Evaluation of Methods for Determining NH_3 and N_2O Emissions from Soil Applied Manure

Alberta
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FOREWORD

Ammonia and nitrous oxide emissions from soils with manure application have potential impacts on air quality and greenhouse effects, respectively. The extent of gas emissions from soil surface is a function of the micrometeorological conditions, including temperature, wind condition and radiation, as well as soil conditions, e.g. temperature and water content, and method of manure application. Both soil water and pH conditions have strong influences on the flux of NH_3 and N_2O from soil surfaces. The chamber method has been used extensively for measuring gas exchange between soil surfaces and the atmosphere and has the advantages of low cost and ease of use.

Three measurement schemes are commonly used for the chamber method: the open chamber method, the closed chamber static method, and the closed chamber dynamic method. All methods employ an inverted chamber covering a small area of soil. The lower edge of the chamber is usually inserted into the soil to a shallow depth. In the open chamber method, pumps are used to provide a steady airflow through the chamber and the concentrations of the target gas are measured at both the inlet and outlet. In the closed chamber static method, the chamber is closed and a chemical absorbent, which acts as a chemical trap, is placed inside the chamber. In the closed chamber dynamic method, the concentration of the target gas in the chamber is monitored over time. The increase in concentration of the gas is used to calculate the rate of gas emissions from the covered soil surface.

This report evaluates the measurement schemes and describes the development of measuring protocols for ammonia (NH_3) and nitrous oxide (N_2O) emissions from soil treated with cattle or hog manure. It consists of theoretical considerations for a new equation and its application. Also, this report explored the possibility of using Tunable Diode Laser (TDL) technology for measuring NH_3 emissions from the soil surface. The project was supported by Alberta Environment's Air Research User Group funding.

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SUMMARY

The chamber method for measuring NH_3 and N_2O emissions from soil was modified and a sampling protocol developed and tested in the field. N_2O and NH_3 emissions were measured using a dynamic chamber method; NH_3 emissions also were measured using a static chamber method. Gas chromatography (GC) was used to determine the concentrations of N_2O and NH_3 in air samples from the dynamic chambers. A solution of 0.02 N H_2SO_4 was used as an absorbent chemical to trap NH_3 emissions from the static chambers. NH_4^+ content was measured by Technicon.

After applications of cattle manure, N_2O emissions from the soil ranged from 0 to 1.28×10^3 $\mu g\ m^{-2}h^{-1}$ and NH_3 emissions ranged from 0 to 1.4×10^3 $\mu g\ m^{-2}h^{-1}$. Ammonia emissions reached 8×10^3 $\mu gm^{-2}h^{-1}$ one day after hog manure was applied and then declined rapidly. The rate of NH_3 and N_2O emissions was positively correlated with the rate of manure application, with the highest rate of manure application for both cattle and hog manure giving the highest rates of emissions.

Measurements from both chamber methods are comparable to data found in the literature, suggesting that the methods are suitable for measuring NH_3 and N_2O emissions in the reported ranges. A direct comparison between the two methods could not be made for NH_3 because the GC failed to detect NH_3 in air samples collected using the dynamic method.

Estimates of NH_3 emissions obtained using the Tunable Diode Laser (TDL) technology and the Backward-time Lagrangian stochastic model were considerably higher than those measured by the chamber methods. Considering the total amount of N applied in the manure, the TDL method seems to overestimate the rates of emissions. This requires further investigation.

More work is needed to refine sampling and analytical protocols. To obtain NH_3 dynamic measurements an analyzer with a detection limit in the range of ppb is required.

1.0 INTRODUCTION

The chamber method has been used extensively for measuring gas exchange between soil surfaces and the atmosphere and has the advantages of low cost and ease of use. It is the method of choice for in-process level studies (Huchinson 1993). The major disadvantage of the method is the small sampling area covered in each measurement. When there are large spatial variations in the rate of gas emissions from soils, the number of replicate measurements required to provide a reasonable field average can be large. Examples of the application of the method are found in Huchinson and Mosier (1981) and Anthony *et al*. (1995).

Three measurement schemes are commonly used for the chamber method: the open chamber method, the closed chamber static method and the closed chamber dynamic method. All methods employ an inverted chamber covering a small area of soil. The lower edge of the chamber usually is inserted into the soil to a shallow depth.

In the open chamber method, pumps are used to provide a steady airflow through the chamber and the concentrations of the target gas are measured at the inlet and outlet. When a steady state is reached the product of the difference in concentrations between the inlet and outlet gas and the airflow is assumed to be the rate of gas emission from the soil surface covered by the chamber. The method requires constant monitoring of airflows and concentrations. In addition, small differences in air pressure between the air in the chamber and the atmosphere, caused by the airflow, may cause significant error, depending on the geometry of the system design (Iritz *et al*. 1997, Fang and Moncrieff 1998).

In the closed chamber static method, the chamber is closed and a chemical absorbent, which acts as a chemical trap, is placed inside the chamber. After the chamber is closed for a time, typically 24 hours, the chemical trap is removed and the total amount of the target gas absorbed in the trap is measured. The amount of gas absorbed, divided by the soil surface area covered by the chamber and time the chamber was closed, is assumed to be the rate of gas emission from the soil. Studies have shown that this method corresponds well with other methods at low gas flux rates but not at high flux rates (Rochette *et al*. 1992).

In the closed chamber dynamic method the concentration of the target gas in the chamber is monitored over time (Huchinson and Mosier 1981, Huchinson 1993, Anthony *et al*. 1995, Rochette *et al*. 1992). The increase in concentration of the gas is used to calculate the rate of gas emission from the covered soil surface. One problem with this method is that the concentration of gas in the chamber does not increase linearly with time. Several processes affect the accumulation of gas in the chamber, including the emission rate, soil properties, and the geometry of the chamber system (Healey *et al*. 1996). To overcome the problem, Anthony *et al*. (1995) developed a diffusion-based equation for analysis of gas concentrations in a closed chamber. They assumed that gas production in soil is concentrated at a fixed depth and did not consider possible leakage around the edges of the chamber. The analysis presented in this report is a new equation for analysis of dynamic chamber measurement data. No assumption is made about the depth distribution of gas sources. Leakage from the chamber is considered as a function of chamber size, depth of insertion into the soil, and soil physical properties.

This report describes the development of measuring protocols for ammonia (NH_3) and nitrous oxide (N_2O) emissions from soil treated with cattle or hog manure. It consists of theoretical considerations for a new equation and its application. We used the closed chamber static method, using a chemical trap, to measure NH_3 and the closed chamber dynamic method to measure N_2O and NH_3. The gas analyzer failed to detect NH_3 in samples from the closed chamber dynamic method; therefore, we only reported N_2O results from this method.

We also explored the possibility of using Tunable Diode Laser (TDL) technology for measuring NH_3 emissions from the soil surface.

2.0 MATERIALS AND METHODS

2.1 Theoretical Analysis for the Chamber Method

In measuring gas emission by a chamber method, a soil chamber is inserted to a shallow depth into the soil. Emission of the target gas from the soil into the chamber results in increases in the concentration of the gas in the chamber. The change in concentration is used to calculate the rate of gas emission. In the simplest application, the rate of gas emission is calculated by:

$$Q = V/A\ (C-C_0)/t \qquad [1]$$

Where Q is the emission rate, V is the volume of the chamber, A is the area of soil covered by the chamber, C is the concentration of the gas in the chamber at time t, and C_0 is the initial concentration at t = 0 (Hutchinson and Livingston 1993).

The calculation assumes that the gas concentration in the chamber increases linearly with time. Research has shown that this is not the case (Hutchinson and Mosier 1981; Hutchinson and Livingston 1993). After the soil surface is covered by a chamber the gas concentration in the chamber increases non-linearly with time and will approach a maximum. Assuming a linear relation may lead to significant error in the calculated rate of gas emission. Strictly speaking, equation [1] only applies at t->0. To overcome the problem, most researchers attempt to use small time measurements to minimize the impact of non-linearity; however, technical difficulties may preclude measurement at sufficiently short time intervals (Anthony and Hutchinson 1990). Another approach is to derive theoretically based, non-linear relations that can be used to fit the experimental data to obtain correct emission rates (Hutchinson and Mosier 1981).

