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Nutrient variability in manures: Implications for sampling and regional database creation

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ABSTRACT: The variability of manure nutrient levels within and across farms makes manure sampling and development of reliable tabular values challenging. The chemical characteristics of beef, dairy, horse, sheep, and chicken solid manures in Colorado were evaluated by sampling six to ten different livestock operations for each manure type and comparing the results to values found in the literature. Due to the semi-arid climate of Colorado, manure tends to be drier and have lower ammonium (NH_4-N) levels and higher phosphate (P_2O_5) and potash (K_2O) levels than those reported in the Midwest. Within-farm variability was assessed by analyzing ten sub-samples from each of nine manure sources. Coefficients of variation were calculated and the sample numbers necessary to achieve 10% probable error were determined. On average, about 25 sub-samples are necessary for nitrogen (N), phosphorus (P), and potassium (K) characterization of solid manures, but determining NH_4-N and nitrate (NO_3-N) concentrations requires over 100 sub-samples to form a representative sample, due to their relatively low concentrations. Data from Colorado, Utah, and New Mexico were combined to form a Mountain West Manure Database. The manure types, with a minimum of 72 farms represented in the database, have narrow confidence intervals. Until we have adequate sample numbers (>72 farms) to establish reliable table values based on local data for all manure types, manure sampling will be recommended.

Keywords: Manure sampling, manure variability, regional manure database

Land-grant universities throughout the United States recommend that farmers sample and analyze animal manure to determine its nutrient content prior to land application. Nutrient management recommendations are dependent on accurate manure nutrient information. Many universities provide table values for use when pro-

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ducers do not have good analyses of their own. But table values commonly used today are 15 to 20 years old and have been re-published so often, that it is often difficult to ascertain their original source (Rieck-Hinz et al. 1996). Few livestock producers actually do site-specific manure sampling, which has led us to question the rationale behind manure sampling.

At least three questions regarding the effectiveness of manure sampling and the use of table values need to be addressed. First, how variable are nutrient contents in manures and how many sub-samples would be required to achieve a representative sample? Second, can table values provide reasonable estimates when farmers do not have analyses from their own operations? Third, do regional differences in climate and management systems require us to develop state-by-state databases of manure nutrient contents?

There are few published data that address these concerns. Powers and Van Horn (2001) emphasized that nutrient excretion can be much more accurately predicted than nutrient concentrations in manure at the time of land application. This is due to variations in climate, storage and handling practices, and other site-specific influences.

Manure nutrient content is known to be variable (Rieck-Hinz et al. 1996), but the implications of that variability for sampling protocols have not been defined, except in a recent paper by Dou et al. (2001). Dou et al. (2001) collected serial samples from dairy, swine, and broiler poultry operations when manure was being loaded for field application. They found that when agitation was used prior to loading, coefficients of variation (CVs) were 6 - 8% within farm, and three to five sub-samples were adequate for a representative composite sample. When no agitation was used, CVs ranged from 20 - 30%, and at least 40 sub-samples were required.

Powers et al. (1975) found that beef manure nutrient concentrations varied about 10-fold for N and P and almost 20-fold for K. Lindley et al. (1988) reported that manure nutrient values can range from 50 - 100% of table values. Dou et al. (2001) also concluded that table values were problematic due to the variability of on-farm data. Variability across herds and within farms has been rightly attributed to differences in feeding practices, breed and age of the animals, manure handling practices, and environmental conditions (Lindley et al. 1988; Clanton et al. 1991; Rieck-Hinz et al. 1996); this wide variability led us to question the use of table values.

Rieck-Hinz et al. (1996) measured manure nutrient content from 14 dairies in north-eastern Iowa using three different manure handling systems and created a modern database of Iowa dairy manure nutrient analyses. The nutrient concentrations found in their study were higher than values previously reported in Iowa and other Midwestern states. They suggested that these differences were attributable to changes in analytical methods, improved sampling methodology, and new feeding, herd, and manure management techniques. Safley et al. (1984) also reported dairy manure N contents approximately 30% higher than those reported previously. In 1996, Rieck-Hinz et al. stated that manure nutrient concentrations that were measured in the 1970s and reported in the 1980s (Sutton et al. 1983; Killorn 1984) were continuing to be used and needed to be updated with more recent data. The most commonly used table values were published by Midwest Plan Service (Loudon 1985) and were recently updated (Lorimor et al. 2000).

