



Creating markets for recycled resources

## Develop standard laboratory stability test

**Project code: ORG0020**

**Date of commencement of research: September 2004**

**Finish date: November 2004**

**Interim report – statistical analysis**

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**Published by:**

**The Waste & Resources Action Programme**

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**Date (published)**

# Objectives

To conduct statistical analysis of Solvita tests results obtained by WRAP and provide:

- The ranges of stability/maturity found for each test
- The interpretation of the results for each test
- Correlation between tests
- Variance between duplicates

# Compost samples

The compost samples that were used are described in Table 1. The samples had a range of age since window formation from 5 to 23 weeks. Three windrows were sampled from two sites rather than two windrows from three sites recommended in the site sampling protocol.

**Table 1 Compost samples**

Code	Screening	Windrow started	Compost sampled	Age
<b>R3-GP</b>	Unscreened	23/04/04	28/06/04	9 weeks
<b>R16/1-GP</b>	0 – 20 mm	16/01/04	28/06/04	23 weeks
<b>R4-GP</b>	Unscreened	14/05/04	28/06/04	6 weeks
<b>W3 -WT</b>	0 – 20 mm	01/03/04	29/06/04	17 weeks
<b>W7/8 -WT</b>	unscreened	24/05/04	29/06/04	5 weeks
<b>W1/2-WT</b>	0 – 40 mm	16/01/04	29/06/04	23 weeks

## Protocols

Sampling and testing protocols were provided to the contractors. Direct Labs and SAC performed respiration tests according to 'Standardised method for the determination of compost stability by measurement of evolved carbon dioxide', a test developed by ADAS for WRAP. The other laboratory used their own methodology.

Solvita tests were to be carried out by all laboratories in accordance with the Solvita protocol. The samples were to have been pre-prepared by the labs in accordance to EN 13040 Section 8.5 (i.e. material passed through a 20 mm sieve). The samples were pre-conditioned by all laboratories apart from Woods End as the pre-conditioning is not part of their protocol.

The labs were also to record bulk density, pH and the temperature at which the test was conducted. The weight of each Solvita sample was to be recorded and the actual moisture content for the Solvita tested after adjustment (if necessary). Samples were to be tested in triplicate. Respiration was to be reported as mg CO<sub>2</sub>/g VS/day.

Data was provided from:

- Direct Labs
- SAC
- Woods End

Correlations were made between the data from Direct Labs and SAC as they were following the same methodology. Where possible, other correlations have been made.

### Direct Labs

Measurements were made on the compost as received, and moisture contents at testing for both Solvita and respiration. The weight of compost used per Solvita test varied between 39 g and almost 80 g, reflecting the various bulk densities of the composts.

## SAC

Incubation for respiration was carried out at 24.5°C on 100 g compost. Compost sample weights for the Solvita tests ranged from 53 g to 82 g. Both tests were carried out at the same moisture contents. SAC reported four suspect titres that could be removed from the respiration data.

## Woods End

The CONEG CO<sub>2</sub> evolution test was used to measure respiration rate of the compost samples. This was carried out at 34°C, some 9°C higher than the temperature used by ADAS. There is a commonly accepted relationship between biological activity and temperature, within this range, of a doubling in activity for every 10°C temperature increase. For comparison therefore, The Woods End data on respiration have been multiplied by a factor of 0.5 x 1.1 to account for the 9°C difference.

## Statistical methods

The stability tests across all laboratories have been analysed using Analysis of Variance (ANOVA) using the mean test result for each sample at each laboratory as a single sample of compost was provided to each laboratory with three replicate tests performed on each. Least significant differences (LSD) at P= 0.05 are shown to compare the mean values between samples and laboratories.

The results from Direct Labs and SAC have also been correlated using ANOVA utilising the replicate data supplied.

Regressions have been performed between test method results from Direct Labs and SAC.

## Compost properties

### Compost pH

There appears to be a difference in method for pH testing between Direct Labs and SAC as the range of pH at Direct Labs was 8.3 to 8.9 and at SAC was 7.0 to 7.4. At Woods End the pH range was 7.8 to 8.1.

