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Freeze Separation To Remove Nutrients from Liquid Swine Manure

by

Amanda Rose Willoughby



A thesis submitted to the Faculty of Graduate Studies and Research in partial fulfillment of the requirements for the degree of Master of Science

in

Geoenvironmental Engineering

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ABSTRACT

Thin-layered freezing was used in a pilot-scale field study for the treatment of liquid swine manure. It was found to be a viable mechanism for extracting a large volume of nutrients from liquid swine manure. More than 75% of the nutrients were recovered and concentrated to one-third the original volume. A significant volume of treated water was produced, which may be a valuable resource for use as recycle or irrigation water on farms. The thin-layered freezing process also removed the objectionable odour of the liquid swine manure.

The technical components of the pilot-scale field system and its operation were examined as a basis for developing a full-scale treatment system at intensive livestock operations. The capital cost for constructing a new system was \$38/m³ of manure treated compared to \$25/m³ treated for retrofitting an existing dual earthen manure storage system. The annual operating costs were found to be \$0.45/m³.

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LIST OF ABBREVIATIONS AND SYMBOLS

| °C | degree centigrade |
|------------------------------|--|
| AEGRF | Applied Environmental Geochemistry Research Facility |
| DOC | dissolved organic carbon |
| EC | electrical conductivity |
| ID | inside diameter |
| ILO | intensive livestock operation |
| К | potassium |
| mS | millisiemens |
| Ν | nitrogen |
| NH₄⁺ | ammonium ion |
| NO ₃ ⁻ | nitrate |
| NO2 ²⁻ | nitrite |
| Р | phosphorous |
| PVC | polyvinyl chloride |
| SRTC | Swine Research and Technology Center |
| S | sulfur |
| SO4 ²⁻ | sulphate |
| TDN | total dissolved nitrogen |
| TN | total nitrogen |

1 INTRODUCTION

1.1 Background

The research conducted in this study focused on the potential for thin-layered freezing to add value to liquid swine manure, which is currently handled as a large volume diluted waste source. To evaluate the viability of this treatment process, the development of a pilot scale thin-layered freezing system at an existing swine operation was necessary. The system was constructed and operated over one winter and spring to determine the percent nutrient extraction as a result of freeze separation and the value of thawed effluent as a fertilizer substitute and reusable water. A review of current literature verified that various freeze separation systems have been used to treat several types of wastes, however there were no examples of previous work using thin-layered freezing to treat liquid swine manure. The following sections are an introduction to the work conducted in this study.

Liquid swine manure is a dilute solution of 1 to 10% solids that contains valuable nutrient components, including nitrogen (N), potassium (K), sulfur (S), and phosphorous (P). Field application of manure can enhance long-term soil productivity, as it is a rich source of organic material, supplies macro and micronutrients to crops and it increases microbial activity thereby increasing nutrient availability (Cassman et al., 1995; Koelsch, 2001). The dilute nature of liquid manure makes it costly to transport, with economical transport distances less than 45 km, and multiple field applications are required to obtain the appropriate nutrient load to crops (McGill, 1997). The result has been the storage of massive volumes of liquid manure.

Physical, chemical, and biological systems that are utilized for treating industrial and human wastewater can be used to treat liquid hog manure. The system that is utilized depends on the pertinent regulations and standards, the volume, composition and concentration of manure, soil type and crop selection, climate and location, and the cost to install and operate the system (Zering, 2000). The majority of farmers utilize a dual earthen manure storage system that incorporates solids

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separation, 9 to 12 months of manure storage and subsequent land application (Day and Funk, 1998, Fleming and Ford, 2002; Jones, 1999; Miner et al., 2000). Based on statistical data from 2001, more than 85% of the hog sector used liquid manure storage systems with a capacity of more than 250 days (Statistics Canada, 2003). Almost 10% of the farms had liquid storage capacity for greater than 400 days.

Freeze separation is the concentration of dissolved solutes and/or suspended solids in aqueous liquids during freezing (Jean et al., 1999; Martel, 1999). The process has been used for centuries to produce valuable drinking water from saline or brackish water in cold regions. In recent years the process has been examined as a low cost natural alternative for treating various municipal and industrial wastewaters. The theory of freeze separation was used to develop a thin-layered freezing system to concentrate the nutrient components into a significantly smaller volume. Increasing the concentration of nutrients and decreasing the volume of liquid for transport increases the economic value of manure and allows further transport distances and more efficient field application. The purified water component may be used for animal drinking water or barn wash water. In addition, the freezing process changes the offensive odour of liquid swine manure, which has implications for the social impacts of locating swine operations (Huber and Palmateer, 1985; Willoughby et al., 2001).

1.2 Objectives

The purpose of this research is to develop a thin-layered freezing system to treat liquid swine manure at intensive livestock operations. The research includes the design and implementation of a pilot scale thin-layered freezing system. Based on the results of the pilot scale system a farm scale system is developed at the conceptual level, including operational procedures and the cost to construct and operate the system.

1.3 Methodology

A pilot scale thin-layered freezing system was designed and constructed at the University of Alberta Swine Research and Technology Center (SRTC) in Edmonton, Alberta. Liquid swine manure was frozen in thin-layers in a freezing pit over winter

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and subsequently thawed over the spring. The concentration of nutrients (nitrogen, phosphorous, sulphur, and potassium) was measured in the raw manure and in the thawed effluent to quantify the percent of nutrients that were extracted and concentrated during the freezing process. The technical components and system operation were also examined as a basis for developing large-scale treatment systems at intensive livestock operations.

Following completion of the field scale study, a thin-layered freezing system was conceptually developed for an average size swine operation in central Alberta. The cost to construct and operate the system was determined for both a new operation and an existing farm that utilizes dual earthen manure storage. The cost to utilize thin-layered freezing per cubic meter of liquid treated was also examined.

Several important recommendations are made based on the results of the pilot scale field test and the associated farm-scale implementation. The significance of nutrient recovery and concentration for the agricultural industry and the need to view manure as a resource and not a waste are discussed.

1.4 Organization of Thesis

This thesis has been written in paper format. Chapter 1 introduces why this study was conducted and outlines the structure of the research. In Chapter 2 the pilot scale field tests are described, including the design and implementation of the field tests, and the results of the tests. Chapter 3 details the requirements for farm-scale implementation of the method based on the results and recommendations described in Chapter 2. The final section, Chapter 4, concludes and summarizes the results of this study and presents the need for additional work.

1.5 References

- Cassman, K.G., Steiner, R., and Johnston, L. 1995. Long-term experiments and productivity indexes to evaluate the sustainability of cropping systems. Chapter 11 in Agricultural Sustainability: Economic, Environmental and Statistical Considerations, V.Barnett, R. Payne, and R. Steiner, Editors. West Sussex, England: John Wiley and Sons.
- Day, D.L., and Funk, T.L. 1998. Processing manure: physical, chemical, and biological treatment. In: Animal Waste Utilization: Effective Use of Manure as a Soil Resource, J.L. Hatfield and B.A. Stewart, Editors. Chelsea Michigan: Ann Arbor Press.
- Fleming, R., and Ford, M. 2002. Comparison of storage, treatment, utilization, and disposal systems for human and livestock wastes. Internal Research Report, Ridgetown College, University of Guelph, Ontario.
- Huber, D. and Palmateer, G. 1985. A joint experimental project between southwest region of the Ministry of the Environment and Group Delta in the storage and renovation of sewage effluent by conversion to snow. Internal Research Report, Ontario Ministry of the Environment, Southwest Region.
- Jean, D.S., Lee, D.J., and Wu, J.C.S. 1999. Separation of oil from oily sludge by freezing and thawing. Water Resources, **33**(7): 1756-1759.
- Jones, J.R. 1999. Animal agriculture: waste management practices: Report to the Honorable Tom Harkin, Ranking Minority Member, Committee on Agriculture, Nutrition, and Forestry, U.S. Senate. United States General Accounting Office, Washington, D.C.
- Koelsch, R. 2001. Lesson 1: Principles of Environmental Stewardship. In Manure Management Curriculum 2002, Saskatoon, Saskatchewan.
- Martel, C.J. 1999. Effect of dissolved solids on freeze-thaw conditioning. In Proceedings of the Cold Regions Engineering 10th International Conference, Lincoln, New Hampshire 16 to 19 August pp: 306-306. Edited by J.E. Zufelt.
- McGill, W.B. 1997. Sustainability of manure management: nutrient retention and cost-benefit analysis. Final Report CAESA Project RES-056-93.
- Miner, J.R., Humenik, F.J., and Overcash, M.R. 2000. Managing livestock wastes to preserve environmental quality. Iowa State University Press, Ames, Iowa.
- Statistics Canada. 2003. Manure storage in Canada 2003: Vol. 1, No. 1. Catalogue no. 21-021-MIE. Agriculture Division, Ottawa.

- Willoughby, A.R., Biggar, K., and Sego, D.C. 2001. Nutrient extraction from hog barn wash water using freeze separation. Internal Research Report, University of Alberta, Department of Civil and Environmental Engineering. Edmonton, Canada.
- Zering, K. 2000. *How to select an alternative manure treatment system*. Presented at the North Carolina Pork Conference, January 12. Fayetteville, North Carolina.

2 FREEZE SEPARATION FOR THE REMOVAL OF NUTRIENTS FROM LIQUID SWINE MANURE: EXPERIMENTAL EVALUATION

2.1 Introduction

Until approximately 20 years ago farms utilized a sustainable approach to manure management; all manure produced was used to supply required nutrients to crops. The last two decades have seen a movement towards intensive livestock operations (ILO) to remain competitive on the world market. The number of farms has decreased across Canada and the average number of animals per farm has increased from 50 to over 1000. These large-scale operations produce far more manure than can be applied as nutrients to lands adjacent to operations. In addition it is not economical to ship liquid manure more than approximately 45 km from its source for field application, and due to the dilute nature of the manure several applications are needed to meet crop nutrient requirements. Coupled with increasingly stringent environmental regulations, farmers are challenged to find an economically feasible means of treating and safely disposing of massive volumes of waste.

Current manure treatment technologies are dominated by anaerobic digestion in a dual earthen manure storage system, which has many drawbacks including odour production, environmental impacts, and long-term storage issues. This research investigates the use of natural freezing to treat liquid swine manure in a thin-layered freezing system at the pilot scale. In the laboratory setting the process demonstrated significant promise as a means of concentrating the nutrient volume (Willoughby et al., 2001). This economically feasible waste-treatment technology reduces the environmental impact of massive earthen manure storage, increases the economic value of manure, and provides an environmentally sustainable solution to manure management. It has the potential to add significant value to the manure as a fertilizer, reduce odour, generate reusable water, and render the solids much easier to handle and manage.

2.1.1 Research Objectives

The purpose of this research was to determine the viability of using freeze

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separation for nutrient removal and purification of the water component of liquid hog manure in a pilot scale field study. The objectives of the research include:

- Determine the percent nutrient extraction as a result of freeze separation based on a complete mass balance of system inflow and outflow;
- Determine the nutrient value of the effluent for use in field applications for crop nutrients;
- Prove out the design of the freezing pit and collection system in the field and determine changes needed for future designs.

2.1.2 Background on Freeze Separation

The thin-layered freezing system developed in this study was based on the theory of freeze separation and freezing point depression of saline or high ionic strength solutions. Freeze separation is the concentration of dissolved solutes and/or suspended solids in aqueous liquids during freezing. It is also referred to as freeze concentration (EPRI, 1987), freeze-thaw conditioning (Jean et al., 1999; Martel, 1999) or freeze crystallization (Suthersan, 1997; Heist, 1981). Chalmers (1959) stated ice crystals grow by the addition of water molecules to its highly crystallographic structure. This structure does not allow the substitution of foreign molecules for hydrogen or oxygen during freezing (Pounder, 1965). As a result, solutes and impurities within the water are rejected ahead of the freezing front and are concentrated in the remaining unfrozen liquid. If the freezing rate is slow, pure ice will form and the remaining solution will be enriched with solutes (Konrad and McCammon, 1990). The increased solute concentration causes a decrease in the freezing point of the enriched solution and results in a freezing temperature below that of pure ice (Andersland and Ladanyi, 2004). This theory has been applied to treat salt water and other industrial wastewaters and formed the basis of laboratory testing on liquid hog manure starting in 2001.