No general agreement exists. There is a lack of quantitative information on proper manure sampling procedures, and little understanding of the variability that exists. With this in mind, we conducted this study with the following objectives:

1. To measure the variability within stockpiles and lagoons of various animal manures and determine the number of sub-samples needed to characterize the nutrient content within a 10% probable error;

2. To compare Colorado manure analyses to the table values we have been using in our publications, which come from Midwestern data; and

3. To develop a database of manures from Colorado samples and determine if we can include manures from neighboring states in a "Mountain West" database.

Methods and Materials

Within-stockpile variability and sub-sample requirements. Ten sub-samples (approximately 0.5 L (0.5 qt) each) from each of five manure stockpiles (beef, dairy, horse, sheep, and chicken) and two finished manure composts (dairy and turkey) were collected in 1996. Each stockpile was sampled from a different farm. Two samples were taken from the top and two from each side of each stockpile (north, south, east, and west). For each pair of samples, one was taken shallowly (0.3 m (1 ft)), and one was taken more deeply (1 m (3 ft)). For the side samples, one of each sample pair was taken from the middle and one from near the bottom of the stockpile. Each sub-sample was analyzed separately for dry matter (D.M.), total nitrogen (N), ammonium (NH_4-N), nitrate (NO_3-N), phosphorus (P), and potassium (K) to determine the variability within the pile or lagoon. Dry matter content was determined by oven-drying the samples at 105°C (221°F) for 24 h; total N was measured by dry combustion; and inorganic N was extracted in 1M KCl land measured by automated colorimetry. Total P and K were analyzed by inductively coupled plasma spectroscopy in nitric-perchloric acid digests, and then results were converted to the oxide forms for presentation (Sparks 1996).

Collected data and equation 1 from Upchurch et al. (1988) and Davis et al. (1995) were used to determine the number of sub-samples needed (N_{est}).

$$N_{est} = t^2CV^2/p^2 \quad (1)$$

where t is Student's t value for a specified probability (in this case, for a 95% confidence interval), CV is the coefficient of variation, and p is a percent probable error (in this case, 10%).

Comparison to Midwestern table values. Beef, dairy, horse, sheep, and chicken solid manures were sampled in 1996 throughout Colorado, including operations located in the South Platte and Arkansas River Basins, and in the Tri-River Area on the Western Slope. Six to ten different livestock operations were sampled for each manure type. Each sample was a composite of six 0.5 L (0.5 qt) sub-samples taken from different locations and depths within the stockpile. The D.M., total-N, NH_4, phosphate (P_2O_5), and potash (K_2O) values measured in these samples and manure sample means from each farm tested in the within-stockpile variability experiment were combined into a database. The same laboratory methods were used as described above for the within-stockpile variability study. Results were compared to values previously used in Colorado extension publications, which came from Midwestern manure samples (Loudon 1985).

Mountain West manure database. Manure samples from three "Mountain West" states (Colorado, New Mexico, and Utah) were combined into one database. The data were gathered from analyses that had been accumulated by extension soil scientists in those states. Samples were not all collected at the same time nor analyzed by the same laboratory. These samples represent a variety of animal types, material types (solid, liquid, and composted), years (1993 to present), and sources. For each state, the number of samples, the mean, and the 90% confidence interval (C.I.) were calculated. When more than one state had samples within a manure type, the

Table 1. Number of sub-samples needed to characterize selected chemical characteristics of Colorado animal manure stockpiles within 10% error at 95% confidence level.

Manure type	N	P	K	NH_4-N	NO_3-N	D.M.*
	number of sub-samples needed†					
Solid manures						
Beef	17	20	32	121	692	3
Dairy	19	49	14	255	1914	22
Horse	17	11	14	211	802	12
Sheep	13	23	19	360	688	7
Chicken	55	31	27	443	147	43
Mean of solid manures	**24**	**27**	**21**	**278**	**849**	**17**
Composts						
Dairy	1	5	119	92	191	2
Turkey	40	26	13	128	440	2
Mean of composts	**21**	**16**	**66**	**110**	**316**	**2**

* D.M. = dry matter

† To determine the number of sub-samples needed (N_{est}), the equation $N_{est} = t^2CV^2/p^2$ was used (Upchurch et al. 1988). The *t* is Student's *t* value for a specified probability; *CV* is the coefficient of variation; and *p* is a percent probable error.

overall mean and C.I. were also determined. Using analysis of variance, we compared four manure types (dairy compost, dairy liquid, dairy solid, and chicken solid) that included samples (a minimum of five per state) from at least two of the three states.

