

December 21, 2009

RD 2

Report 2nd Round 2009

Robert Miller, PhD, Program Coordinator



Compost Analysis Proficiency Program

The 2nd Round of CAP 2009 had seventeen labs enrolled, with thirteen submitting results for Tier I inorganic analyses and ten Tier II, inorganic + biological analyses. We would like to thank all participating laboratories for their cooperation in completing these analyses in a timely manner.

The 2009 the proficiency data report list results by analysis followed by compost material. For 2009 95% Confidence Limits (CL) based on the median $\pm 2.9 \times \text{MAD}$ is used to assess lab bias. Individual lab values exceeding this range are flagged for bias as ***L** or ***H**. This statistical approach is more robust to the influence of data set extreme values and conforms to 95% confidence limits based on two standard deviations (s) for normal populations. The data analysis report lists: the number of lab results reported; minimum value reported, maximum value reported; median value of reported; 95% Confidence Limits; overall reproducibility (R_d) across laboratories; individual reported lab values; repeatability (R_p) of a single lab value; and mean lab value reported. Lab R_p values exceeding three (3) times the R_d , are marked with a colored ***P** adjacent to the R_p results indicating the individual lab precision exceeded the database median for the compost analysis.

Compost Materials

SRC 2009 - D: represented by sub samples *SRC-0910*, *SRC-0911* and *SRC-0912* is a brewery compost obtained from Pioneer Sand and Gravel, labeled as Premium 1, Fort Collins, CO.

SRC 2009 - E: represented by sub samples *SRC-0913*, *SRC-0914* and *SRC-0915* was a commercial compost landscape material marketed by Lowes, Loveland, CO.

SRC 2009 - F: represented by sub samples *SRC-0916*, *SRC-0917* and *SRC-0918* was a sheep manure feedstock material obtained from Cactus Hill Feedlot, Windsor, CO.

Selected Points of Interest:

- Compost repeatability results indicate N-combustion ranges from 10.3% to 18.5% of the median for samples ranging from 0.862 to 2.57% N over the past 5 years.
- CAP sample SRC 2009-F, the ammonium-nitrogen median concentration was 1840 mg kg⁻¹.
- Nickel results indicate a consistent high bias trend for 1 of 9 reporting labs for all three compost materials.

CAP Proficiency Results

Compost total solids median values were 70.3, 71.42, and 70.2% for the three compost materials, respectively. Intra-lab reproducibility (R_d) values for the 105° C for 2.5 hr ranged from 0.40 to 0.69%, indicating a high degree of homogeneity for all three compost materials. Loss on Ignition (LOI) medians ranged from 33.6% to 50.2% with reproducibility (R_d) values ranged from 1.8 to 4.2%. Based on four reporting labs median CEC values, ranged from 11.3 to 24.7 cmol kg⁻¹ across the three compost materials. Saturated paste pH median values for the three materials ranged from 7.82 - 8.40. Sample **SRC-2009-F** had high saturated paste EC value, with a median of 21.6 dS m⁻¹.

Median results reported for EC 1:5 extract ranged 4.6 to 12.1 dS m⁻¹ across the three samples, with R_d values ranging from 2.1 to 4.0%. Confidence Limits (CL) across materials ranged from 6.7 - 56.9% of the median for Ca, Mg, Na. 1:5 PO₄-P concentrations ranged from 5.1 - 51.5 mg L⁻¹ with an average R_d value of 5.0% for the three materials. Extract 1:5 Cl median results was 2248 mg L⁻¹ for sample **SRC-2009-F**.

Four labs provided TKN results and fifteen for N-combustion. Comparison of nitrogen method median values indicates a low bias that ranges from 0.09% to 0.20% for TKN relative to N-combustion across the three samples. TKN R_d values ranged from 1.2 - 4.4% and 1.7 - 5.2% for the N-combustion method for the three materials. Total organic carbon (TOC) median values were 16.5%, 24.0% and 25.0% TOC respectively with the 95% CL averaging 20% TOC. Median C:N ratios ranged 12.2 to 17.8, the highest for sample **SRC-2009-F**. Three laboratories reported total sulfur (S) by combustion, median values which ranged from 0.23 to 1.14% S for the three materials.