### Loss on ignition (LOI)

Volatile solids were measured by each laboratory as loss on ignition and reported on a dry matter basis, see Table 2. The range of LOI measured for each sample was over 10% in two cases and LOI tended to be lower in the measurements made on the SAC samples.

**Table 2 Loss on ignition % DM**

	<b>Direct Labs</b>	<b>SAC</b>	<b>Woods End</b>	<b>Max</b>	<b>Min</b>	<b>Mean</b>
<b>R3GP</b>	41.8	31.0	34.0	41.8	31.0	35.6
<b>R16\1GP</b>	31.8	26.6	30.7	31.8	26.6	29.7
<b>R4GP</b>	31.7	24.5	33.4	33.4	24.5	29.9
<b>W3WT</b>	42.0	38.1	37.7	42.0	37.7	39.3
<b>W7\8WT</b>	34.6	30.0	40.4	40.4	30.0	35.0
<b>W1\2WT</b>	28.5	26.1	29.4	29.4	26.1	28.0
<b>Mean</b>	35.1	29.4	34.3			

### Moisture content

Moisture content of the samples at the time of analysis was measured as shown in Table 3. There is good agreement between laboratories and all moistures are between 40 and 60%. Moisture can limit microbial activity when it is low.

**Table 3 Moisture content %**

	<b>Direct Labs</b>	<b>SAC</b>	<b>Woods End</b>	<b>Max</b>	<b>Min</b>	<b>Mean</b>
<b>R3GP</b>	47.9	45.3	46.7	47.9	45.3	46.6
<b>R16\1GP</b>	49.4	49.7	50.9	50.9	49.4	50.0
<b>R4GP</b>	46.2	46.1	44.5	46.2	44.5	45.6
<b>W3WT</b>	50.8	51.4	52.3	52.3	50.8	51.5
<b>W7\8WT</b>	42.7	44.7	45.7	45.7	42.7	44.4
<b>W1\2WT</b>	49.1	49.8	53.8	53.8	49.1	50.9
<b>Mean</b>	47.7	47.8	49.0			

## Stability tests

Each laboratory conducted tests using the Solvita kits. Pre-conditioning is not part of the Solvita protocol and so was not carried out at Woods End. In almost all cases at Direct Labs and SAC, the Solvita scores were identical for each replicate. Only single results were reported from Woods End.

Direct Labs and SAC carried out respiration tests according to 'Standardised method for the determination of compost stability by measurement of evolved carbon dioxide', a test developed by ADAS for WRAP. Woods End used the CONEG-USA CO<sub>2</sub> method and the data has been transformed to units equivalent to those used by Direct Labs and SAC from mg CO<sub>2</sub>-C/g VS/day to mg CO<sub>2</sub>/g VS/day (single result reported).

### Solvita CO<sub>2</sub>

There was close agreement between Direct Labs, SAC and Woods End in the Solvita CO<sub>2</sub> scores.

**Table 4 Solvita CO<sub>2</sub>**

	Direct Labs	SAC	Woods End	Max	Min	Mean	Age
R3GP	4.7	6	5	6.0	4.7	5.2	9 weeks
R16\1GP	7	7	7	7.0	7.0	7.0	23 weeks
R4GP	5	5	5	5.0	5.0	5.0	6 weeks
W3WT	7	6	6	7.0	6.0	6.3	17 weeks
W7\8WT	6	6	6	6.0	6.0	6.0	5 weeks
W1\2WT	7	7	6	7.0	6.0	6.7	23 weeks
Mean	6.1	6.2	5.8				
LSD (0.05) between samples =			0.79	& between labs =		0.56	

### Solvita NH<sub>4</sub>

There was relatively good agreement on Solvita NH<sub>4</sub> although on three samples Woods End was slightly lower.

