Determining the Most Effective Method of Measuring Compost Maturity

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1.0 Introduction

Measuring compost maturity is the most important test in assessing not only the quality of the compost product, but also indirectly measuring the efficacy of the compost technology itself—unfortunately, despite the importance of the test, most of the recognized tests do not accurately measure compost maturity. A list of maturity tests acceptable to the Province of Nova Scotia prior to 2010 is listed in Table 1.

Table 1. Original List of Maturity Tests Prior to 2010 (NSDOE, 1998).

One of the following four sets of criteria must be met to qualify as mature compost:	
Set 1 (Two of three required)	- Carbon:Nitrogen (C:N) Ratio $\leq$ 25:1 - A respiration rate of <150 mg O_2/h/kg organic matter - Cress and radish germination shall be >90% of the control sample and plant growth shall be $\geq$ 50% of the control sample
Set 2	- Compost must be cured for $\geq$ 21 days - Compost will not re-heat to >20^{o}C above ambient temperature
Set 3	- Compost must be cured for $\geq$ 21 days - Organic matter reduction > 60% by weight
Set 4	- Compost cured (post-thermophilic stage) for six months in aerobic Environment

In 2010, the province of Nova Scotia made a significant step in correcting this issue as the number of acceptable tests was reduced to modified versions of the re-heat test and the respiration test (Table 2), which will be introduced by 2018.

Table 2. Present Maturity Tests for Nova Scotia Product.

One of the following:
- A respiration rate of <400 mg O_2/h/kg organic matter or a respiration rate of <4 mg CO_2-C/day/g organic matter
- Compost will not re-heat to >8^{o}C above ambient temperature

What has led to the present issue of confusion is that there are a number of respiration tests, all of which are considered acceptable that include six tests (including the re-heat test) cited in the US document Test Measures for the Examination of Composting and Compost (TMECC), as well as a respiration test utilized by the Centre De Recherche Industrielle Du Québec. All seven tests depend upon the response of the microbes to the conditions imposed upon them, however, all tests are somewhat different and as a result produce different results—some far more accurate than others.

On a general basis, the respiration rate is the most accurate method of quantifying compost maturity. The test measures the rate at which microorganisms consume oxygen as they chew their way through the organic waste —a faster rate of oxygen consumption indicates the presence of easily biodegradable organic waste (fresh food waste), or in other words a highly unstable product. It is not uncommon for fresh feedstock to have a respiration consumption rate of 10,000 mg of oxygen per kg organic matter per hour. As the composting matter moves through the process and the easily compostable constituents are consumed in the primary process stage, the microbes turn to the less biodegradable matter in the curing stage and as a result, the rate of oxygen consumption declines, producing a characteristic curve (Figure 1). Once most of the readily available organic waste has been consumed to the point where the respiration rate is 400 mg of oxygen per kg organic matter per hour (or 4 mg CO_2-C/g organic matter per day), it is deemed to be mature and is allowed to be released from the facility.

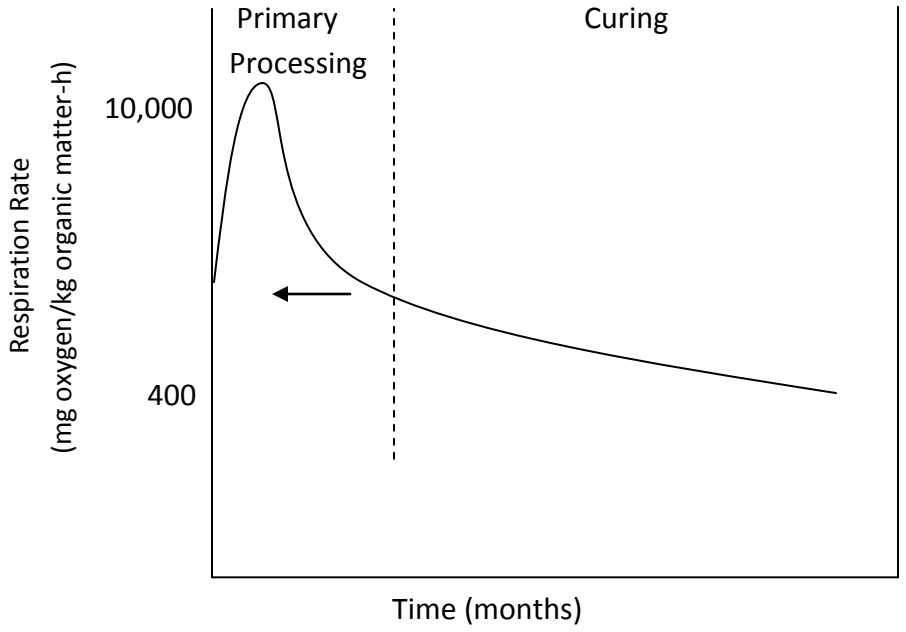

Figure 1. Typical process performance curve in terms of respiration.

