminates subjectivity. Together with sample weighing and conversion of units, the new test attains laboratory-level expectations.

Solvita, maturity, CO_2-respiration

1 Introduction

Production of carbon dioxide and the consumption of oxygen are among the most frequently cited parameters for evaluating compost stability and maturity. Compost respiratory activity is directly related to the presence and quantity of microorganisms and is also dependent on the quantity and quality of the organic and nutrient mix undergoing degradation. Breakdown of organic compounds and release of CO_2 diminishes over time as organic matter becomes less labile. Humus formation represents stabilization and conversion and is a principle reason cited that compost is beneficial to soils. Yet many composts are immature and incompletely humified. Compost may be considered soil and plant-ready, when decay has declined to low levels and humus polymerization is maximised (Schnitzer & Kahn 1987, Schlichting and Blume, 1966). Compost that has not attained this state may be referred to as “raw waste”, “active compost”, or “uncomposted” (BGK,1998). Laboratory respiratory tests, when used properly, are important and helpful to gauge the quality of the process, the composting age and its stability (CIWMB, 2003). For testing and validating biodegradable polymers in composts, measuring CO_2 respiration is a critical parameter (Strotmann et al. 2004).

Lab CO_2 evolution procedures have been adapted to compost recently, but have long been used as indicators of soil fertility (Gainey, 1919). CO_2 evolution techniques date at least to Isermeyer and Koepf (1950, 1952). Anderson (1982) describes at least 10 adaptations of laboratory respirometry methods based on these. The use of CO_2 rate to estimate the activity of aged manures and composts, however, is different to that of using it as an index of soil fertility, and was possibly first proposed by Castellanos (1984). He was able to show a relationship of nitrogen mineralization from manures with laboratory-tested CO_2 respiration, indicating that the less stable products gave greater release of nitrogen. Nakasaki et al. (1985) showed that the amount of dry matter reduction of compost was directly proportional to cumulative CO_2 respiration and microbial cell counts, thusly providing a basis for maturity estimation. Willson and Dalmat (1986) showed a simple laboratory set up using pressure drop inside a sealed vessel as a means to evaluate stability or reactivity of aging compost.

Analyses of CO_2 respiration (and O_2 uptake) for organic materials and compost is expressed a variety and confusion of units. It is customary to report on a volume basis, a total-solids (weight basis) and on a volatile solids (VS) or

total-carbon (TC) basis. The latter (VS, TC) are increasingly common but represent relative and not absolute indicec of respiration, since they only concern the organic fraction. Due that organic content may be high in fresh compost, there is likely to be very little difference in reporting TS or VS unit basis respirometry; however, as compost ages, and the ash content increases, the rate of respiration as a function of organic content decreases nearly exponentially. In this way, a volume based test like Solvita® becomes increasingly sensitive as density of compost increases with age, which is of interest to the end-user, but confounds correlations with gravimetric tests. It is also theoretically possible that total (absolute) respiration may decline but not relative respiration, especially where suboptimal conditions exist or simply where composts meeting basic regulations and harvested and sold immature, an increasingly common practice. Under these circumstances, it is very useful and important to report decomposition in both absolute and relative units, similar to reporting heavy metals relative to TS and OS (BGK, 1998).

The applicability of most laboratory respiration methods for simple field or on-site use, is questionable. Laboratory CO_2 respiration methods employ alkali traps with KOH or NaOH which absorb evolved CO_2 and are titrated with analytically standardized HCl, after precipitating carbonates with excess $BaCl_2$, using phenolphthalein as the endpoint. The accuracy of the endpoint detection, is one or two drops of HCl based on the quality of the titrimeter, and determines the precision of the overall test, in relation to the quantity of sample (Anderson, 1982).

CO_2 trap procedures most definitely require proper laboratory experience and are not intended for inexperienced users, and have not to our knowledge been used successfully for on-site monitoring at compost facilities. For this reason compost samples that are to be tested for stability or maturity by respiration are normally sent to laboratories that specialize in this form of testing. As a result, very little stability testing is performed. In the USA, there are only about 9 or 10 laboratories that are capable of performing such compost respirometry tests, as recently determined by a compost analysis-proficiency program (CAP, 2004). In Germany, compost laboratories that are certified under BGK are more numerous, and compost samples may be optionally examined for respiratory activity using an unspecified oxygen respirometer, presumably either Sapromat or Oxitop (Strotman et al. 2004, BGK 1998, Veeken et al, 2002). The compost stability test, which is mandated for compost members of the association Bundesgütegemeinschaft-kompost (BGK), is the Dewar self-heating test, which differs from respirometry.

