

# NITROGEN CONCENTRATION VARIABILITY IN DAIRY-CATTLE SLURRY STORED IN FARM TANKS

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## ABSTRACT

Spatial and temporal variability in the concentration of total Kjeldahl and ammonia ( $\text{NH}_3 + \text{NH}_4^+$ ) nitrogen (TKN and AMN, respectively) was studied in 8 to 10% total solids content dairy-cattle manure slurry and its centrifuged supernatant during undisturbed storage in covered, reinforced concrete, farm storage tanks. Slurry was stored in two winter-filled tanks for 285 days, and in two additional summer-filled tanks for 146 days. Although concentration variability with time and space was small relative to the initial concentrations, slurry at depths of less than 1 m had consistently lower concentrations than at greater depths, particularly after the initial two months of storage. Mass balance for nitrogen (N) indicated a 9% loss in three of the four tanks. A lower loss (4% N) in the fourth tank was accompanied by a decrease in acetic acid concentration and a rise in slurry pH, at all depths, which was not observed in the other tanks. The lower loss of N from this tank than from the other tanks probably occurred to satisfy chemical equilibria that required a greater retention of ammonia ( $\text{NH}_3$ ) by the carbon dioxide ( $\text{CO}_2$ ) release from acetic acid breakdown. Considerations other than TKN and AMN concentration changes and equilibrium relations may also be important factors for N retention in slurry stored in farm tanks.

**KEYWORDS.** Manure nitrogen, Dairy cattle slurry, Farm storages.

## INTRODUCTION

Utilization of crop nutrients in animal manures is almost essential for the long-term sustainability of mixed farming systems. Nutrient transformations and losses that occur during manure handling need to be known for matching of nutrient applications to crop requirements. Of the three major nutrients, nitrogen (N), phosphorus and potassium, the first is by far the most important because it is subject to greater losses than the latter two since only N can be lost by volatilization as gaseous ammonia ( $\text{NH}_3$ ). Losses and transformations of N in manure slurry stored in farm tanks is discussed in this paper.

Nitrogen concentrations and losses under different types of manure handling and storage systems have been reported previously (Jones et al., 1973; Vanderholm, 1975; Converse et al., 1975; Oatway et al., 1975; Safley, 1980; Williams and Evans, 1981; Bulley and Holbek, 1982; Muck and Steenhuis, 1982; Muck and Richards, 1983; Muck et al., 1984, Converse and Holmes, 1985; Burton and Beauchamp, 1986). Depending on the type of slurry handling and storage system used, and the ambient temperature, the manure N loss reportedly ranges from negligible to 60% during collection, and from 3 to 60% during storage in pits or tanks. Jones et al. (1973) reported that 7.6% of the excreted N in dairy cattle manure was lost during daily scraping and a 101-day storage in a concrete tank. Bulley and Holbek (1982) estimated 6 to 10% loss of N during daily scraping of dairy cattle manure, and a further 10% loss during a 90-day storage in a covered concrete tank. Midwest Plan Service (1985) estimates N loss during manure collection and storage in anaerobic pits to range from 15 to 30% of the excreted N. The wide variation in the reported N losses from stored slurry, sometimes under apparently similar conditions, may be partly because of the difficulty of obtaining reasonably accurate mass balances for N in farm storage tanks when fresh manure is periodically added to the stored manure in the tanks, and the stored slurry is subject to loss by leaching, and dilution by rainwater and snow. These difficulties were essentially avoided in the study reported here.

The objective of our study was to determine the temporal and spatial variations in the composition and properties of dairy cattle manure slurry during undisturbed storage in covered, concrete farm tanks after filling in the winter and in the summer. This article reports the results on concentration changes and mass balances for N, and discusses these results in the context of other composition changes in the slurry.

## STUDY PROCEDURE

### MANURE SOURCE AND STORAGE TANKS

Slurry from pits beneath slotted floors in a 240 milking cow, free-stall, dairy barn was used to fill two concrete tanks in the winter (January) and two additional tanks in the summer (June). The slurry was left undisturbed in these tanks until October/November for a storage period of 285 days for the winter-filled tanks (WF1 and WF2) and 146 days for the summer-filled tanks (SF1 and SF2). It accumulated beneath the slats in the barn for a six-week period prior to its transfer to the storage tanks using the minimum necessary dilution water. Thus the slurry transferred to the tanks was "on average" three weeks old. The total solids (TS) content of the slurry transferred to the tanks ranged from 8.8 to 10.0% by weight. Wood shavings, used for bedding in the

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barn, accounted for about 8% of slurry TS. The cows were fed a combination of alfalfa silage, corn silage, haylage, and/or chopped hay.

