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# Total Nitrogen Content of Dairy Manures in New Mexico

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## Summary

Using dairy manure on agricultural soils requires an accurate analysis of nutrients and manure sampling strategies. The objectives of this study were to: determine the variability of total Kjeldahl nitrogen (TKN), water content and temperature in four types of dairy manure; estimate the effect of sampling location and depth on TKN content; calculate the sample size; and correlate the TKN content in a composite sample with the average of individual samples.

Dairy manure samples were collected from 19 manure piles in seven counties in New Mexico. Samples were taken from five locations and two depths in each pile.

Total Kjeldahl nitrogen content of dairy manure did not depend on the type of manure handling (pile) and ranged from 0.53% N to 2.83% N for all piles and types of manure. The 95% confidence interval for each type of manure ranged from 1.51 to 2.53% N for fresh manure, from 0.09 to 2.02% N for composted manure, from 0.99 to 1.89% N for stockpiled manure, and from 0.28 to 3.02% N for wash solids. Sample depth and sample location in a manure pile did not significantly affect the TKN content of the sample.

For an accurate determination of the TKN content of manure in a manure pile, a minimum of seven samples should be collected and composited from a variety of locations and depths of fresh manure and 30 samples should be collected from stockpiled manure. A minimum of two samples should be collected and composited from an aerobically composted manure pile.

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## INTRODUCTION

The New Mexico dairy industry has generated tremendous amounts of manure as a result of a 172% increase in production (Bethard, 1998). The total number of cows has tripled since 1987, and manure disposal as an organic fertilizer has increased in agricultural soils (USDA, 1997; Dickerson, 1999; Skaggs et al., 1999). Additionally, dairy manure compost is being applied to soils as a nutrient source (Walker, 1999). Thus, manure sampling strategies must be designed to evaluate the nutrient content of manure.

Nitrogen (N) requires critical attention because of its impact on crop productivity and water quality. Total Kjeldahl nitrogen (TKN) can be an accurate predictor of total N content, because the inorganic N content in manure generally is very small when compared to the total N content (Paul and Beauchamp, 1993; Eghball, 2000). Total Kjeldahl nitrogen is a wet oxidation procedure used to determine the organic N present as  $\text{NH}_3$  in soils, plants, and organic residues, such as dairy manure. Also, TKN analysis can include a pretreatment of the sample to convert  $\text{NO}_3^-$ -N and  $\text{NO}_2^-$ -N to  $\text{NH}_3$ , and thus have a total N analysis (Bremner, 1996).

Total N content in dairy manure depends on feed inputs, age of the cows, age of the manure, environmental conditions, manure handling systems, manure storage, and land application methods (Brady and Weil, 1996; Rieck et al, 1996). According to a manure analysis program conducted by the University of Maryland, the average total N content was 2.4% for 400 samples of dairy manure collected from 1985 to 1990 (Brady and Weil, 1996). Sweeten et al. (1982) reported that the N content of 23 Texas High Plains feedlots ranged from 1.16 to 1.96%. They also reported N concentrations of 2.2% for cattle manure, 2.37% for beef feedlots, 2.13% for dairy corrals, and 1.72% for a dairy manure stockpile in Arizona. Zhang and Hamilton (1998) reported values from 1.29 to 1.93% N for feedlot manure. Iversen et al. (1997) found 1.2% N in samples of composted dairy manure.

The composition of manure changes from the time it is excreted to the time it is applied to the field. Scraping, weathering, decomposition, and handling can change the N content. Safley et al. (1984) concluded that TKN values were much lower for scraped

dairy manure than for fresh manure. The scraped manure varied from 2.27 to 5.16% N, whereas fresh manure varied from 2.68 to 7.73% N in samples collected from seven farms in North Carolina.

A representative sample of manure must be collected from a manure pile to obtain an accurate N content for that pile. Therefore, determining a sample size per pile is necessary to avoid a large number of samples and reduce the cost of analysis. Iversen et al. (1997) concluded that 17 subsamples were required to characterize a beef manure stockpile for total N content in order to achieve 95% confidence with 10% probable error. Great variability was found in the inorganic forms of N. They proposed to base N availability estimates from manure solely on total N with a regular program of soil testing.