Results and Discussion

Within-stockpile variability and sub-sample requirements. The variability of samples within a manure stockpile or lagoon differed for the various constituents. Ammonium and nitrate had the greatest coefficients of variation due to their relatively low concentrations. The greater the coefficients of variation, the greater the number of sub-samples required for useful analyses (Table 1). For example, to achieve probable error within 10% for a beef manure stockpile, one would need 17 sub-samples to characterize total N, 20 sub-samples for P, 32 for K, 121 for NH_4-N, and 692 sub-samples for NO_3-N.

The means for required sub-sample numbers for the composts were generally similar to those of the solid manures. For solid manures, it seems possible to estimate the total N, P, and K in a stockpile within 10% probable error with a moderately intensive sampling plan (collecting 21 - 27 sub-samples and combining them to form one composite sample). However, to characterize the NH_4-N and NO_3-N levels in order to predict N availability to crops, the required sub-sample number becomes impractical (> 100). Rieck-Hinz et al. (1996) used four sub-samples per farm in their study, and Dou et al. (2001) suggested a minimum of 40 sub-samples for unagitated manures.

Comparison to Midwestern table values. The solid manures sampled from Colorado operations differed in comparison with those we previously used in our extension publications (Waskom 1994), which originated from sources in the Midwest (Loudon 1985). On a dry weight basis, the total N and NH_4-N contents in Colorado samples were consistently lower than those from the Midwest (Table 2a). Phosphate (P_2O_5) content was higher among the Colorado manures in three out of five cases, while in the other two cases, the contents were about equal. Potash (K_2O) was higher in the Colorado samples than the Midwestern values in three out of five manures, but for the other two manures, the Midwestern samples were higher.

The dry matter contents of the Colorado manures sampled were consistently higher than those reported from the Midwest (Table 2b). On a wet weight or "as is" basis, the Colorado manures had higher total N contents in four out of five cases. Ammonium (NH_4-N) was lower in all of the Colorado manures on a wet weight basis. Colorado P_2O_5 and K_2O contents were higher than Midwestern data for all manure types, when evaluated on a wet weight basis.

The semi-arid and windy climate of Colorado probably leads to greater evaporation of water and volatilization of NH_3* from manure stockpiles, resulting in the higher dry matter values and lower contents of NH_4-N in all of the manures. Phosphate and K_2O contents are probably greater in Colorado manures because of the concentration effect from the greater loss of water. This concentration effect also occurs with organic N, causing the increase in total N con-

Table 2a. Comparison of selected chemical characteristics of solid animal manures from Colorado and Midwest (dry weight basis).

Manure type	Source	n*	Total N	NH_4-N	P_2O_5	K_2O
			kg/Mg†			
Beef	Colorado	11	17	2	18	30
	Midwest‡		20	6	14	25
Dairy	Colorado	8	12	2	15	32
	Midwest		25	11	11	28
Horse	Colorado	9	12	0.5	9	23
	Midwest		15	4	4	15
Sheep	Colorado	9	21	2	19	28
	Midwest		32	9	20	46
Chicken	Colorado	9	25	6	54	28
	Midwest		36	29	54	38

* n = number of samples

† To convert to English units, kg/Mg x 2 = lb/t.

‡ Midwestern values come from Loudon (1985).

Table 2b. Comparison of selected chemical characteristics of solid animal manures from Colorado and Midwest (wet weight basis).

Manure type	Source	n*	D.M.†	Total N	NH_4-N	P_2O_5	K_2O
			— % —	kg/Mg‡			
Beef	Colorado	11	68	12	2	12	20
	Midwest§		52	10	4	7	13
Dairy	Colorado	8	54	6	1	8	17
	Midwest		18	4	2	2	5
Horse	Colorado	9	78	10	0.5	7	18
	Midwest		46	7	2	2	7
Sheep	Colorado	9	69	14	1	13	19
	Midwest		28	9	2	6	13
Chicken	Colorado	9	60	15	4	32	20
	Midwest		45	16	13	24	17

* n = number of samples

† D.M. = dry matter.

‡ To convert to English units, kg/Mg x 2 = lb/t.

§ Midwestern values come from Loudon (1985).

tent in most of the manures. Rieck-Hinz et al. (1996) also evaluated manure nutrient content on both wet and dry weight scales to determine whether the differences in nutrient contents were attributable to differences in moisture content. In their study, statistical analyses of dry weight data resulted in smaller but still significant differences, as compared to the data analysis on an "as is" or wet weight basis. The differences in nutrient contents were not completely attributable to differences in moisture content.

