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The Ohio State University PH.D. 1982

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FACTORS AFFECTING AMMONIA VOLATILIZATION FROM SEWAGE SLUDGE APPLIED TO SOIL IN A LABORATORY STUDY

DISSERTATION

Presented in Partial Fulfillment of the Requirements for
the Degree Doctor of Philosophy in the Graduate
School of The Ohio State University

By

William Cullen Donovan, B.S., M.S.

* * * * *

The Ohio State University
1982

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Studies in Statistics. Professors R. V. Skavaril and P. R. Henderlong

Studies in Hydrology. Professors W. A. Pettyjohn, B. L. Bondurant, and J. H. Lehr

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INTRODUCTION

According to Tarr (1975), the use of urban wastes on farmland was wide-spread in the middle and late 19th century in the United States; and as late as 1912, the city of Baltimore, Maryland collected human wastes and sold them to be distributed to farmers. By the end of the 19th century, however, several factors were at work that changed this. One was the extensive building of sewage systems in cities, with the wastes being discharged into streams on the theory that running water cleans itself. Another factor was public health concerns about the wastes.

By the late 1960's, the public was becoming increasingly concerned about the environmental deterioration of lakes and rivers. One of the major factors contributing to this was the discharge of municipal and industrial wastes into the lakes and streams.

According to the Sixth Annual Report of the Council of Environmental Quality (1976), the 1948 Federal Water Pollution Control Act and the Refuse Act of 1899 defined the federal approach toward water pollution. These particular pieces of legislation focused on ambient water quality, with discharges permitted that did not exceed the estimated assimilative capacity of the body of water. Enforcement was slow and punishment nonexistant.

In 1972, amendments were incorporated in the Federal Water Pollution Control Act which changed the focus of the act from ambient quality and assimilative capacity to the establishment of discharge limits, requiring industrial polluters to achieve the "best practicable treatment control technology currently available" by 1977 and even more stringent "best available technology economically achievable" by 1983. Municipal sewage discharge had to have secondary treatment by 1977 and the best practicable waste treatment technology by 1983. The final goal was to eliminate the discharge of pollutants into navigable waters by 1985. Prior to 1972 federal aid to local governments for the construction of municipal wastewater treatment systems covered up to 55 percent of the construction costs and annual appropriations were running $1 billion. The 1972 amendments increased the federal share to 75%, and $18 billion was authorized for construction over a three-year period.

As a result of the 1972 amendments to the Federal Water Pollution Control Act, municipalities were faced with the disposal of ever-increasing amounts of sewage sludges. Not only did they have to dispose of the existing amounts of sludge generated, but secondary and tertiary treatment resulted in even greater quantities of sewage sludge.

Different methods of waste disposal were available, with the interest in land application of sewage sludges growing due to economic factors. According to Miller (1974), the cost of disposal of liquid sewage sludge on land within reasonable proximity to the treatment plant was much less than any other disposal method.

Sewage sludge is also a valuable source of nitrogen, phosphorus, and potassium. When sewage sludge is applied to agricultural land, it can serve as a source of these nutrients. The phosphorus and potassium are readily available to the plant. About 50%) of the nitrogen in sludge is in the organic form, of which approximately 30% is available in the year it is applied (Ohio Guide for Land Application of Sewage Sludge, 1979). The remainder of the nitrogen is present principally as ammonia which is immediately available. Some of the ammonia may be lost initially through volatilization, reducing the amount of nitrogen supplied to the crop. Since the sewage sludge is applied at application rates below the nitrogen requirements of the crop, any ammonia not lost by volatilization would be utilized. When sludge is land applied, the farmer needs to know the amount of nitrogen available from the sludge to his crops to be able to supply additional nitrogen fertilizer to meet the nitrogen requirements of that crop. Accurate estimates of the extent of ammonia volatilization under different environmental conditions are needed to be able to do this.

The objective of this study was to evaluate the relative effects of soil moisture, incorporated versus unincorporated sludges, different sludge types, vegetative cover, soil pH, and temperature on ammonia volatilization from land applied sewage sludges in a laboratory and growth chamber study.

LITERATURE REVIEW

Figure 1 is a conceptual view of what occurs when sewage sludge is applied to the soil surface before mineralization can occur. An equilibrium exists between tne NH_4^+/NH_3 forms for the ammonia (NH_3) dissolved in the sludge water, as shown by the hydrolysis equation.

The ammonia (NH_3) form can volatilize from the sludge surface as a gas and be lost to the atmosphere. Increasing the NH_3/NH_4^+ concentration in the solution, increasing the temperature, and increasing the pH of the solution mixture would all increase the vapor pressure of ammonia in solution, and thus volatilization. The energy needed for volatilization to occur could easily be supplied by heat adsorbed from the water in the sludge.

The water present in the sludge also evaporates from the sludge surface and the ammonium (NH_4^+) can also move into the soil. The NH_4^+ is dissolved in water, and as the water infiltrates from the sewage sludge into the soil, it carries the dissolved ammonium (NH_4^+) along with it.

The ammonium (NH_4^+), having a positive charge, can also be adsorbed on the surface of soil particles as a result of the negative charges of the cation exchange capacity. The ammonium is then able to come off the surface of the soil particle to balance ammonium (NH_4^+) ions lost from the soil solution; so there is an

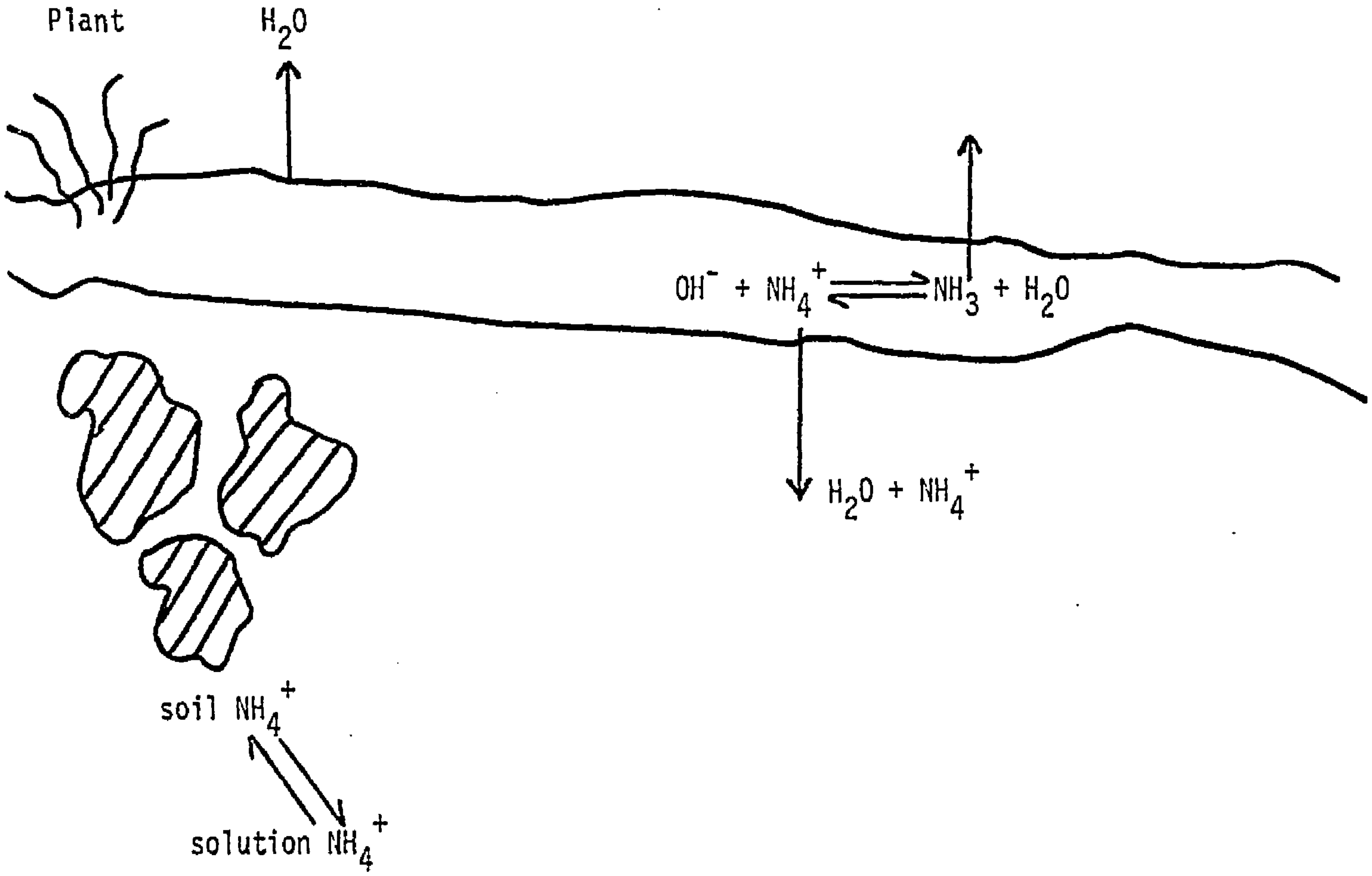

Figure 1. Representation of reactions which occur when sludge ammonia is applied to soil.

equilibrium between the ammonium in the soil solution and the ammonium on the exchange complex of the soil particles.

The physics and chemistry of ammonia volatilization from a dilute solution were recently reviewed by Denmead et al. (1982). Volatilization is an endothermic process. Normal rates of ammonia volatilization are much less than those of water evaporation (about 1/10,000) and are not restricted by the amount of available solar energy which does control the evaporation of water. The amount of energy required for ammonia volatilization could be supplied from the air, water, or soil.

Volatilization results from a difference in vapor pressure between the ammonia in solution and the ambient air. The solution vapor pressure is determined by the concentration of aqueous ammonia and the temperature. The concentration of aqueous ammonia depends on the concentration of ammoniacal nitrogen (aqueous ammonia plus ammonium) in the water, the temperature, and the pH. Increasing the ammoniacal nitrogen in solution, increasing the temperature, and increasing the pH all increase the aqueous ammonia concentration, which would in turn increase the solution vapor pressure and thus volatilization. Ambient ammonia vapor pressure increases linearly with the concentration of aqueous ammonia and exponentially with temperature (Denmead et al., 1982).

If similar mechanisms govern ammonia volatilization as those affecting the evaporation of other volatile materials, there should be some dependence on wind speed. The exchange of gases or vapor across an air-water boundary can be described by:

$$F = k\ (p_o - p_z),$$

where

F = flux density of the gas

k = exchange coefficient

p_o = equilibrium vapor pressure

p_z = vapor pressure of the substance at a height z above the surface

For the evaporation of water, k increases linearly with wind speed. For ammonia, due to its high solubility and NH_4^+/NH_3 aqueous equilibrium, the liquid phase resistance would be small and the gas phase would control the exchange, so k would have the same linear dependence on wind speed as water vapor.

Several environmental variables affect the NH_4^+/NH_3 equilibrium in solution and the NH_4^+/NH_4^+ equilibrium between the soil solution and soil particles. The following variables and the principles behind them are listed in Table 1: soil pH, soil moisture, temperature, vegetative cover, sludge incorporation, and type of sludge. The principal factor governing the NH_4^+/NH_3 equilibrium is soil pH. As the pH increases (or the hydroxyl ion concentration increases) more ammonium reacts to form ammonia and water, thus more ammonia volatilization can occur. The various factors governing soil moisture effects on ammonia volatilization are, again, the NH_4^+/NH_3 equlibrium, the movement of water and ammonium into the

Table 1. The major environmental variables which influence ammonia volatilization and the principles behind them.

	Variable	Principle
1.	pH	$NH_4^+ + OH^- \rightleftharpoons NH_3(g) + H_2O$
2.	Moisture	a) $NH_4^+ + OH^- \rightleftharpoons NH_3(g) + H_2O$
		b) Movement of H_2O into the soil
		c) Evaporation of H_2O
3.	Temperature	$NH_3(g)$ solubility
4.	Vegetation	Entrapment of sludge solids
5.	Incorporation	NH_3 adsorption
6.	Sludge Type	a) Sludge chemistry--NH_3 content, pH
		b) H_2O content

soil, and the evaporation of water. The principal effect of temperature is on the solubility of ammonia in water. The higher the temperature, the less ammonia can be dissolved in a given quantity of water. This is why the ambient ammonia vapor pressure increases linearly with the concentration of aqueous ammonia and exponentially with the temperature. There are several effects of vegetation. The principal effect is on the entrapment of sludge solids in the vegetative cover. Another effect is on the adsorption of the ammonia in the soil solution by rapidly growing plants. When sewage sludges are incorporated, the ammonia is adsorbed on the exchange complex of the soil particles. Finally, different types of sludges affect ammonia volatilizaton through their chemistry, ammonia content, pH, and water content. Each of these variables is discussed in detail in succeeding sections.

Soil Moisture

Soil moisture affects the movement, adsorption, and retention of ammonia in the soil in a number of ways. Much of the research on the effects of soil moisture on ammonia volatilization was done not with manures or sludges but with urea or chemical fertilizers containing ammonia.

Jewitt (1942), using ammonium sulfate as the ammonia source, found a close relationship between the loss of ammonia and the loss of water from the soil. The actual moisture content of the soil did not appear to have an important effect except as it approached air dry conditions. Volatilization stopped when moisture loss stopped, and the rate of fertilizer application greatly affected the rate of ammonia volatilization.

Jewitt proposed that, in alkaline soils, the soil solution contained low concentrations of ammonium ions in equilibrium with those on the base-exchange complex. The NH_3 and H_2O have their own partial pressures and would evaporate together in amounts determined by their relative molar concentrations. The NH_4^+ ion concentration would tend to remain constant due to equlibrium between NH_4^+ ion in solution and on the exchange complex. The OH^- concentration is also buffered by the soil solution. The constant equilibrium between NH_3, NH_4^+, and OH^- ions would result in the ratio of NH_3 volatilization to H_2O loss remaining approximately constant. Jewitt (1942) found that this condition was approximately correct. The ratio of ammonia volatilization to water loss declined slowly, indicating that the reserve of ammonium ions on the base-exchange complex was sufficient only to partly maintain the ammonia concentration in solution. The ammonia was volatilized, as from a dilute solution, at a constant rate proportional to the NH_3 concentration in the soil solution. No specific soil moisture level, above the air-dry condition, was found to be the most favorable for NH_3 volatilization. The base-exchange equlibrium was not influenced by the soil-water ratio and was able to explain the limited influence of the moisture concentration on the reaction.

Wahhab et al. (1956), using ammonium sulfate fertilizer, found similar results as Jewitt -- a relationship between loss of moisture and volatilization of NH_4-N which was indicated by an approximately constant ratio between their percentage losses. The loss of ammonia through volatilization and water by evaporation became constant simultaneously, showing that the volatilization of ammonia was only possible when a loss in moisture occurred. Wahab et al. further speculated that, in an alkaline soil, NH_3 could exist in the soil solution as NH_4OH, hydrated NH_3, $(NH_4)_2CO_3$ or NH_4-HCO_3. The NH_4^+ in the soil solution would be in equilibrium with NH_4^+ on the exchange complex as long as sufficient ammonium ion was present on the exchange complex. Maximum NH_3 volatilization took place at 25% moisture content, with the volatilization decreasing with increasing moisture. Little volatilization of ammonia occurred from air dried soil. Ammonia volatilization at different soil moisture levels showed similar trends over time; however the absolute amount of ammonia lost depended on the moisture content of the soil. No volatilization of NH_3 occurred when moisture loss stopped.

Martin and Chapman (1951), using many different ammonium fertilizers (dried blood, ammonium sulfate, ammonium hydroxide, ammonium nitrate, sodium nitrate and urea), found, like Jewitt, that the specific moisture content of the soil had little effect on ammonia volatilization, except that evaporation of water was necessary for volatilization of ammonia to occur. They used saturated air and dry air and drew it over the soil surface. Little ammonia volatilized from the soil when moisture saturated air was used.

Ernest and Massey (1960) and Volk (1959) found also that more ammonia was lost from urea fertilizer applied to soils when they were allowed to dry. In addition, they found that the maximum ammonia volatilization occurred at a soil moisture half-way between air-dry and field capacity. This finding is very similar to Wahhab et al. (1956) who found that the maximum volatilization of NH_3 occurred at 15 atm. (wilting point).

Both Jewitt (1942) and Martin and Chapman (1951) had found that the soil moisture content above air-dry was not important in terms of ammonia volatilization. However, a careful study of their methods showed that the soils were not brought up to determined percent moisture saturations. Jewitt added a measured amount of water and ammonium sulfate to the soil, allowed the water to totally evaporate, then added additional water. Martin and Chapman's methods were similar. Soils of a determined percent moisture saturation were never used to measure maximum volatilization.

Stanley and Smith (1956) also studied the influence of soil moisture on ammonia volatilization using injected anhydrous ammonia. They found that soil moisture did influence ammonia retention. Volatilization was low from a silt loam at 15 to 18% moisture content, regardless of depth of injection, but increased as the soil was drier or more moist. For a 7.6 cm injection depth, volatilization was higher from a soil at field capacity (23% H_2O) than from an air-dry soil (2% H_2O) due to the evaporation of water from the soil surface. As the water evaporated from the surface, soil water containing the ammonia, was pulled upward and finally reached the soil surface where the ammonia volatilized as water evaporation occurred. In dry soils there was a mass flow of the injected anhydrous ammonia from the injection site and this did not occur. Most of the volatilization occurred in the first hour for a dry soil. At 15 to 18% moisture content little volatilization occurred, and at field capacity ammonia volatilization gradually increased over time until by 36 hours the loss was nearly as great as from the dry soil.

The research linking evaporation of water from the soil and ammonia volatilization, and research on different rates of drying of soils and ammonia volatilization are interrelated. They are in fact different aspects of the same problem, the effect of water evaporation on ammonia volatilization. The maximum periods of volatilization for the different soil moistures are similar.

Laner et al. (1976) found that general evaporative conditions and precipitation appear to be the main factors affecting ammonia volatilization under field conditions. Drying in response to evaporative conditions drives the ammonia volatilization process. Laner et al. studied ammonia volatilization from dairy manure spread in the field under different seasonal conditions.

Additional research has been conducted on the effects of soil drying on ammonia volatilization.

Volk (1959) found volatilization of ammonia from urea, surface applied to dry soil, to be low, but even low levels of moisture still gave considerable ammonia volatilization. He evaluated the effect of the rate of drying on ammonia volatilization. Partial drying at moisture levels near field capacity increased ammonia volatilization for three of the four soils studied. At moisture levels near air-dry conditions, the effect of drying was the reverse, due to the soils being quickly dried below the capacity to support rapid urea hydrolysis. The rate of drying was shown to be a significant factor in the rate of ammonia volatilization from ammonium sulfate, ammonium nitrate, and ammonium hydroxide on calcareous soils (Martin and Chapman, 1951).

Hutchinson and Viets (1969) found that maximum volatilization occurred when beef cattle feedlot surfaces were undergoing rapid drying, and minimum volatilization occurred during periods of precipitation or low evaporation.

Both Jewitt (1942) and Martin and Chapman (1951) reported that the volatilization of ammonia from soil is dependent on the soil moisture loss. Ernst and Massey (1960) found some additional relationships between ammonia and moisture evaporation from the soil by studying humidity effects. The soil was initially at field capacity. Under extremely rapid drying (0% relative humidity), ammonia volatilization was minimal due to the soil becoming nearly air-dry before hydrolysis of the urea was completed, and further hydrolysis was prevented by lack of moisture. This condition would not apply to ammonia volatilization from sewage sludge, especially liquid sludges. Moisture losses were very different for the two intermediate humidity levels (85-90% and 50 -55%), but the soils lost ammonia at nearly the same rate. There was a very slight loss of moisture from soil in a volatilization flask aerated with air at 10% relative humidity, but approximately 10% of the added ammonia was volatilized. Thus, the volatilization of ammonia was not closely related to the rate of drying of the soil. It is important that some evaporation of water was found to occur with the ammonia volatilized. In other words, ammonia volatilization was not found to be totally independent of water evaporation.

Ernst and Massey (1960) also observed a close correlation between ammonia volatilization and initial soil moisture under aeration at 50 to 55% relative humidity. The moisture contents used were 37.5 (saturation), 21, 5, and 1% (air dry). Differences in ammonia volatilized from soils at different moisture contents were significant at the 1% level.

Chao and Kroontje (1964) used a clay and a fine sandy loam to study the relationship between the rates of ammonia volatilized and water evaporated. The same principle was found to operate in both soils. The rate of ammonia volatilization in water saturated and unsaturated air decreased with time. The following equation described this relationship:

$$\frac{N}{t} = at^b$$

where:

N = ammonia volatilized in ug/g

t = time, hours

a,b = constants

Plotting the log of ammonia volatilized per hour versus the log of t gave a straight line for a clay soil in a saturated and unsaturated atmosphere. This showed that the rates of ammonia volatilization in a water-saturated and in a water unsaturated atmosphere followed a similar function.

The rate of water evaporation remained constant for both soils over time with the magnitude of loss depending on the speed and humidity of air flowing across the soil surface, temperature, and soil texture. The rate of ammonia volatilization decreased with time for both a water-saturated and a water-unsaturated atmosphere. This does not agree with the data by Wahhab et al. (1956) who found a constant ratio between ammonia volatilization and moisture loss.

Ryan and Keeney (1975) studied ammonia volatilization from surface applied sewage sludge on quartz sand. They found that the total amount of NH_3 volatilized was not affected by the addition of quartz sand, but the rate of volatilization decreased. The decrease was the same regardless of amount of sand used. They increased the relative humidity of the air passing through the system and significantly reduced the rate of water loss, but observed no effect on the rate or amount of NH_3 volatilized. The decrease in the rate of ammonia volatilized was attributed to a decrease in surface area for volatilization with the addition of sand. They cited a lack of relationship between H_2O loss and NH_3 volatilization. This result is very similar to that found by Chao and Kroontje but it contrary to that found by Wahhab et al.

The cation exchange capacity of the quartz sand was not given, but is assumed to be low. It is possible that the ammonia volatilization declined as the ammonium present in the soil solution was exhausted and what remained on the exchange complex was quickly used up and unable to maintain a constant supply of ammonium in the soil solution. This would explain the results observed.

This discrepancy in the results of Wahhab et al. (1957), Ernst and Massey (1960), Chao and Kroontje (1964), and Ryan and Keeney (1975) over the constancy of the ratio between ammonia volatilization and moisture loss is an area requiring additional research.

The $NH_4^+ + OH^- \rightleftharpoons H_2O + NH_3$ equilibrium as described by Jewitt (1942) and supported by Du Plessis and Kroontje (1964) appears to be valid, at least in clay or silt loam soils. The necessity for some water evporation to also occur with ammonia volatilization is supported by the various researchers.

Rapid ammonia volatilization occurs during periods of rapid water evaporation. The rate of ammonia volatilization is also linked to the initial moisture content of the soil. In short, a link between water evporation and ammonia volatilization does exist, the question being the exact nature of the relationship.

For sewage sludges, the moisture content of the sludge would be strongly influenced by the moisture content of the soil to which it was applied and vice versa. For a very dry soil, the water from the sludge, carrying the ammonium present in it, would rapidly infiltrate and move into the soil. For a saturated soil, the moisture in the sludge would not enter the soil but would remain on the soil surface and be subject to evaporation. Soil moisture content less than saturation would allow some sludge liquid infiltration to occur, with the depth and speed of infiltration increasing or decreasing as the soil moisture content increased or decreased.

Rapid ammonia volatilization could be expected to occur from the sludge during conditions which promote rapid water evaporation, such as from a sludge applied to a saturated soil.

Incorporation

The initial research on the effect of incorporation on ammonia volatilization was performed with chemical fertilizers.

Steenbjerg (1917) found that, with the exception of calcareous soils, placement of ammonia fertilizer at a 6 cm depth completly stopped volatilization irrespective of soil type.

Jackson and Chang (1947) found that anhydrous ammonia was rapidly adsorbed by the soil under laboratory conditions, and that soil moisture and depth of application, as long as the ammonia was incorporated 5.1 cm deep, were of minor importance in retaining it. As long as the anhydrous ammonia was applied 5.1 cm deep or greater (to field moist soils), an increase in pH due to the presence of free calcium carbonate or sodium carbonate did not prevent its retention. They proposed four mechanisms of ammonia retention by soils:

(1) In acid soils: the ammonia would combine with hydrogen on the soil colloid to form ammonium colloid:

$NH_3 + Hx \rightleftharpoons NH_4^+$ (nonvolatile)

where x = soil colloid

(2) In neutral soils (pH 7), NH_3 combines with the soil colloid, which is partially saturated with calcium plus hydrogen ions, to form a calcium ammonium colloid:

$$\underset{(pH\ 7.0)}{NH_3 + CaHx} \rightleftharpoons \underset{(pH\ 8\ to\ 9)}{CaNH_4x\ (nonvolatile)}$$

where x = soil colloid

(3) Ammonia could dissolve in the soil solution.

(4) Ammonia could be adsorbed physically on the surface of soil particles.

They did not perform any experiments to verify the proposed methods of ammonia retention.

Wahhab et al. (1956) placed ammonium sulfate on the soil surface and at one, two, and three cm below the surface. They found that increasing the depth of fertilizer placement reduced ammonia volatilization significantly.

Stanley and Smith (1956) found the retention of ammonia from anhydrous ammonia was high when applied to soils of 15 to 18% moisture content or when placed at least 15.2 cm deep in dry soil. Losses were much greater from a soil at field capacity (23% moisture). When applied 7.6 cm deep, volatilization was slightly higher from a soil at field capacity (23% moisture content) than an air-dry soil (2% moisture content). Retention was the same (2% loss) at a 15.2 or a 22.9 cm depth of incorporation, except for the air-dry soil. With the air-dry soil ammonia volatilization was greater compared to the soil at 15 and 23% moisture content (5% loss at 15.2 cm and 1% loss at 22.9 cm) probably due to a mass flow outward and upward from the injection site of the anhydrous ammonia due to the pressure. Losses on soils at field capacity were speculated to occur due to the upward movement and evaporation of water. For a dry soil, most of the loss occurred during the first hour. With a soil at a 15% moisture content, little loss occurred--only one percent in 36 hours. For a soil at field capacity, there was a gradual loss that was nearly as great after 36 hours as from the dry soil. Ammonia volatilization was found to occur on some soils if the anhydrous ammonia was placed 7.6 to 10.1 cm below the surface; but incorporation greater than 15.2 cm was not recommended. Significant volatilization was still found to occur when the anhydrous ammonia was incorporated 15.2 cm deep where the soil was wet, the soil surface was warmed by the sun, and wind blew across the soil surface--all of which contributed to water evaporation.

Meyer et al. (1961) studied ammonia volatilization from surface applied nitrogen fertilizer and concluded that urea compounds must be incorporated in the soil, due to losses from ammonia volatilization, for maximum benefit from the fertilizer unless rain or irrigation will occur imediately after surface application. The soils examined were neutral to alkaline. Ammonia volatilization from ammonium sulfate and ammonium nitrate was found not to compare to the loss from urea, but was still sufficient to recommend incorporation also.

Gasser (1964) found that volatilization of ammonia was similar from urea and ammonium sulfate applied to calcareous soils. The ammonium sulfate lost ammonia immediately when it was surface applied to calcareous soils, while the ammonia was lost rapidly from the urea only after a period of time. If rain falls before urea has been hydrolyzed, it is washed down into the soil and ammonia volatilization is reduced. Ammonia was lost from the ammonium sulfate whether or not rain occurred. More ammonia was lost from the ammonium sulfate than urea when both were surface applied. Incorporating the ammonium sulfate greatly decreased ammonia volatilizaton, while incorporating the urea had less of a result so that the amount of ammonia lost from the urea was greater. Volk (1959) and Martin and Chapman (1951) also found more total ammonia volatilization from ammonium sulfate than urea when they were surface applied to calcareous soils.

Gasser (1964) also found that ammonia volatilization decreased as the cation exchange capacity of the soils increased. Martin and Chapman (1951) and Volk (1951) found similar results. Martin and

Chapman (1951) used soils that were moistened, then allowed to dry, and moistened again, allowing large losses of ammonia to occur from soils of high cation exchange capacity under alternate wetting and drying (urea fertilizer was used). The work by Meyer et al. (1961) was conducted on irrigated land in Nebraska where alternate wetting and drying cycles could occur on neutral to alkaline soil. This could explain the greater volatilization from urea compounds. The cation exchange capacity was speculated as the property most likely to be related to the soil's ability to retain ammonium nitrogen by Ernst and Massey (1960) and Volk (1959), as well as Martin and Chapman (1951) and Gasser (1964).

King (1973) found that ammonia volatilization was significantly higher from a surface-applied sludge compared to an incorporated sludge. The nitrogen loss for the surface treatment was much greater from the sludge crust than from the soil. King recommended that, when the primary purpose of land application of sludges was disposal, then the sludge should be surface applied; but for maximum nitrogen utilization by crops, the sludge should be incorporated whenever feasible.

Hoff et al. (1981) studied ammonia volatilization from liquid swine manure that was broadcast or injected. Volatilization was considerably less for the injection method, with only 2.5% or less of the ammonia lost. They concluded that where nitrogen conservation was important for maximum fertilizer value, manure should be injected to prevent large ammonia losses through volatilization.

The conclusions of Hoff et al. were similar to King's and agreed with the results of Jackson and Chang (1947) who studied ammonia volatilization from injected anhydrous ammonia.

From the literature, the behavior of sewage sludge and manure when it is incorporated or not incorporated would appear to be similar to chemical fertilizers. When the sewage sludge is incorporated, there is an entrapment effect of the soil on the ammonia present in the sludge water. The ammonia in the sludge water is adsorbed on the surface of soil colloids. This would reduce ammonia volatilization.

Soil pH and Cation Exchange Complex

Any discussion of the principles of ammonia volatilization from soils will involve pH and the cation exchange complex.

A number of authors found that ammonia volatilization was related to pH and calcium carbonate content of soil, with volatilization greatly increased from an alkaline soil.

Steenbjerg (1917) found that ammonia losses from soils fertilized with ammonium sulfate ranged from 5% at pH 6 to 60% at pH 8 in four weeks. Both the pH and calcium carbonate content of the soil were found to be important in determining the extent of ammonia volatilization.

Martin and Chapman (1950) found that from 9 to 51% of added nitrogen in the form of ammonium hydroxide was lost from soils with pH's from 4.5 to 8.0, respectively, and from 1 to 27% was lost from ammonium sulfate applied to the same soils. The pH of the soil was important in determining the degree of loss. Below a pH of 7.2, very little ammonia was lost from either fertilizer application. More ammonia was lost from the ammonium hydroxide because ammonium hydroxide applications raised the surface pH of acid soils to the alkaline range, enabling more volatilization to occur.

Mitsui et al. (1954) found that volatilization of ammonia increased with increasing rates of urea fertilization, the increase being greater when the urea was surface applied or when the pH of the soil was greater than 7.0.

Ernst and Massey (1960) found that increasing the soil pH increased the volatilization of ammonia from urea. Two possible reasons were given: a greater amount of calcium saturation of the soil cation exchange complex with increasing pH, leaving less room on the exchange complex for ammonium formed by the hydrolysis of the urea; and an increased hydroxyl ion activity in the soil solution, favoring ammonia volatilization. When ammonium is present on the soil cation exchange complex, an equilibrium will exist between adsorbed ammonium and ammonium in solution. As mentioned by Jewitt (1942), an $NH_4^+ + OH^- \rightleftharpoons H_2O + NH_3(g)$ equilibrium exists in the soil solution. By increasing the soil pH by liming, the activity of both ammonium and hydroxyl ions are increased, shifting the reaction to the right and increasing ammonia volatilization.

Meyer et al. (1961) found that the amount of ammonia loss from urea, ammonium nitrate, ammonium sulfate, and a solution of urea and ammonium nitrate decreased with decreasing soil pH. Losses were small on an acid soil, severe on a neutral soil, and very severe on a calcareous soil.

Lehr and Weiemael (1961) found that the presence of carbonates was the main factor affecting ammonia loss.

Chin and Kroontje (1963) found that ammonia volatilization may be accelerated due to the presence of bases in the soil.

Ivanov (1964) found that significant losses of nitrogen from surface-applied ammonical fertilizers only occurred from carbonate-containing soils. Losses from weakly alkaline, but not carbonate-containing soils, were not important.

Loftis and Scarsbrook (1969) reported that liming increased ammonia losses from 2 to 9% for a formaldehide urea solution applied to a Norfolk sandy loam. Liming also increased ammonia losses from clay soils with high cation exchange capacity.

Miyamoto et al. (1975) studied the loss of ammonia from anhydrous ammonia in irrigation water. The partial pressure of ammonia in the atmosphere is low, so dissolved ammonia volatilizes upon exposure to the atmosphere. The anhydrous ammonia dissolves in the water and forms ammonia compounds (aqueous NH_3, NH_4OH, NH_4^+, and several ion pairs) with the ammonium hydroxide being directly responsible for volatilization loss. Adding sulfuric acid reduced ammonia loss by neutralizing hydroxyl ions and lowering pH. This reduced ammonia loss by as much as 50%. The reduction in ammonia loss by forming ammonium sulfate was small (a few percent).

Jewitt (1942) found that the rate of ammonia volatilization was greatly influenced by the rate of fertilizer application and little influenced by the moisture content except when it approached air dry levels. The conclusion was that ammonia volatilization from soil depended mainly on the soil pH and base exchange relationships with the ammonium ion.

Wahhab et al. (1957) proposed the theory that ammonia losses from slightly acid soils to which ammonium sulfate had been applied, could have the following equilibrium:

$$(NH_4)_2SO_4 \rightleftharpoons 2NH_4^+ + SO_4^{2-}$$

with:

$$NH_4^+ + OH^- \rightleftharpoons NH_3(g) + H_2O$$

The hydroxyl concentration would be dependent on the pH of the soil system.