The freeze separation process was completed in two stages: winter freezing and spring thaw. During the freezing stage manure is placed in 50 to 75 mm thick layers and allowed to freeze. During freezing, nutrients and dissolved impurities are forced downward ahead of the freezing front, resulting in a thin layer of highly concentrated

nutrient rich fluid. After each layer is frozen another layer is placed on top. Freezing point depression allows the concentrated fluid to remain in the liquid state and form vertical drainage channels through the ice mass, draining to a collection system at the base of the frozen layers. The concentrated fluid is collected and stored in a separate tank. As the air temperature increases above zero in the spring, the ice begins to melt. The initial melt water flushes any remaining impurities with it leaving nearly pure ice. This purified ice thaws as the final melt water in late spring and is collected and stored separately. When all melting is completed, the solids can be removed and treated separately.

2.1.3 Examples of Freeze Separation Treatments

Freeze separation has been utilized in cold regions for centuries as a natural means of producing fresh drinking water from sea ice. Many researchers have developed the conceptual model of freeze separation for the treatment of various aqueous saline solutions and wastewaters. Various aspects of desalination using freeze separation have been investigated by Elmore (1968), Fertuck (1969), Krepchin (1985), Spyker (1981), Terwilliger and Dizio (1970), and Weeks and Ackley (1982). Freeze separation has been investigated as a means of treating municipal wastewater and wastewater treatment sludges (Halde, 1980; Huber and Palmateer, 1985; Martel, 1989, 1993, 1999; Muller and Sekoulov, 1992; and Parker et al., 2000). Mine wastewaters, including mine tailings and acid mine water have been treated using freeze separation (Applied Sciences Laboratory, 1971; Gao, 1998; Stahl and Sego, 1995). Petroleum refinery oil sludge (Jean et al., 1999), pulp mill wastes (Gao, 1998; Grulich, 1969; Kenny et al., 1991), and various industrial wastewaters (Baker, 1970; Campbell and Emmerman, 1972) have been treated using various freeze separation applications.

The use of thin-layered freezing to treat liquid swine manure at the pilot scale has not been investigated previously. Delta Engineering in conjunction with Alberta Agriculture Food and Rural Development (1997) has assessed spray freezing to treat liquid swine manure at the field scale and Gao (1998) investigated impurity rejection and concentration of swine wastewater using spray freezing as a treatment alternative. Thin-layered freezing has been investigated extensively by Martel (1989) to treat and condition wastewater treatment sludges. The use of thin-layered freezing for the treatment of liquid swine manure allows the system to be designed to utilize already existing earthen manure storage systems on intensive livestock operations.

2.1.4 Liquid Swine Manure Characteristics

Liquid swine manure is an odourous large volume waste produced in flushed manure transport systems where water is added to the raw manure for handling and transport (Miner et al., 2000). The liquid is a dilute solution of 1 to 10% solids and is approximately ten times the original volume of manure produced. The chemical and physical makeup depends on the physical plant, feed rations, the manure management system, and animal weight. The manure is a valuable resource that can be used as a substitute for commercial fertilizer due to the nutrient components, including nitrogen (N), potassium (K), sulfur (S), and phosphorous (P). Manure application can enhance long-term soil productivity, as it is a rich source of organic material, supplies macro and micronutrients to crops and it increases microbial activity thereby increasing nutrient availability (Cassman et al., 1995; Koelsch, 2001).

According to McGill (1997) over 155,000 Mg of N and 42,000 Mg of P are produced per year in Alberta and the estimated annual value of nutrients excreted by confined livestock animals in Alberta in 1991 was \$167 million. The dilute nature of liquid swine manure requires multiple rates of application to meet plant crop requirements and transportation costs are only economical over short distances from the source (Prairie Agricultural Machinery Institute, 2001). Thin-layered freezing in the laboratory has shown concentration of the nutrient component to 10 to 15% of the original volume (Willoughby et al., 2001). Decreasing the volume and concentrating the nutrient component significantly increases the value of manure for fertilizer replacement as it decreases the number of applications required and allows further transport from the source.

2.2 Experimental Setup

Permission was obtained from the University of Alberta Swine Research and Technology Center (SRTC) to complete the construction and operation of a thin layered swine manure freezing system during the winter and spring of 2004. Preliminary testing initiated at the SRTC in the winter of 2003 provided insight on technical challenges that had to be resolved for successful implementation of the field system. A detailed description of the 2003 system and results is contained in Field Test 2003 Summary Report by Willoughby et al. (2003). A summary of the 2003 recommendations has been included in Appendix A. Construction of the 2004 system took place during November and December of 2003. This section describes the design, construction and operational procedures of the 2004 thin-layered freezing system.

2.2.1 Test Equipment and System Setup

The SRTC is a state-of-the-art facility that provides a site for integrated swine research on nutrition, reproduction, environmental management and medical research. It houses approximately 300 farrowing sows and up to 1700 young pigs, producing an average of 60, 000 to 80, 000 liters of manure per week. All manure produced at the SRTC is pumped from the barn to a lift station north of the building. It is then hauled offsite at a cost of approximately \$40, 000 per year. The farm is located in the center of a city residential area and as such earthen manure storage of the manure is not feasible. The freeze separation system designed for this research was not intended to manage all of the manure produced during the winter of 2004. The system was designed as a small-scale pilot system to validate laboratory results for nutrient extraction using freeze separation. The results of this pilot study will be used to design a system for a typical farm operation utilizing earthen manure storage and field application of manure following freeze separation (Chapter 3).

The 2004 system was designed based on the recommendations from testing in 2003 and also with the intent of reusing much of the 2003 setup. The design incorporated reuse of the 2003 freezing pit and manure storage tanks. The 2004 thin-layered

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freezing system is illustrated in Figures 2.1, 2.2, and 2.3, and includes four major components: earthen manure storage, the freezing pit, effluent collection system, and layer application equipment.

The freezing pit (Figures 2.2 and 2.4) was an excavated pit lined with a 30-mil polyvinyl chloride (PVC) geomembrane. The purpose of the freezing pit was to contain the liquid swine manure while it freezes and to prevent the migration of any contaminants into the subsurface. To minimize boundary effects during freezing and thawing the dimensions of the pit base were 8.5 m by 9.0 m. Taking into consideration the number of freezing days in a typical Edmonton winter the freezing pit was designed for a maximum ice depth of 2 m with 30 cm of freeboard. The base of the pit was 1.5 m below grade with 1:1 side slopes to allow for construction of the collection system. The base of the pit was designed with a 3 percent grade towards the collection piping.

The effluent collection system collects thawed effluent from the freezing pit and transports it to storage. Figures 2.5 and 2.6 show the layout of the collection piping in the freezing pit and the piping from the pit to the collection sump. Single slotted PVC piping with a 5 cm diameter was used within the pit to collect effluent and transport it to the collection sump via 5 cm solid PVC piping. All of the piping was lined with heat tape in the event that the pipe contents freeze during the winter months. A boot installation was used where the effluent piping exited the PVC liner to prevent migration of manure out of the pit at this junction. The boot installation is shown in Figure 2.7.

A drainage layer of sand and gravel was required over the collection piping within the freezing pit. A 30 cm layer of 1 cm rounded pea gravel was placed over the collection piping followed by a 10 cm layer of washed concrete sand. The drainage layer provided protection for the piping, allowed manure to be placed and frozen without running directly into the collection pipes, and acted as a filter to remove fine particulate as thawed effluent flowed down through the layers.

The collection sump (Figure 2.6) consisted of an insulated 400 L polyethylene barrel containing a level actuated sump pump and heat tracing. The effluent piping entered

the barrel through a bulkhead fitting and was equipped with a shutoff valve to prevent migration of manure into the collection sump during layer application. The level actuator ensured the liquid level in the collection sump did not rise above the effluent piping.

The layer application equipment, illustrated in Figure 2.8, consisted of a galvanized steel dispenser, flexible and rigid piping, and a wastewater pump. The manure dispenser was designed to prevent thermal erosion of the ice during application and to ensure an even and laminar distribution of liquid across the ice surface during application. The dispenser was equipped with wheels to enable movement across the ice surface. The metal surface of the dispenser also provided a means of removing sensible heat from the manure prior to placement.

Several components of the 2004 system were added or changed in order to reuse the 2003 freezing pit. Additions to the system included a collection sump located outside of the freezing pit rather than inside the pit, and a new drainage and collection system. The stages of construction included: earthwork and reconstruction of the pit base and slopes; construction of the effluent collection sump and associated piping; PVC membrane placement and construction of the boot seal around the effluent piping; construction of the drainage system including collection piping with heat tape and gravel and sand placement.

2.2.2 Thin Layered Freezing System Operation

The operation of the thin-layered freezing system can be broken down into two components: manure placement and effluent collection. Based on prior experience, the recommended procedures for successful implementation followed in this experiment are described below. A detailed description of the test implementation is described in Section 2.3.1 of this report.

Prior to the placement of any manure, a thin layer of water had to be frozen to seal off the collection system to prevent the migration of manure through the drainage layers into the collection system during initial manure placement and freezing. The water was sprayed on the sand in three to four applications. The applications were intended to saturate the top 5 cm of sand and were placed when the ambient air

temperature was less than 0 °C to allow the saturated sand to freeze. Water application was stopped when there was sufficient evidence of water pooling on the ice during spraying and when a thin layer of ice covered the entire base of the pit.

After the ice layer was established manure placement commenced. Two conditions were chosen to ensure the manure would freeze and to prevent thermal erosion of underlying previously frozen layers: the minimum daily temperature must be less than -10 °C and the maximum daily temperature should be no greater than 0 °C. If both of these temperatures were forecasted, manure application proceeded. In addition, the manure being placed had to be colder than 2 °C to minimize the amount of sensible heat removal and the time to freeze, and to prevent thermal erosion. The detailed procedures for layer placement are specified in Appendix B. Layer placement continued over the winter months until air temperatures warmed above the specified temperature conditions.

Two types of effluent were collected from the collection sump over the course of the thawing period: nutrient rich effluent and purified or treated water effluent. The nutrient rich effluent was collected early in the spring when the frozen layers reached a temperature warmer than the freezing point depression of the concentrate, approximately -2 to -5 °C. The purified water effluent melted once the temperature of the frozen layers surpassed this temperature range. The transition between nutrient rich effluent (concentrate) and purified water effluent depends on the temperature and consistency of the frozen layers. Based on previous laboratory testing the nutrient rich effluent was dark in color and the electrical conductivity (EC) of the solution was significantly higher than the raw manure placed. The EC of the effluent decreases as the amount of nutrient rich solution remaining in the ice pack decreases, leaving cleaner ice.

Effluent production into the collection sump occurs in early spring. The valve to the collection sump was opened when overnight ambient temperatures exceeded -5 °C on a consistent basis. At this temperature the nutrient rich brine migrated through the frozen layers and into the collection system due to its freezing point depression. Once fluid began to flow into the collection sump the valve was left open to ensure

the liquid level did not rise into the frozen layers. The flow into the sump was monitored daily and the volume collected in the storage tank recorded.

2.2.3 Sampling Methodology

The sampling and analysis protocol was based on the requirement of obtaining an accurate calculation of the amount of nutrients extracted from the manure during the freeze separation process. Several pieces of information were required to obtain the mass balance, including the volume of manure placed, the volume of snow and rain deposited within the pit, the volume of effluent collected during thawing, and the chemical composition of both the raw manure and the effluent. Table 2.1 summarizes the analytical parameters that were obtained for both the raw manure and effluent, and the expected values based on previous laboratory testing using thin layered freezing of liquid hog manure (Willoughby et al., 2001).

Liquid samples were collected during layer placement and effluent collection. The manure placed within the freezing pit was sampled immediately prior to or during placement of each layer. One manure sample was taken for multiple layers If the same batch of manure was used. Once the ice layers began to thaw in the spring, effluent samples were collected on a daily basis and the associated volume of effluent collected that day was recorded. Both raw manure and effluent samples were collected in 500 mL polyethylene bottles and maintained in the dark below 0 °C during transport. During collection the headspace in the samples was minimized to limit the amount of ammonia volatilization from the liquid prior to analysis. The EC and pH of the samples were measured within 48 hours of sample collection. Samples were kept frozen until chemical analysis was performed.