The Dewar test has been represented to be an on-site procedure, however, this is dubious. It is a self-accelerating type procedure measuring heat production against a (required) constant ambient background, and the heat level attained is confounded by evaporative heat loss (Becker 1995, Brinton et al, 1995) and also by differential vessel-wall heat-loss, not controlled for in the flask manufacturing process (McEntee, 2005). Consequently, the test is best conducted with precalibrated Dewar-vessels, in incubator chambers at 20C, and on somewhat dry samples (Becker, 1995, McEntee, 2005), all factors that remove it from on-site consideration. The Sapromat® test indirectly measures oxygen consumption and is considered a specialised (and costly) laboratory device, with similar restraints. To our knowledge it has not been employed for routine compost testing. Oxitop® is a less expensive modular unit recently adapted from biochemical-oxygen-demand (BOD) methods to be used for soils and even more recently for use with high organic materials by employing wide-mouth jars with large rubber seals (Veeken et al, 2002). There is only one laboratory in USA that we are aware of that employs Oxitop® for compost, and the test is definitely not portable and must be performed in dark, temperature controlled chambers to minimize pressure variation. For oxygen consumption testing, specific oxygen uptake rate (SOUR) tests have also been modified from BOD. SOUR testing, after CO_2 titrimetry, may be the simplest approach to asses compost stability, yet it also requires specialised laboratory equipment including polargraphic electrodes and calibrated electronic meters together with incubation vessels, uses artificial (for compost) water-suspensions, and is not adaptable to on-site, field conditions (ASTM 1996, Ianotti et al. 1994).

Lab procedures are accurate when properly used but also time consuming and costly to the compost producer, who may require information about stability frequently for regulatory purposes or as compost shipments are being made (CIWMB, 2002). Liebig et al. (1996) discuss a portable method for collection of CO_2 gas samples for field sampling

of soil respiration for use with portable Dräger-tubes that employ semi quantitative detection. Itävara et al (2000) discuss the technical issues involved in adapting a variety of tests for compost maturity assessment. All tests are limited for their adaptability, quantity of material that can be tested, access to samples, portability or length of time required to obtain meaningful results returned from the laboratory. For this reason, the Solvita® test was developed to work under regular field conditions, with precautions for normal good handling. In this paper, we show an improvement of the procedure to be more accurate and precise.

2 Procedure

Composts may produce enormously variable quantities of gases such as CO_2, VOC, and NH_3 in dependence on age and other factors. A simple field test must therefore be very adaptable. Solvita® was designed to measure respiration over an extremely wide range of concentrations of gases, by using a chemistry-based colorimetric scale and assigning ordinal numbers to visually discernible colour steps for CO_2 and NH_3, as shown in Table 1.

In order to have an easy test and to make it safe to the user, Solvita® also replaced the gravimetric (TS) basis with volume-basis, similar to Dewar (Becker, 1995, Brinton, 1995). This meant that sample weighing was not required. Next, Solvita® replaced the toxic $BaCl_2$ ingredient used in lab respirometry with a phosphate buffer system. Similarly, the ammonia detector of Solvita replaced the boric acid trap with a carbonate buffer system. These changes result in a dual advantage: the tests are indeed very easy and safe to perform plus the chemical CO_2 reaction is largely reversible so that the Solvita® CO_2 chemistry equilibrates constantly to the actual headspace concentration of CO_2 with no endpoint other than time.

Table 1 Solvita Ordinal Numbering of Visual Optical Scale in Relation to Concentration of Gases

Ordinal Number for CO_2 Test Result	Approximate CO_2 conc. mg L^{-1}	Ordinal Number for NH_3 Test Result	Approximate NH_3 conc. mg L^{-1}
8	2,000	5	<100
7	5,000	4	800
6	10,000	3	2,000
5	20,000	2	8,000
4	40,000	1	25,000
3	75,000		
2	140,000		
1	200,000		

