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## Can natural $^{15}\text{N}$ abundance of hog manure be used to trace nitrate leaching?

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### Introduction

Hog manure is a relatively rich source of nitrogen, phosphorous, and organics. Although it is these characteristics of manure that contribute to its notoriety as a significant environmental hazard they also are the key characteristics that make hog manure a potentially valuable fertilizer/soil amendment. For example, when applied in an appropriate manner and incorporated into agricultural soils, hog manure can supply much of the N and phosphorous needed for crop production (Kaminiski & Kaminiski, 1993). In addition, the incorporation of hog manure into some soils can benefit particle aggregation and soil tilth. The organic compounds in manure act as a binding agent which can stabilize soil structure and consequently positively impact water infiltration and water holding capacity while providing resistance to wind and water erosion (Larney et al., 1991). On the negative side, the nitrogen, phosphorous, and organics in hog manure represent potential contaminants that can lead to the accelerated eutrophication of surface waters as well as the fouling of groundwater.

Of all of the N occurring on earth most of it is  $^{14}\text{N}$  (99.634 %), with the remaining 0.366 % existing as  $^{15}\text{N}$ . It is the distribution of  $^{15}\text{N}$  relative to  $^{14}\text{N}$  that is interesting biologically. Nitrogen-15 concentrates in biological tissues as it passes through the food chain. Animals eating plant products will be more enriched in  $^{15}\text{N}$  than the plants they ate. The amount of  $^{15}\text{N}$  in the plants will vary with the concentration of  $^{15}\text{N}$  in the inorganic N taken up through their roots. By measuring the amount of  $^{15}\text{N}$  relative to  $^{14}\text{N}$  in biologically derived tissues ( $\delta^{15}\text{N}$ ) we can get information about where the N originated from. For example, whereas the nitrate derived from soil organic matter normal exhibits  $\delta^{15}\text{N}$  values in the 4 to 9‰ range, commercial nitrate fertilizers typically exhibit  $\delta^{15}\text{N}$  values in the -4 to 4 ‰ range, and the  $\delta^{15}\text{N}$  for nitrate derived from animal wastes is typically in the 10 to 22‰ range (Heaton, 1986).

Nitrate-N is extremely mobile and can be quite easily leached from the soil. Consequently, groundwater contamination by manure-derived nitrate represents a potentially serious environmental problem. The ability to determine the source of nitrate contamination will help us to adjust application rates and management strategies to eliminate or minimize contamination. A number of studies have demonstrated that the measurement of natural abundance levels of stable N isotopes (i.e.,  $\delta^{15}\text{N}$ ) is a useful tool for discriminating among nitrate sources in groundwater (Wells and Krothe, 1989; Komor and Anderson, 1993). Typical  $\delta^{15}\text{N}$  values for animal wastes range from 10 to 22‰ (Heaton, 1986), although will depend largely on what the animal is fed. Furthermore, in order to be useful as a tracer, the manure  $\delta^{15}\text{N}$  value must be considerably different from the soil to which it is applied. Because of biological N transformations occurring in soils, Saskatchewan soils tend to have fairly high  $\delta^{15}\text{N}$

values that may preclude using  $\delta^{15}\text{N}$  to trace manure-nitrogen. A survey of the variation in natural  $^{15}\text{N}$  abundance in soil organic matter for 58 surface soils from central Saskatchewan reported a mean  $\delta^{15}\text{N}$  of  $9.4 \pm 3.0$  ‰ (Karamanos et al., 1981).

In addition to tracing nitrate leaching,  $\delta^{15}\text{N}$  measurements could be useful for quantifying crop uptake of nitrogen.  $^{15}\text{N}$  techniques offer the only direct way of measuring plant uptake from applied N-fertilizers (Zapata, 1990) and commercial fertilizers enriched in  $^{15}\text{N}$  are routinely used to measure N use efficiency by crops.