Several factors contribute to the non-linear increase in gas concentration in the chamber with time. First, as the gas concentration builds up, a small reverse concentration gradient develops in the soil profile. The gradient counteracts the gas gradient in the soil, thus reducing the rate of emission. The increased gas concentration in the soil accounts for some of the gas that otherwise would have been emitted from the soil surface and thus reduces the amount of gas emitted into the chamber. The magnitude of this effect is a function of chamber geometry, depth of gas production in the soil profile, soil air-filled porosity, and the gas diffusion coefficient in the soil. Secondly, because of the increased concentration of gas in the chamber, a concentration gradient builds up between the chamber and the surrounding air. A small leakage flux may develop between the chamber and the surrounding air through the rim of the chamber. The magnitude of this effect is a function of soil gas diffusion coefficient, chamber geometry (diameter, height), and the depth to which the chamber is inserted into the soil. Thirdly, because of changes in soil temperature, the rate of gas production in the soil is not a constant and the gas concentration gradient in the soil may not be at a steady state, as is assumed in most chamber measurements. The non-linearity between chamber gas concentration and time depends on whether the gas production rate in the soil is increasing or decreasing. The magnitude of the effect is a function of soil gas diffusion coefficient and the depth of gas production.

There has been no satisfactory treatment in the literature of the impact of these factors on the results of gas emission measurements with the chamber method. Hutchinson and Mosier (1981) attempted to develop a non-linear relation that accounted for diffusion and the changing concentration gradient in the soil profile during chamber measurement. Several of their assumptions are questionable; for example, the assumption that the gas concentration at a given depth in the soil profile remains constant and changes linearly from this constant concentration to that of the chamber air at the soil surface during measurement.

In light of these problems, rigorous, theoretically-sound relationships are needed that account for the impacts of chamber geometry, soil properties (porosity and diffusion coefficient), and the rate of gas production on the measurement of gas emission using the chamber method.

A schematic of the chamber system is shown in Figure 1. The chamber has a radius “r”, is inserted into the soil to a depth “d”, and has a headspace above the soil surface of height “h”. The volume in the headspace of the chamber is V.

The concentration of gas in the chamber is affected by several processes. As the concentration difference between the air inside the chamber “C” and the air outside the chamber “Ca” develops a small first order leakage is assumed to occur because of the imperfect seal around the edge of the chamber. The leakage is calculated by:

$$q_L = k_L (C - C_a) \qquad [2]$$

where q_L is gas flux due to leakage and k_L is the leak coefficient. As the gas inside the chamber builds up, the concentration gradient between the soil and air in the chamber changes. This leads to changes in gas concentrations in the soil profile.

Because of limitations in obtaining data, we assume that the soil profile is at a steady state at the beginning of the measurement. This assumption eliminates the need to know the distribution of gas production as a function of depth in the soil. A more complete theory that accounts for transient changes in gas production in soils would require information about the distribution of gas production in the soil profile, as well as how gas production changes with time. In most cases, this information is not available.

We assume that the gas concentration throughout the soil profile is at steady state with a gas generation rate of q_0 per unit of soil surface area. Because the soil gas concentration is at steady state, this is also the gas flux at the soil surface. A soil flux, as a result of changes in gas concentration in the soil after placement of the chamber, can be defined as:

$$q_s = A\theta_a \frac{d}{dt}\int_{-\infty}^{0} [C_s(z) - C_0(z)]dz \qquad [3]$$

where A is the area covered by the chamber, θ_a is the air-filled porosity of the soil, $C_0(z)$ is the gas concentration in the soil at steady state before placement of the chamber, and $C_s(z)$ is the gas concentration in soil after chamber placement, assumed to be a function of time. The integration is a theoretical convenience that accounts for possible gas production anywhere in the soil profile. Because of the assumption of a steady state before measurement, the specific depth at which gas is produced is not important. This depth only becomes important when one considers the impact on chamber measurements of dynamic changes, such as the diurnal cycle, in gas production in soils.

Assuming that the measurement process does not significantly change the rate of gas generation in the soil, consideration of the conservation of mass will result in:

$$(C - C_a)V + \int_0^t (q_s + q_L)dt = q_0 At \qquad [4]$$

To obtain q_s, one needs to solve the equation for gas diffusion through the soil. For simplicity, we consider one-dimensional gas diffusion. The equation of gas diffusion through the soil profile then can be written as:

$$\theta_a \frac{\partial C_s}{\partial t} = D \frac{\partial^2 C_s}{\partial z^2} + g(z) \qquad [5]$$

where g(z) is the rate of gas generation as a function of depth z. The mass conservation requirement demands that:

$$\int_{-\infty}^{0} g(z)dz = q_0 \qquad [6]$$

The initial and boundary conditions for the gas diffusion equation are:

$C_s = C_0(z)$, $t=0$
$C_s = C(t)$, $z=0$
$\partial C_s/\partial z = 0$, $z=-\infty$

Our objective is to express changes in gas concentration in the chamber as a function of time; therefore, a detailed solution of gas concentration distribution, as given by the diffusion equation, is not necessary. All that is needed is an expression of q_s.

If the total gas concentration in the soil is expressed as:

$$C_s = C_0(z) + \delta C \qquad [7]$$

where δC is deviation from the steady state concentration profile, then the steady state concentration profile satisfies the diffusion equation:

$$0 = D \frac{\partial^2 C_s}{\partial z^2} + g(z) \qquad [8]$$

with the boundary conditions:

$C_0 = C_a$, $z=0$
$\partial C_0/\partial z = 0$, $z=-\infty$

If the concentration deviation, δC, satisfies the diffusion equation:

$$\theta_a \frac{\partial \delta C}{\partial t} = D \frac{\partial^2 \delta C}{\partial z^2} \qquad [9]$$

with the initial and boundary conditions:

$\delta C = 0$, $t=0$
$\delta C = C - C_a$, $z=0$
$\partial \delta C / \partial z = 0$, $z=-\infty$

then the total concentration $C_s = C_0 + \delta C$ is the solution to the original diffusion problem.

The concentration deviation "δC" is the solution for diffusion into a semi-infinite medium with surface concentration given as $C - C_a$. The quantity we are seeking is:

$$q_s = A\theta_a \frac{d}{dt}\int_{-\infty}^{0} [C_s(z) - C_0(z)]dz = A\theta_a \frac{d}{dt}\int_{-\infty}^{0} \delta C dz \qquad [10]$$

For the differential equation defining δC, the solution is:

$$\int_0^t q_s dt_s = A\theta_a \sqrt{\frac{4\theta D_a}{\pi}}\sqrt{t} \otimes \frac{dC}{dt} \qquad [11]$$

where $\otimes$ means convolution.

We may now revisit the mass conservation equation:

$$(C - C_a)V + \frac{dC}{dt} \otimes A\sqrt{\frac{4\theta_a D}{\pi}}\sqrt{t} + \int_0^t k_L (C - C_a)dt = q_0 A t \qquad [12]$$

Solution for this equation can be found using Laplace transform. The Laplace transform of the above equation is:

$$(\overline{C} - \frac{1}{s}C_a)V + A\sqrt{\theta_a D}\,\frac{1}{s^{3/2}}(s\overline{C} - C_a)\sqrt{t} + \frac{k_L}{s}\left(\overline{C} - \frac{1}{s}C_a\right) = \frac{1}{s^2}q_0 A \qquad [13]$$

where s is the Laplace domain variable, and a symbol with a bar at the top indicates Laplace transform. For example, $\overline{C}$ is the Laplace transform of C. From this equation we can find:

$$\overline{C - C_a} = \frac{A q_0}{k_L s + A\sqrt{\theta_a D}s^{3/2} + V s^2} \qquad [14]$$

The gas concentration in the chamber is found by the inverse Laplace transform. Unfortunately, an explicit expression of the inverse Laplace transform of this equation can not be found. Although numerical inversions can be done, they are inconvenient to use. Therefore, we would seek a simplified approximate solution. For short periods of time, which is the case for most measurements of emissions, the approximate solution is:

$$\frac{(C-C_a)h}{t} \approx q_0 - \frac{4q_0\sqrt{\theta_a D}}{3h\sqrt{\pi}}\sqrt{t} + \frac{1}{2}\left(\frac{\theta_a D}{h^2} - \frac{k_L}{V}\right)t \qquad [15]$$

After the soil surface is covered by the chamber even for a short time period, gas concentrations in the chamber increase non-linearly with time. In chamber applications, the quantity $(C-C_a)h/t$ often is used to calculate the gas emission rate q_0. Our equation shows that this is a true estimate of the q_0 only at infinitely short time periods. Using $(C-C_a)h/t$ generally will produce results that underestimate q_0. The extent of the error is a function of the time, size of the chamber, gas diffusivity in the soil, as well as the air-filled porosity of the soil. For example, Nay *et al.* (1994) found in a controlled laboratory experiment that using chambers and a 78 second dynamic measurement consistently underestimated the true q_0 by 15%. In many field applications, the measurement period is much longer and the error associated with using the linear equation for estimating gas flux will increase.