The storage site consisted of six adjacent, partly below-grade, covered concrete tanks, each 12.3 x 7.2 x 3.0 m deep, built with 0.2 m of the tops extending above ground. The tanks were not watertight as they were built with unsealed wall-to-floor and wall-to-wall joints (Turnbull et al., 1971). After prior cleaning, four of the six tanks were used in the study. During the study period, all six tanks were further covered with 0.15 mm thick black polyethylene sheets to prevent entry of rain or snow into the tanks. Liquid level in the tanks was monitored (Model 5-FW Liquid Level Recorders, Belfort Instrument Company, Baltimore, MD) to determine changes in slurry volume during storage.

### SAMPLE AND DATA COLLECTION, PROCESSING, AND ANALYSIS

Slurry samples were collected from two openings midway along the length of each tank, at depths of 0.3, 1.0, 1.8, and 2.5 m below the slurry surface, 1.5 m away from the walls. Two-liter polyethylene bottles (230 mm x 120 mm diameter, 90 mm mouth opening), clamped at the end of a long-handled sampler, were used for sample collection, with minimum possible disturbance of the slurry. After positioning the bottle at a chosen sampling location, a remotely-operated stopper on the bottle was momentarily lifted to allow the slurry to flow into the bottle. Samples were collected at the time of filling, one week later and about monthly thereafter. A final sample was collected after thorough mixing (4 h recirculation using a pto centrifugal pump with a submerged slurry discharge nozzle) at the end of the storage period prior to hauling. Sixteen samples were obtained at each collection time (2 tanks x 2 locations x 4 depths). Samples were collected one week apart from the winter- and summer-filled tanks.

About 1.5 L of each sample was blended for 45 s in a Waring blender to obtain homogenous subsamples. It was considered preferable to have relatively homogenous as opposed to non-homogenous subsamples of slurry for analysis even though some loss of gaseous ammonia could occur during blending. Based on slurry pH and temperature, less than 1% of the total ammonia in the slurry was estimated to be in the un-ionized form. Loss of ammonia during the blending operation was therefore assumed to be insignificant compared to the total ammonia present in the slurry.

A subsample was centrifuged at 13,700 g for 20 min at 15° C to obtain a supernatant. Slurry temperature and pH were determined at the time of sample collection using a Yellow Springs Instrument Company (Yellow Springs, OH) thermistor probe and Radiometer Model 26 pH meter (Radiometer Copenhagen, Denmark). Total Kjeldahl nitrogen (TKN) and ammonia nitrogen (AMN, free ammonia + ammonium nitrogen) were determined in the slurry and supernatant (Association of Official Analytical Chemists, 1975). Slurry TS were determined by oven drying at 103° C (American Public Health Association, 1981).

Data were analyzed separately for each tank, and for pairs of tanks based on winter- and summer-filling. The least-square significant difference test (t-test) was used to compare mean concentration differences at the beginning and the end of the storage periods. Single degrees-of-freedom comparisons were made for N concentrations at different depths.

### RESULTS AND DISCUSSION

Continuous record of slurry levels indicated that the volume of the stored slurry varied by less than ±1.5% of the initial volume. The unsealed joints in the tanks appeared to have been sealed by the 9 to 10% TS manure slurry used in this study, as discussed earlier by Patni and Jui (1987). It was assumed that the volume of slurry remained essentially constant during the storage period. A dry crust was not formed at the slurry surface.

### SLURRY TEMPERATURE AND pH

Changes in slurry temperature and pH are shown in figures 1 and 2, respectively. The temperature varied in response to changes in the local ambient temperature (Table 1). In spite of subfreezing ambient temperatures, the slurry did not freeze because of the insulation provided by the adjacent ground, snow on the polyethylene and concrete covers, a layer of manure foam on the slurry surface, and a 0.3 m free-space below the tank cover. The slurry temperature varied most near the surface (0.3 m depth) and