2.2.4 Nutrient Mass Balance Determination

The efficiency of nutrient extraction was determined based on a mass balance of the amount of nutrients placed within the freezing pit and the cumulative amount of nutrients collected in the effluent. The mass balance was determined for nitrogen, phosphorous, potassium, and sulfur, because these are the constituents of importance for fertilizer applications. Masses of nutrients were calculated based on measured concentrations and associated volumes either placed or collected. It was

expected that the nutrients would be concentrated into 10 to 30 % of the original volume of manure placed.

2.3 Experimental Results

The following sections describe the results of the 2004 field test including the operation of the system during layer application and effluent collection, the amount of nutrient extraction that occurred due to freeze separation, and changes to the manure odour and solids consistency due to freezing and thawing.

2.3.1 System Operation

System operation began in the first week of January 2004, with the application of three thin layers of water to saturate and freeze off the uppermost portion of the sand to prevent manure migration into the collection system during initial application. Manure application proceeded based on meeting the temperature requirements for freezing a 7.5 cm layer over 24 hours. Initially three thin layers of manure ranging in depth from 2.5 to 3.8 cm were placed to level the ice surface prior to 7.5 cm layer placement. Acceptably cool temperatures allowed the placement of only three full depth layers (Layers 1, 2, and 3). In addition to Layers 1 through 3, several snowfall events required saturation of the accumulation to prevent the development of voids within the frozen layers during subsequent layer application. Table 2.2 summarizes the details of each layer application including total volume placed, temperature at time of placement, and the volumes used to saturate snowfall accumulations. During layer application no fresh manure was observed in the collection sump showing that the sealing layer implemented prior to the application of manure was adequate. A total of 20.98 m³ of manure was frozen within the pit over the winter. A detailed description of each manure application is located in Appendix C.

The thawing period and subsequent effluent collection began earlier than expected when the ambient air temperatures warmed up significantly above normal in the second week of February. As the frozen layers warmed up to temperatures greater than -2 °C the concentrated nutrient brine thawed and migrated through the ice layers to the collection system. The volume of effluent pumped from the pit was monitored and recorded daily. Figure 2.9 shows the initially high effluent EC

compared to raw manure, and the gradual decrease as the effluent changes from concentrated nutrient brine to treated water. The daily volumes placed, effluent pH and EC data are provided in Appendix C. The samples were also analyzed for various chemical constituents to determine the mass balance and effectiveness of nutrient removal. The analytical results are reported and discussed subsequently.

Ambient temperatures returned to acceptable limits for freezing once again during the last week of February. On February 25 400 L of manure was placed, however excessive melting along the geomembrane during the previous warm period allowed the manure placed to drain down between the edge of the frozen layers and the membrane. Thus the edge of the pit required sealing prior to any further manure placement. A snowfall event on March 1, 2004 resulted in the accumulation of approximately 30 cm of snow. In an effort to seal the edges of the frozen layers the snow was saturated with manure and allowed to freeze overnight.

The following morning 1067 L of manure was placed in the pit. Within 3 hours of application the entire manure volume had migrated through the frozen layers. The manure did not appear to be flowing down along the side of the membrane but rather through the frozen layers. The valve to the collection sump was kept closed overnight, in the event that the freshly applied manure might freeze, and was opened the following morning. Within a 24-hour period all but 200 L of the raw manure placed as Layer 4 was removed from the collection sump. Comparison of the EC of this fluid and the raw manure showed little difference, at 16 mS/cm and 17 mS/cm respectively. The effluent value is likely slightly smaller due to dilution effects when traveling through the frozen layers and collection system.

This terminated the manure application phase because a liquid level could not be maintained on the ice surface long enough to freeze the manure. The thawing period at the end of February had caused increased porosity of the frozen layers due to the drainage of nutrient rich brine out of the system. Prior to the application of Layer 4 approximately 25% of the total volume of previously placed manure had been removed from the system as effluent without a significant change in the thickness of the frozen layers. The resulting porous structure of the frozen layers allowed the raw manure to migrate easily down into the collection system. After

March 2, 2004 effluent collection continued as per the procedures previously outlined.

To verify the porous nature of the frozen layers, three 10 cm diameter cores were obtained on March 10, 2004. A Cold Regions Research and Engineering Laboratory (CRREL) core barrel was used to obtain cores from the center of the pit and the north and south ends. The location of the cores, the equipment used, and the porous nature of the ice layers are illustrated in Figures 2.10, 2.11, and 2.12. There was a significant change in the color of the manure, from dark brown-black to almost white, following drainage of the nutrient rich brine from the ice. Large vertical drainage pathways were also evident in the structure of the ice, created by vertical drainage of the nutrient rich brine through the ice.

Temperature data was collected hourly to provide insight into the thermal characteristics of the system during placement, freezing, and thawing. Figure 2.13 shows the temperature data obtained from the thermistors placed in each of the layers during pit operation. The ambient temperature at the ice surface and at the top of the pit is also compared in Figure 2.14 to determine if there was a significant difference between the two locations. Figure 2.14 starts at February 10, 2004 because the thermistor at the top of the pit malfunctioned several times prior to this date, when a new thermistor was installed. The resolution of the thermistors was ±1 °C and taking this into consideration there was no significant difference in temperature between the two locations both when manure was placed and when there was a snow cover on the ice surface. However, as the ambient temperature rose above zero the temperature at the ice surface was in some cases 10 degrees higher than at the top of the pit. This is due to the absorption of solar radiation by the black surface coloring of the frozen manure.

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2.3.2 System Mass Balance and Nutrient Extraction

Raw manure and effluent samples were submitted for analysis to the Limnology Laboratory and Applied Environmental Geochemistry Research Facility (AEGRF) at the University of Alberta. Analytical parameters included total nitrogen (TN), ammonium ion (HN_4^+), total dissolved nitrogen (TDN), nitrite/nitrate ($NO_2^{2^-}/NO_3^-$), potassium (K), sulphate ($SO_4^{2^-}$), dissolved organic carbon (DOC), and total phosphorous. The AEGRF also conducted a cation and anion analysis for comparative purposes. The methods and standards used for the analyses are summarized in Appendix D. The detailed analytical results are located in Appendix E.

A total of 20.98 m³ of manure was frozen in the pit. Approximately 6.8 m³ of rain and snowfall accumulation was calculated based on averaging meteorological data from the Edmonton International Airport and the Edmonton Municipal Airport weather stations. In total approximately 27.5 m³ of effluent was collected over the course of the test. The mass balance for the various analytes, including the mass placed, mass extracted, and percent recovery is summarized in Table 2.3. Of the total nutrients placed the percent recovery following freeze separation was 86.5, 29.4, 62.0, and 92.0% for total nitrogen, total phosphorous, sulphate, and potassium respectively. Of the nitrogen species considered, more than 85% of the nitrogen extracted was in the form of ammonium-nitrogen. The majority of nitrogen exists as ammonium nitrogen, which is plant available during the first year of application as fertilizer. Complete closure on the mass balance was not expected and the basis for the discrepancies will be discussed in Section 2.4.

Figure 2.15 compares the cumulative percentage of nutrients extracted with cumulative percentage of effluent collected. As expected, more than 70% of nitrogen, potassium, and sulfur were recovered in the first third of total effluent collected. The recovery of phosphorous was expected to be low because the majority of it remains with the solids in the freezing pit and not with the effluent collected. Three of the samples tested for ammonium and total nitrogen showed total nitrogen concentration to be less than the ammonium concentration (Appendix

E), which is theoretically not possible. However, these results are within acceptable analytical error of \pm 20%.

Electrical conductivity was measured as a means of approximating the time when the thaw fluid changed from nutrient rich brine to treated water. The change in EC with the cumulative percentage of effluent collected is illustrated in Figure 2.16. The electrical conductivity of raw manure ranged from 15 to 17 mS/cm. The initial EC of the effluent was nearly 40 mS/cm, more than double that of the raw manure. The EC was high when the nutrient rich brine was collected in the early spring. As the ambient temperatures rose and the purified ice began to melt the EC decreased continuously until it reached approximately 5 mS/cm in the melt water from the cleaner ice.

The effect of the ice temperature on the concentration of constituents in the effluent and the hierarchy of constituent melting was examined. Figures 2.17 and 2.18 illustrate the concentration of various nutrients and chemical constituents as a function of maximum daily ice temperature at the base of the ice layers. Fluorine, bromine and magnesium concentrations were all less than 25 mg/L and therefore were not shown on the charts. Sulphate was also very low at less than 50 mg/L for most samples but was included as it is a nutrient requirement for crops. The charts illustrate the effect of freezing point depression on the effluent constituents concentrations. At temperatures near -2 to -3 °C the effluent had very high concentrations as expected in the nutrient rich brine. As the ice temperature increased to near 0 °C the concentrations decreased dramatically due to dilution with melt water. There is also a lag effect, where the change in concentration comes after the ice temperature changes.

2.3.3 Observed Changes in Odour and Solids

Laboratory testing using thin-layered freezing to treat liquid swine manure showed two promising results: a change in the offensive odour of manure after freezing and thawing, and a change in the solids consistency and phosphorous content (Willoughby et al., 2001). Observations on the liquid's odour were made at the time of layer placement, during winter while the manure was frozen and during effluent

collection. During placement the odour was very strong and offensive however, once frozen there was no odour emitted from the pit. During the thawing phase of the experiment the pit also did not emanate any offensive odour. The effluent fluid had a significantly different odour from that of raw manure. It was no longer offensive, but slightly sweet and weaker in strength. Laboratory testing to verify the odour changing capacity of freeze separation treatment for liquid swine manure was beyond the scope of this research. However, its significance could prove very important for minimum distance separations at intensive livestock operations and for the technologies required for the field application of manure.

As previously reported the efficiency of phosphorous extraction using freezing separation was found to be less than 30%. This result was expected due to the adherence of phosphorous to the solids particles in the freezing pit. It is postulated that the phosphorous containing solids could be utilized as a compost additive. The manure used in this field test had a solids content of less than one percent due to problems with the SRTC manure handling system. Consequently there were insufficient solids remaining in the pit after the ice thawed to run tests on their compost additive qualities. The few solids that were remaining were very friable and dry. Future testing is required to explore the effect of freezing and thawing on the solids in manure because the solids content in most liquid swine manure is between 5 and 10%.

2.4 Discussion of Results

The purpose of this field test was to determine the viability of using freeze separation to treat liquid swine manure at a pilot scale. The objectives were three fold: prove out the design and operation of the thin-layered freezing system for field operation; determine the efficiency of nutrient extraction due to freeze separation; and evaluate the nutrient components of the effluent for use as crop fertilizer, irrigation or recycle water.

At the pilot scale the thin-layered freezing system operated very efficiently. Significant time was invested to ensure the system operated with as few man-hours as possible while still maintaining a large enough scale to apply to large-scale operations in the future. The collection and drainage system did not require the use of the installed heat tape due to the proper placement of an ice sealing layer prior to manure application. Future installations may not need to install heat tracing in the collection piping as long as a proper sealing layer is formed at the outset. The manure placement equipment worked well for the pilot scale but will require redevelopment for large-scale operations. Underground insulated piping that runs into the pit at several locations would be more appropriate than utilizing a moving dispenser or system on the ice surface. At very cold temperatures above ground manure placement systems may require significant man-hours to deal with freezing pipes and equipment.

Operationally, four key recommendations from the 2003 testing were reinforced. First, following snowfall events the snow accumulation should be saturated with manure and allowed to freeze prior to the placement of another full layer of manure to ensure no voids develop in the covered frozen manure. Secondly, it is imperative that the manure applied to the surface be at or near 0 °C to prevent thermal erosion of the surface during flooding. Thirdly, a specifically designed dispenser was required to reduce erosion of the ice during manure placement. And finally, isolation of the collection sump from the pit during manure placement is crucial to ensure a free draining system is maintained and that effluent does not rise from the drainage pipes into the previously frozen material.

To improve the system for large-scale operations snowfall accumulation and layer thickness must be addressed. In areas with large snowfall volumes a cover may be required to keep the surface free of accumulation. Snow cover insulates the frozen layers and minimizes melting during warm winter days, however the requirement of saturating the accumulation to prevent void inclusions in the frozen manure may incur more man-hours and decrease the total amount of frozen manure that can be formed over the winter. The increased man-hours should be considered against the cost of installing a cover over the freezing pit or removing the accumulated snow prior to placement of the next layer.