For the quantities in our equation, soil air-filled porosity can be estimated from bulk density and volumetric water content. The gas diffusion coefficient in the soil "D" also can be calculated from soil porosity and air-filled porosity using Millington's equation:

$$D = D_0 \frac{\theta_a^{3.333}}{f^2} \qquad [16]$$

where D_0 is diffusivity in air, typically in the range of 0.1 to 0.2 cm^2/s.

The last remaining parameter is the leak coefficient "k_L". It is a function of the chamber diameter, the depth of insertion of the chamber into the soil, and soil gas diffusivity. An approximate expression is:

$$k_L = \frac{4Dr^2}{1.395d + 0.862r} \qquad [17]$$

2.2 Tunable Diode Laser (TDL) Technology

The Tunable Diode Laser system, operating in the near infrared range, is the best method for making continuous measurements of many gases. The measurements are interference free and can be made with high sensitivity in a fraction of a second (Schiff *et al.* 1994). A TDL system can provide instant measurements of field concentrations of NH_3 over the soil surface in real time. This information can be used, in combination with information on boundary layer resistance calculated from climate and soil characteristics or measured independently, to produce real time measurements of field-averaged gas emission rates.

2.3 Manure Application

Cattle manure was transported from the Highlander Feedlot to the Vegreville research site. The research site is described in Appendix A. To avoid inter-plot interference for emission measurements, cattle manure was applied to only two plots: the plots received 15 ton ha^{-1} and 90 ton ha^{-1} on October 6, 1999. This allowed us to measure air emissions for nine days until October 15, 1999 when the rest of the treatment plots received cattle manure. Hog manure was transported from a hog operation in St. Vincent. The manure, containing approximately 6% solids, was applied on October 22, 1999 at rates of 11, 22, 44, and 66 ton ha^{-1}. The total nitrogen content (based on dry weight) was 1.4% and 4.5% in cattle manure and hog manure respectively.

2.4 Measurements

2.4.1 The Chamber Method

Chamber Construction
Chambers were constructed using 5.8 L plastic buckets with a height of 21cm and a diameter of 20 cm at the top and 17.5 cm at the bottom. The buckets were wrapped with fiberglass insulation and covered with aluminum foil to protect the fiberglass. In the case of dynamic measurements, a pressure release device (air lock) from a home wine making kit was installed in the chamber. Teflon tubing (3 mm in diameter) was inserted through the pressure release port for air sampling. The chamber system is shown in Figure 2.

Sampling for Dynamic Measurements
The chambers were pushed into the soil to a depth of approximately 4.5 cm. At 6-minute intervals, air samples were drawn through the Teflon tubing using a 10 ml airtight syringe (Hamilton Co.). Five samples were taken from each chamber for a total sampling time of 30 minutes. One air sample also was collected before the chamber was closed to obtain a measurement at time zero. The air samples were transferred to 7 ml vacuum containers (Fisher Scientific) and transported to the laboratory. Samples were stored at 4 ºC in the dark until analysis.

Sampling for Static Absorption Measurements
Petri dishes (7 cm in diameter, 1 cm high) containing 25 ml of 0.02 N H_2SO_4 solution were placed on top of a plastic sponge on the soil surface. The sponge allowed gases to diffuse from the soil to the chamber. The chamber was pushed into the soil to a depth of approximately 4.5 cm. After 12 or 24 hours the solution in the Petri dishes was transferred to plastic vials. The vials were transported to the laboratory and stored at 4 °C until analysis. Three chambers (replicates) were used for each treatment.

Nitrous Oxide and Ammonia Analysis Using Gas Chromatography

A gas chromatograph, Model 8610C (SRI Instruments), equipped with a plot cap column (30 cm x 0.53 mm internal diameter) maintained at 40 °C and an electron capture detector (ECD) maintained at 100 mV and 350 °C were used to measure N_2O concentrations in the air samples. Five percent methane in argon (ECD grade - BOC) was used as make-up gas supplied at a pressure of 60 psi and helium (UHP grade - BOC) was used as carrier gas supplied at a pressure of 10 psi. 0.5 ml samples of air were injected on-column using a gas-tight syringe (Hamilton Co.). Detector output was monitored using PeakSimple® for Windows software. Nitrous oxide peaks were integrated manually, peak areas were measured, and N_2O concentrations determined by comparing the areas to those obtained from N_2O calibration gases (Praxair) verified by RSLS 2000 laboratory.

The same GC was equipped with a Haysep-T column and an independent HID detector to measure ammonia; however, the sensitivity of the detector was too low to allow for accurate determination of ammonia in the collected air samples.

Ammonia Analysis in 0.2 N H_2SO_4

The concentration of ammonia in samples from the closed chambers was determined using a Technicon autoanalyzer as described by McKeague 1978.

Static Absorption Measurement

The rate of NH_3 emissions was calculated according to the following equation:

$$\text{Flux } (\mu\text{g m}^{-2}\text{h}^{-1}) = \text{C } (\mu\text{g ml}^{-1}) \text{ x } 25(\text{ml}) / \text{duration (hours)} \qquad [18]$$

where C is the concentration of NH_3 in the samples.

2.4.2 Using TDL System to Measure Ammonia

TDL System

The TDL consists of an integrated transmitter/receiver unit and a remote, passive retroflector array. The operator targets a remote retroflector array by adjusting the TDL laser beam emission direction by means of a two-axis mounting unit. A telescopic sight and an on-board, visible aiming laser assist in pinpointing the direction. The ruby red visible laser is seen as a bright reflection when the laser is focused on the retroflector. The laser light emitted from the transceiver is reflected back from the retroflector and focused onto a photodiode detector with a portion passing through a calibrated reference cell containing ammonia. The two optical signals are compared by a microprocessor and the resulting ammonia concentration calculated for the optical path. The microprocessor comparison of the emission spectra and calibration sequence are unique to Boreal Laser Inc.'s system and add confidence that the absorption measured is due to ambient ammonia. The computed gas concentration is then displayed on the back panel of the instrument in ppmm (parts per million metres) and transmitted to a data logger (Model CR10X, Campbell Scientific Inc.) where the data are collected and stored. The TDL specifications are listed in Appendix B. A field set up is shown in Figure 3.

Meteorological Tower

An on-site, three-metre tripod meteorological tower was used to characterize the atmospheric parameters (Figure 3). The meteorological tower was located on a control plot throughout the study. Wind speed and direction were monitored with a R.M. Young instrument (Campbell Scientific Inc.) at three metres above ground level. Five Met-one wind speed sensors, capable of measuring calm-condition wind velocities (Campbell Scientific Inc.), were used to characterize the vertical wind velocity distribution. The 5 Met-one sensors were located 40 cm, 65 cm, 115 cm, 215 cm, and 315 cm above ground level. Four fine wire thermisters were used to characterize the vertical temperature profile. The thermisters were located 25 cm, 50 cm, 100 cm, and 315 cm above ground level. A Met 107 temperature sensor also was used for a reference temperature. Details of the meteorological equipment are contained in Appendix C. The meteorological parameters were data logged at 5-second intervals and averaged to 10-minute averages by a CR10 data logger with a SDM – SW8A port extender (Campbell Scientific Inc.).

Data Collection

The meteorology tower was set up on September 30, 1999 (Figure 3) at the Vegreville field site. The TDL was set up on a north-south path on the west side (path-length 12 m). The TDL read high background ambient NH_3 values during the day on October 3rd and 4th (Figure 4). These values were attributed to emissions from the plot areas, but could have come from other activities, such as unloading and piling of manure to the east of the experimental area.