As Table 1 indicates, Solvita® is responsive to a large range of concentration in headspace gas. Changa et al. (2003) and Wang (2004) reported comparisons of Solvita to laboratory respiration rates determined gravimetrically and obtained very high linear correlations. These workers noted that it is difficult to reduce volume colorimetric tests to mg CO_2-C g^{-1} VS. For this reason, lab procedures thusly appear to have the advantage of being weighed before data reduction, but the significance for practical interpretation has never been determined. Francou et al. (2005) reported high correlations of Solvita to Dewar self-heating tests, and such a high correlation is expected since both are volume-based tests. It is also understandable that Francou et al. did not show clear relationships when Dewar Reifegrad gave results < II; Solvita® is responsive to very low respiration values and Dewar definitely is not (Brewer and Sullivan, 2003). Recent work shows Solvita with high detectability at low ranges of fungal respiration (Chitraker et al, 2006). By weighing the samples instead of using volume rates, the correlation results may be markedly improved, even though the advantage to the composter is not necessarily increased. Use of a digital devise to accurately measure colour would be a solution to the subjectivity of colour perception. Because the rate of concentration change is exponential with respect of the Solvita ordinal numbers (1 to 8 for CO_2 and 1 to 5 for NH_3), a significant increase in readability and accuracy could be made by more precise measurements. Solvita is normally

read visually as reflected (transmitted) light from a thin 1mm gel surface. The digital colour reader (DCR) approach was considered valid since Solvita chemistry obeys the Beer-Lambert law relating transmission of light to the molar concentration, absorption and path length. The new Solvita DCR has an array of photo-detectors, each with either a red, green, or blue filter, or no filter. The filters for each colour are distributed evenly throughout the array to eliminate location bias among the colours. Internal to the device is an oscillator that produces a square-wave output whose frequency is proportional to the intensity of the chosen colour. RGB (red-green-blue) colour wavelengths are read simultaneously and employed individually and in ratios by internal software to deduce both the ordinal Solvita number and the concentration of selected molecule, CO_2 or NH_3. The Solvita paddles are thus simply inserted into the light path of the diode array (see Figure 1 and Figure 2).

Figure 1 DCR Device for Insertion of Solvita® Gel Paddles after Respiration Test

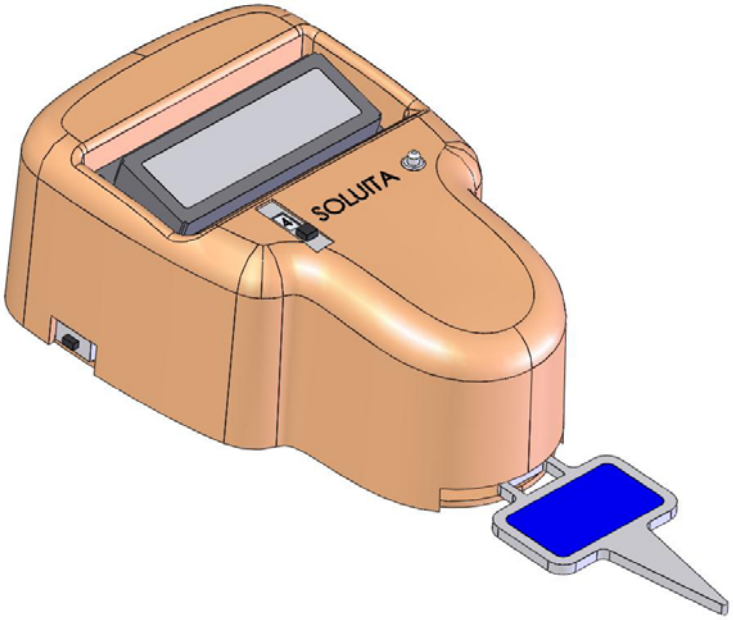
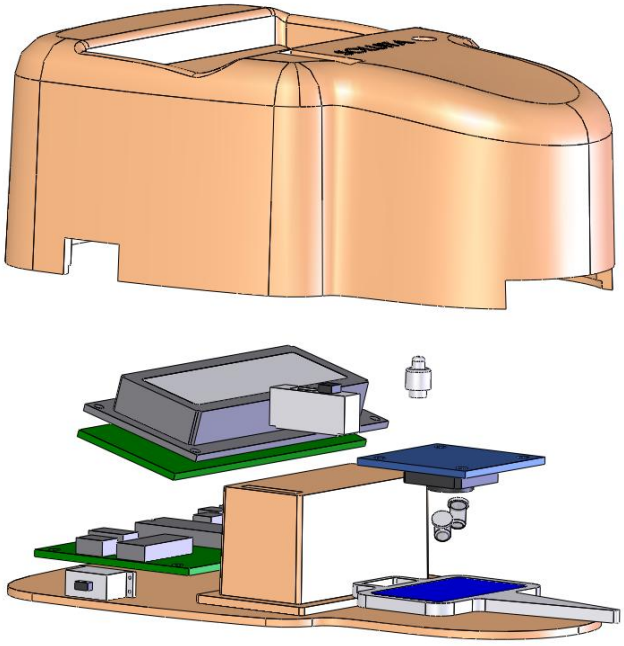


Figure 2 Expanded View of DCR showing Diode Optical Components.