*SRC 2009-F, NH₄-N
was 1840 mg kg⁻¹*

Nitrate and ammonium results were provided by seven and six labs respectively. NO₃-N median results ranged from 4.7 to 508 mg kg⁻¹ and CL was 95% of the median for sample **SRC-2009-E**. NH₄-N median values ranged from 33.1 to 1836 mg kg⁻¹ with CL for NH₄-N averaging 150% of the median. NH₄-N/NO₃-N ratio, calculated for each lab, ranged from 0.60 to 36.0. An average of fifteen labs provided results on total P, K, Ca, Mg and Na. Phosphorus medians concentrations were 0.30, 0.28 and 1.19%, respectively. R_d values averaged 3.5% for P. Potassium median concentrations ranged from 1.6 to 3.4%. Across the three materials Ca median concentrations ranged from 3.0 to 4.2% and Mg values ranged from 0.56 to 1.03%.

Compost Barium median concentrations ranged from 88 - 137 mg kg⁻¹, Cu 9.2 - 28.9 mg kg⁻¹, and Zn 30.8 - 148 mg kg⁻¹ across the three materials. Strontium median concentrations ranged from 198 - 465 mg kg⁻¹; Pb from 2.9 - 8.2 mg g⁻¹; and Ni from 4.1 - 13.0 mg kg⁻¹. Cadmium median concentrations ranged from 0.14 - 0.90 mg kg⁻¹, Co 1.5 - 3.9 mg kg⁻¹ and Cr 4.4 - 11.9 mg kg⁻¹ across the three materials. Arsenic median values ranged from 2.0 - 5.1 ug kg⁻¹ for each of the three samples. Generally CL were greater than 50% of the median for elements: Al, Be, Cd, Cr, Li, Mo, Pb, Sb, V, Hg, As, and Se which is associated with concentrations approaching the method detection limit.

Generally R_d values for Al, B, Ba, Be, Ca, Co, Cu, Fe, K, Li, Mg, Mn, Na, P, Sr, V, and Zn were less than 5% across compost materials.

Tier II results for the 2009 CAP RD2 contents were performed by eight labs, dependent on the test. Sieve size classification (>9.5 mm) were less than zero for two of the three samples. All three materials were blended, but not pulverized or screened, and thus represented compost samples closely representing real world materials. (*Continued Page 4*)



www.dur.ac.uk/geochem/www/group/cleanlab.htm

N-Combustion Precision

Over the past five years the CAP program has compiled an extensive lab proficiency database on the Nitrogen Combustion (Dumas) analysis of compost samples. Data on N-Combustion repeatability and reproducibility were calculated in accordance with International Standard Organization (ISO) 5725-2 (1994) from the results of eleven CAP proficiency rounds conducted from 2006 through 2009 based on data submitted by 12 - 18 laboratories across North America. Listed in Table 1 are the Total Solid and Dumas Nitrogen results of four of the eleven samples evaluated in the CAP Program, listed from high to low total nitrogen content. Listed for Dumas Nitrogen is the median content, within-lab standard deviation (S_r) within-lab repeatability (r), the between-lab standard deviation (S_R), and the between-lab Reproducibility (R). S_r represents the within lab repeatability calculated from the three replicates and r is the repeatability 95% confidence interval. S_R represents reproducibility across laboratories (outliers removed) and R represents the reproducibility 95% confidence interval.



pubs.usgs.gov/of/2002/of02-371/

These results show that at a compost Dumas Nitrogen concentration of 2.57% repeatability was $\pm 10.3\%$ of the median content, whereas at the lowest concentration of 0.862% nitrogen repeatability was $\pm 18.5\%$ of the median concentration. CAP reproducibility was $\pm 22.8\%$ of the median content at the highest concentration and $\pm 49.1\%$ at the lowest sample Dumas nitrogen concentration. These results show method precision changes with concentration for the Dumas Nitrogen method analysis of compost.

This proficiency data will be incorporated in the N-Combustion method in the next revision of the TMECC methods manual which has begun by the US Compost Council.