A dynamic ammonia sample was collected using a sampling pump to draw ambient air through two impingers in series. The NH_3 was absorbed into 0.05 N H_2SO_4. The impinger samples were collected at the mid point of the TDL path on the west side of plot 7 (plot locations are shown in Appendix A) and at the same height as the TDL path. The impinger samples were collected from October 6 to October 22, 1999 in parallel with the TDL measurements. This allowed estimates of NH_3 emissions when the concentration of NH_3 in the air was lower than the detection limit of the TDL system.

2.5 Soil Temperature Measurement

HOBO temperature sensors (Onset Computer Corporation) were installed beside the sampling chambers at the soil surface and at a soil depth of 5 cm (Figures 5 and 6).

3.0 RESULTS AND DISCUSSION

3.1 N_2O and NH_3 Emissions

After manure was applied to the soil, N_2O emissions from both the 15 ton ha^{-1} and the 90 ton ha^{-1} treatments increased immediately (Table 1). For the 90 ton ha^{-1} treatment, N_2O emissions reached 1280 µg m^{-2} h^{-1} two hours after manure application. The high emission rate may result from rapid nitrification of NH_4^+ or denitrification of NO_3 already present in manure. After the initial peak, the N_2O emissions declined for both treatments. On the day following manure application, the N_2O emissions from the treatment plots were slightly higher than those from control plots. The N_2O emission rates measured in this study are comparable to the emission rates of approximately 1000 µg m^{-2} h^{-1} reported by Chadwick *et al*. (2000).

Table 1 N_2O Emissions Determined Using a Dynamic Chamber Method for Various Cattle Manure Treatments. Each value represents a single measurement.

Time after Manure Application (hours)	**N_2O Emission Rate** (µg m^{-2} h^{-1})		
	Control	Treatment	
		15 ton/ha	90 ton/ha
0.5	0.0	0.0	144.0
1.5	0.0	935.9	1246.8
2.0	0.0	1064.1	148.4
2.5	0.0	12.9	774.1
3.5	0.0	323.6	166.2
5.0	0.0	0.0	219.4
23.0	0.0	184.8	452.9
23.5	89.9	0.0	85.6
24.0	0.0	0.0	0.0
25.0	189.0	54.3	0.0
25.5	167.6	0.0	9.2
26.0	0.0	0.0	0.0
26.5	129.1	0.0	57.7
27.0	0.0	259.2	253.7
28.5	0.0	0.0	219.0
29.0	372.5	493.0	0.0
42.5	411.3	0.0	0.0
43.0	414.5	0.0	40.7

Ammonia emissions also increased with increasing application rate for both cattle and hog manure (Tables 2 and 3). Following manure application, the NH_3 emission rate from the plot receiving 90 ton ha^{-1} manure ranged from 600 to 1400 µg m^{-2} h^{-1} over 8 days. It fluctuated with air temperature (Figures 5 and 6). One day after manure application, ammonia emission rates were significantly higher from plots to which hog manure was applied, up to 8000 μg m^{-2} h^{-1}, than from plots receiving cattle manure. However, emissions decreased rapidly over time in association with declining soil and air temperature (Figure 6). Ammonia emission from soil surfaces after manure application is controlled mainly by the gas diffusion process, which is temperature dependent. As soil and air temperature increases, the rate of gas diffusion from soil surface increases.

Table 2 **NH_3 Emissions Determined using the Static Chamber Method for Various Cattle Manure Treatments.** Each value is the mean of three replicates ± SE.

Time After Manure Application (hours)	**NH_3 Emission Rate** (μg m^{-2} h^{-1})									
	Treatment									
	Control		**15 ton/ha**		**30 ton/ha**		**60 ton/ha**		**90ton/ha**	
24	5.7	(±2.7)	3.8	(±0.6)	N/A		N/A		4.6	(± 1.7)
48	24.7	(±6.4)	116.0	(±34.8)	N/A		N/A		728.0	(±135.3)
72	20.7	(±4.5)	178.2	(±59.3)	N/A		N/A		1194.1	(±255.3)
96	1.1	(±0.7)	86.6	(±25.3)	N/A		N/A		629.2	(±181.1)
264	2.7	(±1.0)	32.3	(±3.2)	63.1	(±5.3)	1202.2	(±38.4)	1195.9	(±40.0)
360	6.8	(±1.1)	29.6	(±7.5)	65.3	(±2.4)	984.3	(±322.2)	1424.7	(±156.2)
384	0.9	(±0.2)	32.7	(±12.8)	51.2	(±10.7)	664.8	(±88.0)	1136.5	(±4.5)
432	8.2	(±0.6)	35.6	(±5.2)	65.2	(±1.7)	794.9	(±152.9)	20.0	(~)

Table 3 **NH_3 Emissions Determined Using the Static Chamber Method for Various Hog Manure Treatments.** Each value is the mean of three replicates ± SE.

Time After Manure Application (hours)	**NH_3 Emission Rate** (μg m^{-2} h^{-1})									
	Treatment									
	Control		**11 ton/ha**		**22ton/ha**		**44 ton/ha**		**66ton/ha**	
24	8.2	(±0.6)	35.6	(±5.2)	65.2	(±1.7)	794.9	(±152.9)	20.0	(~)
96	20.3	(±5.7)	1818.3	(±876.4)	1777.9	(±484.9)	6733.6	(±4075.3)	8164.8	(~)
144	7.7	(±2.8)	485.3	(±215.8)	309.9	(±79.3)	1957.7	(±1458.4)	3276.5	(±1373.4)

3.2 Ammonia Emissions Measured Using TDL

Ammonia concentrations along the TDL path length were used with the Backward-time Lagrangian stochastic model (Flesch *et al*. 1995) to determine emission rates from the source area. Ammonia emission rates (*Q)* from the three study plots are shown in Table 4. The values range from approximately 72 to 490 mg m^{-2} h^{-1}. We expected emission rates to depend on the time after manure application, temperature, or wind speed. Unlike results from the static measurement, this clearly was not the case for the plot receiving the application of 90 ton ha^{-1} of cattle manure. For this plot, there was no decrease in the emission rate over time, nor an apparent correlation between rates of NH_3 emission and either air temperature or wind speed. The plot that received 60 ton ha^{-1} of cattle manure showed a more expected pattern of emission rates with rates decreasing over time and an apparent correlation with air temperature.

The assumption that the background ammonia concentration was zero could be a significant source of error in the NH_3 emission estimates. If background concentrations were a significant fraction of the measured concentration, then the calculation would over-estimate the rates of emission by the same fraction. When the laser observations of ammonia concentration (C_L) were used to obtain background emission rates before manure application, very high emission rates were observed (Figure 10). Values of Q ranged from zero to almost 3.6 g m^{-2} h^{-1}, or 172 ton $ha^{-1}year^{-1}$, assuming approximately 200 days in a growing season. These values seem unreasonably high; however, the cause is not clear.

The rates of NH_3 emissions estimated from impinger samples and the Backward-time Lagrangian stochastic model were up to 500 times higher than the results from the static chamber measurements. According to estimates from the Backward-time Lagrangian stochastic model, approximately 14 g of NH_3 would be emitted over a 12-day period from a 1 m^2 area of the plot that received 90 ton ha^{-1} of cattle manure; however, the total nitrogen input was only 25g m^{-2} based on the nitrogen content in manure. The BLS technique requires more work before it can be used with confidence for estimating soil gas emissions from smaller plots.

Table 4 Ammonia Concentrations Measured 0.65 m Above the Soil Surface and Ammonia Emission Rates (*Q*) From Manure Plots. Q values denoted with a * are likely in error because of ammonia "contamination" from adjacent plots or from a wind direction that was not ideal for estimating Q.