Du Plessis and Kroontje (1964) attempted to predict ammonia losses from $NH_4^+/NH_3(g)$ equilibrium. When data was plotted as the log of ammonium-N applied vs. the log of ammonia volatilized, the slope of the line of predicted volatilization was the same as the slope of the line for experimentally determined ammonia volatilization. The measured ammonia volatilization was several times greater, however, than the predicted ammonia volatilization, the difference being due to the continuous removal of the ammonia as it was evolved, maintaining a very low partial pressure of ammonia in the atmosphere of the volatilization flask. This was found to be true for ammonia losses from acid as well as neutral soils. From the relationship between the initial pH of the soil and ammonia volatilization, and the line plot of the final pH at the termination of the experiment vs. the quantity of ammonia lost, the deduction was made that the activity of the hydroxyl ion may contribute directly to ammonia volatilization from soils. The low activity of the hydroxyl ion retards ammonia volatilization in acid soils. In an alkaline soil, an increase in hydroxyl ion concentration would shift the $NH_4^+/NH_3(g)$ equilibrium towards $NH_3(g)$.

Fenn and Kissel (1973) studied ammonia volatilization from surface applied ammonia compounds on calcareous soils. They proposed the following theory of ammonia loss: ammonia compounds aplied to the surface of a calcareous soil react with solid state $CaCO_3$ and form Ca-precipitates:

$$X(NH_4)_zY + NCaCO_3 \rightleftharpoons N(NH_4)_zCO_3 + Ca_nY_x$$

Where:

Y = ammonium anion

N, X, Z depend on valences of anion and cation

The $(NH_4)_2CO_3$ is unstable and decomposes as shown below:

$$(NH_4)_2CO_3 + \rightleftharpoons \underset{2NH_4OH}{\underset{\downarrow\uparrow}{2NH_3}} + H_2O + CO_2$$

The amount of NH_4OH formed depends on the solubility of Ca_nY_x and its rate of formation. If Ca_nY_x is insoluble, the reaction proceeds to the right and more NH_4OH is formed. If Ca_nY_x is soluble, little $(NH_4)_2CO_3$ will exist and the NH_3 volatilization which occurs will depend on the soil's pH.

Teagley and Hossner (1978) studied the mechanism by which NH_3 was lost in the reaction between $(NH_4)_2SO_4$ and $CaCO_3$ on a calcareous Norwood silt loam. They found that the reaction proceeded through an NH_4HCO_3 intermediate instead of $(NH_4)_2CO_3$ as Fenn and Kissel proposed.

Avnimelech and Laher (1977) studied the effects of acidification of the soil solution on the equilibrium process. An OH^- ion is lost for each conversion of NH_4^+ to NH_3, decreasing the pH which in turn decreases the NH_3 fraction. The rate at which this occurs depends on the initial and final concentration of ammonium and the buffer capacity of the soil.

Avnimelech and Laher proposed the following model for ammonia volatilization was proposed:

$$(1 + CB) \cdot [NH_4^+]_f + CB\,[\overline{NH_4^+}]_f = C\,[H^+]_o + CB([\overline{NH_4^+}]_o + [NH_4^+]_o)$$

Where:

$$C = \frac{Kb}{Kw} \cdot K \cdot Pa$$

Where:

Kw = dissociation constant for water (10^{-14})

$K_b = \frac{(NH_4)(OH^-)}{(NH_3)} = 1.8 \times 10^{-5}$

K = proportionality constant

P_a = partial pressure of ammonia in the air

B = buffer factor = $\frac{\Delta(H^+)}{\Delta A}$

Where:

ΔA = amount of acid added (mole/liter)

and:

$\Delta(H^+)$ = change in H^+ activity (mole/liter)

$[NH_4^+]_0$ and $[NH_4^+]_f$ = concentration of ammonium in the soil solution initially and at equilibrium

$[\overline{NH_4^+}]_0$ and $[\overline{NH_4^+}]_f$ = the concentrations for the adsorbed ammonium ions initially and at equilibrium

Avnimelech and Laher found that pH was of prime importance only when the buffer capacity (or CEC) of the soil was high, or when the concentration of the applied NH_4^+ source is low such that acidification does not change the pH for a given CEC. If the buffer capacity is very low, the acidification process decreases pH. If the buffer capacity is very high, acidification of an alkaline soil is very slow, thus large NH_3 losses will occur unless NH_4 concentration is also very high. The buffer capacity would also maintain the pH of an acid soil if an alkaline ammonia source was used.

Four major factors were found to determine the amount of NH_4^+ remaining in the soil at equilibrium: the partial pressure of ammonia in the air; the pH of the soil; the buffer capacity of the soil; and the ammoniun concentration.

The preceding theories examine the $NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O$ equilibrium in soil solutions. Avnimelech and Laher (1977) explained the general principle operating in ammonia volatilization in that they found that pH was of prime importance only under certain conditions (as given above). Other models seemed to deal with volatilization under specific conditions. Fenn and Kissel (1973) and Teagley and Hossner (1978) dealt with ammonia volatilization from calcareous soils or highly buffered soils. Under these conditions pH would be of prime importance. The relationship between pH and ammonia volatilization in soil as reported by DuPlessis and Kroontje (1964) was, again, an example of ammonia volatilization from calcareous soils or highly buffered soils. The soil samples were taken from a liming experiment on Tatum silt loam in Virginia. Wahhab et al. (1957) and Jewitt (1942) both studied alkaline soils.

The relationship between pH, cation exchange capacity, and ammonia volatilization is summarized by Avnimelech and Laher (1977). The cation exchange capacity determines the buffer capacity of the soil, and the buffer capacity determines the stability of the soil pH as ammonia volatilization proceeds. For a high CEC soil, or low concentrations of applied ammonium , the pH is of primary importance because it is stable throughout the period of volatilization. The acidification of the soil is very slow, so large ammonia losses will occur as long as soil pH is high. For a very low CEC soil, the pH would decrease, as well as volatilization, as the soil became more acid due to the $NH_4^+ + OH^- \rightleftharpoons NH_3 + H_2O$ reaction. High CEC would maintain the pH of an acid soil for an alkaline ammonia source. Thus cation exchange capacity (and buffer capacity), soil pH, and ammonia volatilization are all related and affect each other.

Temperature

Martin and Chapman (1951) studied the volatilization of ammonia from surface applied fertilizers and urea. They concluded that the volatilization was highly dependent on pH and temperature. As the temperature increased, the loss of nitrogen increased from a clay loam soil.

Volk (1959) in surface application of urea to turf or bare soil, showed that temperature was a major factor in ammonia volatilization: the colder the soil, the lower the initial loss.

Ernst and Massey (1960) found that increasing the temperature resulted in increasing ammonia volatilization. They studied volatilization of ammonia from urea. For each 8.4°C temperature increase there was a 4.7% average increase in ammonia volatilization. They did not distinguish between the effects of temperature on the rate of urea hydrolysis and the rate of ammonia volatilization from ammonium carbonate formed from the hydrolysis. The incomplete hydrolysis could partially account for less ammonia volatilization for the 7.2 and 15.6°C temperatures. The solubility of ammonia in water is influenced by temperature, with the solubility decreasing as the temperature increases, so the NH_4^+ (adsorbed)/NH_4^+ (in solution) equilibrium in soils may also be affected.

Chin and Kroontje (1963) found that, at higher soil temperatures, both urea hydrolysis and ammonia volatilization were accelerated.

Gasser (1964), Taigainides and White (1969), and Watkins et al. (1972) all found that ammonia volatilization increased with increasing temperatures. Gasser (1964) studied it from urea and ammonium sulfate applied to soil. Taigainides and White (1969) studied it in swine buildings, and Watkins et al. (1972) dealt with it from forest floor soils.

Yaalon (1964) found that spring rain collected at seven Israeli sampling stations contained four times more ammonia than rain sampled in cooler months. He felt the ammonia came from the soil, but speculated that fertilizer could contribute to its total.

Elliott et al. (1971) found that, as temperatures increased in the spring, more ammonia was volatilized from surrounding fields and the beef cattle feedlots they were studying. During cold weather, it decreased.

Fenn and Kissel (1974) found that the effect of temperature on ammonia volatilization from surface applied ammonium fertilizers depended on the type of ammonium compound and the presence of calcium carbonate in the soil. Total ammonium volatilized was modified by temperature over a wide range of ammonium application rates when a compound which formed a precipitate with calcium carbonate, such as ammonium sulfate, was applied to a calcareous soil, but the rate of ammonia volatilization was highly influenced by temperature. High temperatures increased the initial rate of ammonia volatilization, but the ammonia volatilization was reduced later compared to a more moderate temperature. The lowest temperature resulted in the lowest initial ammonia volatilization rate, but the rate became the highest during the last 76 hours of measurement.

When a non-precipitate-forming compound (with calcium carbonate) such as ammonium nitrate was applied to a calcareous soil, increasing temperature increased the volatilization of ammonia. Over a 100 hour period, 14, 18, and 25%, respectively, of the applied ammonium was lost at 12, 22, and 32°C. Both the total ammonia volatilized and the rate of ammonia volatilization were increased by increasing temperature, in contrast to the $(NH_4)_2SO_4$ where only the rate of volatilization was increased.

Ammonium sulfate applied to a non-calcareous soil buffered to the pH of the calcareous soil had ammonia volatilization equivalent to the ammonium nitrate at 12°C and lower than ammonium nitrate at 32°C. Both the total ammonia volatilized and the rate of ammonia volatilization were similar to the behavior of ammonium nitrate applied to the calcareous soil. Therefore, Fenn and Kissel speculated that the majority of the ammonia loss from precipitate-forming ammonium compounds was due to the formation of ammonium carbonate and not because of either initial soil pH or temperature.

Fenn and Kissel developed regression equations to predict ammonia volatilization from the calcareous soil. (Such equations were not developed for the non-calcareous soils). Their use is restricted to a soil similar to the soil used: Houston Black clay loam with a high calcium carbonate content. The family of curves (from which the equations were derived) did not pass through the origin; the result was that the predicted values from 0 to 6 hours were in error. From 6 through 90 hours, the rates of ammonia volatilization predicted were excellent. The relationship developed by Fenn and Kissel is demonstrated by the following equation:

$$X = -18.44 + 1.24t + 0.42T + 0.091U - 0.021t^2 + 1.68 \times 10^{-4}t^3 - 4.71 \times 10^{-7}t^4 - 8.97 \times 10^{-5}U^2$$

$R^2 = 0.98.$

Where:

U = NH_4^+-N application rate in kg/ha

t = time

T = temperature

X = percentage of applied NH_4^+-N lost as NH_3-N

As was previously stated, the intercept of -18.44 eliminates the predictive nature of the equation during the first 6 hours.

The equation has wider use than just for ammonium sulfate. The equation was expanded to include variable ammonium application rates (U) for the precipitate-forming ammonium compounds (including ammonium sulfate). The equation also shows no influence of application rate on ammonia volatilization from a non-precipitate-forming compound such as ammonium nitrate.

Hargrove et al. (1977) measured ammonia volatilized from ammonium compounds (ammonium sulfate and ammoniun nitrate) surface applied to a calcareous soil (Houston Black Clay) in the field. They found (as expected) that less ammonia was volatilized in the spring versus late summer. Consistent diurnal fluctuation in ammonia occurred in both spring and later summer. They also found that total ammonia volatilization was more sensitive to soil temperatures at all application rates under field conditions as compared to laboratory conditions. The ammonium sulfate lost considerably more ammonia than the ammonium nitrate. This agreed with laboratory studies.

In terms of sewage sludges, Beauchamp et al. (1978) studied ammonia volatilization from an anaerobically digested sewage sludge in the field. They found a diurnal flux pattern with maximum values occurring around mid-day. The flux decreased exponentially over time. The flux patterns appeared to be most closely related to air temperature, with the air temperature being most important in the first two or three days after sludge application. The initial soil moisture, soil pH, relative humidity and soil texture became increasingly important later.

Curnoe, as cited in Beauchamp et al. (1978), in a laboratory study, found that temperature was the most important experimental variable for ammonia volatilization. He found that increasing the temperature or soil pH increased ammonia volatilization. The length of his study was not reported.

Vegetation

In terms of the effect of vegetation on ammonia volatilization, a number of papers deal with the effects of rapidly growing plants on ammonia loss.

Lowenstein et al. (1957) reported that nitrogen loss, irrespective of soil treatment, was less in soil seeded with oats. Nitrogen loss in the unseeded soil was 35 to 72%, while in the cropped soil the loss was only 7 to 8%. Ammonium sulfate and alfalfa meal were used as fertilizer sources.

Kresge and Satchell (1959) studied the effect of plant cover an ammonia loss by applying different rates of urea (92 and 277 kg/ha of nitrogen) to bermuda grass. At the 92 kg/ha rate, medium and heavy amounts of plant cover significantly reduced the loss of ammonia compared to bare soil. At 277 kg/ha there were significant differences among the medium and heavy growths of plant cover. The maximum loss from 277 kg of nitrogen per hectare of urea surface applied to heavy grass cover was 7% compared to 23% from bare soil. The respective values were 1.4% and 8% at the 92 kg/ha rate. A review of the paper reveals that the urea was surface applied at the respective rates and was then watered in. The fertilizer was applied to grass stolons that were allowed to grow and root until growth covered the soil surface, so the grass stolons were still actively growing. We are looking at ammonia volatilization from a liquid fertilizer applied to rapidly growing grass. The decrease in ammonia volatilization when applied to the grass cover compared to the bare soil is understandable under these conditions.

King and Morris (1974) grew grass in soil in vertical clear plastic pipes to which different sewage sludge treatments were applied. For all cycles of the experiment, except one, they harvested the grass prior to sludge application. Where the grass was not harvested prior to sludge application, they found low values of ammonia volatilized. They speculated that the actively growing grass served as the sink for ammonia and reduced volatilization.

Mills et al. (1974) found that rapidly growing corn seedlings reduced ammonia losses by at least 60% in alkaline soils and by smaller amounts in acid and neutral soils. The rapid adsorption of ammonium by plant roots, from the ammonium chloride solution applied to the soil, appeared to account for the decrease in ammonia loss.

There are a few papers which deal with the interference of the vegetative cover on fertilizer application to soil. The vegetation prevents or interferes with fertilizer-soil contact.

Volk (1959) applied pelleted and crystal urea and ammonium nitrate to grass sod. Ammonia losses from 92 kg/ha of urea nitrogen averaged 20.6 and 29.3% for pelleted and crystal urea, respectively, while only 0.3% was lost from the ammonium nitrate. The greater losses from the crystal urea were attributed to a tendency of the crystals to cling to the grass instead of penetrating to the soil surface.

Meyer et al. (1961) found large amounts of ammonia were lost from two soils when pelleted urea and a urea-ammonium nitrate solution were surface applied to the soil or to a wheat straw residue on the soil surface. Losses of 20% of the applied nitrogen occurred under the worst conditions. Little ammonia loss occurred from the ammonium nitrate application, with the largest from applications on the wheat straw. The amount of ammonia loss decreased with decreasing soil pH, except for a fertilizer solution sprayed on a wheat straw residue. The soil under the wheat straw residue was acid, but the amount of ammonia volatilization from the fertilizer solution equaled the ammonia lost from fertilizer solutions applied to neutral and alkaline soils. The researchers felt that the majority of fertilizer solution did not penetrate the straw residue to the acid soil, but was exposed on the straw residue and subject to volatilization.

Another approach to examining the effects of vegetative cover on ammonia volatilization was taken by Coffee and Bartholomew (1964). Coffee and Bartholomew, in previous experiments, found that surface layers of mineral soils greater than a few centimeters thick are not very effective in adsorbing surface applied ammonia. Most of the adsorption takes place in the surface centimeter. Moist plant materials may adsorb and retain from 2 to 7 g NH_3-N/kg of residue. Dry plant residues did not adsorb or retain as much ammonia as moist samples. Total ammonia adsorption by the plant materials was related to their moisture contents. The vegetative cover on many sandy soils would equal the soil's ability to adsorb and retain ammonia. For soils with a greater exchange capacity than sandy soils, the soil is much more important in ammonia adsorption.

The effect of vegetative cover on ammonia volatilization from sewage sludges and manures would appear to be similar to the effect on chemical ammonia fertilizer, in that rapidly growing plants would adsorb ammonia present in the soil from the sludge. Vegetative cover such as straw and grass could possibly intercept the solids and some of the liquid fractions of sewage sludges and manures and prevent contact with the soil, increasing volatilization losses. Vegetative cover might also adsorb some of the ammonia present. The adsorption of ammonia by rapidly growing plants or vegetative cover would reduce ammonia volatilization, while the interception of the sewage sludges by the vegetative cover would increase ammonia volatilization.

Equipment Design

Different methods have been used to measure ammonia volatilization. They may be classified as closed or open systems. In a closed system, the soil, plant, and atmosphere are completely enclosed and the concentration of all chemical reactants are measured from their accumulation within the system apparatus or from differences between their initial and final concentrations. In an open system, the soil-plant components are not confined or are only partly confined, and only specific reaction products are monitored (McGarity and Rajaratnam, 1973).

The open system design has been employed principally in field experiments. This design has the least interference from microenvironmental effects within the sampling apparatus. The sampling apparatus subsequently used in this experiment was based on modified open system field designs of Kissel et al. (1977) and Hoff et al. (1981) for this reason.

The importance of ammonia volatilization from ammonia fertilizer has been well researched. Most of the earlier studies were conducted in a laboratory or greenhouse and best approximated closed systems even though only the ammonia volatilized was measured. Few studies were conducted in the field, and only one of which attempted to use an open system method. A problem with an enclosed system is the potential of creating an artificial microenvironment within the equipment used, thus altering ammonia volatilization from what would occur in the surrounding conditions.

Jewitt (1942), Kresge and Satchell (1960), Martin and Chapman (1951), Gasser (1964), and Fenn and Kissel (1973) all used closed system methods for studying ammonia volatilization from ammonia fertilizers.

Jewitt (1942) added different concentrations of ammonium sulfate to thin soil layers in wide-mouth bottles. A slow stream of air was drawn over the soil surface, then passed through dilute sulfuric acid. The ammonia lost was estimated and checked by titration. The air exchange rate inside the bottles was not known.

Kresge and Satchell (1960) performed experiments in both the laboratory and outside using 1.9-liter screw-cap jars in the lab. Soil was added to a 5.1 or 7.5 cm depth. Soil moisture was at 2/3 field capacity, and known concentrations of fertilizer were applied. Dry air was drawn across the soil surface and the ammonia collected by bubbling the air through dilute sulfuric acid (0.03N). For the field studies, 7.6-liter crocks were used. Stolons of bermudagrass were planted in the soil in the crocks and allowed to grow until growth covered the soil surface, then the crocks were sealed with transparent plastic hoods and placed in sunlight. The remainder of the procedure was similar to the laboratory procedure.

Martin and Chapman (1951) used a procedure very similar to the lab procedure of Kresge and Satchell (1960) but the air was first passed through sulfuric acid to remove ambient concentrations of ammonia, and was then drawn through a large tube filled with $CaCl_2$ to remove moisture.

Gasser (1964) put the soil in a sealed 900 ml jar, passed air across the soil surface and then bubbled the air through standard sulfuric acid to trap any ammonia present. The soil layer was 2.5 cm deep, and the sulfuric acid was changed three times weekly.

Fenn and Kissel (1973) used a plexiglass column filled with soil up to approximately 8 cm of the top; the 8 cm space was used for gas exchange. Air was humidified by bubbling through water before entering the head space. A commercial air compressor was used, and the air flow rate was 14 to 16 air exchanges/minute/cylinder. Volatilized ammonia was collected in 200 ml of 2% boric acid and the boric acid titrated with dilute hydrochloric acid.

Of all these studies, only Martin and Chapman (1951) used sulfuric acid to remove ambient concentrations of ammonia from the air before passing it through the system. Only Fenn and Kissel (1973) measured the air exchange rate needed for maximum volatilization and used an exchange rate above this inside the volatilization cylinders. All of the researchers used a system which was closed off from the surrounding environment throughout the experimental period.

The first few reported field studies were performed by Volk (1959) and Kresge and Satchell (1960). The equipment used, however, created artificial environmental conditions inside the area of study.

Volk (1959) used an early open system, a 20.3 by 20.3 cm glass baking dish, 5.1 cm deep, that was placed directly over the treated area for a specified time period during NH_3 volatilization measurement. A square pad of glass wool was attached to the bottom with paraffin and 18 ml of 10% sulfuric acid was added to the square pad. In field tests, the dishes were shielded from sun and rain by a tin cover placed across the dish. No air exchange could occur between the outside air and the air inside the glass baking dishes when they were inverted over the treatment area. The treatment area under the inverted baking dishes were also cut off from other environmental influences by the tin cover placed across the dish.

McGarity and Rajaratnam (1972) used an open volatilization chamber system which seriously attempted to reduce the environmental effects caused by the equipment. They used a cover which could be placed directly over the soil surface. The base, which was driven into the soil, contained a rectangular metal frame with an upper surface carrying a rubber gasket and a cover which was clamped and sealed to the rubber gasket by another metal frame. A windshield and a collecting system of sulfuric acid traps completed the equipment design and were connected to the cover. Air was drawn through the system at 20 l/hr. The major disadvantage with the system was condensation of water on the inside surface of the cover. A heating element was used to eliminate the condensation. During the summer, temperatures inside the cover were higher, but painting the windshield with whitewash partially eliminated the problem. Since ammonia is very soluble in water, losses of ammonia could be underestimated from any condensate dripping back to the soil. Since ammonia volatilization is also very sensitive to temperature, the temperature problem in the summer would also affect the results.

Field approaches other than volatilization containers or chambers have been used to measure the amount of ammonia volatilized.

Denmead et al. (1974) used a micrometeorological technique to measure the ammonia flux from a pasture. The method depended on the accurate determination of air temperature, vapor pressure, and atmospheric pressure.

An aerodynamic approach was used by Beauchamp et al. (1978) to measure ammonia volatilization from newly-applied anaerobically digested sewage sludge in the field. The ammonia fluxes at 10, 50, 100, and 150 cm heights above the study area were measured. This approach was based on an aerodynamic diffusion model.

Neither approach induced any environmental effects in the treatment area, but required accurate measurements of several variables and a fairly large experimental area. The Beauchamp et al. (1978) method, for example, required an area of 0.4 hectares per treatment.

Another method used was measuring ammonia volatilization indirectly through the nitrogen uptake from various nitrogen sources.

Terman and Hunt (1964) used the yields and uptake of nitrogen by corn forage grown in pots in which various nitrogen fertilizers were mixed with the soil or surface applied to moist soils a week prior to planting. Meyer et al. (1967), as part of their study of ammonia losses from fertilized Nebraska soils, used oats grown in plastic containers in both laboratory and greenhouse studies and corn grown in field studies to measure ammonia losses from different ammonia fertilizers.

Again, these methods do not induce artificial environmental variables beyond those which would occur in the lab or greenhouse normally. Such methods are not suitable for short-term ammonia volatilization studies due to the time period required to grow and harvest the crop.

Hargrove et al. (1977) studied ammonia volatilization from ammonium sulfate and ammonium nitrate applied to calcareous soils in the field by using small steel cylinders forced into the soil as volatilization cylinders. A plexiglass top was used to close the cylinder and an air flow rate of 20 air exchanges/minute/cylinder flowed across the soil surface. The air was then bubbled through 175 ml of a 2% boric acid solution to trap the ammonia. The sampling period was 10 minutes every two hours for the first few hours after fertilization and less frequently later. The plexiglass top was removed between sampling periods. The advantage of the system was that the volatilization cylinders were closed off from the surrounding environment for only short periods of time.

Kissel et al. (1977) subsequently improved upon the methods used by Hargrove et al. (1977) by designing and testing an automated system that could measure ammonia volatilization under field conditions without creating an artificial environment. The basic system consisted of a volatilization chamber which was a steel cylinder with a plexiglass lid. The plexiglass lid contained 5 intake ports and one exhaust port and had rubber foam attached to the edges to form a seal. The lid was attached to the steel cylinder by a hinge, and a reversible electric motor opened and closed the lid. A vacuum pump pulled air through the volatilization chamber and into a chemical trap, where it was bubbled through 175 ml of a 2% boric acid solution to trap the ammonia. The air then flowed through the vacuum pump and was exhausted. A system was used to collect individual samples from eight volatilization chambers simultaneously. The sampling period was 12 minutes every 3 hours. And an air flow rate in excess of 19 exchange volumes/minute/cylinder was used.

Hoff et al. (1981) modified the Kissel et al. (1977) system to include a chemical scrubber, with 200 ml of 0.5N sulfuric acid to remove ambient concentrations of ammonia. They also used a manifold system composed of 2 cm PVC water line pipe to enable them to sample 6 volatilization cylinders at one time.

In summary, early methods were ruled out because of their susceptibility to creating an artificial microenvironment within the apparatus. Micrometeorological and aerodynamic approaches were ruled out because of the large experimental area required. Measuring ammonia volatilization indirectly through nitrogen uptake by plants presented a problem in the length of time required to grow the plants. The sampling apparatus subsequently used in this experiment was based upon modified designs of Kissel et al. (1977) and Hoff et al. (1981).

Air Flow

Several researchers have studied the effect of wind speed or air flow on ammonia volatilization.

McGarity and Rajaratnam (1972) describe a closed system to be used for measuring ammonia volatilization in the field. Air could be drawn through the volatilization chamber at a rate of about 20 liters/hour, but they did not attempt to link this to the amount of air needed to be drawn through the system for maximum volatilization. They used a clear plexiglass cover measuring 16.25 x 25 x 62.5 cm.

Fenn and Kissel (1973), used a 30.5 cm long plexiglas column with an inner diameter of 9.8 cm as a volatilization cylinder. An air flow rate of 2 to 3 liters/minute/cylinder was found to be sufficient for maximum ammonia volatilization, but a safety factor two to three times this amount was used. The air flow rate selected was thus 8 to 9 liters/minute/cylinder or 14 to 18 air exchanges/cylinder/minute. This corresponds to a wind velocity of approximately 0.25 km/hour.

Denmead et al. (1974) used a micro-meteorological technique to measure the ammonia flux from pasture grazed by sheep. The wind speed under which their measurements were taken varied from 0.9-3 meters/sec. at 1 meter above the ground.

Hargrove et al. (1977) developed an apparatus to directly measure ammonia volatilization from the soil surface. The area for ammonia volatilization consisted of a metal cylinder 12 mm above the soil surface, 23.5 cm in diameter, sealed with a plexiglass chamber 2 cm tall by 22.4 cm outside diameter. An air flow rate of 20 air exchanges/minute (approximately 20 liters/minute) was selected based on the work by Fenn and Kissel (1973).

Kissel et al. (1977) developed a field sampler for ammonia volatilization. They found that ammonia volatilization increased rapidly as air flow was increased up to about 14 air exchanges/minute. The ammonia loss increased only 1% when the air flow was increased from 14 to 19 exchange volumes/minute. They chose an air flow rate in excess of 19 exchange volumes/minute which corresponds to a wind velocity of 0.25 km/hour. They also performed a series of wind velocity measurements in the field with a hot wire anemometer and found that the wind velocity five cm above the soil surface at the top of bermuda grass stubble was half the wind velocity at two meters above the soil surface. The wind velocity at two meters was 2.1 km/hour. Using the assumption that wind velocity at five cm would be exactly half the wind speed at the two meter height, the wind velocity at five cm above the soil surface in the field would have been greater than 0.25 km/hour (or 20 air exchanges/minute) at least 98.5% of the time.

Beauchamp et al. (1978) used an aerodynamic method to measure ammonia nitrogen volatilization from land-applied sewage sludge. Small anemometers were set up on a mast at heights of 10, 50, 100, and 150 cm above the soil surface according to a diffusion model. The diffusion model was used to predict the wind speed and ammonia concentration product as a function of height. The predicted wind speeds at the different heights were not given.

MATERIALS AND METHODS

Ammonia Collection System

The ammonia collection system used for this series of experiments was based upon equipment designed by Kissel et al. (1977) as modified by Hoff et al. (1981). The system consisted of four parts: (1) a chemical scrubber to remove ambient traces of ammonia; (2) a volatilization cylinder, enclosing the sludge-treated soil, to permit sampling of air above the soil surface; (3) a chemical trap in which the ammonia volatilized from the sludge was trapped; and (4) a vacuum pump that pulled air through the system. In addition, the system also contained two manifolds to conduct air from the chemical scrubber to the volatilization cylinder and from the chemical trap to the pump itself.

Figure 2 illustrates the air flow path through a single volatilization chamber of the system. Outside air was pulled into and through the chemical scrubber where ambient ammonia was removed. The air then flowed through a 70-cm lengh of tygon tubing and into a manifold system. The air then entered the volatilization chamber through five intake ports equally spaced along one-half of the circumference of the cylinder and exited at an outlet across from the inlets. From the cylinder the air then flowed through tygon tubing into the chemical trap, out of the chemical trap, and on into

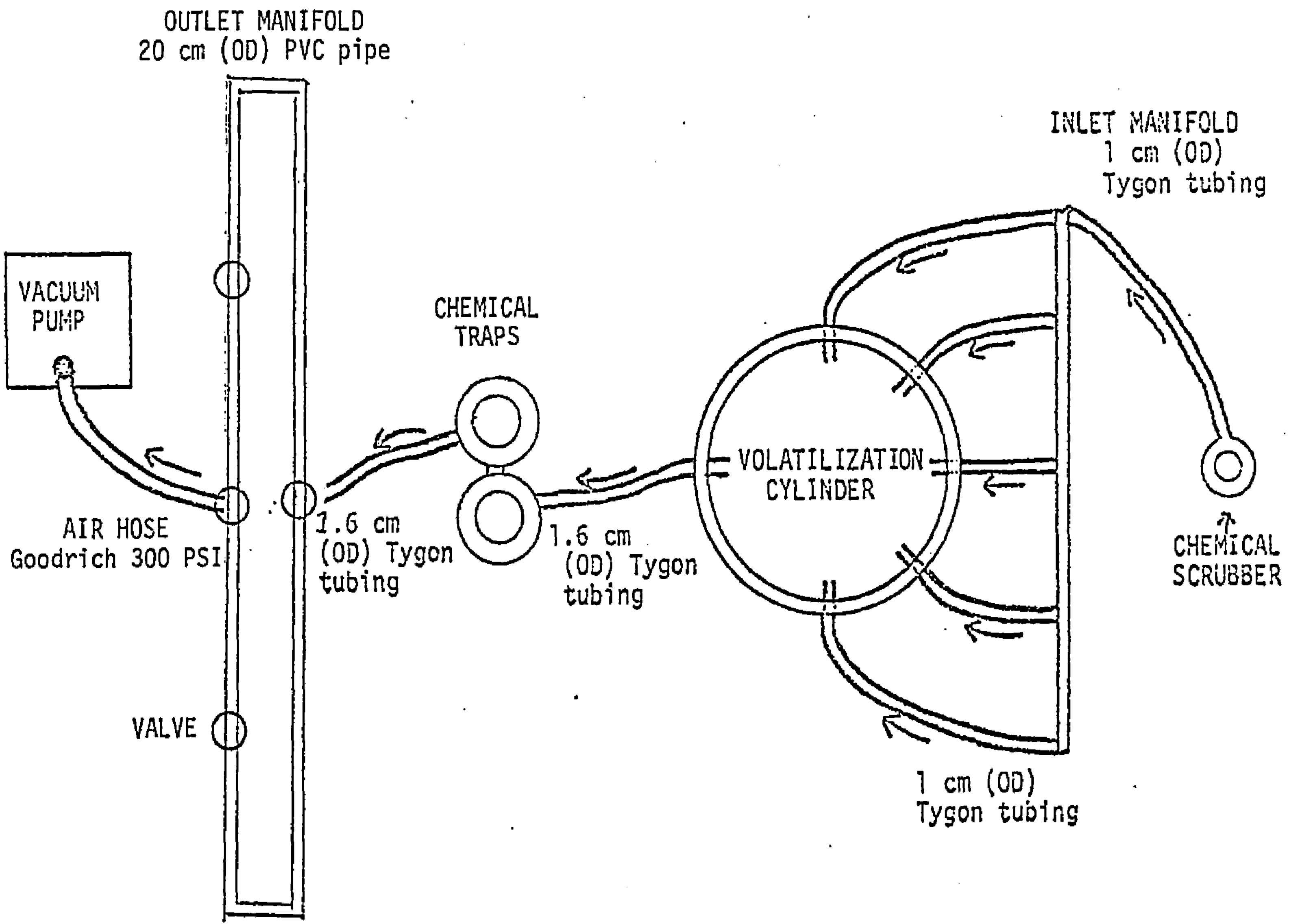

Figure 2. Vertical view of the volatilization apparatus.

another manifold system consisting of 20-cm (OD) PVC water pipe. From the manifold system the air exited to an oil trap and then on to the pump through Goodrich 300 PSI high vacuum tubing.

At the beginning of each sampling period, a pyrex glass lid was pressed down on top of the PVC cylinder to the rim of which Dow Corning high vacuum grease had been applied. The vacuum pump was started and the system checked for leaks. Timing began when the boric acid in the chemical trap began bubbling. The air was pulled through the collection cylinder for 20 minutes, then the pump was turned off and the pyrex glass lid removed. Three collection cylinders were sampled simultaneously. The collection periods chosen were initially and at 1, 3, 6, 12, and 24 hours. Sampling started 15 minutes after sludge application. A total of six collection cylinders were sampled each sampling period during an experiment by switching from one set of three collection cylinders to another (Figure 3 illustrates how all six volatilization cylinders were linked within the system).

A side view of the equipment set-up is shown in Figure 4, and Figure 5 shows the equipment as actually constructed.