2.1 DCR Correlations to CO_2 Concentration and Visual Solvita

Initial testing of the DCR has been conducted at two laboratories: Purdue University, East Lafayette, Indiana and in the soil laboratory of USDA-ARS, Temple, Texas. These trials have provided evidence of high responsiveness, speed, and accuracy consistent with standard laboratory respirometry (Stroshine & Moog, 2006, Rick Haney, *personal communication*). In the latter case, laboratory respiration tests of soil with Solvita DCR correlated at r^2=0.89*** and 0.82*** for 24-h and 28d CO_2 tests.

Fig 2. shows the visual Solvita correlated to concentration of CO2 in a 250cc incubation jar provided with the kit. The test is read through the walls of the clear polystyrene jar, or by removing the paddle from the jar, with correlations between r^2=0.975 and r^2=0.978, respectively, indicating that it makes no difference if one leaves the Solvita paddle in the jar or removes it to be read. It also shows the exponential response of Solvita ordinal colour numbers to the concentration of CO2 – a doubling of concentration for each visually discerned colour change.

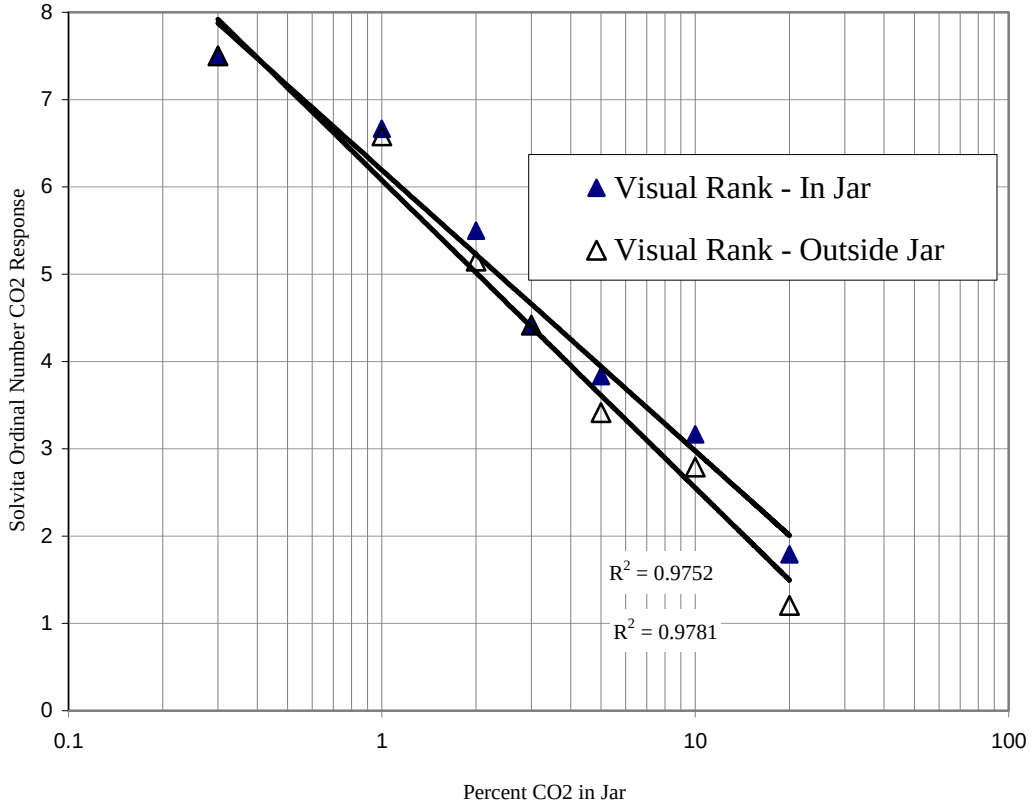


Figure 3 Solvita Visual Ranking in Relationship to Headspace CO_2 Concentration

It is common experience that visually reading Solvita may be performed with greater accuracy the more experience one has. It is also observed that reading Solvita by placing a number of test samples side by side, improves readability, since small differences are easily distinguished. For example, rank-order correlation of staff results indicates ½ unit visual colour changed is reliably viewed. However, from a quantitative lab perspective, this means a 50% rate difference in CO_2-production; therefore, the use of a digital reader capable of distinguishes small units of change would significantly improve precision and reduce variability.

