EFFECTS OF RAINFALL, SLOPE, AND VEGETATION

HEIGHT ON RUNOFF WATER QUALITY FROM

FESCUE PLOTS TREATED WITH

POULTRY LITTER

By

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Thesis Approved: Thesis Advisor

Dean of the Graduate College

PREFACE

The design storm selection procedures were developed to assist researchers in selecting storm events to apply in laboratory or field studies when one or more storm events are desired to use as treatment variables. Provided that sufficient rainfall data is available the researcher can use the methodology described in Chapter IV to derive equally probable storm events from a probabilistic analysis of real rainfall data.

The goal of the laboratory study was to better evaluate management and environmental parameters that have the greatest effect on loss of nutrients to surface water runoff. Support for this research effort was provided by the Soil Conservation Service and the Oklahoma Agricultural Experiment Station. Without this greatly appreciated support a study of this magnitude would not have been possible.

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CHAPTER I

INTRODUCTION

BACKGROUND

Land application of poultry litter to permanent pasture is a widely spread and accepted method of disposing of poultry waste. The poultry industry has seen significant growth in recent years all across the Southeast. Poultry production recently has expanded into Eastern Oklahoma and represents major economic opportunity for the region. Because tourism and recreation has been a vital portion of Eastern Oklahoma's economy, poultry production and associated water quality concerns and the impact on Oklahoma's recreational waters are the focus of many researchers. As a fertilizer, poultry litter is a valuable source of nutrients and can greatly increase forage yields and productivity. In terms of the nutrient constituents that are capable of being utilized by plants, there is comparatively more phosphorus in poultry litter than other types of animal wastes. In other words, poultry litter is typically higher in phosphorus than is required by growing plants if the litter is applied on the basis of nitrogen needs. This is the fundamental problem associated with poultry litter in terms of water quality. Applying litter on the basis of nitrogen needs is also accompanied by phosphorus applications that often exceed crop needs. Having phosphorus levels above that which can be utilized by plants posses a

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potential threat to water quality. Streams and rivers which receive runoff waters from land where poultry litter has been applied are of special concern as well as receiving lakes which serve as phosphorus sinks. For most freshwater systems, including those in Oklahoma, phosphorus is a major limiting nutrient for aquatic algae. Increasing phosphorus loads in these waters enhances aquatic productivity and thus incipient eutrophication can result.

In 1990, the USDA Soil Conservation Service (SCS) in Oklahoma established guidelines for animal waste application based on phosphorus. They recommended that broiler litter applied to permanent pastures should not exceed 200 kg/ha P_2O_5 per year on soils less than 15 percent slope. These guidelines increased the land requirements for litter application and reduced the land available for disposal. For most areas of the country where poultry production thrives as an industry there are few other agricultural alternatives. These areas are usually hilly, rocky terrain and to find suitable disposal sites of less than 15 percent can be difficult - especially if poultry litter applications cannot exceed 200 kg/ha P_2O_5 per year.

A recently completed field study, funded by the Soil Conservation Service (SCS), identified significant parameters affecting the magnitude of the constituents in surface runoff resulting from the land application of poultry litter onto permanent pasture (Huhnke et al., 1993; Storm et al., 1993). This study evaluated the affects of poultry litter application rate, slope, and vegetation condition on nutrient losses in runoff. The results from the field study indicate a high degree of variability within the data. This suggested a need for more controlled conditions.

LABORATORY STUDY

The SCS and the Oklahoma Agricultural Experiment Station provided support for this laboratory study to better evaluate management and environmental parameters that have the greatest effect on loss of nutrients to surface water runoff. The results of this study will then be validated in the field through support provided by SCS and the U.S. Environmental Protection Agency. The information obtained from these laboratory and field studies will be used to develop recommendations for rates and timing of animal waste application and other management techniques to minimize off-site water quality impacts.

The focus of this thesis is the laboratory study which was designed to determine the effects of rainfall, slope, and vegetation height on runoff water quality from fescue plots treated with poultry litter. Small-scale plots were constructed to approximate field conditions using feacue grown in 1.0 meter square boxes. Rainfall was applied using a single nozzle rainfall simulator. Broiler litter was applied at 6.7 Mg/ha which reflects typical field application rates in Eastern Oklahoma. The experimental design consisted of a 3x3x3x2 factorial where rainfall duration, rainfall intensity, and slope were studied at three levels with two levels of vegetation height. The variables involving rainfall were developed from nine years of 15-minute rainfall data from southern Delaware County in Eastern Oklahoma. Design storms presented in Chapter IV are intensity-duration combinations having equal probabilities of occurrence derived from a probabilistic analysis of storms occurring during the nine years of observed rainfall from the study area. The design storms selected to use as experimental variables were modified from those derived in Chapter IV to represent a range of storm events that cover occurrences of short duration and low intensity to long duration and high intensity events. Rainfall intensities were 25, 38, and 51 mm/h. Rainfall durations were 26, 84, and 204 minutes. Slopes were set at 5, 10, and 15 percent; and fescue was cut to a height of 75 and 150 mm. Samples were composite runoff samples for each storm duration and were analyzed for the following water quality parameters: ammonia-N (NH₃-N), nitrate-N (NO₃-N), total Kjeldahl nitrogen (TKN), soluble phosphorus (PO₄-P), total phosphorus (TP), and total solids (TS).

The results of this study will aid in developing best management practices (BMPs) for poultry producers as well as help establish guidelines for future field studies. The effect of scale is very important in how these results are interpreted. For these laboratory conditions, concentrated flow regimes are not realized due to the scale. The observations represent worst case scenarios in that rainfall is applied immediately after application and antecedent moisture conditions were high.

RESEARCH OBJECTIVES

This research involves two major areas of interest: 1) the selection of design storms and 2) the effects of rainfall and management factors on runoff water quality. In chapter IV, a systematic methodology for selecting design storms for rainfall simulator studies is presented. Chapters V, VI, and VII discuss procedures, results, and conclusions of the study where these design storms were applied.

Specifically, the research objectives are:

- Develop a systematic methodology of design storm selection for rainfall simulator studies.
- 2. Determine if rainfall intensity, rainfall duration, slope, and vegetation height have a significant effect on runoff water quality.

CHAPTER II

LITERATURE REVIEW

RAINFALL SIMULATOR STUDIES

Application rates

Considerable research efforts have been devoted to the effects of litter/manure application rates on hydrology and movement of nutrients via runoff. Edwards and Daniel (1993) used fescuegrass plots (1.5m x 6m) and simulated rainfall to study the effects of application rate of broiler litter on total Kjeldahl nitrogen, ammonia nitrogen, nitrate nitrogen, total phosphorus, dissolved reactive phosphorus, total suspended solids, chemical oxygen demand, and electrical conductivity. Application rates were 0, 218, 435, and 870 kg N ha⁻¹ on 5 percent slopes, and rainfall intensities of 5 and 10 cm/h for 30 minutes after runoff began. Litter application rate did not significantly effect runoff depth. Results indicated that increasing the litter rate significantly effected runoff concentrations of all nutrient constituents. Mass nutrient transport of all constituents also increased significantly as application rates increased. Both runoff concentrations and mass nutrient losses were found to increase approximately linearly with increasing application rate.

In a similar study by Edwards et al. (1994), solids transport and erodibility were of primary concern. Litter application and rainfall rates were 0, 5.9, 11.8, and 23.5 Mg ha⁻¹,

and 5 and 10 cm/h, respectively. Solids yield increased with increasing application rate as well as erodibility, which increased linearly ($r^2 = 0.98$) with application rate. Westerman et al. (1983) observed the same effects on runoff volume and poultry litter constituents in a laboratory study using 1.0 m x 0.64 m plots were treatments were applied to bare soil. Loading rates were 3 and 6 Mg/ha and did not significantly influence runoff volumes but was significant in relation to average concentrations and mass transport of all nutrients studied. "There were generally large increases in both concentration and mass transport with addition of manure or litter compared to the controls (bare soil). Doubling the loading rate approximately doubled the concentration and mass transport in runoff of all nutrients."

A field study conducted by Storm et al. (1992) observed a significant effect of poultry litter application rate on fescuegrass plots that measured 1.8 m x 9.8 m. Litter application rates ranged from 0 to 960 kg P_2O_5 ha⁻¹. Runoff volumes decreased as poultry litter applications increased. However, both TP and TKN average concentrations increased as litter application rate increased. Also, the percent litter phosphorus lost in runoff decreased as the litter application rate increased. The same trends were observed for TKN percent loss in runoff to the TKN supplied by the litter as application rates were increased.

Edwards and Daniel (1992) used poultry manure slurry in a field study that utilized fescuegrass plots (1.5 m x 6 m). The results of the study contrasted with other studies that used poultry litter as the nutrient source. Runoff volumes increased significantly as slurry rates increased. The significant effect of slurry application rate was described as 'likely due to both the addition of water to the soil through the slurry and to sealing of the

soil surface by the organic matter in the slurry." Slurry application rates used were 0, 220, and 879 kg N ha⁻¹. Average runoff concentrations and mass losses of slurry constituents were also significant as slurry rates increased. Over this range of application rates, both responses in runoff concentrations and mass losses were linear.

Litter vs Manure

The effects of application rate on runoff and runoff water quality are not only dependent upon the amount of waste applied but the type of waste. Poultry litter is a relatively dry waste as compared to poultry manure slurry. Poultry manure is typically more concentrated in terms of nutrients. Due to the dry nature of poultry litter, runoff is reduced by the litter's ability to absorb rainfall and slow overland flow velocities which increases ponding and thus increases infiltration. This effect is more pronounced at high application rates. On the other hand, poultry manure slurry adds water to the soil profile and the drying of the slurry can act as a sealant on the soil surface. With poultry litter, the bedding (woodchips, rice hulls, straw, ect.) makes up a significant portion of the volume.

Edwards and Daniel (1991) reported average values of N, P, and K for poultry litter and poultry manure. The study reported that poultry litter contains approximately 4.1 percent nitrogen, 1.4 percent phosphorus, and 2.1 percent potassium (dry basis). Also, poultry manure was reported to contain average nutrient constituents of 4.6 percent nitrogen, 2.1 percent phosphorus, and 2.1 percent potassium (dry basis). Average moisture contents for litter and manure were 19 and 91 percent, respectively. In this same reference (Edwards and Daniel, 1991), waste type was used as a variable to evaluate the impact of poultry waste application on infiltration and runoff. Both infiltration and runoff were greatly influenced by the litter and the manure. By increasing the broiler litter rate it took more rainfall to produce runoff. Conversely, layer manure tended to increase runoff and decrease infiltration. Waste type was determined to be the single most important factor that affected hydrologic parameters.

Westerman et al. (1983) used both litter and manure. His observations agree with Edwards and Daniels (1991) in that manure type was significant in influencing runoff volumes. Manure type had a significant effect on both average concentrations and mass transport of soluble P, nitrate, and mass transport of TKN.

<u>Rainfall Intensity</u>

The effects of rainfall intensity on nutrient transport in relation to runoff water quality has been addressed in many studies. Most studies indicate rainfall intensity as a significant treatment variable. In general, nutrient concentrations decrease and mass nutrient loading increase as rainfall intensity increases. The decrease in nutrient concentrations are due to dilution effects. Increases in mass nutrient loading are attributed to greater runoff volumes and runoff rates and thus more energy available for transport. In most studies in which rainfall intensity is a treatment variable, design storms represent relatively high return periods compared to more typical intensities that are more likely to occur. Using rainfall intensities that tend to represent extreme events are valuable in that worst case scenarios are of interest, especially in preliminary studies where little information is available.

Rainfall intensity has been a significant treatment variable in several field studies (Edwards et al., 1994; and Edwards and Daniel, 1993;1992) as well as laboratory studies

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as described by Westerman et al. (1983). These studies used rainfall intensities of 50 and 100 mm/h. Westerman et al. (1983) observed rainfall intensity as having a significant effect on average concentrations of TKN, ortho-P, runoff volume and the ratio of volatile solids to total solids. Rainfall intensity was also significant for the mass transport of all nutrients. In summarizing the effect of rainfall intensity on manure and soil transport from bare soil plots, it was suggested that manure is easily detached and transported. As rainfall intensity increases, the increase in soil transport is greater relative to the increase in manure transport, probably because the manure is a limited source for erosion, with the limit depending upon the loading rate.

Rainfall Duration

Rainfall duration in most experimental studies was fixed. However, when sampling runoff from different time intervals throughout the runoff hydrograph, the effects of rainfall duration are considered. Edwards et al. (1994), Edwards and Daniel (1993, 1992, 1991) used a fixed duration of 30 minutes after runoff began. Westerman et al. (1983) used a fixed duration of 30 minutes and Huhnke et al. (1992) used a fixed duration of 75 minutes. The effects of rainfall duration on surface water quality have not been investigated.

Field Slope

Field slope is a particular point of interest, especially from a confined animal feeding standpoint. Usually in areas where confined animal feeding is concentrated there are few other agricultural alternatives. These are areas that are generally hilly and permanent pasture is the predominant system on which manure is applied.

With most field studies it is difficult to use specific slopes due to the logistics and availability of suitable sites. However, laboratory studies are much better suited to study a range of slopes. Westerman et al. (1983) used a nine percent slope on bare soil in a laboratory study. Studies previously cited by Edwards et al. (1994), Edwards and Daniel (1993, 1992, 1991), used a fixed slope of five percent.

Mitchell and Gunther (1976) conducted a laboratory study using liquid swine manure on bare soil plots (0.91 m square) set at three and nine percent slope to evaluate the effects of manure applications and slope in runoff, erosion, and nitrate loss. The nine percent slope had runoff volumes that were 40 percent greater than the three percent slopes. Nitrate concentrations were also greater for the nine percent slopes than the three percent. Volatile solids concentration and loss decreased with the amount of manure for the three percent slopes, but at the nine percent slopes; the volatile solids concentration increased with the amount of manure but total loss was not effected.

This study agrees with the previous discussion on the high erodibility of manure and the effects of rainfall intensity on the transport of manure particles. By increasing the slope the ability of runoff to transport manure constituents increases. There is also a significant interaction of slope and application rate on runoff volumes and consequently manure transport.

Field slope has also been investigated as a continuos variable by Storm et al. (1992). Slopes ranged from 3 to 15 percent in a field experiment that studied the effects of poultry litter, slope, and other factors on runoff water quality. Two seasons were considered as treatment variables (spring and summer). Observations indicated that slope did not

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significantly affect curve number and runoff volume when averaged across seasons. Slope was a significant factor in total phosphorus average concentrations for the spring runs but was insignificant for the summer runs. However, significant slope interactions were present influencing curve number and runoff volumes for the summer runs.

Vegetative Cover

Vegetative cover is a critical factor in terms of management in minimizing erosion and nutrient losses. Both pasture vegetation and crop residue aid in dissipating the energy of rainfall as well as reduce overland flow velocities which can enhance infiltration rates. Thus by decreasing runoff volume and energy, losses can be minimized.

Laboratory experiments by Westerman et al. (1983) and Mitchell and Gunther (1984) both used bare soil plots to investigate water quality parameters. Field studies reported by Edwards et al. (1994), Storm et al. (1992), and Edwards and Daniel (1993, 1992, 1991) applied selected treatments to fescue grass plots. All of these studies used fixed vegetation levels or bare soil with the exception of Storm et al. (1992). The effects of mowing were investigated in this study where mowed plots were represented by fescue cut to a length of 5 cm and the unmowed were approximately 30 cm in height. Fescue height was a significant factor on average concentrations of total phosphorus. Decreasing trends in concentrations were observed for the taller fescue.

Drying Effects

Edwards et al. (1993) studied the effects of poultry litter drying interval on runoff water quality. Fescue plots were used in this field study and a portable rainfall simulator was utilized to produce a design storm. Drying interval times were 4, 7, and 14 days. It was concluded that drying interval did not significantly affect either concentration or total mass in any constituent lost in the runoff. In a controlled laboratory experiment, it was determined by Crane et al. (1981) that there were substantial losses of nitrogen during application and incubation which was explained by the volatilization of ammonia.

NUTRIENT DYNAMICS

Generally, the greatest pollution potential to receiving waters is the first runoff producing event after poultry litter has been surface applied. However, there are shortterm nutrient transformations that take place where litter constituents such as nitrogen and phosphorus in non-equilibrium states undergo significant changes in approaching equilibrium with the soil interface. Examples would be nitrification and ammonia volatilization, and phosphorus absorption/desorption. Several soil and environmental factors influence transformation rates such as soil and air temperature, soil moisture, soil pH, cation exchange capacity (CEC), wind velocity, and method of application. Hensler et al. (1971) reported that substantial amounts of nutrients were lost when rainfall occurred immediately following manure application. Knowledge of the litter's chemical dynamics and the timing of litter application in relation to forecasted rainfall is an important management consideration.

Short-term Nitrogen Transformations

Crane et al. (1981) studied short-term chemical transformations following land application of poultry manure in an environmentally controlled chamber. Poultry manure application caused an immediate increase in soil pH that was dependent on the loading rate of manure. Nitrification was retarded due to anaerobic conditions produced by the manure and ammonia volatilization resulted in substantial losses of nitrogen during application and incubation.

A nonpoint source model for land areas receiving animal wastes was developed by Reddy et al. (1979) that contains components for mineralization of organic nitrogen and ammonia volatilization. Assuming first-order kinetics for mineralization, and volatilization and rate constants based on available literature, it was concluded that about 50 percent of the N available for mineralization was converted in 3 to 6 weeks for poultry wastes. Ammonia volatilization losses were determined to occur relatively rapid. Approximately 80 percent of the applied ammonia is volatilized during the first week after application. Soils with low CEC were determined to have greater losses of ammonia than soils with high CEC.

Phosphorus Forms and Chemical Transformations

Phosphorus forms are not as dynamic as nitrogen forms in terms of chemical transformations. Soluble phosphorus makes up a large portion of the total phosphorus found in poultry litter and is readily available for plant uptake. The form of soluble phosphorus in solution is determined by the pH. In the acid range $H_2PO_4^{-1}$ predominates, while in the alkaline range HPO_4^{-2} is more abundant (Methods for the determination of

inorganic substances in environmental samples, U. S. EPA). Labile phosphorus is loosely bound to iron, aluminum, and calcium cations in the soil. This form exists in equilibrium with soluble phosphorus and the equilibrium constant is a function of pH and the CEC. If phosphorus is applied at rates greater than what the growing crop can utilize, a buildup of soil P will occur and fewer adsorptive sites will be available for binding P. Since loosely bound labile P is in equilibrium with soluble forms of P ($H_2PO_4^{-1}$ and HPO_4^{-2}), when soil P levels are high the potential for high soluble P levels are great. The excess soluble and labile P may leach downward to a zone that has more attachment sites and then be converted to labile or fixed P, or it maybe transported off-site in surface runoff.

Singh and Jones (1976) studied the effects of low and high organic P residues on sorption and desorption of P by a high P-fixing soil. Poultry manure was among the high organic P residues used in the study. The critical value of P in organic residues which resulted in no tie-up of soil P was 0.3 percent. It was concluded that poultry manure contained organic P in excess of 0.3 percent (1.42 percent P) and decreased soil sorption of P when incubated for 30, 75, and 150 days. The explanation given was that when there is no utilization of soil P, the added P results in higher equilibrium-solution levels. Also, a decrease in soil sorption of P could be the result of sorption sites being tied up by the products of organic matter decomposition.

Long-term Impacts of Manure/Litter Application

Kingery et al. (1994) conducted a study to determine the effects of long-term broiler litter application on environmentally related soil conditions in the Alabama region. Comparisons were made between littered and nonlittered pastures on matching soil series maintained under perennial tall fescue. Soil cores indicated litter application increased organic C, total N, NO₃-N, pH, electrical conductivity, and extractable P, K, Ca, Mg, Cu, and Zn. Elevated levels of organic C and total N were observed from littered soils to depths of 15 and 30 cm, respectively, and pH was found to be 0.5 units higher to a depth of 60 cm as compared to nonlittered soils. Significant NO₃-N accumulations were found to be six times greater on littered versus nonlittered soils to a depth of 60 cm.

Over time, heavy applications of manure/litter can cause accumulations of nutrients in the soil profile, especially NO_3 -N at or near a confining layer. Most importantly, elevated levels of soil phosphorus decrease sorption characteristics of the soil and cause phosphorus to more readily desorb and thus transported in surface runoff.

CHAPTER III

EXPERIMENTAL APPARATUS

FESCUE BOXES

Thirty boxes which measured 1.0 meter square by 0.33 meter deep were constructed with a perforated bottom to hold soil and grow fescue in an effort to simulate permanent pasture typical of Eastern Oklahoma as well as much of the Southeast. The soil type and nutrient status will be discussed in the next section. The boxes were seeded with tall fescue (*Festuca arundinacea*), specifically the Kentucky 31 variety.

The fescue boxes were constructed from a supporting frame of heavy angle iron with treated plywood attached as the sides of the box. Four steel rods provided the support for the bottom of the box. The steel rods also supported a section of expanded metal (4.76 mm) on which a piece of fine mesh polyethylene screening was then attached. The boxes were filled to a depth of 5 cm with a gravel/sand mixture. Coarse sand was then added and spread evenly to an additional depth of 2.5 cm. Porous hose (soaker hose) was lain on top of the sand layer and arranged in a circular fashion with one end capped and the other routed through a hole in the side of the box. The hose extended to the outside where a connection was made to a water supply. The plywood side on the front of the box was cut 2.5 cm lower than the other three sides so runoff could leave the plot. A

flume made of galvanized metal was attached to the lower side to direct runoff. The remaining 25.5 cm were filled with a Baxter silt loam from Delaware County, Oklahoma. To make the fescue boxes easily retrievable, four 10 cm x 10 cm wooden blocks were bolted on the bottom of each box at the corners to allow access by a floor dolly and forklift.

The Baxter soil series' area of occurrence is quite large. The National Cooperative Soil Survey reports it's occurrence as nearly level to gently sloping upland soils in the valleys of the Quachita Mountains and the Arkansas Valley and Ridges. The Baxter silt loam is classified in the hydrologic soil group D. However, the upper horizon of this soil type generally has a much higher permeability than the lower layers in the strata. Soil Conservation Service (SCS) Soil Survey reports a permeability range of 15 mm/h to 51 mm/h for the upper horizon of the Baxter silt loam. The soil to be used for this experiment measured an average permeability of 19 mm/h, which agrees with SCS report.

Three composite soil core samples taken to a depth of 5 cm were sent to the Oklahoma Soil, Water, and Forages Analytical Laboratory for analysis. For a stated yield goal of 6,700 kg/ha fescue the soil was deficient by an average of 200 kg/ha of actual nitrogen, 56 kg/ha of P_2O_5 , and 64 kg/ha of K_2O . The pH tested an average of 4.6 which is the lower end of the range (4.5-7.0) recommended for fescue in Oklahoma (Oklahoma Soil Fertility Handbook, 1977). Table 3.1 shows the results of the soil tests based on a 15 cm furrow slice.

Sample No.	NO3-N (kg/ha)	P (soil test index)	K (soil test index)	pH
rep I	2.24	15	97	4.6
rep II	2.24	15	95	4.6
rep III	2.24	15	86	4.5

Table 3.1Results of composite soils tests taken from the fescue boxes.

To avoid conducting the experiment with fescue boxes in a nutrient deficient condition (primarily P_2O_5) the top 8 cm of the soil surface was removed and amended with a 20:10:10 (N:P:K) commercial fertilizer applied at a rate of 0.028 kg/m². Sixty percent ECCE (effective calcium carbonate equivalent) commercial lime was also applied at a rate of 9.3 Mg/ha to bring the soil pH up to 6.8. The soil tests reported an average soil buffer index (SBI) of 6.5 which was used to calculate the amount of lime needed. The soil that was removed from the boxes and the fertilizer/lime amendment were mixed individually in a rotary mixer and placed back in the boxes.

Soil contained in the boxes were allowed to consolidate naturally over a period of several months. The junction between the soil and inside of the plywood sides was filled and packed with powdered bentonite. When wet, the swelling properties of the bentonite maintained a seal along the sides of the box. Paraffin was melted and used to seal the exposed bentonite along the edges.

Tall fescue seed was broadcasted over the soil surface and the boxes were kept moist by subirrigation through the porous hose. After a complete cover of fescue was established, the forage was allowed to grow for 18 months before treatments were applied. This gave the fescue time to become established and provided satisfactory leaf litter cover at the soil surface. During the establishment period the forage was harvested approximately once a month to maintain plant vigor.

POULTRY LITTER

The term poultry litter refers to the waste that is removed from the broiler houses when a growing cycle is completed. Poultry litter contains bedding, poultry excrement, feathers, wasted feed, ect. that accumulate on the floor of the broiler house during the grow-out period. When the finished birds are marketed to a slaughter facility, they are removed from the houses and generally the floor is scraped with a skid loader. The waste removed at this time is generally the top layer that is usually thin and caked. A more complete clean out of the poultry house is usually done one or two times a year. This type of clean out includes the removal of the underlying bedding material. The litter is then loaded directly onto spreader trucks to apply to pasture or farmland. The poultry litter used in this experiment contained woodchips as the bedding material which is typical of modern broiler production facilities in Eastern Oklahoma.

Approximately 1.0 metric ton of litter was transported from Eastern Oklahoma for use in the experiment. The litter was individually double bagged and tied in 10 liter plastic bags and stored in deep freezers at -10 °C for one year prior to the experiment.

RAINFALL SIMULATOR

The rainfall simulator used is a modified design of the Kentucky Rainfall Simulator described by Moore et al. (1983). The rainfall simulator was designed to allow the fescue boxes to tilt and accommodate different slope settings. The simulator used in this experiment consists of a single oscillating nozzle which travels back and forth between two return pans. The simulator utilizes a Veejet 80100 nozzle with an operating pressure at the nozzle of 41 kPa to achieve the drop size and kinetic energy typical of natural rainfall (Bubenzer, 1980). Uniformity coefficients as described by Christiansen (1942) were computed by:

$$C_{u} = \left[1 - \frac{\sum \left|x_{i} - \overline{x}\right|}{n\overline{x}}\right] 100$$
(3.1)

where C_u is the uniformity coefficient, x_i is the point reading, \overline{x} is the mean of the point values, and n is the number of sampling points within the plot. The uniformity coefficients were $C_u = 99$ for intensities of 25 mm/h, $C_u = 97.9$ for intensities of 38 mm/h, and $C_u = 95.7$ for intensities of 51 mm/h. The high coefficients can be attributed to having only one nozzle and the spray area being only 1 m x 1 m. Larger scale rainfall simulators of this type that are equipped with multiple nozzles and have much larger spray areas have reported uniformity coefficients of 80.2 to 83.7 (Moore et al., 1983).

RUNOFF WEIGHING SYSTEM

Runoff was collected in a series of three tanks. The three tanks fit one inside the other with the smallest tank designed to accommodate runoff volumes from 26 minute durations, the medium size tank for 84 minute durations and the largest tank for 204 minute durations. The tanks rested on a balance that was equipped with two strain gauges on either side. A data-logger recorded the strain gauge readings every 15 seconds for the duration of the storm events. This information was then downloaded to a personal computer to analyze the runoff hydrograph. The hydrograph data were actually the mass flow rate of the runoff throughout the rainfall event.