The objective of this study is to determine if manure-N collected from 15 hog manure lagoons across Saskatchewan is sufficiently enriched or depleted in  $^{15}\text{N}$  to distinguish it from soil-nitrogen. This information will give us the background necessary to assess the feasibility of using natural abundance  $^{15}\text{N}$  levels to trace nitrate leaching and crop uptake of N from manure sources. An additional objective was to determine if different lagoon management features affected the amount of K, P, ammonia and total N in the manure.

## **Methods and Materials**

### ***Manure collection:***

Manure samples were collected from 15 lagoons across Saskatchewan. In the case of double-celled lagoons, only the primary cell was sampled. This cell contains more solid particulate organic matter than the secondary cell and is the cell from which manure is pumped for field applications. Typically two samples were collected from opposite sides of each lagoon, for a total of four samples per lagoon. However, at two of the lagoons only one side of the lagoon was sampled because of problems with the sump pump plugging.

Manure samples were collected from a lagoon using a sump pump connected to polyvinylchloride (PVC) tubing. The tubing was assembled in 1.5-m lengths so that it could be easily transported and cleaned, and the length adjusted to the depth of an individual lagoon. The sump pump was lowered to the bottom of a lagoon by easing it into the lagoon with the PVC tubing. Once the pump reached the lagoon bottom it was raised approximately 0.5 m off of the lagoon floor. The sump pump was primed for 1-2 minutes by pumping effluent through the pump and tubing and returning it back to the lagoon. After priming, approximately 5 L of effluent was collected in a bucket and 1 L subsequently transferred to a collection jar, stored on ice and returned to the laboratory for analysis.

### ***Laboratory analyses:***

To prevent volatilization of ammonia, sub-samples of the manure were acidified to pH 3-4 using concentrated  $\text{H}_2\text{SO}_4$ . The sub-samples were freeze-dried and the remaining residue analysed for  $\delta^{15}\text{N}$  using a TracerMass stable isotope converter interfaced with a RoboPrep sample converter (Europa Scientific, UK).

Composite samples from each side of the lagoon sampled (generally 2 composite samples per lagoon) were analysed for K, P, ammonia, and total N composition. No

nitrate was detected. The manure samples were acid digested (Thomas et al., 1967). Digestion tubes containing glycine were included as controls for quantifying N recovery. Potassium was detected in the digestion solution by atomic absorption spectroscopy, P, ammonia and total N were detected using autoanalysers.

Soil samples from fields amended with four times the recommended rate of manure and field not amended with manure were analysed for  $\delta^{15}\text{N}$ . A composite sample was prepared from four 10-cm depth soil cores (i.d.=40 mm) and replicate 10-mg subsamples analysed on the mass spectrometer for  $\delta^{15}\text{N}$ .

#### ***Statistical analyses:***

Manure samples were grouped according to a number of lagoon characteristics that could potentially affect the form and/or amount of N retained in the lagoon. These four characteristics were: (1) the type of lagoon sampled (single- vs. double-celled); (2) the presence or absence of straw cover; (3) the type of operation (nursery, breeder, finishing, farrow to finish, breeder/finisher); and (4) the operator (Quadra, Big Sky, Heartland, Independent).

Data were analysed by the General Linear Model for the effect of the four lagoon characteristics on the  $\delta^{15}\text{N}$  values of the manure. Mean manure  $\delta^{15}\text{N}$  values were compared to published soil  $\delta^{15}\text{N}$  values and assessed for their potential use as a natural tracer.