Because the respiration rate declines over time as the compost moves through the process, it can be plotted if samples are extracted and tested at various stages of the process. This is, in effect, a method of assessing the performance of a given technology—the faster the process can produce a mature product (reduce the respiration rate to 400 mg oxygen/kg organic matter-h), the more efficient the process (Figure 2).

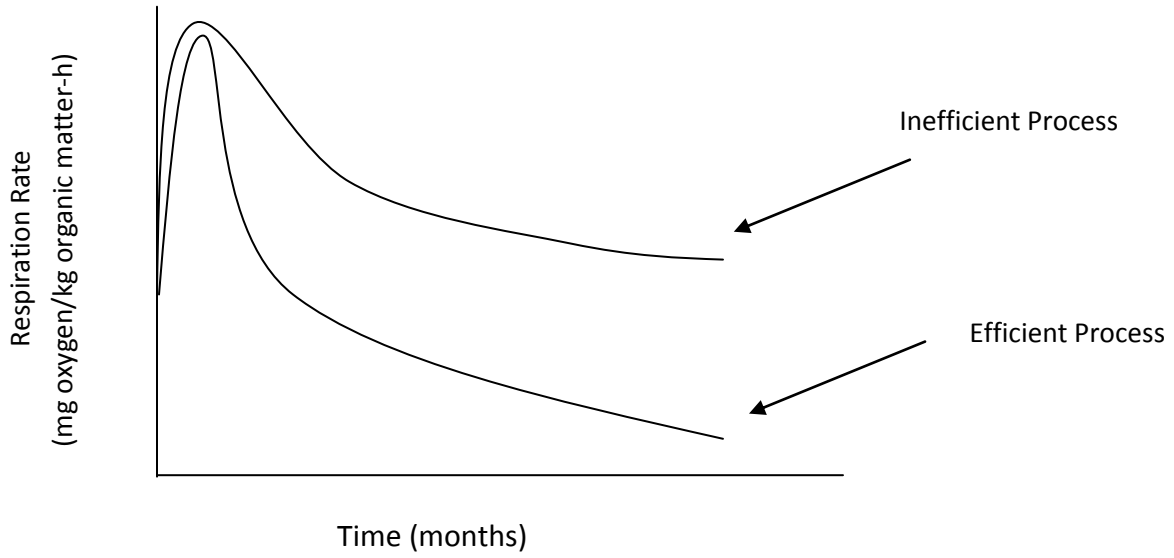

Figure 2. Comparing process performance curves using respiration rate.

Recently, the seven accredited tests were compared by Dr. Arnold on an informal basis using five different compost samples (Figure 3). These results indicate that there is a tremendous difference among the seven techniques used and when one applies the threshold of 4 mg CO_2-C/g organic matter/day, there are clearly tests that are incorrectly declaring the product as mature, while others indicate the product as immature. While it is possible to achieve a false pass, it is impossible to have a false fail unless the equipment is faulty. Thus, it is clearly evident that it is necessary to evaluate these tests in a more formal, scientific process to determine, once and for all, the test(s) that accurately determine compost maturity.