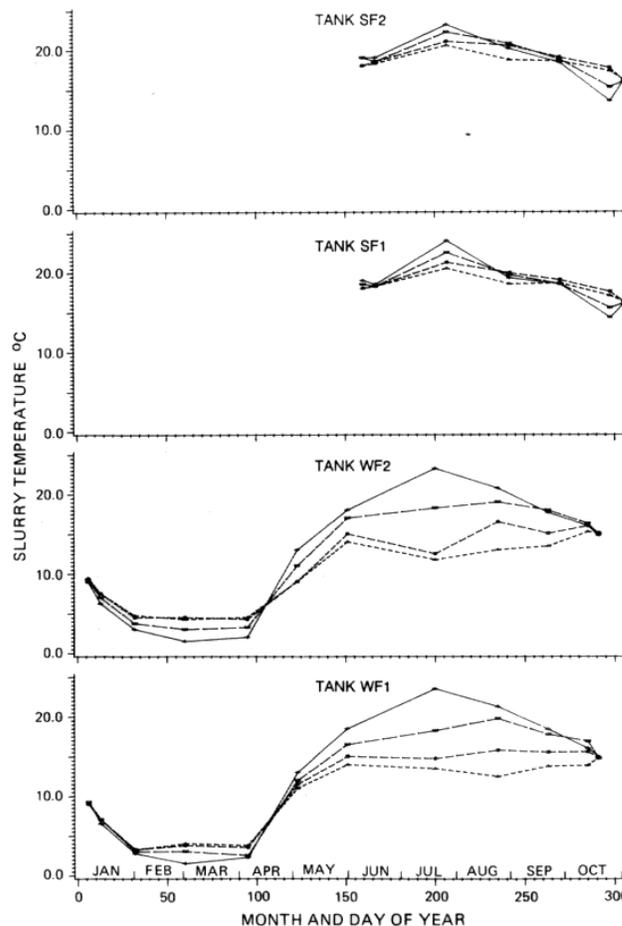
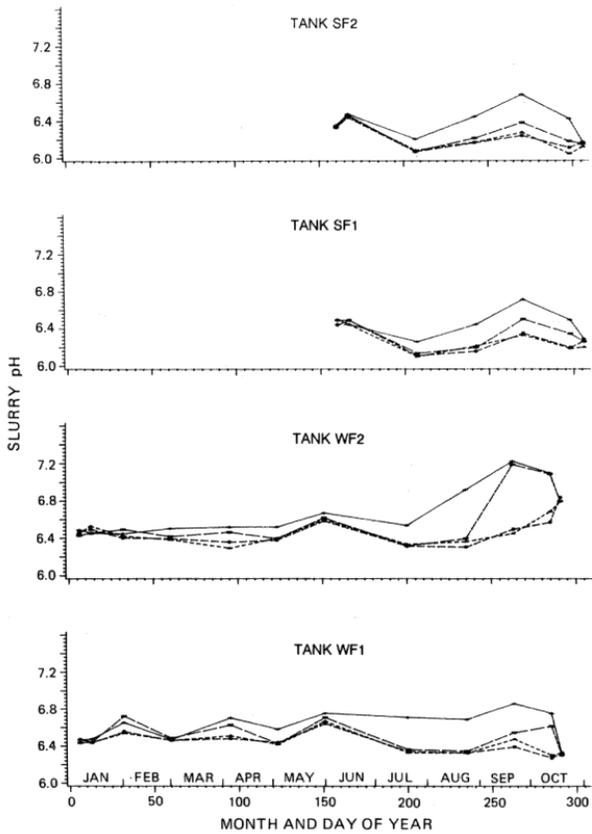


Figure 1—Changes in slurry temperature at depths of 0.3 m (——), 1.0 m (— — —), 1.8 m (— · — ·), and 2.5 m (— · — ·) below the surface. (Note: More broken the line, greater the depth below the surface.) SF = summer filled, WF = winter filled.



**Figure 2—Changes in slurry pH at depths of 0.3 m (—), 1.0 m (— —), 1.8 m (— — —), and 2.5 m (— — — —) below the surface.**

least near the bottom (2.5 m depth). The slurry mean temperature was higher in the summer-filled tanks (16 to 22° C) than in the winter-filled tanks (3 to 17° C) during the storage period. In the latter tanks, slurry temperature did not exceed 16° C at depths greater than 1.8 m.