CHAPTER IV

SELECTION OF DESIGN STORMS

INTRODUCTION

A design storm is the term commonly used to describe a hydrologic event used to design a hydrologic structure. However, a design storm can be extended to the application of hydrologic and water quality models for predicting an "average" pollutant loss, or in this experiment the rainfall event used in a rainfall simulator study. For hydrologic modeling, precipitation processes have been modeled using probabilistic analysis of observed data. Most researchers have concentrated on daily precipitation models due to the unavailability of detailed long-term rainfall data, and the development of crop growth and ET models which typically require only daily rainfall.

The subject of this chapter is to define a systematic methodology for selecting a design storm(s) based on historical rainfall data for rainfall simulator studies. To date the selection of a design storm for rainfall simulator studies has been arbitrary. The methodology describes procedures for deriving a matrix of design storms for a laboratory-scale rainfall simulator study using nine years of 0.25-hour rainfall data. The methodology can also be applied to field-scale rainfall simulator studies that require one design storm or an extensive matrix of design storms.

The design storm selected for a particular experiment is a function of the process of interest, such as erosion, movement of pesticides or nutrients, and related physical and chemical processes. The effect of scale is very important when selecting an appropriate design storm, e.g. laboratory, plot, field, or watershed scale. Infiltration, ponding, chemical and physical interactions with the soil matrix and the surface vary temporally as well as spatially. Therefore, the selection of an appropriate design storm is essential.

The selection of the proper rainfall event, or design storm, for rainfall simulator studies dramatically affects the outcome and conclusions from an experiment. Typically only a few storms each year are responsible for the majority of the pollutants that reach the stream. From a USDA-ARS plot study in Kingdom City, Missouri, Burwell and Kramer (1983) identified 2 out of 24 years of erosion that accounted for approximately 60 percent of the total soil loss. Hjelmfelt et al. (1986) found that over the 24 year period of record, 19 of the largest soil loss events produced 50 percent of the total sediment yield. Therefore, the selection of an appropriate design storm is crucial.

There are four characteristics that define a rainfall event: duration, volume, temporal and spatial intensity distribution. The distribution of rainfall intensity is very important to runoff-related processes. Because most storms are average intensities over the duration of the event do not equal the maximum intensity at some point during the event. Most rainfall simulator studies assume a uniform intensity. As the study of rainfall/runoff driven processes expand, the temporal distribution of rainfall will obviously be considered in rainfall simulator studies. For this study, however, a uniform rainfall intensity is assumed and thus the methodology will identify design storms with a specified rainfall depth and duration.

A 3x3 matrix of design storms was calculated by the procedures in this chapter. However, the design storm matrix can be either expanded or downsized to fit the needs of other experiments. The rows and columns of the matrix represent three mean durations and three mean depths, respectively. Therefore, the nine elements of this matrix are design storms of a determined depth and duration. The rainfall simulator was calibrated for intensity, determined by oscillations per second, so the depth-duration matrix is converted to an intensity-duration matrix.

PROCEDURES

Defining Storm Events

Nine years (1974-1982) of rainfall data were obtained from a gaging station in Southern Delaware County, Oklahoma. Data resolution was 0.25 h and 2.54 mm. Individual storms were processed using an arbitrary critical duration of one hour. The critical duration is the minimum period of time with no precipitation before a sequence of data is declared a single event (Griffin, 1988). The intensity distribution of observed rainfall data is seldom continuous and the majority of precipitation occurs in bursts separated by periods of little accumulation (Huff, 1967). The effect of scale is very important. The intent of this study is to utilize single events on a laboratory-scale. Therefore, it is desirable to preserve the precision of the data and avoid combining a series of single events into one larger event. The determination of the critical duration can be approached analytically or it can be an arbitrary decision. Analytical determination is usually calculated by one of two methods identified by Griffin (1988): rank correlation or exponential.

For our application, only runoff producing events are of interest. The design storms produced by this method must exceed the infiltration rates of the soil in the fescue boxes. Steady-state infiltration was estimated and design storms were chosen that exceed this value. The variability between boxes, in terms of infiltration and drainage characteristics, were minimized by saturating the boxes prior to rainfall. Steady-state infiltration was approximated using rainfall simulator experiments described below.

Infiltration Experiments

A storm of 38 mm/h for 0.5 h was arbitrarily selected and applied to the thirty boxes to estimate steady-state infiltration. The Delaware County soils map lists the maximum infiltration rate of the Baxter silt loam to be 51 mm/h. The duration of the storm was chosen for convenience. The boxes were thoroughly saturated before being placed under the rainfall simulator. The slope of the boxes was set at 5 percent. Runoff was weighed every 15 seconds in the collection tank and the runoff data were downloaded to a computer to calculate a mass flow rate for each box.

The mass flow rate was divided by the surface area of the box and the density of water to obtain a steady-state runoff rate. Steady-state infiltration rates were calculated by subtracting steady-state runoff from rainfall intensity. The arithmetic mean of the steady-state infiltration rates was 19 mm/h. Figure 4.1 shows a relative frequency histogram of the steady-state infiltration rates (Table a.1, Appendix A).

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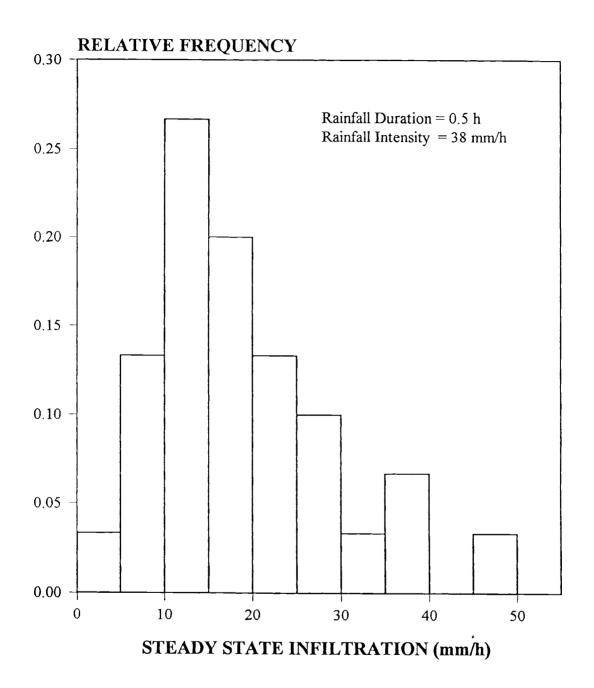


Figure 4.1 Relative frequency histogram of steady state infiltration rates of fescue boxes.

Analysis of Storm Durations

A critical duration of one hour defined 423 individual storm events from the nine-year rainfall record. A lognormal probability density function (pdf) was fit to these durations, given as:

$$p(x) = \frac{\exp\left[-\frac{1}{2}(\ln x - \mu_{y})^{2} / \sigma_{y}^{2}\right]}{(2\pi x^{2} \sigma_{y}^{2})^{\frac{1}{2}}}$$
(4.1)

where μ_y is the mean and σ_y^2 is the variance of the natural logarithms. Figure 4.2 shows the relative frequency histogram of storm durations and the fitted lognormal pdf. The rainfall data were recorded every 0.25 h, and thus it was unreasonable to consider the area under the pdf that was less than this value in the analysis. The time increment of 0.25 h will be denoted as threshold duration (TD). Thus, the pdf was truncated at TD. The probability of an observation greater than or equal to TD was numerically integrated and divided into three equal areas. The first moment for each area interval with respect to the origin were $\overline{D}_1 = 0.44$ h, $\overline{D}_2 = 0.85$ h, and $\overline{D}_3 = 1.67$ h. Figure 4.3 shows the lognormal pdf and calculated mean durations. Table 4.1 is a summary of these computations.

Duration Interval	Prob	Lower Bound (h)	Upper Bound (h)	Mean D (h)
Di	0.32	0.25	0.64	0.44
D ₂	0.32	0.64	1.15	0.85
D_3	0.32	1.15	7.25	1.67

Table 4.1 Mean Storm Durations Defined from Lognormal PDF

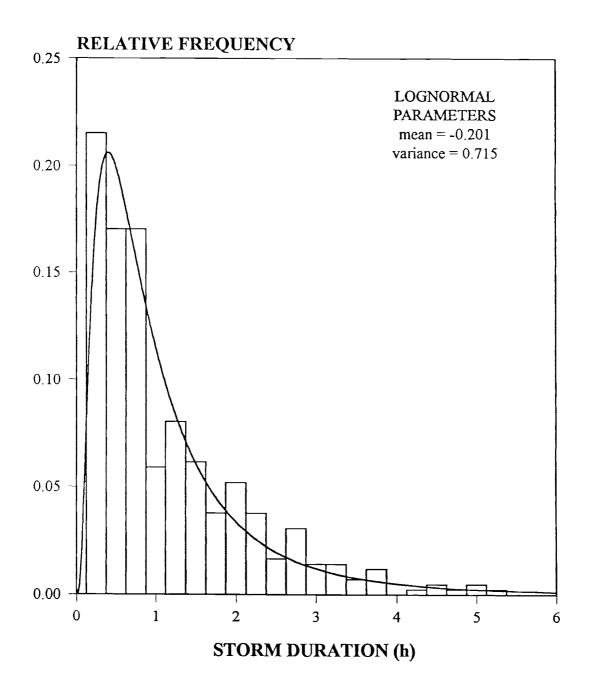


Figure 4.2 Relative frequency histogram of storm durations from Delaware county rainfall data and a fitted lognormal PDF.

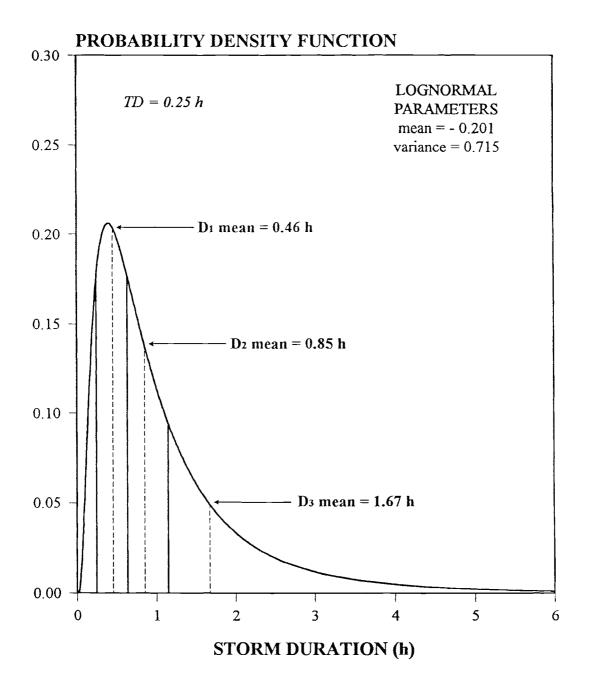


Figure 4.3 Defined mean storm durations by calculating the first moment of each area interval with respect to the origin.

Analysis of Rainfall Depths

Once \overline{D}_1 , \overline{D}_2 , and \overline{D}_3 are determined, the rows of the design storm matrix are complete. To fill the columns of the matrix, the same general procedure is followed to arrive at three mean rainfall depths that correspond to each mean duration.

Storms whose durations lie between the lower and upper bounds of each of these intervals were grouped into separate data sets. These three data sets will be referred to as D_1 , D_2 , or D_3 rainfall depths. From the 423 individual storms identified, 162 storms were in the range 0.25 and 0.64 h (D_1), 131 were between 0.64 and 1.15 h (D_2), and 130 were between 1.15 and 7.25 h (D_3). Separate relative frequency histograms were examined for D_1 , D_2 , and D_3 and lognormal pdfs were fit to the data. To obtain better fits to the rainfall depth pdfs, D_1 rainfall depths were transformed by subtracting 3.81 mm, D_2 rainfall depths were transformed by subtracting 3.18 mm, and the D_3 rainfall depth PDF's were obtained by trial and error. In effect, this shifts the values closer to the origin. The transformations were selected by trial and error until reasonable fits were obtained. Figures 4.4, 4.5, and 4.6 show the relative frequency histograms and the fitted lognormal pdfs for D_1 , D_2 , and D_3 rainfall depths, respectively.

Each mean duration, \overline{D}_1 , \overline{D}_2 , and \overline{D}_3 was multiplied by the mean steady-state infiltration rate to obtain depth threshold values. The respective threshold depths were denoted as Td₁, Td₂, and Td₃. Therefore, Td₁ equals 8.66 mm, Td₂ equals 16.2 mm, and Td₃ equals 31.7 mm. Due to the transformations of the rainfall depths, Td₁, Td₂, and Td₃ must also be transformed before applying them to the pdfs. By subtracting 3.81 mm from Td₁, 3.18 mm from Td₂, and 6.35 mm from Td₃, the transformed threshold depths are Td₁' = 4.85 mm, Td₂' = 13.1 mm and Td₃' = 25.4 mm.

For each lognormal pdf, the probabilities of observations greater than the threshold depths $(Td_1', Td_2', and Td_3')$ were numerically integrated and divided into three equal areas. The first moment for each area interval was calculated with respect to the origin to yield three mean depths having equal probabilities of occurrence (Figure 4.7, 4.8, and 4.9). Table 4.2 summarizes the computations needed to determine the mean depths (denoted as \overline{d}) required for completing the design storm matrix. Tables 4.3 gives the depth-duration design storm matrix, and Table 4.4 gives the intensity-duration matrix derived from depth-

duration.

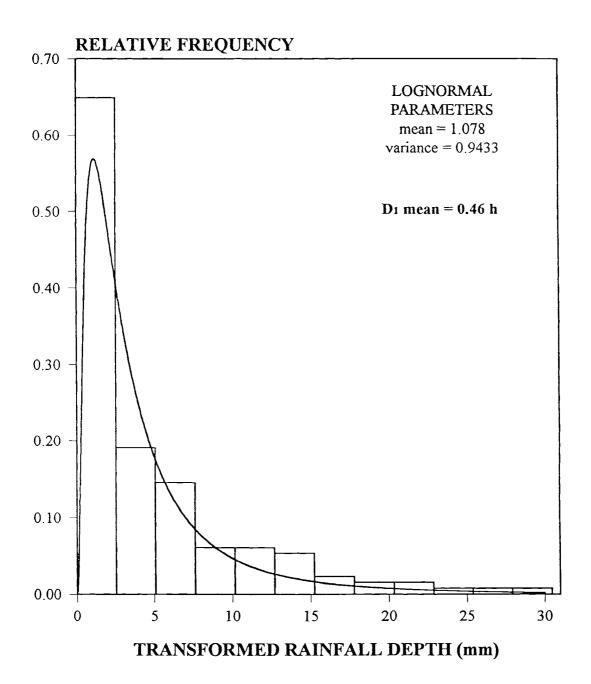


Figure 4.4 Relative frequency histogram of transformed rainfall depths that occured within the storm duration interval represented by D₁.

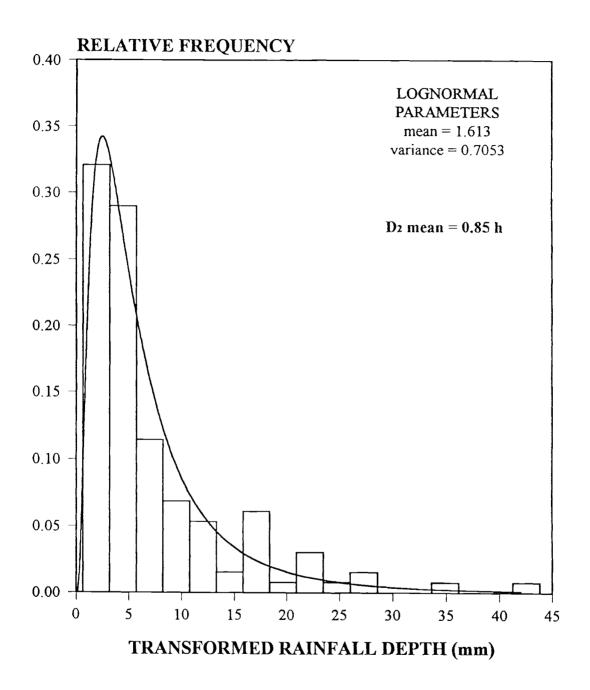


Figure 4.5 Relative frequency histogram of transformed rainfall depths that occured within the storm duration interval represented by D₂.

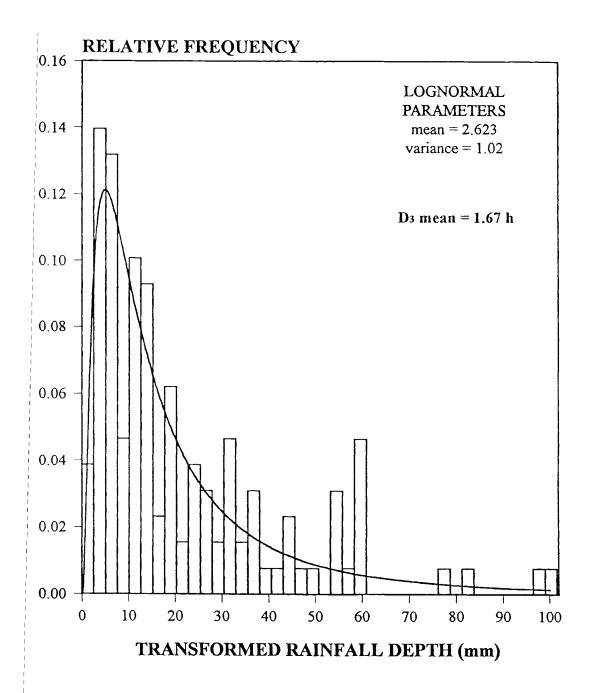


Figure 4.6 Relative frequency histogram of transformed rainfall depths that occured within the storm duration interval represented by D₃.

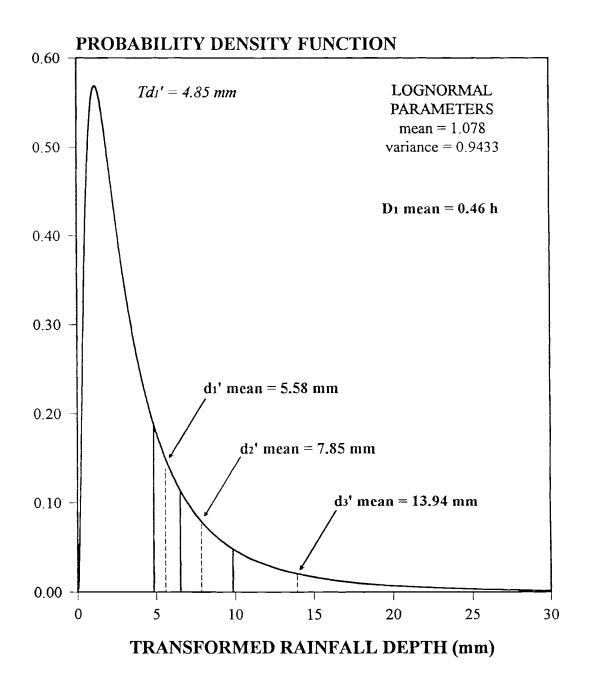


Figure 4.7 Defined mean rainfall depths that correspond to D₁ by calculating the first moment of each area interval with respect to the origin.

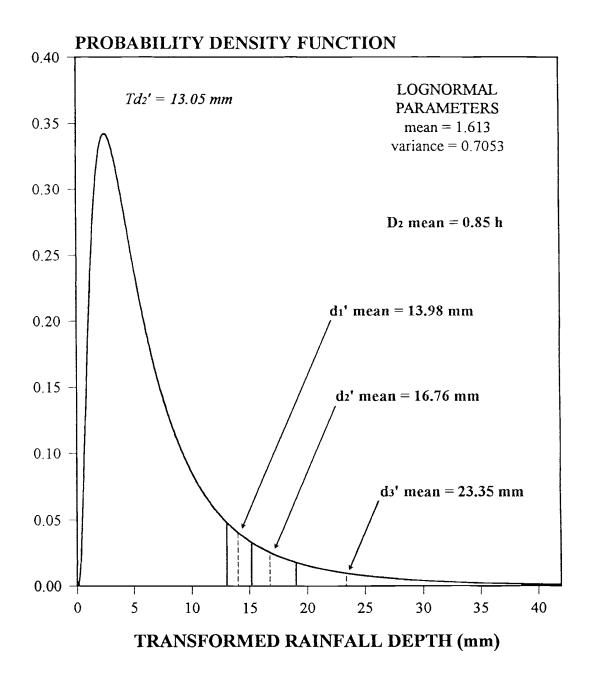


Figure 4.8 Defined mean rainfall depths that correspond to D₂ by calculating the first moment of each area interval with respect to the origin.

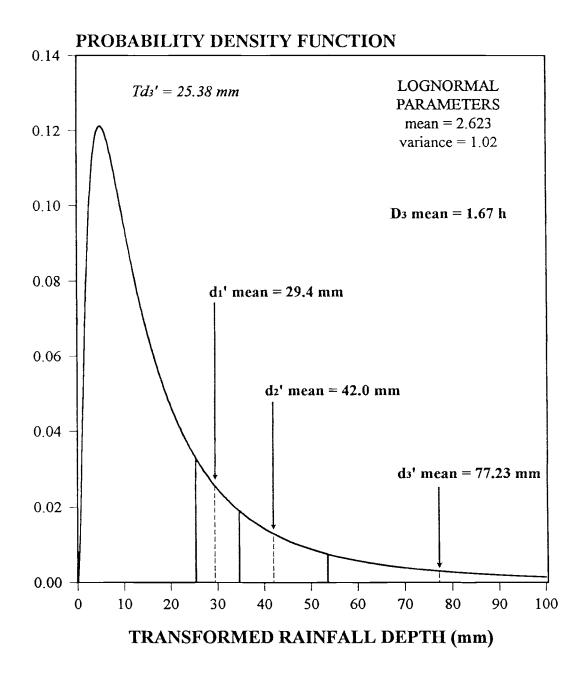


Figure 4.9 Defined mean rainfall depths that correspond to D_3 by calculating the first moment of each area interval with respect to the origin.

$\overline{\mathbf{D}}_1 = 0.44 \ \mathbf{h}$						
Depth Interval	Prob	Lower Bound	Upper Bound	Mean d'	Mean * d (mm)	Intensity (mm/h)
d1	0.099	4.85	6.53	5.58	9.39	20.6
d_2	0.099	6.53	9.88	7.85	11.7	25.6
d ₃	0.099	9.88	∞	13.9	17.8	38.9

Table 4.2 Mean Rainfall Depths and Intensities Defined for Storm Duration Intervals

* $\overline{d} = \overline{d}' + 3.81$

 $\overline{\mathbf{D}}_2 = 0.85 \text{ h}$

Depth Interval	Prob	Lower Bound	Upper Bound	Mean d'	Mean * d (mm)_	Intensity (mm/h)
dı	0.029	13.1	15.2	14.0	17.2	20.1
d_2	0.029	15.2	19.0	16.8	19.9	23.3
d ₃	0.029	19.0	x	23.4	26.5	31.1

* $\overline{d} = \overline{d'} + 3.18$

$D_3 =$	1	.67	h

Depth Interval	Prob	Lower Bound	Upper Bound	Mean d'	Mean * d (mm)	Intensity (mm/h)
d_1	0.091	25.4	34.7	29.4	35.8	21.4
d ₂	0.091	34.7	53.6	42.0	48.4	29.0
d ₃	0.091	53.6	00	77.2	83.6	50.1

* $\overline{d} = \overline{d}' + 6.35$

Table 4.3	Depth-Duration Desig	n Storm Matrix
	- opin Duranon Doolg	

		Storm Depth, mm				
Storm Duration	dı	d ₂	d3			
$\overline{\mathrm{D}}_{\mathrm{I}} = 0.44 \mathrm{h}$	9.4	11.7	17.8			
$\overline{D}_2 = 0.85 \text{ h}$	17.2	19.9	26.5			
$\overline{\mathrm{D}}_3 = 1.67 \mathrm{h}$	35.8	48.4	83.6			

Table 4.4 Intensity-Duration Design Storm Matrix

		Storm Intensity, mm/h				
Storm Duration	i ₁	i ₂	i ₃			
$\overline{\mathbf{D}}_1 = 0.44 \text{ h}$	20.6	25.6	38.9			
$\overline{\mathrm{D}}_2 = 0.85 \mathrm{h}$	20.1	23.3	31.1			
$\overline{\mathrm{D}}_3$ = 1.67 h	21.4	29.0	50.1			

CHAPTER V

PROCEDURES

EXPERIMENTAL DESIGN

This laboratory study employed a 3x3x3x2 factorial design where rainfall duration, rainfall intensity, slope, and vegetation height; respectively, were investigated at levels defined in the following sections. Also, the application rate of poultry litter is discussed but not investigated as a variable.

Storms Selected for Laboratory Experiment

The intensity-duration matrix shown in table 4.4 shows intensities across durations that were very similar with the exception of the 50.1 mm/h intensity storm. Therefore, to maximize the efficiency of the experiment, i.e. minimize the number of rainfall events, three levels were used instead of nine in order to obtain replications of rainfall intensity. Considering the fact that adding litter to the plots would decrease runoff, the lowest intensity used in the experiment was 25 mm/h which is about the average of the second column of intensities. The highest intensity used was 51 mm/h which is very close to the last intensity shown in column three but it is also a multiple of 25. The moderate intensity was 38 mm/h, which is the midpoint between 25 and 51 mm/h.

The storm durations chosen for the experiment were kept at three levels, but the magnitudes were modified. The durations originally developed had equal probabilities of occurrence if rainfall occurred. Because this experiment was the first of this nature to consider a range of intensities as well as durations, it was more desirable to consider a larger range of durations that represented high as well as low probabilities of occurrence. The design storm durations computed by the method demonstrated in Chapter IV were not used in this experiment except for the 26 minute duration. Knowledge of how management practices affect runoff water quality across a wide range of storm durations will provide critical information in the design of future field studies.

For this experiment, the 0.44 h (26 minutes) duration would represent the short duration events. The longest duration defined as 1.67 h (100 minutes) would be roughly doubled to 204 minutes to represent long duration storms. The midpoint between the two extremes (roughly 84 minutes) would be the moderate duration. Probabilities of storm durations occurring within the intervals represented by 26, 84, and 204 minutes are 0.43, 0.39, and 0.12, respectively. The three levels of rainfall intensities and rainfall durations are summarized as follows:

Rainfall Intensity @ 25 mm/h for durations of 26, 84, and 204 minutes

Rainfall Intensity @ 38 mm/h for durations of 26, 84, and 204 minutes

Rainfall Intensity @ 51 mm/h for durations of 26, 84, and 204 minutes

Table 5.1 shows depth/duration and return periods of events obtained from a rainfall frequency atlas of the United States (United States Department of Commerce, 1961) for Delaware county in Eastern Oklahoma. To compare the design storms used in the

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experiment to those obtained from rainfall frequency atlas, the design storm's depth/duration are given below.

Duration: 26 minutes (0.43 h)	Depths: 11 mm, 17 mm, and 22 mm
Duration: 84 minutes (1.4 h)	Depths: 36 mm, 53 mm, and 71 mm
Duration: 204 minutes (3.4 h)	Depths: 86 mm, 130 mm, and 173 mm

Table 5.1	Depth/duration and	return periods f	or Delaware County

Return Period (years)	Duration (h)	Depth (mm)
1	0.5	30
2	0.5	38
1	1	38
2	1	46
5	1	58
1	2	46
2	2	58
5	2	71
10	2	84
25	2	97
50	2	109
1	3	51
2	3	61
5	3	81
10	3	94
25	3	109
50	3	119
100	3	135

Slope and Vegetation Height

This laboratory experiment investigated slopes at levels of 5, 10, and 15 percent. These levels are multiples of five that incrementally cover the range of slopes adopted by the SCS in 1990 where poultry litter was not recommended to exceed 200 lb P_2O_5 /acre on slopes less than 15 percent. Different slopes were obtained by tilting the rainfall simulator's floor to the appropriate angle.