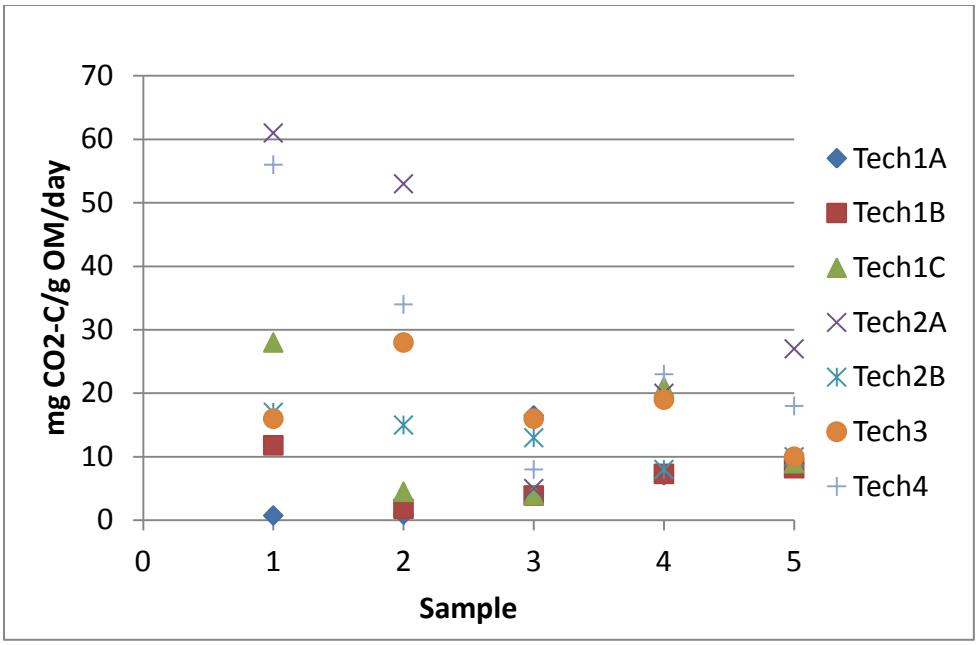

Figure 3. Comparing the results of seven techniques on five compost samples.

Like Nova Scotia in 2010, the province of Ontario recently completed a revision of their compost guidelines and, like Nova Scotia, reduced the number of maturity tests. Unlike Nova Scotia, the Ontario guidelines went a step further, for although they still declared respiration rates as the test category of choice, they also gave the Minister the discretion to determine which respiration method has to be completed and not necessarily leave it up to the compost facility. Consequently, both provinces have an interest to jointly determine the most effective maturity test.

2. PURPOSE AND OVERVIEW OF STUDY

In light of new compost maturity standards phasing in for Ontario on July 1, 2015, this study was initiated to better understand how different maturity tests performed with mature and immature compost samples. The Ministry of Environment & Climate Change (MOECC) tested compost maturity using four different respiration tests (Solvita, CO_2 evolution, O_2 consumption, BOD) for 10 SSO compost samples at various stages during curing from five SSO facilities and included one mature SSO+biosolids compost. Composite samples were split and sent blindly to each of 5 labs (including but not limited to A&L Canada Labs, BNQ (CRIQ), MOECC lab and Acadia University) to compare results between labs and between different methods, and to determine the typical rate of false passes for immature composts for each method.

3. SAMPLE SELECTION AND COLLECTION

Samples were collected at four SSO facilities in Ontario and one pilot site (SSO + biosolids), taking one composite sample at each of two stages of compost production (i.e. immature vs. mature):

1. Post-primary stage (a few days or week after material was moved to curing area)
2. Finished compost at end of curing period, nearly ready for market

The samples taken at the front end of the curing process post-primary (i.e. unfinished stage) allowed comparison of the tests under conditions where the false positives (passing of immature compost) may arise in known immature compost. The mature samples were included to test the range of values determined and check incidence rate of false passes.

Composite samples were collected pooling 10 separate grab samples from one meter into the pile at each random location across the pile and mixed according to the MOECC compost quality standards protocol (s. A1.1.8). After coning and quartering the 30-40 L composite sample was split into five and shipped (on ice in some cases) to the laboratories listed above for a round-robin style analytical study. Samples were single replicates only, and sent blind regarding maturity status). It was deemed not to do multiple independent samples of the piles because the project goal was not to determine the heterogeneity of the compost pile but rather the reproducibility of the methods.

4. RESPIRATION METHODS

Once the samples were collected, they were sent to respective laboratories for analysis; the test methods applied to the samples included the following:

CO_2 Evolution. CO_2 production from compost was measured using passive diffusion as per the TMECC method (05.08-B; Testing Methods for Evaluation of Compost and Composting 2002). The method suggests that samples be adjusted to proper moisture if necessary (40-55%) via squeeze test and samples allowed to equilibrate at 25 – 28°C for 24 to 72 hr prior to testing. CO_2 evolved was collected (passively) in an NaOH solution and the NaOH titrated daily for four days at 34°C to determine levels of CO_2 produced. Results are averaged over the 4 days for a value expressed as mg CO_2-C per g dry weight organic matter (OM) per day. Organic matter (loss on ignition; LOI) and moisture were determined as at MOECC and CRIQ labs on fresh subsamples as received.

