COMPARISON OF METHODS TO DETERMINE CARBON AND NITROGEN IN SOIL EXTRACTS

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Objectives

1. Compare carbon and nitrogen contents of 0.5M K_2SO_4 extraction measured by various analytical procedures
2. Determine the reliability and ease of use of these analytical devices

Methods*

Extractions

1. Soil were processed for soluble and microbial biomass C and N following Vance et al.1 using 0.5 K2SO4 , 1:1.5 soil/sol, and 3 day CHCl3 fumigation
2. Samples analyzed fresh or stored frozen 1-4yrs

Carbon Analysis

1. **COD**-Chemical Oxygen Demand digestion (CHEMetrics, Inc.) photometric measurement with a Beckman Spectrophotometer DU 7400 or an AquaFast Orion AQ4000
2. **TOC-**Direct combustion with platinum catalyst and NDIR for CO_2 on a Lachat IL550 TOC/TN

Nitrogen Analysis:

1. **TPN**-Microwave[2] or autoclave[3] assisted total persulfate nitrogen digestion followed by NO_3^- determination with ALPKEM continuous flow injection analysis
2. **TN**-Direct combustion and detection of total N with an inline NO electrochemical detector (ECD) on the IL 550 TOC/TN

Statistical Comparisons:

Pearson correlations and simple linear regressions were calculated on soluble C and N in mg C or N g^{-1} soil and mg Microbial biomass C or N (Kc =0.4 Kn =0.45) g^{-1} soil

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Results

Table 1. Simple Linear regression and Pearson's Product Moment Correlation (PPM) coefficients for comparison of mg C g^{-1} soil determined by COD vs. TOC, mg Microbial biomass carbon (MBC) g^{-1} soil determined by COD vs. TOC, mg N g^{-1} soil determined by TPN vs. TN through direct combustion, and mg microbial biomass nitrogen (MBN) g^{-1} soil determined by TPN vs. that determined by TN.

Variables								
Explanatory	Dependant	Year	Slope	Intercept	R^2	PPM	F*	N
COD	TOC	2002	0.5675	0.0272	0.9332	0.9660	5296.46	381
		2003	0.8524	0.0082	0.7906	0.8891	1438.12	383
		2004	0.8536	0.0121	0.9284	0.9636	4903.14	380
		2005	0.7485	0.0076	0.8961	0.9466	3216.97	375
		2006	0.8219	-0.0009	0.8962	0.9467	3290.30	383
		ALL	0.7116	0.0233	0.8352	0.9139	9626.93	1902
MBC	MBC	2002	0.2061	0.3706	0.0837	0.2893	17.08	189
		2003	0.1517	0.4008	0.0301	0.1736	5.87	191
		2004	0.8105	0.0215	0.5318	0.7293	211.31	188
		2005	0.4088	0.2820	0.1826	0.6150	41.22	183
		2006	0.4933	0.2893	0.1910	0.4370	44.62	191
		ALL	0.2732	0.3479	0.1807	0.4251	207.32	942

Variables								
Explanatory	Dependant	Year	Slope	Intercept	R^2	PPM	F*	N
TPN	TN	2002	0.7956	0.0035	0.9174	0.9578	4220.73	382
		2004**	0.3052	0.0084	0.2681	0.5178	67.40	186
		2005	1.1143	-0.0071	0.9324	0.9656	5134.79	374
		ALL	0.8881	-0.0002	0.9069	0.8523	9158.49	942
MBN	MBN	2002	0.5206	0.0359	0.3529	0.5940	101.97	189
		2005	0.7236	0.0410	0.3328	0.5769	89.80	182
		ALL	0.3947	0.0615	0.1732	0.4162	77.31	371

* Significance for all tests was $P<0.0001$ except for 2003 MBC which was $P = 0.0163$.
** TPN digestion and subsequent comparison were performed only on non-fumigated soil samples.

- **Pooling of fumigated (F) or unfumigated (NF) samples greatly improved fit of data to regression and correlation**
- **Intercept for direct C and N contents close to zero and slopes < or near 1 show methodologies used were comparable**
- **Low fit of correlation and regressions for the MBC and MBN by different methods was due to lack of lower values**

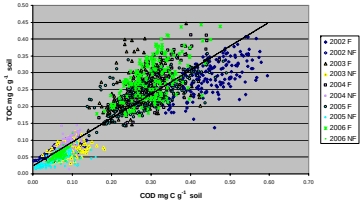

Figure 1. Comparison of total organic carbon of fumigated (F) and unfumigated (NF) samples measured by COD and TOC methods for five years of annual samples.

- **The good fit of the regression (black line) was dependant on including samples of low to high range in the calculation**
- **Greater variability in the data is evident at higher concentrations**

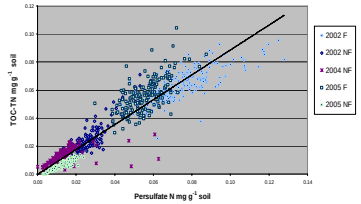

Figure 2. Comparison of total nitrogen of fumigated (F) and unfumigated (NF) samples measured by persulfate digestion and analysis of nitrate and TN by direct combustion for three years of data.

- **The nitrogen data showed much better regression both within year and for all years.**
- **Again greater variability occurred at higher concentrations**

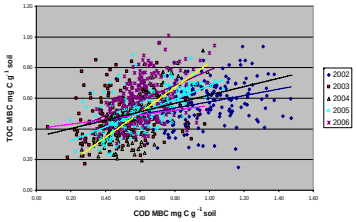

Figure 3. Comparison of microbial biomass carbon (MBC) determined from the subtraction of unfumigated from fumigated C measurements by COD or TOC analysis for five years of data.

- **The regressions by year (colored lines) show the great amount of variability in the data.**
- **We attribute low fit of the data to greater variation connected with calculating MBC from two (F and NF) separate samples.**

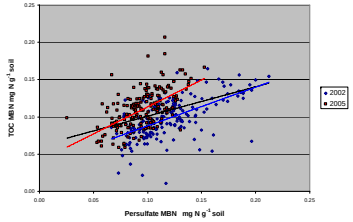

Figure 4. Comparison of microbial biomass nitrogen (MBN) determined from the subtraction of unfumigated from fumigated N measurements by persulfate digestion or TN analysis for two years of data.

- **The fit of the regressions (color lines indicate by year) to the data is somewhat better than that of the MBC regressions however also show the greater variability in calculations from two individual samples.**

Recommendations

- The tight regression lines passing through or near the origin point for the soluble C and N data indicate TOC or COD and TN or persulfate digestion produce comparable and reliable measurements of C and N in soil extracts.
- Evaluation of the MBC and MBN regression and correlation data suggests caution in interpreting data from mixed methodologies; treatment comparisons should be made with data produced from one method only.
- The direct combustion of extraction solutions is the more cost effective and less hazardous approach. Not only can C & N be obtained simultaneously, but production of caustic chemical waste, particularly as it applies to the COD method, is eliminated.

References

1. Vance, E.D., Brookes, P.C. and Jenkinson, D.S. 1987. An extraction method for measuring soil microbial biomass C. Soil Biol. Biochem., 19, 703-707.

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3. Cabrera, M.L. and M.H. Beare. 1993. Alkaline persulfate oxidation for determining total nitrogen in microbial biomass extracts. Soil Sci. Soc. Am. J. 57:1007-1012.

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