The lack of information about N content and sample size per pile of dairy manure in New Mexico motivated the present study. The objectives were to: determine the TKN content in different types of dairy manure, and to determine whether sampling location and depth have a significant effect on the N content within manure piles; estimate the average TKN content for selected types of dairy manure in New Mexico with a 95% confidence interval; calculate the number of samples within a pile required to adequately estimate TKN content for each manure type; and determine if the TKN content in a composite sample is correlated with the average TKN content of individual samples taken from each manure pile.

## MATERIALS AND METHODS

Manure samples were collected in July 1998 from dairies and farms in Bernalillo, Chaves, Doña Ana, Eddy, Lea, Quay and Socorro counties (fig.1). These counties have a total of 144,000 cows that represented 71% of the dairy cows in New Mexico (USDA, 1997). Eleven dairy farms were selected at random and represented 7% of all dairy farms in New Mexico (Bethard, 1998). A total of 19 piles of dairy manure were sampled from the following types: fresh manure that was scraped weekly inside the pen; stockpiled manure that was stored at least six months outside the pen; aerobically composted dairy manure; and wash solids that included solids screened from the wastewater system prior to entering a liquid storage lagoon.

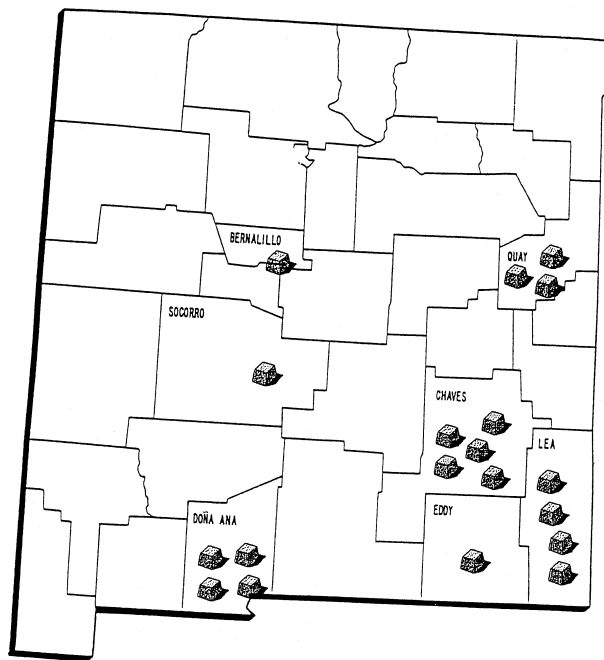


Figure 1. Map showing sampling locations in New Mexico.

## Manure Sampling

The number of samples collected per pile of manure varied from six to 21 according to the manure type and the pile shape (table 1). Two general pile shapes were sampled, round and rectangular. In addition to the sampling scheme described below for round and rectangular piles, composite samples were collected from each pile to a depth of 12 in. The temperature of each manure pile was also measured at each side of the pile with a 1 yd Rheostat compost thermometer.

Manure samples were taken from the north, east, south, west, and from the top of the round piles. At each location within a pile, one sample was taken from a shallow depth (0-12 in.) and another from a deep depth (12-40 in.). A total of 10 samples were collected per pile. In four fresh piles from Lea County (table 1), only shallow samples could be taken because the manure was too wet and compacted to sample at greater depths.

The lengths of rectangular and windrow piles were approximately 100 yd with widths less than 3 yd. Windrow piles were less than 2 yd. tall, while rectangular piles were greater than 2 yd. in height. Rectangular and windrow piles were sampled in the same way as round piles. Samples were taken from two sides, either east and west or north and south. Five samples from each side at two depths (0 to 12 in. and 12 to 40 in.) were collected for a total of 20 samples. Additionally, a composite sample from the 0 to 12 in. depth was collected from the same sides of each pile.