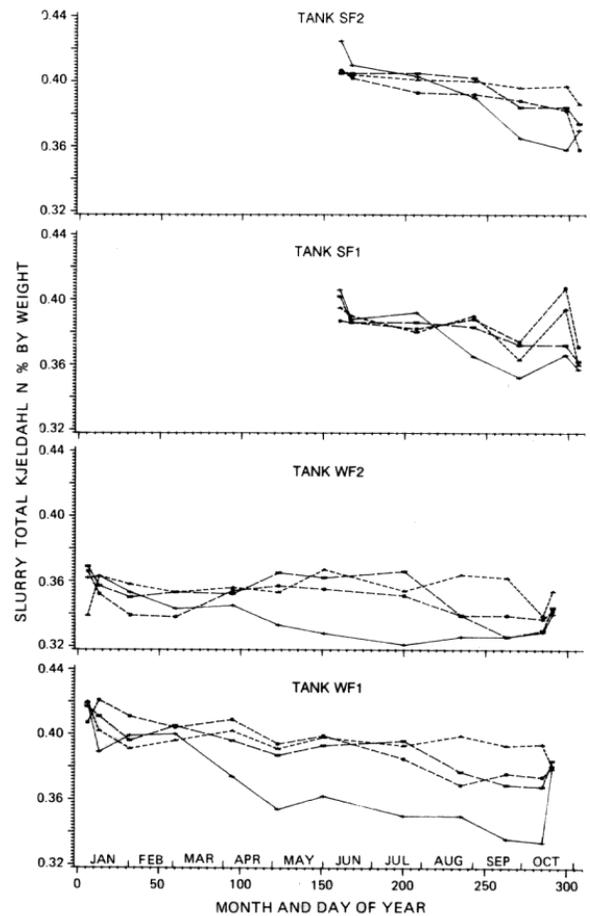
The slurry pH was mostly below 6.8 in all tanks (fig. 2) and varied little below 1.0 m depth. After about fifty days of storage, the pH was significantly ( $P < 0.01$ ) higher at the 0.3 m depth than at greater depths.

The pH changes were similar in the two summer-filled tanks, and also in the two winter-filled tanks until August after which the pH tended to rise at all depths in tank WF2. The relatively small pH change in all tanks suggests that the slurry was well-buffered.

The spatial and temporal variation in the slurry temperature and pH may have provided a different environment for biochemical reactions in different parts of the storage tanks. The slurry temperature and pH were generally less than the recommended optimum temperature of 35 to 37° C and pH of 7.0 to 7.2 for methanogenic cultures (Mah, 1981).

### NITROGEN CONCENTRATION CHANGES

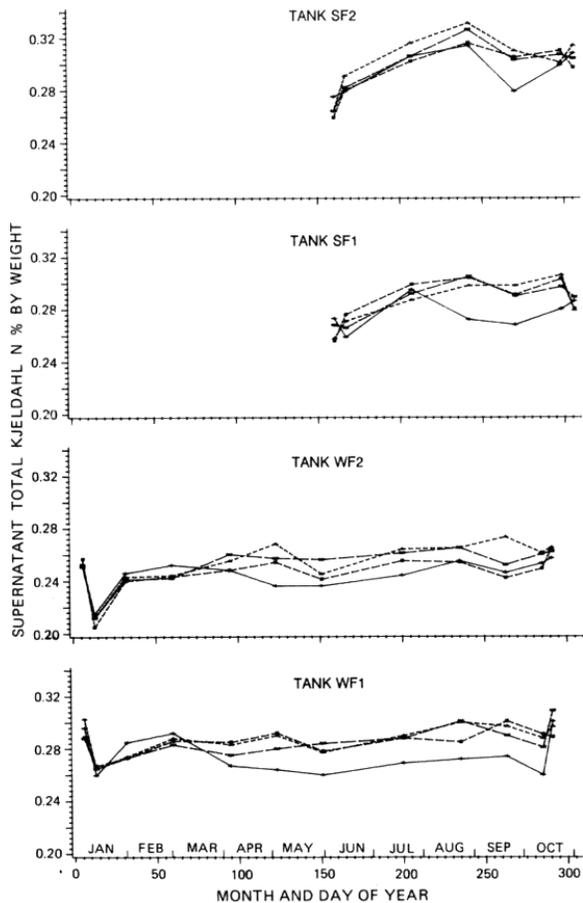
Concentration changes in the slurry and the supernatant are shown in figures 3 and 4 for TKN, and figures 5 and 6 for AMN, respectively. The TKN and AMN concentrations were determined on a weight basis, and are reported as such. The density of manure, as determined from the weight of a fixed volume (approximately 300 mL) of several subsamples of the raw slurry ranged from 0.966 to 1.045 kg L<sup>-1</sup>. This variation was partly due to the presence of entrapped gas bubbles, and some feed and bedding in the slurry. Each concentration point in figures 3 to 6 is the mean of two values (from two samples, one from each of two openings in the tank cover) for a given depth and time. In spite of the concentration variability in figures 3 to 6, some features were common in both the winter- and summer-filled tanks. First, the concentration change with time and depth was small relative to the initial



**Figure 3—Changes in slurry total Kjeldahl nitrogen concentration at depths of 0.3 m (—), 1.0 m (— —), 1.8 m (— — —), and 2.5 m (— — — —) below the surface.**