Vegetation heights used represent short and tall conditions at levels of 75 and 150 mm, respectively. In this experiment the vegetation was clipped using hand shears. The percent cover and forage yields were also estimated for each fescue box used in the experiment. The forage data are contained in Appendix B were sampling procedures are discussed as well.

Poultry Litter Application

For this experiment, the application rate was fixed at 6.7 Mg/ha. Before the litter was applied to the fescue boxes it was completely thawed and thoroughly mixed. The exposure time after application was not to be a variable in this experiment so litter was applied immediately before rainfall was applied. A digital balance was used to weigh 673 and 400 grams of litter from the plastic bag. The sample weighing 673 grams was applied to an individual fescue box. The litter was applied by hand to the box surface with special attention devoted to the uniformity of application. The sample weighing 400 grams was stored at 4° C for approximately four months until the litter analysis was completed. Litter samples obtained from two fescue boxes were composited in order to decrease the number of samples for analysis.

Analysis of Litter Samples

Litter samples (400 grams) were collected at the same time that litter was applied to the fescue boxes. The litter sample, mentioned in the previous section, was taken to the laboratory for analysis of TKN, TP, and percent moisture. Table 5.2 shows TKN, TP, and percent moisture for the litter applied to the fescue boxes.

Table 5.2

Poultry Litter Analysis

Sample No.	Moisture (%)	N, wet (%)	P, wet (%)	N, dry (%)	P, dry (%)
1	25	4.27	1.22	5.69	1.58
2	20	4.35	1.23	5.44	1.59
3	19	4.45	1.18	5.49	1.53
4	21	4.25	1.20	5.38	1.56
5	21	4.13	1.20	5.23	1.55
6	23	4.25	1.21	5.51	1.56
7	20	4.35	1.23	5.43	1.58
8	23	4.48	1.23	5.82	1.59
mean	21.5	4.32	1.21	5.50	1.57

ANTECEDENT SOIL MOISTURE

Antecedent soil moisture conditions can become a problem in conducting any type of hydrologic experiment differing antecedent soil moisture conditions can add considerable variability to an experiment. From the infiltration experiments, steady-state infiltration data are listed for every fescue box in Appendix A, table a.1; the arithmetic mean was 19 mm/h with a standard deviation of 12.7. Most of the infiltration rates were grouped closely about the mean. However, a considerable range existed between box 23 (1 mm/h) and box 16 (49 mm/h). These infiltration rates can change over time but the high variability among fescue boxes observed from the infiltration experiments was high.

In an effort to reduce this variability, the boxes were thoroughly saturated and allowed to drain 72 hours prior to applying poultry litter. Vegetation was also cut to the appropriate height prior to saturating the boxes. From the thirty fescue boxes available for use as experimental units, only 18 were needed to satisfy the number of treatment combinations. From a total of thirty, 18 fescue boxes were assigned at random to each treatment combination. The steady state infiltration rates of the boxes selected for the experiment are shown in Appendix A, table a.2.

RAINFALL SIMULATOR RUNS

The experiments were conducted during the month of December, 1994. Average high temperatures inside the greenhouse were approximately 24° C and lows about 16° C. After a 72 hour draining time and litter applied to the fescue box, rainfall was applied at the appropriate design storm. The boxes were placed in the rainfall simulator by a forklift. A plastic tarp was attached to the front of the fescue box and extended over the runoff weighing system to shield it from stray rainfall. The rainfall simulator was calibrated daily throughout the experiment. In addition, each rainfall simulator run was checked with a small volumetric rainguage to maintain consistency among design storms.

Collecting Runoff Samples

All fescue boxes received rainfall for a total of 204 minutes. At the end of 26 minutes, the inner tank was removed and the runoff was allowed to flow into an intermediate tank which was removed at 84 minutes. The rest of the runoff was collected in the main tank for the remaining 204 minutes. Each tank was stirred vigorously with a variable speed electric stirrer for 2 to 3 minutes. After the runoff was thoroughly agitated, a parastolic pump was used to gather a 0.5 liter composite sample from each tank. The composite samples were then treated with 0.5 ml of concentrated sulfuric acid, a small portion (45 ml) of the runoff was filtered for the analysis of inorganic constituents and the remainder stored at 4°C until totals were analyzed. Detailed sample handling procedures are described in Appendix C.

<u>Sample Analysis</u>

Samples were analyzed using colorimetric methods in a spectrophotometer. Samples were handled and analysis was conducted according to specified quality control guidelines described at the end of each procedure located in Appendix C where detailed laboratory procedures for the analysis of ammonia, nitrate, total Kjeldahl nitrogen, ortho-phosphate, total phosphorus, and total solids are contained.

Background Levels

Before treatments were applied to the fescue boxes, background levels of the nutrients considered were obtained from each box to serve as a control for the experiment. A design storm of 38 mm/h for a duration of 30 minutes was used to collect the background samples. The results from the analysis of background levels for are shown in table 5.3. Levels of NH₃ were higher than expected due to turbidity problems. The sample volumes were increased to lower the detection limit in an effort to pick up trace NH₃. This was not successful, the actual levels of NH₃ were nearly zero.

Treatment				_						
Box #	Inten (mm/h)	VegH (mm)	Slp (%)	Runoff (liters)	NH₃* (mg/l)	TKN (mg/l)	NO3 (mg/l)	PO ₄ (mg/l)	TP (mg/l)	TS (mg/l)
26	25	75	5	3.6	2.1	12	0	0	0	1500
23	25	150	5	5.0	1.8	14	0	0	0	3600
28	38	75	5	3.2	1.8	13	0	0	0	4300
30	38	150	5	3.2	2.1	11	0	0	0	4200
13	51	75	5	1.4	2.8	14	0	0	0	3700
4	51	150	5	3.0	1.8	13	0	0	0	4400
3	25	75	10	3.4	1.8	14	0	0	0	4300
27	25	150	10	4.0	1.8	14	0	0	0	4300
9	38	75	10	2.3	1.8	14	0	0	0	4200
7	38	150	10	2.0	1.8	12	0	0	0	1800
16	51	75	10	1.5	1.6	14	0	0	0	3500
11	51	150	10	1.7	1.2	12	0	0	0	3800
17	25	75	15	2.2	2.1	14	0	0	0	4100
5	25	150	15	2.8	1.8	13	0	0	0	3700
29	38	75	15	4.5	1.6	11	0	0	0	4100
19	38	150	15	4.2	1.8	14	0	0	0	4000
8	51	75	15	3.1	1.6	13	0	0	0	3900
10	51	150	15	1.5	1.6	11	0	0	0	3700

 Table 5.3
 Concentrations in Runoff (before the listed treatments were applied)

* Concentrations of NH_3 are misleading, the background samples contained very small amounts of NH_3 . The detection limits for the Nessler method are 0.4 mg/l. Since the samples contained very low levels of NH_3 , the sample volumes used in the analysis were increased to increase the mass NH_3 . Increasing the sample volumes produced interferences due to turbity. The levels of NH_3 in the samples were very small, no color was observed when the analysis was performed. However, the background samples were cloudy (turbid) which yielded a higher absorbence in the spectrophotometer than the blank sample. The increase in absorbence and thus an increase in sample concentration is attributed to turbidity in the sample and not color developed from the presence of NH_3 .

CHAPTER VI

RESULTS AND DISCUSSION

DATA ANALYSIS

The first statistical test performed on the data was an analysis of variance, ANOVA. The ANOVA is an arithmetic process for partitioning a total sum of squares into components associated with recognized sources of variation (Steel and Torrie, 1980). The terms' main effects (treatment variables) and response variable are commonly used in the discussion of an ANOVA. Recognized sources of variation of an ANOVA are treatment variables or main effects. These two terms are used interchangeably. Designations given to the treatment variables in this experiment are listed below along with the corresponding abbreviations used in the statistical analysis.

> Rainfall Duration (Dur) Rainfall Intensity (Inten) Vegetation Height (VegH) Slope (Slp)

Response variables are the measured parameters on which variation due to treatment variables are observed. The response variables measured in this experiment are listed below. Runoff components are listed by the abbreviations first followed by the common name in parenthesis. All quantitative measures of nitrogen and phosphorus forms are reported as actual N and P.

Runoff Volume NH₃-N (ammonia-N) NO₃-N (nitrate-N) TKN (total Kjedahl nitrogen) PO₄-P (soluble phosphorus) TP (total phosphorus) TS (total solids)

Due to the size and scope of the experimental design, i.e. the number of treatment combinations and sample analysis required, replicating treatments was not feasible because of available manpower, availability of additional fescue boxes, and time constraints. Since this experiment was not replicated, there is only one set of measured response variables per treatment combination. This does not allow a measurement of the error between fescue boxes because every box received a different combination of treatments. The experimental error includes the variation among fescue boxes except that due to treatments.

An assumption of the ANOVA is treatment affects on measured observations are additive. If there is no replication of the experiment and thus no measure of experimental error, the highest order interaction can be used for the experimental error. However, the assumption that there is no three-factor (Inten x VegH x Slp) interaction is required to use this as the error. Since treatment effects are additive then the mean square of Inten x

51

VegH x Slp would contain the mean square error plus any effects due to a three-factor interaction. Even though the assumption was made of no interaction, the mean square of Inten x VegH x Slp will be larger than the mean square error. For this reason the statistical tests will be rather conservative.

The significance levels as indicated by the ANOVA are presented as one of four symbols for every response variable. Tables of significance indicate (***) when probabilities of a greater F are less than 0.025, (**) when probabilities of a greater F are less than 0.05, (*) when probabilities of a greater F are less than 0.10, and (ns) to indicate not significant.

The F test is a simultaneous testing of hypotheses about many differences in means with the overall rejection on one or more unspecified differences. In other words, the null hypothesis of the ANOVA F test is equal treatment means. If this null hypothesis is rejected at the specified level of significance (p = 0.05), then at least one of the means was statistically different (p < 0.05). To identify which treatment means were significantly different, a Fisher's protected lsd (least significant difference) was performed on treatment means that were declared unequal from the ANOVA. To remain consistent thoughout the analysis, the lsd was determined at $\alpha = 0.05$ for treatment means declared unequal at p = 0.05 from the ANOVA. Due to the conservatism in the statistical tests, treatment effects that were significant at p = 0.1 were also reported in the ANOVA summary tables.

To statistically evaluate the significance of duration as an experimental variable, data from all three storm events were combined into one data set and duration was designated as a fourth variable. Inten x VegH x Slp x Dur was used as the error term to test the significance of duration on response variables. The same assumptions apply that were stated previously involving the use of a high order interaction as an error term in the ANOVA.

In the analysis of the data, more emphasis is placed on data from each of the storm durations rather than considering duration as a variable. Because, from each fescue box, runoff samples for the 26, 84, and 204 minute durations were obtained. This means that samples obtained from the treatment combinations of Inten, VegH, and Slp are independent. However samples obtained from durations of 26, 84, and 204 minutes at the same levels of Inten, VegH, and Slp are not independent because those samples came from the same fescue box.

ZERO RUNOFF VOLUME

Two of the fescue boxes did not produce runoff at 26 minutes. Runoff volumes were zero for the following treatment combinations:

Inten @ 25 mm/h, Slp @ 5 percent, and VegH @ 150 mm

Inten @ 51 mm/h, Slp @ 5 percent, and VegH @ 150 mm

For the ANOVA procedure, the zero runoff volumes and zero response variables were included from the 26 minute durations. Performing a Fisher's protected lsd at $\propto = 0.05$ on treatment means (zero values excluded) that were highly significant in terms of the ANOVA (where zero values were included) showed many mean response variables to not be significant. There were no zeros to represent response variables at 84 and 204 minutes, and thus the ANOVA and the Fisher's protected lsd procedure were more in agreement.

RUNOFF VOLUMES

Runoff volumes were influenced greatest by the level of rainfall intensity (p < 0.025) for all three durations (figure 6.1). Slope was significant in influencing mean runoff volumes at 26 (p < 0.1) and 84 (p < 0.05) minute durations. For 84 minute durations, mean runoff volumes from 15 percent slopes were significantly different than means at 10 and 5 percent (20, 15, and 13 liters, respectively). Figure 6.2 shows the effect of slope on mean runoff volumes across durations. Vegetation height was not significant at durations of 26 and 204 minutes. Vegetation height was significant for 84 minute durations with a runoff volume mean of 14 liters for the tall vegetation (150 mm) and 19 liters for the short vegetation height. Figure 6.3 shows runoff volume means for the levels of fescue height for all three durations. The results of the ANOVA and significance levels are summarized in table 6.1. Table 6.2 is a summary of treatment means that were significantly different ($\alpha = 0.05$) and the trends observed as levels of each main effect were increased.

·	Rainfall Duration (min)					
treatment variable	26	84	204			
Rainfall Intensity	***	***	***			
Vegetation Height	*	**	*			
Slope	*	**	ns			
Rainfall Intensity x Vegetation Height	**	* *	*			
Rainfall Intensity x Slope	**	*	ns			
Slope x Vegetation Height	ns	**	***			

Table 6.1 Summary of ANOVA for Total Runoff Volumes From all Storm Durations

*** p < 0.025, ** p < 0.05, * p < 0.1

Table 6.2 Significant* Trends for Runoff Volume Means

Rainfall Duration (min)			
26	84	204	
+	+	+	
	-		
	+		

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

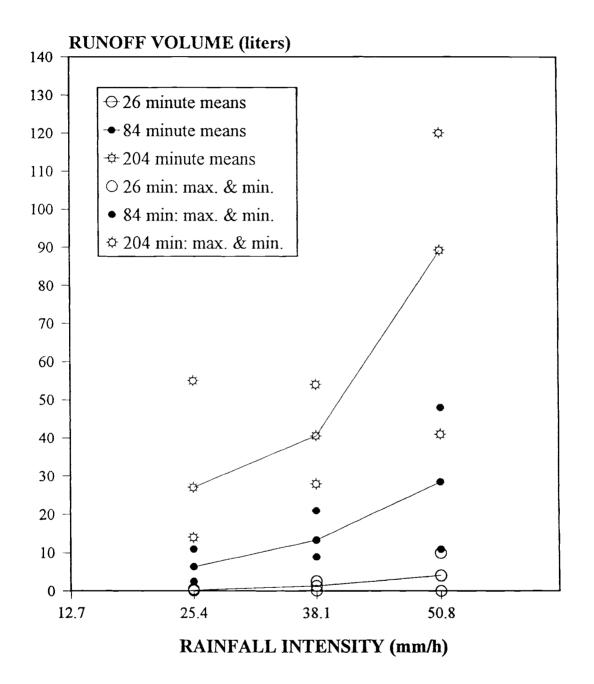


Figure 6.1 Mean, maximum, and minimum runoff volumes at each rainfall intensity for 26, 84, and 204 minute durations.

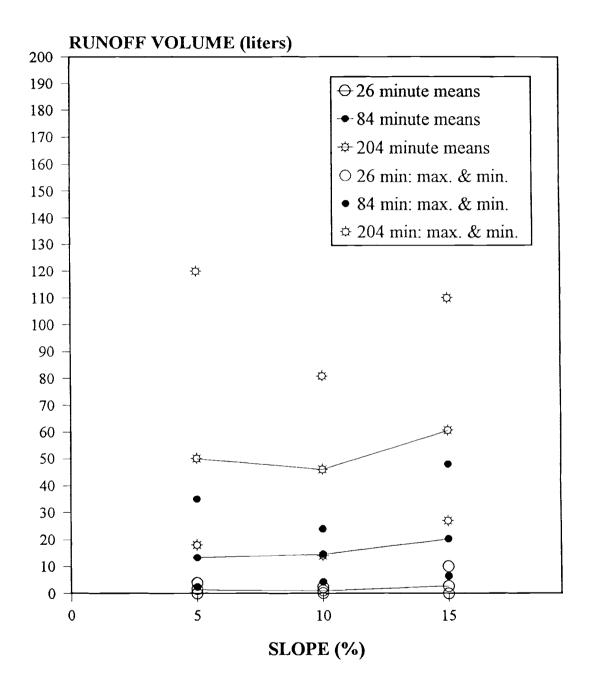


Figure 6.2 Mean, maximum, and minimum runoff volumes at each slope for 26, 84, and 204 minute durations.

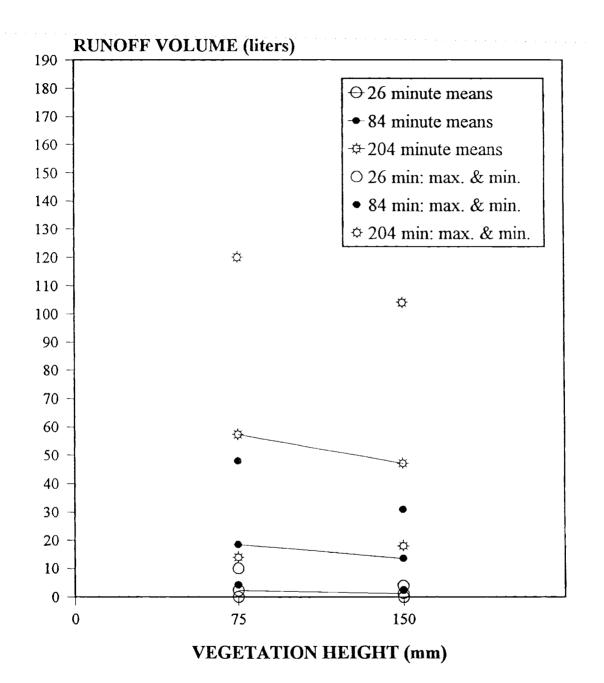


Figure 6.3 Mean, maximum, and minimum runoff volumes at both vegetation heights for 26, 84 and 204 minute durations.

26 MINUTE DURATIONS

Mass Transport

Table 6.3 shows a summary of the ANOVA for mass nutrient transport from the 26 minute durations. All treatment variables were significant for mass transport of all runoff components except NO₃-N. The interactions Inten x VegH and Inten x Slp were also significant for all nutrient mass transport with the exception of NO₃-N. Slp x VegH was not significant. Fisher's protected lsd on significant treatment means where zero values were excluded is summarized in table 6.4.

Table 6.3

26 Minute Design Storm	Summary of ANO	VA for Mass	Transport
------------------------	----------------	-------------	-----------

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	***	***	ns	***	***	***
Vegetation Height	**	**	ns	**	**	**
Slope	**	**	ns	**	*	*
Rainfall Intensity x Vegetation Height	**	*	ns	*	*	**
Rainfall Intensity x Slope	**	***	ns	**	**	**
Slope x Vegetation Height	ns	ns	ns	ns	ns	ns

*** p < 0.025, ** p < 0.05, * p < 0.1

Table 6.4

treatment variable NH₃-N TKN NO₃-N PO₄-P TP TS Rainfall Intensity ++++ +Vegetation Height Slope ++

Significant* Trends of Mass Transport Means for 26 Minute Durations

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

Increases in rainfall intensity strongly influenced increases in mass transport. All mass loading means increased by a factor of about 2.5 when rainfall intensity was increased from 38 mm/h to 51 mm/h. Increases in mean mass loading did not follow the same pattern when rainfall intensity was increased from 25 to 38 mm/h. For NH₃-N, PO₄-P, and TP, increases in mass loading were about 7.5 times higher for the 38 mm/h intensity than for the low intensity. TKN increased by a factor of 6 as rainfall intensity was increased from 25 to 38 mm/h and total solids increased by a factor of 4. A summary of significant mass loading means at each rainfall intensity is shown in table 6.5a.

Vegetation height had no significant effects on mass loading means for NH₃-N or total solids. Significant differences in means were observed for TKN, PO₄-P, and TP.

Nearly three times more PO_4 -P mass and TP mass were transported from boxes where the fescue was trimmed to 75 mm compared to the 150 mm height. Also, twice as much TKN mass was transported from the short vegetation than from the tall. Table 6.5b shows means from 26 minute durations of significant mass nutrients and the vegetation height.

The effect of slope was only significant for TKN and TP. Mass TKN loading means at 15 percent were significantly higher than both means at 10 and 5 percent. Mass loading for TP was also significantly higher at 15 percent slopes than at 5 percent. Slopes of 10 percent were not significantly different than either 5 or 15 percent slopes. Table 6.5c summarizes the effect of slope on TKN and TP. For both TKN and TP, the mean loading increased by a factor of 1.4 and 2.5 when slopes were increased from 5 to 10 percent and from 10 to 15 percent, respectively. Figures 6.4, 6.5, and 6.6 show graphically the effects of rainfall intensity, vegetation height, and slope, respectively, on means declared significantly different in terms of mass transport for all nutrient means listed in tables 6.5(a,b,c).

	Rainfall Intensity (mm/h)						
mass component	25	38	51				
NH3-N	20.4 mg	147 mg	377 mg				
TKN	63.3 mg	368 mg	903 mg				
PO₄-P	2.3 mg	17.6 mg	54.4 mg				
TP	3.6 mg	28.2 mg	71.6 mg				
TS	2,140 mg	8,770 mg	23,100 mg				

Table 6.5a Significant mass loading means at 26 minutes for each rainfall intensity

Table 6.5b Significant mass loading means at 26 minutes for the short and tall vegetation

	Vegetation Height (mm)			
mass component	75	150		
TKN	623 mg	285 mg		
PO ₄ -P	37 mg	13 mg		
ТР	51 mg	19 mg		

Table 6.5c Significant mass loading means at 26 minutes for each slope

	Slope (%)					
mass component	5	10	15			
TKN	234 mg	324 mg	820 mg			
ТР	18 mg	25 mg	64 mg			

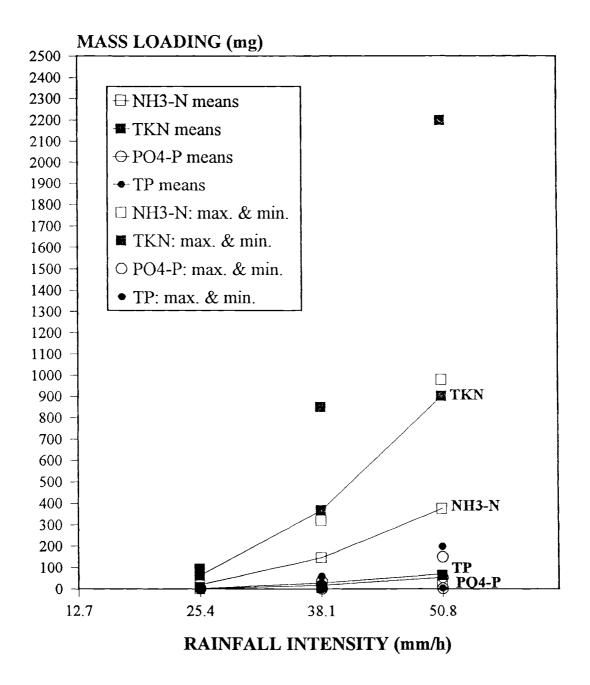


Figure 6.4 Significant mass loading means, maximum, and minimum values of nitrogen and phosphorus forms at each rainfall intensity for 26 minute durations.

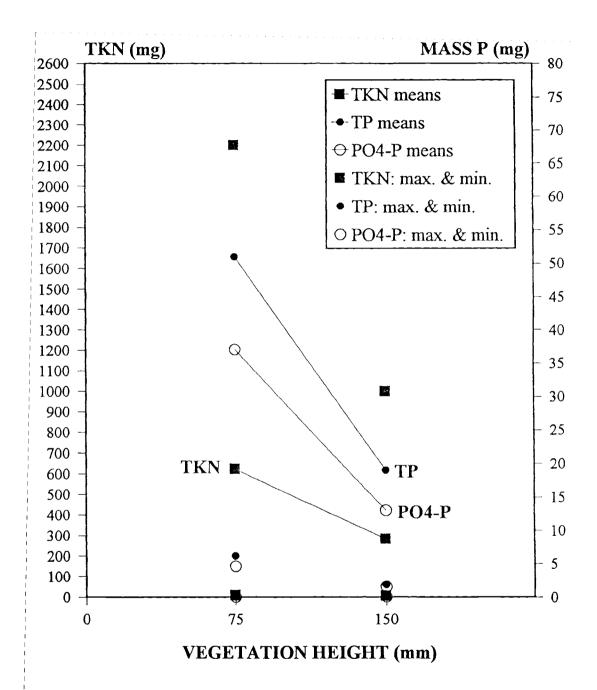


Figure 6.5 Significant mass loading means, maximum, and minimum values of nitrogen and phosphorus forms at both vegetation heights for 26 minute durations.

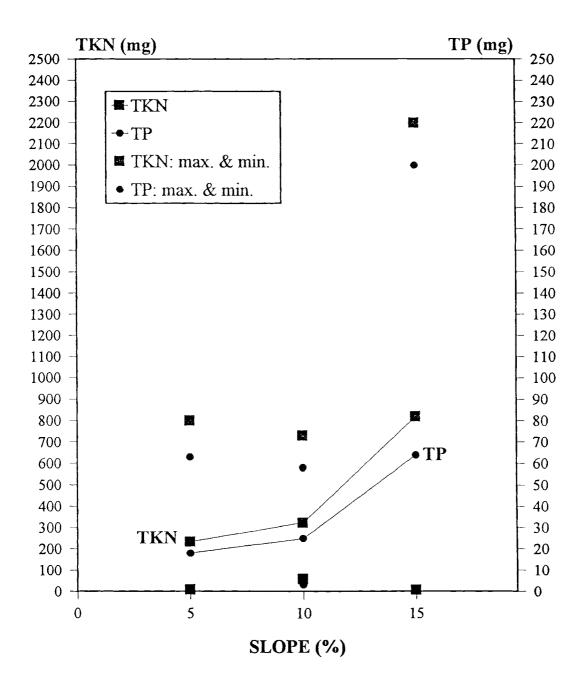


Figure 6.6 Significant mass loading means, maximum, and minimum values of TKN and TP at each level of slope for 26 minute durations.

Average Concentration

The results of the ANOVA show intensity (p < 0.10), vegetation height (p < 0.025), and slope (p < 0.025) as being significant treatment factors influencing average concentrations of TP. Also, slope was a significant (p < 0.025) factor in average concentrations of TKN and TS as well as vegetation height being significant (p < 0.10) on TKN. The number of significant variables decreased when the lsd was performed. A summary of the ANOVA and significant treatment means as well as trends for 26 minute durations are shown in tables 6.6 and 6.7, respectively.

Table 6.6

26 minute Design Storm Summary of ANOVA for Average Nutrient Concentrations

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	ns	ns	ns	ns	*	ns
Vegetation Height	ns	*	ns	ns	***	ns
Slope	ns	**	ns	ns	***	**
Rainfall Intensity x Vegetation Height	ns	ns	ns	ns	ns	ns
Rainfall Intensity x Slope	ns	ns	ns	ns	ns	ns
Slope x Vegetation Height	ns	ns	ns	ns	ns	ns

The effect of vegetation height on total phosphorus was the only significant treatment on average concentrations for 26 minute durations. The concentration of total phosphorus at the tall vegetation height (12 mg/l) which was almost half the concentration at the short vegetation height (21.4 mg/l).