Chemical Scrubber

The chemical scrubber consisted of a 500-ml narrow-mouth polyethylene bottle containing 300 ml of 0.5<u>N</u> H_2SO_4. A #4 rubber stopper with two 4 mm holes containing a #12 (extra coarse) air inlet dispersion tube and a 6 mm glass outlet tube was used to close

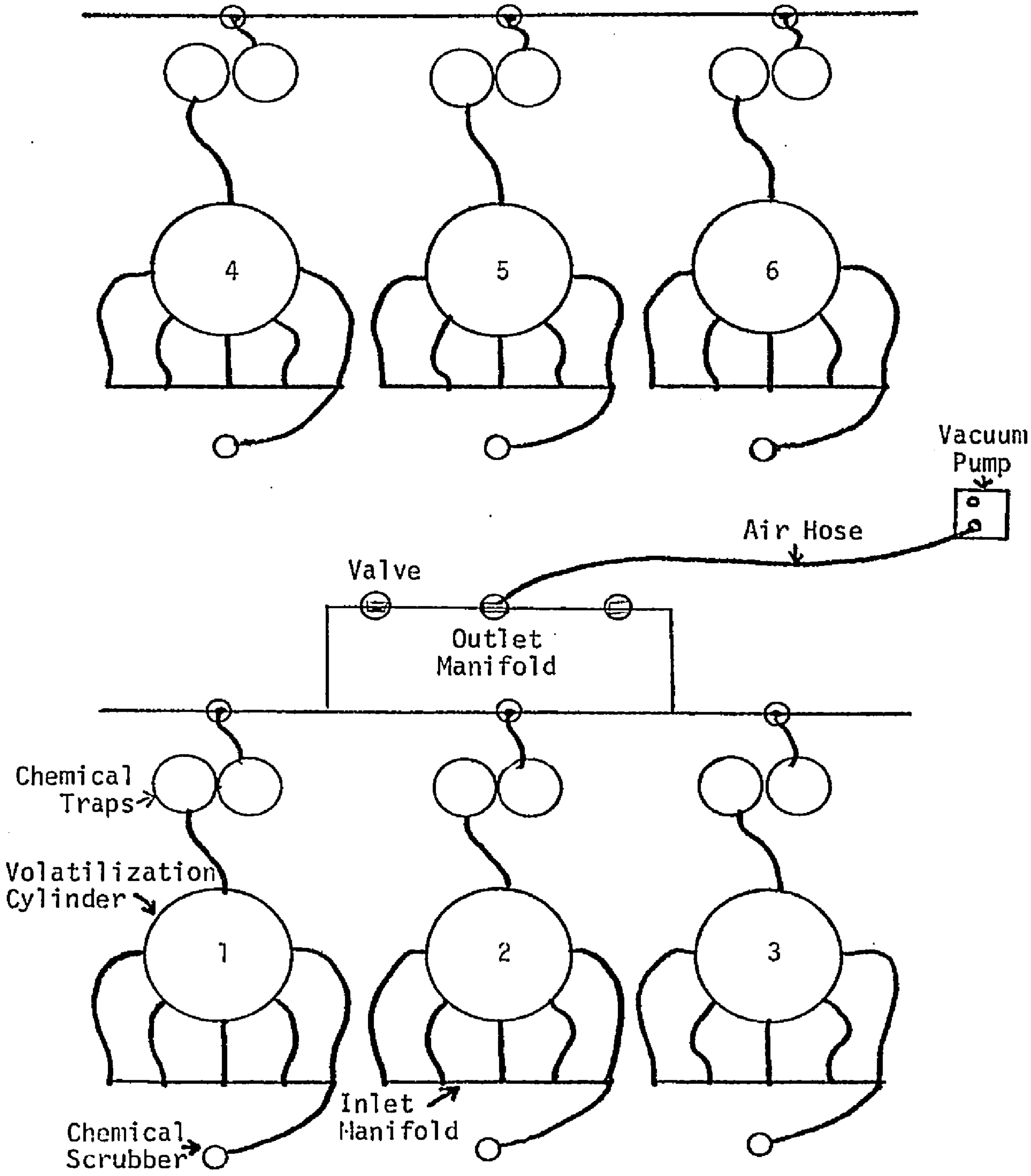

Figure 3. The manifold system for operating six volatilization cylinders.

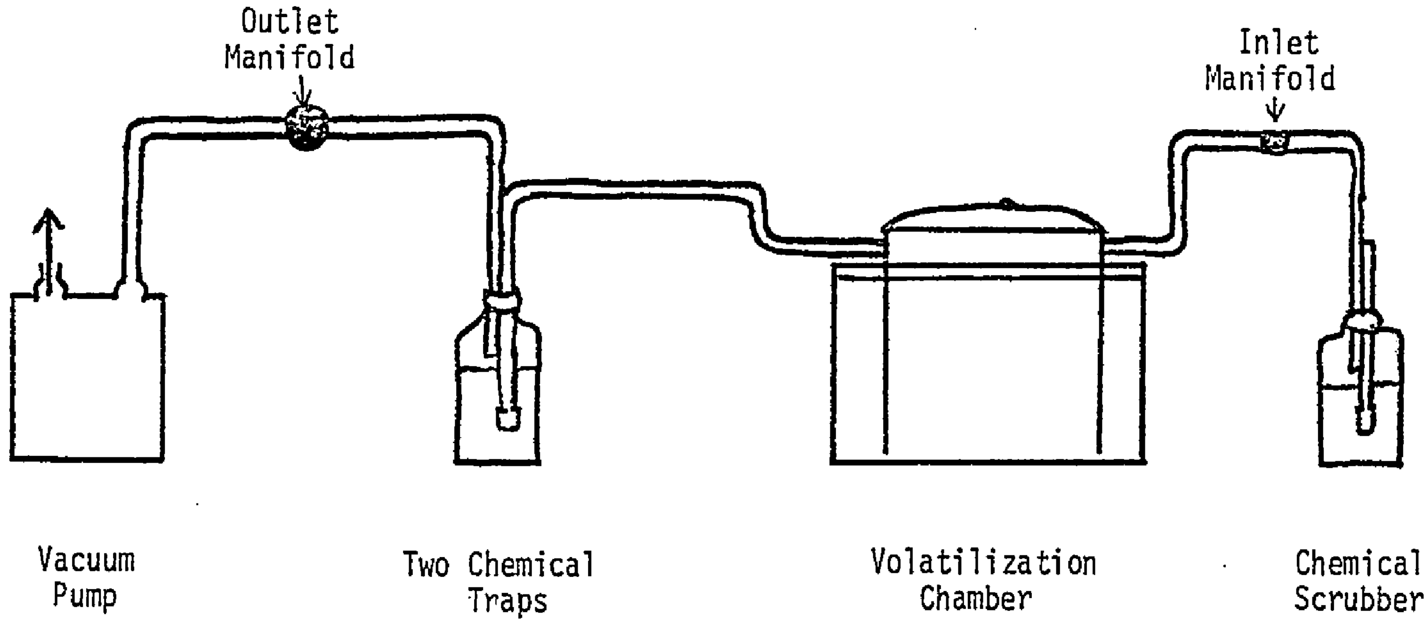

Figure 4. Side view of the volatilization apparatus.

Figure 5. The volatilization apparatus as used in the laboratory.

the bottle. The outside air entered the polyethylene bottle through the air inlet dispersion tube and exited through the 6 mm glass outlet tube.

Volatilization Chamber

The volatilization chamber consisted of a 30 cm diameter PVC cylinder cut to a length of 25 cm. The cylinder was inserted to a depth of 21 cm in the soil so that the upper 4 cm of the cylinder was above the soil surface. Five 5-cm (internal diameter) air inlet tubes were evenly spaced around one-half of the circumference of the cylinder with one 12-mm (internal diameter) outlet directly across from the inlets (Figure 2). The five inlet tubes were connected to 0.6 cm (internal diameter) 1.0 cm (outside diameter) tygon tubing through polyethylene quick connects allowing the tubes to easily snap into place. The 12 mm outlet used a copper connector to connect the tygon tubing to the cylinder.

The cylinder was placed inside a polyethelene pan measuring 47 cm by 35 cm on the sides by 18 cm deep. The soil was then placed both inside and outside the cylinder until the desired depth was reached. The volatilization cylinder was closed by a 30 cm pyrex glass lid sealed with Dow Corning high vacuum grease around the edges.

Chemical Trap

The chemical trap consisted of two 2.5-liter glass acid bottles (thoroughly washed) connected in series. Each bottle contained 800 ml of 2% boric acid. The second bottle was present to slow the air flow and to provide additional capacity should a larger flow be required. The first bottle connected to the cylinder was found to trap all of the ammonia volatilized whereas the second bottle trapped only minute traces. The bottles were closed with #4 rubber stoppers with two 4 mm holes. The air entered through a #12 (extra coarse) air inlet dispersion tube placed under the surface of the boric acid (so the air would bubble through it) and exited by a 6 mm (internal diameter) glass tube placed just inside the mouth of the bottle.

Pump

A Lebold Heraus S30 vacuum pump with a free air displacement of 760 liters/minute pumped the air through the system. The manifold system consisted of 20 cm (internal diameter) PVC water line pipe with a regular PVC valve at five inlets (Figure 2). Two extra valves were used to balance the system with individual valves for each volatilization cylinder. The total system enabled three volatilization cylinders to be sampled at one time with an air exchange of 18 volumes per minute per cylinder. An oil trap was made for the pump by filling a 20-liter polyurethane reagent bottle with crushed marble chips and placing a high pressure Goodrich 300 PSI hose from the pump inside the bottle with the outlet buried at the bottom of the marble chips and another high pressure Goodrich 300 PSI hose from the bottle to an outside vent.

Equipment Calibration

The ammonia collection system was calibrated by mixing solutions of known ammonium chloride concentration with 0.5N NaOH. Acid was added later to stop the generation of NH_3. The efficiency of the system was determined by using semi-micro Kjeldahl analysis to determine both the amount of NH_3 remaining in the solution after the addition of NaOH had stopped and the amount of NH_3 trapped in the boric acid in the chemical trap. A Gilmont #11 flow meter was used to adjust the air flow through the cylinders. Since three cylinders were sampled simultaneously, the air flow was adjusted to the same relative amount for each cylinder. Only relative air flows were used in measuring the efficiency of the system. The air flows were called relative since they were used to adjust the flow of air through the cylinders and manifold system and not to measure the air flow through each cylinder. The absolute amount of air flowing through the cylinder was determined later after a relative rate of air flow was selected that gave consistent results. A Roger Gilmont flow meter size #15 was used to measure absolute flow rate in liters/minute.

Table 2 gives the NH_3 recovered at different air flow rates for different amounts of NH_3 generated. There was no apparent effect of

Table 2. Recovery of NH_3-N at different rates of NH_3 generation and different relative air flow rates.

Relative Flow Rates (cm^3/min.)	NH_3-N Generated (ug)	NH_3-N Recovered (ug)	Percent NH_3-N Recovered
50	36,600	20,000	54.6
	16,800	10,000	59.5
	7,100	5,700	80.3
			64.8
100	64,100	35,100	54.8
	32,400	19,600	60.5
	10,000	8,200	82.0
	6,600	7,300	110.6
			77.0
150	47,000	29,200	62.1
	28,700	18,200	63.4
	12,400	13,700	110.5
	6,900	8,200	118.8
			88.7
200	50,400	31,900	63.3
	29,700	22,200	74.7
	10,500	8,200	78.0
	5,300	4,600	86.8
			75.7
250	37,600	41,500	110.4
	21,700	21,000	96.8
	11,000	11,800	107.3
	6,700	8,200	122.4
			109.2
300	53,000	24,000	45.3
	28,000	15,400	55.0
	14,700	8,900	60.5
	6,500	5,900	90.8
			62.9

air flow on percent NH_3-N recovery, but recoveries tended to be highest for the lowest NH_3-N generated (80.3-122.4%). The 50 cm^3/minute air flow was chosen for the experiments because it produced the least drying and disturbance of the soil surface during sampling, and reduced the tendency for air to be pulled through the soil pan and into the cylinder during the 20-minute sampling period.

The NH_3-N recovered during the experimental runs was less than the lowest NH_3-N generated during calibration (Table 2). The percent recovery for the lowest NH_3-N generated at the 50 cm^3/minute relative air flow selected for the experimental runs was 80.3%. Therefore, NH_3 recovery for the experimental runs should be close to or greater than the 80%. No attempt was made, however, to correct the experimental data to 100% NH_3-N recovery.

The flow rate through each cylinder was 18 air exchanges/minute during the 20-minute sampling period. This value was measured with a Roger Gilmont #15 size flow meter and coresponds to a wind velocity of 0.22 km/hour for the path length from the inlet to the outlet of the volatilization cylinder. This flow rate ensured that maximum volatilization occurred during the sampling periods according to the results of Kissel et al. (1977) and Fenn and Kissel (1973).

The air flow within my laboratory, between sampling periods, was 0.11 km/hour using the air flow path length across the room. A ventilating fan with a capacity of 514 liters/sec. and an air conditioner with a capacity of 123 liters/minute were run during the experiments. The 0.11 air flow rate was calculated by measuring the dimensions of the laboratory, calculating the volume, and dividing the result by the sum of the capacities of the fan and air conditioner. The capacity of the air conditioner was obtained by consulting a General Electric Company technical specialist and is a conservative estimate. Thus the air flow in the laboratory during non-sampling periods during the 24-hour experimental period (0.11 km/hour) was not as great as that in the cylinders during sampling periods (0.22 km/hour). Fenn and Kissel (1973), using a closed volatilization system in the laboratory, found that maximum volatilization occurred at an air flow of 0.06 to 0.07 km/hour, while Kissel et al. (1977), using an ammonia collection system in the field, found that ammonia volatilization reached a maximum around 0.18 km/hr. Therefore, the air flow rate within the laboratory during non-sampling periods was greater than the minimum air flow rate required for maximum volatilization as determined in the lab by Fenn and Kissel (1973), but was less than the flow rate for maximum volatilization measured in field studies by Kissel et al. (1977). The estimates used to determine the air flow within the room were conservative, so actual air flow was greater than the 0.11 km/hour calculated for the non-sampling period, and should not have greatly limited NH_3 volatilization, if at all.

Temperature Control

The experiments were conducted in a laboratory with sealed windows, so ventilation was provided by running a exhaust fan with a capacity of 514.4 liters/sec. Beginning with Experiment 3 (Soil pH), an air conditioner also ran during the experiments to provide ventilation and temperature control. The experiment on different temperatures (Experiment 5) was conducted in a growth chamber which was automatically and constantly ventilated.

The control temperature was difficult to maintain in the laboratory due to the laboratory's size and design. The laboratory temperature was 18°C for Experiment 1 (Soil Moisture). It was 18°C for the first run in Experiment 2 (Sludge Incorporation) but was 22, 21, 22, and 23°C when the experiment was repeated four more times. For Experiment 3 (Soil pH) the laboratory temperature was 27, 25, and 26°C, respectively, for the three replications of the experiment. Experiment 4 (Sludge Type) had a uniform temperature of 26°C for all three replications of the experiment. The fifth experiment (Temperature) was conducted in a growth chamber and subject to temperature control. The sixth experiment (Part A--Vegetative Cover, large sludge particles)--was repeated four times, and the temperatures were 26, 26, 25, and 25°C, respectively. Experiment 6 (Part B--Vegetative Cover, liquid sludge)--had temperatures of 23, 25, and 22°C for the three repetitions of the experiment.

An important consideration is whether these different temperatures affected the results of the experiments. The same range of temperatures experienced in the actual laboratory during experimentation did not exceed the range of temperatures studied in Experiment 5. By looking at Experiment 5 (Temperature), we can determine the effect of various temperature ranges used in the experiments on ammonia volatilization. The temperature range of the experiments overall was not sufficient to significantly influence the results of the experiments (see discussion of Experiment 5 in the Results and Discussion section).

Preparation of Soil and Sludge

A Crosby silt loam was moistened to the desired water content prior to being placed in the plastic pan containing the PVC cylinder. The soil was then placed both inside and outside the cylinder and firmed. Additional soil was added and firmed until the desired level of 4 cm below the rim of the volatilization cylinder was reached. The pan, cylinder, and soil were then immediately covered with plastic to prevent moisture loss until the experiment began. This method of soil preparation and soil addition to the pans was followed consistently throughout the experiments.

The soil in each pan was totally replaced between each series of experiments with new soil screened with a 2 mm mesh sieve and moistened to the desired level. The top 11 cm of soil in each pan and volatilization cylinder was replaced with new prepared soil moistened to the desired water content between each experiment of the series.

The sludge was applied by adding a carefully weighed amount of sludge to a measured amount of distilled water and mixing as completely as possible to give a 10% solids content. A partially dewatered, anaerobically digested, Columbus Jackson Pike sludge with 17.3% solids initially was used for Experiments 1-5, while a sludge from the same source with 27.7% solids was used for Experiment 6 and the part of Experiment 1 dealing with air-dried soil. The application rate was 5 dry metric tons per hectare of a sludge with a 10% solids content, with the exception being Experiment 4 (sludge type) where a 5% solids content was used and an application rate of 2.5 dry metric tons per hectare. Half of the measured amount per pan was applied to the soil surface outside the PVC cylinder and half inside, as the area inside the cylinder was 50% of the total surface area of the pan. This method of sludge preparation and application was followed consistently for all of the experiments with the exception of Experiment 4.

The sludge was prepared within 15 minutes of the start of the experiment. The plastic covering the soil was removed, and the sludge was applied to the different cylinders. The individual cylinders were closed with pyrex glass lids and the pump turned on. The sampling period was 20 minutes and began when the chemical trap started bubbling. The sampling interval was staggered: initially, 1, 3, 6, 12, and 24 hours after the application of the sludge. Because the sludge was mixed prior to the start of the sampling period, sampling started 15 minutes after sludge application. This method was followed consistently in all experiments. When the 20-minute period was completed, the pump was turned off and the chemical traps allowed to cease bubbling prior to removing the pyrex glass lids and exposing the volatilization cylinder to the atmosphere. The volatilization cylinders were left uncovered until the next sampling period.

All of the liquid sludge used in a replication of an experiment was reduced to a 10% solids content by mixing with distilled water in an 18.9-liter container, with all the sludge applied to the soil surface of the volatilization chambers and all sludge samples used for analysis taken from the mixed sludge in the container. This process was used for Experiments 3 (soil pH), 5 (temperature), and 6 (vegetative cover), but was not used for Experiment 4 (sludges) because of the different sludges used. For Experiments 1 (soil moisture) and 2 (incorporation), individually prepared sludge samples were mixed separately prior to their application to the soil in the pans and volatilization cylinders. A composite sample taken from the individual sludge batches was taken for subsequent laboratory analysis. The process of mixing all the liquid sludge used in an experiment in an 18.9 liter container, then withdrawing the sludge for application to the volatilization chambers and for sampling analysis was adopted after Experiment 2 (incorporation). A potential source of sampling variability using individual batches of sludge for each cylinder could have been variations in the ammonia content of the batches (even though all sludge was taken from the same container collected at the same time from the same treatment plant). By mixing all sludge used in an experiment with distilled water in the same container, this variable could be eliminated.

The design of the equipment used was such that only three volatilization cylinders could be sampled at one time. Goodrich 300 PSI high vacuum tubing was connected to the manifold system for one set of volatilization cylinders. The sludge was then applied to the volatilization cylinders and pans, the lids were closed, and the pump turned on. The next set of cylinders was prepared just prior to the end of the sampling period for the first set of volatilization cylinders. When the sampling period ended, the air line to the pump was quickly disconnected from the first manifold system and reconnected to the second system for the second set of cylinders and the sampling period began again. The chemical scrubbers were filled with 300 ml of fresh $0.5\underline{N}$ H_2SO_4 prior to the start of each experiment. They were filled again when needed during the experiment or if the experiment was repeated more than three times. The 800 ml of boric acid in the bottles in the chemical traps were refilled after each sampling period and the samples analyzed within one hour (see section on Laboratory Analysis).

Soil Samples

The Crosby silt loam used in this study was taken from experimental field plots near The Ohio State University Don Scott Airport. Soil for all experiments except pH was collected from a plot with a soil pH of 6.7. Soil was collected from two other plots with pHs of 5.1 and 7.5, respectively, for the pH experiment.

Samples were taken to a depth of 20 cm. Enough soil was taken originally to fill six plastic 88-liter garbage cans. The soil was dried thoroughly, mixed, and sieved with a 2 mm mesh sieve to remove debris and ensure a uniform size. The original soil collected was used through the first three experiments. Additional soil was collected from the same plot, air-dried, and screened for the last two experiments.

Sludge Samples

Samples of dewatered, anaerobically digested, sludge were collected at the Jackson Pike sewage treatment plant in Columbus, Ohio. The sludge was scraped off a conveyer belt used to transport the sludge away from the centrifuge and stored in 18.9-liter plastic containers with plastic locking lids. As discussed previously, two different batches of sludge were collected. The first batch was used in experiments 1, 2, and 3, part of Experiment 4 and Experiment 5. The sludge for the above experiments was collected immediately prior to the start of the first experiment. The second batch of sludge was collected and used for the experiment on vegetative cover and for additional sets of experiments with air-dry soil (see Experiment 1, soil moisture). For the experiment with different sludges, samples of sludges were collected at treatment plants in Medina and Ashland, Ohio, stored in plastic containers, and sent to Columbus.

The compost sample was collected in plastic bags from the composting facility at the Columbus, Ohio Southerly treatment plant. All sludge samples were stored in a walk-in-cooler at 1.1°C until used.

For the experiment on sludges, the application rate was 2.5 dry metric tons per hectare, as stated previously, with the solids content dependent on the percent solids of the sludge used. The solids content was 5% for the Jackson Pike anaerobically digested sludge, 1.57% for the Medina aerobically digested sludge, 1.96% for the Ashland lime-stabilized primary sludge, 61.2% for the Columbus compost made from primary sludge, and 17.3% for the dewatered Jackson Pike anaerobically digested sludge. The object was to apply the sludges at application rates that could be encountered in the field at solids contents indicative of the sludge.

Laboratory Analysis

The volatilized ammonia was collected in 800 ml of 2% boric acid and titrated with standardized HCl. The samples were titrated within 1 hour of their collection.

Five ml samples of prepared sludge from each experiment were analyzed for NH_3 content by using a semi-micro Kjeldahl distillation apparatus. The 5 ml samples were transferred to digestion flasks by using a 5-ml sludge pipette (a pipette modified to have a large discharge end allowing liquid sludge to be easily drawn up or removed from the pipette). The steam generator was a large flask containing heating coils submerged in distilled water. Magnesium oxide was added to the samples to raise pH and generate NH_3 from NH_4^+. Five ml sludge samples were used since previous experiments had determined that this sample size contained sufficient ammonia that could be accurately duplicated when titrated with the standardized HCl. The distillate was collected in 50 ml Erlenmeyer flasks to which 5 ml of 2% boric acid containing a mixed indicator (bromocresol green and methyl red) had been added. Distillation continued until 30 ml of sample was collected. The sample was titrated with standardized HCl. An average of eight sludge samples were run for each experiment and a mean value was calculated from the samples. The detection limit for this procedure was 120 ug NH_3-N.

Statistical Analysis

The Statistical Analysis System (SAS) package at The Ohio State University Instruction and Research Computer Center was used to write four computer programs. The first program converted the raw titration data into ug of ammonia. The replicate values were then used to plot ammonia volatilized versus time for each treatment. The computer then gave the best fit line to the data. The integrated form of the best-fit line was used in the second program to calculate the area under the line and the results were checked by graphical method. This calculation yielded NH_3 volatilized for any period up to 24 hours. A third program checked for distinctness or separateness of individual lines representing individual treatments in the experiments at the 0.05 level according to a method outlined by Neter and Wasserman (1974).

An additional method was used in which the 24-hour ammonia volatilization sums were calculated for the individual treatment replications by using the best fit regression method used previously, integrating the regression equation and calculating the area under the curve generated by the regression equation. The sums were converted to NH_3-N volatilized over 24 hours as percent of total NH_3-N applied. The percents were used in a one-way ANOVA to see if significant differences existed between the treatments. A Duncan's Multiple Range test was then used to locate the treatment source of the difference.

The 24-hour ammonia volatilization sums were also used to check for a potential predictive relationship between levels of treatments for these experiments with continuous variables (temperature, pH, soil H_2O, time of incorporation). The sums were converted to NH_3-N volatilized over 24 hours as percent of total NH_3-N applied. The percents were used as data points and a best fit method used to generate a regression equation.

Finally, all 24-hour ammonia volatilization sums (expressed as percent of total NH_3-N applied) for each replication of the control in each experiment were used in a one-way ANOVA to see if significant differences existed between the controls in the different experiments.

Experimental Design

A series of experiments were run in which all treatment variables were kept constant except the variable being studied. Experiments included: soil moisture, time of incorporation, soil pH, sludge type, temperature and vegetative cover, and the treatments are summarized in Table 3.

The experimental treatments were replicated from 3-18 times, depending on the number of treatments in an individual experiment. Since a total of six cylinders could be run at one time, the number of runs which were required to give the necessary replications varied with each experiment. In each experiment, however, all of the treatments were included in each run and were randomized as to their positions on the experimental apparatus.

Experiment 1: Soil Moisture

The effects of initial soil moisture on ammonia volatilization from surface applied sewage sludge was studied. The experiment had a total of six replications of each treatment. The soil moisture levels selected for study were those at 0, 0.1, and 15 atm. The percent moisture by weight at these tensions were 32, 22, and 10%, respectively, for the Crosby soil. All other parameters were held constant. The soil in each pan was totally replaced between each experiment with new soil moistened to the desired water content. This was the only series of experiments in which the soil was totally replaced between each run. This practice was discontinued

Table 3. A summary of the NH_3 volatilization experiments.

	Experiment	Reference Conditions[*]	Variable Conditions	Replications
1.	Soil Moisture	0.1 atm.	0, 15 atm. Air-dry (31 atm.)	6 12
2.	Time of Incorporation	Unincorporated[†]	0, 1, 3, 6, 12 hours after application	5
3.	Soil pH	6.7	5.1, 7.5	6
4.	Sludge Type	Columbus anaerobically digested, liquid	Ashland lime-stabilized primary Medina aerobically digested Columbus anaerobically digested, dewatered Columbus composted primary	3
5.	Temperature	$26.7^{o}C$[‡]	12.8, $18.3^{o}C$	6
6.	Vegetative Cover			
	a) Large sludge particles	bare	Wheat straw Kentucky bluegrass sod	8
	b) Homogenized sludge	bare	Wheat straw Kentucky bluegrass sod	6

[*] These conditions were kept constant in each experiment except for the variables (e.g., temperature) being studied.

[†] In this experiment, sludge was incorporated just before the 24-hour sampling.

[‡] In the laboratory for the other experiments, temperature was 26 ± $2^{o}C$.

after the experiment on soil moisture due to the length of time and the large quantity of soil required. Instead, the top 11 cm of soil in each pan and volatilization cylinder were replaced with prepared soil between runs. An additional experiment was performed with air-dry soil at a moisture content of 5.9%. The experiment was run twice with six cylinders of air dry soil per run for a total of twelve replications.

Experiment 2: Sludge Incorporation

Experiment 2 was designed to study the effects of time of sludge incorporation on ammonia volatilization. For this particular experiment a series of five runs were made. Each run consisted of six treatments (periods of incorporation) for a total of five replications for each treatment. There were six times of incorporation: immediately (or 15 minutes), 1, 3, 6, 12, and 24 hours after the sludge application. The sludge was incorporated immediately prior to starting the 20-minute sampling period. A three-pronged hand cultivator was used to incorporate the sludge. The length of each prong was 3.5 cm., so the sludge and soil were mixed to that depth. The 24-hour incorporation was essentially the same as the unincorporated control treatment used in the other experiments as the amount of NH_3 being volatilized by the end of the 24 hour period was quite low.

Experiment 3: Soil pH

The effect of different soil pHs on ammonia volatilization was studied by using Crosby silt loam soil samples with field pHs of 5.1 and 7.5, as well as the control with a pH of 6.7. The soil was completely replaced prior to the start of the experiment with soil of the desired pH. The top 11 cm of soil was replaced between each replication. A series of three runs were made with two replications per treatment per run for a total of six replications per treatment.

Experiment 4: Sludge Type

The fourth experiment examined the differences in ammonia volatilization from different sewage sludges. For purposes of identification, the sludges were named for the city or treatment plant from which they were taken (see Table 4) . The sludges were applied at 2.5 dry metric tons/hectare with a solids content dependent on the sludge instead of the 5 metric tons/hectare used in the other experiments. This rate was chosen because of the low solids content of some of the sludges and the resulting large amount of sludge and water that would need to be applied at the higher application rate. The ammonia content, NH_3-N applied per cylinder and sludge volume applied per cylinder are given in Table 4. For Experiment 4, a series of three runs were made, with each run consisting of five treatments (sludge type) giving a total of three replications for each treatment.

Table 4. Sludge treatment and NH_3 and solids content of the sewage sludges studied in Experiment 4.

Sludge Name	Sludge Treatment	NH_3-N Content (ug/g dry solids)	Solids Content %	Amount Applied Per Cylinder for a 2.5 Dry Ton Per Hectare Application:	
				NH_3-N (ug)	Volume (ml)
Ashland	Lime-stabilized liquid primary sludge, pH 12	11,400	2.0	204,700	900
Medina	Aerobically digested liquid sludge	7,400	1.6	133,200	1120
Columbus	Anaerobically digested liquid sludge	8,100	5.0	142,300	350
Dewatered Columbus	Anaerobically digested, centrifuged sludge	8,300	17.3	147,000	102
Columbus Compost	Composted primary sludge	900	61.2	16,500	30

Experiment 5: Temperature

For Experiment 5, a growth chamber was used to study the effect of temperature on ammonia volatilization from sludges. Three temperatures were used: 12.8, 18.3, and 26.7°C. Each run consisted of one treatment (one temperature) with six replications per treatment. The growth chamber was set at the selected temperature during the 24-hour volatilization run and then reset for the subsequent temperature treatments.

A Foxboro Recording Thermometer was used to record the temperature on a circular graph. The temperature was recorded constantly throughout the experimental period. The growth chamber was old, so some variability was present at the temperature settings. For the 12.8°C setting, the temperature initially varied from 13.0° to 14.5° and then dropped to 12.0°C. Over half of the experimental period was conducted at 12.0°C. For the 18.3°C setting, for the first third of the experimental period the temperature was 17.8°C, and for the next two thirds it was near 18.9°C. For the 26.7°C setting the temperature was near 26.9°C for the first third of the experimental period, when it then fell briefly to 24.5°C, rose briefly to 28.9°C, and then dropped to near 25.9°C for the remainder of the experimental period.

Experiment 6: Vegetative Cover

Experiment six was conducted to measure the effect of vegetative cover on ammonia volatilizaton. Wheat straw and a Kentucky blue grass sod were compared with bare soil.

For this experiment, the soil inside and outside the PVC cylinders that were to contain straw and sod was scooped out until the soil level was 6 cm below the rim of the PVC cylinders. The grass sod placed inside the cylinders raised the soil level to 4 cm from the rim of the PVC cylinders, the standard condition in the other experiments. The straw was placed inside the cylinders until the straw surface was 4 cm below the rim of the PVC cylinders. The level of bare soil was 4 cm below the rim of the PVC cylinder.

The experiment on vegetative cover was divided into two sets of experiments: one with sludge containing large sludge particles and another with well-mixed, homogenous sludge (without large sludge particles).

A second batch of Columbus Jackson Pike sludge had to be collected since the original supply (at 17.3% solids) had been depleted in previous experiments. This newly collected sludge had a solids content of 27.7% and was mixed with distilled water to give a solids content of 10%. The application rate was 5 dry metric tons/hectare.

A total of four runs were made with two replications per treatment in each run for a total of eight replications per treatment. It was observed during these runs that the second batch of sludge contained large sludge particles which were retained by the sod and straw treatments. To evaluate this effect, the sludge was carefully homogenized after dilution to 10% solids and a new series of three runs were made with two replications of each treatment per run for a total of six replications per treatment.

RESULTS AND DISCUSSION

The data collected for each experiment were analyzed and are reported in the following manner:

1. The individual amounts of ammonia (ug NH_3-N/cylinder) applied and recovered at each sampling period are reported by replication, treatment, and experiment in the Discussion and are also presented graphically.

2. A regression curve of NH_3-N volatilized (in units of percent of NH_3-N applied) versus sampling period was fitted to the data for all replicates of an experimental treatment, and the regression equations and goodness-of-fit statistics are presented in the Appendices. A statistical procedure described by Neter and Wasserman (1974) was used to determine whether the regression curves for individual treatments were significantly different from each other (termed distinct in this procedure) at the 0.05 level of significance.

3. The total NH_3-N volatilized in 24 hours as percent of the NH_3 -N applied for each treatment replication in the experiments was determined by integrating the regression equation (determined from the data points of that treatment replication) and calculating the area under the curve (of the regression equation) for the 24-hour

period. These totals were used in a one-way analysis of variance, and the Duncan's Multiple Range Test was used to determine significant differences between treatments at the 0.05 level. It should be noted here that the test for line distinctness may show that two treatments have different patterns of NH_3 volatilization over time (different regression curves), yet still yield the same amount of NH_3-N lost in the 24-hour period.

The pattern of ammonia volatilization varied over time for all experiments. There was a large amount volatilized right after the sludge had been applied (initial sampling). The peak period of volatilization usually occurred at the one-hour sampling, then the loss declined with time. By the end of the six-hour period, volatilization had declined to the same or below that obtained initially. The amount volatilized continued to decline over time to a value near zero at 24 hours. Specific patterns are included in the discussion of each experiment.

Experiment 1: Soil Moisture

NH_3-N sampled per cylinder are given in Table 5, and are shown graphically in Figure 6. The greatest ammonia loss occurred from sludge applied to the soil at 0 atm. (32% moisture). Peak ammonia loss occurred at one hour and decreased gradually throughout the experimental period, but was consistently higher throughout than the other soil moisture treatments. The 15 atm. treatment was second in terms of ammonia loss throughout the experimental period, followed

Table 5. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 1: Soil Moisture.