Solvita Maturity Index. Solvita tests were conducted using the Solvita test kits and followed the supplier's manual (Woods End Laboratories Inc., ME, USA; i.e. TMECC Method 05.08-E). Compost was adjusted to proper moisture (as above) and placed passively (no circulation) in air-tight chambers with coloured gel paddles and situated in the dark. The NH_3 and CO_2 evolution colourimetric reading on paddles were determined after a 4-hr incubation period at 20 - 25°C using a hand-held spectrophotometer device for better accuracy. The Maturity Index was calculated as described in the manufacturer's protocol.

O_2 Consumption BNQ Test. The CRIQ lab conducted a dynamic gas production test according to BNQ/CAN 0413-220. Immediately upon receipt of a sample, a 20-g solids per liter suspension was prepared in water with no equilibrium period prior to initiating the test. The suspension was fully oxygenated and the volume of gas production was measured for 18-24 h

at 25°C. Evolved CO_2 was not measured directly: CO_2 was trapped in the reactor and the difference in chamber pressure was converted to an equivalent of oxygen on a mol basis of 1:1. Hourly results were converted to O_2 consumption and both O_2 consumption and respiration rate were plotted over time (respiration graphs shown in Appendix 1).

MOECC BOD Test. A modified BOD test was done by the MOECC Laboratory Services (BOD3182) by making a series of dilute compost slurries (from 10 mg to 250 mg of moist compost in 100 g distilled water) and tested within 7 days (held at room temperature). The three tests were run in static vessels in a 300 mL oxygenated buffer for 5 days in the dark at 20°C (without stirring). DO readings were taken using an oxygen electrode with depleted O_2 levels remaining between 35 to 75% of saturated levels. Units reported as mg O_2/L were converted to those of the Ontario quality standards (i.e. mg O_2/kg volatile solids dry wt (i.e. OM/ hr).

Modified BOD Respiration Test (Paul Arnold, Acadia University).
The test is an original test, modified from the standard biological oxygen demand (BOD) test traditionally used for wastewaters. Fifty grams of compost was placed in 19 L of tap water in 20 L carboy and fitted with a stir bar, an optical dissolved oxygen (ODO) probe and an aeration hose attached to a fish tank aerator. With constant stirring, the suspended compost slurry was incubated at 25°C continually for 3 days and aerated for 30 min every 8-hrs (or 1 hour every 4 hours for immature samples). O_2 levels were maintained above 2 mg/L. ODO readings were plotted over time and the three slopes for each 8-hr interval (six slopes for each 4-hr interval for immature samples) were averaged to calculate O_2 consumption rates per g organic matter (dry weight) per hr for reporting compost respiration.

5. RESULTS

The various test results were compiled and compared; those results are summarized in Table 3 while Figure 4 directly compares the results between the CRIQ and A&L Labs. In addition to comparing the results from established labs with those from Dr. Arnold's respiration test (summarized in Table 3), a number of alternative respiration tests were also completed on the same samples from the Ontario Ministry of Environment by Dr. Arnold. These tests, listed in the Test Methods for the Examination of Composting and Compost (TMECC), in addition to tests with the Arthur Respirometer, provide further evidence of the wide range of results produced from tests that are intended to be similar.

The test methods and a brief description follows:

i) TMECC 05.08-A: Specific Oxygen Uptake rate (SOUR)
The test measures the change in the oxygen content of the headspace above 250 mL of pre-incubated compost in a single test at 34^{o}C for 90 minutes.

ii) TMECC 05.08-B: Carbon Dioxide Evolution Rate
The test measures the four-day average of carbon dioxide released from a beaker containing 25 g of pre-incubated sample that is absorbed in an adjacent beaker of sodium hydroxide solution, both of which are enclosed in a 4 L jar at 34^{o}C.

iii) TMECC 05.08-D: Dewar Self-Heating Test
The test measures the temperature differential between 2 L of a pre-conditioned compost sample in an insulated thermos, and the ambient temperature, over a 5-10 day period.

iv) TMECC 05.08-E: Solvita Maturity Index
The test estimates the carbon dioxide and ammonia released through a colourimetric reaction from a sample in a half-filled sealed 250 mL jar over a 4-h period.

v) Arthur Respirometer
Carbon dioxide, released from an aerated water-compost slurry at 25^{o}C, is absorbed in potassium hydroxide in a closed loop. The loss of carbon dioxide creates a slight vacuum that increases over time to produce a rate of respiration. This method is employed by the CRIQ.