The samples were stored in plastic bags and placed in a cooler with ice for transportation to the soil microbiology laboratory, whereupon the samples were stored in a freezer until analysis. The water content of the manure samples was determined gravimetrically by drying at 150°F until constant weight. The water content in the manure samples was calculated as:

$$\text{Water content (\%)} = ((\text{wet wt.} - \text{dry wt.}) / (\text{wet wt.})) * 100$$

Dried samples were finely ground and sieved (40 mesh) for chemical analysis. Manure samples were digested with  $\text{H}_2\text{SO}_4$  and analyzed for TKN using a colorimetric technique (Technicon Instruments Corporation, 1974) with the Technicon Autoanalyzer II (Technicon Instruments Corporation, Tarrytown, N.Y.).

**Table 1. Type and shape of dairy manure piles and sampling scheme.**

County	District	Type of Manure	Pile Name	Pile Shape	Sample Depth	Sample Number
Socorro	Bernardo	Stockpiled	Stabo	Round	a <sup>+</sup>	11
Bernalillo	Albuquerque	Stockpiled	Stson	Round	a	11
Doña Ana	Mesquite	Stockpiled	Stvrmix	Round	a	11
Doña Ana	Mesquite	Stockpiled	Frstar	Round	a	11
Doña Ana	Mesquite	Fresh	Stvium	Round	a	11
Doña Ana	Mesquite	Fresh	Frvesta	Round	a	11
Eddy	Cottonwood	Stockpiled	Sthaf	Round	a	11
Chaves	East Grand Plains	Stockpiled	Stnat	Windrow	a	21
Chaves	East Grand Plains	Composted	Conat	Windrow	a	21
Chaves	East Grand Plains	W. Solids	Wanat	Windrow	a	21
Chaves	Mountain States	Stockpiled	Sber	Rectangle	b	20
Chaves	Midway	Stockpiled	Stvaca	Round	b	11
Lea	Hobbs	Fresh	Frelow1	Round	b	6
Lea	Hobbs	Fresh	Frelow2	Round	b	6
Lea	Hobbs	Fresh	Frermid	Round	a	6
Lea	Hobbs	Fresh	Frhigh	Round	a	6
Quay	Clovis	Composted	Corag	Windrow	a	21
Quay	Clovis	Stockpiled	Strag1	Round	a	11
Quay	Clovis	Stockpiled	Strag2	Round	a	21

<sup>+</sup>a = Two sample depths, from 0-12 inches and 12-40 inches; b = One sample depth from 0-12 inches.

## Statistical Analysis

Statistical analyses were performed using the GLM procedure of the Statistical Analysis System (SAS, 1997). The pile averages of TKN, water content, and temperature were analyzed manure type using a completely randomized design. Because the manure piles did not have the same number of samples, locations and depths, an analysis of variance also was conducted separately for each manure type to determine the effect of location and depth on the variables measured. This separate analysis could not be performed for wash solids, because only one pile was sampled. The TKN, water content, and temperature means were compared using Fisher's Least Significant Difference (LSD). The correlation between composite samples and the average of the individual samples from the same pile was estimated for TKN and water content.

A 95% confidence interval was calculated about the mean TKN for each manure type, using the pooled estimate of the variance from the combined analyses of manure types.

Sample size (n) per pile for future studies was calculated for each manure type using the coefficient of variation (CV) estimated from the separate manure analyses. Student's t-statistic (t) was set at 2.26 (t.025, 9) and the probable error (p) was set at 0.10, similar to Iversen et al. (1997). The sample sizes were computed according to:

$$n = \frac{t^2(CV)}{p^2}$$

In addition to the sample size computations, the effect of increasing the number of samples per pile on the within pile standard error was illustrated by plotting the estimated standard error against sample size for each manure type. This estimated standard error (SE) was calculated as:

$$SE = \frac{S}{\sqrt{n}}$$

Where S is the estimated standard deviation within piles.