Mountain West manure database. The database of manure samples from Colorado, New Mexico, and Utah is summarized in Table 3. Although the data set includes a large number of manure types, few of them include large sample numbers from more than one state. Only one manure type (solid beef manure) has more than 100 samples in the database, and these are mostly from one state (Colorado).

Analysis of variance was used to compare four manure types that included samples (minimum of five) from at least two states (Table 4). Significant differences were found among states within each manure type tested. Solid dairy manure differed among states in all four characteristics measured. New Mexico solid dairy manure consistently had the highest D.M., N, P_2O_5, and K_2O contents. Utah had the lowest concentrations, and Colorado samples were intermediate. The other three manure types had differences among states for only one characteristic. Dairy compost had differences in dry matter content, solid chicken manure was significantly different in total N, and liquid dairy manure had significant differences in K_2O.

Another measure of similarity is the confidence interval (C.I.), which is a measure of the probability that a sample will fall within an upper and lower limit (Table 3). For the one case in which we had over 100 samples (solid beef manure), the 90% C.I.s were extremely narrow. For example, the mean total N content was 12 kg/Mg (23 lb/t), with a C.I. of 10 - 12 kg/Mg (21 - 24 lb/t). We can interpret this to mean that nine out of ten beef manure stockpiles will have an N content between 10 and 12 kg/Mg (21 and 24 lb/t). For means with small sample sizes, the C.I.s were often larger, making our table values less precise.

Table 3. Means and 90% confidence intervals (C.I.) of selected chemical characteristics (wet weight basis) of manures from Colorado, New Mexico, and Utah.

	D.M.*			N†			P_2O_5†			K_2O†		
Manure type	**n‡**	**Mean**	**C.I.**	**n**	**Mean**	**C.I.**	**n**	**Mean**	**C.I.**	**n**	**Mean**	**C.I.**
Solids												
Beef	103	62	60-65	103	12	10-12	84	12	11-12	84	16	16-17
Dairy	78	63	59-66	62	12	12-14	51	9	8-10	51	18	16-20
Chicken	14	64	56-72	14	24	16-31	14	31	27-34	14	20	18-23
Hog	6	43	20-67	6	6	1-10	3	22	0-64	3	4	0-16
Horse	9	78	73-82	9	10	8-11	9	7	6-8	9	18	17-20
Llama	3	79	66-92	3	14	6-22	3	12	8-16	3	27	10-44
Sheep	12	67	59-67	12	14	12-16	10	13	11-15	10	20	17-22
Turkey	9	90	84-96	9	32	28-36	9	38	32-46	9	18	16-20
Liquids												
Dairy	18	1	0-2	23	0.6	0.4-0.8	23	0.2	0.1-0.5	23	0.6	0.4-0.8
Hog	9	0	0-0	9	0.2	0.1-0.4	9	0.1	0.1-0.1	9	0.6	0.4-0.8
Composts												
Dairy	38	77	74-80	36	12	10-13	36	11	10-12	36	22	18-24
Turkey	6	68	66-71	6	19	16-22	6	40	36-44	6	23	19-26
Chicken	12	54	45-62	12	13	12-14	12	35	28-41	12	16	14-19

* Means and C.I. in %.

† Means and C.I. in kg/Mg for solids and composts; kg/1000 L for liquids. To convert to English units, kg/Mg x 2 = lb/t, and kg/1000 L x 8.32 = lb/1000 gal.

‡ n = number of samples

Table 4. Analysis of variance and confidence intervals associated with selected chemical characteristics of manures from Colorado, New Mexico, and Utah.

Manure type	State	Dry matter n[*]	Dry matter Mean	Dry matter C.I.	Total N n	Total N Mean	Total N C.I.	P_2O_5 n	P_2O_5 Mean	P_2O_5 C.I.	K_2O n	K_2O Mean	K_2O C.I.
			— % —			— kg/Mg or kg/1,000 L —			— kg/Mg or kg/1,000 L —			— kg/Mg or kg/1,000 L —	
Dairy compost	CO	11	69 A[†]	64-75	11	10 A[‡]	8-12	11	11 A	8-14	11	20 A	14-24
	NM	25	80 B	77-84	23	12 A	11-14	23	10 A	9-12	23	23 A	20-26
Dairy liquid	CO	18	1	—	18	0.6 A	0.4-0.8	18	0.2 A	0-0.5	18	0.4 A	0.1-0.6
	UT	—	—	—	5	0.7 A	0.1-1.2	5	0.5 A	0-0.8	5	1.4 B	1.0-1.9
Dairy solid	CO	22	48 B	42-53	22	10 B	8-12	11	8 A	6-10	11	16 AB	12-20
	NM	48	73 A	69-76	32	16 A	14-17	32	10 A	9-12	32	20 A	17-22
	UT	8	44 B	35-53	8	6 B	4-10	8	4 B	2-6	8	10 B	5-14
Chicken solid	CO	8	60 A	49-71	8	15 A	8-22	8	32 A	27-38	8	20 A	16-23
	UT	6	69 A	55-82	6	36 B	28-44	6	29 A	23-34	6	22 A	17-26