The layer thickness used in this test was between 6 and 7 cm based on previous laboratory testing, which allowed the layer to freeze completely in less than 24

hours. Layer thickness can be adjusted in the field based on ambient temperature. Colder days may allow the thickness to be increased another 3 to 4 cm whereas warmer days would require a decrease in layer thickness to ensure it freezes completely and does not erode the frozen layer below. The goal is to freeze as much manure as possible over the winter period and minimize the storage required in summer months.

The efficiency of nutrient removal in the field setting was less than that obtained in the laboratory. In the case of nitrogen, sulfur and potassium more than 70% of each constituent was concentrated in the first third of effluent collected. The key to operating a successful system at the field scale will be in determining the cut off between nutrient rich brine and purified water for irrigation or recycle purposes. Figure 2.16 shows that in this experiment the EC decreased to approximately raw manure values within the first 30% of effluent collected. Using daily measurements of effluent EC the operator can determine when the effluent reaches raw manure EC equivalencies. From that point the effluent should be stored in a separate location from the nutrient rich brine to maintain the increased economic value of concentrating the nutrients in the brine and to prevent recontamination of the purified water that may be reusable for other purposes around the farm.

Complete closure was not obtained on the mass balance for the various nutrient constituents. In the case of nitrogen, volatilization of ammonium from the system is believed to be a major cause of nitrogen loss. In an open system this loss cannot be controlled. Potassium loss was not significant as a closure of nearly 93% was obtained. It should be noted that the potassium and sulphate concentrations were very low and therefore any small analytical error translates into a large error in the recovery calculation. Phosphorous extraction was expected to be low due to its adherence to solids remaining in the freezing pit. Other constituents such as bromine and magnesium had very low concentrations, which may incur large error in the mass balance.

The EC of the effluent near the final stages of thaw was close to 5 mS/cm whereas it was expected to be less than 1 mS/cm. This may be a residual effect of the placement of Layer 4, which ran through the system without freezing. In Figures

2.16 and 2.17 an increase in the concentration of effluent in all species is seen between March 3 and March 9, 2004 and then a gradual return to decreasing levels. Layer 4 was placed on March 3, 2004 and by running through the system without freezing it may have contaminated the purified ice and increased the EC to higher than expected.

The main reason for undertaking this research was to add value to a large volume waste and to develop a more sustainable manure management system for large operations. Freeze separation maximizes the value of effluent by concentrating the nutrients into a smaller volume, which translates into decreased cost for transportation and application. The P:N ratio in raw manure is generally higher than plant requirements (Prairie Agricultural Machinery Institute 2001). As reported earlier the P:N ratio is greatly reduced through the use of freeze separation and can be of significant environmental importance in protecting watersheds from nutrient overloading.

In addition to the lowered P:N ratio, more than 85% of the nitrogen concentrated into the nutrient rich brine existed as ammonium-nitrogen, which is plant available in the first year of application. It is important to note that liquid swine manure is a highly variable fluid and the constituents and concentrations depend on many factors. Land application as fertilizer or irrigation water must involve testing both the soil and effluent in accordance with pertinent standards and regulations to accurately determine crop requirements and to ensure over application does not occur.

The difference between TN and inorganic nitrogen is the organic-nitrogen component also known as TKN or Total Kjehdhal Nitrogen. The Limnology Laboratory results were less than 0.05 mg/L for nitrite/nitrate with more than 85% of the nitrogen existing as ammonium nitrogen. The remaining 10 to 15% of nitrogen species existed as organic-nitrogen or TKN. The difference in results for nitrite between the Limnology Laboratory and AEGRF is due to the use of different analytical techniques and the age of samples. The AEGRF samples were analyzed more than 30 days after the Limnology analysis was complete, leaving time for the conversion of ammonium nitrogen to nitrite. The two data sets were not combined because of the extreme difference in results. It is common to see high levels of

nitrite and low levels nitrate in liquid swine manure due to the bacteriological environment of the liquid during storage (MMSC, 2002).

Gao (1998) and Elmore (1968) showed that the order in which constituents melt out of the ice is temperature dependent. The number of samples analyzed in this test was insufficient to determine a temperature dependency in the melting of various constituents. Several samples per day would be required to quantify this relationship.

The scope of this research did not allow for testing the quality of late spring thaw water for use as recycle water, irrigation water, or animal drinking water. The testing showed very high levels of dissolved organic carbon above the level that would qualify for use in swine facilities as drinking water. Due to the 'all in all out' nature of the SRTC operation high levels of chlorine were found in the effluent, which may hinder its use as irrigation or recycle water. Additional tests, including both chemical and bacteriological, are required to evaluate the quality of the purified effluent for use as farm recycle water, animal drinking water, or irrigation water. These preliminary results suggest that further treatment would be required for use as animal drinking water or irrigation water. However, in times of drought the purified effluent would prove a valuable resource and merits further investigation.

The liquid swine manure used in this field test contained less than 1% solids compared to the actual 5 to 10% solids in adequately mixed manure. One of the key advantages of the thin-layered freezing system is that all materials produced in the process are reusable. During a normal freeze separation operation a significant volume of solids would remain at the base of the freezing pit. Previous laboratory testing showed that the moisture content of the solids following freeze separation was decreased to less than 5%, the nature of the solids was highly friable, and the phosphorous content was maintained (Willoughby et al., 2001). Further testing is required to support these findings and to confirm whether the solids would provide a valuable compost additive after freeze separation treatment of swine manure.

2.4.1 Limitations of Test Method

At the pilot field scale the test method was very successful. Operational limitations included working in extreme cold conditions, dealing with pipes and equipment freezing, and adequately mixing manure to maintain the solids content. Future designs must utilize underground-insulated piping and heated structures for equipment to prevent freezing. Adequate agitators or mixers are required within manure storage tanks to prevent solids from settling prior to layer placement. A significant limitation for this system is its dependency on climate. Warm winters result in limited freezing time. This decreases the amount of manure that can be treated and increases the amount of raw manure storage required in the summer and fall. A very significant result of the freezing system is the change in odour of the manure once it has been frozen. Decreasing the amount of frozen manure during a warm winter results in increased storage of raw manure and the production of offensive odours. As with any large volume waste the shear size of the operation may be a limitation in terms of man-hour costs. This issue will be addressed in the following chapter, which deals with the development of a farm-scale application and the monetary value of concentrating the nutrient component of the manure.

2.5 Conclusions

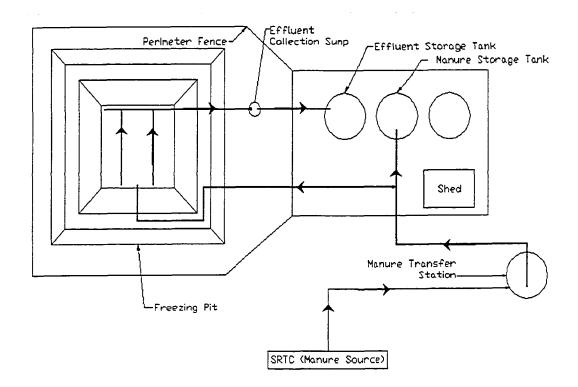
The high cost of storing and treating large volumes of liquid swine manure, coupled with increasingly stringent environmental regulations has driven research into finding a more economical and environmentally sustainable manure management system for intensive livestock operations. The purpose of this study was to develop and test a pilot scale freeze separation system to treat liquid swine manure. The objectives included proving out the field system and operation, determining the efficiency of nutrient extraction, and quantifying the value of the treated manure for use as a fertilizer substitute or as farm recycle water. The study has been successful in its objectives, from which several conclusions can be drawn:

Thin-layered freezing is a viable mechanism for extracting a large volume of valuable nutrients from liquid swine manure in a field setting. More than 70% of the nutrients were recovered and concentrated to one-third the original volume.

- The majority of nitrogen existed as ammonium-nitrogen, which is plant available in the first year of field application and therefore a valuable substitute for commercial fertilizer.
- A significant volume of relatively pure ice remained after collection of the nutrient rich brine, which may be valuable resource for use as recycle or irrigation water on farms.
- The freezing and thawing process removed the objectionable odour of liquid swine manure to non-offensive.
- The process added value to a large volume waste by decreasing the volume of nutrient containing liquid for transport and application and rendering the solids more easily handled for composting or disposal.
- Future thin-layered freezing system designs require the incorporation of buried and insulated piping to prevent freezing of manure transported within the pipes.

The major advantages of freeze separation include: low overhead costs due to the use of existing farm infrastructure, the use of ambient freezing and thawing conditions and gravity drainage separation, decreased odour, and the production of three usable products from one with limited application. Thin-layered freezing produces high nutrient value fertilizer, reusable water, and phosphorous containing solids that may be used as a compost additive. With the ever increasing number of intensive livestock operations in Alberta and through-out Canada, freeze separation is a cost-effective means of sustainable manure management that utilizes all components of the waste and may prove to be an invaluable asset to the industry.

2.6 Figures



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Figure 2.1 Plan view of thin-layered freezing system used for nutrient removal from liquid swine manure.

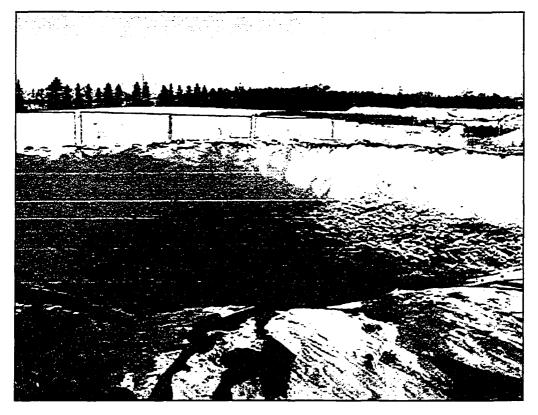


Figure 2.2 Freezing pit prior to manure placement in early December 2004.

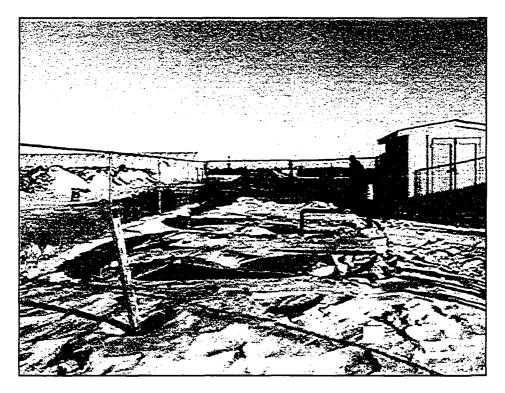


Figure 2.3 Manure and effluent storage tanks and equipment shed.

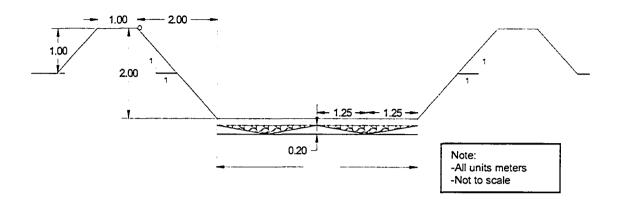


Figure 2.4 Cross-section of freezing pit showing sand and gravel collection system at pit base.

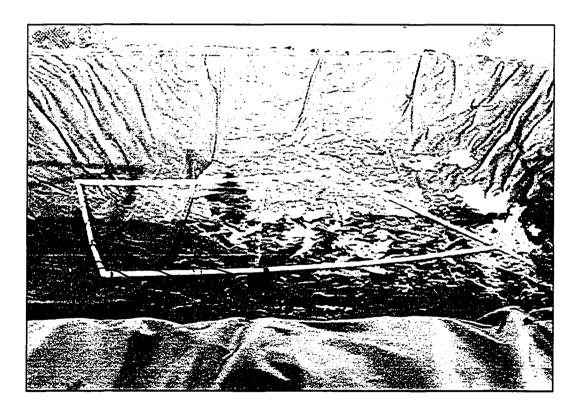


Figure 2.5 Collection system piping prior to placement of gravel and sand drainage layers.