The carbon dioxide evolution, SOUR and respiration tests were run over a number of days to determine the variation based upon time.

Table 3. Compost maturity results – data from MOECC and Acadia University (dry wt basis)

COMPOST SITE ID	Compost Status	Days Curing[a]	H_2O% (wet wt)	OM % (dry wt)	A&L Solvita[g]	MOE-BOD (mg O_2/ kg OM/h)	A&L CO_2 Evolution (mg CO_2 –C / g OM/d)	A&L CO_2 Evolution (mg O_2 / kg OM/h)	CRIQ Respirometer (mg O_2/ kg OM/h)	Dr. Arnold's Respiration Test[e] (mg O_2/ kg OM/h)
Site H (Dec 9, 2013)										
C1	Highly Mature[b]	90	48	69	7	24	nd		200	271[f]
C2	Highly Mature[b]	90	50	66	6	23	nd		180	186[f]
Site A (Feb 3, 2014)										
C1	Immature	3	46	51	5	120	6.8	756	1370	604
C2	Mature[c]	59	39	46	7	15	2.6	289	320	334
Site B (Mar 10, 2014)										
C1		3	25	71	5	690	6.8	756	3780	5154
C2	20 days, Immature	20	23	67	6	460	4.8	533	3580	4158
Site C (Mar 31, 2014)										
C1	1 day, Immature	1	49	88	4	1020	8.6	956	810	1242
C2	3 months	84	38	80	6	124	5.5	611	998	1187
Site D (May 26, 2014)										
C1	1 day after time/temp	1	52	67	8	8	0.7	78	166	232
C2	3.5 weeks Mature[d]	25	47	62	8	26	1.2	133	236	266

a Curing is defined to begin on the first day the last portion of a compost batch is removed from the active phase (Ontario Compost Quality Standards, 2012)

b Compost had passed maturity tests weeks before this sample was taken

c Mature compost, considered mature by producer and ready to be tested to meet standards of finished compost.
d Site D sample will be considered mature by producer once screened in next day or two.
e First 24 h
f Consists of two 15-minute averages at 24 and 48 h after test began in triplicate
g Solvita index raw (1) to mature (8)