### **Results and discussion**

The overall mean  $\delta^{15}\text{N}$  value for the 52 manure samples was  $11.1 \pm 2.8$  ‰ and was well within the reported range for manure (i.e., 10-22 ‰; Heaton, 1986). Overall, the manure  $\delta^{15}\text{N}$  values were too similar to published  $\delta^{15}\text{N}$  values for soils in Saskatchewan ( $9.4 \pm 3.0$  ‰, n=38; Karamanas et al., 1981) to be of general value for tracing manure derived N through the soil-water-plant continuum (Table 1). Specifically, the variability of manure  $\delta^{15}\text{N}$  overlaps with the  $\delta^{15}\text{N}$  for soil. Furthermore, the application of manure to soils in the field did not result in  $\delta^{15}\text{N}$  values that were significantly different from the check plots where no manure was added (Table 2). However, measuring  $\delta^{15}\text{N}$  on the total soil may have masked differences in  $\delta^{15}\text{N}$  among specific N pools as a result of manure application. Measurement of  $\delta^{15}\text{N}$  in specific N pools was beyond the scope of this study. That soil  $\delta^{15}\text{N}$  values tended to be higher immediately after manure application, suggests that the manure may have altered existing  $\delta^{15}\text{N}$  pools. The higher  $\delta^{15}\text{N}$  in the soils sampled immediately after manure application compared to the same soils amended one year previously and sampled just prior to the second manure application may be a cumulative effect of repeated manure application. Alternatively, it could also represent a reduction in soil  $\delta^{15}\text{N}$  over time as the result of plant uptake of inorganic N enriched in  $^{15}\text{N}$ , or the leaching of  $^{15}\text{N}$ -nitrate below the 10-cm depth that the soil cores were sampled.

Table 1.  $\delta^{15}\text{N}$  values according to lagoon management characteristics for swine manure sampled from 15 lagoons across Saskatchewan. Between 2 and 4 samples were analysed for each lagoon.

		Mean $\delta^{15}\text{N}$ (‰)	SD	n
Lagoon	Single-celled	11.1	1.8	16
	Double-celled	10.9	3.6	38
Cover	None	10.8	3.3	10
	Straw	11.7	2.8	44
Operation <sup>1</sup>	Nursery	10.2	1.9	8
	Breeder	12.2	2.3	8
	Finishing	3.9	2.2	4
	Farrow to finish	11.9	2.8	30
	Breeder-finishing	10.3	1.2	4
Operator	Quadra	11.9	2.7	26
	Big Sky	11.1	1.8	16
	Heartland	8.4	4.5	12
	Independent	11.8	4.7	2
Overall mean		11.1	2.8	52

<sup>1</sup>Significant at  $\alpha=0.05$ .

Table 2. A comparison of  $\delta^{15}\text{N}$  values for soils amended with swine manure vs. unamended (check) soils.

		Treatment	$\delta^{15}\text{N}$ (‰)	n
Prior to manure application <sup>1</sup>	Check		6.6 (0.5)	4
	4x recommended rate		6.8 (0.5)	4
Post manure application <sup>2</sup>	Check		6.6 (0.7)	4
	4x recommended rate		7.2 (0.4)	4

<sup>1</sup>Soils sampled on October 6, 1998. Manure injected sub-surface in October, 1997.

<sup>2</sup>Soils sampled on October 22, 1998. Manure injected sub-surface October

The usefulness of  $\delta^{15}\text{N}$  values to identify the source of nitrate contaminants is complicated by the fact that N isotopes are fractionated in the soil environment; i.e., natural transformations of soil N cause changes in the  $\delta^{15}\text{N}$  value between the substrate and the end products. Because of differences in the chemistry of compounds containing  $^{14}\text{N}$  and  $^{15}\text{N}$ ,  $^{14}\text{N}$  compounds are used preferentially. Consequently, processes such as denitrification result in increased values of  $\delta^{15}\text{N}$  for the residual nitrate that is left in the soil and decreased  $\delta^{15}\text{N}$  values for the nitrous oxide and N gases that escape to the

atmosphere (Smith et al., 1991; Farrell et al., 1996). Because so many potential transformation processes exist in the soil (e.g., denitrification, nitrification, volatilization, immobilization and mobilization) it is difficult to predict the nature of the resulting  $\delta^{15}\text{N}$  value for a particular soil N pool. In order to prove useful as a tracer the difference between  $\delta^{15}\text{N}$  values of the soil and the manure will probably need to be in the order of at least 5 ‰ units.