Table 1. Precision Data for CAP Program Dumas Total Nitrogen Method

Sample	Total Solids Content	Dumas Nitrogen (%)				
		Content	Within lab Std	Within lab Repeatability ¹	Between Lab Std	Between Lab Reproducibility
	%	%	S_r	r	S_R	R
SRC 2008-A	52.7	0.862	0.057	0.160	0.151	0.423
SRC 2009-A	68.8	1.25	0.037	0.104	0.180	0.504
SRC 2009-C	51.2	1.72	0.041	0.114	0.111	0.308
SRC 2007-D	70.7	2.57	0.095	0.266	0.210	0.588

¹ International Organization for Standardization. Accuracy (trueness and precision) of measurement methods and results. Part 2. Basic method for the determination of repeatability and reproducibility of a standard measurement method. Report 5725-2 1994:42pp ISO Geneva.



Nickel Observations

ROBERT O. MILLER

Soil and Crop Sciences Dept
C 117 Plant Science Bldg
Colorado State University
Fort Collins, CO 80523

Phone: 970-686-5702

Fax: 970-686-5709

Email: rmiller@lamar.colostate.edu

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Observations for 2009 Round 2 indicate the three compost materials had a range in median nickel concentrations ranging from 4.1 to 13.0 mg kg⁻¹ (see Figure 1). Listed is the median (solid lines) and standard deviation (error bar) for each compost material. Sample **SRC 2009-D**, shows a wide distribution in Cu values ranging from 9.5 to 20 mg kg⁻¹, for labs #1 through #9 with confidence limits of ± 3.7 mg kg⁻¹. Lab #1 was found to have a low bias, for **SRC 2009-A**, whereas labs # 8 and #9 had a high bias. Lab #3 was inconsistent with a low bias on samples **SRC 2009-E** and **SRC 2009-F**. Generally standard deviation error bars across labs were higher for sample **SRC-2009-D** over the two compost samples. Lab #9 consistently had the highest error bars for all three materials. The high bias and error bars maybe associated with compost sample pulverizing equipment.

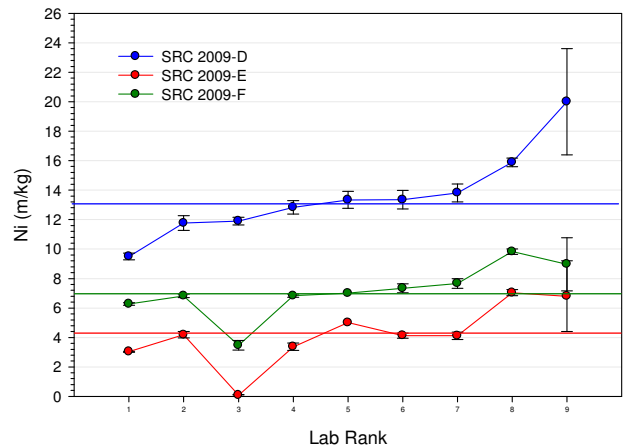


Figure 1. Ni for three compost materials, CAP 2009 RD2.

CAP Proficiency Results (Continued)

Eight laboratories provided results on seedling germination and growth. Median seedling cucumber emergence was 100, 91 and 5.0% for the three materials respectively, with R_d values averaging 3.5%. Poor seedling vigor was likely associated with the high EC, in particular sample **SRC-2009-F**. Total solids respirometry (% w/w basis) was provided by five labs with rates of 53.0 to 57%, and R_d values averaged 2.1% for sample **SRC-2009-E**. LOI OM respirometry values ranged from 31.8 - 50.6% with R_d values averaging 3.0%.

Carbon Dioxide Evolution I indicated a range of 0.26 - 2.30 (mg/gTS/d) across the three materials. SOUR has been deleted from the database as no data has been provided. Solvita Maturity Index data tests data was provided by no labs. Biological Available Carbon (BAC) and I and II was provided by one lab. Pathogen analyses was provided for Fecal Coliform Bacteria by an average of seven labs with a median value of 700 MPN/g for sample **SRC-2009-E**.



Source: swww.ianr.unl.edu/ianr/csas/graphic/turn1.jpg



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The 2009 CAP Program is supported through funding from the US Composting Council.