## RESULTS AND DISCUSSION

### Manure TKN Content

When the average TKN content for each pile was analyzed statistically for all manure types, TKN content was not different ( $p=0.18$ ) among manure types (table 2). When the fresh, stockpiled and composted manures were analyzed separately, sample location within piles of manure, sample depth, and the interaction of location by depth had no detectable effect on TKN content. However, the variability of TKN content among piles was significant for all three types of dairy manure. This analysis could not be performed for washed solids as only one pile was sampled. These results suggest that the TKN content is more influenced by manure handling (pile) than manure type or location and sampling depth in the pile.

The average sample TKN content for each manure type is given (table 3). The composted manure sample average was the lowest of the four manure types in our study. Failure to detect a difference among the manure types may be a result of having sampled only two compost piles and one wash solids location. In part, the low TKN content of composted manure can be attributed to loss of N through volatilization during the composting process. Eghball et al. (1997) found that N loss during composting ranged from 19 to 42% and was related to the initial manure N content.

The TKN content in all piles varied from 0.53% (10.6 lb N ton<sup>-1</sup> manure) to 2.83% (56.4 lb N ton<sup>-1</sup> manure) on a dry weight basis and from 0.43 to 2.06% (4.3 to 20.6 kg N ton<sup>-1</sup> manure) on a wet weight basis (table 4). Water content in manure dilutes the TKN content. These values are in the range of N content reported by Sweeten et al. (1982) and Zhang and Hamilton (1998) for similar manure types. The variation detected in the present study may be related to the different ages of manure, handling systems, and moisture content during storage.

This information could be useful to estimate the manure application rate, if mineralization rates, soil test nitrogen, and crop requirements are known. Deficiencies can be corrected with inorganic fertilizer. In this way, nutrients can be managed for optimum crop production and environmental protection.

**Table 2. Analysis of variance for the effects of type of manure pile, sample location and sample depth on the TKN, water content, and temperature of manure samples.**

Source of Variation	Total Kjeldahl Nitrogen	Water Content	Temperature <sup>+</sup>
Manure Type <sup>++</sup>	NS	**	NS
Sample Location <sup>‡</sup>	NS	NS	**
Sample Depth <sup>‡</sup>	NS	NS	na
Location *Depth <sup>‡</sup>	NS	NS	na

<sup>+</sup>Temperature was compared at only one depth (0-12 inches).

<sup>++</sup>From the statistical analysis on pile means for all manure types.

<sup>\*\*</sup> Significant at the 0.01 probability level.

<sup>‡</sup>Analyzed separately for each manure type (stockpiled, fresh and composted).

<sup>na</sup>Not analyzed.

**Table 3. Mean values and standard errors of four types of dairy manures collected at 11 farms in New Mexico.**

Type of Manure	TKN (%)		Water (%)		Temp. (°F)		Piles Sampled
	Mean	S.E.	Mean	S.E.	Mean	S.E.	
Fresh	2.07 a <sup>+</sup>	0.26	33.7 b	3.5	117 a	3.4	6
Wash Solids	1.65 a	0.64	46.4 b	8.5	147 a	8.4	1
Stockpiled	1.42 a	0.20	19.0 a	2.7	126 a	2.8	10
Composted	1.06 a	0.45	25.2 ab	6.0	111 a	0.0	2

<sup>+</sup>Within columns, means followed by the same letter are not significantly different ( $P>0.05$ ) according to Fisher's protected LSD test.

### Water Content and Temperature

Water content differed among manure types ( $p<0.01$ ) but not by location within the pile or depth for any of the manure types ( $p>0.05$ ) (table 2). Wash solids had the greatest water content and the stockpile manure the lowest (table 3). Water content among piles of manure was highly variable and ranged from 6 to 46% (table 4).

Temperature was not different among manure types, but it was affected by sample location within stockpiles (tables 2 and 3). High temperatures in the fresh, wash solids, and stockpiled manure piles indicate that active, thermophilic decomposition was occurring.