Table 6.7

26 Minute Duration Trends of Significant* Nutrient Concentration Means

NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
				-	
-	NH ₃ -N	NH3-N TKN	NH3-N TKN NO3-N	NH3-N TKN NO3-N PO4-P	

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

84 MINUTE DURATIONS

Mass Transport

Rainfall intensity was highly significant (p < 0.025) for all runoff components but NO₃-N. NO₃-N was significant (p < 0.10) but not to the degree that the other components were influenced. Vegetation height was highly significant for NH₃-N (p < 0.025), PO₄-P (p < 0.05), and total solids (p < 0.025) and lesser significance (p < 0.10) for TKN and TP. Slope was highly significant for NH₃-N (p < 0.025) and significant for TKN and PO₄-P (p < 0.10). A summary of the ANOVA and significant treatment means as well as trends for 84 minute durations are shown in tables 6.8 and 6.9, respectively.

Table 6.8

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	***	***	*	***	* * *	***
Vegetation Height	***	*	ns	**	*	***
Slope	***	*	ns	*	ns	ns
Rainfall Intensity x Vegetation Height	***	*	ns	*	*	***
Rainfall Intensity x Slope	ns	ns	ns	ns	ns	ns
Slope x Vegetation Height	**	*	ns	*	*	***

84 Minute Design Storm Summary of ANOVA for Mass Transport

treatment variable	NH3-N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	+	+		+	+	+
Vegetation Height	-			-		-
Slope	+	+		+		

84 Minute Duration Trends of Significant* Mass Transport Means

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

Rainfall intensity was significantly different for all levels (25, 38, and 51 mm/h) and all runoff components considered (NH₃-N, TKN, PO₄-P, TP, and TS) with the exception of NO₃-N. Mass loading means approximately doubled for each increase in rainfall intensity. Table 6.10a and figure 6.7 presents the mean mass loading for each nutrient and the levels of rainfall intensity.

Vegetation height was significant for NH_3 -N, PO_4 -P, and total solids. Approximately, 60 % more mass was transported from the short vegetation than the tall. Table 6.10b and figure 6.8 show the mean mass loadings for each nutrient at both vegetation heights.

Slope was only significant for mass loading means of TKN, NH_3 -N, and PO_4 -P (table 6.10c). The highest mass loadings were observed at 15 percent slopes for NH_3 -N and PO_4 -P. Largest observed means for TKN were at the 10 percent slopes although the mean

loading at 15 percent slopes was not significantly different. For TKN, significant differences were observed between slopes at 5 and 10 percent. Means of nitrogen and phosphorus forms for 84 minute durations at each level of slope are graphically presented in figure 6.9.

	Raint	all Intensity (m	.m/h)	
mass component	25	38	51	
NH ₃ -N	484 mg	882 mg	1620 mg	
TKN	1230 mg	2160 mg	4150 mg	
PO ₄ -P	84 mg	169 mg	310 mg	
ТР	101 mg	208 mg	369 mg	
TS	32,100 mg	65,600 mg	141,000 mg	

Table 6.10a Significant mass loading means at 84 minutes for each rainfall intensity

Table 6.10b Significant mass loading means at 84 minutes for the short and tall vegetation

	Vegetation Height (mm)					
mass component	75	150				
NH3-N	117 mg	826 mg				
PO ₄ -P	224 mg	151 mg				
TS	90,700 mg	68,600 mg				

 Table 6.10c
 Significant mass loading means at 84 minutes for each slope

mass component	Slope (%) 5 10 15					
NH₃-N	762 mg	954 mg	1270 mg			
TKN	1990 mg	2860 mg	2700 mg			
PO ₄ -P	148 mg	181 mg	233 mg			

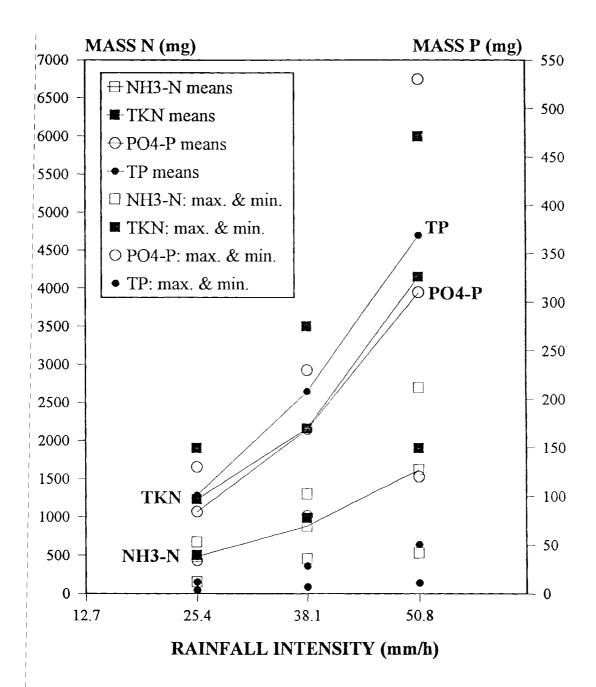


Figure 6.7 Significant mass loading means, maximum, and minimum values of nitrogen and phosphorus forms at each rainfall intensity for 84 minute durations.

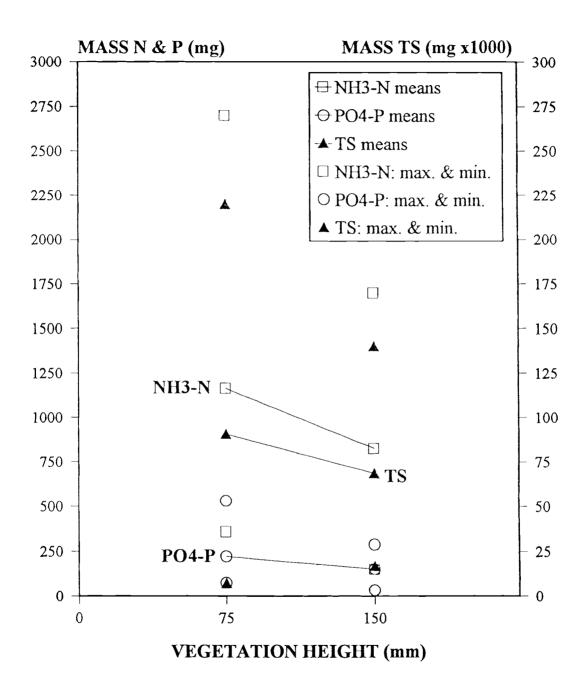


Figure 6.8 Significant mass loading means, maximum, and minimum values of NH3-N, PO4-P, and TS at both vegetation heights for 84 minute durations.

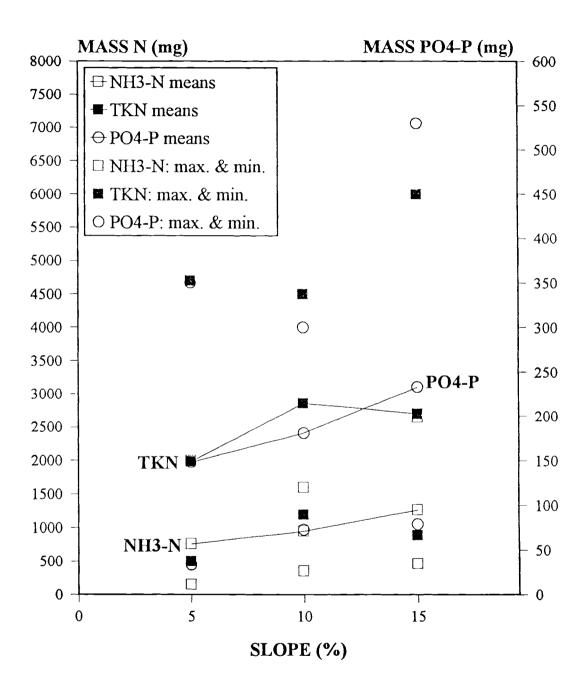


Figure 6.9 Significant mass loading means, maximum, and minimum values of nitrogen and phosphorus forms at each level of slope for 84 minute durations.

Nutrients whose average concentrations were affected for 84 minute durations were NH₃-N and TKN. Rainfall intensity was a highly significant factor for NH₃-N (p < 0.025) and significant for TKN (p < 0.10). NH₃-N (p < 0.10) was the only constituent that was affected by vegetation height at the 84 minute duration. Slope was significant for both NH₃-N (p < 0.025) and TKN (p < 0.05). A summary of the ANOVA for average concentrations and significant treatment means as well as trends for 84 minute durations are shown in tables 6.11 and 6.12, respectively.

Table 6.11

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	***	*	ns	ns	ns	ns
Vegetation Height	*	ns	ns	ns	ns	ns
Slope	***	**	ns	ns	ns	ns
Rainfall Intensity x Vegetation Height	***	ns	ns	ns	ns	ns
Rainfall Intensity x Slope	***	ns	ns	ns	ns	ns
Slope x Vegetation Height	**	ns	ns	ns	ns	ns

84 Minute Design Storm Summary of ANOVA for Average Concentrations

treatment variable	NH ₃ -N	TKN	NO3-N	PO ₄ -P	TP	TS
Rainfall Intensity	-	-				
Vegetation Height						
Slope	+	+				

Significant* Trends of 84 Minute Average Concentration Means

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

Fisher's protected lsd was performed on average concentration means from treatments that were determined to be significant from the ANOVA (table 6.11). NH₃-N concentration means that were averaged for each rainfall intensity were significantly different at $\alpha = 0.05$ for each level of intensity. The same was true for TKN average concentration means but the means averaged at each level of rainfall intensity were not significantly different. The mean TKN concentration at 25 mm/h was statistically different ($\alpha = 0.05$) than the mean at 51 mm/h but the mean concentration for 38 mm/h was not statistically different than either the low or high intensities. As rainfall intensity was increased the mean average concentration of NH₃-N and TKN decreased. Table 6.13a and figure 6.10 shows the means of NH₃-N and TKN for each level of rainfall intensity. Means averaged for each level of slope were also significantly different ($\alpha = 0.05$) for the same forms of nitrogen that were affected by rainfall intensity. Mean concentrations of NH₃-N for 5 percent slopes were different than slopes at 10 and 15 percent. For TKN concentrations, 10 percent slopes were different than both 5 and 15 percent. Table 6.13b and figure 6.11 shows the mean average concentrations for both NH₃-N and TKN for each level of slope.

Table 6.13a Significant concentration means at 84 minutes for each rainfall intensity

	Rainfall Intensity (mm/h)					
component	25	38	51			
NH3-N	76.7 mg/l	65.8 mg/l	56.6 mg/l			
TKN	202 mg/l	161 mg/l	155 mg/l			

 Table 6.13b
 Significant concentration means at 84 minutes for each slope

5	10	15
57.2 mg/l	71.8 mg/l	70.2 mg/l
159 mg/l	214 mg/l	146 mg/l
	_	

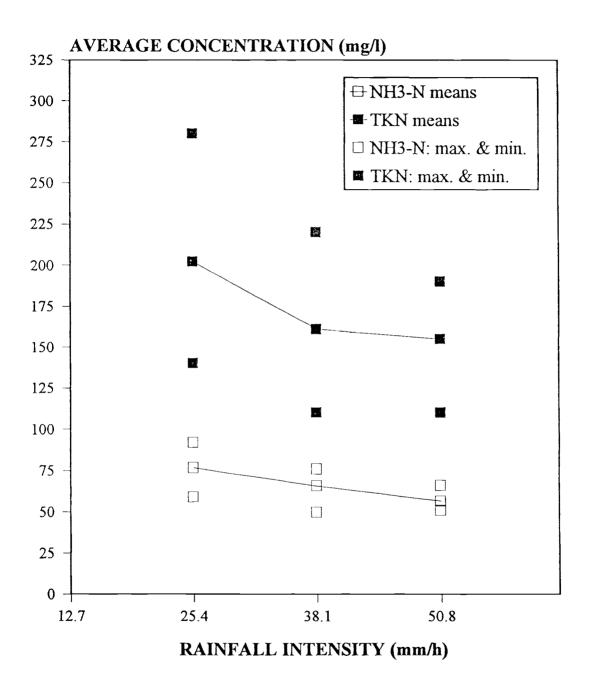


Figure 6.10 Significant concentration means, maximum, and minimum values of nitrogen forms at each rainfall intensity for 84 minute durations.

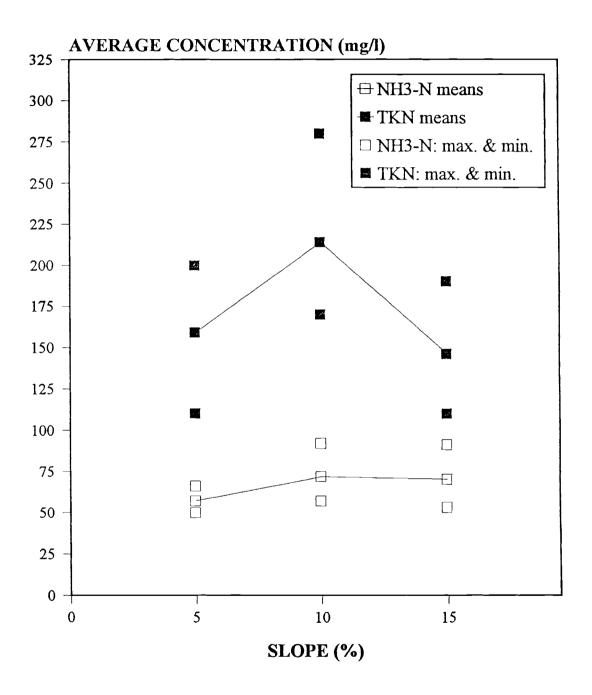


Figure 6.11 Significant concentration means, maximum, and minimum values of nitrogen forms at each level of slope for 84 minute durations.

204 MINUTE DURATIONS

Mass Transport

From the ANOVA, rainfall intensity was determined to be significant for all runoff components including NO₃-N. Vegetation height was significant for NH₃-N and TKN (p < 0.10). The significance of vegetation height in relation to PO₄-P (p < 0.05) and TP (p < 0.025) were higher than for NH₃-N and TKN. NO₃-N and total solids mean mass loadings were not affected by the height of vegetation. Slope was highly significant for NH₃-N (p < 0.025) and also significant for TKN (p < 0.10). The summary of the ANOVA and Fisher's protected lsd procedure on significant treatment means are shown in tables 6.14 and 6.15, respectively.

Table 6.14

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	***	***	***	***	***	***
Vegetation Height	*	*	ns	**	***	ns
Slope	***	*	ns	ns	ns	ns
Rainfall Intensity x Vegetation Height	*	ns	ns	ns	***	ns
Rainfall Intensity x Slope	**	***	*	ns	**	ns
Slope x Vegetation Height	***	***	ns	**	***	**

204 Minute Design Storm Summary of ANOVA for Mass Transport

treatment variable	NH3-N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	+	+	+	+	+	+
Vegetation Height				-	-	
Slope	÷	+				

Significant* Trends of Mass Transport Means from 204 Minute Durations

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

Mass loading means for each level of rainfall intensity are significantly different ($\alpha = 0.05$) for NH₃-N and TP. The mass loading mean from the highest level of rainfall intensity (51 mm/h) was significantly different ($\alpha = 0.05$) than the moderate (38 mm/h) and low (25 mm/h) intensities for TKN, NO₃-N, PO₄-P, and total solids mass. Table 6.16a and figure 6.12 shows the means of each significant nutrients mass loading at each level of rainfall intensity.

As rainfall intensity increased from 25 to 38 mm/h, most nutrient mass loading for NH_3-N , PO_4-P , TP and total solids increased by a factor of 1.3 whereas TKN increased by a factor of 1.2 and NO_3-N did not change. The mean nutrient mass loadings increased by a larger amount as rainfall intensity increased from 38 to 51 mm/h. NH_3-N , TKN, PO_4-P , and TP mass means nearly doubled from 38 to 51 mm/h. Other constituents increased by

greater amounts from the moderate rainfall to the high intensity such as total solids which increased by a factor of 2.4 and NO₃-N which increased in mass by 2.7 times the mass at the low and moderate intensities.

Differences in mass loading means due to levels of vegetation height were observed for PO_4 -P and TP. All of which had significantly greater mass loadings for the short rather than the tall vegetation height. The mass loading means for both forms of phosphorus and the height of vegetation is shown in table 6.16b and figure 6.13.

The effect of slope was significant ($\propto = 0.05$) for NH₃-N where mass transport from 15 percent slopes was greater than both 5 and 10 percent slopes, which were not significantly different. Differences in TKN mass loading means were observed between slopes of 5 and 10 percent. Means at 15 percent slopes were not statistically different than either 5 or 10 percent. Table 6.16c and figure 6.14 summarizes the effect of slope on NH₃-N mass transport at the 204 minute duration.

	Rain	Rainfall Intensity (mm/h)				
mass component	25 38		51			
NH3-N	1210 mg	1600 mg	3120 mg			
TKN	3020 mg	3510 mg	7170 mg			
NO ₃ -N	86 mg	87 mg	231 mg			
PO ₄ -P	240 mg	301 mg	588 mg			
TP	258 mg	332 mg	614 mg			
TS	125,000 mg	161,000 mg	379,000 mg			

Table 6.16a Significant mass loading means at 204 minutes for each rainfall intensity

Table 6.16b Significant mass loading means at 204 minutes for the short and tall fescue

	Vegetation Height (mm)				
mass component	75	150			
PO ₄ -P	422 mg	332 mg			
TP	444 mg	359 mg			
IP	444 mg	359 n			

Table 6.16c Significant mass loading means at 204 minutes for each slope

		Slope (%)		
mass component	5	10	15	
NH3-N	1730 mg	1800 mg	2390 mg	
TKN	4160 mg	4970 mg	4570 mg	

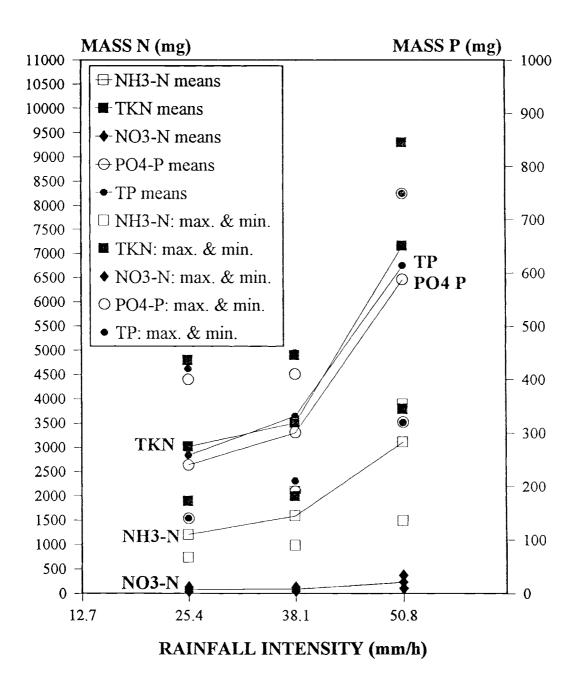


Figure 6.12 Significant mass loading means, maximum, and minimum values of nitrogen and phosphorus forms at each rainfall intensity for 204 minute durations.

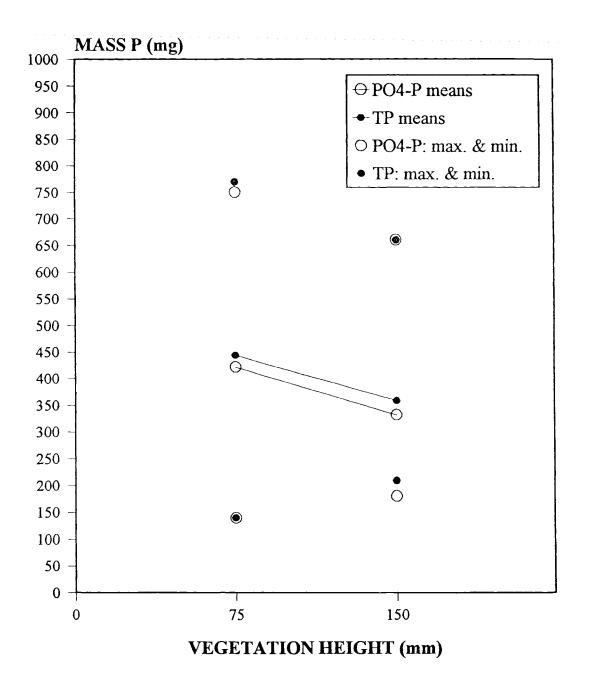


Figure 6.13 Significant mass loading means, maximum, and minimum values of phosphorus forms at both vegetation heights for 204 minute durations.

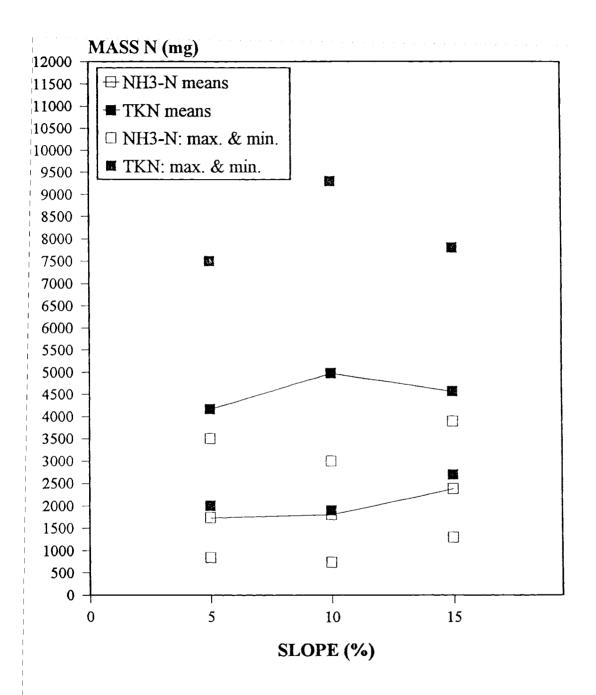


Figure 6.14 Significant mass loading means, maximum, and minimum values of nitrogen forms at each level of slope for 204 minute durations.

Average Concentration

From the ANOVA summary (table 6.17), rainfall intensity is a significant treatment factor for all nutrients. Nutrients of significance are NH₃-N and TKN (p < 0.025), NO₃-N (p < 0.05), and PO₄-P, TP, and total solids (p < 0.10). The effects of vegetation height were not apparent at the 204 minute durations. Slope was significant for all nutrients except PO₄-P and TP. Highly significant nutrients affected by slope are NH₃-N, total solids (p < 0.025) and TKN (p < 0.05). NO₃-N was not highly significant (p < 0.10) in terms of slope.

Table 6.17

treatment variable	NH ₃ -N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	***	***	**	*	*	*
Vegetation Height	ns	ns	ns	ns	ns	ns
Slope	***	**	*	ns	ns	***
Rainfall Intensity x Vegetation Height	***	*	***	ns	*	ns
Rainfall Intensity x Slope	***	ns	*	ns	ns	ns
Slope x Vegetation Height	ns	ns	*	ns	ns	ns

204 Minute Design Storm Summary of ANOVA for Average Concentrations

treatment variable	NH3-N	TKN	NO ₃ -N	PO ₄ -P	TP	TS
Rainfall Intensity	-	-	-			-
Vegetation Height						
Slope	+	+	+			-
Slope	+	+	+			-

Significant* Trends of 204 Minute Average Concentration Means

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

- decreasing trend where means are significantly different.

Fisher's protected lsd was performed on average concentration means from treatments that were determined to be significant from the ANOVA (table 6.17). The number of nutrients significantly affected by the various treatment factors decreased when means were compared for differences using the lsd test. Table 6.18 shows significant concentration means for 204 minute durations and the trends observed as the levels of each treatment variable were increased. The nitrogen forms and total solids means were statistically different when compared between rainfall intensities and slopes. Rainfall intensity decreased average concentrations of all significant nutrients. Slope had the opposite effect in that average concentrations increased as the levels of slope increased with the exception of total solids whose concentrations decreased as slope increased. Concentration means of NH₃-N were significantly different ($\alpha = 0.05$) at level of rainfall intensity. Concentration means of TKN and NO₃-N at 25 mm/h were different than means at the moderate (38 mm/h) and high (51 mm/h) intensities which were not different. Total solids concentrations at 25 mm/h were significantly different ($\alpha = 0.05$) than mean concentrations at 38 mm/h. Mean total solids concentrations at 51 mm/h were not statistically different than either of the means at the low (25 mm/h) and moderate (38 mm/h) intensities. Table 6.19a and figure 6.15 shows the mean concentrations of the significant runoff components at each level of rainfall intensity.

Mean concentrations of NH₃-N at the 5 percent slope were significantly lower than mean concentrations at 10 percent slopes. NH₃-N concentrations at 15 percent slopes were not statistically different than the 5 percent slope concentrations and but were significantly lower than concentrations at 10 percent. TKN and NO₃-N concentrations were highest at 10 percent slopes. Significant differences in means for TKN and NO₃-N were between 10 percent and both 5 and 15 percent slopes. Total solids concentrations were highest at 5 percent slopes and decreased for slopes of 10 and 15 percent. Table 6.19b and figure 6.16 shows means of the significant nutrients at 5, 10 and 15 percent slopes.

	Rainfall Intensity (mm/h)						
component	25	38	51				
NH3-N	47.9 mg/l	39.4 mg/l	35.3 mg/l				
TKN	123 mg/l	87.6 mg/l	84.2 mg/l				
NO3-N	3.53 mg/l	2.19 mg/l	2.77 mg/l				
TS	4660 mg/l	4070 mg/l	4340 mg/l				

Table 6.19a Significant concentration means at 204 minutes for each rainfall intensity

Table 6.19b Significant concentration means at 204 minutes for each slope

component	5	Slope (%) 10	15	
NH3-N	37.5 mg/l	43.6 mg/l	41.6 mg/l	
TKN	96.7 mg/l	116 mg/l	82.3 mg/l	
NO ₃ -N	2.44 mg/l	3.47 mg/l	2.59 mg/l	
TS	4700 mg/l	4540 mg/l	3830 mg/l	

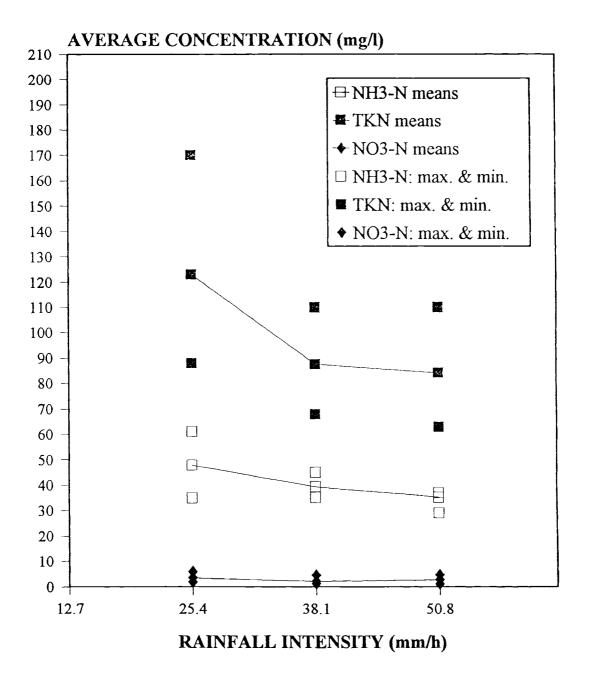


Figure 6.15 Significant concentration means, maximum, and minimum values of nitrogen forms at each rainfall intensity for 204 minute durations.