**TABLE 1. Monthly mean ambient temperatures recorded at a weather station located 8 km from the study site**

Month	Mean max	Mean	Mean min	Month	Mean max	Mean	Mean min
				°C			
January	-10.5	-15.4	-20.3	June	21.9	16.9	11.8
February	-5.1	-9.8	-14.4	July	27.3	20.6	13.8
March	1.5	-3.7	-8.8	August	22.8	17.4	11.9
April	9.5	4.5	-0.6	September	19.9	14.9	9.8
May	21.0	15.2	9.4	October	14.1	9.2	4.2



**Figure 4—Changes in supernatant total Kjeldahl nitrogen concentration at depths of 0.3 m (—), 1.0 (— —), 1.8 m (— — —), and 2.5 m (— · — ·) below the surface.**

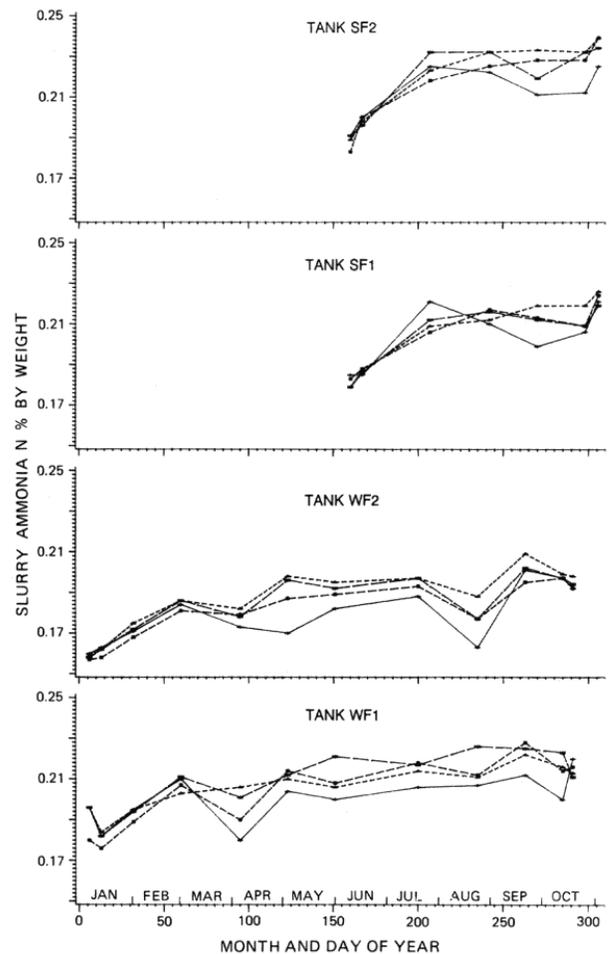
concentration. Second, there was a general trend of decrease in slurry TKN concentration (fig. 3), and increase in slurry AMN and supernatant TKN and AMN (figs. 4,5,6). Third, the rate of concentration change with time was relatively greater for AMN than for TKN. Fourth, concentration variation with depth was small during the initial 50 to 60 days of storage, after which concentrations at the 0.3 m depth were, on average, significantly ( $P < 0.01$ ) lower than at greater depths. Fifth, the supernatant TKN concentration ranged from 65 to 80% of that in the slurry, whereas the AMN concentration tended to be similar in the slurry and the supernatant.

The increase in slurry AMN concentrations in tanks WF1 and WF2 during the January-to-April period (fig. 5) indicates that the low slurry temperatures (fig. 1) during this period did not inhibit mineralization of slurry organic nitrogen (ORN). A general lack of substantial changes in TKN concentrations in these tanks during the same period (fig. 3) suggested that loss of N from the tanks during the winter months was small. The TKN concentration changes in tanks SF1 and SF2 (fig. 3) suggested that most of the N loss from these tanks occurred after July.