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
		ug NH_3-N/cylinder					
		0 atm. (32% soil moisture)					
1	235200	1500	1700	1400	1000	700	100
2	235200	1700	1800	800	800	200	200
3	230700	1300	1800	1100	900	400	200
4	230700	700	1000	800	800	800	500
5	201700	2100	3000	2600	2100	1800	900
6	201700	1800	2300	2400	1700	1400	1000
Mean	222533	1517	1933	1517	1217	833	483
		0.1 atm. (22% soil moisture)					
1	235200	2300	2200	1500	1700	900	200
2	235200	0	600	500	500	600	200
3	230700	1200	1200	1200	700	400	100
4	230700	1600	2000	1400	1100	700	400
5	201700	700	1100	1000	1100	900	700
6	201700	1400	1600	1300	1100	1000	500
Mean	222533	1200	1450	1150	1033	750	350

Table 5. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
		ug NH_3-N/cylinder					
				15 atm. (10% soil moisture)			
1	235200	1200	1700	1500	1100	900	0
2	235200	700	1100	900	700	500	100
3	230700	2500	2200	1700	1200	700	200
4	230700	1600	2100	1400	1200	700	200
5	201700	1000	1300	1100	1100	1100	1100
6	201700	1600	2100	1400	1200	600	400
Mean	222533	1433	1750	1333	1083	750	333

Table 5. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
				ug NH_3-N/cylinder			
				31 atm. (6 % soil moisture)			
1	204100	500	600	500	500	100	0
2	204100	300	600	400	400	0	0
3	204100	0	400	300	400	100	0
4	204100	400	700	700	600	400	100
5	204100	400	600	500	400	200	0
6	204100	100	300	400	400	100	0
7	203500	300	500	500	300	100	0
8	203500	400	500	600	300	0	100
9	203500	100	300	300	300	100	0
10	203500	500	800	400	500	100	0
11	203500	400	800	400	400	100	0
12	203500	300	300	100	200	0	0
Mean	222533	308	533	425	391	108	17

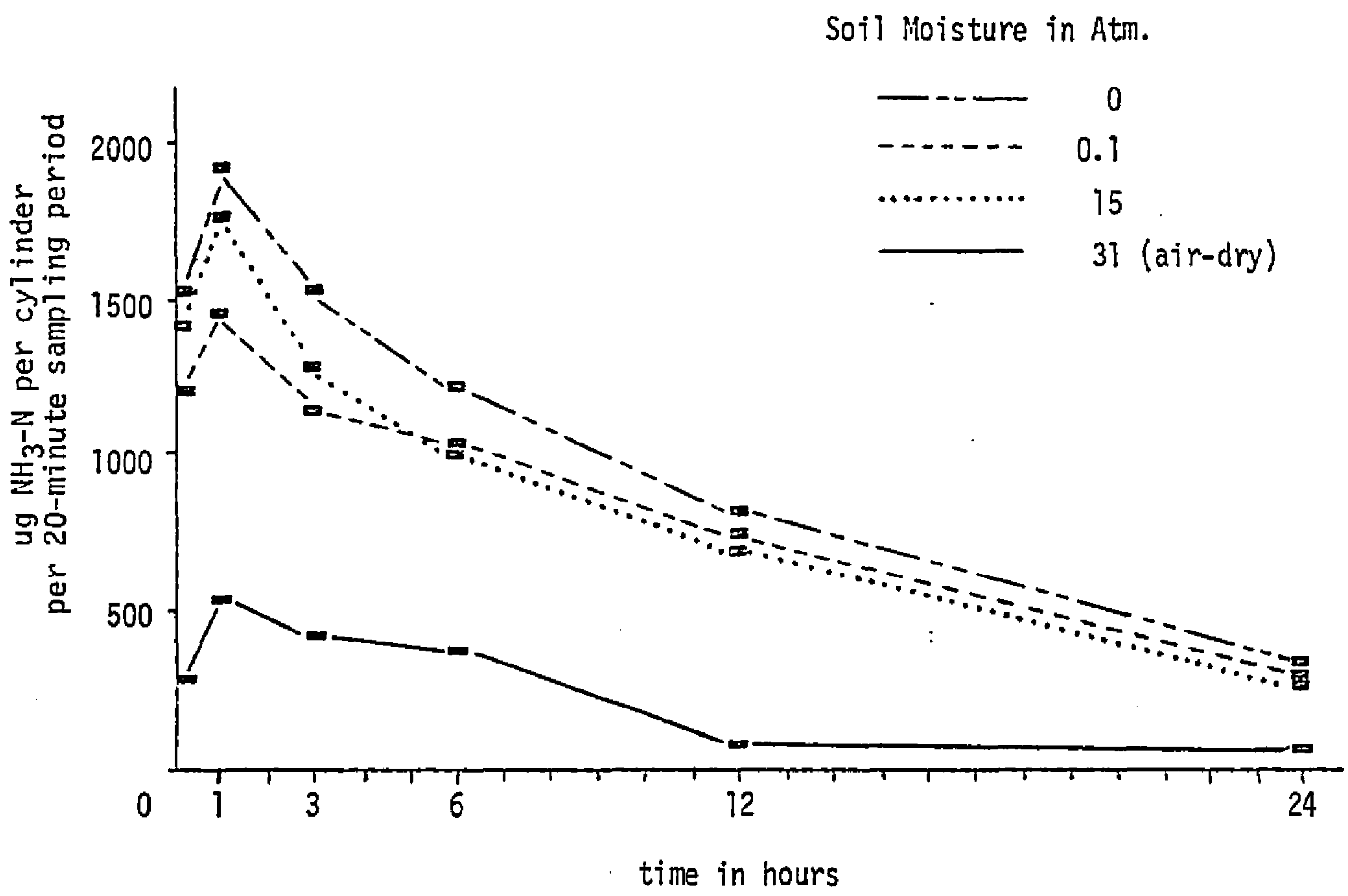

Figure 6. NH_3-N volatilized versus sampling period for sewage sludge applied to soils at 0, 0.1, and 15 atm., and air-dry initial moisture levels.

closely by the 0.1 atm. treatment. The air-dry soil gave the lowest NH_3 loss at each sampling period.

The pattern of volatilization for the air-dry treatment was statistically significant from the other three treatments, but the pattern of volatilization for the 0, 0.1 and 15 atm. treatments were not different from each other (Table 6). Significantly less NH_3 loss occurred in the 24-hour sampling period from the air-dry soil than from higher moisture contents (Table 6). NH_3 loss was 4 to 5 times lower from the air-dry soil than from the soils at higher initial moisture contents.

The additional water added to the soil with the sludge (10% solids; 90% H_2O) increased the percent soil moisture: for the air dried soil (31 atm.), the percent soil moisture increased from 6 to 8.5%; for the 15 atm. soil, from 10 to 12.8%; for the soil at 0.1 atm., the percent soil moisture increased from 22 to 24.2%; and for the saturated soil, percent soil moisture increased from 32 to 34.5% based on the assumption that the sludge liquid interacted with the entire volume of soil in the bin.

Figure 7 gives the moisture retention curve for the Crosby silt loam soil used in the experiments. The addition of liquid with the sludge lowered the moisture tensions of the soil for the different treatments as follows:

0 atm.	no change
0.1 atm.	decreased to 0.07 atm.

Table 6. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for sewage sludge applied to soils at 0, 0.1 and 15 atm., and 31 atm. (air-dry) initial moisture levels.

Treatment	NH_3-N Volatilized in 24 Hours as Percent of NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
0 atm.	31.6	a	a
0.1 atm.	25.9	a	a
15 atm.	26.8	a	a
31 atm. (air-dry)	6.4	b	b

*Means followed by the same letter are not significantly different at the 0.05 level.

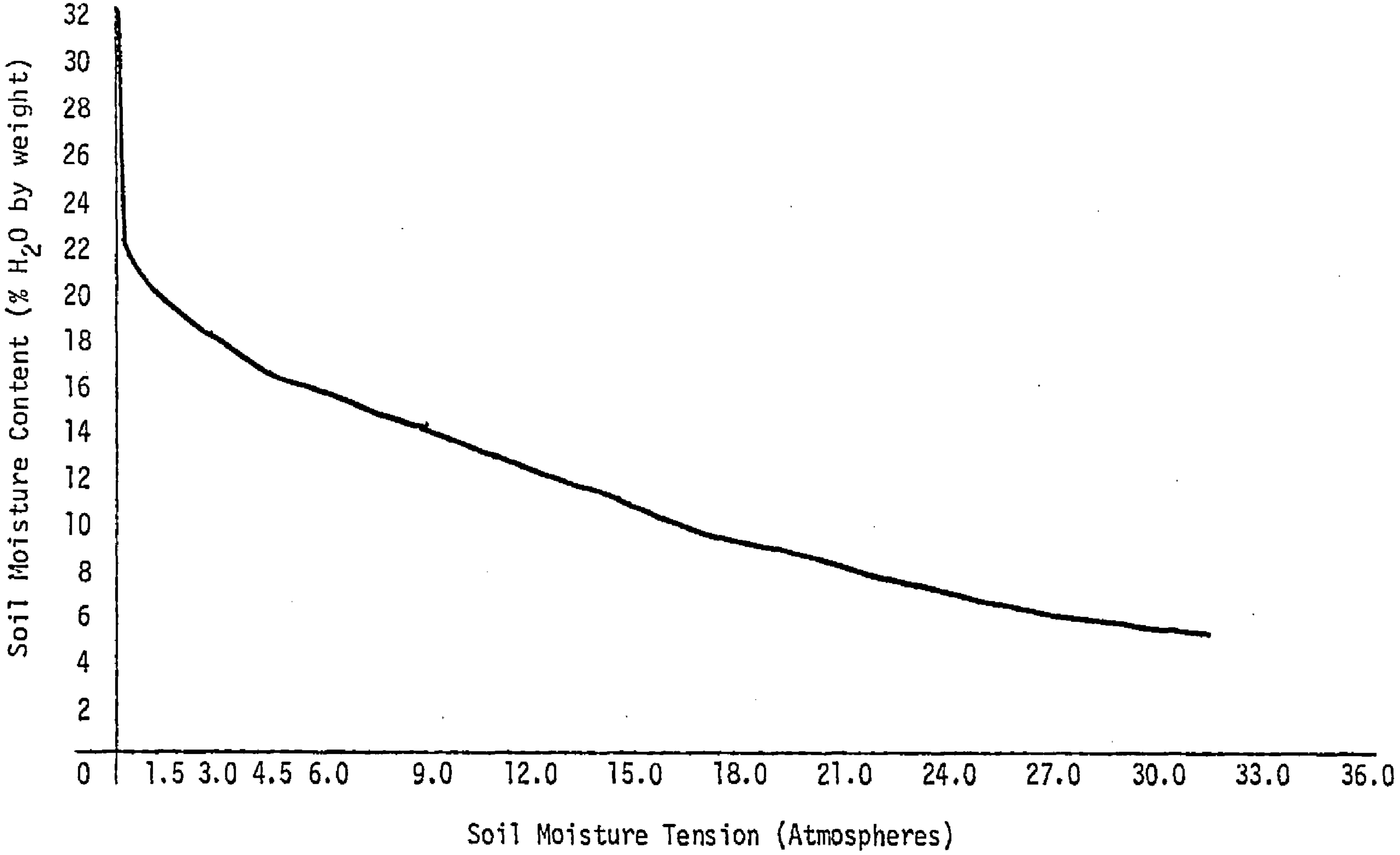

Figure 7. Soil moisture retention curve for the Crosby silt loam soil used in the NH_3 volatilization experiments.

15 atm. decreased to 10.5 atm.

air-dry (31 atm.) decreased from 31 atm. to 18.9 atm.

The 0 and 0.1 atm. treatments were essentially the same after sludge was applied, but the increased H_2O content after sludge application does not explain why the 15 atm. treatment had the same NH_3 loss as the more saturated soils. The literature has shown that there are several competing mechanisms which determine the effects of soil moisture on NH_3 volatilization. In the case of the 0 and 0.1 atm. treatments, there was a free liquid surface during most of the 24 hour sampling period, and Wahhab et al. (1957), Jewitt et al. (1947) and others have shown that the evaporation of water is important in NH_3 volatilization. With these two treatments, also, there was a minimum of contact between the sludge liquid containing the dissolved NH_3 and the soil. This reduced the ability of the soil to absorb and hold NH_4^+.

In the case of the 15 atm. and air-dry treatments, moisture tensions were high enough after addition of the sludge to absorb the sludge liquid. The only difference noted between these two treatments was the observation that the air-dry soil (31 atm.) absorbed the liquid sludge more rapidly and to a greater depth than the 15 atm. soil. Absorption of the sludge liquid to a shallow depth would have resulted in a much higher moisture content and lower moisture retention than was calculated for the entire volume of soil in the pan. Also, movement of the sludge liquid containing NH_3 to a greater depth in the air-dry soil, increased the depth of soil through which NH_3 gas would have to diffuse to the surface.

A statistically significant increase in NH_3-N volatilization with increasing initial soil moisture was given by regression equations in linear, quadratic, and cubic forms, respectively (Table 7). As shown in Table 7, the cubic equation had the greatest R^2 value and the predicted values most closely approximated the measured values.

Experiment 2: Sludge Incorporation

The results showed that NH_3-N volatilization decreased when sludge was incorporated (Table 8 and Figure 8). The sludge was incorporated immediately prior to sampling and the control for the experiment was the sludge incorporated at 24 hours.

For the sludge which was incorporated immediately (0 hours), the peak period of loss occurred initially, indicating that the main process operating here was the exposure of the sludge to the atmosphere. Once the sludge was incorporated, other factors aiding continued NH_3-N volatilization were the evaporation of moisture from the sludge as it dried and diffusion of NH_3 to the surface. Also, sludge incorporation with the soil did not totally seal it off from further contact with the atmosphere since some of the sludge (due to the mixing process) was still at or near the soil surface and thus able to continue to volatilize.

Table 7. The R^2, significance and some predicted and actual values for the linear, quadratic, and cubic regression equations for 6% (31 atm.), 10% (15 atm.), and 22% (0.1 atm.) soil moisture.

Equation Type	R^2	Significance	Percent Moisture	Actual Value (mean) %*	Predicted Value %*
linear: % NH_3-N volatilized/cylinder = 6.64 + 0.86 (% Soil Moisture)	0.37	p = 0.0003	6	6.9	11.8
			10	26.6	15.2
			22	25.7	25.4
quadratic: % NH_3-N volatilized/cylinder = -4.86 + 2.75 (% Soil Moisture) - 0.05 $(\text{\% Soil Moisture})^2$	0.42	p = 0.0006	6	6.9	9.7
			10	26.6	16.9
			22	25.7	27.2
cubic: % NH_3-N volatilized/cylinder = -58.99 + 15.41 (% Soil Moisture) - 0.05 $(\text{\% Soil Moisture})^2$ + 0.01 $(\text{\% Soil Moisture})^3$	0.55	p = 0.0001	6	6.9	7.1
			10	26.6	27.3
			22	25.7	28.5

*NH_3-N volatilized over 24 hours as percent of total NH_3-N applied.

Table 8. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 2: Sludge Incorporation.

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
		ug NH_3-N/cylinder					
				Incorporation Initially			
1	263100	200	300	300	100	0	0
2	264200	0	0	0	0	0	0
3	307800	600	600	600	100	0	0
4	290200	400	0	0	0	0	200
5	294500	1000	900	500	700	300	200
Mean	283960	440	360	280	150	60	80
				Incorporation in 1 Hour			
1	263100	1600	300	100	0	0	0
2	264200	1600	100	0	0	100	0
3	307800	3100	900	500	400	0	0
4	294500	2900	800	400	500	300	100
5	290200	1300	1500	200	100	0	200
Mean	283960	2100	720	240	200	80	60

Table 8. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Incorporation 3 Hours After Application			
1	263100	1300	2200	300	100	0	0
2	264200	2500	2000	200	0	100	0
3	307800	1500	1600	500	100	0	0
4	294500	3200	3800	700	500	300	0
5	290200	100	200	100	100	0	200
Mean	283960	1720	1960	360	160	80	40
				Incorporation 6 Hours After Application			
1	263100	2400	3100	2800	500	0	0
2	264200	2100	2100	1700	0	0	0
3	307800	2900	5200	5100	800	0	0
4	294500	2100	3200	3100	600	100	0
5	290200	800	500	200	0	0	100
Mean	283960	2060	2820	2580	380	20	20

Table 8. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
		ug NH_3-N/cylinder					
			Incorporation 12 Hours After Application				
1	263100	600	1300	1000	1000	0	0
2	264200	1600	1600	1700	1200	0	0
3	307800	900	1200	1500	800	100	0
4	294500	2400	3300	2800	2200	400	0
5	290200	500	500	400	500	0	0
Mean	283960	1200	1580	1480	1140	100	0
			Incorporation 24 Hours After Application				
1	263100	1100	1800	1100	1200	700	0
2	264200	1500	2000	1500	1300	800	0
3	307800	3900	3700	3200	2300	1200	100
4	294500	2900	3700	3300	2700	2000	400
5	290200	900	700	500	300	0	100
Mean	283960	2060	2380	1920	1560	940	120

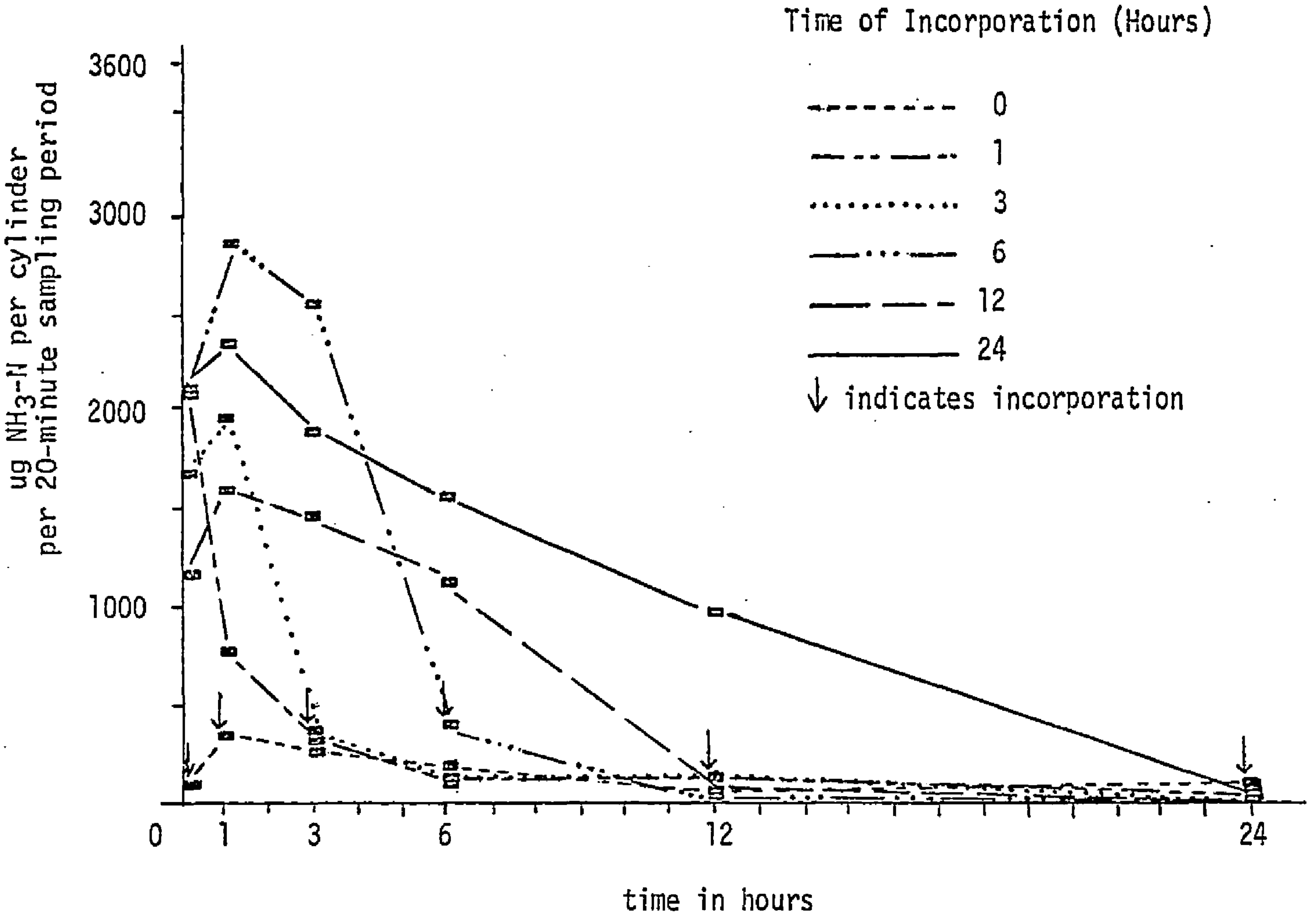

Figure 8. NH_3-N volatilized versus sampling period for sludge incorporated 0, 1, 3, 6, 12, and 24 hours after application.

The peak period of loss occurred at the one hour sampling period, for the sludges incorporated at 3, 6, 12, and 24 hours. For the sludge incorporated initially and at the one hour sampling period, the peak periods of volatilization occurred at the initial reading. As Figure 8 shows, at each period of incorporation, NH_3-N loss was dramatically reduced. The NH_3-N volatilized gradually declined over the experimental period for all periods of incorporation.

The greatest reductions in ammonia volatilization occurred by incorporating the sludge immediately. Table 9 indicates that the pattern of volatilization for the sludge incorporated immediately was distinct from that of the sludge incorporated at 24 hours. NH_3-N volatilization decreased gradually over time for the sludge incorporated immediately and at 24 hours in contrast to the other incorporation treatments which decreased abruptly after they were incorporated. NH_3-N volatilization for the sludges incorporated initially, 1 and 3 hours later was statistically significant from the NH_3-N volatilization from the sludge incorporated at 24 hours. Less volatilization occurred over the 24-hour experimental period when the sludge was incorporated within three hours than at 24 hours (Table 9). The eight to four fold reduction in volatilization with immediate, 1 hour, and 3 hour incorporation (Table 9) versus the 24-hour incorporation indicates that significant nitrogen conservation can be achieved through timely incorporation of the sludge after its

Table 9. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for sewage sludge incorporated in soil initially and at 1, 3, 6, 12, and 24 hours.

Periods of Incorporation	NH_3 Volatilized Over 24 Hours as % of total NH_3-N Applied	Test of line Equality*	Duncan's Multiple Range Test*
Initial (15 min.)	3.1	a	a
1 hour	4.0	b	a
3 hours	6.3	b	a
6 hours	14.2	bc	ab
12 hours	13.6	bc	ab
24 hours	25.8	c	b

*Means followed by the same letter are not significantly different at the 0.05 level.

application. The practicality of immediate sludge incorporation will be discussed later.

There was a linear increase ($p = 0.0001$), $R^2 = 0.43$, in volatilization with time of incorporation (see Appendix B):

$$\%\ NH_3\text{-}N \text{ volatilized/cylinder} = 3.62 + 0.87 \text{ (time of incorporation in hours)}$$

For incorporation at 1, 6, and 24 hours, the calculated values for percent NH_3-N volatilized are 4.5, 8.8, and 24.5%, respectively. This compares to actual values of 4.0, 8.8, and 25.8% respectively.

Experiment 3: Soil pH

Figure 9 shows that increasing soil pH increases ammonia volatilization, with the lowest pH (5.1) having the lowest amount of ammonia volatilized and the highest pH (7.5) having the greatest amount (Table 10). The general shapes of the curves, however, were quite similar, and were not distinct (Table 11).

Table 11 shows values of 20.0, 21.0, and 23.6% of applied NH_3-N volatilized for soil pH's of 5.1, 6.7, and 7.5, respectively. The volatilization loss at pH 7.5 was significantly higher than the losses at the lower pH's. As shown by Table 12, the quadratic equation would seem to be the best choice for the pH range 5.1 to 7.5. For the pH value of 6, however, the predicted value is 13.60 for the quadratic and 15.4 for the linear equation, The predicted value for the quadratic equation thus declines between pH 5.1 and pH 6.7 while

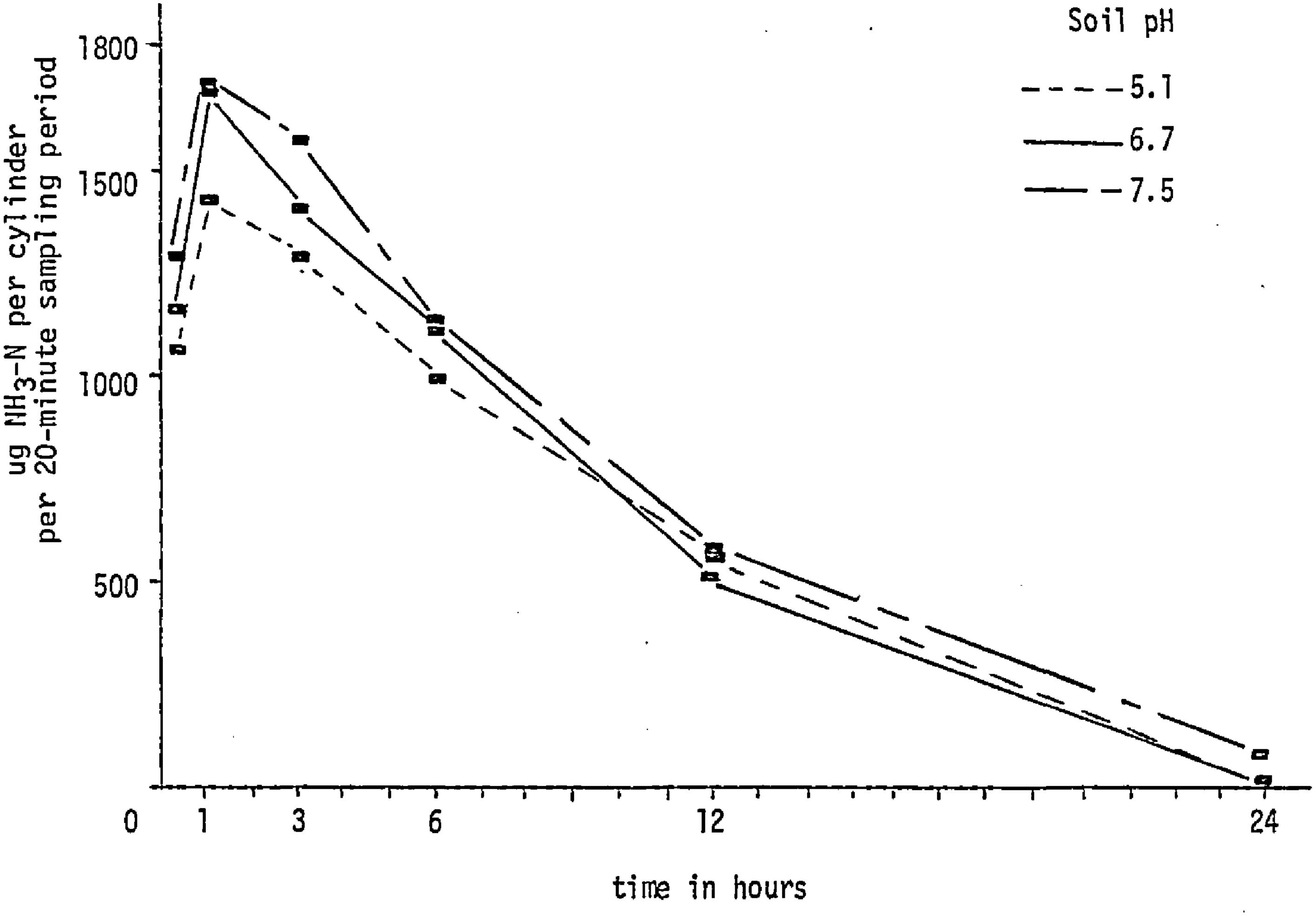

Figure 9. NH_3-N volatilized versus sampling period for sewage sludge applied to soils with pH's of 5.1, 6.7, and 7.5.

Table 10. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 3: Soil pH.

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
		ug NH_3-N/cylinder					
				pH = 5.1			
1	317200	1500	1600	1000	700	500	0
2	311400	700	1100	900	700	400	0
3	303700	1300	1400	1300	700	400	0
4	317200	1200	1200	1600	1500	900	0
5	311400	800	1700	1400	1100	600	0
6	303700	1000	1700	1700	1300	500	0
Mean	310767	1083	1450	1317	1000	550	0
				pH = 6.7			
1	317200	1400	1800	1500	1000	400	0
2	311400	1200	1500	1200	1100	400	0
3	303700	1500	1900	1500	1200	500	0
4	317200	1300	2100	1700	1200	700	0
5	311400	600	1200	1000	900	500	0
6	303700	1000	1700	1600	1300	500	0
Mean	310767	1167	1700	1417	1117	500	0

Table 10. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				pH = 7.5			
1	317200	1500	1900	1300	700	300	0
2	311400	1100	1300	1000	700	400	0
3	303700	1500	1700	2000	1300	700	200
4	317200	1600	1900	1600	1500	900	0
5	311400	1000	1500	1900	1400	800	0
6	303700	1100	2000	1700	1200	600	200
Mean	310767	1300	1717	1583	1133	567	67

Table 11. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for sewage sludge applied to soil pH's of 5.1, 6.7, and 7.5.

Soil pH	NH_3-N Volatilized Over 24 Hours as % of Total NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
5.1	20.0	a	a
6.7	21.0	a	a
7.5	23.6	a	b

*Means followed by the same letter are not significantly different at the 0.05 level.

Table 12. The R^2, significance, and some predicted and actual values for the linear and quadratic regression equations for soil pH's of 5.1, 6.0, 6.7, and 7.5.

Equation Type	R^2	Significance	pH	Actual Value (mean) %*	Predicted Value %*
linear: % NH_3-N volatilized/cylinder = 2.65 + 2.12 (pH)	0.30	p = 0.028	5.1	14.4	13.5
			6.0	--	15.4
			6.7	15.0	16.7
			7.5	20.1	20.5
quadratic: % NH_3-N volatilized/cylinder = 90.94 - 26.68 (pH) + 2.30 $(pH)^2$	0.45	p = 0.022	5.1	14.4	14.7
			6.0	--	13.6
			6.7	15.0	15.0
			7.5	20.1	20.1

*NH_3-N volatilized over 24 hours as percent of total NH_3-N applied.

the linear equation predicts an increase as pH rises from 5.1 to 6.7.

Previous work (Ivanov, 1964) has shown that the greatest effect of soil pH on ammonia volatilization occurs at high pH's, and particularly when the soil is calcareous. The pH 7.5 Crosby soil used in this study did not have free carbonates. One other factor may have reduced the effect of soil pH on NH_3 loss. The soil in this experiment had an initial moisture content of 0.1 bar, which has been previously shown to reduce the contact between the sludge liquid and the soil. This would reduce the ability of the soil to change the solution pH which was 7.2 for the sludge itself.

Experiment 4: Sludge Type

There were large differences in the ammonia volatilized from the different sludges (Table 13 and Figure 10), but some of these differences are due to the fact that different amounts of NH_3-N were applied for the different sludges (see Table 4). The same approximate amounts of NH_3-N were applied for the Medina aerobically digested, Columbus anaerobically digested, and dewatered Columbus anaerobically digested sludges (Table 13). Because of one replication, the amount of NH_3-N applied for the Ashland lime-stabilized liquid primary sludge was higher than the other sludge treatments. If this replication were excluded, then the amount of NH_3-N applied was similar to the other treatments. The greater NH_3-N present in the third replication could be due to a greater

Table 13. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 4: Sludge Type.

		NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
Replicate	NH_3-N Applied	0	1	3	6	12	24
		ug NH_3-N/cylinder					
				Columbus			
1	142900	500	500	200	100	100	0
2	145500	500	500	300	300	0	0
3	138400	800	900	600	400	300	100
Mean	142267	600	633	367	267	133	33
				Dewatered Columbus			
1	140700	400	700	400	100	0	200
2	152100	1200	1200	800	500	100	0
3	148400	700	800	500	200	0	100
Mean	147067	767	900	567	267	33	100
				Medina			
1	129800	0	100	0	0	0	100
2	129800	0	0	0	0	0	0
3	139400	0	0	0	0	0	0
Mean	133000	0	33	0	0	0	33

Table 13. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Columbus Compost			
1	16500	0	0	0	0	0	0
2	16500	0	0	0	0	0	0
3	16500	0	0	0	0	0	0
Mean	16500	0	0	0	0	0	0
				Ashland			
1	154500	2000	1000	200	200	100	100
2	154000	4500	2100	900	500	300	100
3	306300	5700	3700	1700	800	600	400
Mean	204933	4067	2267	933	500	333	200

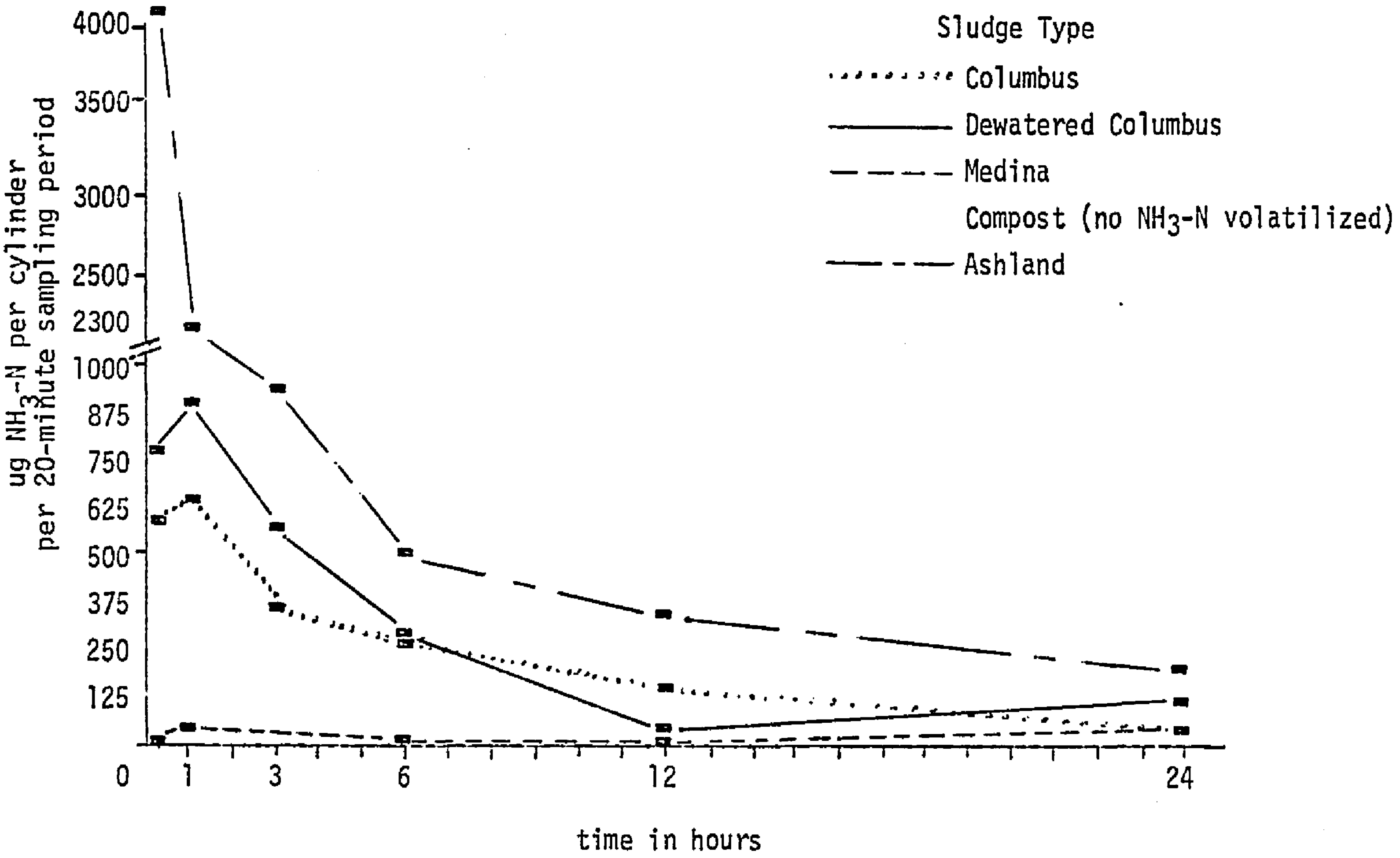

Figure 10. NH_3-N volatilized versus sampling period for an Ashland primary lime-stabilized sludge, a Columbus anaerobically digested sludge, a composted Columbus primary sludge, a Medina aerobically digested sludge, and a dewatered Columbus anaerobically digested sludge applied to soil.

concentration of sludge solids than in the other two replications. The sludge for the replication was taken after the container containing the Ashland sluge was inverted to thoroughly mix the sludge solids and liquid. By the third replication, there was simply less liquid sludge to invert and mix with the solids present in the bottom of the container. Approximately 1.5 times as much NH_3-N was applied with the Ashland lime-stabilized liquid primary sludge as for the Medina aerobically digested, Columbus, and dewatered Columbus anaerobically digested sludges. Only 0.12 times as much NH_3-N was applied with the Columbus compost as with the Medina and Columbus and dewatered Columbus sludges.

The Ashland sludge (Figure 10) had very high NH_3-N losses throughout the 24 hour period. The ammonia values dropped off rapidly for the six hour sampling and then more gradually over the remainder of the sampling period. Even if the third replication value is omitted, values for the other two replications still show higher NH_3-N loss compared to the other sludges for the sampling period. The pH of the Ashland sludge, a lime-stabilized sludge, is 12, and this was responsible for the rapid loss of ammonia. The smell of the sludge was consistently very strong from the initial to the final sampling and beyond. The sludge continued to consistently lose ammonia even after the experiment was terminated. Table 14 shows that 15.8% of the ammonia was volatilized in 24 hours. If the third replication were omitted, the amount of NH_3-N volatilized

Table 14. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for a Columbus anaerobically digested sludge, a dewatered Columbus anaerobically digested sludge, a Medina aerobically digested sludge, a composted Columbus primary sludge, and an Ashland lime-stabilized primary sludge.

Sludges	Type of Sludge	NH_3 Volatilized Over 24 Hours as % of Total NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
Columbus	anaerobically digested	8.3	a	a
Dewatered Columbus	anaerobically digested	7.8	a	a
Medina	aerobically digested	0.4	a	a
Columbus Compost	primary	none detected	a	a
Ashland	aerobically digested, lime-stabilized	15.8	b	b

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*Means followed by the same letter are not significantly different at the 0.05 level.

increased from 15.7 to 20.97%. This was the largest percentage of ammonia volatilized for any sludge treatment in Experiment 4.

The pattern of ammonia volatilization for the different sludges fell into three groups: Ashland; the two Columbus sludges; and the Medina sludge and Columbus compost. However, only the Ashland sludge gave a pattern of volatilization that was statistically significant from the others (Table 14). The Ashland sludge had significantly more NH_3 loss in the 24 hour sampling period than did the other sludges. The lack of statistical significance in NH_3 loss among the other sludges was due, in part, to the lower number of replications per treatment (three) used in this experiment and to the very high volatilization of the Ashland sludge compared to the others. The number of replications for this experiment was limited by the number of treatments and the amounts of the different sludges available.

Of the different sludges studied, the lime-stabilized material is the least common for land application in Ohio and other areas. Although it is a good source of phosphorus and nitrogen, these results indicate that much of the NH_3 (which usually accounts for 60-70% of the available N) can be lost if not immediately incorporated. Other problems with lime-stabilized sludge such as potential odor and physical handling reduce the suitability of this material for application to cropland.

Experiment 5: Temperature

Three temperatures were studied: 12.8, 18.3, and 26.7°C. The 12.8 and 18.3°C temperatures were chosen for several reasons. Volk (1959, 1968), in a series of experiments, found that ammonia volatilization was retarded by a temperature of 7.2°C but increased significantly above 15.6°C. Ernst and Massey (1960) studied ammonia volatilization from surface applied urea at 7.2, 15.6, 23.8, and 32.2°C and found that each 8.4°C increase significantly increased ammonia volatilization. The three temperatures chosen for this study (12.8, 18.3, and 26.7°C) gave sufficient range to measure any differences in ammonia volatilized due to temperature while taking into consideration the limitation of the growth chamber used. The growth chamber was old and I was advised to be careful in using low temperatures so as to avoid a mechanical failure, since the temperature experiments were conducted in August.

The first experiment was conducted August 5, 1981, with the growth chamber temperature at 18.3°C. The second experiment was conducted on August 7, 1981 with a temperature of 26.7°C; and the third experiment was conducted on August 9, 1981 with a temperature near 12.8°C. The 18.3 and 26.7°C temperatures were held constant throughout the experimental periods. The 12.8°C temperature was hard to achieve and hold constant throughout the experimental period. The measured temperature setting in the growth chamber varied from a high of 14.4°C for a brief period to a low of 11.8°C. Half of the experimental period occurred at 11.8°C.

The data are summarized in Table 15 and presented graphically in Figure 11. The pattern of volatilization for the 26.7 and 18.3°C temperatures (Figure 11) were similar, with the ammonia volatilization values greater for the 26.7°C temperature compared to the 18.3°C temperature. The principal effect seems to be at the initial sampling, where volatilization at 18.3°C was approximately 2/3 that at 26.7°C. At the one-hour sampling, the respective values are closer, with the 18.3°C temperature value being 80% of the NH_3-N volatilization at 26.7°C. The patterns of volatilization were distinct from each other (Table 16) and from the 12.8°C treatment. The percent of applied NH_3-N volatilized in 24 hours from the 26.7°C temperature was 13.6% compared to 9.8% from the 18.3°C temperature (Table 16), but these differences were not statistically significant.

The pattern of volatilization for the 12.8°C temperature shows much lower volatilization and declines much more gradually than at the two higher temperatures. For the 24 hour period, 2.3% of the applied ammonia was volatilized (Table 16), which was statistically significant compared to the other two temperatures.

As the temperature increased, there was an increase in the 24-hour percent NH_3-N volatilization loss ($p = 0.0009$; $r^2 = 0.63$):

$$\%\ NH_3\text{-N volatilized/cylinder} = -28.88 + 3.31\ (\text{temperature}) - 0.07\ (\text{temperature})^2$$

For temperatures of 12.8, 18.3, and 26.7°C, the predicted percent

Table 15. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 5: Temperature.

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Temperature of 12.8°C			
1	326200	100	100	100	200	100	0
2	326200	100	300	0	300	0	0
3	326200	100	500	0	0	0	0
4	326200	400	400	0	300	200	0
5	326200	300	300	0	0	0	0
6	326200	500	400	400	500	200	0
Mean	326200	250	333	83	217	83	0
				Temperature of 18.3°C			
1	326700	500	800	800	500	400	100
2	326700	300	900	1000	700	300	100
3	326700	600	1200	700	800	300	200
4	326700	800	1000	700	400	300	0
5	326700	1200	1500	1000	800	300	0
6	326700	1200	1400	1200	700	400	100
Mean	326700	767	1133	900	650	333	167

Table 15. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
		ug NH_3-N/cylinder					
		Temperature of 26.7°C					
1	354300	1400	1800	1300	1600	800	300
2	354300	1200	1300	1300	800	600	0
3	354300	1200	1500	1500	700	500	100
4	354300	900	1100	700	800	300	0
5	354300	1100	1400	1100	1000	300	0
6	354300	1100	1500	900	800	300	100
Mean	354300	1150	1433	1133	950	467	83

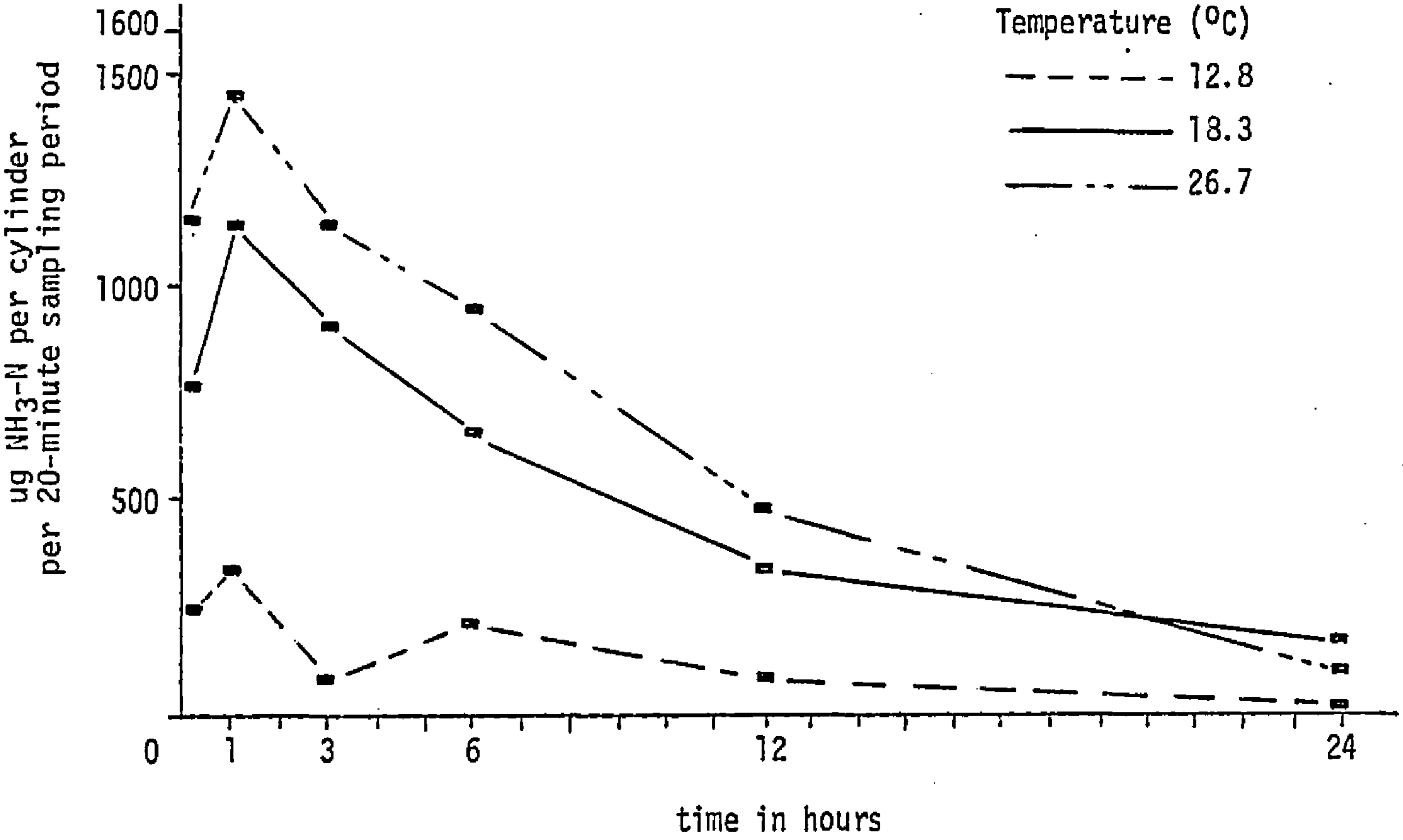

Figure 11. NH_3-N volatilized versus sampling period for sewage sludge applied to soils at temperatures of 12.8, 18.3, and 26.7°C.

Table 16. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for sewage sludge applied to soils at temperatures of 12.8, 18.3, and 26.7°C.

Temperature (°C)	NH_3 Volatilized Over 24 Hours as % of Total NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
+12.8	2.3	a	a
+18.3	9.8	b	b
+26.7	13.6	c	b

*Means followed by the same letter are not significantly different at the 0.05 level.

NH_3-N volatilized was 2.9, 9.9, and 13.1%, respectively. The actual values were 2.3, 9.8, and 13.6%, respectively (see Appendix E).

In Ohio, it is common to spread sludge year-round since many sewage treatment plants do not have sufficient storage capacity to avoid spreading for more than a month or so. Sludge spread during the winter is not incorporated because of frozen soil, but this data would indicate that NH_3 volatilization losses would be quite low during these periods. On the other hand, much sludge is spread in the summer months, particularly on fields from which wheat has been harvested and on hay and pasture lands. Surface temperatures during this period can greatly exceed the maximum temperature of 26.7°C studied here, and volatilization losses would be expected to be much greater than the 13.6% obtained for that temperature.

Experiment 6: Vegetative Cover (Large Sludge Particles)

Sampling began on September 2, 1981 and a total of four experiments were conducted. A new batch of the Columbus dewatered sludge with a solids content of 27.7% was collected just prior to this experiment and proved difficult to thoroughly mix with distilled water. Small chunks of sludge remained even after thorough mixing. The vegetative cover had two forms: wheat straw and a Kentucky blue grass sod cut to a height of 3.5 cm.

The data are given in Table 17 and in Figure 12. The peak pattern of NH_3 loss for both the straw and sod was greater and of longer duration than for the bare soil (Figure 12). The straw and

Table 17. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 6: Vegetative Cover (Large Sludge Particles).

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Bare Soil			
1	176400	400	100	300	200	0	0
2	176400	100	200	400	200	100	0
3	179000	300	800	500	300	200	0
4	179000	400	700	500	300	300	0
5	183500	300	400	300	300	200	0
6	188900	400	500	400	100	0	0
7	183500	400	500	400	300	300	0
8	188900	100	300	300	100	0	0
Mean	181950	300	437	387	225	137	0

Table 17. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of: 0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Kentucky Blue Grass Sod			
1	176400	300	200	400	200	200	0
2	176400	300	700	600	400	200	0
3	179000	400	600	600	300	400	0
4	179000	400	500	500	400	400	0
5	183500	500	700	700	700	500	200
6	183500	300	500	500	400	400	0
7	188900	300	600	600	300	100	0
8	188900	0	500	500	200	0	0
Mean	181950	313	537	550	363	275	25

Table 17. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Wheat Straw			
1	176400	300	500	600	400	300	0
2	176400	100	400	500	300	200	0
3	179000	500	700	500	400	400	0
4	179000	400	500	500	400	400	100
5	183500	400	700	800	800	1000	400
6	183500	200	500	400	500	400	100
7	188900	600	700	700	500	400	0
8	188900	600	600	600	200	0	0
Mean	181950	387	575	575	437	387	75

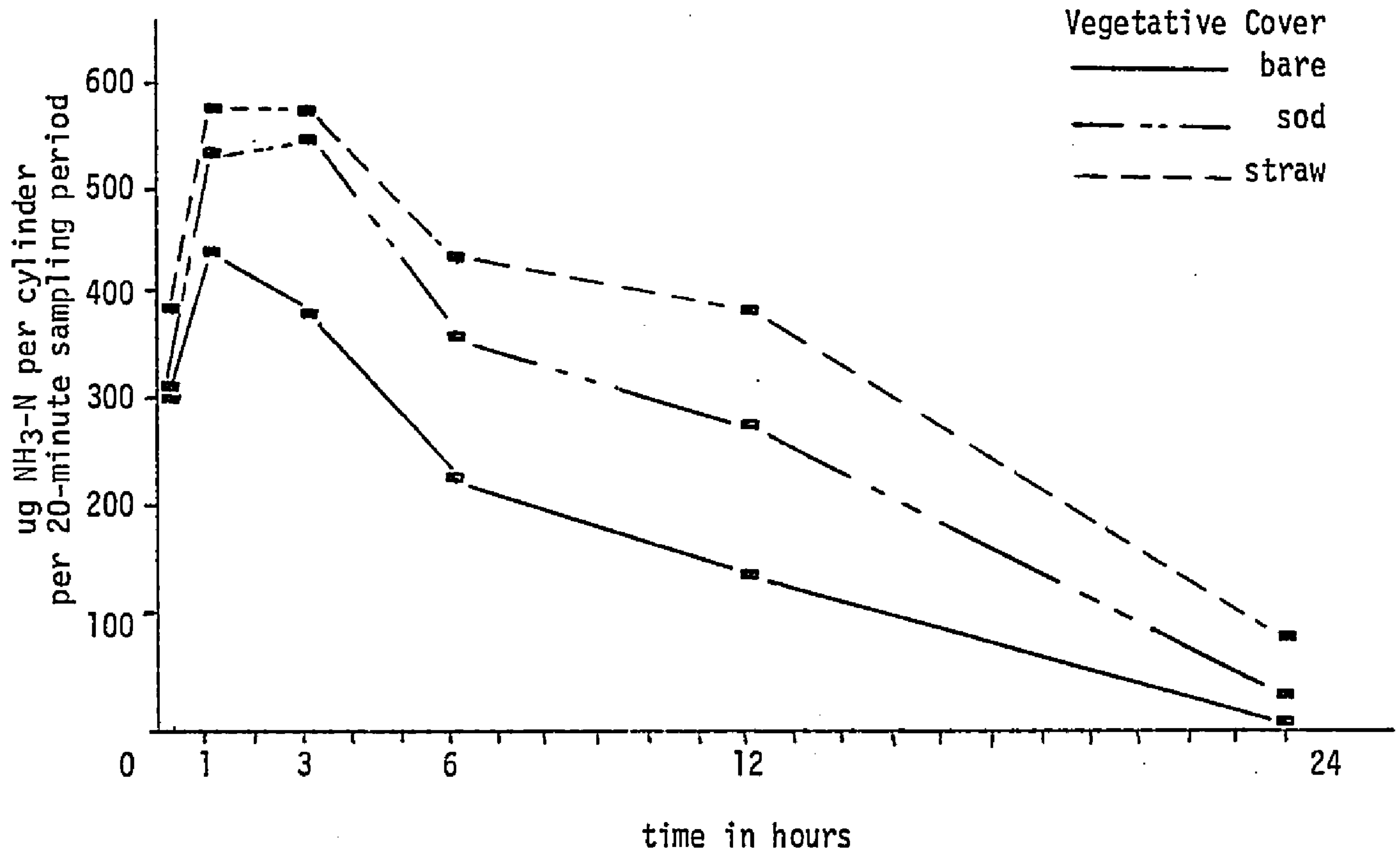

Figure 12. NH_3-N volatilized versus sampling period for sewage sludge containing large sludge particles applied to a straw-covered soil, a sod, or bare soil.

sod seemed to reach a plateau in ammonia volatilization from the one hour to the three hour reading, and then declined over time, while the bare soil had a peak reading at one hour and then declined over time. The volatilization pattern for the two vegetative cover treatments were statistically distinct (Table 18) from that of the bare soil but not from each other. The volatilization from the bare soil was statistically significant (6.4% of the NH_3-N applied) compared to the volatilization from the soil with the wheat straw cover (14.3%), but the sod treatment was not statistically different from the other two (Table 18).

When sewage sludge was surface applied to the straw covered soil, the straw retained some of the sewage sludge chunks preventing them from making contact with the soil. The straw acted as a physical barrier. Meyer et al. (1961) found that, when a urea-ammonium nitrate fertilizer solution was sprayed on a straw residue covering an acid soil, the amount of NH_3 loss was similar to an alkaline or neutral soil due to the straw physically intercepting the solution and the volatilization occurring from the spray on the straw surface and not from the spray on the soil surface. Something similar appears to have been occurred here. The straw had the largest percent ammonia volatilization loss and the grass was next. An additional factor besides the interception of part of the sewage sludge was that the grass sod was very thick, and possibly the air circulation through and around the grass sod was not as efficient as through the straw, allowing more volatilization to occur from the

Table 18. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for sewage sludge containing large sludge particles applied to soils with vegetative cover (wheat straw or sod) and a bare soil.

Vegetative Cover	NH_3-N Volatilized Over 24 Hours as % of Total NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
Wheat Straw	14.3	a	a
Sod	11.3	a	ab
Bare Soil	6.4	b	b

*Means followed by the same letter are not significantly different at the 0.05 level.

straw. The grass was growing in a vertical direction, while the straw was in a horizontal direction, allowing more of the sludge and water mixture to be physically intercepted by the straw compared to the grass, thus presenting a larger volatilization surface from the straw. The grass sod was cut to 3.5 cm before the experiment began, and it is possible that the grass sod was a sink for some of the sludge ammonia and this decreased volatilization; but active growth was not observed until after the experiment was terminated.

Ammonia loss was least from the bare soil, and evidently the contact of the sewage sludge mixture with the soil prevented part of the loss.

Experiment 6: Surface Cover (Liquid Sludge)

The experiment was repeated as described for the first part of Experiment 6. The sludge was mixed with distilled water, allowed to stand for five minutes, then again mixed with distilled water. The repeated mixing process was sufficient to eliminate the large sludge particles.

The results (Table 19 and Figure 13) show that volatilization from the bare soil peaked at 1 hour then declined throughout the rest of the experiment. It had the highest peak volatilization rate for any of the treatments, but rapidly declined and had lower values than the other treatments from the third hour reading on. The straw treatment reached a plateau in NH_3-N volatilized from the first hour reading to the third hour reading then declined over time. The sod

Table 19. NH_3-N (ug/cylinder) applied and recovered in the 20-minute sampling periods by replication for Experiment 6: Vegetative Cover (Homogenous Sludge).

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Bare Soil			
1	194900	200	400	500	400	0	0
2	219100	500	700	500	400	200	0
3	219100	0	500	400	200	0	0
4	222300	200	600	600	300	200	0
5	222300	400	700	600	200	0	0
6	194900	500	500	500	500	300	0
Mean	212100	300	567	517	333	117	0
				Kentucky Blue Grass Sod			
1	194900	0	0	200	100	0	0
2	194900	200	100	200	300	0	0
3	219100	400	500	500	400	0	0
4	219100	400	1000	1200	1000	700	200
5	223000	300	500	700	500	500	0
6	222300	200	300	500	100	0	0
Mean	212100	250	400	550	400	200	33

Table 19. (continued)

Replicate	NH_3-N Applied	NH_3-N Recovered in 20-minute Sampling Period at Intervals (Hours) of:					
		0	1	3	6	12	24
	ug NH_3-N/cylinder						
				Wheat Straw			
1	194900	400	300	300	200	0	0
2	194900	800	1000	800	700	500	0
3	219100	300	600	400	400	100	100
4	219100	100	300	300	100	100	0
5	222300	100	500	600	200	100	0
6	222300	100	500	700	400	300	0
Mean	212100	300	533	517	333	183	17

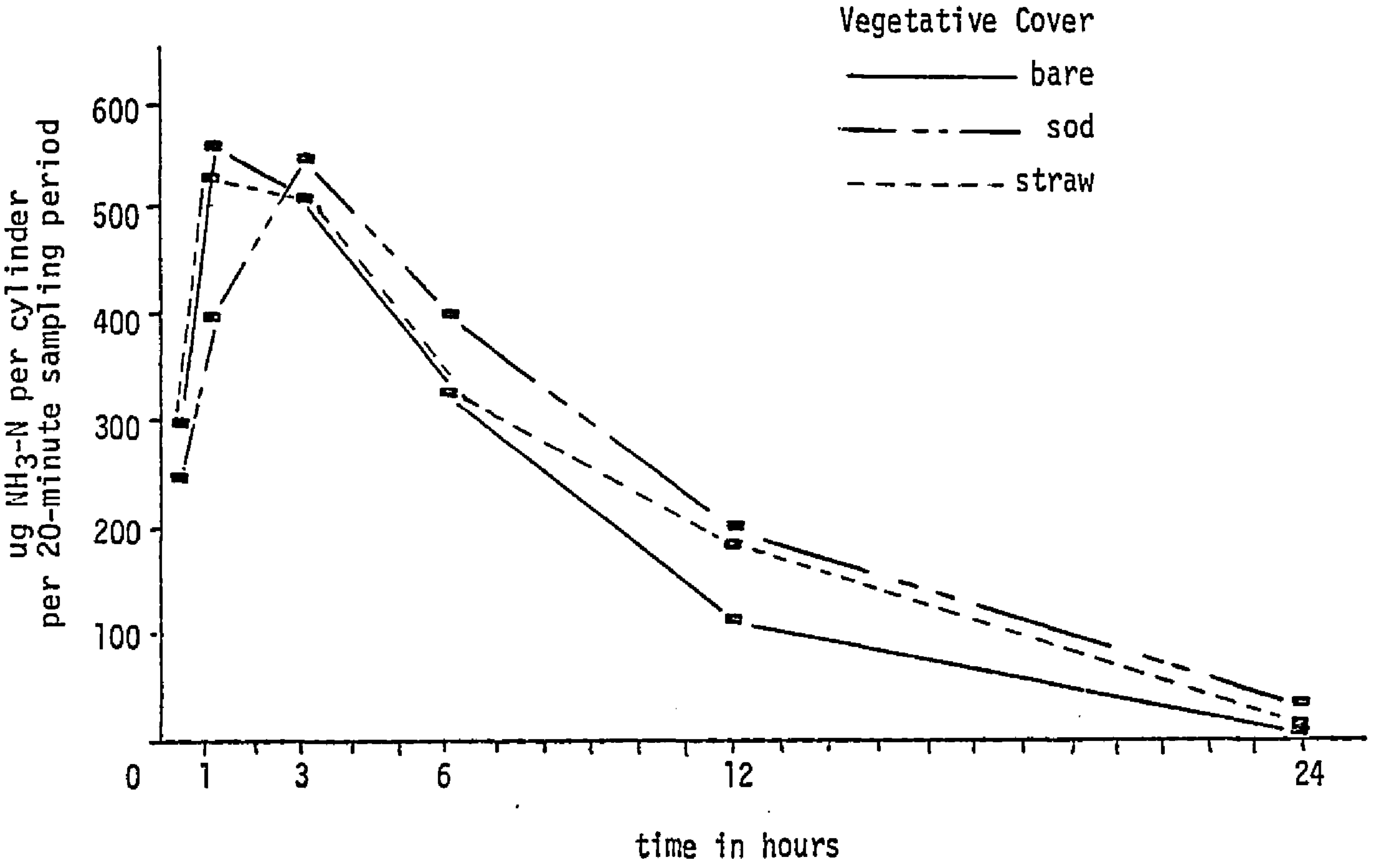

Figure 13. NH_3-N volatilized versus sampling period for a liquid sewage sludge applied to a straw-covered soil, sod, or bare soil.

treatment had a peak reading at three hours and then declined. The volatilization patterns for the three treatments, however, were not distinct (Table 20) nor were there any statistically significant differences in the amounts of NH_3 volatilized in the 24-hour experimental period.

The wheat and sod delayed the peak periods of NH_3 volatilization compared to the bare soil as they did in the first part of the experiment with sludge containing sludge particles. The main difference, however, was that there was greater overall volatilization with the vegetation treatments in the first part of the experiment (large sludge particles, Figure 12), but little or no effect of these treatments with the more homogenized sludge. Becaue of its high water solubility, almost all of the ammonia in liquid sludge is associated with the liquid fraction and little is on the sludge solids. If sludge solids were trapped on the surface of the straw and sod, volatilization of NH_3 from the particles themselves would probably not be enough to account for the differences observed. Another possible explanation, however, is that the solids may have prevented the sludge liquid from rapidly moving through the vegetative cover to the soil surface by plugging the spaces between the straw particles or blades of grass. In addition, the sludge solids are organic and would have retained a high percentage of moisture even after most of the sludge liquid had moved through the vegetative layer and into the soil. Ammonia could have volatilized from the absorbed water.

Table 20. NH_3-N volatilized as percent of NH_3-N applied and tests of significance for a well mixed sewage sludge applied to soils with vegetative cover (wheat straw or sod) and a bare soil.

Vegetative Cover	NH_3-N Volatilized Over 24 Hours as % of Total NH_3-N Applied	Test of Line Equality*	Duncan's Multiple Range Test*
Wheat Straw	9.1	a	a
Sod	0.1	a	a
Bare Soil	6.4	a	a

*Means followed by the same letter are not significantly different at the 0.05 level.

Although the effects of vegetative cover on NH_3 volatilization from sewage sludges were found to be small, and affected by the physical characteristics of the sludge itself, these effects could be important in the field. Many sludges are land-applied as filter cake or centrifugal sludge which contain around 10-25% solids, and the sludge particle effects noted in this research could be more significant with sludges of this type. Also, application of sludges to land with vegetative cover (wheat stubble, corn stalk residues, hay or pasture land) is a recommended practice wherever feasible to reduce soil compaction and rutting by the applicator truck and to minimize runoff.

Comparisons Between Experiments

In order to determine if comparisons could be made between experiments, an analysis of variance and Duncan's Multiple Range Test were performed on the means of ammonia volatilized for the reference treatments of all experiments. There were no significant differences between the reference treatments for the experiments on soil pH, temperatures, sludge types, and vegetative cover (Table 21). There were also no significant differences between the reference treatments for the experiments on soil moisture and incorporation. However, the reference means for these two experiments were significantly different than the means of the other four experiments.

Table 21. NH_3-N volatilized as percent of NH_3-N applied and Duncan's Multiple Range Test for the reference treatments for the different experiments.

Treatment	NH_3-N Volatilized in 24 Hours as Percent of NH_3-N Applied	Duncan's Multiple Range Test*
Soil Moisture	26.6	a
Sludge Incorporation	25.6	a
pH	15.0	b
Temperature	13.1	b
Different Sludges	8.4	b
Vegetative Cover (liquid)	6.4	b
Vegetative Cover (large sludge particles)	6.3	b

*Means followed by the same letter are not significantly different at the 0.05 level.

In the soil moisture and incorporation experiments, the sludges were pre-mixed in individual batches for each cylinder, while in the other experiments all of the sludge for an experiment was prepared as one batch. This difference in preparing the sludges may have, in part, accounted for the differences in reference treatment means observed (Table 21).

Although the primary objective of this study was to examine, in a series of experiments, the relative effects of a number of variables, it is also useful to compare treatments for all of the experiments. The validity of these comparisons, however, are determined by the significance or non-significance of the reference treatments for each experiment (Table 21).

One means of comparing treatment amounts for the different experiments is to rank them in terms of their effects on NH_3 volatilization (expressed as percent of NH_3-N applied) as given in Table 22.

Considering all experimental treatments together (Table 22), sludge type, initial soil moisture, temperature, and time of incorporation tended to have greater effects on NH_3 volatilization than the other factors studied.

The predictive equations for the continuous variables discussed previously were used to calculate the effect on NH_3 volatilization of typical changes encountered under field conditions (e.g., a 10°C change). The results are given in Table 23.

Table 22. The relative ranking of treatments among all the experiments by magnitude of ammonia volatilization.

Rank	Treatment	NH_3-N Volatilized in 24 Hours (% of NH_3-N Applied)
1	0 atm. (32% soil moisture)	31.6
2	15 atm. (10% soil moisture)	26.8
3	0.1 atm. (22% soil moisture)	25.9
4	24 Hours Incorporation	25.8
5	Soil pH 7.5	23.6
6	Soil pH 6.7	21.0
7	Soil pH 5.1	20.0
8	Ashland primary lime-amended sludge	15.8
9	Wheat Straw (large sludge particles)	14.3
10	Sludge Incorporated at 6 Hours	14.2
11	Temperature of 26.7°C	13.6
12	Sludge Incorporated at 12 Hours	13.6
13	Sod (large sludge particles)	11.3
14	Temperature of 18.3°C	9.8
15	Straw (homogenous, liquid sludge)	9.1
16	Sod (homogenous, liquid sludge)	9.1
17	Columbus Anaerobically Digested Sludge	8.3
18	Air-dried Soil (6% soil moisture)	6.4
19	Bare Soil (large sludge particles)	6.4
20	Bare Soil (homogenous, liquid sludge)	6.4
21	Sludge Incorporated at 3 Hours	6.3
22	Sludge Incorporated at 1 Hour	4.0
23	Sludge Incorporated Initially	3.1
24	Dewatered Columbus Anaerobically Digested Sludge	2.6
25	Temperature of 12.8°C	2.3
26	Medina Aerobically Digested Sludge	1.3
27	Compost	0.0

Table 23. The calculated incremental effects of the continuous variables studied (temperature, soil pH, soil moisture content, and time of incorporation) on NH_3 volatilization.