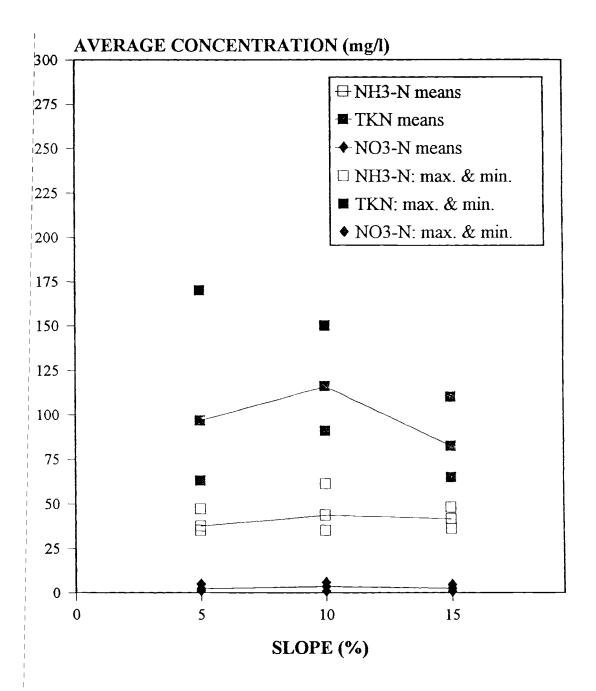


Figure 6.16 Significant concentration means, maximum, and minimum values of nitrogen forms at each slope for 204 minute durations.

NUTRIENT RATIOS

The ANOVA summary in table 6.20 shows slope being highly significant for the ratio NH₃-N/TKN for both 84 (p < 0.05) and 204 (p < 0.025) minute durations. For 84 minute durations the ratio PO_4 -P/TP was also significantly (p < 0.10) influenced by slope. Table 6.21 is a listing of means for each main effect that are significantly different using an lsd ($\propto = 0.05$) procedure on treatments that were indicated to be significant from the ANOVA. The ratio PO₄-P/TP was not significant in terms of differences in means at each slope. However, NH₃-N/TKN was significant for both 84 and 204 minute durations having increases in ratios as levels of slope were increased. Evidently, the amount of NH₃-N in relation to TKN increased as slope increased from 10 to 15 percent. At 84 minute durations no differences were observed in NH₃-N/TKN means at 5 and 10 percent slopes (0.37 and 0.34, respectively) although the mean at 15 percent was significantly different (0.48). The same relationships were observed at 204 minute durations for NH₃-N/TKN means at 5 (0.41), 10 (0.37) and 15 (0.51) percent slopes. Table 6.22 and figure 6.17 summarizes these results for each level of slope.

	Duration (min)								
		26			84			204	
treatment				_	_		_	-	<u>P₂O₄-P</u>
variable	TKN	TKN	TP	TKN	TKN	TP	TKN	TKN	<u>TP</u>
Rainfall Intensity	ns	ns	ns	ns	ns	ns	ns	ns	ns
Vegetation Height	ns	ns	ns	ns	ns	ns	ns	ns	ns
Slope	ns	ns	ns	**	ns	*	***	ns	ns
R. Intensity Veg. Height	ns	ns	ns	ns	ns	ns	ns	ns	ns
R. Intensity x Slope	ns	ns	ns	ns	ns	ns	ns	ns	ns
Slope x Veg. Height	ns	ns	ns	ns	ns	ns	ns	ns	ns

Summary of ANOVA for Nutrient Ratios at 26, 84, and 204 Minute Durations

	26			Duration (min) 84			204		
treatment variable	<u>NH3-N</u> TKN		<u>P₂O₄-P</u> TP	<u>NH3-N</u> TKN	<u>NO3-N</u> TKN	<u>P₂O₄-P</u> TP	<u>NH3-N</u> TKN	<u>NO3-N</u> TKN	<u>P₂O₄-P</u> TP
Rainfall Intensity									
Vegetation Height									
Slope				+			+		

Trends of Significant* Nutrient Ratio Means From 26, 84, and 204 Minute Durations

* significance determined by Fisher's protected lsd using $\infty = 0.05$

+ increasing trend where means are significantly different.

Table 6.22	Significant NH ₃ -N/TKI	N ratio means at	84 and 204	minutes for each slop	be
	0				

	Slope (%)				
nutrient ratio	5	10	15		
84 min NH3-N/TKN	0.37	0.34	0.48		
204 min NH3-N/TKN	0.41	0.37	0.51		
			_		

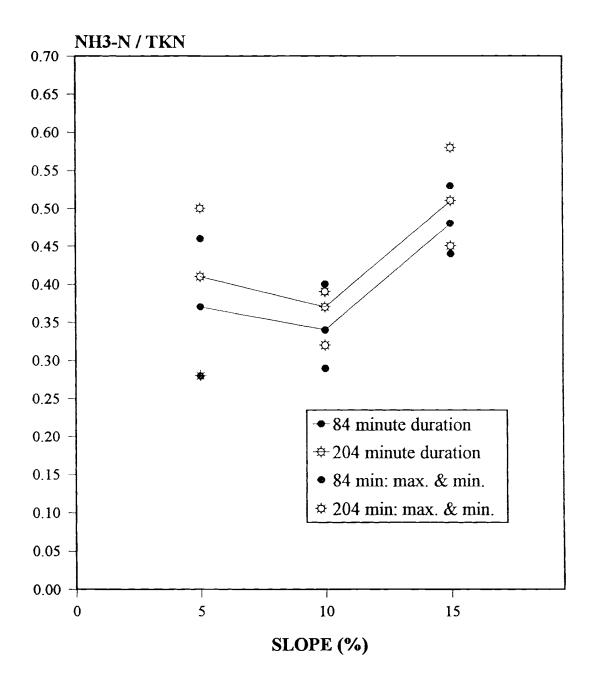


Figure 6.17 Significant nutrient ratio means, maximum and minimum values of NH3-N/TKN at each slope for 84 and 204 minute durations.

STORM DURATION EFFECTS

In order to statistically evaluate the significance of duration as an experimental variable, data from all three storm events were combined and duration was designated as a treatment variable having three levels; 26, 84, and 204 minutes. An ANOVA was performed on the combined storm data. Inten x VegH x Slp x Dur was used as the error term to test the significance of duration on each response variable. Although Inten x VegH x Slp was used as the error term to test the significance of rainfall intensity, vegetation height and slope on each response variable, the four factor interaction is more appropriate for testing all four treatment variables.

Runoff Volumes

The effect of storm duration was the most significant variable in influencing runoff volumes (figures 6.1, 6.2, 6.3). Table 6.23 shows all treatment variables in the order in which the highest significance was observed on which runoff volumes were effected.

Table 6.23

Ranking Significance of Each Treatment Variables Influence on Runoff Volumes

DURATION*	INTENSITY‡	VEGETATION HEIGHT‡	SLOPE‡		
p > F: 0.0001	p > F: 0.0006	p > F: 0.0533	p > F: 0.0751		

* Inten x VegH x Slp x Dur used as error term

‡ Inten x VegH x Slp used as error term

Mass Transport

Storm duration had the greatest impact on mass transport of all nutrients for all treatment variables (rainfall duration, rainfall intensity, slope and vegetation height). All nutrients, excluding NO₃-N, were significant as influenced by rainfall intensity for all design storm durations (26, 84, and 204 minutes). As storm duration and rainfall intensity increased, nutrient mass loadings also increased. Figures 6.18 through 6.22 show the significantly different means of each nutrient as influenced by rainfall intensity for the three storm durations. The height of vegetation significantly affected mass loading means of PO₄-P for all three of the storm durations (figure 6.23). Slope significantly influenced mass loading means of TKN for all three durations as shown in figure 6.24. In general, as storm duration and slope increased, the mass loading of TKN also increased. Table 6.24 shows the ranking significance of each response variable for each main effect where duration is included as a variable in the ANOVA.

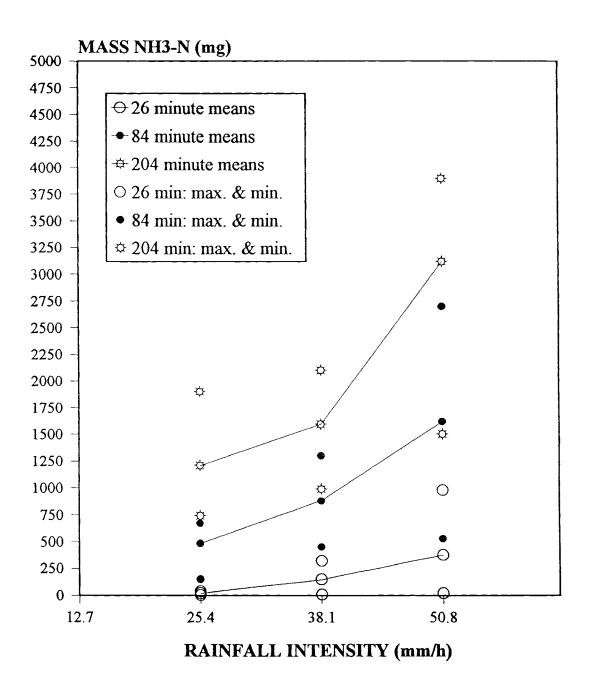


Figure 6.18 Mass loading means, maximum and minimum values of NH3-N that are significantly different at levels of rainfall intensity for every storm duration.

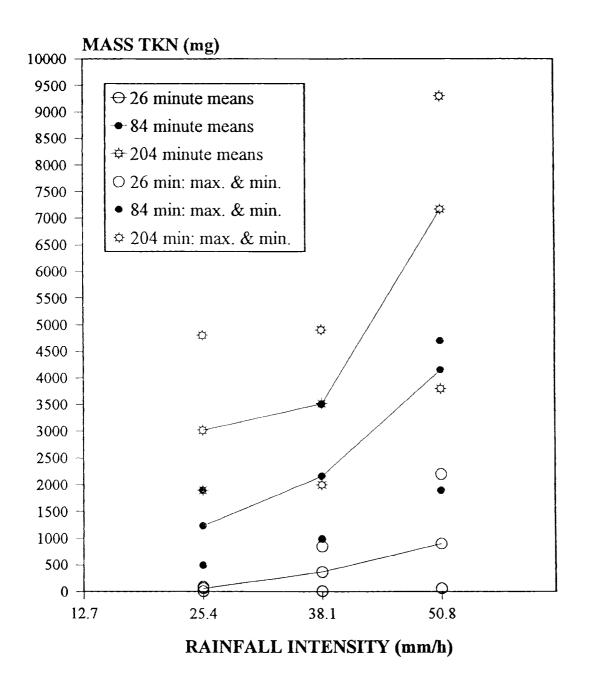


Figure 6.19 Mass loading means, maximum and minimum values of TKN that are significantly different at levels of rainfall intensity for every storm duration.

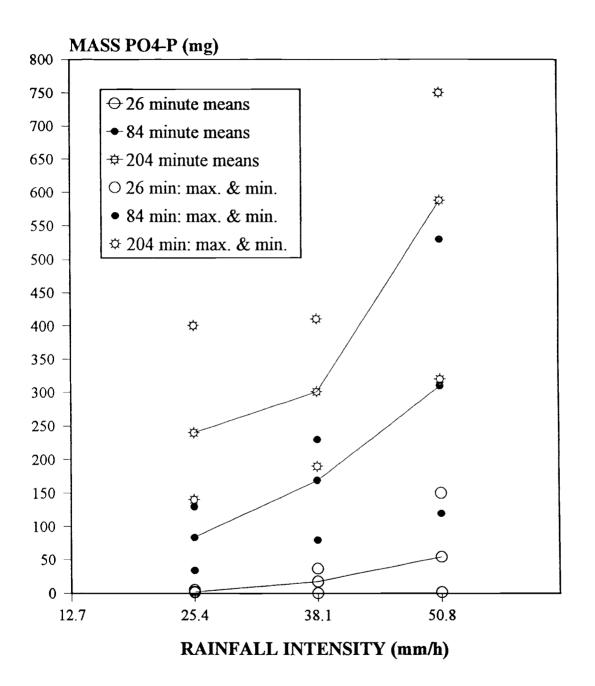


Figure 6.20 Mass loading means, maximum and minimum values of PO4-P that are significantly different at levels of rainfall intensity for every storm duration.

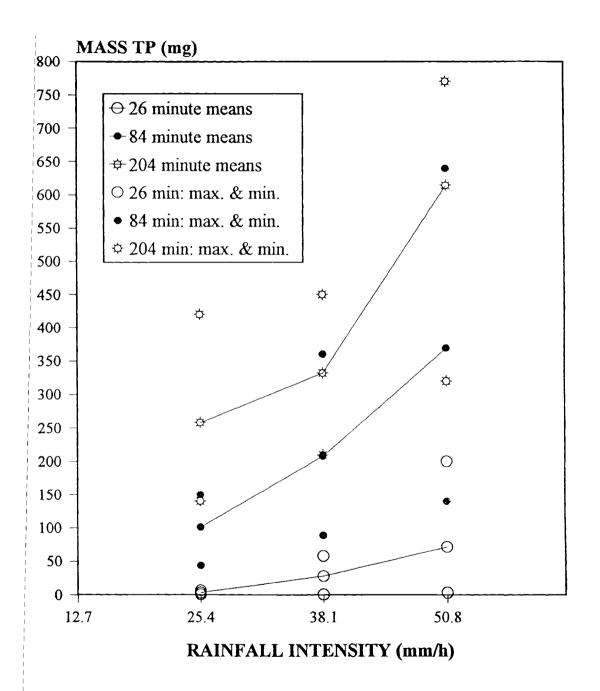


Figure 6.21 Mass loading means, maximum and minimum values of TP that are significantly different at levels of rainfall intensity for every storm duration.

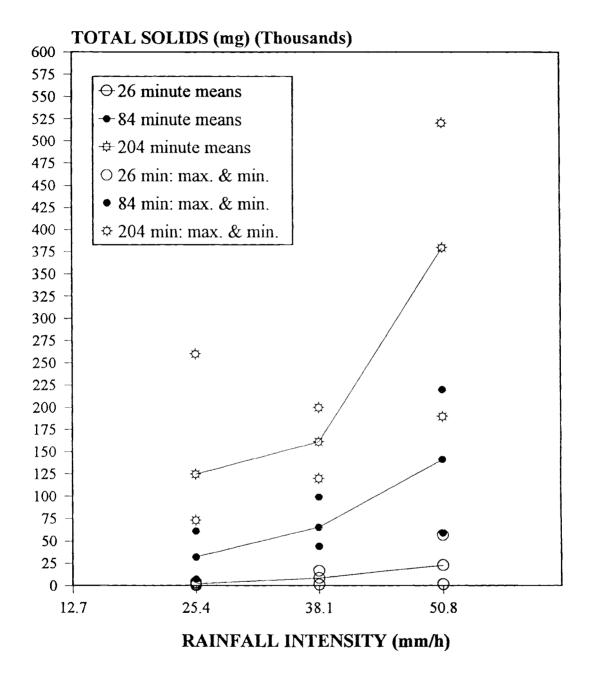


Figure 6.22 Total solids mass loading means, maximum and minimum values that are significantly different at levels of rainfall intensity for every storm duration.

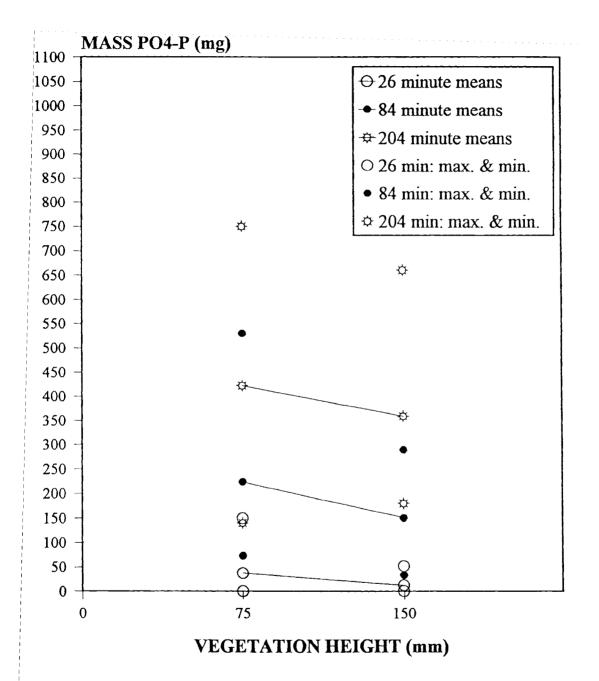


Figure 6.23 Mass loading means, maximum and minimum values of PO4-P that are significantly different at each level of vegetation height for all storm durations.

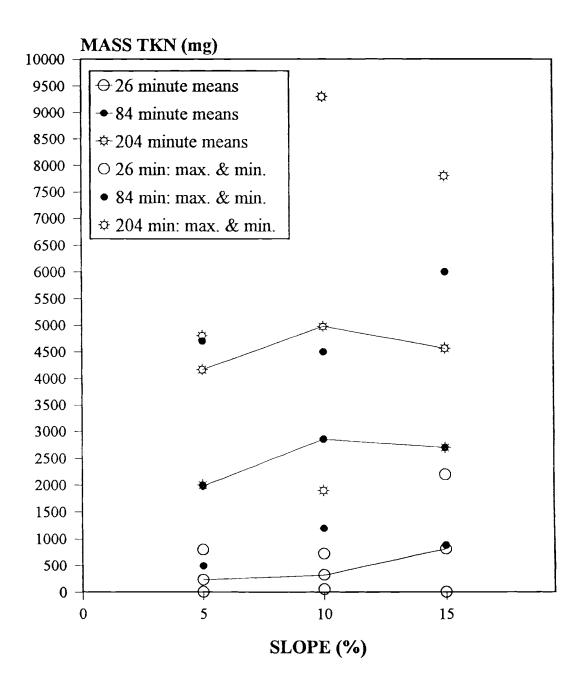


Figure 6.24 Mass loading means, maximum and minimum values of TKN that are significantly different at each level of slope for every storm duration.

Table 6.24

DURATION*	INTENSITY‡	VEGETATION HEIGHT‡	SLOPE‡		
		$\frac{NH_{3}-N}{(p < F: 0.0108)}$			
1		TP (p < F: 0.0224)			
NH ₃ -N	TS	PO_4-P	NO ₃ -N		
($p < F: 0.0001$)	($p < F: 0.0009$)	(p < F: 0.0240)	($p < F$: 0.1626)		
	TP	TKN	PO_4-P		
	(p < F: 0.0011)	(p < F: 0.0412)	($p < F: 0.1700$)		
TS	PO ₄ -P	TS	TP		
($p < F: 0.0001$)	($p < F: 0.0015$)	(p < F: 0.0490)	(p < F: 0.2243)		
NO ₃ -N	NO ₃ -N	NO ₃ -N	TS $(p < F: 0.6241)$		
($p < F: 0.0001$)	($p < F: 0.0311$)	($p < F: 0.1281$)			

Ranking Significance of Mass Nutrient Transport for Each Treatment Variable (bold type indicates significance at p < 0.10)

* Inten x VegH x Slp x Dur used as error term

‡ Inten x VegH x Slp used as error term

All factors involving rainfall (intensity and duration) were highly significant for the mass transport of all nutrients considered. The height of vegetation was significant for all nutrients except NO₃-N. Slope was only significant for NH₃-N and TKN mass loading. Since these tests of significance are rather conservative, considering that the error terms used are larger than a true error term, probabilities of a greater F that are less than 0.10 are considered significant.

Average Concentration

None of the nutrients mean concentrations were significant for the same treatment variables (rainfall intensity, vegetation height, and slope) for all three durations (26, 84, and 204 minutes). Therefore, discussions involving significance due to rainfall intensity, vegetation height, and slope will be addressed on a duration by duration basis as discussed in the previous sections of this chapter. Average concentration data were pooled for all durations and were analyzed using the ANOVA procedure and the four-factor interaction (Inten x VegH x Slp x Dur) as the error term. Storm duration had the greatest impact on average concentrations of all nutrients considered over all other experimental variables (rainfall intensity, slope and vegetation height). Table 6.25 shows the ranking significance of each nutrient concentration for all four treatment variables.

Table 6.25

DURATION*	INTENSITY [†]	VEGETATION	SLOPE‡
	+	HEIGHT [‡]	0-0-24
TKN	TP	TP	TKN
(p < F: 0.0014)	(p < F: 0.1236)	(p < F: 0.0285)	(p < F: 0.0131)
TP	TS	TKN	TP
($p < F: 0.0016$)	(p < F: 0.1940)	(p < F: 0.1243)	(p < F: 0.0160)
NO ₅ -N	NH3-N	PO ₊- P	$NH_{3}-N$
($p < F: 0.0071$)	(p < F: 0.3031)	(p < F: 0.1727)	($p < F: 0.0654$)
$NH_3-N (p < F: 0.0074)$	TKN	NH3-N	NO3-N
	(p < F: 0.3571)	(p < F: 0.2899)	(p < F: 0.1387)
TS	NO ₃ -N	TS	PO₄-P
(p < F: 0.0114)	(p < F: 0.5267)	(p < F: 0.4979)	(p < F: 0.2267)
PO ₄ -P	PO₄-P	NO ₃ -N	TS
($p < F: 0.0305$)	(p ≤ F: 0.5671)	(p < F: 0.5214)	(p < F: 0.2713)

Ranking Significance of Average Nutrient Concentrations for Each Treatment Variable (bold type indicates significance at p < 0.10)

* Inten x VegH x Slp x Dur used as error term

‡ Inten x VegH x Slp used as error term

The effects of rainfall, in terms of average concentrations, were highly significant only for duration. Intensity was not significant for any of the nutrients considered although duration was significant for all nutrients. Vegetation height was significant only for average concentrations of TP. Slope was significant in terms of TKN, TP and NH₃-N average concentrations.

CHAPTER VII

SUMMARY

DESIGN STORM SELECTION

The methodology for design storm selection that was described in Chapter IV utilized historical rainfall data to probabilistically determine mean depth and duration. The rainfall depths and durations have equal probabilities of occurrence beyond specified threshold values. The design storm matrix can be either expanded or downsized to match a particular experimental design by increasing or decreasing the number of equal probability areas under the pdfs.

The use of threshold values to define areas under the pdfs are beneficial by restricting mean values for only those regions relevant to an experiment. In this experiment, 1m x 1m boxes with fescue are saturated when the treatments and design storms are applied. Only runoff producing events are relevant to our experiment so steady-state infiltration is used as the threshold in determining the design storms. Therefore, the design storms selected are equally probable events of a greater intensity than the mean steady-state infiltration rate. If the analysis of depth had been done without a threshold value then design storms throughout the whole range of depths would have been determined. If the

experiment's efficiency is to be maximized, this would not have been appropriate since all events do not produce runoff.

Several factors will greatly influence the type of design storms generated by this method. The type of historical data used is extremely important. The rainfall record must be sufficiently long to yield an adequate number of individual events to statistically justify this type of analysis. Also, the data resolution, in terms of time and depth, can be preserved in the design storm matrix depending on the selection of an appropriate critical duration. In addition, the effect of scale is crucial in determining the design storms for a particular application.

LABORATORY STUDY

In this laboratory study, a series of design storms were applied to 1 meter square fescue boxes that had been treated with poultry litter at a rate of 6.7 Mg/ha. Because this experiment was the first of this nature to consider a range of intensities as well as durations, it was more desirable to consider a larger range of durations that represented high as well as low probabilities of occurrence. The design storm durations computed by the method demonstrated in Chapter IV were not used in this experiment except for the 26 minute duration.

The boxes were set at 5, 10, and 15 percent slopes and the fescue was trimmed to lengths of 75 and 150 mm. Rainfall was applied at intensities of 25, 38, and 51 mm/h for durations of 26, 84, 204 minutes. Composite runoff samples were collected and analyzed for the following water quality parameters: NH₃-N, NO₃-N, TKN, PO₄-P, TP, and TS.

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Rainfall intensity was a significant treatment variable. This observation agrees with previous studies by Edwards and Daniel, 1993; 1992, Edwards et al., 1994, and Westerman et al., 1983. Slope was also a significant treatment variable which was previously studied by Storm et al., 1992 and Mitchell and Gunther, 1976. Also, vegetation height was significant and confirms earlier work by Storm et al., 1992.

<u>Runoff Volumes</u>

For rainfall durations of 26 minutes, rainfall intensity was the only significant factor effecting runoff volumes. Runoff volumes from 84 minute durations were significantly influenced by all main treatment factors but predominantly rainfall intensity and to lesser degrees slope and fescue height.

As was expected, runoff volumes increased significantly as rainfall (intensity and duration) increased. The effects of slope and vegetation height that were observed at 84 minutes became more pronounced at 204 minutes but the variability for the long durations was greater so these effects were not statistically significant. Differences in runoff volumes between slopes of 5 and 10 percent were not significantly different but volumes increased significantly for all durations for slopes of 15 percent. The taller vegetation significantly lowered runoff volumes for all durations.

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MASS TRANSPORT

In general, the mass transport of all nutrients followed the trends that were observed for runoff volumes under the same treatment conditions. These observations were expected since runoff is the transport mechanism by which nutrients move off the plot. Mass transport of all nutrients were maximized at high intensity rainfall and long duration, at slopes of 10 or 15 percent and short vegetation.

Trends in mass transport for both nitrogen and phosphorus forms (except NO_3 -N) increased linearly for levels of rainfall intensity for 26 and 84 minute durations. However, sharp increases at high intensity rainfall at 204 minute durations were observed for the same nutrients.

As rainfall duration increased, slope became less significant for TKN and TP. In other words, significant differences in mass transport means at 26 minute durations were found between 10 and 15 percent slopes for TKN and TP. As durations were increased to 84 minutes, significant differences in mass transport means of TKN were observed between 5 and 10 percent slopes with means at 15 percent not being different than 10 percent slopes. In contrast, mass constituents such as NH₃-N and PO₄-P were significant in terms of slope for the 84 minute durations but deviated from what was observed for TKN and TP. Soluble nutrients(NH₃-N and PO₄-P) mass transport means did not follow the same trends and increased linearly as slope was increased. The results also indicate as rainfall duration increases the benefit of applying litter on lesser slopes is decreased for the transport of mass TKN and TP. The tall vegetation effectively reduced mass loading of all nutrients especially phosphorus forms. Tall vegetation significantly reduced PO_4 -P mass for all durations and TP mass transport was reduced at 26 and 204 minute durations. As rainfall durations were increased the effect of vegetation height on mass transport became more pronounced with largest differences observed for the 84 minute durations.

Effects of Rainfall on Mass Transport

Rainfall intensity was significant at all durations for the mass transport of every nutrient except NO₃-N for 26 and 84 minute durations. In general, the trends of mass transport plotted for every duration at each level of rainfall intensity followed the trends observed in runoff volumes from the same treatments. This was expected since runoff volume is the means of transport.