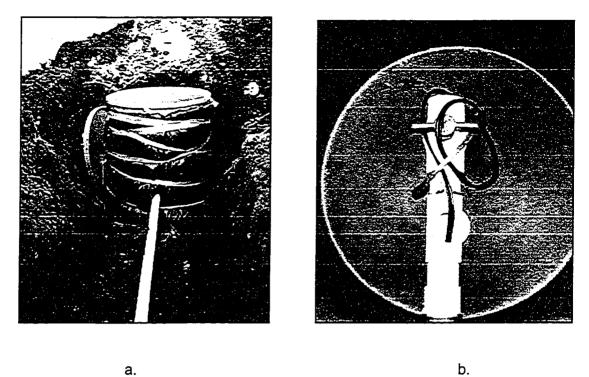




Figure 2.6 a. Collection sump and piping prior to backfill; b. Heat tape and collection sump shutoff valve



Figure 2.7 Impervious boot installation for effluent piping at geomembrane junction.

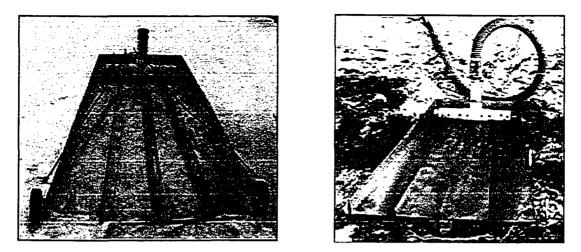


Figure 2.8 Layer placement equipment shown prior to and during manure placement in the pit.

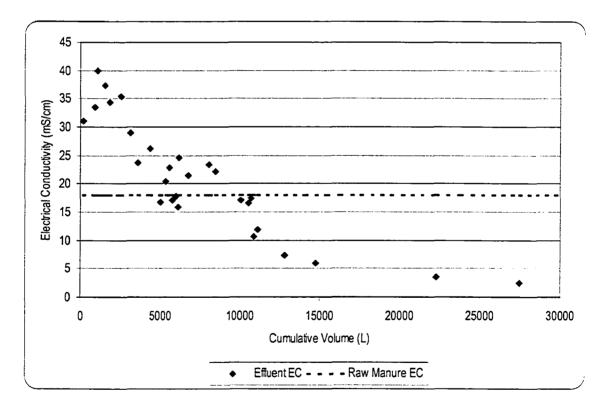


Figure 2.9 Effluent electrical conductivity versus cumulative effluent volume showing an increase in solute concentration due to freeze separation.

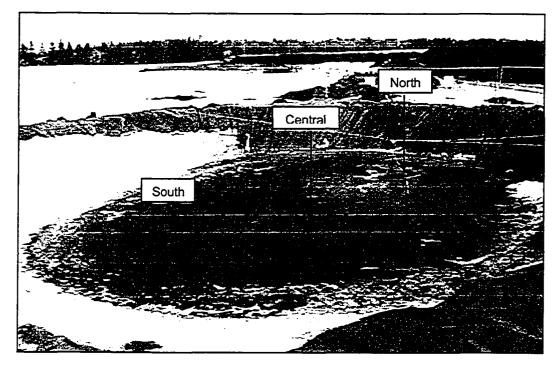


Figure 2.10 Freezing pit following completion of layer placement. The arrows indicate the location of ice cores.

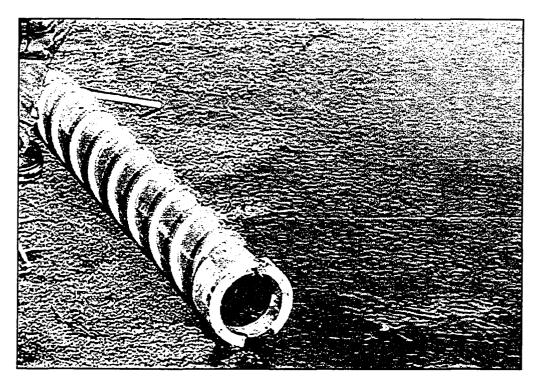
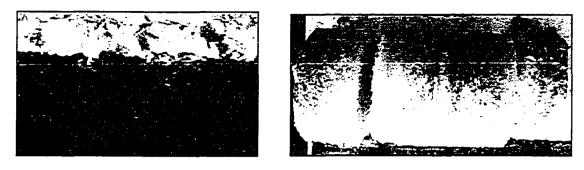
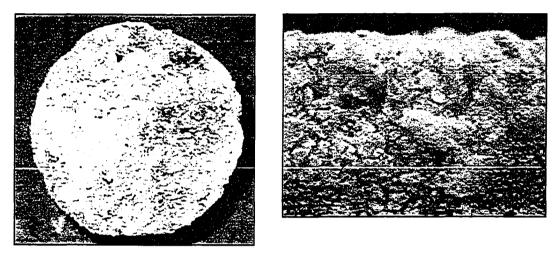


Figure 2.11 CRREL barrel used to obtain ice cores for examination of porous structure.



а.

b.



C.

d.

Figure 2.12 a. Raw liquid swine manure at time of placement in pit; b. Ice core taken after nutrient rich brine had drained from ice (ice surface is on the left side of photo); c. Porous nature of core showing vertical drainage paths created by nutrient rich brine; d. Close-up view of porous ice structure.

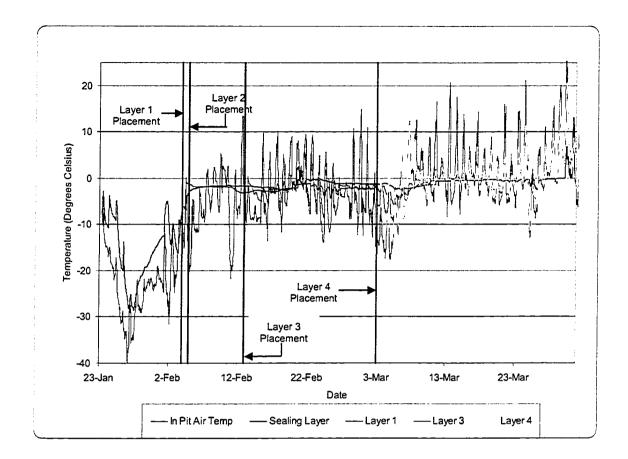


Figure 2.13 Temperature profile of frozen layers through test progression and date of layer placement..

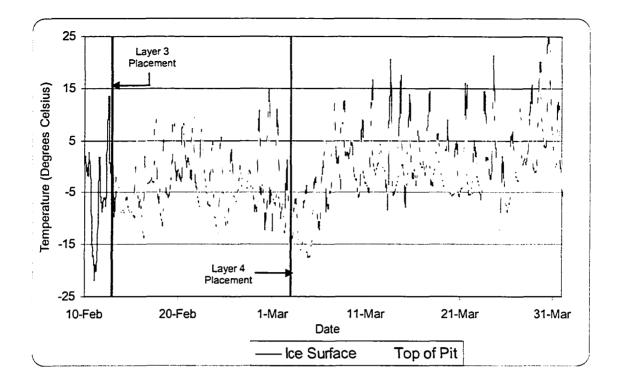


Figure 2.14 Comparison of ambient temperature at ice surface and top of pit.

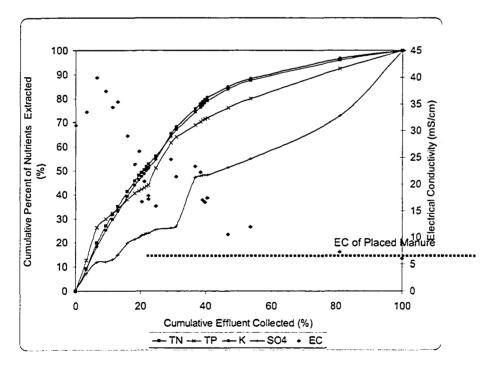


Figure 2.15 Cumulative percentage of nutrients extracted and the electrical conductivity of effluent versus the percentage of total effluent collected.

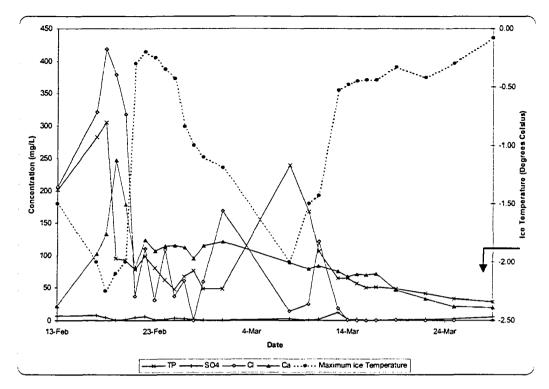


Figure 2.16 Effect of temperature on the concentration of selected effluent constituents for concentrations less than 450 mg/L.

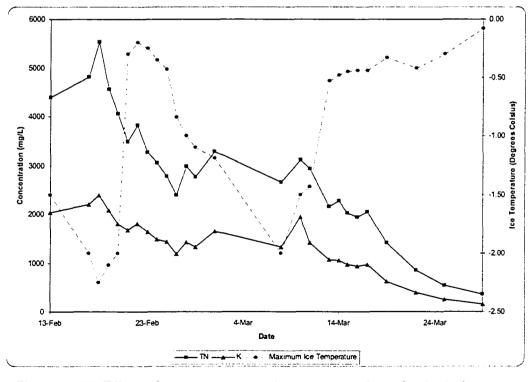


Figure 2.17 Effect of temperature on the concentration of selected effluent constituents for concentrations less than 5500 mg/L.

2.7 Tables

| | Expected Values* | | |
|--------------------------------|------------------|-------------------|--|
| Parameter | Raw Manure | Nutrient Effluent | |
| | (mg/L) | (mg/L) | |
| Total Nitrogen (TN) | 850 | 3000 to 5000 | |
| Sulphate (SO₄) | 10 to 20 | 100 to130 | |
| Potassium (K⁺) | 300 | 1000 to 1400 | |
| Total Phosphorous (TP) | 150 | 0 to 50 | |
| Dissolved Organic Carbon (DOC) | 300 to 500 | 3000 to 5000 | |

| Table 2.1 | Expected values of liquid swine manure analysis before and after thin- |
|-----------|--|
| | layered freezing treatment. |

*Based on previous laboratory testing (Willoughby et al., 2001)

| Table 2.2 | Volume of manure | placed and ambien | it temperature at time | of placement. |
|-----------|------------------|-------------------|------------------------|---------------|
|-----------|------------------|-------------------|------------------------|---------------|

| Layer Number | Volume Placed (m³) | Ambient Temperature (°C) |
|----------------------------|-----------------------|-----------------------------|
| 1 | 5.65 | -10 |
| 2 | 4.13 | -18 |
| 3 | 1.60 | -6 |
| Manure for Snow Saturation | 9.60 | -11 to -31 |

| Parameter | Total Mass Placed (kg) | Total Mass Extracted (kg) | % Recovery |
|---------------------------------------|---------------------------|------------------------------|------------|
| Based on Limno | logy Laboratory Analys | is | |
| TN as N | 55.0 | 47.6 | 86.5 |
| NH₄ ⁺ as N | 39.5 | 40.6 | 103 |
| TDN as N | 48.5 | 42.1 | 86.8 |
| NO ₂ +NO ₃ as N | 0.00 | 0.00 | 0.00 |
| TP as P | 6.90 | 2.03 | 29 |
| DOC | 58.7 | 63.2 | 108 |
| к | 24.7 | 22.7 | 92.0 |
| Based on Geoer | nvironmental Laboratory | Anlaysis | - |
| SO₄ as S | 0.48 | 0.30 | 62 |
| NO ₂ as N | 36.3 | 11 | 31 |
| NO ₃ as N | 0.00 | 0.01 | >100 |
| Br | 0.09 | 0.15 | 165 |
| CI | 1.7 | 1.5 | 82 |
| F ⁻ | 0.00 | 0.00 | 0.00 |
| PO ₄ | 7.6 | 1.3 | 17 |
| NH₄ as N | 62 | 40 | 64 |
| Ca ⁺⁺ | 2.2 | 1.6 | 75 |
| Mg ⁺⁺ | 0.11 | 0.24 | 211 |
| K⁺ | 26 | 23 | 87 |
| Na ⁺ | 8.3 | 8.3 | 100 |

Table 2.3 Summary of mass balance for thin-layered freezing system.