**Table 5 Solvita NH<sub>4</sub>**

	Direct Labs	SAC	Woods End	Max	Min	Mean	Age
R3GP	5	5	5	5.0	5.0	5.0	9 weeks
R16\1GP	5	5	5	5.0	5.0	5.0	23 weeks
R4GP	4.3	4	3	4.3	3.0	3.8	6 weeks
W3WT	5	5	4	5.0	4.0	4.7	17 weeks
W7\8WT	5	5	4	5.0	4.0	4.7	5 weeks
W1\2WT	5	5	5	5.0	5.0	5.0	23 weeks
Mean	4.9	4.8	4.3				
LSD (0.05) between samples =			0.62	& between labs =		0.44	

## Solvita Index

For Direct Labs and SAC, the Solvita indices were the same or, on two samples, only one unit apart. Woods End results were also generally consistent with those of Direct Labs and SAC.

**Table 6 Solvita Index**

	Direct Labs	SAC	Woods End	Max	Min	Mean	Age
R3GP	4.7	6	5	6.0	4.7	5.2	9 weeks
R16\1GP	7	7	7	7.0	7.0	7.0	23 weeks
R4GP	5	5	4	5.0	4.0	4.7	6 weeks
W3WT	7	6	6	7.0	6.0	6.3	17 weeks
W7\8WT	6	6	6	6.0	6.0	6.0	5 weeks
W1\2WT	7	7	6	7.0	6.0	6.7	23 weeks
Mean	6.1	6.2	5.7				
LSD (0.05) between samples =			0.82	& between labs =		0.58	

## Respiration

Utilising the data that included all of the replicates at SAC gave the results shown in Table 7a. Omitting the four replicates gave the results shown in Table 7b. SAC recorded greater CO<sub>2</sub> evolution than Direct Labs. The Woods End method gave a much greater reading than from the other three laboratories due to temperature and so a correction factor was used. Statistics refer to the adjusted data.

**Table 7a CO<sub>2</sub> evolution mg CO<sub>2</sub>/g VS/day – all replicates**

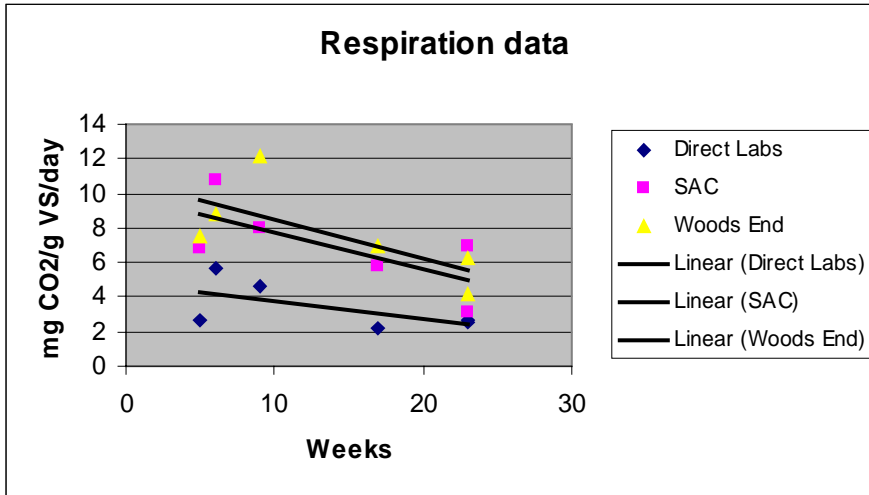
	Direct Labs	SAC	Woods End original	Woods End adjusted	Max	Min	Mean	Age
R3GP	4.6	8.0	22.1	12.2	12.2	4.6	8.3	9 weeks
R16\1GP	2.7	3.1	7.7	4.2	4.2	2.7	3.3	23 weeks
R4GP	5.7	10.8	16.0	8.8	10.8	5.7	8.4	6 weeks
W3WT	2.2	5.7	12.6	6.9	6.9	2.2	5.0	17 weeks
W7\8WT	2.7	6.8	13.7	7.5	7.5	2.7	5.7	5 weeks
W1\2WT	2.5	7.0	11.4	6.3	7.0	2.5	5.2	23 weeks
Mean	3.4	6.9	13.9	7.7				
LSD (0.05) between samples =			2.47	& between labs =		1.75		