Manure Loading (t/ha)	**Date**	**Observation Start Time**	**Duration** (min)	**NH_3 C_m** (mg m^{-3})	**Wind Direction** (deg)	**Air Temperature** (°C)	***Q*** (mg m^{-2} h^{-1})
B90	06-Oct-99	11:45	255	0.176	108	7	115
B90	07-Oct-99	13:30	150	0.117	273	17	76
B90	08-Oct-99	8:45	180	0.514	250	7	331
B90	08-Oct-99	11:45	255	0.108	256	12	115
B90	12-Oct-99	13:30	155	0.177	188	11	540*
B90	13-Oct-99	11:00	285	0.105	284	14	144
B90	18-Oct-99	11:00	310	0.097	241	8	72
B60	19-Oct-99	10:50	140	0.268	286	14	414
B60	19-Oct-99	13:10	190	0.303	294	17	490
B60	20-Oct-99	8:35	275	0.209	255	16	241
B60	20-Oct-99	13:15	180	1.049	293	18	2023*
B60	21-Oct-99	8:40	260	0.181	280	10	122
B60	21-Oct-99	13:00	180	0.228	230	11	137
B60	22-Oct-99	9:10	350	0.150	170	19	655*

4.0 CONCLUSIONS

We adopted and modified the chamber method for measuring NH_3 and N_2O emissions from soil. A sampling procedure was developed and tested in the field. N_2O emissions ranged from 0 to 1.28×10^3 mg m^{-2} h^{-1} after manure application. NH_3 emissions ranged from 0 to 1.4×10^4 mg m^{-2} h^{-1}. The highest manure application rate for both cattle and hog manure resulted in the highest NH_3 and N_2O emissions. The results from TDL measurements were unreasonably high. Further investigation is needed before the TDL technique can be used to estimate NH_3 emissions from the soil surface.

More work is needed to refine sampling and measuring protocols. To obtain NH_3 dynamic measurements (air samples) an analyzer with a detection limit in the ppb range is required.

5.0 RECOMMENDATIONS FOR FUTURE STUDY

Future work should focus on:

- Refining the sampling protocol for both dynamic and static measurements
  - Number of replicates.
  - Number of sampling points.
  - Effect of temperature on measurements during summer time.
- Modify a GC to measure N_2O and CH_4 simultaneously.
- Select an analyzer that can detect ppb levels of NH_3.
- Quantify N_2O and NH_3 emissions associated with manure treatments on agricultural land.

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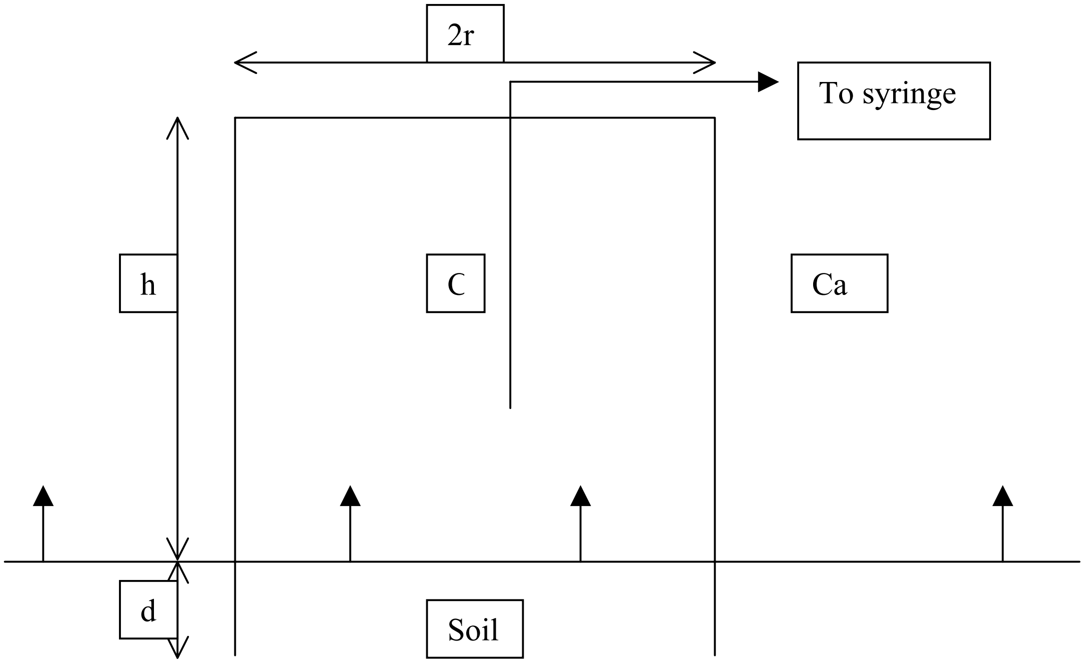


Figure 1 **Schematic of Chamber System for Gas Exchange Measurements**

Figure 2 **The Chamber was Installed in the Field**

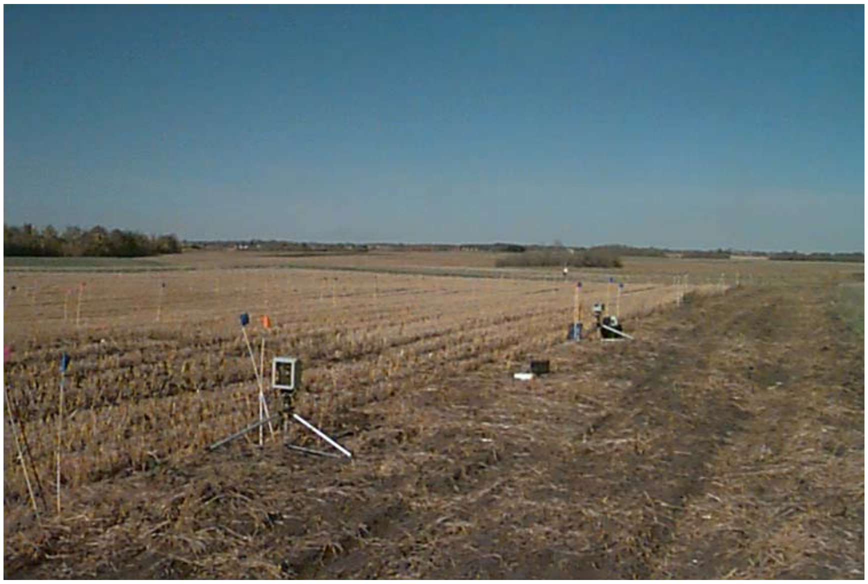
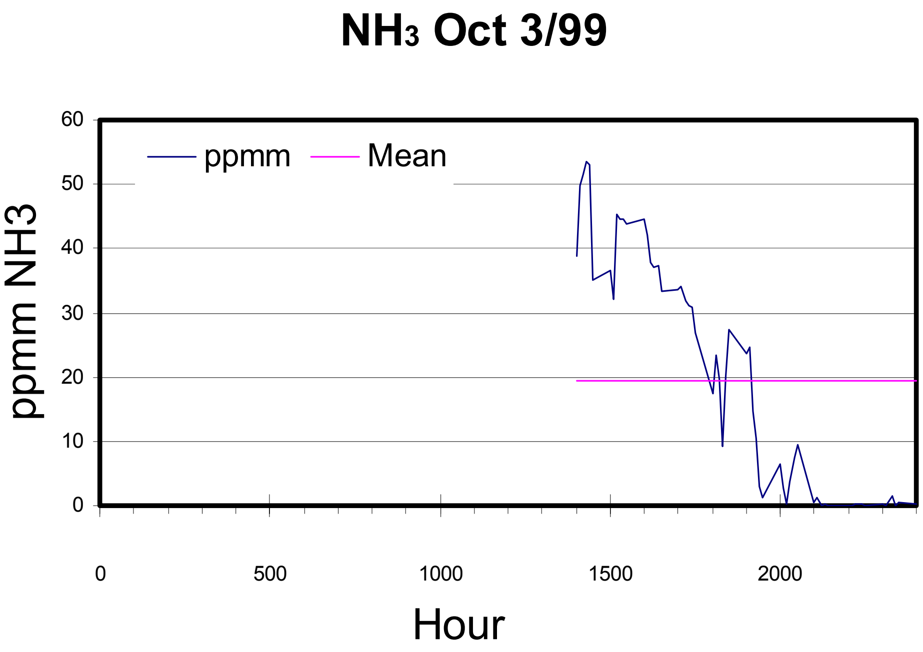


Figure 3 TDL Monitoring on the Vegreville Site (top picture) and Meteorological Tower Setup in the Control Plot on the Vegreville Site (bottom picture)