2.8 References

- Alberta Agriculture Food and Rural Development (AAFRD). 1997. Assessment of the potential for SnowfluentTM to treat liquid hog manure, Interim Report. Conservation and Development Branch.
- Andersland, O.B. and Ladanyi, B. 2004. *Frozen ground engineering*. Second Edition, Hoboken, New Jersey: John Wiley & Sons.
- Applied Science Laboratory, Inc. 1971. *Purification of mine water by freezing*. US EPA, Water Quality Offices, Water Pollution Control Research Series: 14010 DR7.
- Baker, R.A. 1970. Trace organic contaminant concentration by freezing IV: ionic effects. Water Research, 4: 559-573.
- Campbell, R.J. and Emmerman, D.K. 1972. *Freezing and recycling of plating rinse water*. Industrial Water Engineering, **June/July**: 38-39.
- Cassman, K.G., Steiner, R., and Johnston, L. 1995. Long-term experiments and productivity indexes to evaluate the sustainability of cropping systems. Chapter 11 in Agricultural Sustainability: Economic, Environmental and Statistical Considerations, V.Barnett, R. Payne, and R. Steiner, Editors. West Sussex, England: John Wiley and Sons.
- Chalmers, B. 1959. How water freezes. Scientific American, 200: 114-122.
- Electrical Power Research Institute (EPRI). 1987. Industrial Application of Freeze Concentration Technology. EPRI Report EM-5232, June 1987.
- Elmore, W.W. 1968. Water purification by natural freezing. M.Sc Thesis, Department of Chemical Engineering, University of Wyoming, Laramie, Wyoming.
- Fertuck, L. 1969. Water purification by freezing in reservoirs, Work done during the season 1968-1969. Saskatchewan Research Council Report E 69-12, November 1968.
- Gao, W. 1998. Partial freezing by spraying as a treatment alternative of selected industrial wastes. Ph.D. Thesis, Department of Civil and Environmental Engineering, University of Alberta, Edmonton, Alberta.
- Grulich, G. 1969. Improved methods of spent sulfite liquor disposal. Ph.D. Thesis, Dartmouth College, Dartmouth, Nova Scotia.

- Halde, R. 1980. Concentration of impurities by progressive freezing. Water Research, 14: 576-580.
- Heist, J.A. 1981. Freeze crystallization applications for wastewater recycle and reuse. In Water – 1980. American Instutute of Chemical Engineers Symposium Series, 200(77), Edited by G.F. Bennett: 259-272.
- Huber, D. and Palmateer, G. 1985. A joint experimental project between southwest region of the Ministry of the Environment and Group Delta in the storage and renovation of sewage effluent by conversion to snow. Ontario Ministry of the Environment, Southwest Region.
- Jean, D.S., Lee, D.J., and Wu, J.C.S. 1999. Separation of oil from oily sludge by freezing and thawing. Water Resources, **33**(7): 1756-1759.
- Kenny, R., Gorgal, R.G., Marlineau, D., and Prahacs, S. 1991. Freeze crystallization of TEMCELL's BOTMP effluent. In: 1991 Environment Conference Publication by Canadian Pulp and Paper Association, Montreal, Quebec, 141-145.
- Koelsch, R. 2001. Lesson 1: Principles of Environmental Stewardship. In Manure Management Curriculum 2002, Saskatoon, Saskatchewan.
- Konrad, J.M. and McCammon, A.W. 1990. Solute partitioning in freezing soils. Canadian Geotechnical Journal, Volume **27**(6): 726-736.
- Krepchin, I.P. 1985. Fresh water from the sea. High Technology, September: 64-65.
- Martel, C.J. 1989. Development and design of sludge freezing beds. American Society for Civil Engineering Journal of Environmental Engineering, **115**(4): 799-808.
- Martel, C.J. 1993. Fundamentals of sludge dewatering in freezing beds. Water Science Technology, **28**(1): 19-35.
- Martel, C.J. 1999. *Effect of dissolved solids on freeze-thaw conditioning*. In Proceedings of the Cold Regions Engineering 10th International Conference, Lincoln, New Hampshire 16 to 19 August 1999: 306-306. Edited by J.E. Zufelt.
- McGill, W.B. 1997. Sustainability of manure management: nutrient retention and cost-benefit analysis. Final Report CAESA Project RES-056-93.
- Miner, J.R., Humenik, F.J., and Overcash, M.R. 2000. Managing livestock wastes to preserve environmental quality. Iowa State University Press, Ames, Iowa.

- Muller, M. and Sekoulov, I. 1992. Wastewater reuse by freeze concentration with a falling film reactor. Water Science Technology, **26**(7-8): 1475-1482.
- Parker, P.J., Collins, A.G., and DeWolfe, J.R. 2000. *Freeze-thaw residuals conditioning*. Journal of American Water Works Association, 92(4): 168-181.

Pounder, E.R. 1965. *The physics of ice*. London: Pergamon Press.

- Prairie Agricultural Machinery Institute. 2001. The pork producer's guide to managing swine manure: a practical guide for farmers and applicators. Saskatchewan, Canada.
- Spyker, J.W. 1981. Desalination of brackish water by spray freezing progress report 1979-80. Saskatchewan Research Council, SRC Technical Report No. 126, SRC Publication No. E-835-1-B-81, June, 1981.
- Stahl, R.P. and Sego, D.C. 1995. Freeze-thaw dewatering and structural enhancement of fine coal tails. Journal of Cold Regions Engineering, 9(3): 135-151.
- Suthersan, S.S. 1997. Remediation Engineering Design Concepts. CRC Press Incorporated, New York.
- Terwilliger, J.P. and Dizio, S.F. 1970. Salt rejection phenomena in the freezing of saline solutions. Chemical Engineering Science, **25**: 1331-1349.
- Weeks, W.F. and Ackley, S.F. 1982. *The growth, structure, and properties of sea ice.* U.S. Army Cold Regions Research and Engineering Laboratory Monograph 82-1.
- Willoughby, A.R., Biggar, K., and Sego, D.C. 2001. Nutrient extraction from hog barn wash water using freeze separation. University of Alberta, Department of Civil and Environmental Engineering. Edmonton, Canada.
- Willoughby, A.R., Biggar, K., and Sego, D.C. 2003. Field Test 2003 Summary Report. University of Alberta, Department of Civil and Environmental Engineering. Edmonton, Canada.

3 Utilization of Freeze Separation to Recover Nutrients from Liquid Swine Manure: Farm Application

3.1 Introduction

Intensive livestock operations (ILOs) across Canada produce billions of liters of manure per year. Land application of these volumes may not be possible due to limited available land space, high costs to transport, and for the environmental impacts of nutrient loading. This can result in long-term storage of the waste in earthen manure storage. The high cost of storing and treating large volumes of liquid swine manure, coupled with increasingly stringent environmental regulations has focused research into finding more economical and environmentally sustainable manure management systems for intensive livestock operations. The impetus for this study was to evaluate an alternative means of treating liquid hog manure was to increase the economic value of the large volume manure product. Chemical, physical, and biological treatment options used to manage industrial and human wastewaters are available to treat liquid swine manure but they usually require large capital investments and operating budgets that are not economically feasible for producer's in the Canadian marketplace. The majority of ILOs operations in Alberta utilize anaerobic digestion in a dual earthen manure storage system to treat or store liquid hog manure. Field application of manure is conducted to alleviate increasing volumes in storage. The dilute nature of manure and the high cost of transport and field application make for an economically undesirable operation.

A pilot-scale test using thin-layered freezing to treat liquid swine manure was completed at the University of Alberta Swine Research and Technology Center (SRTC) during the winter and spring of 2004. The system was able to concentrate the nutrient component of liquid swine manure to one third of its original volume, change the odour characteristics of the manure and treated effluent, and produce a large quantity of treated water for irrigation or reuse purposes. At the pilot scale the system was shown to increase the economic value of manure and provide an environmentally sustainable solution to manure management. This research investigates the development of the thin-layered freezing system at the farm scale and the cost to install and operate the system at an average size swine farm in

Alberta. The research is focused on farms in Alberta but is applicable to any cold climate regions.

3.1.1 Research Objectives

The purpose of this research is to determine the cost to install and operate a thinlayered freezing system for the treatment of liquid swine manure on swine production operations in Alberta. The objectives of the research include:

- Design a thin-layered freezing system to treat liquid swine manure at intensive livestock operations.
- Estimate the capital and operational costs to construct and operate a thinlayered freezing system at a 3000 head swine facility;
- Calculate the cost per cubic meter to treat liquid swine manure using thinlayered freezing;

3.1.2 Current Treatment Technologies and Manure Management Systems

With modifications physical, chemical, and biological systems that are utilized for treating industrial and human wastewater can be used to treat liquid hog manure. The system that is utilized depends on the pertinent regulations and standards, the volume, composition and concentration of manure, soil type and crop selection, climate and location, and the cost to install and operate the system (Zering, 2000). Table 3.1 summarizes the physical, chemical, and biological systems that can be used to treat or process manure (CETAC-West, 1999; Day and Funk, 1998; Westerman and Zhang, 1995; Zhang and Westerman, 1995). A detailed description of each of these systems is beyond the scope of this research.

Table 3.1 outlines many technologies to treat swine manure, however the majority of farmers do not use a treatment system and rely on 9 to 12 months of manure storage with land application (Day and Funk, 1998, Fleming and Ford, 2002; Jones, 1999; Miner et al., 2000). Based on statistical data from 2001, more than 85% of the hog sector used liquid manure storage systems with a capacity of more than 250 days (Statistics Canada, 2003). Almost 10% of the farms had liquid storage capacity for greater than 400 days.

In Alberta, current practices are governed by the *Agricultural Operation Practices Act* (Province of Alberta, 2000), which regulates the development of new and expanding livestock operations. The design standards for manure storage in Alberta are summarized in Appendix F. In addition, beneficial management practices (BMPs) have been developed for manure storage to reduce environmental risks and to increase environmental benefits of agricultural operations (Statistics Canada, 2003). The BMPs include runoff prevention, ground and surface water protection, odour and air pollution minimization, supplying sufficient storage to prevent nutrient overapplication, and prevention of nutrient losses during storage. Manure management practices in Alberta and Canada are moving towards sustainable system paradigms that view manure as a resource and not a waste.

3.1.3 Freeze Separation to Treat Liquid Swine Manure

The thin-layered freezing system developed to treat liquid hog manure was based on the concept of freeze separation, which was discussed in detail in Chapter 2. The farm scale system was developed utilizing the same principles as the pilot scale system with minor modifications due to the increased size and scope of the operation. The system design and operational procedures will be discussed in Section 3.2 and 3.3. The major advantages of freeze separation include: low overhead costs due to the use of existing farm infrastructure, the use of natural processes, decreased odour, and the production of three usable products from one with limited application. Thin-layered freezing has produced high nutrient value fertilizer, reusable water, and phosphorous containing solids that may be used as a compost additive. The development of a thin-layered freezing system at the farm scale would provide a cost-effective means of sustainable manure management that utilizes all components of the waste.

3.2 System Design

The thin-layered freezing system was developed with a focus on utilizing existing farm infrastructure. With more than 85% of current swine farms utilizing manure storage a dual lagoon freezing system was chosen. Figure 3.1 illustrates the design of a typical liquid manure storage pit based on Alberta standards and regulations.

For a typical freeze-separation system, each operation would require evaluation to determine the best use of existing infrastructure and the most cost effective design. In most cases, farms operating a dual earthen manure storage system would require evaluation of the earthen manure storage lining, addition of an effluent collection system and manure placement equipment, and the purchase of adequately sized earthen storage pits for the nutrient rich brine and treated water. For ease of calculation and design the system developed for this research was based on new construction of all components for a 3000 head operation in central Alberta.

The system size depends on the volume of manure produced in one year at the facility, the requirements for freeboard, rainfall and storage capacity, and the volume of nutrients and treated water that can be used each year. Based on average statistical data for North American swine operations, the volume of manure produced over one year for a 3000 head operation was estimated to be approximately 6.13 x 10^3 m^3 (Miner et al., 2000; Statistics Canada, 2003). The following sections summarize the design of the freezing pit and raw manure storage, the effluent collection system, and manure placement equipment. Output from the spreadsheets used to design this system are located in G.