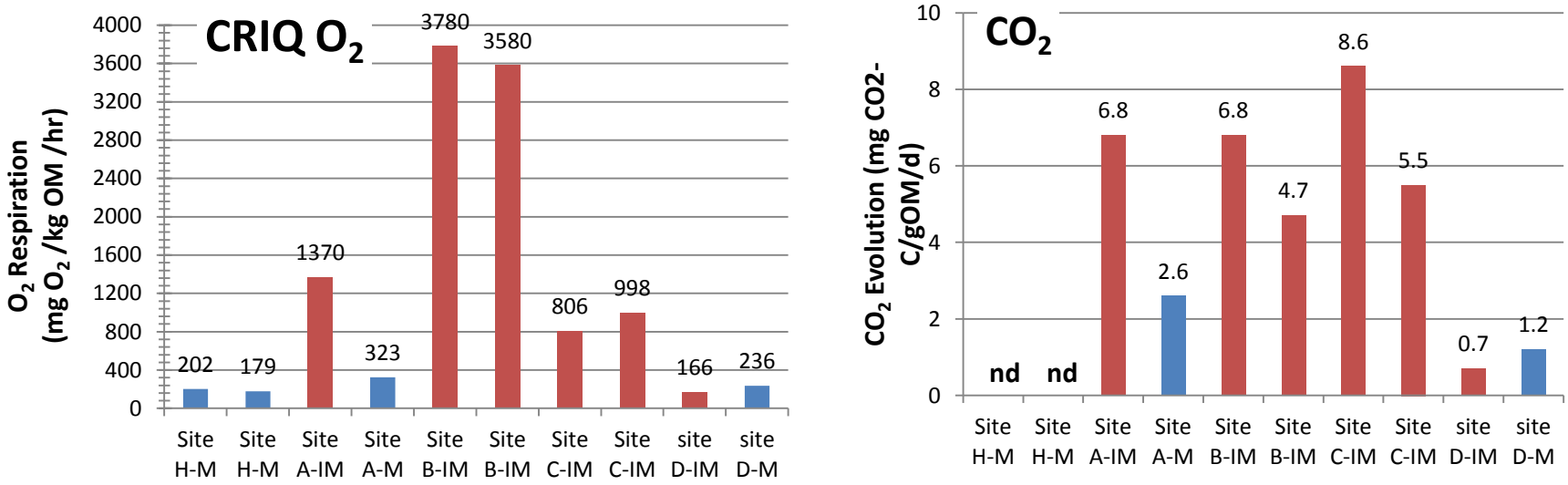

FIGURE 4. Respiration Data from Table 1 for BNQ O2 (CRIQ) and TMECC CO2 evolution (A&L Canada) for mature (blue, “M”) and immature (red, “IM”) composts.

Table 4. Compost maturity results from additional tests on Ontario samples.

COMPOST SITE/ID	Moisture % (wet wt)	OM % (dry wt)	pH	Solvita CO_2	Solvita NH_3	Solvita Overall	Dewar Reheat ΔT	Test Day	CO_2 Evolution (mg O_2/kg OM/h)	SOUR (mg O_2/kg OM/h)	Respirometer (mg O_2/kg OM/h)
Site A (Feb 3, 2014)											
C1	44 45	45 51	8.1 8.2	2-3 2-3	4 4	2.5 2.5					
C2	38 35	42 48	8.3 8.5	6 5-6	3 3	5 4.5					
Site B (Mar 10, 2014)											
C1	25 26	72 72	7.2 7.2	5 6	5 5	5 6	43°C (day 4)	1 2 3 4	197 412 226 168	323 118 104 277	649 (160 min) 1752 (120 min) 2428 (120 min) 1134 (120 min)
C2	21	66	6.2 6.1	8	5	8	1.5°C (day 4)	1 2 3 4	20 - 26 30	64 -	7.1 (262 min) 2416 (173 min) 2191 (165 min) 1366 (158 min)
Site C (Mar 31, 2014)											
C1	53	84	5.8				49°C (day 2)	1 2 3 4	672 1294 1926 1323	2736 2090 1078 1425	1775 (145 min) 577 (144 min) 1250 (111 min) 1092 (125 min)
Site D (May 26, 2014)											
C1	55	69		8	5	8	2°C (day 6)	1 2 3 4	84 75 15 22	235* 87 71 107*	51 (180 min) 29 (150 min) 31 (150 min)
C2 mature	49	59		6 5	5 5	6 5	5.5°C (day 2)	1 2 3 4	64 70 48 37	237 176 139 108	40 (310 min) 83 (200 min) 33 (160 min) 57 (215 min)

*Not reduced by blank

6. OBSERVATIONS & CONCLUSIONS

Clearly, the high degree of variation in the test results of identical samples underscores the original premise—that the acceptance of the wide variety of compost maturity test results will continue to produce confusion and uncertainty within the regulatory regime as well as within the industry. From the test results reported, a number of observations can be made:

- Of the original test methods, the CRIQ-BNQ respiration test appeared to give the best performance with respect to clearly failing immature composts and passing mature compost with a high level of resolution to differentiate between the levels of maturity. The shortcomings with this test were its short duration (<24h) and absence of acclimatization period; it gave one false pass (of 7-d old steaming hot compost).
- The modified BOD test of Dr. Arnold's is a simpler respiration test is still comparable to the CRIQ's respiration test, averaging slightly higher results which indicate a more sensitive test.
- The CRIQ test and P.Arnold's respiration test, typically produce respiration rates higher than alternative tests.
- The CO_2 evolution test performed well in general – failing immature compost for the most part- but the resolution power was much lower and there are aspects that need to be better standardized for accuracy. The experience of the lab conducting the CO_2 test was a big factor: a second lab had poor performance (falsely passing all immature samples by the CO_2 evolution test), likely because they lacked experience conducting this test.
- The BOD test significantly under-estimated product maturity.
- The Solvita test appeared to have a less discriminatory power and have a number of shortcomings. It is a good field tool but not good for demonstrating compliance. This view was also shared by TMECC and other jurisdictions.
- There was still one immature sample (a very fresh steaming hot sample) that was able to "falsely pass" all of the tests, possibly due to sampling timing and conditions or the fact that the test was not of a long enough duration to allow the microbes to acclimatize and begin the consumption of the available organic matter.
- Higher respiration rates are truer, because oxygen consumption or carbon dioxide production cannot be falsified, while lower rates can be due to tests that don't provide for supportive conditions or aren't sensitive enough.
- In comparing the results of different maturity tests, the difference between less accurate and more accurate tests will be more obvious with less mature products; the more mature the product, the closer all tests will be to producing similar results.
- We should be clear to establish the difference between measuring carbon dioxide production and oxygen consumption because the two are not typically equal. When we measure oxygen consumption, we measure the oxygen used to make carbon dioxide as well as the oxygen used to make oxidized products of other elements such as NO_3 and SO_4.