It should also be pointed out from the chart (see Figure 1), that the range of test results for Solvita is extremely broad, with a Solvita #8 (considered to be “mature” or “highly stable” compost) corresponding to about 2,000 ppm CO_2, a Solvita #7 is 5,000 ppm, and so on, such that a Solvita #2 is between 15 and 20% CO_2.

The new approach to using Solvita® either with visual colour chart or DCR is to employ the kit to determine the time to attain 50% depletion of oxygen. The amount of time it requires to reach a Solvita #3 colour (2.50 – 3.25 by DCR), will correspond to 50% oxygen depletion, under similar sample: container circumstances. For in-vessel composting, with limited headspace, oxygen depletion determines the time to set aeration cycles. By employing a simple approach with the Solvita kit as indicator, the timing of aeration cycles based on expected depletion on the test jar (or nay test jar of any volume : sample ratio) may be anticipated.

The Solvita® DCR collect data for light reflectance after a 0.3-sec burst of low-voltage brilliant diode light. This reading is converted by on-board software into respective units of concentration and units if Solvita. Figure 4 shows the best-fit curve of the new DCR employing a mathematical parameter of red-blue-green light capture, regressed linearly against the Solvita ordinal number with an r^2=0.984**. For each configuration, whether reading CO_2 or NH_3, these internal calibrations are performed automatically.

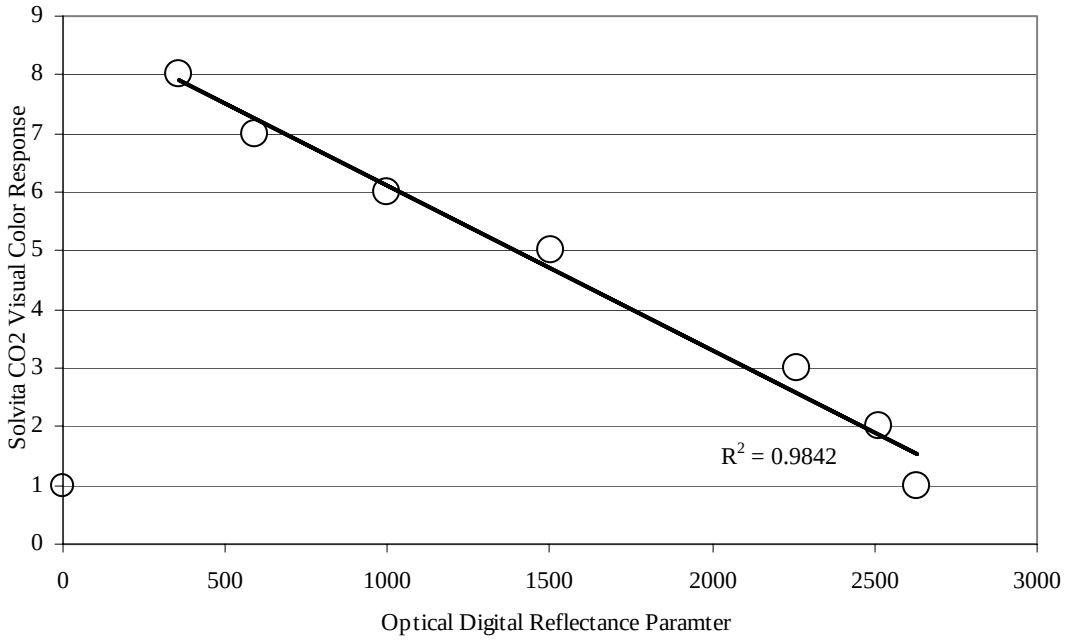


Figure 4 Digital Colour Reader Response in Relationship to Solvita® Ordinal Numbers.

How does the Solvita® DCR attain this range of sensitivity from 0.1 to 25% CO_2? Normally, laboratory respirometry must be predetermined for range of carbon dioxide expected; this influences choice of the CO_2-trap in terms of NaOH concentration, HCL titration and so on, in order to avoid over-saturation, otherwise the test has to be repeated.

The optoelectronic sensor employed in Solvita precisely measures light using an array of photodiodes, with the output of digital square waves. Thus, the unit has an input dynamic range of 160dB; that is, it can measure light over a range of 100,000,000-to-1. In this way we have created a test system in Solvita reader based on both the patented broad-spectrum chemistry and the high-dynamic range optics at a low cost to represent a range of CO_2 that is consistent and relevant to compost performance over the entire range of fresh very active to very cured. These data help explain unusual results reported for Solvita as being insensitive to moderately high respiration rates (Adani,

2003). Based on our data, and independent university trials, we predict that Solvita may represent the dynamic respiration range (DRI) covering the entire scale accurately from about 50 to 2,500 mg O_2 kg^{-1} VS h^{-1} DRI, as indicated in the following graph (see Figure 5). Because Solvita colours represent ordinal numbers assigned to an exponential detection range, we should expect to obtain a straight line correlation in most cases of respiration over a range of samples from low to high.