**Table 4. Water content and total Kjeldahl nitrogen (TKN) for all manure piles.**

Type of Manure	Water Content	TKN+ Dry Basis	TKN++ Wet Basis	TKN¶ Content Dry Basis	TKN¶¶ Content Wet Basis
-----%-----					
----- lb N ton <sup>-1</sup> manure -----					
Stockpiled	22.5	1.32	1.02	26.4	20.5
Stockpiled	23.9	1.00	0.76	20.0	15.2
Stockpiled	27.2	2.83	2.06	56.4	41.1
Stockpiled	19.1	0.53	0.43	10.6	8.6
Stockpiled	18.0	1.85	1.52	37.0	30.3
Stockpiled	33.8	0.77	0.51	15.4	10.2
Stockpiled	13.1	2.26	1.96	45.0	39.1
Stockpiled	6.1	1.36	1.27	27.1	25.4
Stockpiled	13.1	1.35	1.17	26.9	23.4
Stockpiled	13.7	0.94	0.81	18.7	16.1
Fresh	25.1	1.11	0.83	22.1	16.6
Fresh	32.6	1.89	1.28	37.8	25.5
Fresh	37.1	1.92	1.21	38.3	24.1
Fresh	34.0	2.59	1.71	51.7	34.1
Fresh	37.7	2.55	1.59	50.8	31.7
Fresh	35.9	2.39	1.53	47.8	30.6
Compost	39.1	1.23	0.75	24.6	15.0
Compost	11.2	0.89	0.79	17.8	15.8
Wash Solids	46.4	1.65	0.88	32.9	17.7

+Dry basis is from laboratory analysis.

++Wet basis: TKN (%) = dry N (%) - ((dry N (%) \* moisture(%))/100)

¶Dry basis: TKN (lb/ton) = dry TKN (%) \* 10 \* 907/454

¶¶Wet basis: TKN (lb/ton) = wet TKN (%) \* 10 \* 907/454

The composted manure pile temperature is closer to the ambient air temperature because decomposition had already occurred.

### Variation of TKN Content and Sample Size

Figure 2 shows that as the sample size increases, the standard error decreases. For stockpiled and fresh manures within each pile, this standard error decreases and remains between 0.05 and 0.1, when the sample size is larger than five samples. The sample size estimation varied from two to 30 samples among manure types (table 5).

The stockpiled and fresh manures had large standard deviations, probably reflecting the haphazard method of piling the manure and lack of mixing. In contrast, the composted manure had a small standard deviation that reflects mixing during the composting process. Figure 2 shows that as sample size increases, the standard error for composted piles decreases below 0.03, when the sample size is larger than three. The small variation of TKN content in composted manure suggests the use of a small sample size (n = 2).

These results suggest that 30 samples per pile is adequate for stockpiled manure and seven samples for fresh manure to achieve a representative sample with 95% confidence. However, the sample size could be smaller for composted manure than for stockpiled and fresh manure, because standard errors in composted manure are relatively constant at small sample sizes (fig. 2). The sample size

**Table 5. Confidence intervals, standard deviations, and sample size for manure sampled at 11 farms in New Mexico.**

Type of Manure	Number of Piles	Confidence Interval (95%)+	TKN Mean (%)	Standard Deviation	Sample Size Estimation
Stockpiled	10	0.99, 1.85	1.42	0.35	30
Fresh	6	1.51, 2.63	2.07	0.22	7
Composted	2	0.09, 2.02	1.06	0.06	2
Wash Solids	1	0.28, 3.02	1.65	0.72	++
All Piles	19		1.60		

+t 0.025, 15-1

++Only one manure pile was sampled.

estimated in this study (30 samples) is larger than the 17 samples reported by Iversen et al. (1997) for total N content in dairy manure.

### Composite Sample

A strong linear relationship ( $r=0.92$ ) was found between the composite sample and the average of individual samples for the TKN content (fig. 3). These data suggest that TKN obtained in the composite sample covered the N content variability in each manure pile. A similar linear response ( $r=0.93$ ,  $p<0.01$ ) was found for the correlation between the composite samples and the average of individual samples for water content. These findings clearly suggest that, for TKN analysis, using a composite sample instead of several individual samples of a manure pile may reduce the cost of chemical analysis and the time for sample collection.