3.2.1 Freezing Pit and Raw Manure Storage

The system was designed to freeze 12 months of manure production in one winter. To accommodate this, two lagoons were required, one for freezing and the other for raw manure storage. The manure produced during winter operation (December, January, and February) would be frozen during that winter, leaving 9 months of raw manure production requiring storage. Cross sections of the freezing pit and raw manure storage are illustrated in Figures 3.2 and 3.3. The dimensions of the freezing pit were calculated based on a rectangular volume without considering the volume of manure placed along the side slopes. The extra volume allows for 9% volume expansion during freezing and snow and rainfall accumulation within the pit. The maximum depth to which manure could be frozen in the freezing pit for central Alberta conditions was determined to be 3 m based on normal freezing indices published by Boyd (1976).

In this design both the freezing pit and earthen manure storage were lined with a 30mil polyvinyl chloride (PVC) agricultural geomembrane to prevent seepage from the structures. A geotextile mat was placed beneath the PVC membrane to provide added protection from puncture for the membrane. Boot installations were specified around any piping entering or exiting the membrane to prevent seepage through these locations. To maximize the lifespan of the geomembranes and minimize degradation by solar radiation it is recommended that the embankments and side slopes be covered with 0.3 m of topsoil or a geotextile cover (Louey, 2004). Where competent clay is located, a compacted clay liner may be used instead of the geomembrane system. The difference in capital cost for these two options is discussed in Section 3.5.

To ensure proper mixing of the manure prior to placement in the freezing pit, agitators were placed in the raw earthen manure storage. The number and size of agitators depends on the size and depth of the earthen manure . To provide protection from the agitator equipment, a geotextile fabric should be placed over the PVC liner followed by 0.1 m of pea gravel and 0.1 m of washed concrete sand.

3.2.2 Manure Placement Equipment

The manure placement system was designed to place one layer of manure in the freezing pit within two hours. The freezing pit layout and piping configuration are illustrated in Figures 3.6 and 3.7. The Hazen-Williams equation for full flowing circular pressure conduits was used to size the piping. Layer placement was designed for single pump operation, however installation of a second backup pump is recommended as this is a critical piece of equipment for operation. The pumps are located within a heated shed between the freezing pit and raw manure storage.

Manure is pumped from the raw manure storage into a header system that distributes the liquid into six 75 mm diameter PVC pipes that enter the side of the freezing pit 0.4 m from the top of the embankment (Figure 3.7). The placement pipes were designed with a 2% grade towards the freezing pit to ensure gravity drainage of the pipes following manure placement. Removable light gauge galvanized steel troughs were designed to transfer the manure from the pipe outlet

to the ice surface. The troughs provide a means of removing sensible heat from the manure and allowing laminar flow of the liquid onto the ice surface. The detailed procedures for layer placement and equipment operation are discussed in Section 3.3.

3.2.3 Effluent Collection System

The effluent collection system was designed for full flowing pipes under atmospheric pressure. Figure 3.4 shows a plan view of the collection system piping and storage tanks, and Figure 3.5 shows a cross section of the collection system at the base of the freezing pit. To size the collection piping, the maximum and minimum thaw rates were determined based on experience with the 2004 pilot test. Table 3.2 summarizes the estimated melt times and the calculated thaw rates. The maximum nutrient thaw rate of 3 x 10^{-3} m³/s governed design of both the slotted collection pipes and the header pipes. A total of 10 single slotted 50 mm (inside diameter, ID) PVC pipes placed at a 1% grade were selected to collect the thawing fluid and convey it to 2 header pipes at opposite ends of the pit. The header pipe was 75 mm ID solid PVC piping.

The drainage layer was included over the collection piping for three purposes: to protect the drainage pipes from damage, to act as a filter and prevent the buildup of fines and particles in the collection pipes, and to allow the formation of an ice sealing layer prior to manure placement. The drainage layer consists of a 0.3 m layer of 9 mm rounded pea gravel covered by a 0.2 m layer of washed concrete sand.

From the header pipes thawed fluid flows by gravity to the collection sump located outside of the freezing pit. The collection sump was designed as a covered tank with the base 5 m below the ground surface. To maintain a free draining system and prevent liquid from rising back into the freezing pit, a level actuated pump was located at the bottom of the tank to transfer the thawed fluid to nutrient brine storage or to treated water storage. The maximum fluid depth in the sump is 1 m. Once this depth is reached the level actuator starts the pump and the fluid volume is removed. To minimize the occurrence of pipe freezing, all pipes outside of the freezing pit were

buried and insulated. The collection sump was equipped with a ladder for accessing the pump.

Two pits are required to store the nutrient rich brine and the treated water. The storage volumes required were calculated based on the estimate that 30% of the treated manure would be thawed as nutrient rich brine in the early spring and 60% would be thawed as treated water in late spring and early summer. To allow for snow and rain accumulation in the pit the estimated volumes were increased by 10%. The storage volumes required for nutrient rich brine and treated water were 2,000 and 4,000 m³, respectively. Both storage pits require installation of an adequate liner system and the nutrient rich brine storage requires a cover to prevent the loss of ammonia. The operational procedures pertaining to thaw fluid transfer are discussed in Section 3.3.

3.3 System Operation

The thin-layered freezing system was designed to freeze manure during the months of December, January, and February. The number of freezing days will increase or decrease depending whether the winter is cold or warm. Figure 3.8 illustrates the layout of the thin-layered freezing system as described in Section 3.2. Prior to the placement of a full layer of manure, a thin layer of water must be frozen to seal off the collection system at the base of the freezing pit from the freshly applied manure. The thin ice layer prevents the migration of manure through the drainage layers into the collection system during initial manure placement and freezing. Using a series of removable sprinklers, water should be sprayed onto the sand in sufficient applications to provide a 1 to 2 cm layer of ice over the entire base of the freezing pit. Ponding of water on the ice surface and a lack of water in the collection sump are sufficient proof that an adequate sealing layer has been formed. The applications are intended to saturate the top 5 cm of sand and must be placed when the ambient air temperature is less than -5 °C to allow the saturated sand to freeze. The collection sump isolation valve should remain in the open position to allow excess water to drain from the system during formation of the sealing layer.

Once the ice layer is established manure placement can proceed as outlined in Appendix H. To place a 7.5 cm layer of manure two conditions must be met: the minimum daily temperature must be less than -10 °C and the maximum daily temperature should be no greater than 0 °C. If both of these temperatures are forecasted, manure application may proceed. In addition, the manure being placed must be colder than 2 °C to minimize the amount of sensible heat removal and the time to freeze, and to prevent thermal erosion. Layer placement continues over the winter months until air temperatures warm above the outlined temperature conditions, or the maximum ice depth of 3 m has been reached. The collection sump isolation valve must be closed during manure placement to prevent manure from running into the collection sump before it is frozen.

Effluent collection will begin in early spring when the average daily ambient air temperature is greater than approximately -2 °C, which is the approximate freezing point depression of the nutrient rich brine. The isolation valve to the collection sump should be opened a couple of weeks prior to this time to ensure there is no fluid buildup in the freezing pit and that the system is free draining. Effluent should be pumped to the nutrient rich brine storage until the electrical conductivity (EC) decreases below the raw manure EC measured at the time of layer placement. The EC of the effluent should be measured at least twice a week to determine the change from nutrient rich brine to treated water. The range of expected EC values for raw manure, nutrient rich brine, and treated water are 10 to 20 mS/cm, 20 to 40 mS/cm, and 0 to 15 mS/cm, respectively.

By midsummer the entire frozen mass should be melted, leaving a volume of dried solids at the base of the freezing pit. The isolation valve to the collection sump must be kept open to allow any rainfall accumulation to flow through the freezing pit and collection system freely. Rainfall accumulation should be directed to the treated water storage tank. The volume of solids remaining depends on the initial solids content, which ranges from 5 to 10% for liquid manure. The solids can be scraped from the surface of the sand using a small tractor with a bucket attachment. Care must be taken to ensure the integrity of the geomembrane and its soil cover along

the freezing pit embankments. Replacement sand may need to be added following solids removal to maintain a level surface at the pit base.

Operation during the remainder of the summer and fall involves monitoring the raw manure storage and utilizing the nutrient rich brine, treated water, and treated solids. The use of any product for soil nutrient amendment requires testing of the product and the soil to ensure crop nutrient requirements are not exceeded. The procedures for determining nutrient requirements are beyond the scope of this research and will not be discussed.

3.4 Expected Results

The ability of the thin-layered freezing system to extract and concentrate the nutrients in liquid hog manure was demonstrated at the laboratory scale and in the field at a pilot scale. Similar results are expected for a full scale farm application, including: concentration of the nutrient component to approximately 30% of the original volume of manure frozen, production of treated water for use as irrigation or recycle water, and the production of treated solids with a high phosphorous content and a dry and friable consistency.

The results obtained by using thin-layered freezing are dependent on many factors including the size and type of operation, the number of actual freezing days, the amount of snowfall accumulation over the winter, and the concentration of nutrients in the raw manure used. The expected results summarized in this section are based on experiences with the 2004 pilot scale system at the SRTC and laboratory testing completed by Willoughby et al. (2001). Table 3.3 summarizes the expected results for treating liquid swine manure at a 3000 head operation in central Alberta. The nutrient calculations are detailed in Appendix I.

The maximum capacity of the system would result in the placement of approximately 37 layers of manure. Approximately 1840 m³ of nutrient rich brine would be produced containing 3900 kg of nitrogen, 16 kg of phosphorous, 1380 kg of sulfur, and 690 kg of potassium. The system would also produce 3680 m³ of treated water containing significantly less nutrient mass. As suggested in previous work the nutrient rich brine would provide a valuable and economical substitute for

commercial fertilizer due to its high nutrient concentration and significantly decreased volume, and the treated water could be used as irrigation water or recycle water on the farm.

3.5 Annual Cost to Install and Operate

The thin-layered freezing system was designed to treat liquid swine manure at intensive livestock operations that utilize manure storage. The cost associated with constructing and operating this system depends on the existing infrastructure and the ability of the farmer to utilize all components of the treated manure. The cost of treatment would decrease in relation to the quantity of existing infrastructure on a specific farm operation. The costs associated with constructing and operating the thin-layered freezing system were calculated for two different scenarios: constructing and operating a new system, and retrofitting an existing dual earthen manure storage system. The construction costs for a new system and for retrofitting an existing dual earthen manure storage system are summarized in Tables 3.4 and 3.5, respectively. Costs were obtained from suppliers and professionals in the Edmonton area and may be subject to change depending on season, availability, and location.

Capital investments for existing storage operations include, but are not limited to: earthwork for freezing pit and raw manure storage, lining the freezing pit and raw manure storage, the purchase and installation of effluent collection components, and the purchase and installation of transfer pumps, piping, and storage tanks. The recurring expenses such as electricity and utilities, repairs and maintenance, and costs associated with professional oversight were not included. The capital costs associated with constructing the system developed for this research totaled approximately \$235,000 or \$38/m³ of manure treated. The largest capital cost incurred in the system design is the installation of a geomembrane system to prevent seepage of nutrients and contaminants from the earthen storage pits. Where competent clay is located an engineered compacted clay liner may be installed to reduce capital costs to \$24/m³. If an existing farm operation utilizing a dual earthen manure storage system incorporated this treatment system the initial capital cost would be approximately \$152,000 or \$25/m³, which is significantly less than constructing the entire system. The United States Department of Agriculture Natural Resources Conservation Service (2004) published data on the cost of installing a typical liquid manure storage and management system for an operation with approximately 2075 animal units. The estimated installation costs in Canadian dollars was \$510 000, or \$245 per animal unit. In comparison, the thin-layered freezing system installation costs were less than half that at \$235,000 or \$78 per animal unit. These values are the initial net costs and have not been amortized over any period.

Thin-layered freezing utilizes natural freezing and thawing processes, therefore operational costs are limited to pump utility costs and man-hours for operation and maintenance. The annual operating cost, as summarized in Table 3.6, was calculated based on operational man-hours and does not include utility costs. The utility costs associated with operating the pumps and agitators and the operational cost of land application of the nutrient rich brine or treated water were not included in this cost because they are costs normally associated with existing operations. The annual operating cost for treating manure using thin-layered freezing was found to be \$2700 or \$0.45/m³.