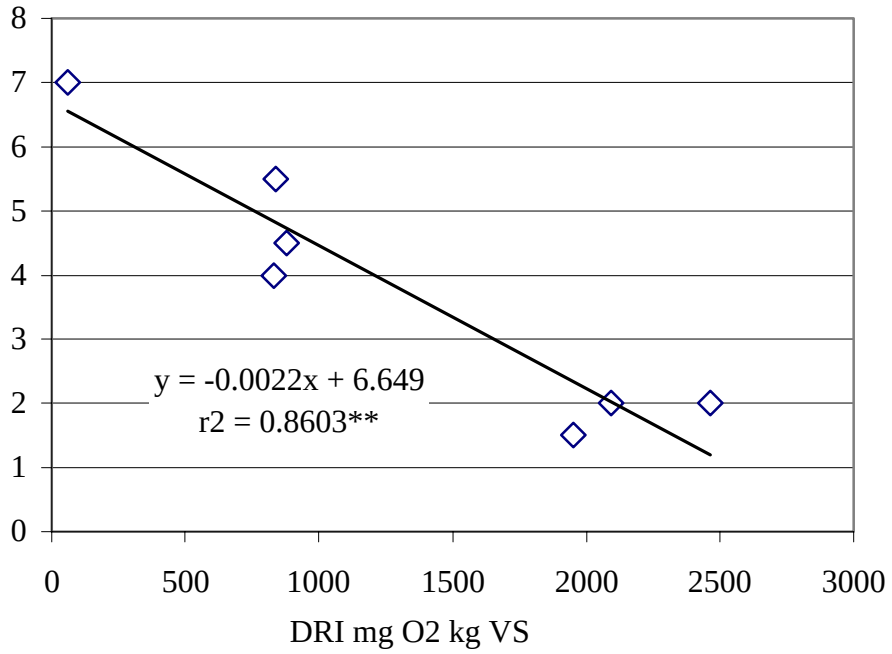


Figure 5 Solvita® Ordinal CO_2 Scale Correlated with ASTM-DRI of MSW Compost

The sensitivity of the DCR detector is made evident may repeating measurements with the same Solvita® gel paddle in increments of 30-min, using a slotted lid device as previously developed (Stroshine and Moog, 2006).
The DCR in the case of very active composts shows a linear responsiveness from 2- to 6 hours of respiration (see Figure 6). For each compost sample, this relationship may be effectively and inexpensively explored in this manner. There is considerable interest in developing quick tests that are not only simple, but also effective over short periods of time (Brewer and Sullivan, 2003). The only limits for Solvita in very short term testing (< 2 h) are the time it takes for CO_2 to fill all the pore space of the sample+jar.

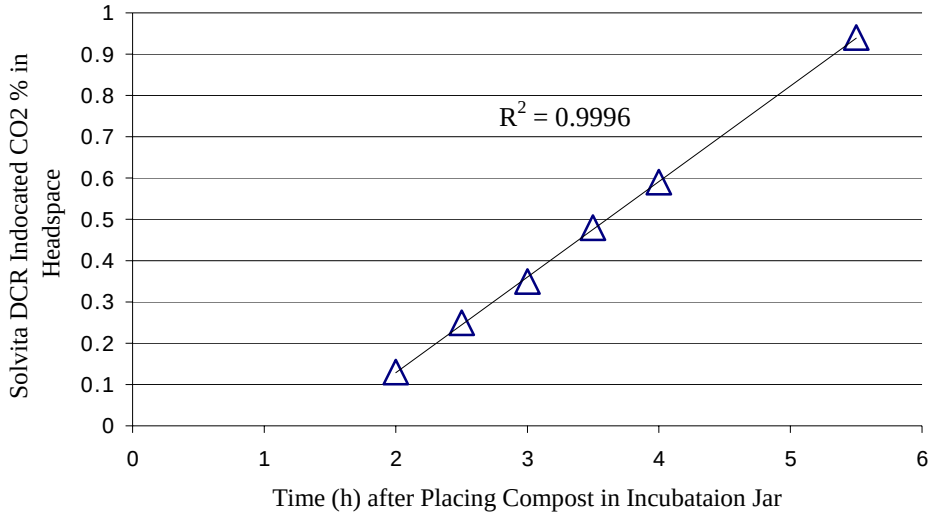


Figure 6 Respiration Response of Active Compost as Indicated by Solvita® DCR over Time.