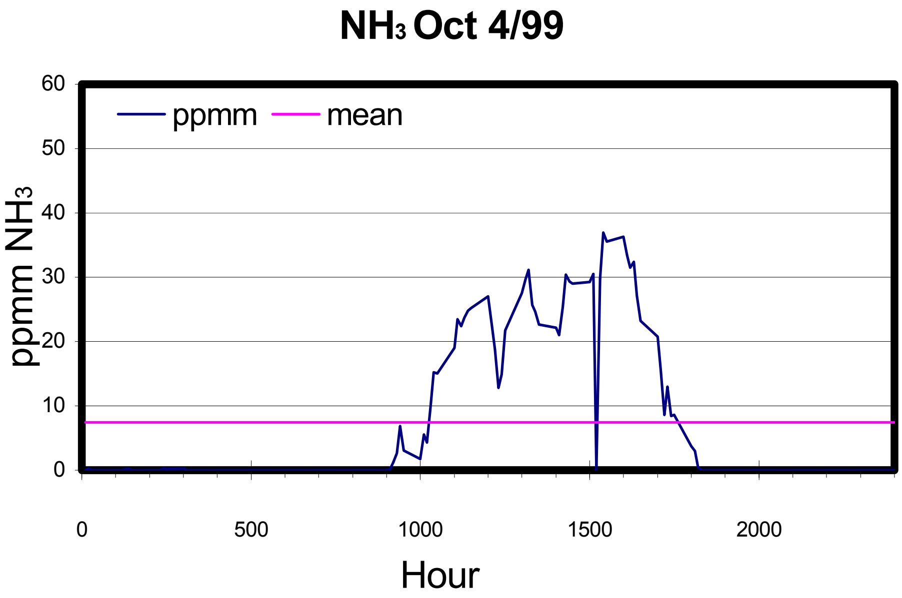


Figure 4 Background NH_3 Levels Measured Using TDL (p.m. October 3 and 4, 1999)

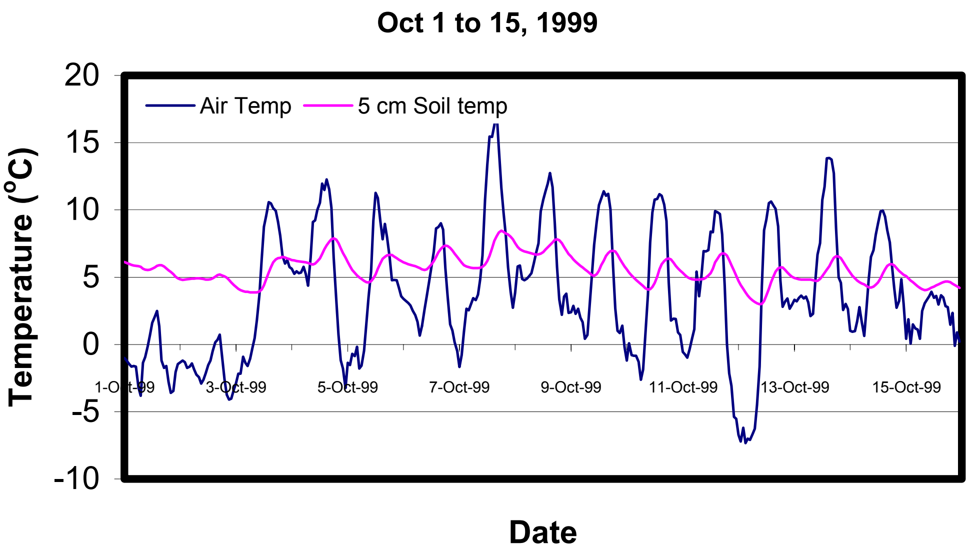

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Figure 5 **Soil and Air Temperatures Before and After Cattle Manure Application**

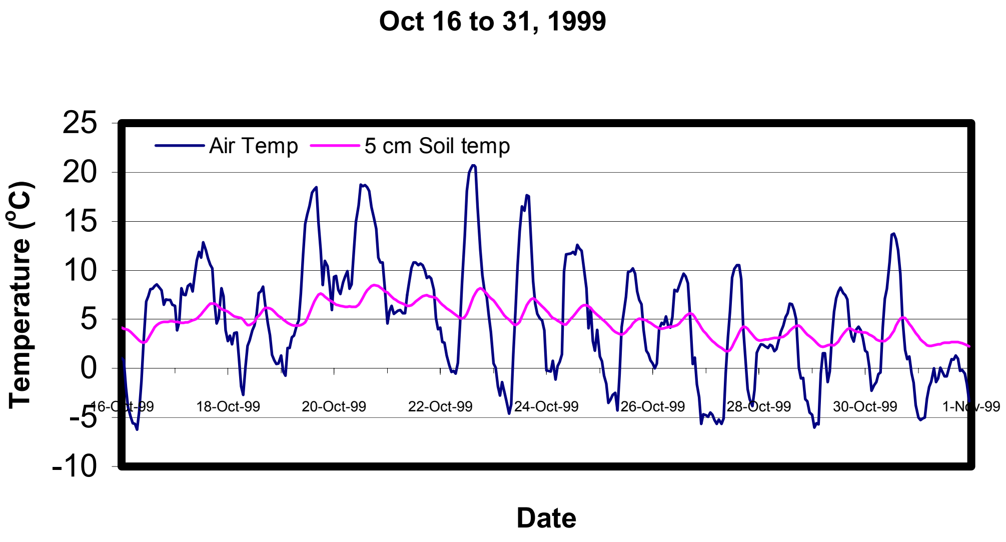
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Figure 6 **Soil and Air Temperatures Before and After Hog Manure Application**

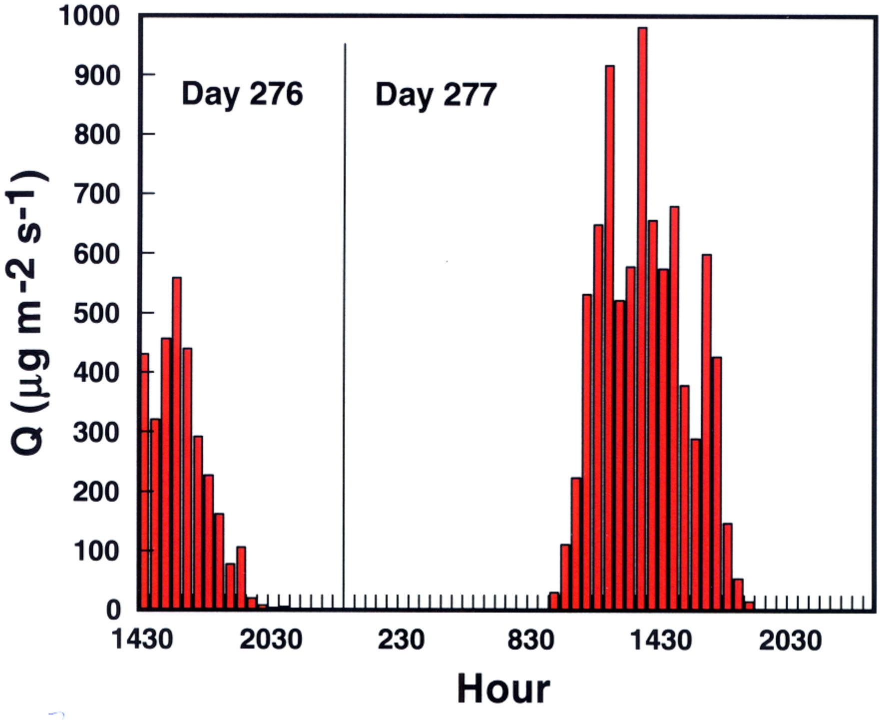


Figure 7 **Ammonia Emission Rate Estimated from the Experimental Area on October 4th (Day 276) and 5th (Day 277) before Manure Applications**. The concentrations were measured using TDL

7.0 APPENDICES

7.1 Appendix A. Description of Research Sites and Experiment Design Operated by Alberta Research Council

Site 1 ARC Vegreville Research Site

Objectives

- Determine the maximum application rates of cattle and hog manure on a Chernozemic soil
- Evaluate the impacts of manure on soil properties.
- Assess the potential impact of manure on groundwater.

Site Description

The site is located at the Alberta Research Council, Vegreville (SW13-52-15-W4). The experimental plot is on a gently undulating field of well-drained soil with a 2% slope. The entire site slopes downward from the northeast to the southwest corner.