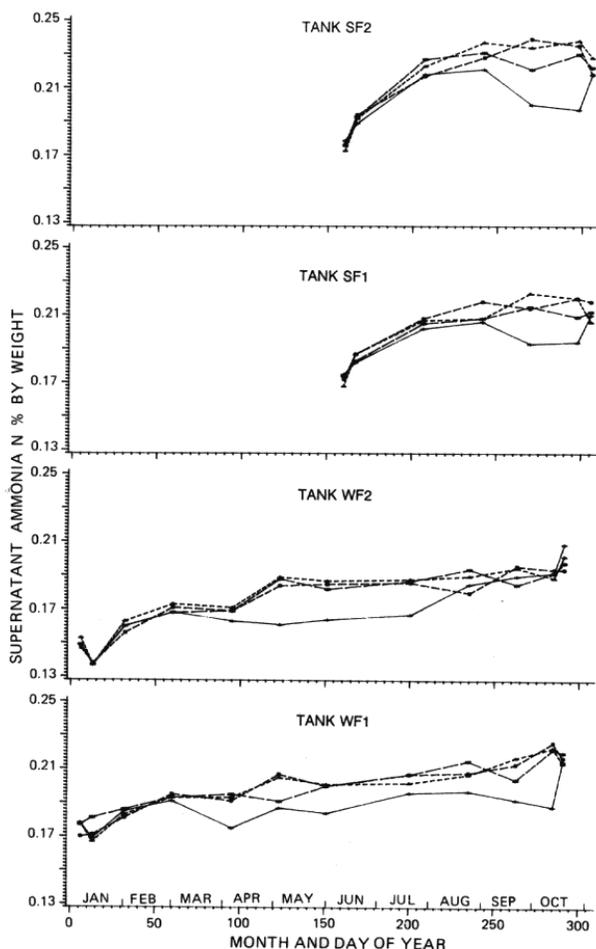
Table 2 shows that N concentrations and the AMN/TKN ratios changed significantly ( $P < 0.05$ ) during storage in all cases except for supernatant TKN in the winter-filled tanks. The initial TKN and AMN concentration was lower in Tank WF2 than in the other tanks because of a greater dilution of manure during its transfer from the barn to this tank. The initial TS concentration was 9.8, 8.8, 10.0, and 10.0% by weight in tanks WF1, WF2, SF1, and SF2, respectively. The changes that occurred in the solids content of the stored slurry have been described earlier (Patni and Jui, 1987). The increase in AMN concentrations and AMN/TKN ratios in the slurry tended to be greater in the summer-filled than in the winter-filled tanks. This suggested that mineralization of ORN to AMN was greater in the former than in the latter, partly because of the generally higher slurry temperature in tanks SF1 and SF2 than in tanks WF1 and WF2 (fig. 1). The AMN/TKN ratio was higher in the supernatant than in the slurry. A dynamic equilibrium value of nearly 0.75 for this ratio was apparently reached at the end of the storage period in the supernatant in all tanks, independent of the date of filling (Table 2). In both the slurry and the supernatant, there was no significant ( $P > 0.05$ ) variation in the AMN/TKN ratio with depth (data not presented).

#### MASS BALANCE FOR NITROGEN IN THE TANKS

Slurry stored in concrete tanks of the type used in our study is predominantly anaerobic and there is little or no oxidized N, that is, nitrate and nitrite present in it (Overcash et al., 1983). Consequently, TKN may be considered to be essentially equivalent to total



**Figure 5—Changes in slurry ammonia nitrogen concentration at depths of 0.3 m (—), 1.0 (— —), 1.8 m (— — —), and 2.5 m (— · — ·) below the surface.**



**Figure 6—Changes in supernatant ammonia nitrogen concentration at depths of 0.3 m (—), 1.0 (— — —), 1.8 m (— · — ·), and 2.5 m (· · · ·) below the surface.**

N in such tanks. Furthermore, ORN in the slurry is practically equivalent to TKN minus AMN because the N in the main N-containing materials in livestock manures (proteins in the undigested feed, intestinal bacteria and cells, urea and ammonia) is determined in the TKN test.

Because of the spatial variation in the slurry TKN and AMN concentrations (figs. 3 to 6), it was not possible to obtain a reasonably reliable mass balance for N in the unmixed slurry at various times during storage. A mass balance for N in well-mixed slurry in the tanks at the beginning and end of the storage period showed that the percentage loss of TKN and gain of AMN in the stored slurry was similar in tanks SF1 and SF2 but not in tanks WF1 and WF2 (Table 3).

Nitrogen loss in tank WF1 was 8.4% (84 kg) compared to 4.4% (38 kg) in tank WF2. This difference was too large to be explained by the variability in analysis and sampling. Mass balance for AMN, was carried out independently of the TKN balance since slurry TKN and AMN were determined separately. It showed that AMN increased by 22.5% (86 kg) in tank WF2 compared to only 11.9% (55 kg) in tank WF1 (Table 3). This suggested a possible presence of some factor in tank WF2 which helped to increase AMN retention in this tank compared to tank

**TABLE 2. Mean TKN and AMN concentrations, and AMN/TKN ratios, in the slurry and the supernatant, initially and after mixing at the end of the storage period\***