Considering NH₃-N mass transport means plotted for every duration at each level of rainfall intensity (figure 6.204-1), the means for low and moderate intensity at each level of duration appear to be evenly spaced relative to one another. When NH₃-N means at the high rainfall intensity are compared across durations the increases are much larger than the increases in means for low and moderate intensities. Means of TKN mass transport (figure 6.204-2) were similar to trends of NH₃-N.

Mass transport of PO_4 -P means (figure 6.204-3) followed similar trends observed for NH₃-N means which increased drastically at high intensity rainfall for all durations but particularly 204 minute durations. Total phosphorus mass (figure 6.204-4) shows means at 26 and 84 minute durations to be approximately linear. However, the trend at 84

minutes increases by a greater amount. Trends of TP mass at 204 minutes increase drastically for high intensity rainfall as was observed for all runoff components considered.

Mass transport means of total solids (figure 6.204-5) were greatly increased when high intensity rainfall was combined with the 204 minute duration. The relationship between low and moderate intensities and 84 and 204 minute durations were parallel. Total solids mass at 26 minutes was relatively low at all rainfall intensities as compared to 84 and 204 minute durations.

Slope Effects on Mass Transport

Mass transport means of TKN were the only nutrient that was significantly different in terms of slope for all durations (figure 6.204-7). For 26 minute durations, 15 percent slopes drastically increased mass TKN loading whereas there were no differences observed between slopes of 5 and 10 percent. TKN trends from durations of 84 and 204 minutes were similar but were the inverse of TKN trends at 26 minutes. Mass TKN means for 84 and 204 minute durations increased sharply as slope increased from 5 to 10 percent and then dropped slightly at slopes of 15 percent. It appears that 15 percent slopes more readily initiated runoff for 26 minute duration events. At durations of 84 and 204 minutes, 5 percent slopes had the least TKN mass transported. Mass TKN means at 5 and 10 percent slopes were significantly different and there was not any significant differences between means at 10 and 15 percent slopes.

Total phosphorus mass transport for 26 minute durations were virtually identical to trends of TKN mass at 26 minutes. Mass loading means of NH₃-N and PO₄-P at 84 minutes were approximately linear and increased as levels of slope increased. The effects

of slope seem to be the same for all nutrients when slope increased from 5 to 10 percent. Increasing the slope from 10 to 15 percent resulted in a continued linear increase in means of soluble forms of N and P (except NO₃-N). TKN and TP means did not increase linearly between levels of slope. For both constituents means increased sharply at 15 percent slopes for the 26 minute durations. At durations of 84 and 204 minutes where TKN was the only significant nutrient, sharp increases were observed between slopes of 5 and 10 percent and no difference was observed between means at 10 and 15 percent.

Effects of Vegetation Height on Mass Transport

For all nutrients considered, the tall vegetation decreased mass nutrient loading when compared to the short condition. The only nutrient where significant differences in means were observed for all durations was PO_4 -P (figure 6.204-6). Durations of 26 minutes showed means of TP and TKN mass reduced under tall vegetation conditions as well. For 84 minute durations, NH₃-N mass was significantly decreased as well as total solids mass (figure 6.84b). Both total phosphorus and soluble phosphorus mass loading were significantly decreased by the tall vegetation for 204 minute durations (figure 6.204b).

AVERAGE CONCENTRATION

In this experiment mainly nitrogen forms were influenced in terms of average concentrations due to treatment variables. One exception was the impact of tall vegetation in decreasing TP concentrations for 26 minute durations. Rainfall duration increased the significance of intensity and slope on NH₃-N and TKN. The only significant nutrient for 26 minute durations was TP due to the effect of vegetation height.

As rainfall durations increased to 84 and 204 minutes more nutrients became significant due to intensity and slope effects. The effects of rainfall intensity and slope on average concentrations were contrasting. As rainfall intensity increased for durations of 84 and 204 minutes, concentrations of NH₃-N and TKN decreased. Downward trends of NH₃-N were linear and trends of TKN dropped sharply from low to moderate rainfall and then leveled off so differences between NH₃-N and TKN at moderate and high levels of rainfall across 84 and 204 minute durations were nearly parallel.

Conversely, increases in slope increased concentrations of NH₃-N with largest increases between 5 and 10 percent slopes for 84 and 204 minutes durations. Concentrations of NH₃-N then leveled off with no difference between slopes of 10 and 15 percent. TKN concentration trends were similar for 5 and 10 percent slopes at 84 and 204 minute durations but then dropped off to lowest levels at 15 percent slopes. Lowest concentrations at 15 percent slopes can be explained by dilution from increased runoff. If the mass transported from the fescue box does not increase proportionally with an increase in runoff volume than the overall effect will be of diluting the nutrient's

concentration. Conversely, the peak in concentrations at 10 percent slopes is due to the lower mean runoff volumes which apparently increased concentrations TKN.

Effects of Rainfall on Average Concentration

Rainfall intensity was not significant in terms of average concentrations of any nutrient for 26 minute durations. However, as rainfall intensity increased for durations of 84 and 204 minutes, average concentrations of NH₃-N, NO₃-N and TKN decreased. Concentrations of TKN dropped sharply between low and moderate intensities and no differences were observed between the moderate and high levels of intensity for 84 and 204 minute durations. Concentrations of NH₃-N at 84 and 204 minutes decreased linearly as rainfall intensity increased. NO₃-N concentrations also decreased but were significant only at 204 minute durations. These trends can be explained by increases in runoff volumes from increasing rainfall intensities and consequently diluting the concentrations if the mass transported does not proportionally increase with runoff volumes. This suggests that the supply of water via rainfall exceeds the source availability of the nutrient for transport.

Slope Effects on Average Concentration

Slope had significant impacts on TKN and NH₃-N at durations of 84 and 204 minutes. NO₃-N was also significant but only for 204 minute durations. For 84 minute durations, TKN concentrations increased sharply as slopes increased from 5 to 10 percent and then decreased sharply for 15 percent slopes. For an explanation of the peak in TKN concentrations at the 10 percent slope, the runoff volumes that occurred at 84 and 204 minutes need to be considered. The runoff volumes at each level of slope were lowest at 10 percent slopes for the 204 minute durations. The peak in TKN concentrations at 204 minutes and 10 percent slopes occurred due lower runoff volumes. The lower runoff volumes in effect concentrated the runoff leaving the plot because of the TKN constituents in the litter being readily available for transport. Increases in runoff volumes at 84 minutes due to increases in slope were nearly linear. The peak in TKN concentrations at 84 minutes and 10 percent slopes is unexplained.

Concentration means of NH₃-N and NO₃-N increased and were significantly different between slopes of 5 and 10 percent but then leveled off for slopes of 10 and 15 percent. This trend was observed for NH₃-N at both 84 and 204 minute durations and for NO₃-N only at the 204 minute duration. The leveling off of mean concentrations between 10 and 15 percent slopes can be attributed to dilution effects of increased runoff volumes. More mass is actually being transported but the increase in runoff due to increasing slope (15 percent) yields a minimal change in average concentrations for NH₃-N and NO₃-N.

Effects of Vegetation Height on Average Concentration

The height of the vegetation only effected mean total phosphorus concentrations at 26 minutes. The tall vegetation lowered the concentrations of TP by one-half of what was observed under short vegetation conditions. The taller vegetation probably reduced the amount of large litter particles detached and transported by decreasing overland flow velocities and dissipating a larger portion of the kinetic energy of the rainfall than did the short vegetation. This would decrease the organic forms of phosphorus in the runoff and thus decrease the concentration of TP. This is further supported by the fact that neither tall nor short vegetation had an impact on soluble phosphorus (dissolved P) which makes

up a significant portion of the total phosphorus. In this study, soluble phosphorus was about 75 percent of the total phosphorus in runoff from 26 minute durations. Other than the impact on TP at 26 minutes, no other nutrient concentrations were effected by vegetation height for any duration.

Chapter VIII

CONCLUSIONS

Rainfall Effects

Rainfall intensity rather than the total rainfall depth is a greater factor in influencing movement of runoff constituents from the fescue boxes. In general, the mass transport of all measured runoff constituents (except NO₃-N) appear to increase linearly as rainfall intensity increases. The average concentrations of most runoff constituents decreased linearly as rainfall intensity increased. These linear trends were observed for all three storm durations. Increasing rainfall intensity had a dilution effect on nutrient concentrations if the mass transported did not proportionally increase with runoff volumes.

Rainfall duration influenced the significance of rainfall intensity, slope, and vegetation height on mass transport and average concentration. As rainfall duration increases the effect of rainfall intensity and vegetation height increase. As rainfall duration increases, slope becomes less significant in influencing mass transport and average concentrations of TKN and TP. For long duration storm events, regardless of the intensity, the effect of vegetation height in improving runoff water quality is greater than that of slope.

Slope Effects

Slope had a greater effect on influencing losses of nitrogen forms over other runoff constituents. However, for storm events of short duration, regardless of the rainfall intensity, slopes less than 10 percent greatly reduced the losses of TKN and TP. In other words for short duration storm events, slope as a management factor has the greatest impact on reducing total losses if the grade is 10 percent or less.

Vegetation Effects

The taller vegetation decreased losses of all nutrients but had the most significant impact on phosphorus forms. Mass transport of phosphorus forms was significantly reduced for the tall vegetation and for all storm durations. Vegetation height did not effect average concentrations of other nutrients. Vegetation height has a greater impact on phosphorus losses over slope. Also, when storm events of long duration are considered, vegetation height is a more important management factor than slope in reducing losses of all runoff constituents.

<u>Design Storm Selection</u>

The design storm matrix derived in Chapter IV can be either expanded or downsized to match a particular experimental design. As detailed long term rainfall records become more available, the selection of design storms need to become more than just an arbitrary decision. Rather, a selection should be based on a probabilistic analysis where data resolution, scale and application are considered.

Chapter IX

RECOMMENDATIONS FOR FURTHER RESEARCH

The results obtained from this laboratory study are not fully applicable to field situations in terms of actual losses in runoff. Rather the trends and significance of the various treatment factors are most important and valuable in applying to field situations. Due to scale, the results should not be applied directly to the field until field studies are conducted to validate the conclusions because concentrated flow regimes are not realized on a 1.0 meter square plot. The box size of the laboratory experiment were 1 meter square which is rather small compared to the 1.8×9.8 meter plot size used in preliminary field studies by Huhnke et al. (1992). Plots of this size would be sufficient to study treatments effects on a more appropriate scale to extrapolate results to the field. Rainfall duration needs to be investigated in more detail as well as a series of rainfall events. Rainfall intensity appears linearly related to mass nutrient transport and average concentration. For further studies where multiple rainfall intensities might be employed a recommendation would be to replicate fewer levels of rainfall intensity rather than have many levels of intensities with less replication. Three intensities worked very well for this experiment. For larger scale rainfall simulators used in field studies, multiple intensities could be difficult to manage due to calibration and repeatability. For this reason I would recommend that two intensities be used at which the rainfall simulator performs well. Then concentrate on replicating those two intensities for either short or moderate durations. Long durations like that which was used in this study would not be necessary unless the point of interest in the study required it.

For studies conducted in the field where vegetation height is variable and trimming is done prior to the experiment one must cut the grass well in advance to avoid skewing runoff results by enhancing nutrient levels at the soil surface due to plant sap. If possible sites should be chosen for field experiments where replications of slope as a treatment factor could be done with minimal variation between setups. Suggestions on reducing variability would be to conduct infiltration experiments and estimate moisture conditions to select sites that are as uniform as possible in terms of hydrologic characteristics. Conducting infiltration experiments would also give the investigator some idea what design storms might be appropriate to use in the experiment. If sufficient rainfall data is available the procedures outlined in chapter IV could be used to select events that would be typical of that geographic area. In estimating field infiltration rates, researchers could then determine what range of design storm intensities would more likely yield the runoff volumes desired.

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APPENDIX A

RUNOFF DATA

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Table A.1

	<u> </u>
	SSI
Box #	(mm/h)
1	13
2 3	24
3	15
4 5	10
5	9
6	14
7	24
8	19
9	32
10	20
11	31
12	34
13	38
14	11
15	39
16	49
17	29
18	39
19	8
20	17
21	15
22	6
23	1
24	25
25	9
26	9
27	4
28	10
29	2
30	10
mean	19.0
mean standard	
deviation	12.7

Steady State Infiltration Rates of all Fescue Boxes

Table A.2

<u> </u>	SSI
Box #	(mm/h)
3	15
4	10
5	9
7	24
8	19
9	32
10	20
11	31
13	38
16	49
17	29
19	8
23	1
26	9
27	4
28	10
29	2
30	10
······	
mean	18.0
standard	
deviation	13.5

Steady State Infiltration Rates of Boxes Randomly Selected for Laboratory Study

Flow Weighted Mass Transport Data from 26 Minute Durations

	Treatm	lent					-	_		
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH ₃ (mg)	TKN (ma)	NO ₃	PO ₄ (mg)	TP	TS (mg)
		(1111)	70	(mers)	(ing)	(mg)	(mg)	(ing)	(mg)	(mg)
26	25	75	5	0.50	36	92	5.8	5.1	6.8	2900
23	25	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
28	38	75	5	0.1	5.2	9.2	0.1	0.5	0.7	1600
30	38	150	5	1.5	26	37	2.6	2.0	2.1	6500
13	51	75	5	4.00	400	800	10	41	63	23000
4	51	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
3	25	75	10	0.127	24	58	3.6	1.7	3.0	1100
27	25	150	10	0.42	34	95	7.1	1.2	4.0	4100
9	38	75	10	1.41	200	530	23	32	48	9800
7	38	150	10	2.54	320	730	31	31	58	17000
16	51	75	10	1.70	180	460	39	24	35	12000
11	51	150	10	0.50	19	65	3.3	1.9	3.3	3600
17	25	75	15	0.025	0.5	0.0	0.0	0.0	0.0	0.0
5	25	150	15	0.045	8.1	7.7	1.5	1.1	0.5	470
29	38	75	15	2.30	300	850	60	37	58	16000
19	38	150	15	0.21	22	55	4.1	2.1	3.6	1800
8	51	75	15	10.4	980	2200	170	150	200	57000
10	51	150	15	3.67	350	1000	4.6	52	61	20000

Flow Weighted Average Concentration Data from 26 Minute Durations

	Treatment									
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH₃ (mg/l)	TKN (mg/l)	NO ₃ (mg/l)	PO₄ (mg/l)	TP (mg/l)	TS (mg/l)
26	25	75	5	0.50	71	180	12	10	14	5800
23	25	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
28	38	75	5	0.09	55	98	0.5	5.7	7.9	18000
30	38	150	5	1.50	17	25	1.7	1.3	1.4	4300
13	51	75	5	4.00	90	200	2.5	10	16	5800
4	51	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
3	25	75	10	0.127	190	460	28	14	24	8300
27	25	150	10	0.42	82	230	17	2.8	9.6	9800
9	38	75	10	1.41	140	370	16	23	34	7000
7	38	150	10	2.54	130	290	12	12	23	6600
16	51	75	10	1.70	100	270	23	14	21	7100
11	51	150	10	0.50	38	130	6.6	3.8	6.6	7200
17	25	75	15	0.03	21	0.0	1.1	1.4	0.0	0.0
5	25	150	15	0.05	180	170	33	24	10	11000
29	38	75	15	2.30	130	370	26	16	25	7000
19	38	150	15	0.21	100	260	20	10	17	8500
8	51	75	15	10.4	94	210	16	15	19	5500
10	51	150	15	3.67	96	270	1.3	14	17	5400

Flow Weighted Mass Transport Data from 84 Minute Durations

Treatment										
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH ₃ (mg)	TKN (mg)	NO ₃ (mg)	PO ₄ (mg)	TP (mg)	TS (mg)
26	25	75	5	11	660	1900	100	130	150	61000
23	25	150	5	2.5	150	500	7.8	34	44	17000
28	38	75	5	12.09	800	1900	29	180	210	67000
30	38	150	5	9.0	450	990	21	80	89	44000
13	51	75	5	34.5	2000	4700	68	350	420	170000
4	51	150	5	10.5	530	1900	29	120	140	59000
3	25	75	10	4.26	360	1200	34	73	84	7200
27	25	150	10	6.46	590	1500	81	74	110	40000
9	38	75	10	9.49	700	2100	52	150	170	49000
7	38	150	10	20.65	1200	3500	53	210	360	99000
16	51	75	10	23.9	1600	4400	270	300	370	140000
11	51	150	10	23.0	1300	4500	200	270	330	140000
17	25	75	15	6.52	470	890	37	79	87	30000
5	25	150	15	7.29	670	1400	59	120	130	37000
29	38	75	15	16.6	1300	2500	160	230	240	77000
19	38	150	15	12.46	900	2000	77	160	190	58000
8	51	75	15	47.87	2700	6000	300	530	640	220000
10	51	150	15	31.35	1700	3400	79	290	320	130000

Flow Weighted Average Concentration Data from 84 Minute Durations

 _	Treatm	ent		———		· <u> </u>				
Box "	Inten	VegH	Slp	Runoff	NH ₃	TKN	NO_3	PO ₄	TP	TS
	(mm/h)_	(mm)	%	(liters)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)
26	25	75	5	11	60	170	10	12	14	5500
23	25	150	5	2.5	59	200	3.1	14	18	6900
28	38	75	5	12.09	66	160	2.4	15	17	5500
30	38	150	5	9.0	50	110	2.3	8.9	9.9	4900
13	51	75	5	34.5	58	140	2.0	10	12	5000
4	51	150	5	10.5	51	180	2.8	11	13	5600
3	25	75	10	4.26	85	280	8.0	17	20	1700
27	25	150	10	6.46	92	230	12	12	17	6200
9	38	75	10	9.49	74	220	5.5	16	18	5200
7	38	150	10	20.65	57	170	26	10	17	4800
16	51	75	10	23.9	66	190	11	13	16	5800
11	51	150	10	23.0	57	190	9.0	12	14	5900
17	25	75	15	6.52	73	140	5.7	12	13	4700
5	25	150	15	7.29	91	190	8.1	16	18	5100
29	38	75	15	16.6	76	150	9.7	14	14	4600
19	38	150	15	12.46	72	160	6.2	13	15	4700
8	51	75	15	47.87	56	130	6.2	11	13	4500
10	51	150	15	31.35	53	110	2.5	9.2	10	4100

Flow Weighted Mass Transport Data from 204 Minute Durations

	Treatm	ent	-			· · · ·		_		
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH3 (mg)	TKN (mg)	NO ₃ (mg)	PO ₄ (mg)	TP (mg)	TS (mg)
26	25	75	5	54.5	1900	4800	130	400	420	260000
23	25	150	5	18	840	3000	86	240	260	96000
28	38	75	5	39.5	1600	3900	59	410	450	180000
30	38	150	5	27.5	990	2000	50	190	210	120000
13	51	75	5	120	3500	7500	150	700	750	520000
4	51	150	5	41	1500	3800	120	320	320	190000
3	25	75	10	14.14	740	1900	40	140	140	73000
27	25	150	10	18.5	1100	2800	110	180	220	78000
9	38	75	10	29.04	1200	3100	64	240	250	120000
7	38	150	10	53.8	1900	4900	69	310	440	200000
16	51	75	10	79.9	3000	7900	370	590	660	390000
11	51	150	10	81.2	3000	9300	310	660	660	400000
17	25	75	15	29.8	1300	2700	57	230	240	120000
5	25	150	15	27.2	1300	2900	91	250	260	120000
29	38	75	15	40.1	1800	3600	180	330	320	160000
19	38	150	15	53.6	2100	3600	100	330	320	180000
8	51	75	15	109.6	3900	7800	300	750	770	380000
10	51	150	15	103.5	3900	6700	110	500	530	390000

Flow Weighted Average Concentration Data from 204 Minute Durations

	Treatm	ent								
Box	Inten	VegH	Slp	Runoff	NH₃ (ma/l)	TKN	NO ₃	PO_4	TP	TS (ma(l))
#	(mm/h)	(mm)	%	(liters)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)	(mg/l)
26	25	75	5	54.5	35	88	2.4	7.3	7.8	4800
23	25	150	5	18	47	170	4.8	13	15	5300
28	38	75	5	39.47	42	98	1.5	10	11	4700
30	38	150	5	27.5	36	72	1.8	7.0	7.8	4500
13	51	75	5	120	29	63	1.3	5.8	6.2	4300
4	51	150	5	41	36	94	2.9	7.9	7.9	4600
3	25	75	10	14.14	53	130	2.8	10	9.9	5200
27	25	150	10	18.5	61	150	5.9	9.7	12	4200
9	38	75	10	29.04	40	110	2.2	8.2	8.8	4300
7	38	150	10	53.81	35	91	1.3	5.8	8.2	3700
16	51	75	10	79.9	37	99	4.7	7.4	8.2	4900
11	51	150	10	81.16	36	110	3.9	8.1	8.1	5000
1 7	25	75	15	29.81	43	92	1.9	7.8	8.1	4100
5	25	150	15	27.2	48	110	3.3	9.1	9.5	4400
29	38	75	15	40.1	45	90	4.5	8.2	7.9	4000
19	38	150	15	53.6	40	68	1.9	6.2	6.0	3300
8	51	75	15	109.6	36	71	2.9	6.9	7.1	3500
10	51	150	15	103.5	37	65	1.1	4.8	5.1	3800

	Tre	atment				
Box #	Inten (mm/h)	Fescue (mm)	Slp %	<u>NH3-N</u> TKN	<u>NO3-N</u> TKN	<u>РО₄-Р</u> ТР
26	25	75	5	0.39	0.06	0.75
23	25	150	5	na	na	na
28	38	75	5	0.56	0.01	0.73
30	38	150	5	0.70	0.07	0.94
13	51	75	5	0.45	0.01	0.65
4	51	150	5	na	na	na
3	25	75	10	0.40	0.06	0.58
27	25	150	10	0.36	0.07	0.29
9	38	75	10	0.39	0.04	0.68
7	38	150	10	0.44	0.04	0.54
16	51	75	10	0.38	0.08	0.68
11	51	150	10	0.29	0.05	0.58
17	25	75	15	na	na	na
5	25	150	15	1.05	0.19	2.37
29	38	75	15	0.36	0.07	0.65
19	38	150	15	0.39	0.07	0.59
8	51	75	15	0.45	0.08	0.78
10	51	150	15	0.35	0.01	0.86

Nutrient Ratio Data from 26 Minute Durations

	Tre	atment				
Box #	Inten (mm/h)	Fescue (mm)	Slp %	<u>NH3-N</u> TKN	<u>NO3-N</u> TKN	<u>РО₄-Р</u> ТР
26	25	75	5	0.35	0.06	0.85
23	25	150	5	0.30	0.02	0.78
28	38	75	5	0.42	0.02	0.86
30	38	150	5	0.46	0.02	0.90
13	51	75	5	0.42	0.01	0.83
4	51	150	5	0.28	0.02	0.87
3	25	75	10	0.30	0.03	0.86
27	25	150	10	0.40	0.05	0.68
9	38	75	10	0.33	0.03	0.89
7	38	150	10	0.34	0.02	0.60
16	51	75	10	0.36	0.06	0.81
11	51	150	10	0.29	0.05	0.83
17	25	75	15	0.53	0.04	0.91
5	25	150	15	0.47	0.04	0.89
29	38	75	15	0.50	0.06	0.98
19	38	150	15	0.45	0.04	0.83
8	51	75	15	0.44	0.05	0.83
10	51	150	15	0.49	0.02	0.92

Nutrient Ratio Data from 84 Minute Durations

_	Tre	atment				
Box #	Inten (mm/h)	Fescue (mm)	Slp %	<u>NH3-N</u> TKN	<u>NO3-N</u> TKN	<u>РО4-Р</u> ТР
26	25	75	5	0.40	0.03	0.94
23	25	150	5	0.28	0.03	0.92
28	38	75	5	0.42	0.02	0.91
30	38	150	5	0.50	0.03	0.91
13	51	75	5	0.47	0.02	0.94
4	51	150	5	0.38	0.03	1.0
3	25	75	10	0.39	0.02	1.02
27	25	150	10	0.41	0.04	0.81
9	38	75	10	0.37	0.02	0.93
7	38	150	10	0.38	0.01	0.70
16	51	75	10	0.38	0.05	0.90
11	51	150	10	0.32	0.03	1.0
17	25	75	15	0.47	0.02	0.96
5	25	150	15	0.45	0.03	0.95
29	38	75	15	0.50	0.05	1.05
19	38	150	15	0.58	0.03	1.02
8	51	75	15	0.50	0.04	0.98
10	51	150	15	0.58	0.02	0.95

Nutrient Ratio Data from 204 Minute Durations

	Treatm	nent								
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH₃ (mg/l)	TKN (mg/l)	NO3 (mg/l)	PO ₄ (mg/l)	TP (mg/l)	TS (mg/l)
26	25	75	5	0.50	71	180	12	10	14	5800
23	25	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
28	38	75	5	0.09	55	98	0.5	5.7	7.9	18000
30	38	150	5	1.50	17	25	1.7	1.3	1.4	4300
13	51	75	5	4.00	90	200	2.5	10	16	5800
4	51	150	5	0.00	0.0	0.0	0.0	0.0	0.0	0.0
3	25	75	10	0.127	190	460	28	14	24	8300
27	25	150	10	0.42	82	230	17	2.8	9.6	9800
9	38	75	10	1.41	140	370	16	23	34	7000
7	38	150	10	2.54	130	290	12	12	23	6600
16	51	75	10	1.70	100	270	23	14	21	7100
11	51	150	10	0.50	38	130	6.6	3.8	6.6	7200
17	25	75	15	0.03	21	0.0	1.1	1.4	0.0	0.0
5	25	150	15	0.05	180	170	33	24	10	11000
29	38	75	15	2.30	130	370	26	16	25	7000
19	38	150	15	0.21	100	260	20	10	17	8500
8	51	75	15	10.4	94	210	16	15	19	5500
10	51	150	15	3.67	96	270	1.3	14	17	5400

Sample Concentration Data from 26 Minute Durations

	Treatm	ent		-		<u> </u>				
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH3 (mg/l)	TKN (mg/l)	NO ₃ (mg/l)	PO ₄ (mg/l)	TP (mg/l)	TS (mg/l)
26	25	75	5	11	59	171	9.4	12	14	5500
23	25	150	5	2.5	59	199	3.1	14	18	6900
28	38	75	5	12.09	66	159	2.5	15	17	5500
30	38	150	5	9.0	57	127	2.4	10	12	5000
13	51	75	5	34.5	53	129	1.9	10	12	4900
4	51	150	5	10.5	51	181	2.8	11	13	5600
3	25	75	10	4.26	82	276	7.4	17	20	1500
27	25	150	10	6.46	93	232	12.1	12	17	5900
9	38	75	10	9.49	61	194	3.6	15	15	4900
7	38	150	10	20.65	48	151	1.2	10	17	4500
16	51	75	10	23.9	63	179	10.3	13	15	5700
11	51	150	10	23.0	57	195	9.0	12	15	5900
17	25	75	15	6.52	73	137	5.7	12	13	4700
5	25	150	15	7.29	91	194	7.9	16	18	5100
29	38	75	15	16.6	67	117	7.1	14	12	4200
19	38	150	15	12.46	72	158	6.0	13	15	4600
8	51	75	15	47.87	45	102	3.5	10	12	4300
10	51	150	15	31.35	47	85	2.7	9	9	3900