2.2 Ammonia Detection with Solvita® test and DCR Response

Ammonia (NH_3) in compost is a sign if incomplete stabilization of nitrogen in relation to the amount of carbon present but unavailable. A surplus of nitrogen (C:N < 25) can cause elevated ammonia in composting. Ammonia may be noxious to compost workers, is harmful to farm animals exposed to it in barns, and can be toxic to many plants depending on its concentration in soil and compost. NH_3 is often responsible for the "burn" effect after heavy surface application to some crops. High ammonia content, however, may also indicate a potential positive crop response when applied correctly to heavy-feeder field crops such as maize. Ammonia emissions have been found to be a nuisance at some compost facilities in England causing harm to designated habitat sites (ENDS, 2005), and ammonia is presently regulated along with VOCs for compost by the California Air Board (CIWMB, 2002). Therefore, accurate quick-tests may be a very important ingredient for compost management.

Under normal conditions of composting, ammonia may increase rapidly in the early period of decomposition, then decline or disappear completely as the compost matures. The pH of the compost tends to follow this cycle attaining 8.5-9.0 when the ammonia is high. Both high pH and high ammonia may inhibit microbial activity and therefore compromise the testing of respiration by any procedure. Increase of ammonia and NOx may also interfere with Oxitop® and Sapromat® tests by changing the pressure of the system in the opposite direction of CO_2-induced pressure drop. The interference may be large, since NH_3 may rise to 2,000-10,000 ppm within the first 24 h of a test, a level of gaseous production equivalent to CO_2 rate in active compost.

The Solvita kit employs a modified boric-carbonate reaction that enables a wide range of detection (see Figure 7.) The range of response was designed to account for ammonia expected in manures and active composts.

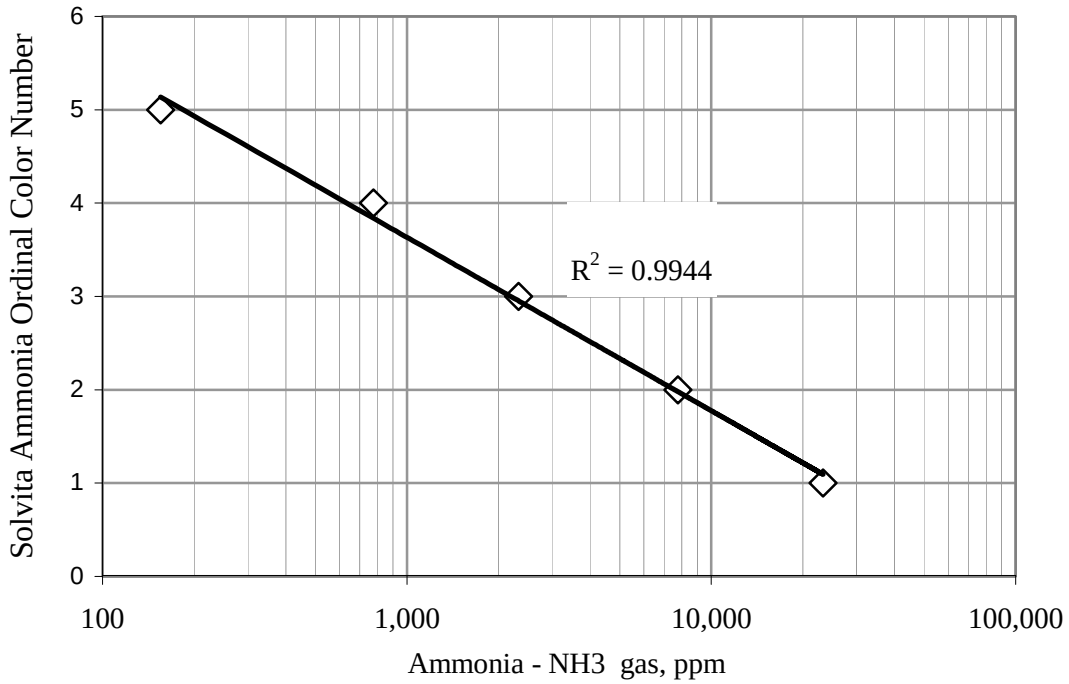


Figure 7 Solvita Ordinal Numbers Assigned to Test Range of NH_3 Gas in Head Space of Incubation Jars.

To determine the applicability of light-diode technology to detection of Solvita ammonia, we tested RGB sequences of transmitted light, with the following excellent correlations (see Figure 8).