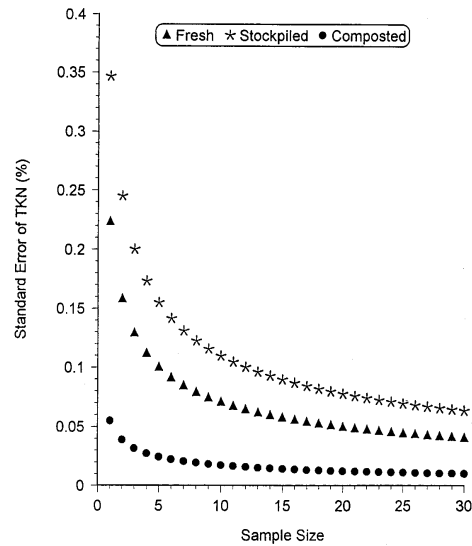


Figure 2. Standard error of the total Kjeldahl nitrogen (TKN) as a function of the sample size in three types of dairy manure.

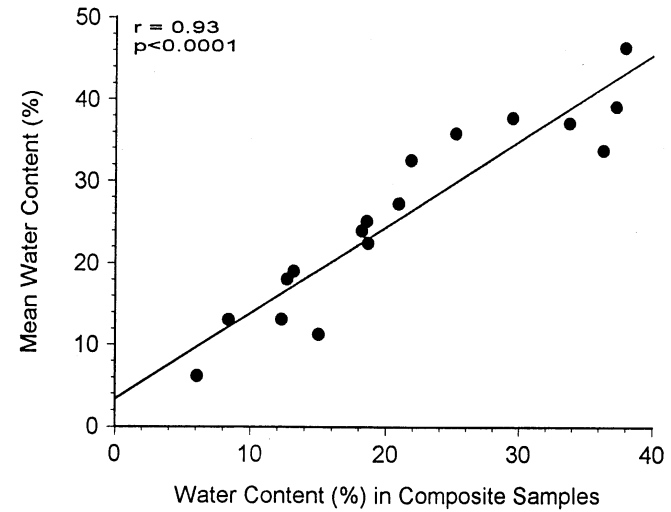
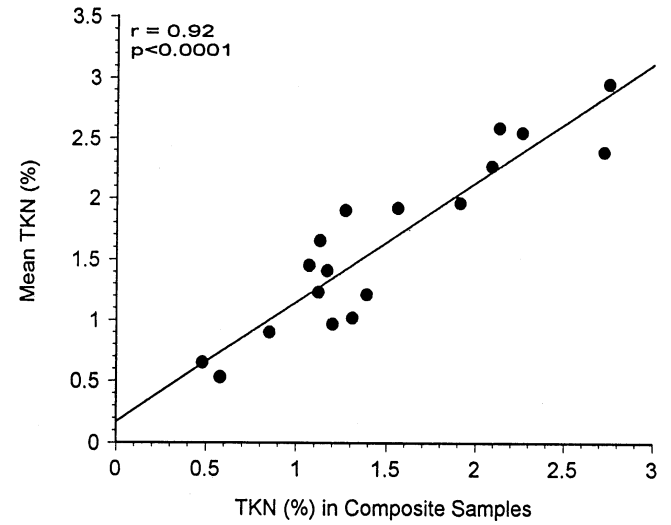


Figure 3. Relationship between the water content and TKN measured in composite samples and the average calculated with individual samples of dairy manure.



## CONCLUSIONS

TKN content of dairy manure did not depend on the type of manure handling (pile) and ranged from 0.53% N to 2.83% N for all piles and types of manure. The confidence interval for each type of manure at 95% confidence ranged from 1.51 to 2.53% N for fresh manure, from 0.09 to 2.02% N for composted manure, from 0.99 to 1.89% N for stockpiled manure, and from 0.28 to 3.02% N for wash solids. Sample depth and sample location in a manure pile did not significantly affect the TKN content of the sample.

To accurately determine the TKN content of manure in a manure pile, a minimum of seven samples should be collected and composited from a variety of locations and depths of fresh manure, and 30 samples are needed for stockpiled manure. A minimum of two samples should be collected and composited from an aerobically composted manure pile.

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