Of the lagoon factors investigated for potentially affecting manure  $\delta^{15}\text{N}$  values, only the type of operation had an affect (Table 1) and this was attributable to manure collected from finishing barns. Only four samples from a single finishing barn operated by Heartland were analysed. Although it cannot be generalized that manure from all finishing barns will have  $\delta^{15}\text{N}$  values that are lower than soil values, this single result does indicate that there is potential for matching a manure source with a specific field in order to trace leaching and/or plant uptake of manure derived nitrate.

A tremendous amount of variability existed for nutrient compositions of manure from the different lagoons (Table 3). The type of operation affected K, P, ammonia and total N and was probably due to animal age and food source. For all of the nutrients, manure from the finishing barns and breeder/finishing barns tested the lowest amounts. However, again only one lagoon from each of these operations was sampled, and may represent an anomalous situation.

Covering the surface of a lagoon with barley straw is used as to effectively control odors (PAMI, 1993). It appears that at least some of the odor control is due to a reduction in ammonia lost from the lagoon (Table 3). Manure from lagoons with straw cover had approximately twice the amount of ammonia than lagoons with no straw cover. This was also reflected in total N amounts, with manure from straw covered lagoons having approximately twice as much total N as lagoons with no straw cover. Although the inclusion of straw cover affected the amounts of N in a manure sample, it did not affect the  $\delta^{15}\text{N}$  value of the manure.

### ***Summary:***

The study was conducted as a preliminary survey to assess the feasibility of using  $\delta^{15}\text{N}$  values of manure to trace nitrate movement through the soil-plant-water and to assess the variability of manure  $\delta^{15}\text{N}$ . Overall,  $\delta^{15}\text{N}$  values for manure and soil were too similar to be of general use as a tracer. Furthermore, substantial variation was associated with manure  $\delta^{15}\text{N}$  values limiting their generalized use. However, it appears that manure samples and soil samples could be selected for widely different  $\delta^{15}\text{N}$  values, providing a 'custom match' for tracing manure derived nitrate.

Table 3. Nutrient analysis according to lagoon management characteristic for swine manure sampled from 15 lagoons across Saskatchewan. One or two composite samples were analysed for each lagoon.

		K ( $\mu\text{g/g}$ )	P ( $\mu\text{g/g}$ )	NH <sub>4</sub> ( $\mu\text{g/g}$ )	Total N ( $\mu\text{g/g}$ )	n
Lagoon	Single-celled	1260 (500)	1646 (2066)	1539 (723)	2926 (1658)	7
	Double-celled	1288 (406)	924 (1321)	1956 (931)	3068 (1878)	20
Cover	None	1575 (355)	1466 (1529)	1615 (731) b	2578 (1373) b	5
	Straw	1213 (414)	1030 (1563)	2874 (838) a	4929(2239) a	22
Operation	Nursery	1410 (284) a	165 (22) a	1387 (566) ab	1779 (673) bc	3
	Breeder	1535 (320) a	3773 (1505) b	1480 (114) ab	4387 (736) a	4
	Finishing	619 (12) b	245 (248) a	1055 (7) b	1488 (217) bc	2
	Farrow to finish	1364 (353) a	858 (1126) a	2282 (863) a	3415 (1869) ab	16
	Breeder-finishing	568 (17) b	97 (44) a	587 (4) b	847 (31) c	2
Operator	Quadra	1303 (360)	723 (1093)	2266 (955)	3273 (2060)	12
	Big Sky	1260 (500)	1645 (2065)	1539 (722)	2926 (1658)	7
	Heartland	1163 (490)	1389 (1843)	1175 (187)	2483 (1622)	6
	Independent <sup>1</sup>	1824	750	2610	4114	1
Overall mean		1280 (423)	1111 (1537)	1848 (888)	3030 (1790)	27

<sup>1</sup>Only one sample was analysed, so no standard deviation could be calculated.

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