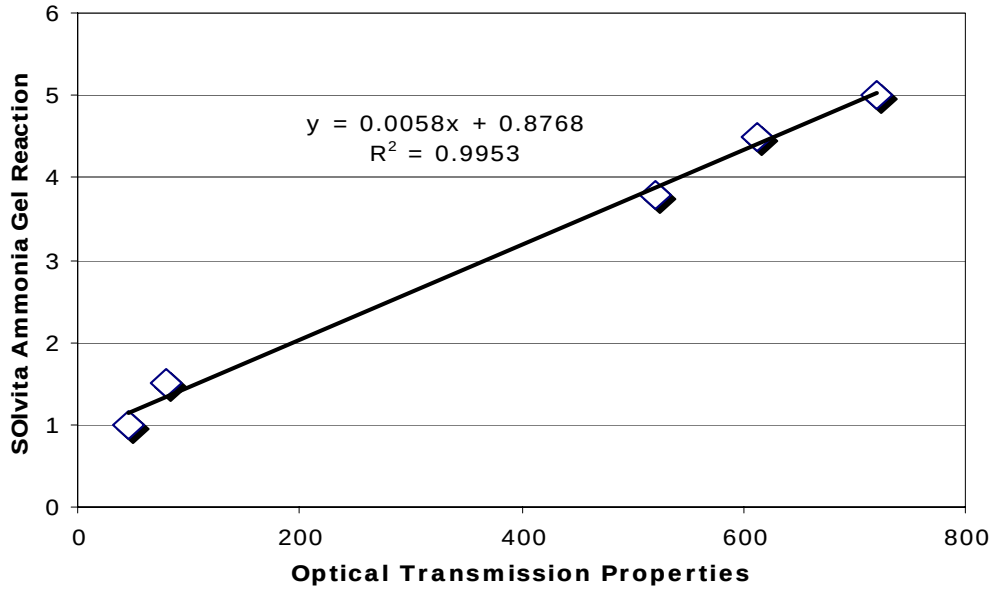


Figure 8 Solvita NH3 Ordinal Scale Value in Relation to DCR Optical Responsiveness

3 Conclusions

The usefulness of on-site and rapid Solvita® testing has been demonstrated in worldwide experience with this test kit over more than 10-years. Limitations of the original procedure exist in attaining lab-level quantisation due partly to subjectivity of colour perception and the use of volume-based samples. By employing an array of photo detectors, each with coloured filters distributed evenly throughout the array to eliminate location bias, we are able to rapidly bounce light off the Solvita gel and attain highly accurate readings, thereby improving usefulness.

Solvita chemistry has been designed to encompass a very wide range of gaseous molecules, mainly because compost varies enormously in maturity and stability. Even compost on the market today may be highly immature, cause odours and induce plant toxicity. Thus there will always be a need to measure compost stability without knowing what range of result is expected in the sample. Lab and field procedure need this flexibility or they will be costly and time consuming. To make the DCR as useful as the Solvita chemistry, internal to the DCR optical device is an oscillator, which produces a square-wave output, the frequency being proportional to the intensity of the chosen colour. This gives it an enormous range of response with very low voltage consumption. We are further able to read RGB colour wavelengths simultaneously within 0.5-sec total, and these values are then employed individually and in ratios together with internal software to accurately deduce both the ordinal Solvita number and the concentration of selected molecule, CO_2 or NH_3 over the entire dynamic range. Therefore, this procedure has capability of detection possibly beyond most conventional laboratory equipment, at a fraction of the cost. Interferences are similar to those that confound other respirometry and oxygen consumption tests including production of ammonia. For Solvita® this is dealt with since the latter that is read simultaneously but separately to CO_2 (there is no ammonia problem if composts are < pH 7.8). We have also determined a probably interference for NOx, resulting from reduction of nitrate-N during the test. This is very rare and usually only encountered in biosolids composts and in very matured composts high in NO_3 that have suddenly been remoistened, as in the lab test. Under these circumstances the same caution and care in identifying samples and their history before laboratory testing, must be exercised.

Finally, since the relationship to CO_2-% concentration in the headspace is indicated by Solvita, the test may now be adapted to produce quantisation as mg CO_2-C g VS d^{-1}, or other suitable index, as recommended by compost respirometry procedures. This only requires that the compost also be weighed into the Solvita jar. More effort to show the reliability and close relationship to more expensive laboratory CO_2 and O_2 methods may prove that Solvita is a very useful and cost-effective test in the field as well as in the lab. <>

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