[*] n = number of samples

[†] States with means with a common letter within manure type are not significantly different by analysis of variance ($p < 0.05$).

[‡] To convert to English units, kg/Mg x 2 = lb/t, and kg/1000 L x 8.32 = lb/1000 gal.

The minimum number of farms required to be sampled in order to reduce the C.I. to an acceptable range, can be predicted by graphing the range of the 90% C.I. as a function of the sample number (Figure 1). Due to the shapes of the curves, transformations were used to optimize the fit of the regression equations. Table 5 summarizes the best fit regression equations and their implications. The equation with the best fit is for D.M. (R^2=0.81), but it applies to the solid manures only. Adding composts to the solid manure equations for N and K_2O did not reduce the R^2 or p-values. The equation for P_2O_5 fits the data for solid, composted, and liquid manure.

Rieck-Hinz et al. (1996) created a database for a sub-region of Iowa with 14 farms. Based on our information, we recommend a minimum of 25 farms for manure database creation in the Mountain West in order to achieve 90% C.I. ranges of 10% D.M. and 5 kg/Mg (10 lb/t) for the nutrients. Including 72 farms in each database (for each manure type) would reduce the ranges in the 90% C.I.s to 5% D.M. and 2.5 kg/Mg (5 lb/t) for each of the nutrients (Table 5).

Summary and Conclusions

Dry matter and nutrient content varied considerably within manure types. About 25 sub-samples were usually sufficient to characterize total N, P_2O_5, and K_2O within a 10% error in our study. It is impractical to sample manure for NH_4-N and NO_3-N due to their low levels and high coefficients of variation in semi-arid areas. This data should not be extrapolated to include regions where NH_4-N makes up a large fraction of the total manure N content. Based on our limited dataset, it appears that nutrient management planners will achieve greater accuracy in semi-arid areas using total N values alone for predictions of N availability to crops.

Colorado manure nutrient contents and dry matter were different from the Midwestern values we had been using in our extension publications. We have since updated our extension publications appropriately (Waskom and Davis 1999). Dry matter content was higher in Colorado due to the semi-arid climate and higher evaporative demand. The P_2O_5 and K_2O contents were greater in Colorado samples than those of the Midwest-ern region. Ammonium-N (NH_4-N) was lower in Colorado, probably due to greater volatilization in this dry and windy climate.

Comparisons of Colorado manures to those from New Mexico and Utah were inconclusive. Almost half of the characteristics measured were significantly different among states. These differences may be largely due to small sample sizes and outliers in the relatively small data sets.

For now, Colorado State University Cooperative Extension plans to base most recommendations on Colorado samples. As the database sizes increase and the C.I.s are reduced, we expect that differences among states will decrease and Colorado table values will become more valuable. Until a given manure type has 72 or more samples and tight confidence intervals, we will continue to recommend that livestock producers sample and test their livestock manure. However, if producers will not sub-sample adequately (n > 20), they should use the new Colorado-based table values for best results and follow up with soil testing.

Table 5. Prediction of sample numbers needed for a manure database to achieve ranges in the 90% confidence interval of 10 and 5% for dry matter (D.M.) and 5 and 2.5 kg/Mg (10 and 5 lb/t) for N, P_2O_5, and K_2O.