Variable	Increment	Change in NH_3-N Volatilized in 24 Hours (% of NH_3-N Applied)	Difference
Temperature	12.6 to 22.6°C	2.9 to 12.8	9.9
	16.7 to 26.7°C	8.3 to 13.1	4.8
Soil pH	pH 5.1 to 6.1	13.5 to 15.6	2.1
	pH 6.1 to 7.1	15.6 to 17.7	2.1
Soil Moisture Content	6 to 10%	7.1 to 27.3	20.2
	10 to 22%	27.3 to 28.5	1.2
	22 to 32%	28.5 to 32.1	3.6
Time of Incorporation	0 to 3 hrs.	3.6 to 6.2	2.6
	0 to 12 hrs.	3.6 to 14.1	10.5
	o to 24 hrs.	3.6 to 24.5	20.9

Incorporation of the sludge significantly reduced ammonia loss, and the magnitude of the reduction was greater than the effects of the other variables except for the loss from air-dry soil. Air-dry soil would only be encountered for short periods in the summer; therefore, during the majority of time when sludge might be spread, immediate incorporation can nullify the effects of the other variables on ammonia volatilization.

CONCLUSIONS

The study of the factors affecting ammonia volatilization from sewage sludge applied to soil in the laboratory was divided into six experiments. From these experiments the following conclusions were made:

1. Soil moisture at 15 atm. tensions or lower resulted in enhanced ammonia volatilization. Ammonia volatilization was significantly lower from air-dry soil.
2. Ammonia volatilization from different periods of incorporation increased linearly with time, so sewage sludge should be incorporated as soon as possible to minimize NH_3 loss.
3. There was a trend for larger NH_3-N volatilization with increasing pH. Ammonia volatilization was significantly greater from the soil at pH 7.5.
4. An Ashland primary lime-stabilized sludge with a pH of 12 lost over 15.8% of the ammonia present in the sludge and would be a less reliable source of NH_3-N for crops than the other sludges studied.
5. Ammonia volatilization increased with increased temperatures and was significantly lower from the 12.8°C

temperature than from the 18.3 and 26.7°C temperatures.

6. Vegetative cover, compared to a bare soil, significantly increased ammonia volatilization from sludges containing solid sludge particles. There was no significant effect of vegetation, however, on NH_3 loss from a well-homogenized liquid sludge.

Implications of the Results

Central Ohio has four definite seasons, and of the variables studied here, temperature, soil moisture, vegetative cover, and opportunity for sludge incorporation vary with the season (Table 24).

Sludge incorporation is the most important factor in all four seasons in that as soon as the sludge is incorporated, ammonia volatilization is greatly reduced. Those conditions that would allow immediate incorporation, such as spring or fall plowing and planting, would significantly reduce ammonia volatilization. The longer the time span between field application and incorporation, the more significant the other factors become.

Soil moisture is another important factor in reducing ammonia volatilization, in that a very dry soil would allow rapid infiltration of sludge water and the ammonia present in the sludge water into the soil, significantly reducing ammonia volatilization. This occurs below soil moistures at 15 atm. tension, so the soil would

Table 24. The relative seasonal effects of temperature, soil moisture, vegetative cover, and sludge incorporation on ammonia volatilization from sewage sludges under field conditions in Ohio.

	Winter		Spring		Summer		Fall	
	Effect[*]	Intensity	Effect	Intensity	Effect	Intensity	Effect	Intensity
Opportunity for Sludge Incorporation	+	H	-	H	+	M	-	M
Soil Moisture	+	H	+	H	-	H	-	M
Temperature	-	H	-	L	+	H	+	M
Vegetative[‡] Cover	+	L	-	L	+	L	+	M

[*] +: opportunity for NH_3 volatilization increases; -: opportunity for NH_3 volatilization decreases.

[†] The magnitude or intensity of the effect. H = high, M = medium, L = low.

[‡] Assumes that most of the agricultural land is in cultivated crops with only a limited acreage of hay or pasture.

‡Assumes

have to be very dry, a condition which occurs more often in summer and fall. At moisture levels above 15 atm. tension moisture conditions enhance instead of reduce volatilization.

Any crop residue, such as wheat stubble, that prevents the sludge from coming in contact with the soil would enhance ammonia volatilization from the sludge. A rapidly growing crop, such as winter wheat in the spring, or hay or pasture, would adsorb some of the ammonia present in the sludge water and reduce ammonia volatilization.

During the winter, temperature would significantly reduce ammonia volatilization from the sludge. During the summer, temperature would significantly increase ammonia volatilization from the sludge, but if the sludge is incorporated or the soil is very dry, the overall volatilization will be less than that predicted by temperature alone.

Of the four factors, only sludge incorporation can be controlled by the farmer during normal farm operations without significantly changing his production schedule. Crop residue or vegetative cover is also subject to control, but would require changing production practices to reduce the residue or changing sludge application schedules to avoid spreading on grass or wheat stubble. Many times this is simply not feasible.

Soil moisture and temperature are not subject to control by the farmer or sludge operator, yet affect sludge management by preventing land application when field conditions are too wet or snow-covered. The farmer and sludge operator must accept these environmental conditions and be aware of their effect on ammonia volatilization so adjustments in supplemental nitrogen fertilizer can be made.

Winter conditions result in lower ammonia volatilization from the sludge. Incorporation of the sludge would not be possible due to the frozen ground and the soil would be wet, both of which would enhance maximum volatilizaton. However, the lower temperatures would reduce ammonia loss from the sludge and compensate for the effects of the other factors.

In the spring the sludge could be readily incorporated if applied ahead of tillage. Soil moisture would be high as would temperature, and both would enhance volatilization of ammonia from the sludge, but this would be effectively reduced by incorporation. There would be less sludge application on crop residue in the spring and ammonia loss would be minimized. The spring would be a season where ammonia volatilization is low or high depending on the awareness of both the farmer and sludge operator and the effort taken to avoid those conditions which favor ammonia volatilization.

In the summer, volatilization would tend to be high due to the high temperatures, but drier soil would help to reduce ammonia volatilization due to the rapid infiltration of sludge water (and ammonia present in the water). The important factor again would be sludge incoproration. In the summer, incorporation would not be as likely as in the spring or fall (due to farming practices), so volatilization would be higher.

In the fall, conditions would favor reduced ammonia volatilization. Sluge would be applied to dry soil and incorporation could be done before fall plowing and planting. However, temperatures would be warm and some crop residue would be present in the fields, both of which would increase volatilization loss. The reduced soil moisture and opportunity for sludge incorporation would result in a low to moderate ammonia volatilization for the fall period.

Whereas some of the variables studied, such as incorporation, may be subject to management control, others such as temperature and soil moisture are not. The manager of a sludge application operation and the farmer need to be aware of the magnitude of ammonia volatilization that can occur as a result of these factors and either attempt to minimize them through management or supplement with chemical fertilizer the total amount of nitrogen required by the crop.

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APPENDIX A

Experiment 1: Soil Moisture

Table 25. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for the different soil moistures. Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X†
1	ROW1	1	24.60
2	ROW2	1	16.35
3	ROW3	1	25.83
4	ROW4	1	25.26
5	ROW5	1	39.54
6	ROW6	1	28.05
7	ROW7	1	.
8	ROW8	1	.
9	ROW9	1	.
10	ROW10	1	.
11	ROW11	1	.
12	ROW12	1	.
13	ROW13	2	30.69
14	ROW14	2	15.03
15	ROW15	2	15.51
16	ROW16	2	25.86
17	ROW17	2	33.03
18	ROW18	2	34.05
19	ROW19	2	.
20	ROW20	2	.
21	ROW21	2	.
22	ROW22	2	.
23	ROW23	2	.
24	ROW24	2	.
25	ROW25	3	23.10
26	ROW26	3	13.65
27	ROW27	3	17.55

Table 25. (continued)

```
OBS     ROW      TREAT      X

28      ROW28      3      23.31
29      ROW29      3      62.49
30      ROW30      3      52.44
31      ROW31      3        .
32      ROW32      3        .
33      ROW33      3        .
34      ROW34      3        .
35      ROW35      3        .
36      ROW36      3        .
37      ROW37      4       7.32
38      ROW38      4       5.22
39      ROW39      4       6.00
40      ROW40      4      15.51
41      ROW41      4       8.64
42      ROW42      4       6.45
43      ROW43      4       5.97
44      ROW44      4       5.61
45      ROW45      4       5.52
46      ROW46      4       7.38
47      ROW47      4       6.69
48      ROW48      4       2.34
```

*Treatments: 1 = 0 atm. (32% H_2O); 2 = 0.1 atm. (22% H_2O); 3 = 15 atm. (10% H_2O); 4 = 31 atm. (air-dry, 6% H_2O.

+x = NH_3-N volatilized as percent of NH_3-N applied.

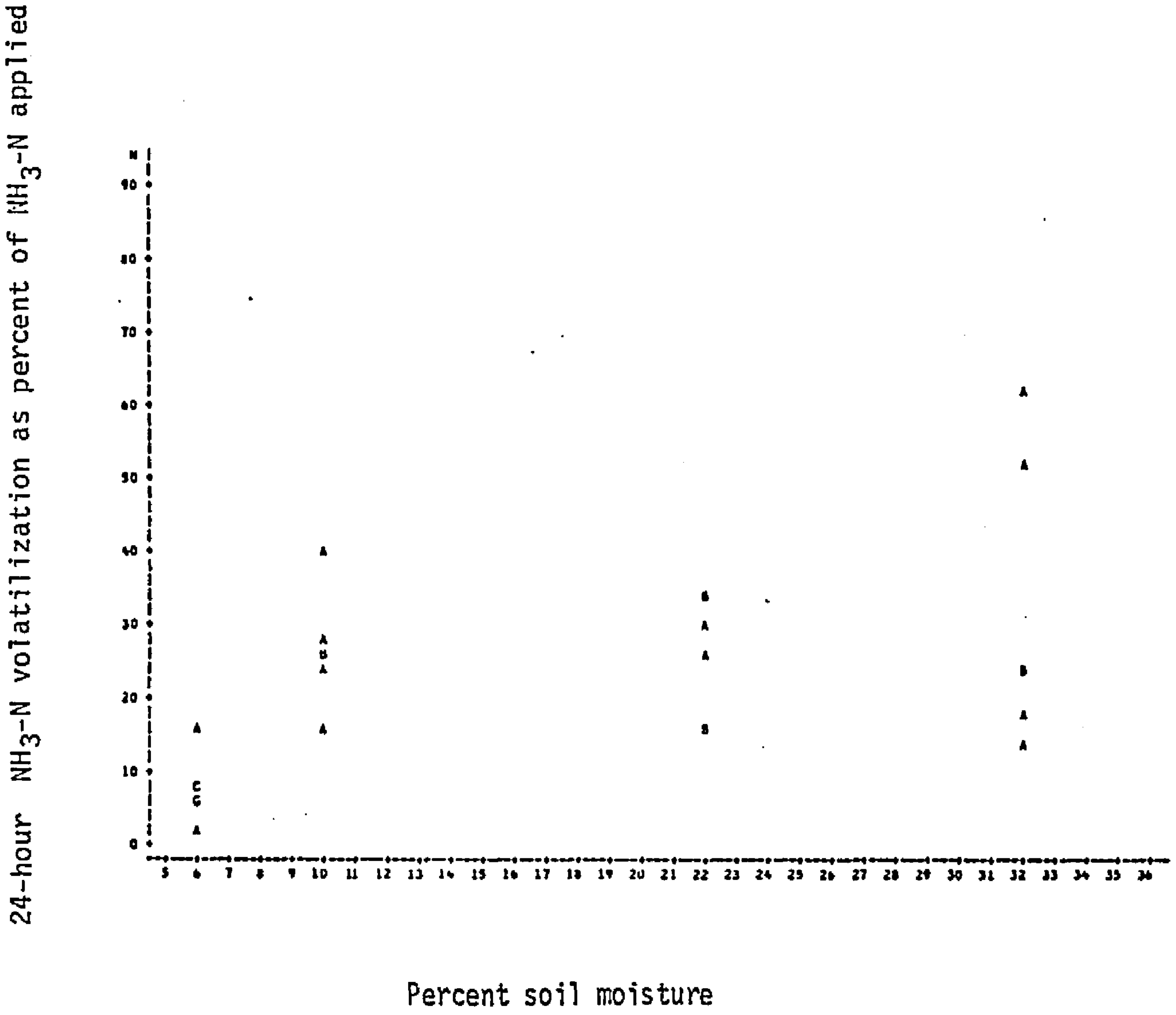

Figure 14. Replicate values of NH$_3$-N volatilized for each soil moisture treatment.

Legend: A = 1 observation; B = 2 observations; C = 3 observations; G = 7 observations.

Table 26. One-way analysis of variance for Experiment 1: Soil Moisture.

```
DEPENDENT VARIABLE: X

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     3         3392.57410500        1130.85803500          10.50          0.0001        0.547725        52.8703

ERROR                    26         2801.36572500         107.74483558                        STD DEV                         X MEAN

CORRECTED TOTAL          29         6193.93983000                                         10.38002098                    19.63300000

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF            TYPE IV SS      F VALUE      PR > F

TREAT                     3         3392.57410500        10.50      0.0001          3         3392.57410500        10.50      0.0001
```

Table 27. Regression analysis (linear) for Experiment 1: Soil Moisture.

```
DEPENDENT VARIABLE: N

SOURCE                  DF      SUM OF SQUARES       MEAN SQUARE      F VALUE        PR > F      R-SQUARE          C.V.

MODEL                    1       2302.44663846     2302.44663846        16.57        0.0003      0.371726       60.0472

ERROR                   28       3891.49319154      138.98189970                    STD DEV                      N MEAN

CORRECTED TOTAL         29       6193.93983000                                  11.78905847                 19.63300000

SOURCE                  DF          TYPE I SS    F VALUE    PR > F         DF      TYPE IV SS    F VALUE     PR > F

SOILMOIS                 1       2302.44663846      16.57    0.0003          1   2302.44663846      16.57     0.0003

                                  T FOR HO:      PR > |T|         STD ERROR OF
PARAMETER           ESTIMATE     PARAMETER=0                        ESTIMATE

INTERCEPT         6.63532012            1.72       0.0959           3.85102397
SOILMOIS          0.85511052            4.07       0.0003           0.21009058
```

Table 28. Regression analysis (quadratic) for Experiment 1: Soil Moisture

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES           MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     2         2625.06188185         1312.53094092           9.93          0.0006        0.423811        58.5595

ERROR                    27         3568.87794815          132.18066475                        STD DEV                         N MEAN

CORRECTED TOTAL          29         6193.93983000                                          11.49698503                    19.63300000

SOURCE                   DF             TYPE I SS      F VALUE       PR > F         DF            TYPE IV SS      F VALUE      PR > F

SOILMOIS                  1         2302.44663846        17.42       0.0003          1          660.70813998         5.00      0.0338
SOILMOIS*SOILMOIS         1          322.61524339         2.44       0.1299          1          322.61524339         2.44      0.1299

                                               T FOR H0:       PR > |T|           STD ERROR OF
PARAMETER                       ESTIMATE       PARAMETER=0                          ESTIMATE

INTERCEPT                    -4.86310829             -0.59       0.5611             6.26285615
SOILMOIS                      2.74964518              2.24       0.0338             1.22986033
SOILMOIS*SOILMOIS            -0.05146363             -1.56       0.1299             0.03295419
```

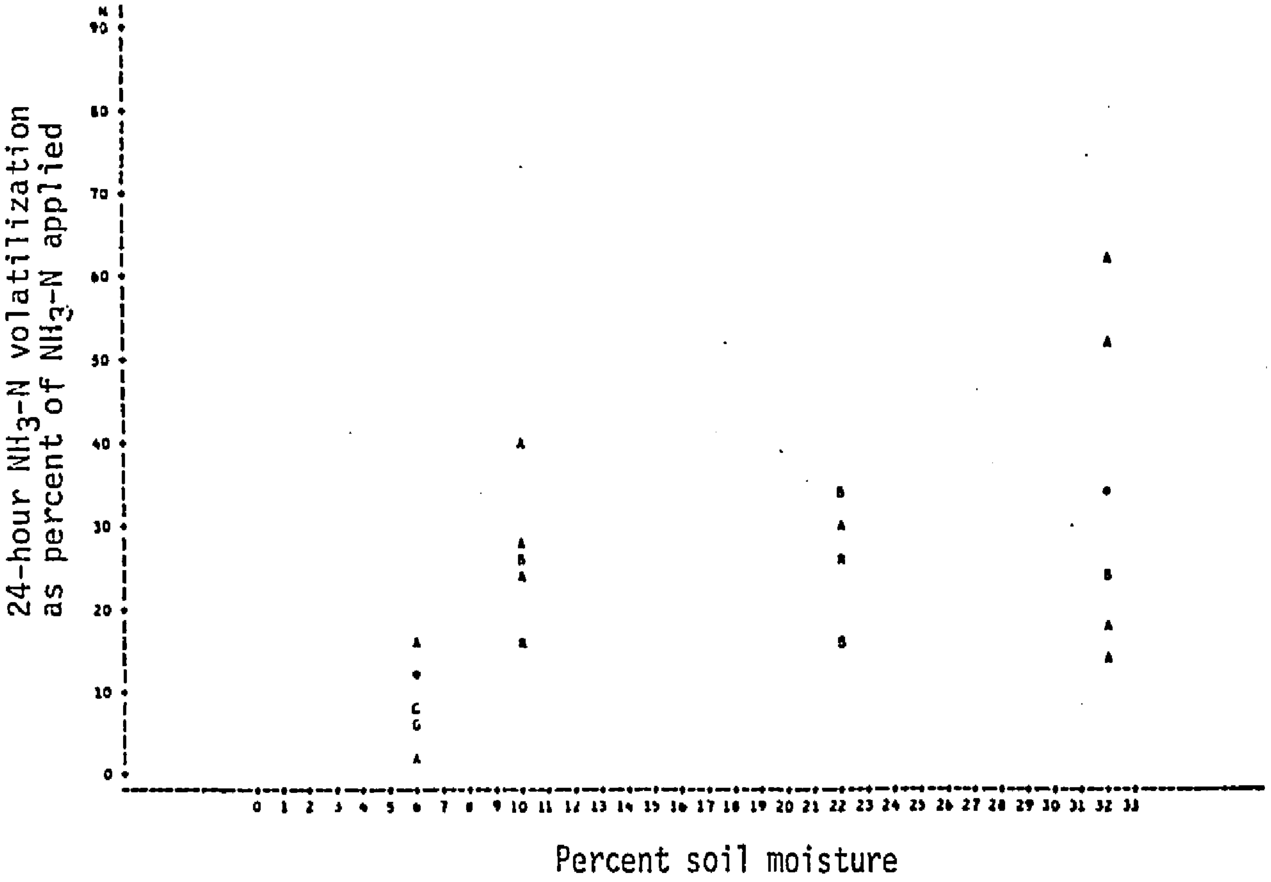

Figure 15. Predicted linear regression equation for Experiment 1: Soil Moisture.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

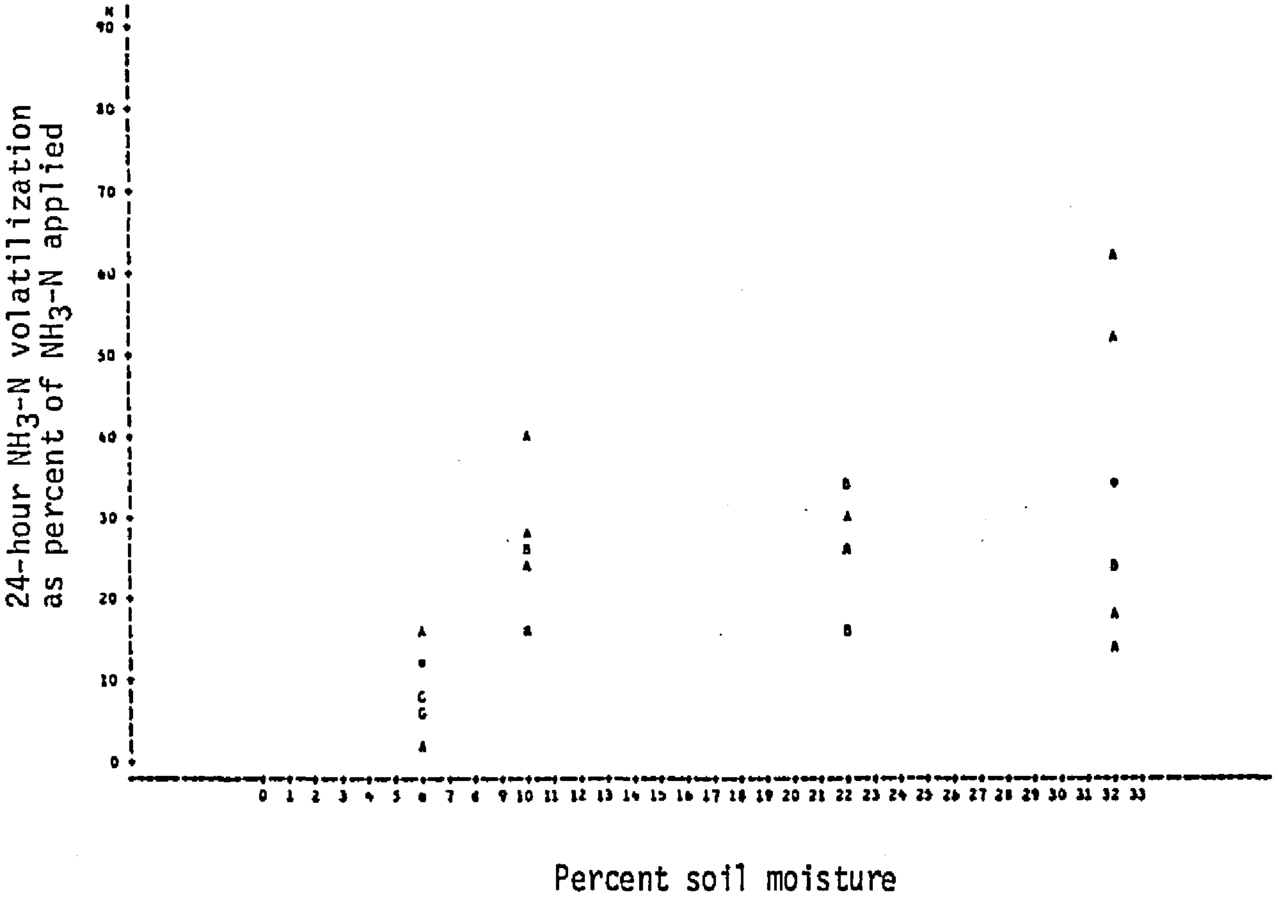

Figure 16. Predicted quadratic regression equation for Experiment 1: Soil Moisture.

Legend: A = 1 observation; B = 2 observations; C = 3 observations; G = 7 observations.

Table 29. Regression analysis (cubic) for Experiment 1: Soil Moisture.

```
DEPENDENT VARIABLE: N

SOURCE                   DF          SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     3           3392.57410500        1130.85803500          10.50          0.0001        0.547725        52.8703

ERROR                    26           2601.36572500         107.74483558                        STD DEV                         N MEAN

CORRECTED TOTAL          29           6193.93983000                                         10.38002098                    19.63300000

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF          TYPE IV SS      F VALUE      PR > F

SOILMOIS                  1         2302.44663846        21.37      0.0001          1       1078.09695324        10.01      0.0040
SOILMOIS*SOILMOIS         1          322.61524339         2.99      0.0954          1        864.84703811         8.03      0.0088
SOILMO*SOILMO*SOILMO      1          767.51222315         7.12      0.0129          1        767.51222315         7.12      0.0129

                                                  T FOR HO:      PR > |T|           STD ERROR OF
PARAMETER                          ESTIMATE      PARAMETER=0                          ESTIMATE

INTERCEPT                      -58.99096154            -2.73       0.0112            21.60896610
SOILMOIS                        15.40690476             3.16       0.0040             4.87062529
SOILMOIS*SOILMOIS               -0.81755420            -2.83       0.0088             0.28856598
SOILMO*SOILMO*SOILMO             0.01328233             2.67       0.0129             0.00497657
```

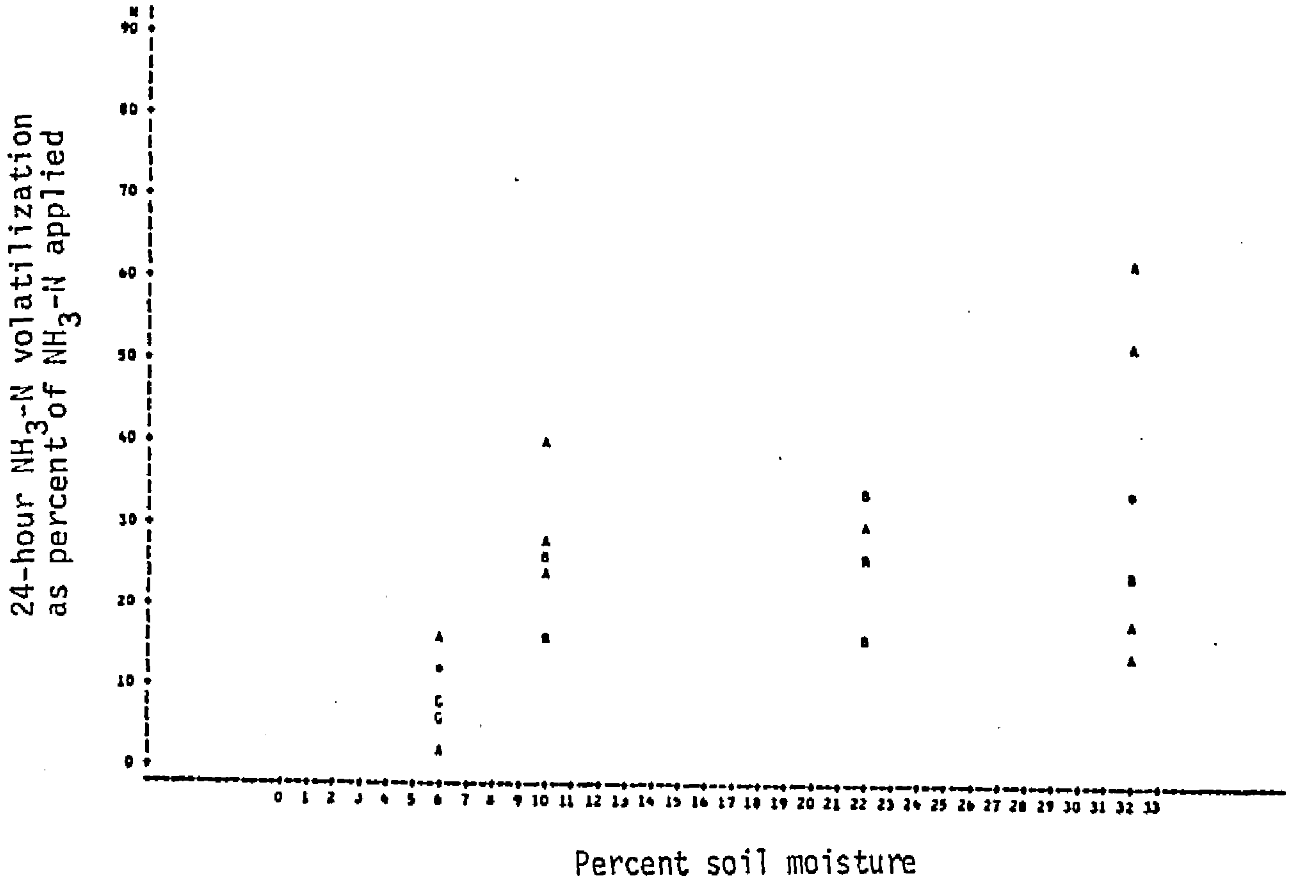

Figure 17. Predicted cubic regression equation for Experiment 1: Soil Moisture.

Legend: A = 1 observation; B = 2 observations; C = 3 observations; G = 7 observations.

APPENDIX B

Experiment 2: Time of Incorporation

Table 30. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replications for the different levels of incorporation. Calculated from the regression equation of percent volatilization versus time of sampling for each replicate

OBS	ROW	TREAT*	X
1	ROW1	1	1.44
2	ROW2	1	0.00
3	ROW3	1	2.19
4	ROW4	1	1.32
5	ROW5	1	1.74
6	ROW6	2	2.01
7	ROW7	2	0.00
8	ROW8	2	4.92
9	ROW9	2	8.85
10	ROW10	2	2.40
11	ROW11	3	7.44
12	ROW12	3	1.98
13	ROW13	3	5.34
14	ROW14	3	17.37
15	ROW15	3	2.43
16	ROW16	4	15.60
17	ROW17	4	8.10
18	ROW18	4	22.68
19	ROW19	4	17.16
20	ROW20	4	2.46
21	ROW21	5	10.62
22	ROW22	5	9.51
23	ROW23	5	10.47
24	ROW24	5	27.75
25	ROW25	5	4.44
26	ROW26	6	19.62
27	ROW27	6	15.03

Table 30. (continued)

OBS	ROW	TREAT	X
28	ROW28	6	33.87
29	ROW29	6	48.42
30	ROW30	6	3.90

*Treatments: 1 = incorporation initially or 0; 2 = incorporation 1 hour after sludge application; 3 = incorporation 3 hours after sludge application; 4 = incorporation 6 hours after sludge application; 5 = incorporation 12 hours after sludge application; and 6 = incorporation 24 hours after sludge application.

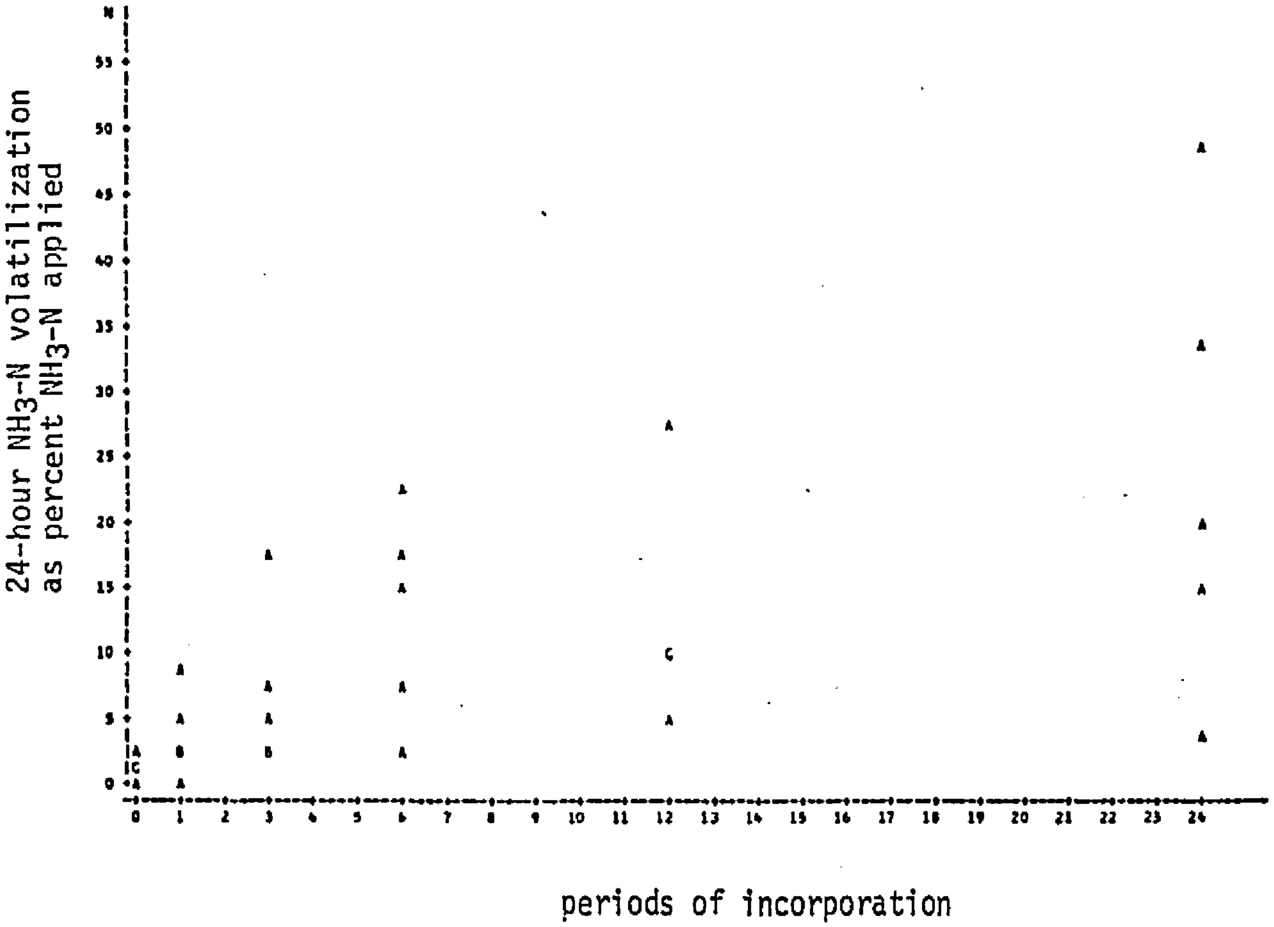

Figure 18. Replicate values of NH_3-N volatilized for each period of incorporation.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 31. One-way analysis of variance for Experiment 2: Different periods of Incorporation.

```
DEPENDENT VARIABLE: X

SOURCE              DF      SUM OF SQUARES       MEAN SQUARE       F VALUE       PR > F       R-SQUARE          C.V.