Parameter	Mean and standard deviation			
	(285-d storage)		(146-d storage)	
	Tank WF1†	Tank WF2	Tank SF1‡	Tank SF2
<u>Slurry</u>				
TKN, % by wt				
initial	0.42 ± 0.01	0.36 ± 0.01	0.40 ± 0.01	0.41 ± 0.01
final	0.38 ± 0.00	0.34 ± 0.01	0.36 ± 0.01	0.37 ± 0.01
AMN, % by wt				
initial	0.19 ± 0.01	0.16 ± 0.01	0.18 ± 0.01	0.19 ± 0.00
final	0.21 ± 0.00	0.19 ± 0.00	0.22 ± 0.01	0.23 ± 0.01
AMN/TKN				
initial	0.46 ± 0.02	0.44 ± 0.02	0.46 ± 0.02	0.46 ± 0.01
final	0.56 ± 0.01	0.56 ± 0.01	0.61 ± 0.03	0.63 ± 0.03
<u>Supernatant</u>				
TKN, % by wt				
initial	0.29 ± 0.01 <sup>ns</sup>	0.25 ± 0.01 <sup>ns</sup>	0.26 ± 0.01	0.27 ± 0.01
final	0.30 ± 0.01	0.26 ± 0.01	0.28 ± 0.01	0.31 ± 0.01
AMN, % by wt				
initial	0.18 ± 0.00	0.15 ± 0.01	0.17 ± 0.01	0.18 ± 0.01
final	0.22 ± 0.00	0.20 ± 0.01	0.21 ± 0.01	0.22 ± 0.01
AMN/TKN				
initial	0.60 ± 0.01	0.59 ± 0.03	0.65 ± 0.02	0.66 ± 0.02
final	0.73 ± 0.03	0.77 ± 0.04	0.75 ± 0.03	0.73 ± 0.03

Difference between the initial and final values is significant ( $P < 0.05$ ) in all cases except for initial values with the superscript "ns". Each mean value is derived from 8 observations per tank (2 locations x 4 depths).

† WF = winter-filled. ‡ SF = summer-filled.

WF1, even though the storage conditions in both tanks were similar. It is noteworthy that the decrease in ORN was similar in the two winter-filled tanks, and also in the two summer-filled

**TABLE 3. Mass balance for slurry TKN, AMN and ORN during storage**

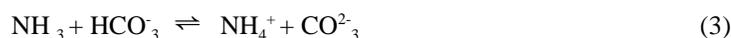
Nitrogen Type	285-d storage		146-d storage	
	Tank WF1	Tank WF2	Tank SF1	Tank SF2
<u>Total Kjeldahl nitrogen (TKN)</u>				
Initial, kg	1005	871	962	952
Final, kg	921	833	878	862
Loss during storage, kg	84	38	84	90
Loss as % of initial TKN content	8.4	4.4	8.7	9.5
<u>Ammonia nitrogen (AMN)</u>				
Initial, kg	464	383	440	437
Final, kg	519	469	538	545
Increase during storage, kg	55	86	98	108
Increase as % of initial AMN content	11.9	22.5	22.3	24.7
<u>Organic nitrogen ORN = TKN - AMN</u>				
Initial, kg	541	488	522	515
Final, kg	402	364	340	317
Decrease during storage, kg	139	124	182	198
Decrease as % of initial ORN content	25.7	25.4	34.9	38.4

tanks. The initial ORN decreased by an average 36.6% in the summer-filled tanks compared to 25.6% in the winter-filled tanks, indicating a greater mineralization of ORN in the former, presumably because of higher slurry temperature during storage. Whereas the decrease in slurry ORN was greater in the summer-filled than in the winter-filled tanks, the opposite was observed for slurry TS and total carbon (TC), as reported earlier (Patni and Jui, 1987). The initial TS and TC in the slurry decreased by an average 20.7 and 22.4%, respectively, in the summer-filled tanks compared to an average 24.9 and 27.0%, respectively, in the winter-filled tanks.

Volatilization of free or un-ionized ammonia (NH<sub>3</sub>) in solution has been suggested as a mechanism for N loss from manure slurries (Overcash et al., 1983). Based on the slurry temperature and pH, and assuming that equilibrium constants for aqueous solutions of NH<sub>3</sub> are applicable to slurries, the un-ionized NH<sub>3</sub> was less than 1% of the AMN in all tanks.