Sample Concentration Data from 84 Minute Durations

	Treatm	ent								
Box #	Inten (mm/h)	VegH (mm)	Slp %	Runoff (liters)	NH3 (mg/l)	TKN (mg/l)	NO ₃ (mg/l)	PO ₄ (mg/l)	TP (mg/l)	TS (mg/l)
26	25	75	5	54.5	29	66	0.6	6	6	4600
23	25	150	5	18	45	161	5.0	13	14	5100
28	38	75	5	39.47	31	72	1.1	8	9	4300
30	38	150	5	27.5	29	53	1.6	6	7	4300
13	51	75	5	120	18	33	1.0	4	4	4100
4	51	150	5	41	30	64	2.9	7	6	4300
3	25	75	10	14.14	39	71	0.6	7	6	6700
27	25	150	10	18.5	44	105	2.5	9	9	3200
9	38	75	10	29.04	23	51	0.6	4	4	3800
7	38	150	10	53.81	20	43	0.5	3	3	3000
16	51	75	10	79.9	25	62	1.9	5	5	4500
11	51	150	10	81.16	28	83	1.9	7	6	4600
17	25	75	15	29.81	35	80	0.8	7	7	3900
5	25	150	15	27.2	33	77	1.6	6	6	4100
29	38	75	15	40.1	24	47	0.7	4	3	3700
19	38	150	15	53.6	30	40	0.6	4	3	2900
8	51	75	15	109.6	20	28	0.3	4	2	2700
10	51	150	15	103.5	31	46	0.5	3	3	3600

Sample Concentration Data from 204 Minute Durations

	Treatm	ent			
Box	Inten	VegH	Slp	% N	% P
#	(mm/h)	(mm)	%	removed	removed
26	25	75	5	0.3	0.08
23	25	150	5	0.0	0.0
28	38	75	5	0.03	0.01
30	38	150	5	0.14	0.03
13	51	75	5	2.79	0.76
4	51	150	5	0.0	0.0
3	25	75	10	0.21	0.04
27	25	150	10	0.35	0.05
9	38	75	10	1.90	0.58
7	38	150	10	2.62	0.7
16	51	75	10	1.72	0.42
11	51	150	10	0.24	0.04
17	25	75	15	0.0	0.0
5	25	150	15	0.03	0.01
29	38	75	15	3.13	0.7
19	38	150	15	0.20	0.04
8	51	75	15	8.16	2.41
10	51	150	15	3.46	0.74

Percent Total Nitrogen and Phosphorus Lost from 26 Minute Durations

	Treatm	ent			
Box	Inten	VegH	Slp	% N	% P
	(mm/h)	(mm)	%	removed	removed
26	25	75	5	6.88	1.81
23	25	150	5	1.75	0.53
28	38	75	5	6.64	2.53
30	38	150	5	3.48	1.07
13	51	75	5	16.4	5.06
4	51	150	5	6.64	1.69
3	25	75	10	4.25	1.01
27	25	150	10	5.44	1.33
9	38	75	10	7.41	2.05
7	38	150	10	12.2	4.34
16	51	75	10	16.1	4.46
11	51	150	10	16.2	3.98
17	25	75	15	3.19	1.07
5	25	150	15	5.02	1.57
29	38	75	15	9.15	2.89
19	38	150	15	7.15	2.29
8	51	75	15	21.7	7.72
10	51	150	15	12.0	3.86

Percent Total Nitrogen and Phosphorus Lost from 84 Minute Durations

	Treatm	ent			
Box	Inten	VegH	Slp	% N	% P
#	(mm/h)	(mm)	%	removed	removed
26	25	75	5	17.0	5.06
23	25	150	5	10.6	3.13
28	38	75	5	13.6	5.43
30	38	150	5	7.06	2.53
13	51	75	5	26.3	9.04
4	51	150	5	13.5	3.86
3	25	75	10	6.68	1.69
27	25	150	10	10.0	2.65
9	38	75	10	10.9	3.01
7	38	150	10	17.1	5.31
16	51	75	10	28.5	7.96
11	51	150	10	33.1	7.96
17	25	75	15	9.49	2.89
5	25	150	15	10.3	3.13
29	38	75	15	13.0	3.86
19	38	150	15	12.7	3.86
8	51	75	15	27.9	9.28
10	51	150	15	23.4	6.39

Percent Total Nitrogen and Phosphorus Lost from 204 Minute Durations

APPENDIX B

FORAGE DATA

Tables

Page

B.1 Percent cover and forage yield of the fescue boxes used in the experiment.--- 147

FORAGE ANALYSIS

The fescue boxes that were used in the experiment were evaluated on the basis of the percentage of blade, litter, and ground cover on the box surface. The measuring device was a simple point measurement system where a pointed rod was placed downward toward the soil surface. The rod was guided by a rigid frame that had 10 sets of holes. The frame was set on the box surface and rods were slowly pushed through the guides toward the soil surface. The first material that the tip of the rod touched was the measured recording for that sampling point. As mentioned, there were three possible types of material to characterize the cover of the plots, they are defined as follows:

BLADE - any living green material such as a leaf or stem

LITTER - any nonliving material such as leaf litter

GROUND - bare uncovered soil

Once the first ten measurements were taken, the frame was rotated on the vertical axis 90° and another ten measurements were taken in the same manner. The total of 20 sampling points were used to determine the percentage of blade, litter, and ground. Table B.1 includes the results obtained by sampling each fescue box immediately after the rainfall simulator runs.

The quantity of forage present at the time treatments were applied to the fescue boxes was also estimated by clipping all the forage from a small area of each fescue box. The area clipped from each box was defined by a square frame with inside dimensions measuring 10 cm x 10 cm. The square frame was tossed on the box surface and all of the forage that was rooted inside the frame was harvested, bagged and dried at 95° C for 24 hours. Table B.1 contains these estimates expressed as kg/ha on a dry matter basis.

Box #	Blade (%)	Litter (%)	Ground (%)	Forage Yield (kg/ha)
26	45	55	0	3620
23	65	30	5	3900
28	35	55	10	3990
30	75	25	0	7510
13	45	50	5	4290
4	65	30	5	4170
3	50	40	10	1990
27	65	30	5	5100
9	50	35	15	3320
7	35	65	5	5510
16	50	40	10	4270
11	75	25	0	3750
17	55	40	5	2510
5	75	25	0	1890
29	55	40	5	3400
19	80	20	0	4250
8	50	35	15	3170
10	80	20	0	4080

Table B.1 Percent cover and forage yield of the fescue boxes used in the experiment.

APPENDIX C

LABORATORY ANALYTICAL PROCEDURES

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SAMPLE COLLECTION, PRESERVATION, AND STORAGE

Sample collection in general is an issue that has to be addressed by the researcher. The method of collection should be such that the sample to undergo analysis be representative of the population from which it was sampled. In this case whether it be runoff or manure/litter samples, the volume of sample collected should be sufficient to perform all the required analysis plus an additional amount to provide for any quality control needs, split samples or repeat examinations. Table A.1 shows the volumes of sample required to carry out all required analysis for this experiment. The containers for sampling and storage should be made of polyethylene or pyrex glass. Wide mouth containers are preferred. Samples should be analyzed as soon as possible after collection because most reliable results are obtained on fresh samples. If they cannot be analyzed immediately, they should be preserved upon collection. For composite samples, each aliquot should be preserved at the time of collection. In order to maintain sample integrity, certain guidelines and procedures should be followed to increase the confidence of the researcher in experimental data obtained. Hold times of samples and storage conditions are summarized in Table A.2.

SAMPLE FILTERING PROCEDURES

Samples were filtered prior to any inorganic analysis using a two-stage filtration system. Using a clean syringe about 50 ml of sample were drawn from a 0.5 l container which held a composite runoff sample. Two filter canisters were then attached to the end of the syringe containing the withdrawn volume of sample. The sample was forced through the filter canisters into a 45 ml EPA vial from which the inorganic analysis would be performed. The first filter canister contained two glass microfibre filters the first of which was 1.0 micron (Whatman, 2.1 cm) in size and the second filter in the direction of the sample flow was 0.75 microns (Whatman, 2.1 cm). The second filter canister contained two filters also, the first of which was 0.75 (Whatman, 2.1 cm) microns and the second 0.5 microns(MSI, 2.5 cm). The filtered sample needs to pass a 0.5 micron filter but to make the filtering process more efficient and save on 0.5 micron filters, a larger one was first used in the filtering process to remove the larger particles.

Sample Type	Analysis	Volume
Lunoff Sample	Total Kjeldahl Nitrogen	
	Total Phosphorus	500 ml
	Total Solids	
	Ammonia	
	Nitrate	50 ml
	Orthophosphates	
oultry Litter	Moisture content	
	Total Phosphorus	500 g
	Total Nitrogen	U

Table C.1 Sample Size

Table C.2 Maximum recommended holding times for samples and storage conditions (U. S. Environmental Protection Agency. 1982. <u>Handbook for Sampling and Sample Preservation of Water and Wastewater</u>. EPA-600/4-82-029.)

Sample Type	Analysis	Storage	Holding Time
Runoff Sample	Total Kjeldahl Nitrogen Total Phosphorus	Cool to 4° C, H_2 SO ₄ to pH<2	28 days
	Ammonia	Cool to 4° C, H ₂ SO ₄ to pH<2	28 days
	Nitrate+Nitrite	Cool to 4°C	28 days
	Ortho-P	Filter immediately, Cool to 4 ⁰ C	48 hrs
	Total Solids	Cool to 4 ^o C	7 days
Poultry Litter	Moisture Content	Cool to 4 [°] C	7 days
	Total Phosphorus	Cool to 4°C	7 days
	Total Kjeldahl Nitrogen	Cool to 4 ^o C	7 days

ANALYSIS OF RUNOFF SAMPLES

Determination of Orthophosphate - Ascorbic Acid Method

This method covers the determination of ortho-phosphorus in drinking water, surface water and domestic and industrial wastes. The minimum detectable concentration of the method is 0.01 mg P/L. The detectable P ranges are as follows:

Approx. P range <i>mg/L</i>	Light path cm
0.30 - 2.00 0.15 -1.30 0.01 - 0.25	0.5
0.01 - 0.25	5.0

Ammonium molybdate and potassium antimonyl tartrate react in acid medium with orthophosphate to form a heteropoly acid- phosphomolybdic acid - that is reduced to a intensely colored molybdenum blue by ascorbic acid.

Interferences:

Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. Concentrations as low as 0.1 mg As/L interfere with phosphate determination. Hexavalent chromium and NO₂⁻ interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L. Sulfide (Na₂S) and silicate do not interfere at concentrations of 1.0 and 10 mg/L.

Apparatus:

- 1. Spectrophotometer or filter photometer for suitable measurements at 650 or 880 nm with a light path of 1 cm or longer.
- 2. Acid washed glassware: All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered until further needed again. If this is done, the treatment with 1:1 HCl is only required occasionally. <u>Commercial detergents should never be used.</u>

Reagents:

- 1. Sulfuric acid, 5N: Dilute 70 ml concentrated H_2SO_4 to 500 ml with distilled water.
- Potassium antimonyl tartrate solution: Dissolve 1.3715 g K(SbO)C₄H₄O₆. 1/2 H₂O in 400 ml distilled water in a 500 ml volumetric flask and dilute to volume. Store in a glass stoppered bottle.
- 3. <u>Ammonium molybdate solution</u>: Dissolve 20 g (NH₄)₆Mo₇O₂₄. 4H₂O in 500 ml distilled water. Store in a glass stoppered bottle.
- 4. <u>Ascorbic acid</u>, 0.1*M*: Dissolve 1.76 g ascorbic acid in 100 ml distilled water. The solution is stable for about 1 week at 4°C.
- 5. <u>Combined color reagent</u>: Mix the above reagents in the following proportions for 100 ml of the combined reagent: 50 ml 5*N* H₂SO₄, 5 ml potassium antimonyl tartrate solution, 15 ml ammonium molybdate solution, and 30 ml ascorbic acid solution.
 - Note: Let all reagents reach room temperature before they are mixed and mix in the order given. Also mix after addition of each reagent. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4h.
- 6. <u>Stock Phosphate solution</u>: Dissolve in distilled water 219.5 mg anhydrous KH_2PO_4 and dilute to 1000 ml; 1.00 ml = 50.0 µg PO_4^{3-} -P.
- 7. <u>Standard phosphate solution</u>: Dilute 50.0 ml stock phosphate solution to 1000 ml with distilled water; $1.00 \text{ ml} = 2.5 \mu \text{g P}$.
- Note: All the reagents must be prepared in ammonia free water. Deionized water of 2 mega ohms is preferred.

Procedure:

Color development:

Pipette an aliquot containing 1 to 40 μ g of P into a 50 ml volumetric flask. Dilute the sample to approximately 40 ml with distilled water, and add 8 ml of the combined color reagent. Dilute to volume and mix well. After 10 min, measure the absorbance at 880 nm, using reagent blank as the reference solution.

Preparation of calibration curve:

Prepare a calibration curve from a series of six standards within the phosphate range of 0.15 to 1.3 mg P/L. Use distilled water blank with the combined reagent to make photometric readings for the calibration curve. Plot absorbance vs. phosphate concentration to give a straight line passing through the origin.

QA/QC:

- 1. Two triplicate samples are run for every 25 samples.
 - *one of the triplicates is a spiked sample
 - *one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.
- 2. One blank is run per 25 samples. Process blank exactly as samples.
- 3. At least one standard(from the standard phosphate solution) are run for every 25 samples.
- 4. If the standard(s) do not agree within $\pm 2\%$ of the true value, prepare a new calibration curve.
- 5. Process standards and blank exactly as the samples. Run at least one blank and one phosphate standard with each set of samples. If the standards do not agree within ± 2% of the true value, prepare a new calibration curve.

References:

- Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-115-116; Method 4500-P E.
- Methods for the determination of inorganic substances in environmental samples (EPA/600/R 93/100), Aug 1993. Method 365.2.

Procedures for the Determination of Total Phosphorus - Ascorbic Acid Method

This method covers the determination of total phosphorus in drinking water, surface water, domestic and industrial wastes. The minimum detectable concentration of the method is 0.01 mg P/L. The detectable P ranges are as follows:

Approx. P range mg/L	Light path cm
0.30 - 2.00 0.15 - 1.30 0.01 - 0.25	0.5 1.0 5.0

Ammonium molybdate and potassium antimonyl tartrate react in acid medium with orthophosphate to form a heteropoly acid-phophomolybdic acid that is reduced to a intensely colored molybdenum blue by ascorbic acid.

Interferences:

Arsenate is determined similarly to phosphorus and should be considered when present in concentrations higher than phosphorus. Concentrations as low as 0.1 mg As/L interfere with phosphate determination. Hexavalent chromium and nitrite interfere to give results about 3% low at concentrations of 1 mg/L and 10 to 15% low at 10 mg/L. Sulfide (NA₂S) and silicate do not interfere at concentrations of 1.0 and 10 mg/L.

Apparatus:

- 1. Spectrophotometer or filter photometer for suitable measurements at 650 or 880 nm with a light path of 1 cm or longer.
- Acid washed glassware: All glassware used should be washed with hot 1:1 HCl and rinsed with distilled water. Preferably, this glassware should be used only for the determination of phosphorus and after use it should be rinsed with distilled water and kept covered. If this is done, the treatment with 1:1 HCl is only required occasionally. <u>Commercial detergents should never be used.</u>

Reagents:

- 1. <u>Sulfuric acid</u>, 5N: Dilute 70 ml of conc. H_2SO_4 to 500 ml with distilled water.
- Potassium antimonyl tartrate solution: Dissolve 1.3715 g K(SbO)C₄H₄O₆•1/2H₂O in 400 ml of distilled water in a 500 ml volumetric flask and dilute to volume. Store in a glass-stoppered bottle.
- <u>Ammonium molybdate solution</u>: Dissolve 20 g (NH₄)₆Mo₇O₂₄•4H₂O in 500 ml of distilled water. Store in a glass-stoppered bottle.
- 4. <u>Ascorbic acid</u>, 0.1 M: Dissolve 1.76 g ascorbic acid in 100 ml distilled water. The solution is stable for about 1 week at 4°C.
- 5. Combined color reagent:

Mix the above reagents in the following proportions and order for 100 ml of the combined color reagent:

- 50 ml 5N H₂SO₄ 5 ml - potassium antimonyl tartrate solution 15 ml - ammonium molybdate solution 30 ml - ascorbic acid solution
- Note: let all reagents reach room temperature before they are mixed. Mix thoroughly after the addition of each reagent. If turbidity forms in the combined reagent, shake and let stand for a few minutes until turbidity disappears before proceeding. The reagent is stable for 4 hours.
- 6. <u>Digestion reagent</u>: Dissolve 134 g K₂SO₄ in 650 ml water and 200 ml of conc. H₂SO₄. Add, with stirring, a solution prepared by dissolving 2 g red mercuric oxide, HgO, in 25 ml of 6N H₂SO₄. Dilute the combined solution to 1 liter with distilled water. Keep at a temperature close to 20° C to prevent crystallization.
- 7. <u>Stock phosphate solution</u>: Dissolve in distilled water 0.2195 g anhydrous KH_2PO_4 and dilute to 1000 ml (1.00 ml = 50.0 μ g PO_4^{-3} -P).
- 8. <u>Standard phosphate solution</u>: Dilute 50.0 ml stock phosphate solution to 1000 ml with distilled water; $(1.00 \text{ ml} = 2.5 \ \mu\text{g PO}_4^{-3}\text{-P})$.

Note: All the reagents must be prepared in ammonia free water. Deionized water of 2 mega ohms is preferred.

Procedure:

Digestion

To 20 ml of sample placed in a digestion tube, add 5 ml of digestion reagent and mix. Place 4 to 6 glass boiling beads in the digestion tube to promote smooth boiling. Place tubes in block digestor and set the temperature between 100 and 105° C. Maintain this temperature until the water portion of the mixture vaporizes (about 8 to 10 hours). Once the water has boiled off there should be 2 to 3 ml of digestion reagent and sample constituents in the bottom of the digestion tube. Turn the temperature up to 360° C and digest sample until copious white fumes are observed and the digested sample is clear of very pale yellow. Cool sample, add 5 to 10 ml of distilled water and swirl until salts go back into solution. Transfer the 5 to 10 ml solution to a glass vial whose weight has been tarred on a digital scale. Use a clean glass disposable pipette to transfer the solution. Rinse the digest vial. Continue the rinse process until 26.20 g of solution are contained within the tarred glass vial. (by experimentation it was determined that 26.20 g correspond to 25 ml, it is much easier to attain 25 ml of solution by use of a scale than to measure volumetrically) Dispose of the transfer pipette after each sample transfer to prevent cross contamination between samples.

Color development

The pH of the sample must be adjusted to around 7.0 (it has been determined that one drop of 6N NaOH per 0.5 ml of aliquot will satisfactorily raise the pH to 7.0). Pipette an aliquot containing 1 to 40 μ g (0.20 to 1.25 mg/L) of P into a 50 ml volumetric flask. Dilute the sample to approximately 40 ml with distilled water, and add 8 ml of the combined color reagent. Dilute to volume and mix well. After 10 minutes, measure the absorbance at 880 nm, using reagent blank as reference solution.

Preparation of calibration curve

Prepare a calibration curve from a series of six standards within the phosphate range of 0.15 to 1.3 mg PO_4^{-3} -P/L. Use distilled water blank with the combined color reagent to make photometric readings for the calibration curve. Plot absorbance vs. phosphate concentration to give a straight line.

Calculation:

Determine the concentration of PO_4^{-3} -P of samples using the standard curve. Make necessary adjustments to the calculation to account for dilutions and express the result as mg PO_4^{-3} -P/L.

QA/QC:

1. Two triplicate samples are run for every 25 samples.

*one of the triplicates is a spiked sample
*one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.

- 2. One blank is run per 25 samples. Process blank exactly as samples.
- 3. At least one standard (from the standard phosphate solution) are run for every 25 samples.

4. If the standard(s) do not agree within $\pm 2\%$ of the true value, prepare a new calibration curve.

Reference:

Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-115 - 116; Method 4500-P E.

Determination of Ammonia - Nesslerization Method

Direct nesslerization is used for purified drinking waters, natural water, and highly purified waste-water effluents, all of which should be low in color and have NH₃-N concentrations exceeding 20 μ g/L. Direct nesslerization is used for purified drinking waters, natural water and highly purified waste-water effluents

The graduated yellow to brown colors produced by the nessler-ammonia reaction absorb over a wide wavelength range. The yellow color characteristic of low ammonia nitrogen concentration (0.4 to 5 mg/L) can be measured with acceptable sensitivity in the wavelength region from 400 to 425 nm when a 1-cm light path is available. A light path of 5 cm extends measurements into the nitrogen concentration range of 5 to 60 μ g NH₃-N/L.

Interferences:

Pretreatment before direct nesslerization with zinc sulfate and alkali precipitates calcium, iron, magnesium, and sulfide, which form turbidity when treated with nessler reagent. The floc also removes suspended matter and sometimes colored matter. addition of EDTA or Rochelle salt solution inhibits precipitation of residual calcium and magnesium ions in the presence of the alkaline reagent. However, use of EDTA demands an extra amount of nessler reagent to insure a sufficient nessler reagent excess for reaction with the ammonia.

Apparatus:

- 1. Colorometric equipment: One of the following is required:
 - a. Spectrophotometer for use at 400 to 500 nm and providing a light path of 1 cm or longer
 - b. Filter photometer, providing a light path of 1 cm or longer and equipped with a violet filter having maximum transmittance at 400 to 425 nm. A blue filter can be used for higher NH₃-N concentrations.
- 2. pH meter, equipped with a high-pH electrode.

Reagents:

- <u>EDTA reagent</u> (stabilizer reagent): Dissolve 50 g disodium ethylenediamine tetraacetate dihydrate in 60 ml of distilled water containing 10 g NaOH. If necessary apply gentle heat to complete dissolution. Cool to room temperature and dilute to 100 ml. (EDTA reagent prevents calcium or magnesium precipitation in undistilled samples after the addition of an alkaline reagent)
- 2. <u>Nessler reagent</u>: Dissolve 100 g HgI₂ and 70 g KI in a small quantity of water and add this mixture slowly, with stirring, to a cool solution of 160 g NaOH dissolved in 500 ml water. Dilute to 1 L. Store in rubber-stoppered borosilicate glassware and out of sunlight to maintain reagent stability for up to a year under normal laboratory conditions. Check reagent to make sure that it yields the characteristic color with 0.1 mg NH₃-N/ L within 10 min after addition and does not produce a precipitate with small amounts of ammonia within 2 hours.
- 3. <u>Stock ammonium solution</u>: Dissolve 3.189 g anhydrous NH₄Cl, dried at 100°C, in water, and dilute to 1000 ml; 1.00 ml = 1.00 mg N = 1.22 mg NH₃.
- <u>Standard Ammonium solution</u>: Dilute 10.00 ml stock ammonium solution to 1000 ml with water; 1.00 ml = 10.00 μg N = 1.22 μg NH₃.

Note: All the reagents must be prepared in ammonia free water. Deionized water of 2 mega ohms is preferred.

Procedure:

Color development:

Pipet out a suitable aliquot* of sample in a 50 ml volumetric flask. Add about 30 ml distilled water. Add one drop(0.05 ml) of EDTA reagent per 0.25 ml of sample aliquot. Mix gently. Add 2.0 ml of nessler reagent. Make up to volume.

*Problems have been encountered with turbidity forming. By using a small sample volume, from 0.25 to 0.5 ml, this problem can be eleviated. The samples analyzed thus far have had fairly high ammonia concentrations. If samples are encountered that contain low concentrations of ammonia the turbidity problem will have to be dealt with in some other manner.

The samples should be mixed thoroughly. The reaction should be allowed to proceed for at least 10 min after addition of nessler reagent. Measure absorbance with spectrophotometer using a wavelength of 400 to 425 nm for 1-cm light path and at 450 to 500 nm for 5-cm light path. Prepare standard curve using the standard ammonium solution in the acceptable sensitivity range $(0.4 \text{ to 5 mg NH}_3\text{-N/L})$.

Calculation:

Determine the concentration of NH_3 -N of samples using the standard curve. Make necessary adjustments to the calculation to account for dilutions and express the result as mg NH_3 -N/L.

QA/QC:

- Two triplicates are run for every 25 samples.
 *one of the triplicates is a spiked sample
 *one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.
- 2. One blank is run for every 25 samples.
- At least three standards(from the standard ammonium solution) are run for every 25 samples.
 *if the standards do not agree within ± 2 % of the true value, prepare a new calibration curve.
- 4. The standards developed should be tested with EPA quality control samples; the samples analyzed value must fall within the stated 95 % confidence interval.
- 5. As far as possible the conditions such as temperature and reaction time should be kept the same for samples, standards and blank. If NH₃-N is very low, use a 30-min contact time for sample, standards and blank.
- 6. The calibration curve should be recalculated for each new batch of nessler reagent.

Reference:

Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-75-80; Method 4500-NH₃ C.

Methods for the determination of inorganic substances in environmental samples (EPA/600/R-93/100), Aug 1993. Method 350.2.

Procedures for the Determination of Total Kjeldahl Nitrogen - Nesslerization Method

Total Kjeldahl Nitrogen(TKN) is defined as the of free-ammonia and organic nitrogen compounds which are converted to ammonium sulfate $(NH_4)_2SO_4$ under the conditions of digestion. Nitrogen in the form of azide, azine, azo, hydrazone, nitrate, nitrite, nitrile, nitro, nitroso, oxime, and semicarbazone are not accounted in the total kjeldahl nitrogen. The converted ammonia can be easily determined colorimetrically.

The graduated yellow to brown colors produced by the nessler-ammonia reaction absorb over a wide wavelength range. The yellow color characteristic of low ammonia nitrogen concentration(0.4 to 5 mg/L) can be measured with acceptable sensitivity in the wavelength region from 400 to 425 nm when a 1 - cm light path is available. A light path of 5 cm extends measurements into the nitrogen concentration range of 5 to 60 μ g/L.

Interferences:

Pretreatment before direct nesslerization with zinc sulfate and alkali precipitates calcium, iron, magnesium, and sulfide, which form turbidity when treated with nessler reagent. The floc also removes suspended matter and sometimes colored matter. Addition of EDTA or Rochelle salt solution inhibits precipitation of residual calcium and magnesium ions in the presence of the alkaline reagent. However use of EDTA demands an extra amount of nessler reagent to insure a sufficient nessler reagent excess for reaction with the ammonia.

Apparatus:

1. Colorometric equipment

One of the following required:

- -Spectrophotometer for use at 400 to 500 nm and providing a light path of 1 cm or longer.
- -Filter photometer, providing a light path of 1 cm or longer and equipped with a violet filter having maximum transmittance at 400 to 425 nm. A blue filter can be used for higher NH₃-N concentrations.
- 2. pH meter, equipped with a high-pH electrode.

Reagents:

- <u>EDTA reagent</u> (stabilizer reagent): Dissolve 50 g disodium ethylenediamine tetraacetate dihydrate in 60 ml of distilled water containing 10 g NaOH. If necessary apply gentle heat to complete dissolution. Cool to room temperature and dilute to 100 ml. (EDTA reagent prevents calcium or magnesium precipitation in undistilled samples after the addition of an alkaline reagent)
- 2. <u>Nessler reagent:</u>

Dissolve 100 HgI₂ and KI in a small quantity of distilled water and add this mixture slowly, with stirring, to a cool solution of 160 g NaOH dissolved in 500 ml distilled water. Dilute to 1 liter. Store in a rubber-stoppered borosilicate glassware and out of sunlight to maintain reagent stability for up to a year under normal laboratory conditions, check

reagent to make sure that it yields the characteristic color with $0.1 \text{ mg NH}_3\text{-N/L}$ within 10 minutes after addition and does not produce a precipitate with small amounts of ammonia within 2 hours.