MODEL                5      1710.17424000       342.03484800          4.17       0.0072       0.464764       87.9325

ERROR               24      1969.48944000        82.06206000                     STD DEV                     X MEAN

CORRECTED TOTAL     29      3679.66368000                                     9.05881118                 10.30200000

SOURCE              DF         TYPE I SS     F VALUE    PR > F      DF        TYPE IV SS      F VALUE       PR > F

TREAT                5      1710.17424000       4.17    0.0072       5     1710.17424000         4.17       0.0072
```

Table 32. Regression analysis (linear) for Experiment 2: Sludge Incorporation.

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     1         1571.29749755        1571.29749755          20.87          0.0001        0.427022        84.2311

ERROR                    28         2106.36618245          75.29879223                        STD DEV                         N MEAN

CORRECTED TOTAL          29         3679.66368000                                          8.67748767                    10.30200000

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF            TYPE IV SS      F VALUE      PR > F

INC                       1         1571.29749755        20.87      0.0001          1         1571.29749755        20.87      0.0001

                                       T FOR H0:      PR > |T|           STD ERROR OF
PARAMETER               ESTIMATE      PARAMETER=0                          ESTIMATE

INTERCEPT             3.61701290             1.68       0.1047             2.15673878
INC                   0.87195484             4.57       0.0001             0.19087928
```

Table 33. Regression analysis (quadratic) for Experiment 2: Sludge Incorporation.

```
DEPENDENT VARIABLE: N

SOURCE                   DF         SUM OF SQUARES          MEAN SQUARE        F VALUE           PR > F        R-SQUARE           C.V.

MODEL                     2          1592.29810838         796.14905419          10.30           0.0005        0.432729        85.3485

ERROR                    27          2067.36557162          77.30983599                         STD DEV                        N MEAN

CORRECTED TOTAL          29          3679.66368000                                           8.79260121                   10.30200000

SOURCE                   DF              TYPE I SS      F VALUE      PR > F          DF             TYPE IV SS      F VALUE      PR > F

INC                       1          1571.29749755        20.32      0.0001           1           221.63124570         2.87      0.1019
INC*INC                   1            21.00061083         0.27      0.6065           1            21.00061083         0.27      0.6065

                                         T FOR H0:     PR > |T|           STD ERROR OF
PARAMETER               ESTIMATE       PARAMETER=0                          ESTIMATE

INTERCEPT             2.75211503              1.00       0.3248             2.74400316
INC                   1.24015174              1.69       0.1019             0.73244785
INC*INC              -0.01533638             -0.52       0.6065             0.02942554
```

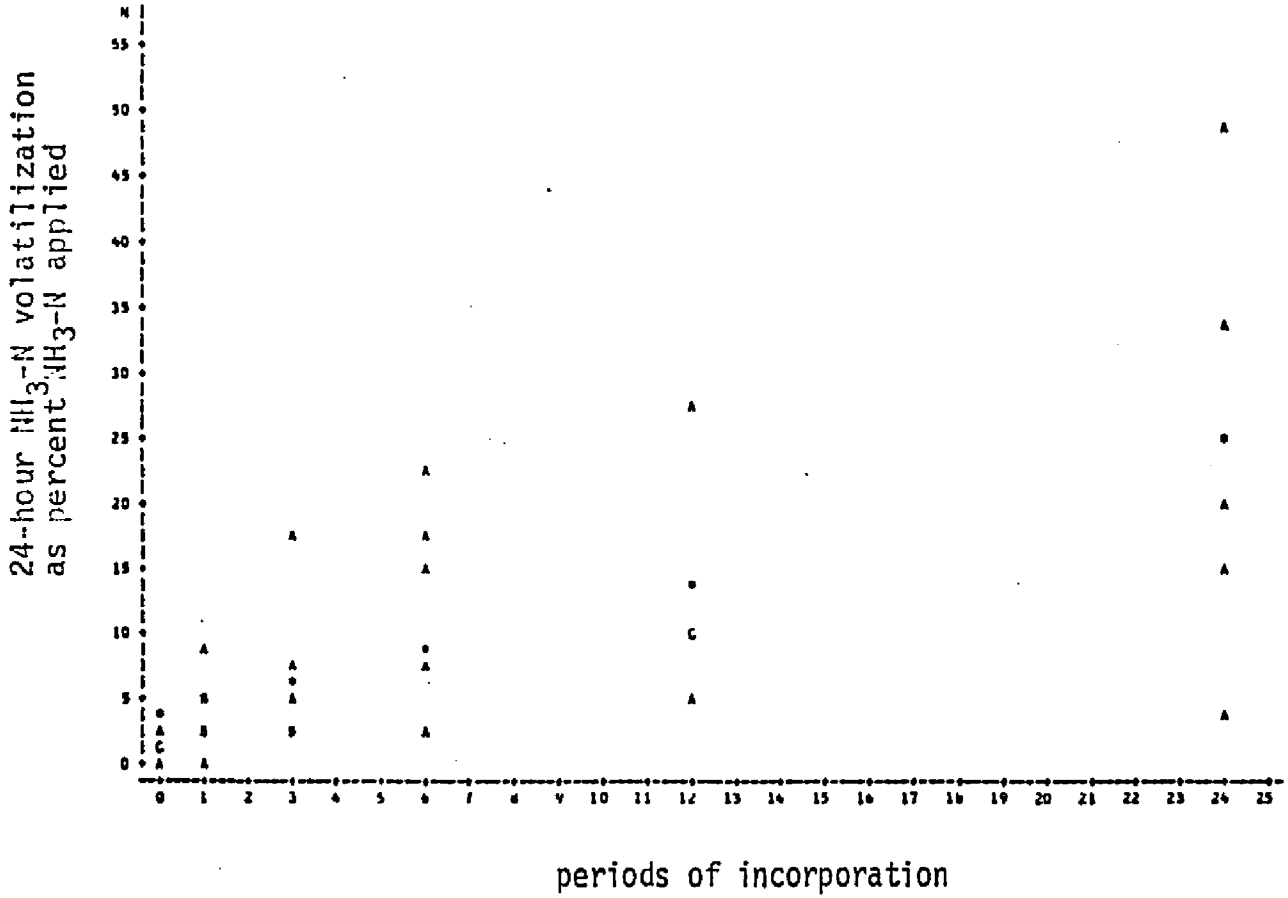

Figure 19. Predicted linear regression equation for Experiment 2: Sludge Incorporation.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

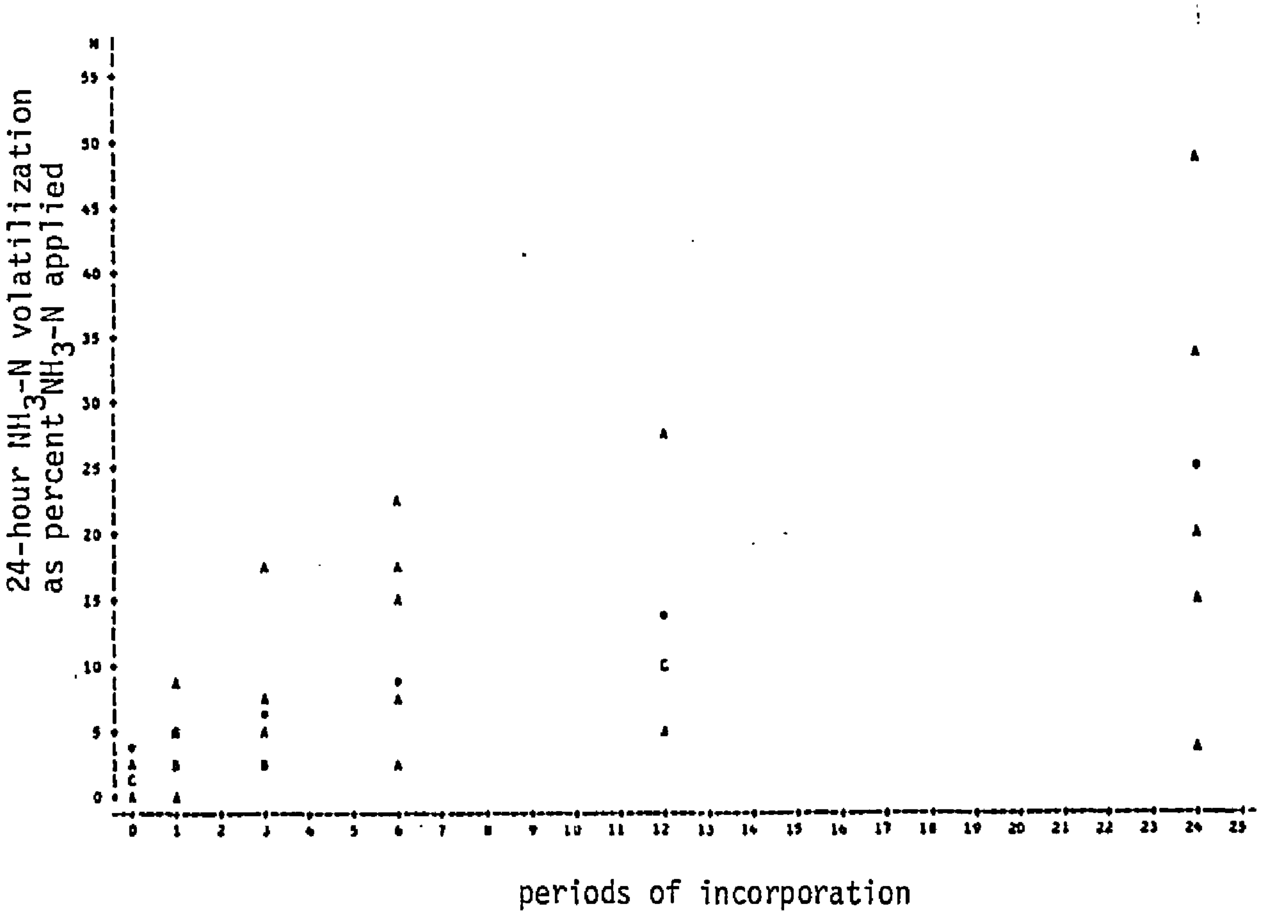

Figure 20. Predicted quadratic regression equation for Experiment 2: Sludge Incorporation.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 34. Regression analysis (cubic) for Experiment 2: Sludge Incorporation.

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     3         1690.93526113         563.64508704           7.37          0.0010        0.459535        84.8945

ERROR                    26         1988.72841887          76.48955457                        STD DEV                         N MEAN

CORRECTED TOTAL          29         3679.66368000                                          8.74583070                    10.30200000

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF            TYPE IV SS      F VALUE      PR > F

INC                       1         1571.29749755        20.54      0.0001          1          219.40440335         2.87      0.1023
INC*INC                   1           21.00061083         0.27      0.6047          1          108.63310789         1.42      0.2441
INC*INC*INC               1           98.63715276         1.29      0.2665          1           98.63715276         1.29      0.2665

                                         T FOR HO:      PR > |T|           STD ERROR OF
PARAMETER                 ESTIMATE      PARAMETER=0                          ESTIMATE

INTERCEPT               0.78387659             0.24       0.8103             3.23323068
INC                     3.27216106             1.69       0.1023             1.93202647
INC*INC                -0.28163609            -1.19       0.2441             0.23632592
INC*INC*INC             0.00774399             1.14       0.2665             0.00681939
```

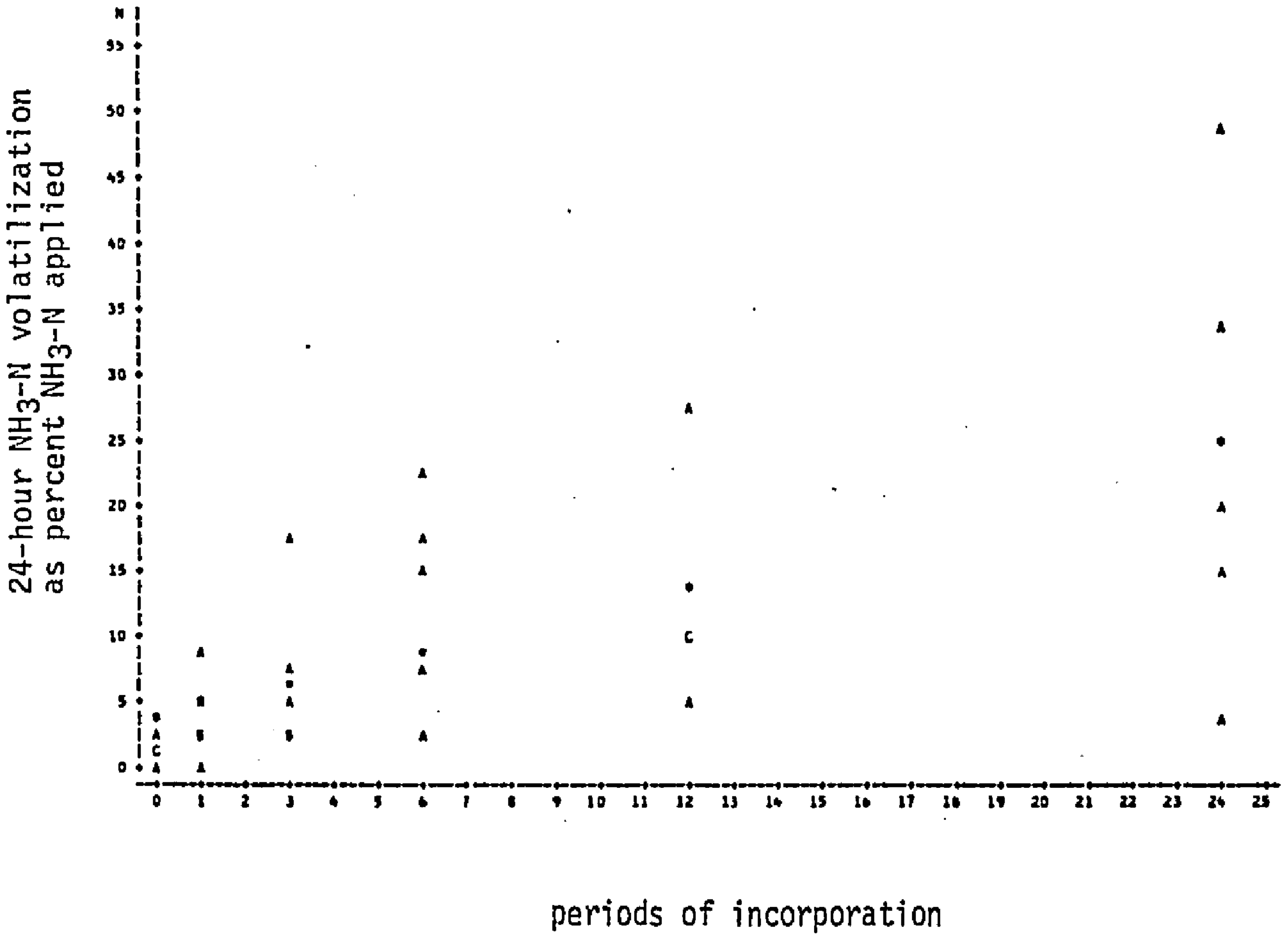

Figure 21. Predicted quadratic regression equation for Experiment 2: Sludge Incorporation.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

APPENDIX C

Experiment 3: Soil pH

Table 35. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for the different soil pH's. Calculated from the regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT *	X
1	ROW1	1	20.67
2	ROW2	1	9.76
3	ROW3	1	11.43
4	ROW4	1	15.96
5	ROW5	1	16.65
6	ROW6	1	11.64
7	ROW7	2	17.79
8	ROW8	2	13.11
9	ROW9	2	13.11
10	ROW10	2	13.14
11	ROW11	2	16.26
12	ROW12	2	16.41
13	ROW13	3	20.58
14	ROW14	3	.
15	ROW15	3	.
16	ROW16	3	20.52
17	ROW17	3	18.09
18	ROW18	3	21.30

*Treatments: 1 = soil pH 5.1; 2 = soil pH 6.7; 3 = soil pH 7.5.

Table 36. One-way analysis of variance for Experiment 3: Soil pH.

```
DEPENDENT VARIABLE: X

SOURCE                  DF        SUM OF SQUARES         MEAN SQUARE       F VALUE          PR > F       R-SQUARE           C.V.

MODEL                    2           90.56947500         45.28473750          5.24          0.0215       0.446172        18.3480

ERROR                   13          112.42282500          8.64790962                       STD DEV                        X MEAN

CORRECTED TOTAL         15          202.99230000                                        2.94073254                   16.02750000

SOURCE                  DF           TYPE I SS     F VALUE      PR > F          DF          TYPE IV SS      F VALUE       PR > F

TREAT                    2           90.56947500      5.24      0.0215           2          90.56947500         5.24       0.0215
```

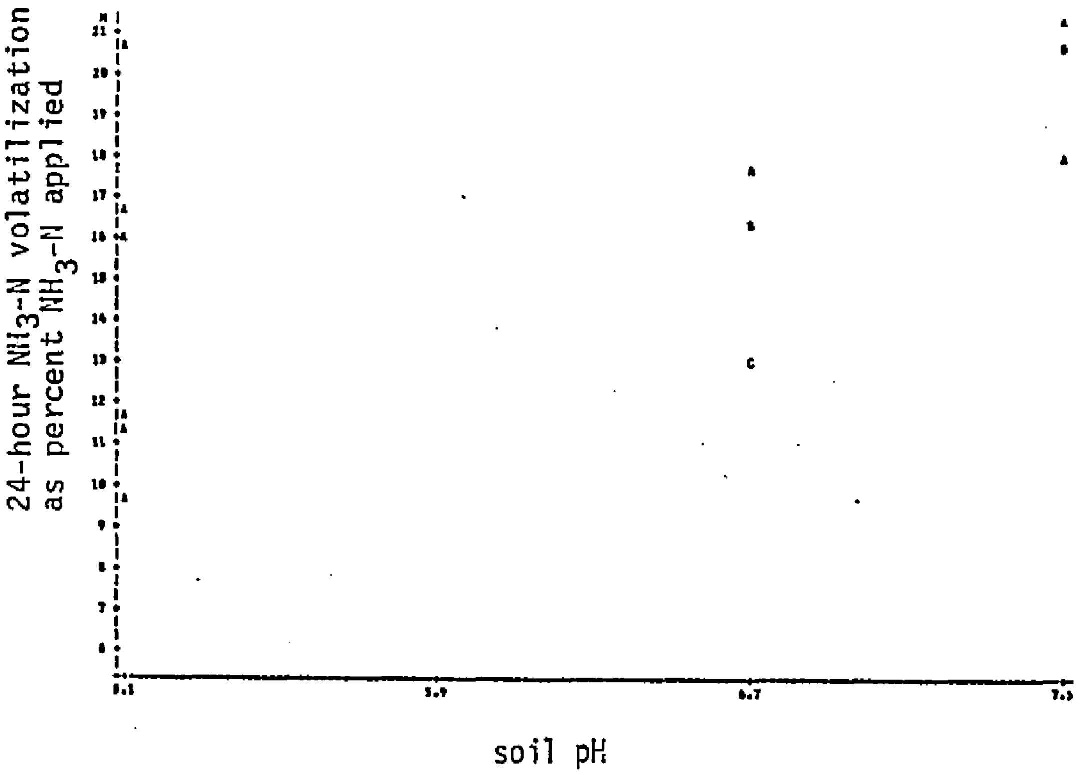

Figure 23. Replicate values of NH_3-N volatilized for each soil pH of 5.1, 6.7, and 7.5.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 37. Regression analysis (linear) for Experiment 3: Soil pH.

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F       R-SQUARE           C.V.

MODEL                     1           61.12991748          61.12991748           6.03          0.0277       0.301144        19.8611

ERROR                    14          141.86238252          10.13302732                        STD DEV                        N MEAN

CORRECTED TOTAL          15          202.99230000                                          3.18324164                   16.02750000

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF             TYPE IV SS         F VALUE      PR > F

PH                        1           61.12991748          6.03      0.0277          1           61.12991748            6.03      0.0277

                                       T FOR HO:      PR > |T|           STD ERROR OF
PARAMETER               ESTIMATE      PARAMETER=0                            ESTIMATE

INTERCEPT             2.65115044             0.48    0.6375            5.50387096
PH                    2.12323009             2.46    0.0277            0.86444977
```

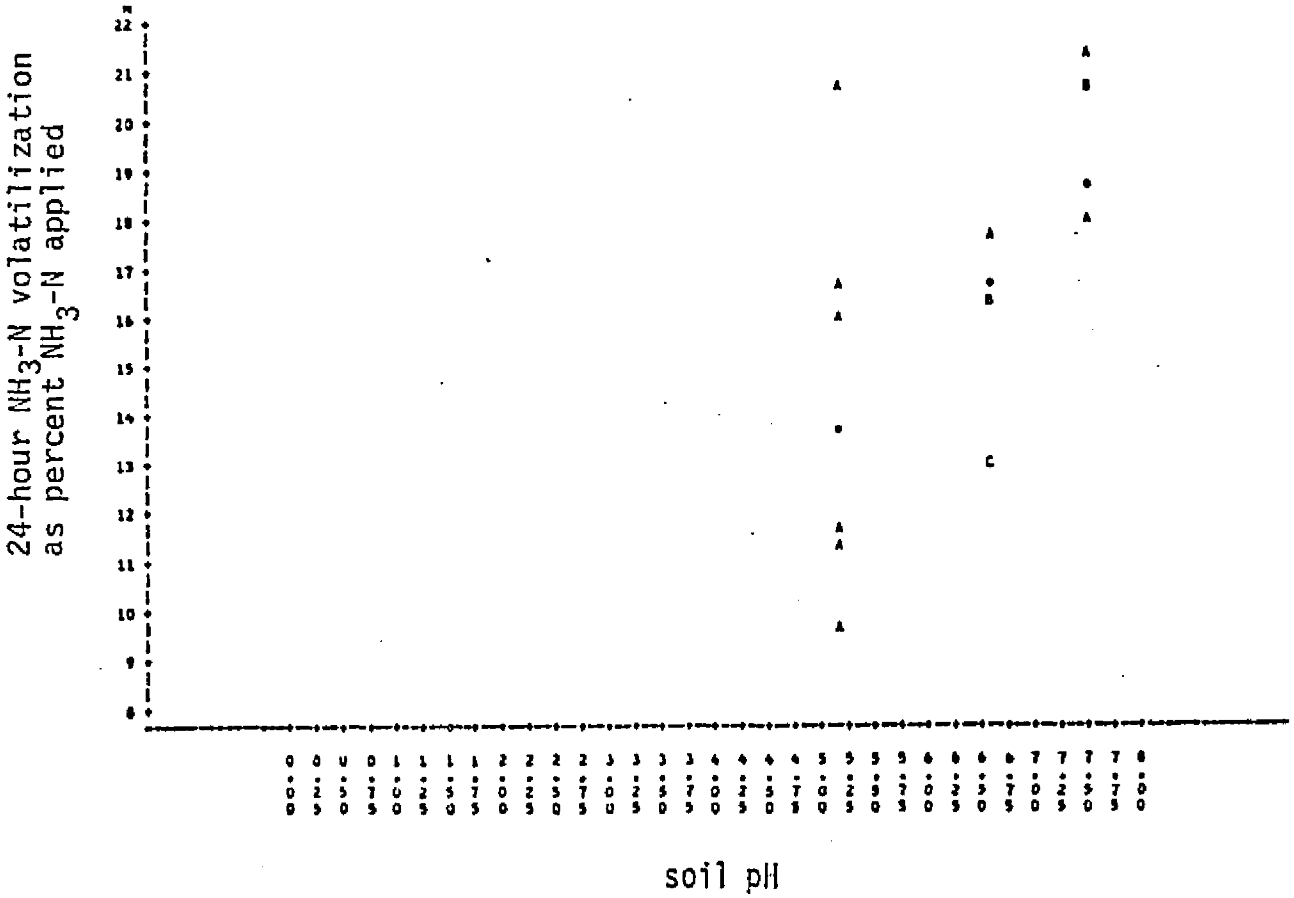

Figure 24. Predicted linear regression equation for Experiment 3: soil pH's of 5.1, 6.7, and 7.5.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 38. Regression analysis (quadratic) for Experiment 3: Soil pH.

```
DEPENDENT VARIABLE: N

SOURCE                DF      SUM OF SQUARES          MEAN SQUARE        F VALUE           PR > F        R-SQUARE          C.V.

MODEL                  2         90.56947500          45.28473750           5.24           0.0215        0.446172       18.3480

ERROR                 13        112.42282500           8.64790962                          STD DEV                       N MEAN

CORRECTED TOTAL       15        202.99230000                                            2.94073284                  16.02750000

SOURCE                DF         TYPE I SS      F VALUE      PR > F          DF          TYPE IV SS      F VALUE        PR > F

PH                     1       61.12991748         7.07      0.0197           1         25.19314342         2.91        0.1116
PH*PH                  1       29.43955752         3.40      0.0879           1         29.43955752         3.40        0.0879

                                    T FOR H0:     PR > |T|            STD ERROR OF
PARAMETER            ESTIMATE     PARAMETER=0                            ESTIMATE

INTERCEPT         90.94276396            1.89       0.0813             48.12239326
PH               -26.67872671           -1.71       0.1116             15.63073822
PH*PH              2.29813665            1.85       0.0879              1.24556323
```

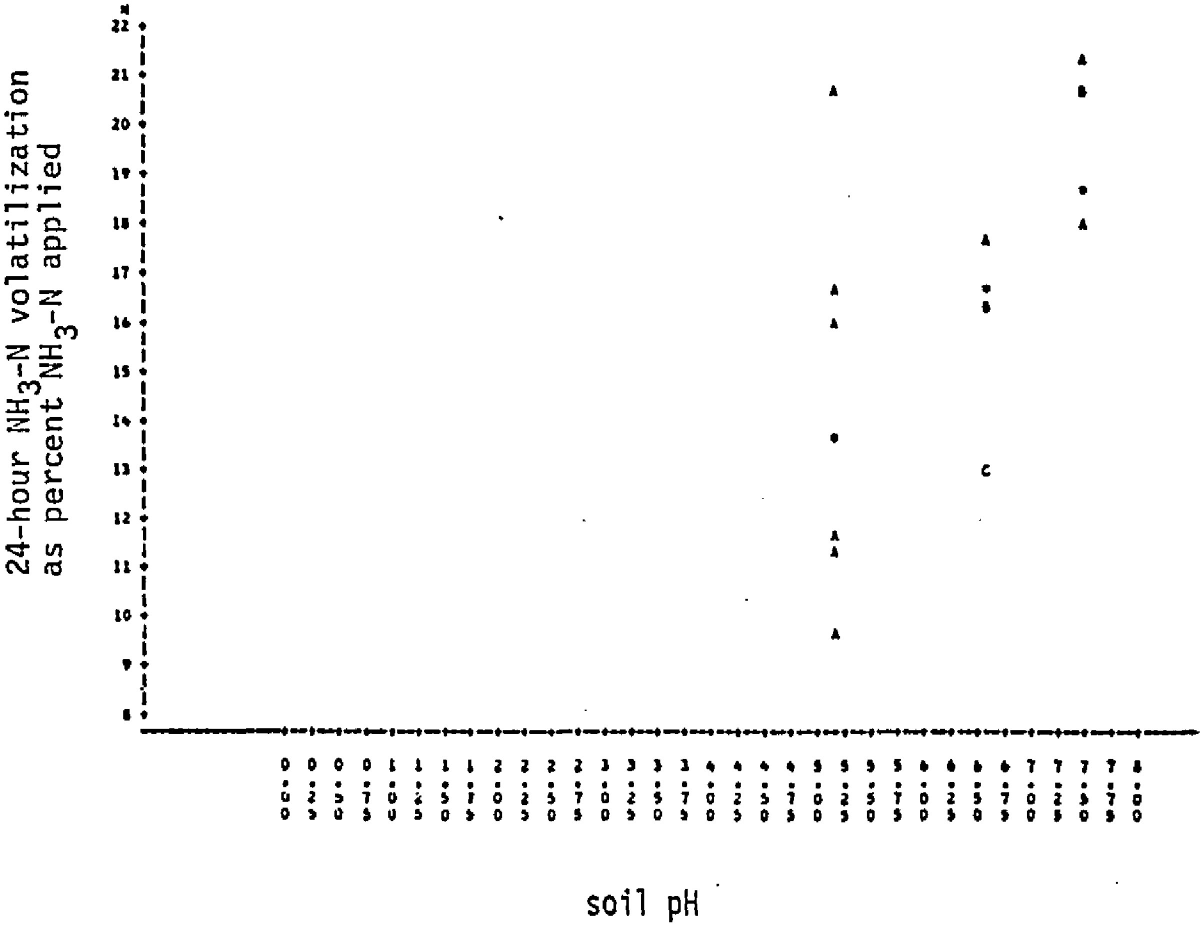

Figure 25. Predicted regression equation for Experiment 3: Soil pH.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

APPENDIX D

Experiment 4: Sludge Type

Table 39. The 24-hour NH_3-N volatilization as percent NH_3-N applied by replication for the different sludge types. Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X
1	ROW1	1	15.39
2	ROW2	1	36.12
3	ROW3	1	17.94
4	ROW4	2	4.71
5	ROW5	2	0.00
6	ROW6	2	0.00
7	ROW7	3	0.00
8	ROW8	3	0.00
9	ROW9	3	0.00
10	ROW10	4	16.11
11	ROW11	4	14.34
12	ROW12	4	45.09
13	ROW13	5	29.43
14	ROW14	5	65.91
15	ROW15	5	61.71

*Treatments: 5 = Ashland primary lime-stabilized sludge; 4 = Columbus anaerobically digested liquid sludge; 3 = Columbus compost made from primary sludge; 2 = Medina aerobically digested sludge; 1 = Dewatered Columbus anaerobically digested sludge.

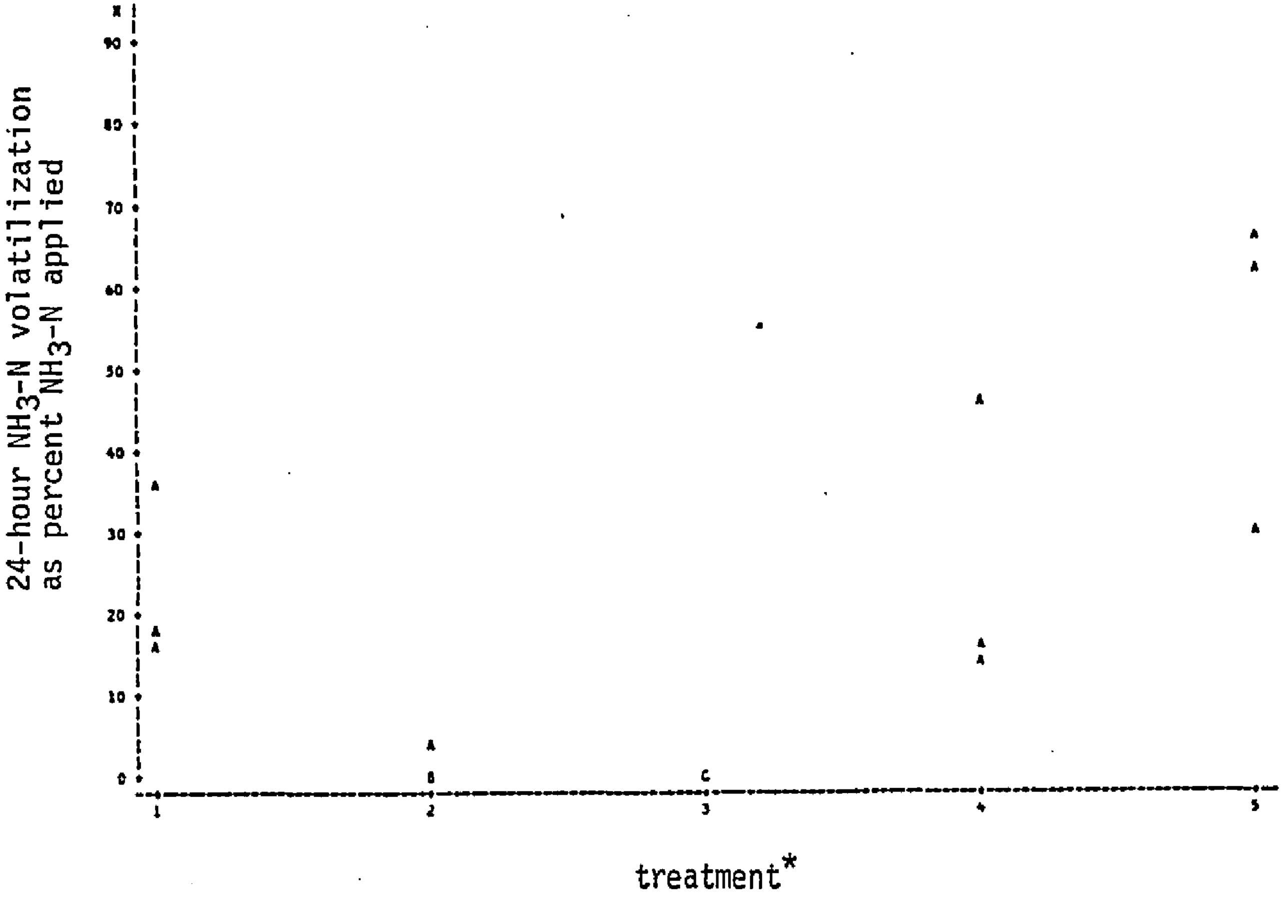

Figure 26. Replicate values of NH_3-N volatilized for the five different sludge types.