**Table 7b CO<sub>2</sub> evolution mg CO<sub>2</sub>/g VS/day – omitting four SAC replicates**

	Direct Labs	SAC	Woods End original	Woods End adjusted	Max	Min	Mean	Age
R3GP	4.6	8.0	22.1	12.2	12.2	4.6	8.3	9 weeks
R16\1GP	2.7	3.1	7.7	4.2	4.2	2.7	3.3	23 weeks
R4GP	5.7	10.2	16.0	8.8	10.2	5.7	8.2	6 weeks
W3WT	2.2	4.5	12.6	6.9	6.9	2.2	4.5	17 weeks
W7\8WT	2.7	6.0	13.7	7.5	7.5	2.7	5.4	5 weeks
W1\2WT	2.5	5.2	11.4	6.3	6.3	2.5	4.7	23 weeks
Mean	3.4	6.2	13.9	7.7				
LSD (0.05) between samples =			2.28	& between labs =		1.61		

Graph 1 shows how the trendlines for each laboratory against age of compost. The ADAS data set and trend line are lower than those for SAC and Woods End adjusted.

**Graph1 Respiration data**



**Comparison between Direct Labs and SAC results**

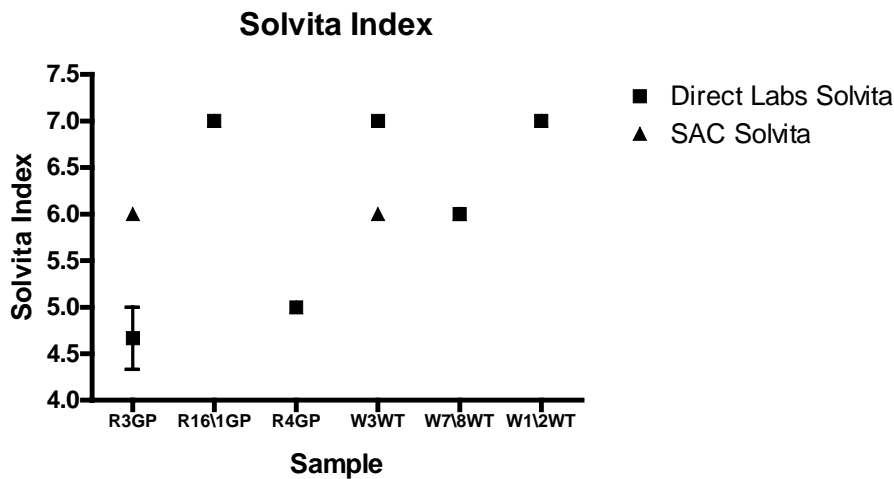
An analysis of the data generated by Direct Labs and SAC utilising the replicate data was carried out.

**Solvita**

Source of Variation	Degrees of Freedom	Sum of Squares	Mean square	Probability
Laboratories	1.0	0.02778	0.02778	not significant
Samples	5.0	21.47	4.294	< 0.0001
Interaction	5.0	4.139	0.8278	< 0.0001
Residual (error)	24.0	0.6667	0.02778	
Total	35.0	26.31		

The data showed that there was good agreement between laboratories and that the differences between the samples were significant and detected by the method. (In graph 2 where only one symbol is seen for a sample, both laboratories had the same result).