Koelliker and Miner (1973) discussed NH<sub>3</sub> loss from liquid manure surface by the desorption process in which the driving force for the rate of NH<sub>3</sub> loss is the difference in NH<sub>3</sub> partial pressure in the liquid and gas phases. In our study, restricted air movement under the tank cover, and the manure foam on the slurry surface may have limited this driving force. A carbon balance for the stored slurry indicated that 2300 to 3100 kg of TC was lost from the slurry in the tanks during storage (Patni and Jui, 1987). Some movement of NH<sub>3</sub> to the surface may have occurred in the mass flow associated with the formation and release of CO<sub>2</sub> and CH<sub>4</sub> from the slurry due to volatilization of carbon. Thus, stripping of NH<sub>3</sub> by CO<sub>2</sub> and CH<sub>4</sub> may have been an additional mechanism for N loss.

Since the slurry storage conditions and the decrease of ORN were similar in tanks WF1 and WF2, it would be reasonable to assume that the amount of AMN produced by ORN mineralization was also similar in the two tanks (Table 3). The greater retention of AMN in tank WF2 than in tank WF1 could have resulted from a drastic decrease in the concentration of acetic acid at all depths in tank WF2 after July (fig. 7). This would also explain the rise in slurry pH at all depths after July (fig. 2). There was probably a greater amount of CO<sub>2</sub> released in the slurry in tank WF2 than in the other tanks because of acetic acid breakdown after July, and this relative excess of CO<sub>2</sub> could have led to a greater retention of NH<sub>3</sub> to satisfy the following ionic equilibria (Perry, 1963):



Further studies are required to verify this hypothesis. It is clear that several complex and interacting factors are involved in N loss from stored slurries, and that considerations other than changes in N concentration alone may also be important.

## CONCLUSIONS

The following conclusions may be drawn for undisturbed storage of about 10% TS dairy-cattle manure slurry in below-grade, covered, concrete farm tanks, under climatic conditions of southern Ontario.

Temporal and spatial variation of TKN (which may be considered to be equivalent to total N) in the slurry tends to be small irrespective of summer or winter-filling of the tanks, particularly during the initial two months of storage. When uniform nitrogen application rate is the only criterion used for land utilization of slurry, and in the absence of a thick crust formation on the slurry surface, the extra effort and expense of thorough mixing of slurry prior to land application may not always be justified.

Concentrations of TKN and AMN in both the slurry and its liquid fraction tend to be slightly lower in the top 1 m depth than at greater depths after prolonged undisturbed storage of slurry in farm tanks.

During storage, the slurry TKN decreases but the slurry AMN as well as the liquid-fraction TKN and AMN increase. Nitrogen loss from stored slurry cannot be avoided even when the slurry is not disturbed during storage in covered tanks. Loss of N amounts to about 4 to 9% of the initial N content.

The extent of N loss due to NH<sub>3</sub> stripping by CO<sub>2</sub> and CH<sub>4</sub> released in fermenting slurry needs further research.

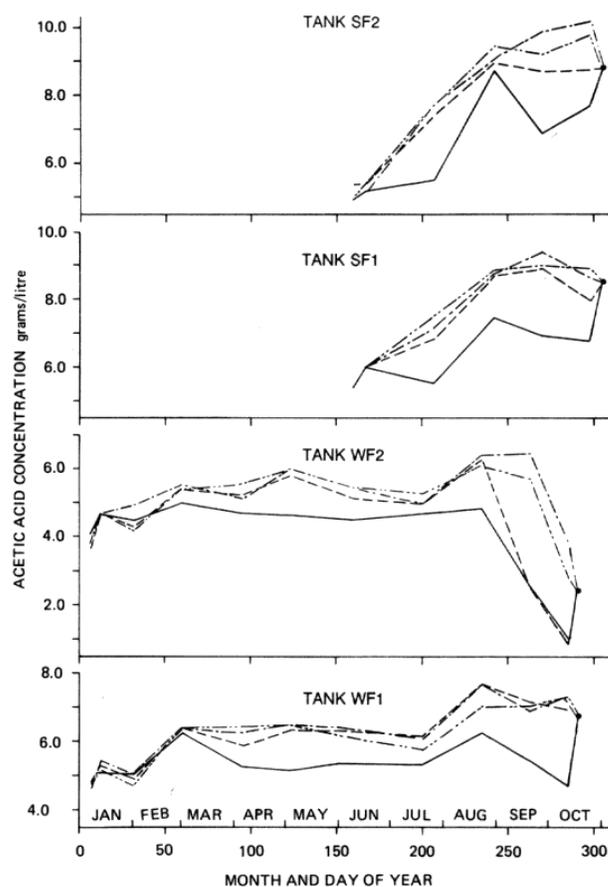


Figure 7—Changes in concentration of acetic acid with time at depths of 0.3 m (—), 1.0 m (---), 1.8 m (-·-·-), and 2.5 m (- - - -) below the surface.

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