The Vegreville site belongs to the Malmo series of an Eluviated Black Chernozem (Table 5). The soil is well drained and developed on fluvial-lacustrine material over till. The soil has an Ap of approximately 15 cm overlying an Ahe horizon approximately 5 to 10 cm thick. At the highest point of the field plot, the Ahe horizon is missing. An AB transition zone (about 10 cm thick) can be found at a lower slope, followed by a BC and Cca horizon with strong effervescence. In general, the soil profile suggests an Hobbema soil, but this classification depends on the depth to the underlying till, which is inconsistent throughout the plot.

Soil profiles are similar from the upper to the lower slope positions; however, the upper slope position is characterized by a shallower A horizon (15 cm) and a shallower depth to the Cca horizon (80 cm depth).

There was concern that soil properties would vary with soil position because of the slight slope across the site; however, no discernable trend was observed across the site in any measured soil parameter. No correlation was observed between measured soil parameters and sampling position. Therefore, only average parameters are reported.

Table 5 Soil Profile of a Lower Slope Position at Vegreville Site

Horizon	Depth (cm)	Description
Ap	0-15	Silt loam, granular structure, 7.5% organic matter, <1% coarse fragments, non-saline (E.C. = 0.6 dS/m), slightly basic (pH = 8), CEC = 27 cmol+/kg
Ahe	15-25	Silt loam, weakly prismatic, 4.9% organic matter, <1% coarse fragments, non-saline (E.C. = 0.7 dS/m), slightly basic (pH = 8), CEC = 23 cmol+/kg
Btj	25-35	Sandy clay loam, Weak boundary, <1% coarse fragments, non-saline (E.C.= 0.6 dS/m), slightly basic (pH = 8), CEC = 23 cmol+/kg
BC	35-110	Sandy clay loam, <1% coarse fragments, non-saline (E.C.= 0.6 dS/m), slightly basic (pH = 8), CEC = 19 cmol+/kg
Cca	110+	Sandy clay loam, massive structure, <1% coarse fragments, non-saline (E.C. = 0.8 dS/m), slightly basic (pH = 8), CEC = 15 cmol+/kg

Experimental design

Treatments

application rates:	4
manure types:	2
cattle manure:	15, 30, 60, 90 tons ha^{-1}
hog manure:	11, 22, 44, 66 tons ha^{-1}
control:	received no manure application

Crop

barley

Experimental layout

A randomized complete block design was used for the experiment. The experiment included a total of 9 treatments, each with 4 replicates. The experimental area covered approximately 132 m by 57 m. Each experimental plot was 12 m by 12 m. The treatments were separated by a 3 m buffer zone. The layout of experimental design is shown in Figure 8.

Site 2: St. Vincent Site

Objectives

- Determine optimal application time of both liquid (hog) and solid (cattle) manure on a Luvisol soil.
- Evaluate the impacts of manure on soil properties.
- Assess the impacts of manure application on surface runoff.

Site Description

The field site is located 0.6 km east of St. Vincent, Alberta. There are several small family-run cattle and hog operations near the site but no larger-scale feedlots. A one-acre site is located on an east-facing slope with a relatively constant 5% slope across the site. The site was cropped to barley the previous year and received intermittent manure treatments at low (5 to 10 ton ha^{-1}) rates.

The soil conditions on the St. Vincent site vary with slope position. Table 6 describes soil conditions in the upper slope position. The soil is an Orthic Gray Luvisol belonging to the Lacorey soil series. It is a typical well-drained soil developed on a moderately fine till. There is no strong evidence of an Ah and Ae horizon because of the mechanical mixing of the soil during agricultural operations. The thickness of the B horizon and depth to underlying C horizon varies with slope position. In the lower slope position, the soil profile is more characteristic of the Fergy soil series, which is an Eluviated Black Chernozem developed on parent material similar to the Lacorey soil. Of particular interest will be the higher concentration of surface organic matter and poorer drainage associated with the lower two replicates on the slope as compared with the upper replicates. Surface organic matter content changes from an average of 6.6% in the lower slope position to 3.6% in the upper slope position.

Experimental design

Treatments
- manure types
  - hog: 20 ton ha^{-1}
  - cattle: 40 ton ha^{-1}
- application times:
  - spring, fall, and winter for hog manure
  - spring and fall for cattle manure
- control:
  - received no manure application

Crop
- hay or barley (rotation during experiment)

Experimental layout

A randomized complete block design was used for the site with a total of 6 treatments, each with 4 replicates. The size of each experimental plot was 10 m by 10 m. The blocks were separated by a 5 m buffer zone. Surface runoff from the entire plot was monitored continuously for one replicate from each treatment using ISCO samplers. For other replicates, a rain barrel was placed at the base of the plot to collect all runoff from a 1 m by 10 m subplot. The rain barrels were emptied and sampled periodically for total amount of collected runoff and sub-sampled for sediment analysis. Surface runoff was analyzed for total sediment: sediments were analyzed for nitrogen and phosphorus concentrations. The layout of the experimental design is shown in Figure 9.

Table 6 Typical Soil Profile for the Upper Slope at the St. Vincent Site

Horizon	Depth (cm)	Descriptions
Ap	0-15	Loam (% sand/silt/clay = 48/37/15), medium granular structure, 3.6% organic matter, 1% coarse fragments, non-saline (E.C. = 0.5 dS/m), neutral pH (7.3 in water), cation exchange capacity = 14 cmol+/kg
Bt_1	15-45	Fine sandy loam (% sand/silt/clay = 59/21/19), weak to moderately strong medium blocky structure, 3.2% organic matter, 3% coarse fragments, non-saline (E.C. = 0.3 dS/m), neutral pH (7.5 in water), cation exchange capacity = 15 cmol+/kg
Bt_2	45-80	Fine sandy loam (% sand/silt/clay = 59/21/19), weak blocky structure, 3% coarse fragments, non-saline (E.C. = 0.3 dS/m), neutral pH (7.5 in water), cation exchange capacity = 11 cmol+/kg
BC	80-90	
Ck	90+	Fine sandy loam (% sand/silt/clay = 53/34/13), massive structure, 3.2% organic matter, 3% coarse fragments, non-saline (E.C. = 0.3 dS/m), neutral pH (7.5 in water), cation exchange capacity = 11 cmol+/kg