- Digestion reagent: Dissolve 134 g K₂SO₄ in 650 ml water and 200 ml of conc. H₂SO₄. Add, with stirring, a solution prepared by dissolving 2 g red mercuric oxide, HgO, in 25 ml of 6N H₂SO₄. Dilute the combined solution to 1 liter with distilled water. Keep at a temperature close to 20° C to prevent crystallization.
- 4. <u>Stock ammonium solution</u>: Dissolve 3.189 g anhydrous NH₄Cl, dried at 100° C, in water, and dilute to 1000 ml. (1.00 ml = 1.00 mg N = 1.22 mg NH₃)
- <u>Standard ammonium solution</u>: Dilute 10.00 ml stock ammonium solution to 1000 ml with water. (1.00 ml = 10.00 μg N = 1.22 μg NH₃)

Procedure:

Digestion

To 20 ml of sample placed in a digestion tube, add 5 ml of digestion reagent and mix. Place 4 to 6 glass boiling beads in the digestion tube to promote smooth boiling. Place tubes in block digestor and set the temperature between 100 and 105° C. Maintain this temperature until the water portion of the mixture vaporizes(about 8 to 10 hours). Once the water has boiled off there should be 2 to 3 ml of digestion reagent and sample constituents in the bottom of the digestion tube. Turn the temperature up to 360° C and digest sample until copious white fumes are observed and the digested sample is clear or very pale yellow(straw colored for samples high in organic matter). Cool sample, add 5 to 10 ml of distilled water and swirl until salts go back into solution. Transfer the 5 to 10 ml solution to a glass vial whose weight has been tarred on a digital scale. Use a clean glass disposable pipette to transfer the solution. Rinse down the sides of the digestion tube with 5 to 10 ml of distilled water and transfer this solution to the tarred glass vial. (by experimentation it was determined that 26.20 g correspond to 25 ml, it is much easier to attain 25 ml of solution by use of a scale than to measure volumetrically) Dispose of the transfer pipette after each sample transfer to prevent cross contamination between samples.

*Note: 26.20 g = 25 ml at room temperature (allow sample to fully cool before transfer)

Color development:

Pipette out a suitable aliquot¹ of sample in a 50 ml volumetric flask. Add about 30 ml of distilled water. Then add one drop of 6N NaOH to raise the pH^2 . Add one drop(0.05 ml) of EDTA reagent per 0.25 ml of sample aliquot. Mix gently. Add 2.0 ml of nessler reagent. Make up to volume.

¹ Problems have been encountered with turbidity forming. By using a small sample volume, from 0.25 to 0.5 ml, this problem can be eleviated. The samples analyzed thus far have had fairly high ammonia concentrations. If samples are encountered that contain low concentrations of ammonia the turbidity problem will have to be dealt with in some other manner.

² It was determined that one drop of 6N NaOH per 0.5 ml of sample would raise the pH to about 7.0. When the nessler reagent is added the pH should increase to about 10.5.

The samples should be mixed thoroughly. The reaction should be allowed to proceed for at least 10 minutes after the addition of nessler reagent. Measure absorbance with a spectrophotometer using a wavelength of 400 to 425 nm for 1-cm light path and at 450 to 500 nm for 5-cm light path. Prepare a standard calibration curve using the standard ammonium solution in the acceptable sensitivity range(0.4 to 5 mg NH₃-N/L).

Calculation:

Determine the concentration of NH_3 -N of samples using the standard curve. Make necessary adjustments to the calculation to account for dilutions and express the result as mg NH_3 -N/L.

QA/QC:

- Two triplicates are run for every 25 samples.
 *one of the triplicates is a spiked sample
 *one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.
- 2. One blank is run for every 25 samples. Process blank exactly as samples.
- 3. At least three standards(from the standard ammonium solution) are run for every 25 samples.
 *if the standards do not agree within ± 2 % of the true value, prepare a new calibration curve
- 4. The standards developed should be tested with EPA quality control samples; the samples analyzed value must fall within the stated 95 % confidence interval.

Reference:

- Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-75-80; Method 4500-NH₃ C.
- Methods for the determination of inorganic substances in environmental samples (EPA/600/R 93/100), Aug 1993. Method 350.2.

Determination of Nitrates - Cadmium Reduction Method

Nitrate is reduced almost quantitatively to nitrite in the presence of cadmium(Cd). This method uses commercially available Cd granules treated with copper sulfate to form a copper coating. The nitrite produced thus is determined by diazotizing with sulfanilamide and coupling with N-(1-naphthyl)-ethylenediamine to form a highly colored azo dye that is measured colorimetrically. A correction may be made for any nitrite present in the sample by analyzing without the reduction step. The applicable range of this method is 0.01 to 1.0 mg NO₃-N/L.

Interferences :

- 1. Suspended matter in the reduction column will restrict sample flow. Pre-filter turbid samples through glass fibre or 0.45 micron pore diameter membrane filters.
- 2. Concentrations of Fe, Cu or other metals above several mg/ L lower the reduction efficiency. Add EDTA to samples to eliminate this interference.
- 3. Oil and grease will coat the surface of Cd. Remove with an organic solvent.
- 4. Residual chlorine can interfere by oxidizing the Cd column reducing its efficiency. Use 1 ml of any of the following reagents to remove 1 mg/ L residual chlorine in 500 ml sample.
 a. <u>Sodium sulfite solution</u>: Dissolve 0.9 g Na₂So3 in water and dilute to 1 L. Prepare fresh daily.

b. <u>Sodium thiosulfate</u>: Dissolve 3.5 g Na₂S₂O₃ 5H₂O in water and dilute to 1 L. Prepare fresh weekly.

Apparatus :

- 1. *Reduction column*: The column can be constructed by joining a 10-cm length of 3.5 mm-ID tubing to a 25-cm length of 3.5 mm-ID tubing. Add a TFE stopcock with metering valve to control flow rate.
- 2. Spectrophotometer, for use at 543 nm, providing a light path of 1 cm or longer.

Reagents :

- 1. Cd granules: 40-to 60-mesh Cd granules.
- 2. <u>Dilute Hydrochloric acid</u>, 6N: Dilute 50 ml of concentrated HCl to 100 ml with deionized water.
- 3. <u>Copper sulfate solution</u>, 2%: Dissolve 10 g of CuSO₄ 5H₂O in 250 ml of deionized water and dilute to 500 ml.
- <u>Concentrated NH₄Cl-EDTA</u>: 13 g of ammonium chloride and 1.7 g of disodium ethylenediamine tetraacetate in 900 ml of deionized water. Adjust the pH to 8.5 with ammonium hydroxide. Dilute to 1 L.

- 5. <u>Dilute NH₄Cl-EDTA</u>: 600 ml of concentrated NH₄Cl-EDTA solution and make up to 1 L with deionized water.
- 6. <u>Color reagent</u>: In a 1 L volumetric flask add 10 g sulfanilamide and 1 g N(1-naphthyl)ethylenediamine dihydrochloride to a mixture of 100 ml of concentrated phosphoric acid and 800 ml of deionized water. Bring to volume with deionized water. Store in a brown bottle under refrigeration.
- <u>Stock NO₃ solution</u>: Dissolve 7.218 g KNO₃ in distilled water and dilute to 1000 ml. Preserve with 2 ml of chloroform per liter. This solution is stable for 6 months and contains 1.00 mg NO₃-N /ml.
- 8. <u>Standard NO₃ solution</u>: 10 ml of stock NO₃ solution diluted to 1000 ml with distilled water (10 ppm), then 25 ml of this solution diluted to 250 ml (1 ppm).
- 9. Standard 0.1 ppm NO₃ solution: 25 ml of standard NO₃ solution is diluted to 250 ml.
- <u>Stock NO₂ solution</u>: Dissolve 6.072 g KNO₃ in 500 ml of distilled water and dilute to 1000 ml. Preserve with 2 ml of chloroform per liter. This solution is stable for 3 months and contains 1.00 mg NO₂-N / ml.
- 11. <u>Standard 0.1 ppm NO₂ solution</u>: 10 ml of stock NO₂ solution is diluted to 1000 ml. Measure 1 ml of this solution and dilute to 100 ml with distilled water.
- Note: Prepare all dilutions with nitrate free water of the highest purity possible(2 mega ohm preferred)

Procedures:

Building of columns :

Use old Cd or fresh Cd to produce a column 18.5 cm long. The conditioning of the Cd (old or new) is done in the following way:

The Cd has to be under water at all times. If using old Cd remove Cd by keeping water in the column and emptying the Cd into a beaker with water. Add water to the top of the column and slightly tap on the outside to help wash out the Cd.

Put in 30 ml of 50:50 HCl (2) in a 250 ml erlenmeyer flask. Under the hood scoop Cd out (with a spatula) from the beaker into the 50:50 HCl flask. Swirl 5 minutes, pour off and add another 30 ml of 50:50 ml HCl and swirl for 5 minutes and then again pour off. Add remainder of dilute HCl to the flask, swirl and pour off. The Cd should be silver in color.

Add deionized water to the flask enough to cover the Cd and rinse by swirling about 10 times until the water is clear.

Add $CuSO_4$ solution (3) about 100 ml to the flask and swirl for 2-3 minutes. Blue color may fade. Decant and add another 100 ml of $CuSO_4$ and swirl for 2-3 minutes and decant.

Repeat this process until the $CuSO_4$ solution develops a dark brown to black precipitate. The Cd also should be dark brown to black in color.

Rinse Cd with 25 ml of water for about 10 times until the water stays clear. Make sure that the water is covering the Cd at all times.

Put in new plugs of glass wool at the end of each tube and place columns upright in the stand. Place one spoonful of Cd into the top of each column, tapping the glass lightly. Repeat this until all the Cd is evenly distributed in the columns. The height of the Cd column should be 18.5 cm.

Wash each column with about 200 ml of dilute NH₄Cl-EDTA(5).

Charge each column with standard NO_3 solution (8):

Measure 25 ml of the standard NO_3 solution (8) and add 75 ml of concentrated NH_4Cl -EDTA (4) and run through each column. Rinse with 100 ml of dilute NH_4Cl -EDTA (5).

Column efficiency:

Check each column with standard 0.1 ppm NO₃ solution (9):

Measure 25 ml of standard 0.1 ppm NO₃ solution (9) and add 75 ml of concentrated NH₄Cl-EDTA (4). Then take 10 ml of solution and add 50 ml of dilute NH₄Cl-EDTA (5) and run through each column, rinsing column with 25 ml of dilute NH₄Cl-EDTA. Catch solution in a 100 ml volumetric. Add 4 ml of color reagent (6) within 15 minutes to the volumetric flask and bring up to volume. Allow 15 minutes for the color reagent to develop color before reading on the UV spec at 540 nm.

While running NO_3 standard solution through columns note starting time and ending time of solution through columns. There should be 60 ml of solution being run the column. The columns should run 7-10 ml of solution through the column in 1 minute. Do not count the time it takes to rinse with the 25 ml of dilute NH_4CI -EDTA.

Compare standard 0.1 ppm NO₃ solution to standard 0.1 ppm NO₂ solution (11). Measure out 25 ml of standard 0.1 ppm No₂ solution and add 75 ml of concentrated NH₄Cl-EDTA. Then measure out 10 ml of this solution into a volumetric flask and add 50 ml of dilute NH₄Cl-EDTA and 4 ml of color reagent, then bring to volume with deionized water. Do not run the NO₂ solution through the columns.

All NO₃ solutions reduced through the column and NO₂ solutions should read the same absorbance on the UV spec. This is to verify the reduction column efficiency. Reactivate the Cd when efficiency of reduction falls below 75%.

Blank:

Run a blank of 60 ml dilute NH_4Cl -EDTA through one column and rinsing with 25 ml of dilute NH_4Cl -EDTA. Catch in a 100 ml volumetric flask and add 4 ml of color reagent. Bring it up to volume.

Sample analysis:

Rinse column with 50 ml of dilute NH_4Cl -EDTA solution. Do not allow air into the column. Allow solution to drain to near the top of the metal.

Add 10 ml of sample (less if NO_3 conc. is high) and 50 ml NH_4Cl -EDTA into a erlenmeyer flask. Pour the contents of the flask into the reduction column and catch in a 100 ml volumetric flask.

Add 25 ml of dilute NH4Cl-EDTA to the reduction column and catch in the same volumetric flask.

Add 4 ml color reagent to the volumetric flask with the sample within 15 minutes and bring to volume. Allow the color to develop at least 10 minutes. Read the sample within 2 hours at 540 nm.

Standards:

- <u>Concentrated NO₃ solution</u>: Dissolve 0.3609 g KNO₃ in 1 liter of deionized water. This solution contains 50 μg N /ml as NO₃. Keep under refrigeration.
- <u>Dilute NO₃ solution</u>: Add 20 ml of concentrated NO₃ solution to 500 ml volumetric flask and bring up to volume. This contains 2 μg N/ml as NO₃. Keep under refrigeration.
- 3. Add 50 ml of dilute NH₄Cl-EDTA to each of the following and pour into the reduction column. Proceed with the color development in the similar manner as for the sample. Read the color developed at 540 nm.

Calculations:

Obtain the standard curve by plotting absorbance of standards against the NO₃ concentration. The sample concentrations are calculated from the standard curve.

The eluant coming from the Cd column contains NO_2 initially present in the sample plus that formed from reduction of NO_3 present in the sample. The NO_2 concentration can be separately determined and corrected for by going through all the steps mentioned above for the sample analysis except the reduction process of the Cd column.

Alternative option is to remove the NO_2 content of the sample before it is passed through the Cd column. This is done by treating the sample with 1 ml of sulfamic acid solution (Dissolve 2 g of <u>sulfamic acid</u> in 100 ml of water. Store it under refrigeration.)

QA/QC:

- 1. Two triplicates are run for every 25 samples.
 - *one of the triplicates is a spiked sample
 - *one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.
- 2. One blank is run for every 25 samples. Process blank exactly as samples.
- 3. At least one standard (from the standard NO₃-N solution) are run for every 25 samples.

*if the standards do not agree within ± 2 % of the true value, prepare a new calibration curve, and check column efficiency.

4. The standards developed should be tested with EPA quality control samples; the samples analyzed value must fall within the stated 95% confidence interval. If the samples fall outside the confidence interval, they are rerun. If the EPA standards still fall outside the confidence interval new working standards are prepared.

Precision and Bias:

In a single laboratory using wastewater samples at concentrations of 0.04, 0.24, 0.55 and 1.04 mg NO_3^- + NO_2^- -N/L, the standard deviations were +0.005, +0.004, +0.005, and +0.001, respectively. In a single laboratory using wastewater with additions of 0.24, 0.55, and 1.05 mg NO_3^- + NO_2^- -N/L, the recoveries were 100%, 102%, and 100%, respectively.

Note:

- The Cd granules used are usually of the size 1 mm in diameter by 2 mm in length or smaller. Suitable Cd metal can be obtained from fisher scientific Co. (catalog no.c-3, purified, granular Cd). If granules <1 mm in diameter are used, column flow rates may have to be increased to ensure complete recovery of added NO₃ because of potential reduction of NO₂ formed in the column. Control of column flow rates is important because slow rates (~8 ml/min) lead to reduction of NO₂, whereas very rapid rates do not give complete reduction of NO₃.
- 2. If the Cd metal is exposed to air through failure to maintain liquid on top of the metal, the reduction column should be repacked, because air bubbles in the column seriously interfere with NO₃ reduction.
- 3. Although the solution pH is not critical during the reduction step, buffered extracts should be adjusted to near pH 7 to ensure quantitative conversion of NO_3 to NO_2 . The solution pH after addition of the color reagent should be around 1.5.
- 4. The rate of color development in this method is very rapid; full color development is obtained in 10 min at 25°c. The color formed is stable for several hours. It is assumed that the fading observed using this method is probably due to exposure of the azo compound to light. It is recommended therefore, that samples prepared for colorimetric measurement be stored in dark if they cannot be analyzed soon after development of the color.
- 5. It is essential that controls be included in each series of analysis to allow for the color produced by the reagents employed.
- 6. The coupling reagent used in this method has the disadvantage that is it becomes discolored and tends to precipitate even when stored in the dark. This decreases the reagent blank and decreases the sensitivity and precision of the method. However, the reagent is stable for at least 1-2 months if it is stored with protection from light in a refrigerator. It is better if the color reagent is prepared monthly afresh.

References:

- Standards methods for the Examination of water and waste water, 18th Edition p. 4-89, method 4500 NO₃-E.
- Page, A. L., R. H. Miller, D. R. Keeney. 1982. <u>Methods of soil analysis, Part 2. Chemical and microbiological properties of soil analysis</u>. Amer. Soc. Agron, Inc. and Soil Sci. Soc. Amer.Inc, Madison, Wisc.

Procedures for the Determination of Total Solids

A well-mixed sample is evaporated in a weighed dish and dried to constant weight in an oven at 103 to 105° C. The increase in weight over that of the empty dish represents the total solids.

Interferences:

- 1. Highly mineralized water with a significant concentration of calcium, magnesium, chloride, and/or sulfate may be hygroscopic and require prolonged drying, proper desiccation, and rapid weighing.
- 2. Exclude large, floating particles or submerged agglomerates of nonhomogeneous materials from the sample.
- 3. Disperse visible floating oil and grease with a blender before withdrawing a sample portion for analysis.
- 4. Because excessive residue in the dish may form a water-trapping crust, limit sample to no more than 200 mg residue.

Apparatus:

- 1. Drying dishes: 10-Ml Aluminum disks or porcelain crucibles.
- 2. <u>Desiccator</u>, provided with a desiccant containing a color indicator of moisture concentration.
- 3. Drying oven, for operation at 103 to 105°C.
- 4. <u>Analytical balance</u>, capable of weighing to 0.1 mg.

Procedures:

- 1. Heat clean evaporating dish to 103 to 105°C for 1 h. Store dish in desiccator until needed. Weigh immediately before use.
- 2. Choose a sample volume that will yield a residue between 10 and 200 mg. When very low total suspended solids are encountered (less than 10 mg/L), less residue may be

collected; compensate by using a high-sensitivity balance (0.002 mg). Transfer a measured volume of well-mixed sample to preweighed dish and evaporate to dryness in a drying oven. Stir the sample during transfer. If necessary, add successive sample portions to the same dish after drying. Lower temperature when evaporating in the drying oven to approximately 2°C below boiling to prevent splattering.

- 3. After evaporation, dry sample for at least 1 h at 103 to 105° C.
- 4. Cool dish in desiccator to balance temperature.
- 5. Weigh the dish.
- 6. Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained, or until weight loss is less than 4% of previous weight or 0.1 mg, whichever is less. When weighing dried sample, be alert to change in weight due to air exposure and/or sample degradation. Duplicate determinations should agree within 5% of their average.

Calculations:

 $(A - B) \times 1,000$

mg total solids/L =

sample volume, ml

where:

A = weight of sample + dish, mg, and B = weight of dish, mg.

QA/QC:

- 1. One duplicate is run for every 40 samples.
- 2. One blank is run daily. A blank sample is prepared by adding 100 ml distilled water.

Precision and Accuracy:

Single-laboratory duplicate analyses of 41 samples of water and wastewater were made with a standard deviation of differences of 6.0 mg/ L.

Reference:

Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.2-54; Method 2540 B.

ANALYSIS OF POULTRY LITTER

Procedures for the Determination of Poultry Litter Moisture Content

A well-mixed sample is dried to constant weight in a convection oven at 60°C. The decrease in weight represents the moisture.

Interferences:

- 1. Some volatile solids may be lost with prolonged drying time.
- 2. Due to excessive residue in the dish a water-trapping crust may form. Limit sample to no more than 2 grams of residue.

Apparatus:

- 1. Drying dishes.
- 2. <u>Convection drying oven</u>, for operation at 60° C.
- 3. <u>Desiccator</u>, provided with a desiccant containing a color indicator of moisture concentration.
- 4. Analytical balance, capable of weighing to 0.1 mg.

Procedures:

- 1. Heat drying dishes to 103 to 105°C for 1 h. Store dishes in desiccator until needed. Weigh immediately before use.
- 2. Choose a sample volume that will yield a residue between 1 to 2 grams. Transfer wellmixed sample to preweighed dish.
- 3. Dry sample overnight at 60° C in a convectional oven.
- 4. Cool dish in desiccator to balance temperature.
- 5. Weigh.

Calculations:

A - BMoisture, $\% = \frac{A - B}{A - C} \times 100$

where,

- A = weight of sample + dish, mg, wet,
- B = weight of sample + dish, mg, dry and
- C = weight of dish, mg.

QA/QC:

One duplicate is run for every 40 samples.

Reference:

Standard Methods for the Examination of Water and Wastewater, 16th Edition, p.93; Method 209 A.

Dry Manure Analysis Methods, Agricultural Services Lab, Sep 1, 1992.

Procedures for the Determination of Total Phosphorus for Poultry Litter samples

A well-mixed sample is air dried and ground in a Grinding Mill (20 mesh). This sample also used in testing TKN.

Determine moisture content of the sample according to procedure outlined under Determination of Poultry Litter Moisture Content, page 157 of Appendix b.

The analysis of total phosphorus of the sample is carried out as per the procedures outlined on page 141, Determination of total phosphorus for runoff water samples.

Reagents:

- 1. $6NH_2SO_4$: Dilute 16.7 ml conc. H_2SO_4 to 100 ml.
- 2. Digestion reagent:

Dissolve 134 g K_2SO_4 in 650 ml water and 200 ml conc. H_2SO_4 . Add, with stirring, a solution prepared by dissolving 2 g red mercuric oxide, HgO, in 25 ml $6N H_2SO_4$. Dilute the combined solution to 1 L with water. Keep at a temperature close to 20^oC to prevent crystallization.

Procedure:

- 1. Air dry the sample and grind in a Wiley Mill (20 mesh).
- 2. Weigh 1.00 g of dried, ground sample into a 100-ml digestion tube.
- 3. Add 10 ml of digestion reagent. Try to analyze a sample which has an amount of 0.05 to 1.0 mg P for ideal color development.
- 4. Allow the samples sit overnight for predigestion in a fume hood. Samples can be allowed to sit just 1 hour if there is no time for predigestion. However, some samples may foam up or splatter upon digestion. Extra care must be taken if adequate predigestion time is not allowed like wearing long sleeved gloves and increasing the temperature gradually.

- 5. Place tubes in block digestor and digest sample for 1 hour at 160°C and then for 1½ hours at about 380°C till white fumes are observed and the digested sample is clear or very pale yellow in color. Cool sample and dilute to 25 ml with distilled water.
- 6. The color development of the digested sample and the calibration curve is carried out similar to the ascorbic acid method as outlined for the analysis of water samples.

QA/AC:

- 1. One triplicate sample is run for every 25 samples.
- 2. One blank is run daily.
- 3. One EPA standard is run for every 25 samples.
- 4. Spike at least one sample for every 50 samples.
- 5. Process standards and blank exactly as the samples. Run at least one blank and one phosphate standard with each set of samples. If the standards do not agree within ± 2% of the true value, prepare a new calibration curve.

Reference:

Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-108 to 4-113; Method 4500-P.C.

Dry Manure Analysis Methods, Agricultural Services Lab, Sep 1, 1992.

Methods for the determination of inorganic substances in environmental samples (EPA/600/R-93/100), Aug 1993. Method 365.2.

Procedures for the Determination of Total Kjeldahl Nitrogen for Poultry Litter

A well-mixed sample is air dried and ground in a Grinding Mill (20 mesh). This sample also used in testing Total Phosphorus.

Determine moisture content of the sample according to procedure outlined on page 157 of Appendix b Determination of Poultry Litter Moisture Content.

The analysis of TKN of the sample is carried out as per the procedures outlined on page 147 **Determination of Total Kjeldahl Nitrogen** for runoff samples.

Reagents:

1. $\underline{6NH_2SO_4}$: Dilute 16.7 ml conc. H_2SO_4 to 100 ml.

2. Digestion reagent:

Dissolve 134 g K₂SO₄ in 650 ml water and 200 ml conc. H₂SO₄. Add, with stirring, a solution prepared by dissolving 2 g red mercuric oxide, HgO, in 25 ml 6N H₂SO₄. Dilute the combined solution to 1 L with water. Keep at a temperature close to 20^oC to prevent crystallization.

Procedure:

- 1. Weigh 1.00 g of dried, ground sample into a 100-ml digestion tube. Carefully add 10 ml digestion reagent to digestion tube containing sample.
- 2. Allow the samples sit overnight for predigestion in a fume hood. Samples can be allowed to sit just 1 hour if there is no time for predigestion. However, some samples may foam up or splatter upon digestion. Extra care must be taken if adequate predigestion time is not allowed.
- After predigestion, place tubes in block digestor and digest sample for 1 hour at 160°C and then for 1½ hours at about 380°C till white fumes are observed and the digested sample is clear or very pale yellow in color. Cool sample and dilute to 25 ml with distilled water.
- 4. The color development of the digested sample and then calibration curve is carried out similar to the nesslerization method as outlined for water sample analysis.

QA/AC:

- 1. Two triplicates are run for every 25 samples.
 - *one of the triplicates is a spiked sample*one triplicate is determined at the start of the analysis and the other triplicate is prepared at the end.
- 2. One blank is run for every 25 samples. Process blank exactly as samples.
- 3. At least three standards(from the standard ammonium solution) are run for every 25 samples.
 *if the standards do not agree within ± 2 % of the true value, prepare a new calibration curve
- 4. The standards developed should be tested with EPA quality control samples; the samples analyzed value must fall within the stated 95 % confidence interval.

Reference:

- Standard Methods for the Examination of Water and Wastewater, 18th Edition, p.4-96: Method 4500-Norg C.
- Methods for the determination of inorganic substances in environmental samples (EPA/600/R-93/100), Aug 1993. Method 351.3.

VITA

Clint H. Olson

Candidate for the Degree of

Master of Science

Thesis: EFFECTS OF RAINFALL, SLOPE, AND VEGETATION HEIGHT ON RUNOFF WATER QUALITY FROM FESCUE PLOTS TREATED WITH POULTRY LITTER

Major Field: Biosystems and Agricultural Engineering

Biographical:

- Personal Data: Born in Waynoka, Oklahoma, on January 31, 1969, the son of Chris and Marilynn Olson.
- Education: Graduated from Waynoka High School, Waynoka, Oklahoma in May 1987; received Bachelor of Science degree in Agricultural Ecology from Northwestern Oklahoma State University, Alva, Oklahoma in May 1992. Completed the requirements for the Master of Science degree with a major in Biosystems and Agricultural Engineering at Oklahoma State University in July 1995.
- Experience: Raised on a farm and ranch near Waynoka in Woods County Oklahoma; assisted his father in farm and ranch operations; employed by Oklahoma State University, Department of Biosystems and Agricultural Engineering as a graduate research assistant; Oklahoma State University, Department of Biosystems and Agricultural Engineering, 1992 to present
- Professional Memberships: ASAE The Society for Engineering in Agriculture, Food, and Biological Systems