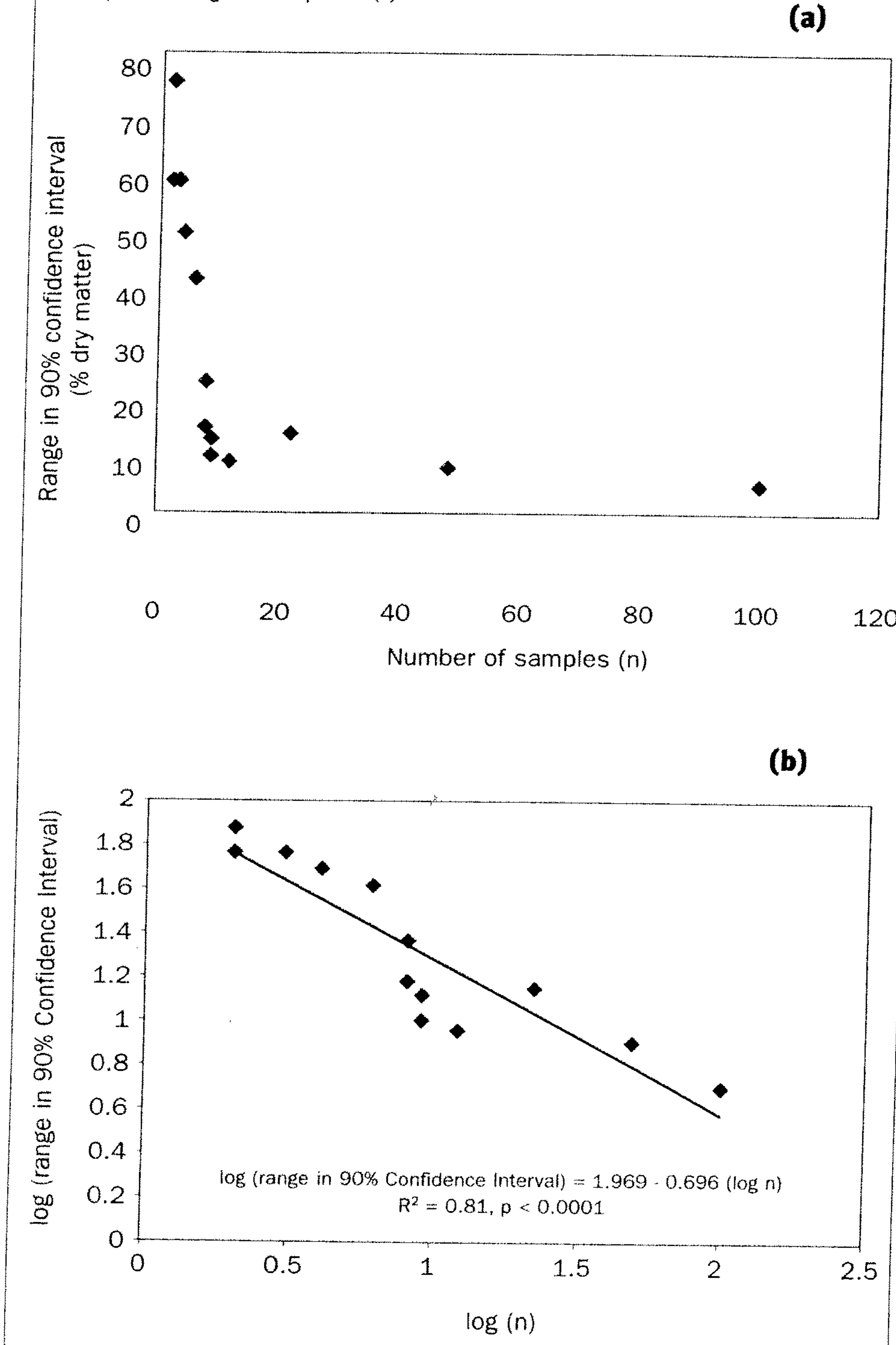
Manure characteristic	Best fit equation[*]	R^2	p	Manure types included	Required n to achieve defined range in confidence interval: 5 kg/Mg[**]	2.5 kg/Mg[***]
D.M.	$\log y = 1.969 - 0.696 \log x$	0.81	< 0.0001	Solid	25	67
N	$-1/y = -0.071 - 0.003 x$	0.39	0.0041	Solid, compost	10	43
P_2O_5	$\log y = 1.717 - 0.649 \log x$	0.32	0.0144	Solid, compost, liquid	11	37
K_2O	$-1/y = -0.055 - 0.002 x$	0.48	0.0019	Solid, compost	22	72

[*] x = number of samples (farms); y = range in 90% confidence interval.

[**] 10% for D.M.

[***] 5% for D.M.

Figure 1
Range in the 90% confidence interval (for manure dry matter) as a function of the number of samples in the database for each solid manure type (a), and the log transformation of those data to improve the regression equation (b).



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