For instance, the oxidation of sludge without nitrification is:
$C_{10}H_{19}O_3N + 12.5O_2 \rightarrow 10\ CO_2 + 8H_2O + NH_3$

And with nitrification is:
$C_{10}H_{19}O_3N + 14.5O_2 \rightarrow 10\ CO_2 + 9H_2O + NO_3^- + H^+$

Although the amount of CO_2 is the same, the amount of oxygen consumed is higher by 2 mols.

Therefore, if we want to measure total oxygen consumption in terms of carbon dioxide production, we would have to multiply the carbon dioxide production by a factor of 14.5/12.5=1.16.

Unfortunately, the present conversion between the oxygen limit of 400 mg O_2/kg OM/h and the carbon dioxide limit of 4 mg CO_2-C/g OM/day (which is actually an equivalent carbon dioxide limit of 3.6 mg CO_2-C/g OM/day if the O_2:CO_2 ratio is 1:1) does not recognize this difference and in fact magnifies the difference further. If the established oxygen consumption limit is 400 mg O_2/kg OM/h, then the equivalent carbon dioxide limit would be 3.1 mg CO_2-C/g OM/day if an allowance for additional oxidized products were included:

$$\frac{400\ mg\ O_2}{kg\ OM/h} * \frac{12\ g/mol}{32\ g/mol} * \frac{24\ h}{day} * \frac{1\ kg}{1000\ g} * \frac{1\ mol\ O_2\ (without\ nitrification)}{1.16\ mol\ O_2\ (with\ nitrification)} = 3.1\ mg\ CO_2\text{-}C/g\ OM/day$$

- Assuming all respiration and compost maturity equipment is functioning properly for all tests, higher oxygen consumption or carbon dioxide production values are always presumed to be more accurate.
- Unfortunately, all respiration tests are not “fail-safe” with a “safe” respiration reading being higher than it actually may be. Less accurate tests will inevitably produce respiration results lower than they actually are thereby passing a sample that may fail with more accurate tests.
- Although the Dewar (re-heat) test is usually a reliable means of indicating compost activity (if sufficient time is provided), it cannot easily translate to a respiration rate (a doubling of the re-heat temperature does not equal a doubling of the respiration rate).
- Regardless of which test is used, all tests (including the CRIQ respiration test) **have** to be run until at least the respiration rate begins to decline (although the decline may only be a local maximum and it may take considerably more time to produce an overall maximum if the sample is microbiologically unhealthy).
- The practice of requiring compost samples to be chilled and sealed in plastic bags for delivery prior to respiration tests should be questioned. The unnatural cooling and suffocation that occurs places the microorganisms in an unhealthy state that usually requires a longer test period before the maximum respiration rate is reached. Placing samples in uncooled, breathable plastic bags will not significantly reduce the respiration rate response and will definitely produce a result sooner than if the samples are cold and anaerobic upon delivery.
- Regardless of which test is used, at least a common temperature should be established among all tests.

7. FURTHER WORK

At present, the modified BOD test has been adopted in the BNQ's draft revisions for the testing of compost maturity. It is presently under public review with comments concerning the adoption of the test due in the fall of 2015.

If the test is accepted and proves to be more sensitive, the adoption of a more accurate test, especially a test that measures oxygen consumption (which is always higher than the corresponding carbon dioxide production due to the additional oxidation of non-carbon compounds) could be cause to reassess the existing threshold of 400 mg/kg OM-h. Although it is arguable that certain, less sensitive receiving environments could withstand less mature product, the establishment of different rates for different applications would be very difficult to control from an administrative perspective. Perhaps the threshold could be changed, but it should remain a single number and it should be understood how the existing limit was established before any changes are made.