Treatment: 1 = Dewatered Columbus anaerobically digested sludge; 2 = Medina aerobically digested sludge; 3 = Columbus compost made from primary sludge; 4 = Columbus anaerobically digested liquid sludge; and 5 = Ashland primary lime-stabilized sludge.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 40. One-way analysis of variance for Experiment 4: Different Sludge Types.

```
DEPENDENT VARIABLE: X

SOURCE                  DF      SUM OF SQUARES        MEAN SQUARE       F VALUE          PR > F      R-SQUARE           C.V.

MODEL                    4      5465.78940000      1366.44735000          8.21          0.0033      0.766682        63.0667

ERROR                   10      1663.36020000       166.33602000                        STD DEV                     X MEAN

CORRECTED TOTAL         14      7129.14960000                                       12.89713224                20.45000000

SOURCE                  DF         TYPE I SS      F VALUE     PR > F        DF        TYPE IV SS      F VALUE       PR > F

TREAT                    4      5465.78940000        8.21     0.0033         4     5465.78940000        8.21       0.0033
```

APPENDIX E

Experiment 5: Temperature

Table 41. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for the different temperatures. Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X
1	ROW1	1	4.32
2	ROW2	1	1.65
3	ROW3	1	0.15
4	ROW4	1	3.30
5	ROW5	1	.
6	ROW6	1	4.83
7	ROW7	2	9.66
8	ROW8	2	10.50
9	ROW9	2	10.56
10	ROW10	2	7.20
11	ROW11	2	10.53
12	ROW12	2	11.13
13	ROW13	3	23.85
14	ROW14	3	12.72
15	ROW15	3	12.18
16	ROW16	3	9.12
17	ROW17	3	10.95
18	ROW18	3	10.02

---

*Treatments: 1 = temperature of 12.8°C; 2 = temperature of 18.3°C; and 3 = temperature of 26.7°C.

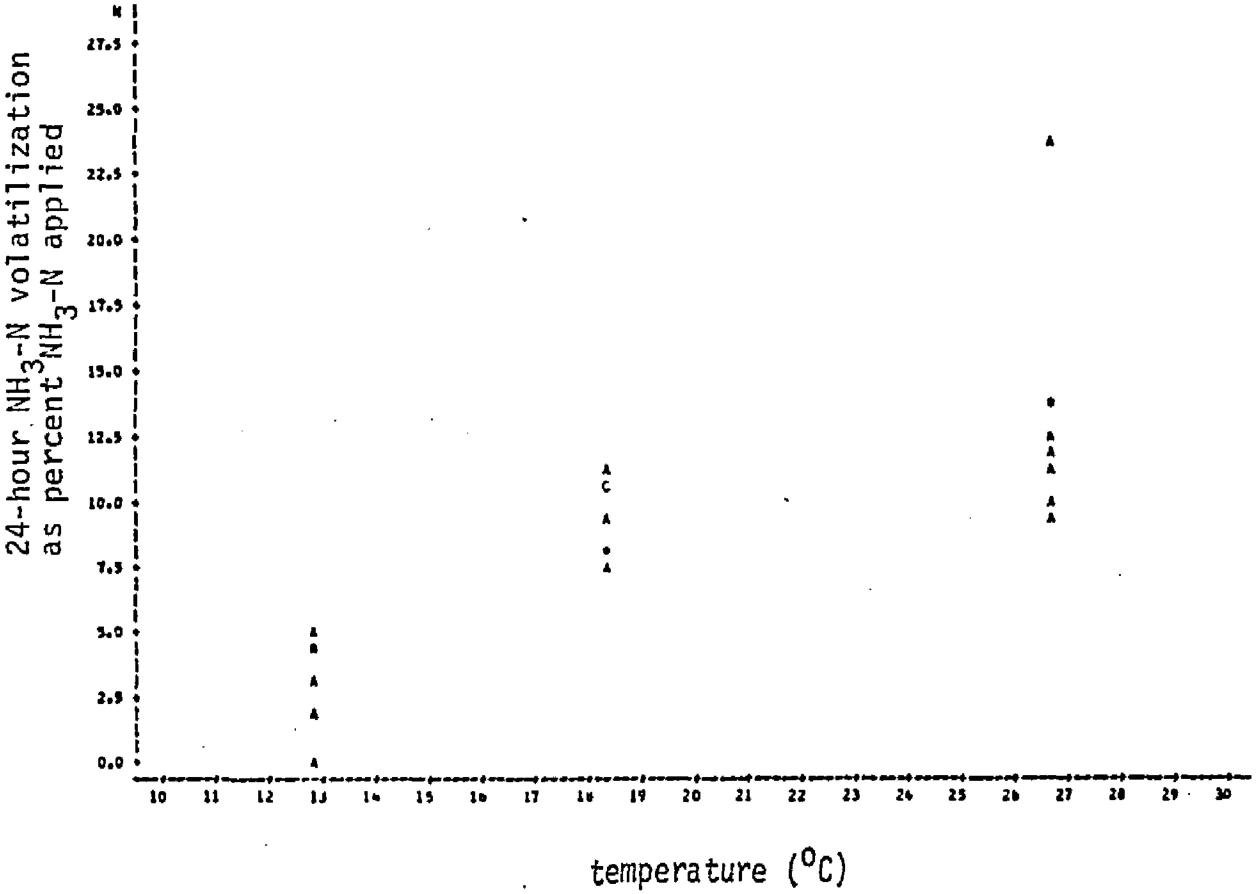

Figure 27. Replicate values of NH_3-N volatilized for temperatures of 12.8, 18.3, and 26.7°C.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 42. One-way analysis of variance for Experiment 5: Temperatures of 12.8, 18.3, and 26.7°C.

```
DEPENDENT VARIABLE: X

SOURCE                DF       SUM OF SQUARES       MEAN SQUARE      F VALUE       PR > F     R-SQUARE         C.V.

MODEL                  2         297.13309412      148.56654706        12.12       0.0009     0.633968      38.9791

ERROR                 14         171.55440000       12.25388571                   STD DEV                    X MEAN

CORRECTED TOTAL       16         468.68749412                                  3.50055506                8.98058824

SOURCE                DF            TYPE I SS     F VALUE     PR > F        DF         TYPE IV SS     F VALUE     PR > F

TREAT                  2         297.13309412       12.12     0.0009         2       297.13309412       12.12     0.0009
```

Table 43. Regression analysis (linear) for Experiment 5: Temperatures of 12.8, 18.3, and 26.7°C.

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE        F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     1          263.08285288         263.08285288          19.19          0.0005        0.561318        41.2255

ERROR                    15          205.60464124          13.70697608                        STD DEV                         N MEAN

CORRECTED TOTAL          16          468.68749412                                           3.70229335                     8.98058824

SOURCE                   DF             TYPE I SS      F VALUE      PR > F         DF             TYPE IV SS      F VALUE     PR > F

TEMP                      1          263.08285288        19.19      0.0005          1           263.08285288        19.19     0.0005

                                       T FOR H0:      PR > |T|           STD ERROR OF
PARAMETER               ESTIMATE      PARAMETER=0                          ESTIMATE

INTERCEPT            -4.66534355            -1.44       0.1695             3.24601658
TEMP                  0.69557138             4.38       0.0005             0.15876920
```

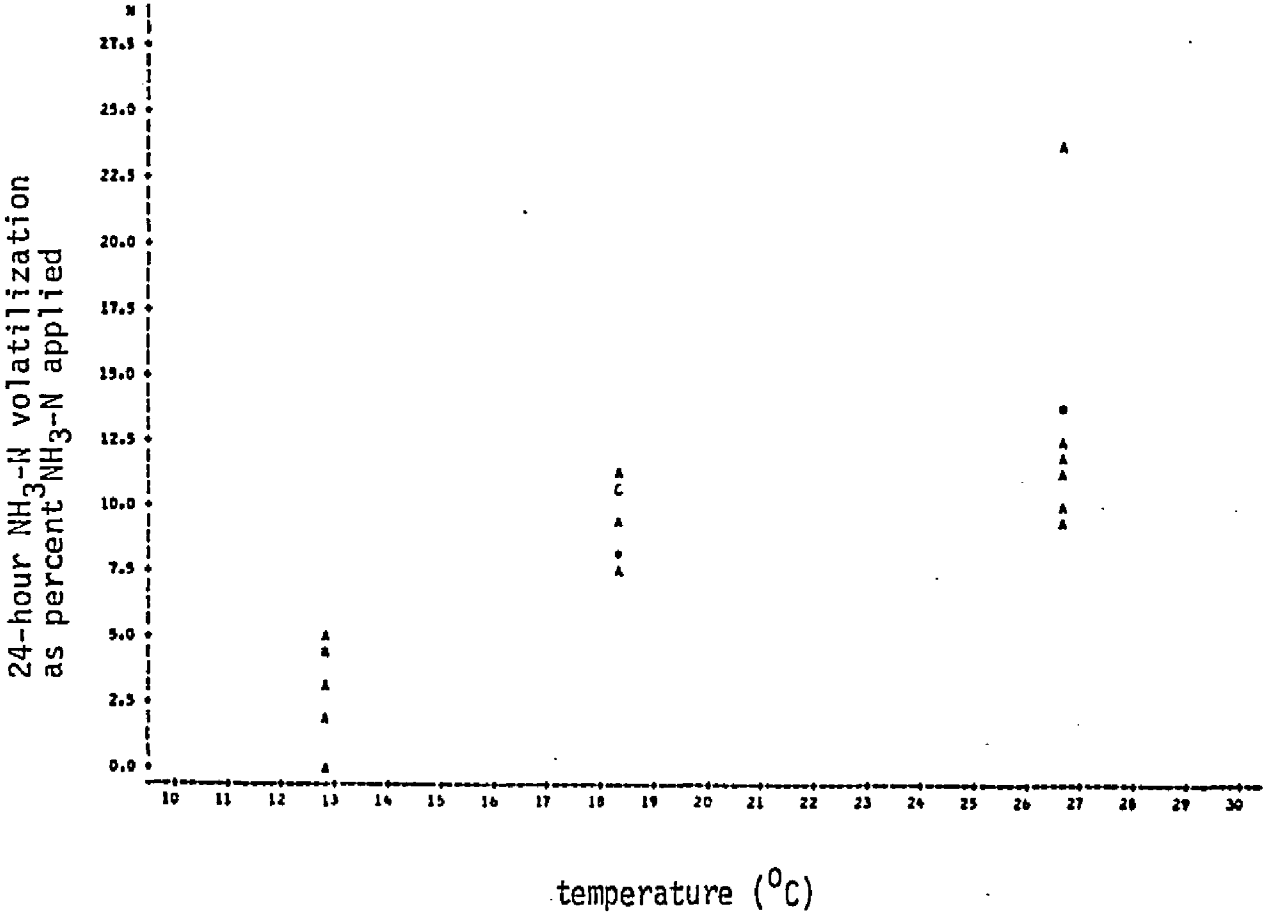

Figure 29. Predicted quadratic regression equation for Experiment 5: Temperatures of 12.8, 18.3, and 26.7°C.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 44. Regression analysis (quadratic) for Experiment 5: Temperatures of 12.8, 18.3, and 26.7°C.

```
DEPENDENT VARIABLE: N

SOURCE                   DF        SUM OF SQUARES          MEAN SQUARE       F VALUE          PR > F        R-SQUARE           C.V.

MODEL                     2          297.13309412         148.56654706         12.12          0.0009        0.633968        38.9791

ERROR                    14          171.55440000          12.25388571                       STD DEV                         N MEAN

CORRECTED TOTAL          16          468.68749412                                         3.50055506                     8.98058824

SOURCE                   DF             TYPE I SS      F VALUE      PR > F        DF           TYPE IV SS      F VALUE      PR > F

TEMP                      1          263.08285288        21.47      0.0004         1          54.06304781         4.41      0.0543
TEMP*TEMP                 1           34.05024124         2.78      0.1177         1          34.05024124         2.78      0.1177

                                       T FOR H0:      PR > |T|          STD ERROR OF
PARAMETER               ESTIMATE      PARAMETER=0                          ESTIMATE

INTERCEPT           -28.88015715            -1.95       0.0719           14.83534434
TEMP                  3.31241942             2.10       0.0543            1.57700101
TEMP*TEMP            -0.06511726            -1.67       0.1177            0.03906366
```

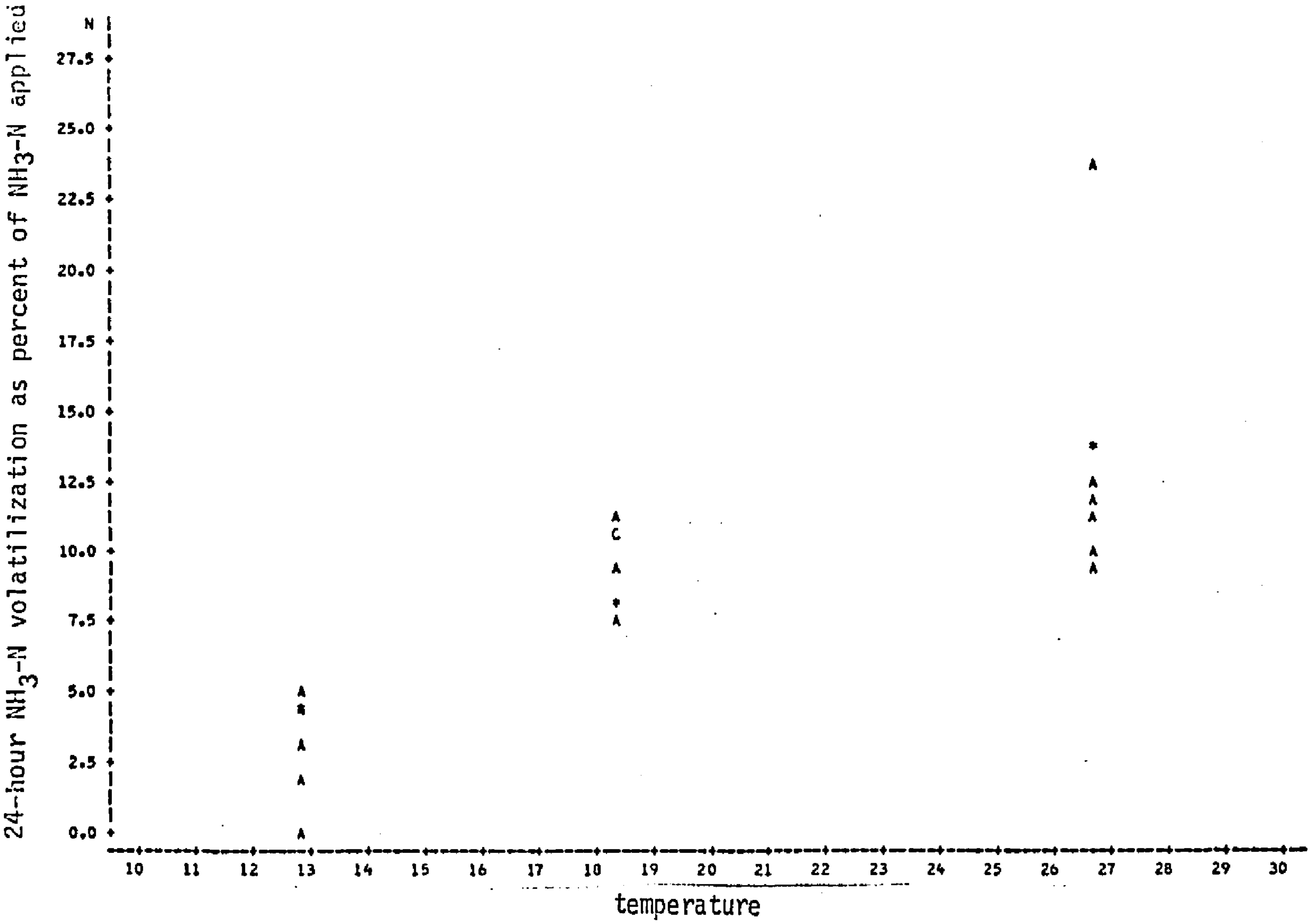

Figure 29. Predicted quadratic regression equation for Experiment 5: Temperatures of 12.8, 18.3, and 26.7°C.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

APPENDIX F

Experiment 6: Vegetative Cover

Table 45. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for vegetative cover (large sludge particles). Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X
1	ROW1	1	11.22
2	ROW2	1	7.95
3	ROW3	1	14.94
4	ROW4	1	14.16
5	ROW5	1	32.82
6	ROW6	1	13.80
7	ROW7	1	14.40
8	ROW8	1	4.17
9	ROW9	2	8.25
10	ROW10	2	10.77
11	ROW11	2	12.75
12	ROW12	2	13.47
13	ROW13	2	20.88
14	ROW14	2	12.54
15	ROW15	2	7.56
16	ROW16	2	3.09
17	ROW17	3	3.24
18	ROW18	3	5.82
19	ROW19	3	9.27
20	ROW20	3	10.41
21	ROW21	3	8.40
22	ROW22	3	2.91
23	ROW23	3	9.30
24	ROW24	3	2.13

*Treatments: 1 = wheat straw; 2 = Kentucky blue grass sod; 3 = bare soil.

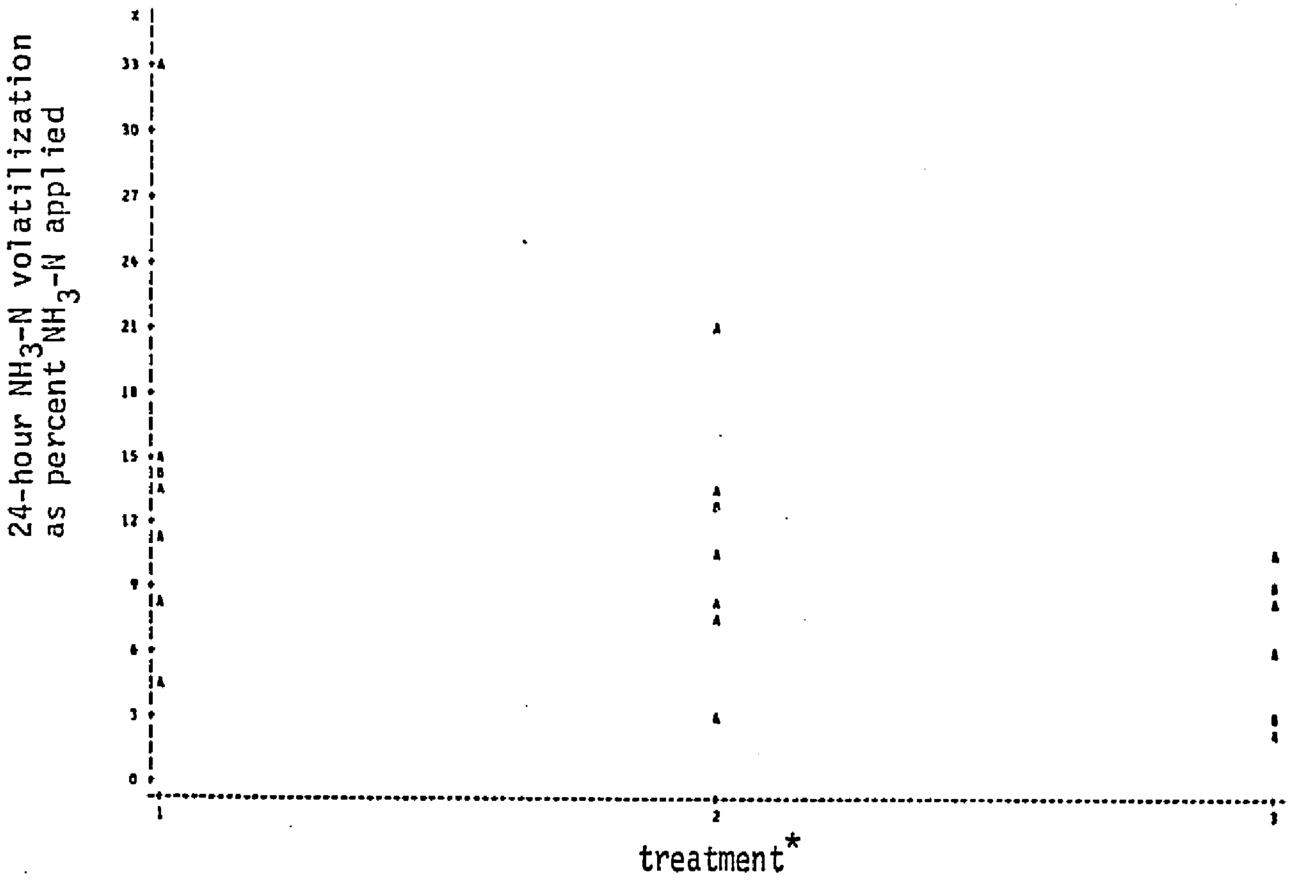

Figure 30. Replicate values of NH_3-N volatilized for each treatment in Experiment 6: Vegetative Cover (Large Sludge Particles).
Treatment: 1 = wheat straw; 2 = Kentucky blue grass sod; 3 = bare soil.
Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 46. One-way analysis of variance for Experiment 6: Vegetative Cover (large sludge particles).

```
DEPENDENT VARIABLE: X

SOURCE                 DF       SUM OF SQUARES          MEAN SQUARE        F VALUE         PR > F       R-SQUARE          C.V.

MODEL                   2         243.99382500         121.99691250           3.35         0.0545       0.241963       56.9509

ERROR                  21         764.39913750          36.39995893                       STD DEV                       X MEAN

CORRECTED TOTAL        23        1008.39296250                                         6.03323785                  10.59375000

SOURCE                 DF           TYPE I SS      F VALUE     PR > F           DF         TYPE IV SS      F VALUE      PR > F

TREAT                   2        243.99382500         3.35     0.0545            2       243.99382500         3.35      0.0545
```

Table 47. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for vegetative cover (liquid or homogenous sludge). Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X
1	ROW1	1	3.72
2	ROW2	1	17.58
3	ROW3	1	7.38
4	ROW4	1	4.35
5	ROW5	1	4.59
6	ROW6	1	9.60
7	ROW7	2	0.81
8	ROW8	2	2.94
9	ROW9	2	4.29
10	ROW10	2	22.92
11	ROW11	2	12.33
12	ROW12	2	3.27
13	ROW13	3	5.40
14	ROW14	3	8.46
15	ROW15	3	2.97
16	ROW16	3	6.30
17	ROW17	3	3.63
18	ROW18	3	11.19

*Treatments: 1 = wheat straw; 2 = Kentucky blue grass sod; 3 = bare soil.

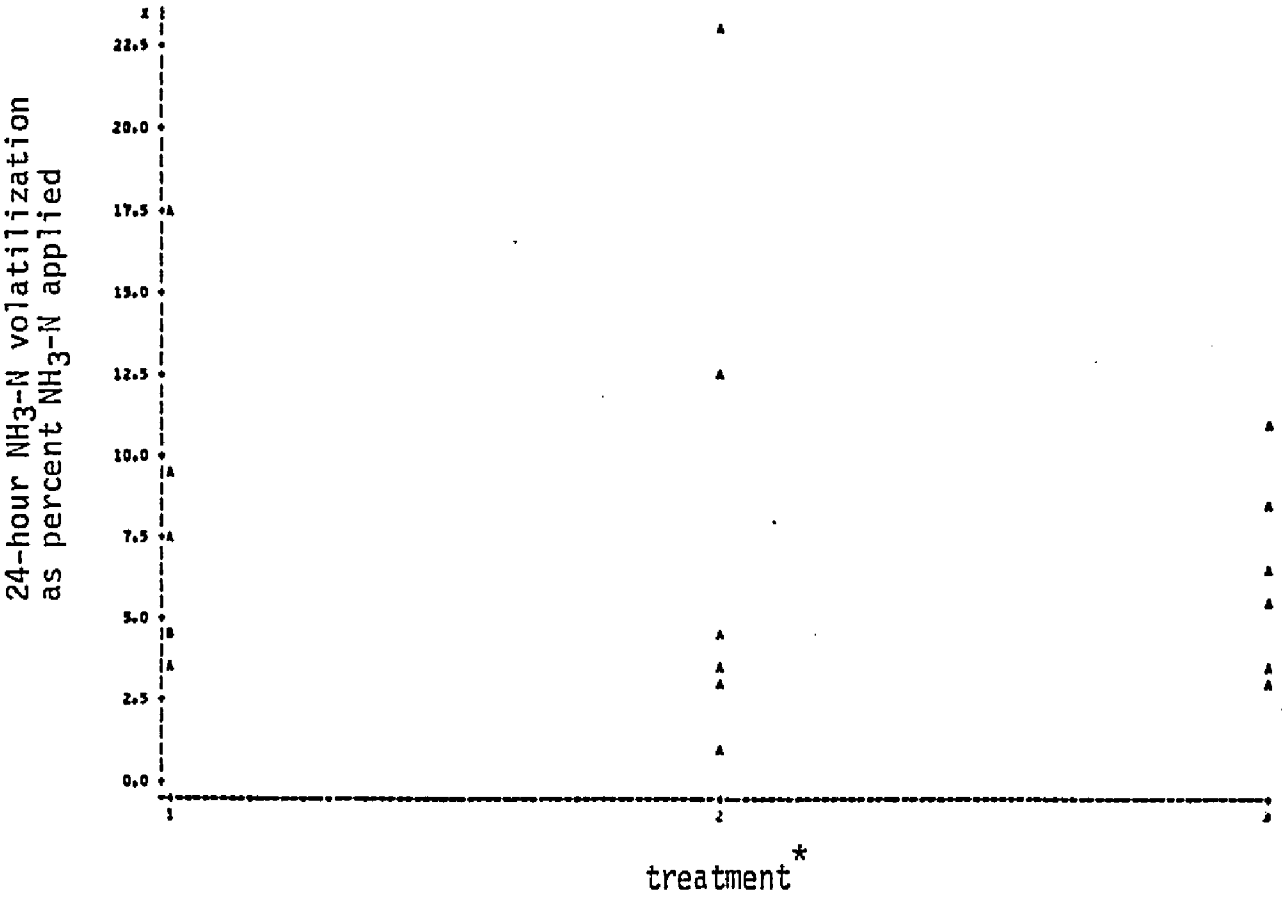

Figure 31. Replicate values of NH_3-N volatilized for each treatment in Experiment 6: Vegetative Cover (Homogenous or Liquid Sludge).

Treatment: 1 = wheat straw; 2 = Kentucky blue grass sod; 3 = bare soil.

Legend: A = 1 observation; B = 2 observations.

Table 48. One-way analysis of variance for Experiment 6: Vegetative Cover (homogenous or liquid sludge.

```
DEPENDENT VARIABLE: X

SOURCE               DF       SUM OF SQUARES          MEAN SQUARE       F VALUE        PR > F       R-SQUARE         C.V.

MODEL                 2           8.91670000           4.45835000          0.12        0.8844       0.016246      81.9811

ERROR                15         539.93655000          35.99577000                     STD DEV                     X MEAN

CORRECTED TOTAL      17         548.85325000                                       5.99964749                 7.31833333

SOURCE               DF          TYPE I SS      F VALUE      PR > F          DF         TYPE IV SS      F VALUE      PR > F

TREAT                 2         8.91670000         0.12      0.8844           2         8.91670000         0.12      0.8844
```

APPENDIX G

Control Treatments

Table 49. The 24-hour NH_3-N volatilization as percent of NH_3-N applied by replication for the controls of Experiments 1 through 6. Calculated from regression equation of percent volatilization versus time of sampling for each replicate.

OBS	ROW	TREAT*	X
1	ROW1	1	3.24
2	ROW2	1	5.82
3	ROW3	1	9.27
4	ROW4	1	10.41
5	ROW5	1	8.40
6	ROW6	1	2.91
7	ROW7	1	9.30
8	ROW8	1	2.13
9	ROW9	2	5.40
10	ROW10	2	8.46
11	ROW11	2	2.97
12	ROW12	2	6.30
13	ROW13	2	3.63
14	ROW14	2	11.19
15	ROW15	2	.
16	ROW16	2	.
17	ROW17	3	23.85
18	ROW18	3	12.72
19	ROW19	3	12.18
20	ROW20	3	9.12
21	ROW21	3	10.95
22	ROW22	3	10.02
23	ROW23	3	.
24	ROW24	3	.
25	ROW25	4	24.60
26	ROW26	4	16.35
27	ROW27	4	25.83

Table 49. (continued)

OBS	ROW	TREAT	X
28	ROW28	4	25.26
29	ROW29	4	39.54
30	ROW30	4	28.05
31	ROW31	4	.
32	ROW32	4	.
33	ROW33	5	17.79
34	ROW34	5	13.11
35	ROW35	5	13.14
36	ROW36	5	13.11
37	ROW37	5	16.26
38	ROW38	5	16.41
39	ROW39	5	.
40	ROW40	5	.
41	ROW41	6	19.62
42	ROW42	6	22.05
43	ROW43	6	33.87
44	ROW44	6	48.42
45	ROW45	6	3.90
46	ROW46	6	.
47	ROW47	6	.
48	ROW48	6	.
49	ROW49	7	5.37
50	ROW50	7	4.78
51	ROW51	7	15.03
52	ROW52	7	.
53	ROW53	7	.
54	ROW54	7	.

Table 49. (continued)

OBS	ROW	TREAT	X
55	ROW55	7	.
56	ROW56	7	.

*Treatment: 1 = Vegetative Cover (solid sludge particles); 2 = Vegetative Cover (homogenous or liquid sludge); 3 = Temperature (12.8, 18.3, and 26.7°C); 4 = Soil Moisture; 5 = pH; 6 = Different Periods of Incorporation; 7 = Five Different Sludges.

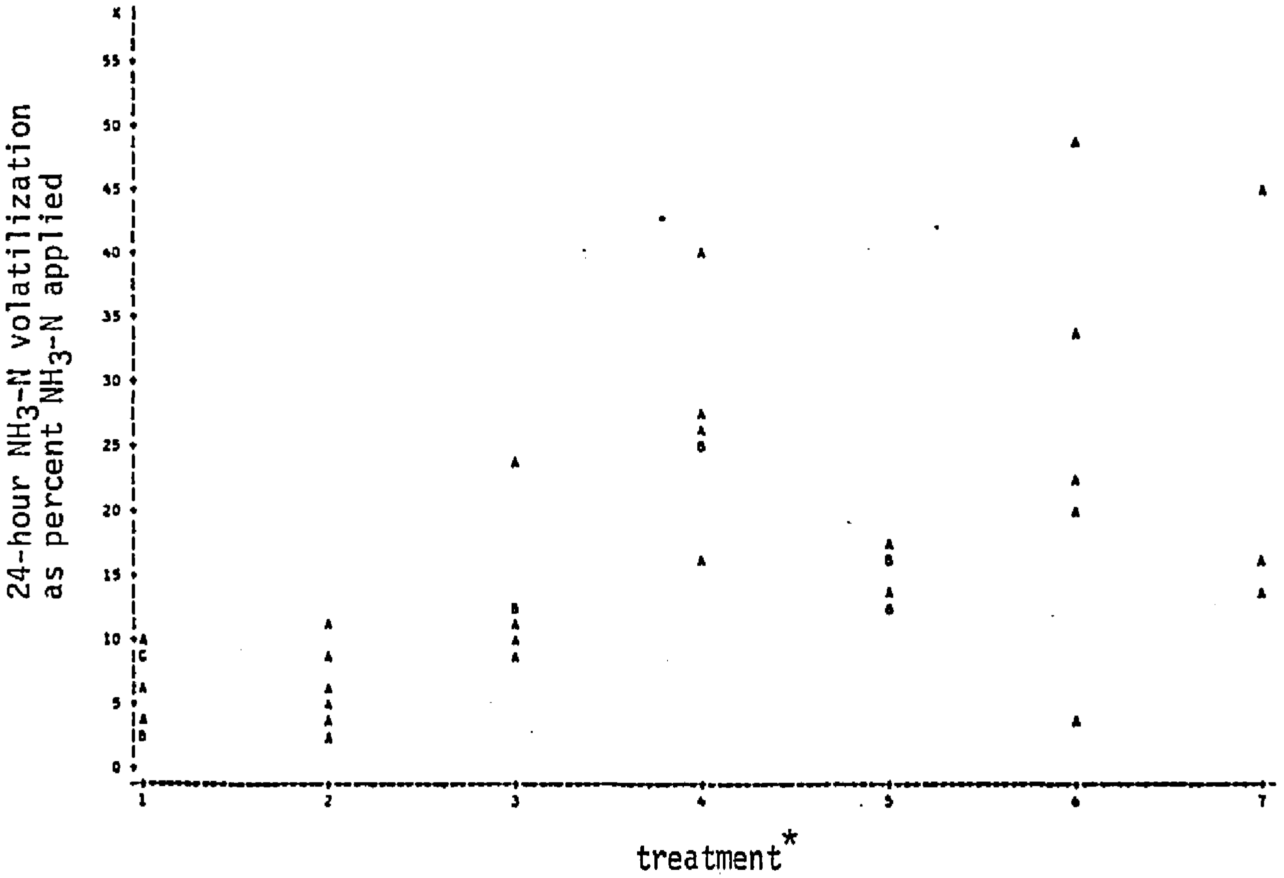

Figure 32. Replicate values of NH_3-N volatilized for the control treatments for Experiments 1 through 6.

Treatment: 1 = Vegetative cover (solid sludge particles); 2 = Vegetative cover (homogenous or liquid sludge; 3 = Temperature (12.8, 18.3, and 26.7°C); 4 = Soil Moisture; 5 = Soil pH; 6 = Different periods of sludge incorporation; 7 = Five different sludges.

Legend: A = 1 observation; B = 2 observations; C = 3 observations.

Table 50. One-way analysis of variance for the control treatments of Experiments 1 through 6.

```
DEPENDENT VARIABLE: X

SOURCE              DF        SUM OF SQUARES        MEAN SQUARE       F VALUE         PR > F        R-SQUARE          C.V.

MODEL                6         2725.80846000       454.30141000          6.58         0.0001        0.544630       53.5189

ERROR               33         2279.07498000        69.06287818                      STD DEV                       X MEAN

CORRECTED TOTAL     39         5004.88344000                                      8.31040782                  15.52800000

SOURCE              DF           TYPE I SS      F VALUE     PR > F        DF         TYPE IV SS      F VALUE      PR > F

TREAT                6         2725.80846000       6.58     0.0001         6      2725.80846000         6.58      0.0001
```