**Graph 2 Solvita index**

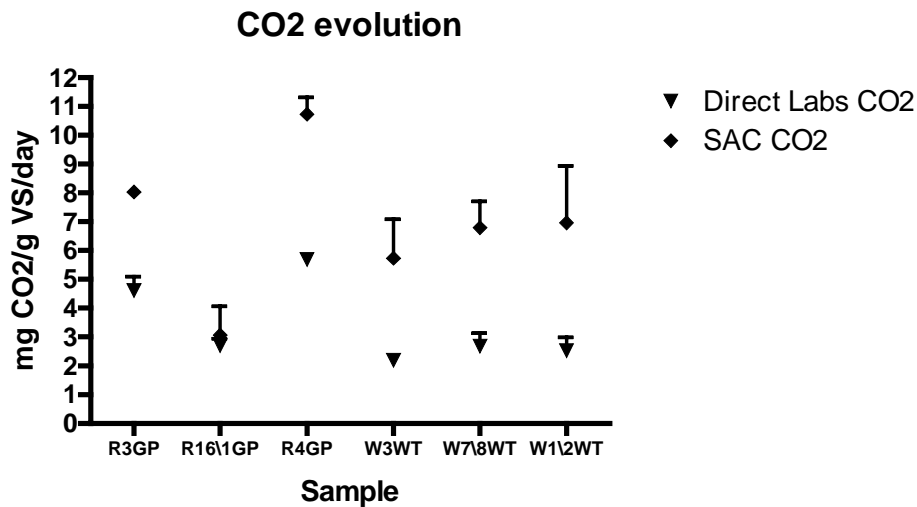


## Respiration

Source of Variation	Degrees of Freedom	Sum of Squares	Mean square	Probability
Laboratory	1.0	109.4	109.4	< 0.0001
Sample	5.0	105.8	21.16	< 0.0001
Interaction	5.0	20.20	4.041	not significant
Residual (error)	24.0	52.24	2.177	
Total	35.0	287.7		

The respiration data confirms that there were significant differences between the two laboratories in the results as well as there being significant differences between samples.

Graph 3 CO2 evolution



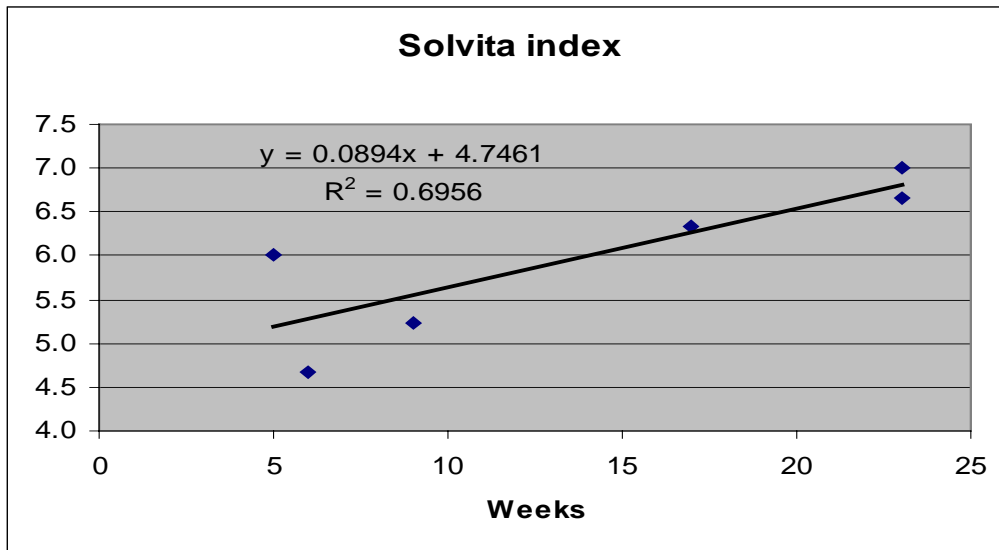
## Interpretation

Although there was a large range in ages of the compost samples, from five to 23 weeks, the activity of the compost was not great in the young samples. Activity decreases greatly in the first few weeks of composting and a greater range of activity could have been usefully incorporated by using a very young compost, at about two weeks old.

Moisture content can also limit the activity of the microorganisms and, although moisture was above 40% in the test samples, it would be useful in the future to test samples at between 50 and 60% moisture with pre-conditioning.

The Solvita tests did pick out the oldest compost samples as shown in Graph 4 (averages from all three laboratories). The five week old compost was the driest sample which may have resulted in a higher index than expected.

**Graph 4 Solvita index – all laboratories**



For respiration, measured as CO<sub>2</sub> evolution, the average data from all laboratories show the trend of decreasing microbial activity with age of compost (Graph 5). Again the five week old sample seems to be lower in activity than expected, possibly due to low moisture content. However, there are differences in test results between the laboratories for the samples and the reason for this needs to be determined if the method is to become a standard.

**Graph 5 CO<sub>2</sub> evolution – all laboratories**