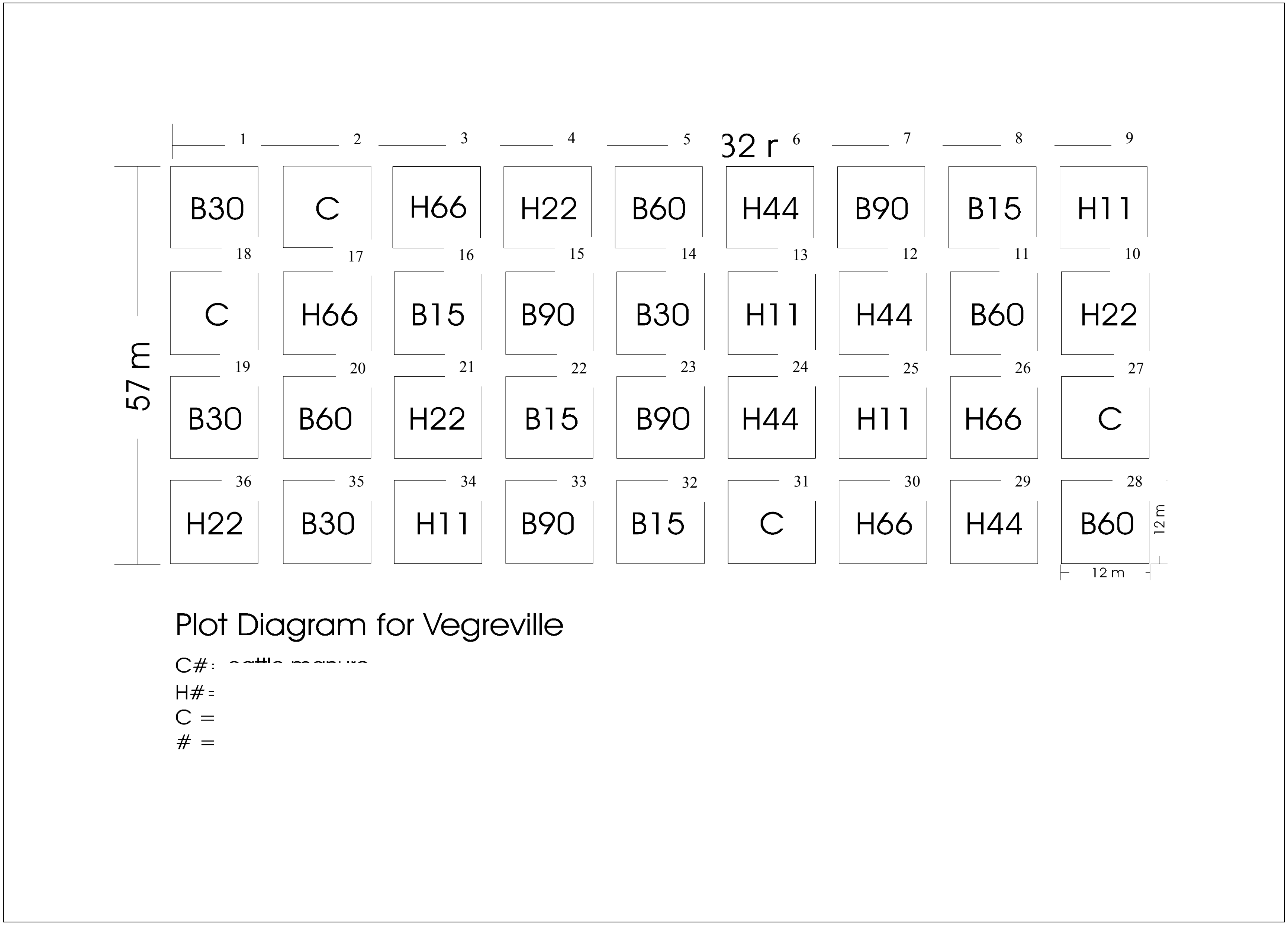
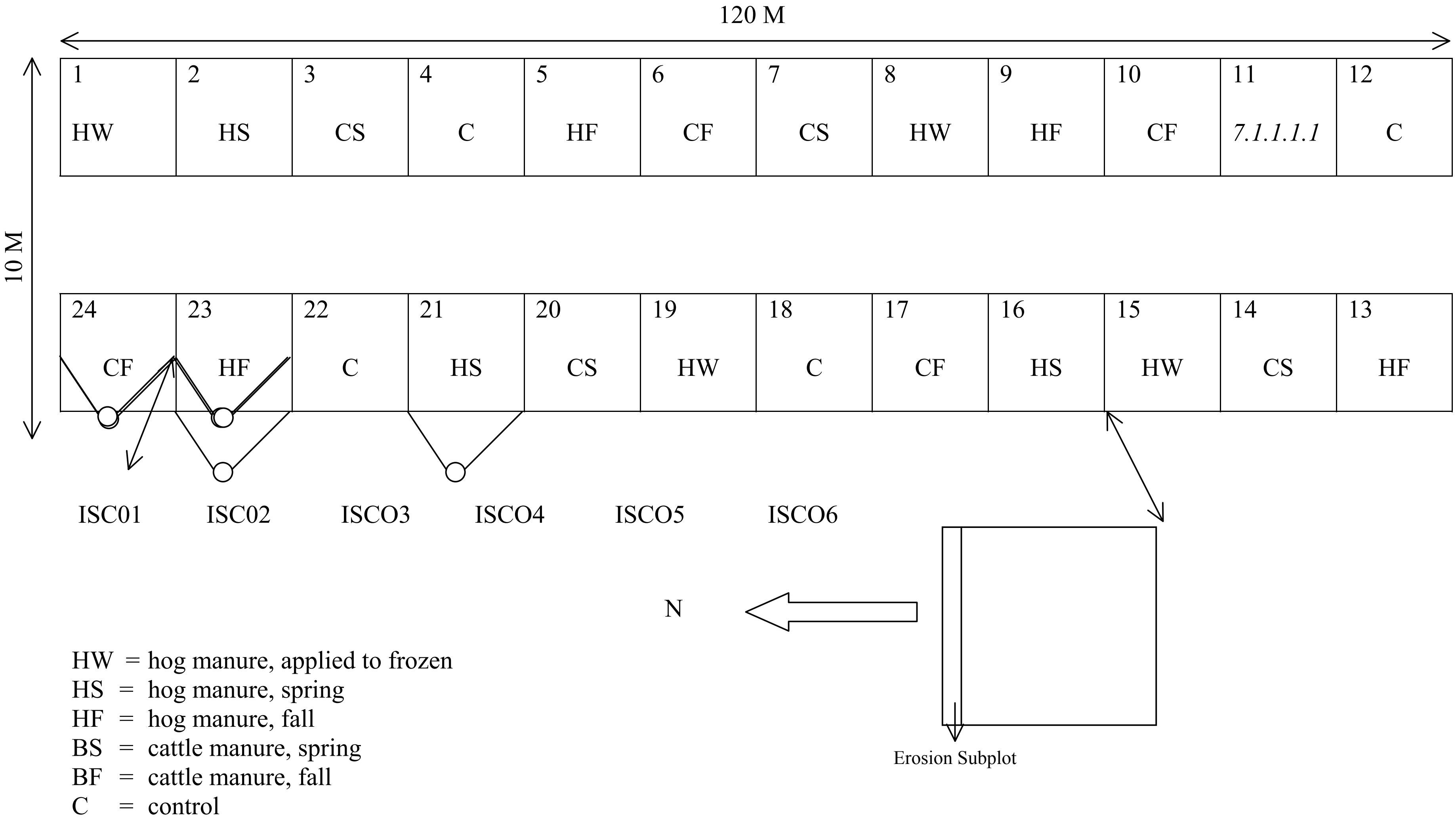


Figure 8 **Plot Diagram for the ARC Vegreville Research Site**



HW = hog manure, applied to frozen
HS = hog manure, spring
HF = hog manure, fall
BS = cattle manure, spring
BF = cattle manure, fall
C = control

Figure 9 Plot Diagram for the St. Vincent Site

7.2 Appendix B. Gas Finder (TDL) Specifications

Physical Data

Weight	5kg
Dimensions (L x W x H)	26 x 20 x 16 cm
Power Requirements	2A @ 12Vdc
Operating Temperature Range	-30’°C to +50’°C
Sighting Optics	3 – 9 x 40 scope (additional on-board visible aiming laser)
Display Options	2 x 20 character Liquid Crystal Display 2 x 20 character Vacuum Fluorescent Display
Data I/O Interface Options	RS232, 9600 baud (N-8-1) 4-20 mA Current Loop
Laser Type	semiconductor laser
Eye Safety Class	Class I or Class IIIa (as per ANSI Z136)

Performance Specifications

Sensitivity NH_3	5 ppmm (parts per million metres). This translates into 0.5 ppm NH_3 at the minimum path length of 10 m and 0.005 ppm at the maximum path length of 1000 m.
Scan Rate	1 sample/sec
Distance Range	10 – 1000 m
On Stream Efficiency (OSE)	>95% per month (target)

7.3 Appendix C. Meteorological Equipment

Quantity	Item Description	Model
5	Met-One Wind Speed Sensor	014A
1	R.M. Young Wind Monitor	05103-10
1	Keyboard display	CR10KD
2	Blue serial patch cables (for keyboard display and storage module)	
1	Themistor, 10’	107
1	Serial computer/datalogger patch cables	
6	Met-One/datalogger patch cables	
2	12-V car batteries	
2	Rubbermaid “Action Packer” storage cases for car batteries	
6	Fine wire thermocouples	
1	Metal bar (~2 m long) with 5 wires to connect fine wire TC’s	
1	Datalogger	CR10X
1	Serial computer/datalogger patch cables	
1	Power cable	
1	Datalogger	CR10
1	Switch closure module	SDM-SW8A
1	CSI 12-V battery with power leads	
1	Tripod	CM10
2	~1.5 m crossarms, ¾”	
1	~40 cm bar, threaded on one side	
7	¾” to ¾” crossover clamps	nu-rail
1	¾” to 1” crossover clamp	nu-rail
1	R.M. Young Wind Monitor mounting piece	
3	~60 cm stakes	
1	Lightning rod and grounding wire	