The economic value of the nutrient rich brine is based on the replacement value of nitrogen and phosphorous in commercial fertilizers. Previous work by McGill (1997) estimated the replacement value of nitrogen and phosphorous in manure exceeded \$160 million dollars annually. The estimated value of nitrogen and phosphorous per ton was \$640 and \$1450, respectively. Utilizing these values the estimated worth of nitrogen and phosphorous in the nutrient rich brine was \$2500 and \$100, respectively. Based on these nutrient values the geometric rate of return for investing in thin-layered freezing was found to be 2.3% over a 5-year period. The rate of return calculation is located in Appendix J.

In addition to the added value of concentrating the nutrient component, decreased transport costs allow for further haul distances and the possibility of regional distribution of the fertilizer substitute. The economic value of providing a reusable source of water for farm operation or irrigation was not substantiated in this study, but the benefits of increasing water supply during times of drought cannot be overlooked.

3.6 Discussion and Limitations of Application for Albertan Farmers

It has been shown that thin-layered freezing systems have the ability to increase the value of a large volume of waste. There are several points that must be considered if the system is utilized at the farm scale. The trend of increasingly warm winters in the prairies means a significant decrease in the volume of liquid manure that can be treated over the winter and an increase in required storage. The high variability in the average number of freezing days will have an impact on estimating the return that farmers can get from investing capital into the treatment system and may be viewed as a major uncertainty of the system.

Farmers that utilize dual earthen manure storage already may not want to invest in a treatment system that relies on cold winters. In areas where there are a number of farms in close proximity capital costs could be significantly reduced if close proximity farms pool resources to utilize the same system. The full benefits of treatment and low operating costs would be a benefit to all farms involved. There is also the possibility of commercial opportunities for management companies to set up and run the treatment system for multiple farms at a specified cost per cubic meter of manure treated. This would leave the farmers with an annual treatment cost and it would transfer the responsibility of the capital costs to the management company.

One of the most promising results of freezing and thawing manure was its ability to significantly change the odour characteristics of manure. However, the system requires up to 9 months of raw manure storage in an earthen pit. The benefits of odour change for the treated manure may be offset locally by the resulting odours from summer storage of raw manure. However, the large reduction in odour may improve opportunities for placement at locations more distant from the farm. It is suggested that covers may be utilized during the spring and summer on the raw manure storage to minimize odour production, and also on the nutrient rich brine storage pit to minimize nutrient loss to the atmosphere through volatilization.

Several authors have discussed the effect of precipitation on the freeze separation process. During winter operation snowfall may occur during layer placement and freezing. Snow cover on the surface of ice decreases the rate of heat loss to the air

and inhibits the rate of ice formation (Ashton, 1980). Fertuck (1968) discussed how snow cover slowed the rate of ice formation resulting in a smaller quantity of ice with a lower effluent brine concentration. Snow cover would decrease the concentration of separated nutrients and it would decrease the amount of treated water produced during thin-layered freezing of manure. To minimize the effects of snowfall during freezing the entire freezing pit would have to be covered. Significant capital costs are required to build a structure to cover the freezing pit. Instead, the system proposed was developed with operational procedures to minimize the effects of snowfall. Saturating and melting snow from each snowfall event with manure and allowing it to freeze did not significantly impact the effectiveness of freeze separation at the pilot scale. Farmers looking to invest in thin-layered freezing must examine the capital costs of placing a cover over the area compared to the reduced effectiveness of freezing produced by snow cover and drifting and the man-hours required to deal with each snowfall event over the winter.

One of the key advantages of utilizing thin-layered freezing is the production of three usable products from one large volume waste. Nutrient rich brine can be used to replace commercial fertilizer applications, the treated water may be used as recycle or irrigation water or as swine drinking water, and the solids may be used as a compost additive. The very nature of sustainable manure management programs sees the utilization of all components of the waste. In that respect the freeze separation process forms a valuable component of a sustainable manure management program. The economics of this system require a significant capital investment at the outset but dollars are not the only consideration in the value of a process. The value of replacing commercial fertilizers with farm produced manure nutrients may outweigh the downside of an initially large capital investment.

3.7 Conclusions

The movement towards sustainable manure management has driven research into developing treatment systems that add value to manure, changing it from a large volume waste to a large volume resource. The purpose of this study was to determine the cost to install and operate a thin-layered freezing system for the treatment of liquid swine manure at swine production operations in Alberta. The

objectives included designing a thin-layered freezing system to treat liquid swine manure at intensive livestock operations, estimating the capital and operational costs to construct and operate the system, and determining the cost per cubic meter to treat liquid swine manure. A thin-layered freezing system was designed for a 3000 head swine operation in central Alberta, including both the technical components and operational procedures. The capital cost for constructing a new system was \$38/m³ of manure treated compared to \$25/m³ treated for retrofitting an existing dual earthen manure storage system. The annual operating costs were found to be \$0.45/m³ of treated manure and the geometric rate of return for investing in thin-layered freezing was found to be 2.5% over a 5-year period. The system requires a significant capital investment in the first year of operation, but this must be balanced with the regional and environmental benefits of adding value to a large volume waste in a sustainable management paradigm.

3.8 Figures

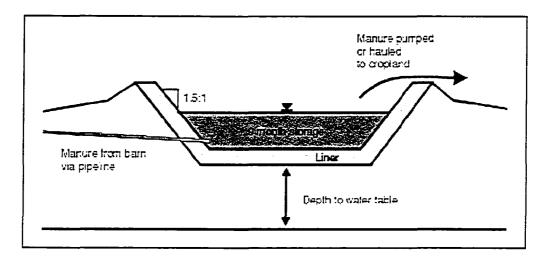


Figure 3.1 Typical design of liquid manure storage in Alberta (Permission obtained from: AAFRD, 2000).

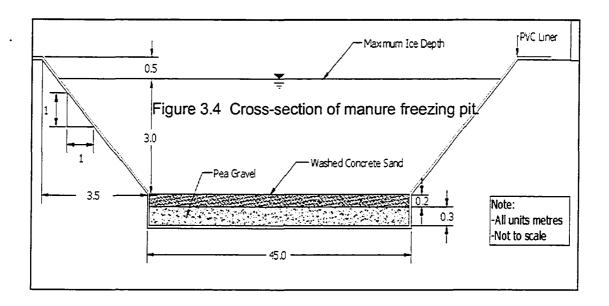


Figure 3.2 Cross-section of manure freezing pit. Pit base is 45 m by 45 m and was designed to freeze 12 months of manure in one winter.

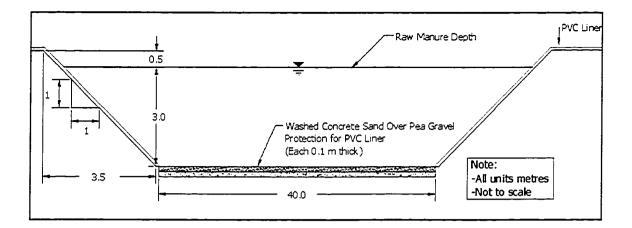


Figure 3.3 Cross-section of raw manure storage. The base is 40 m by 40 m and was designed to contain 9 months of produced raw manure.

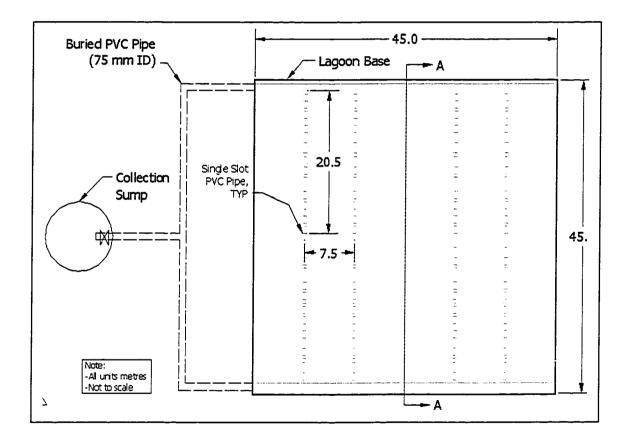


Figure 3.4 Plan view of effluent collection system showing slotted collection pipes, header pipes, and collection sump.

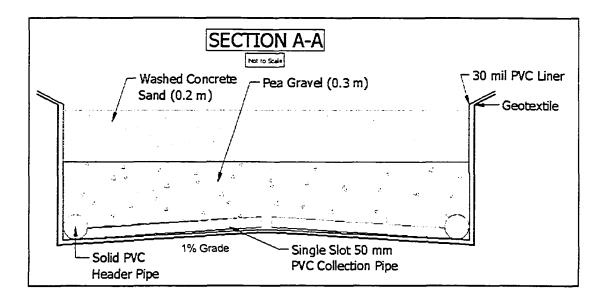


Figure 3.5 Cross section schematic of effluent collection system.

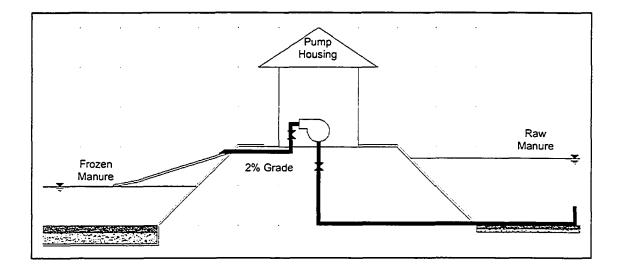
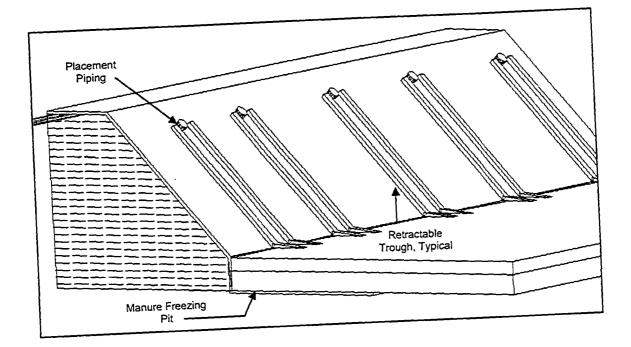
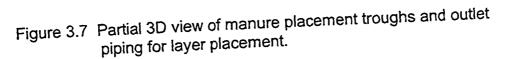
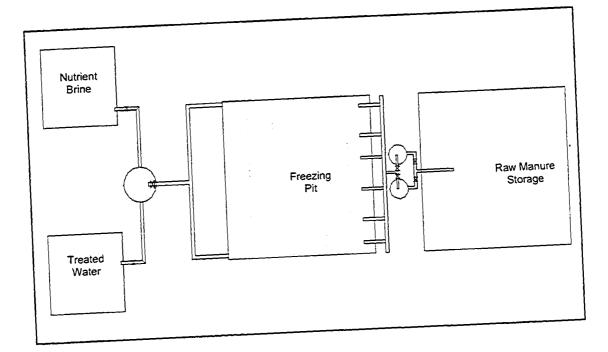
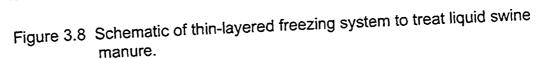


Figure 3.6 Cross-section of manure placement equipment, raw manure storage, and manure freezing pit. Raw manure agitator not shown as it is a removable unit located on the right side of the raw manure storage.









3.9 Tables

| Table 3.1 | Summary of physical, chemical, and biological treatment for manure (Day |
|-----------|---|
| | and Funk, 1998). |

| Technology | Available Systems | Treatment Products |
|----------------------------------|---|---------------------------------|
| Physical Treatment | | |
| Solid-Liquid Separation | Sedimentation, screening, centrifuging | Slurry, liquid, solids |
| Drying | Solar, mechanical dryers | Solids |
| Incineration | Fluidized bed, rotary kiln | Ash |
| Constructed Wetlands | | Treated water, solids |
| Biological Treatment | | |
| Anaerobic Treatment | Lagoons, digestors, septic tanks | Biogas, sludge, treated water |
| Aerobic Treatment | Oxidation ponds, aerated lagoons, oxidation ditches | Nitrate nitrogen, treated water |
| Composting | Conventional, thermophilic aerobic, vermicomposting | Compost |
| Chemical Treatment | | |
| Odour Control | Various additives | Odour reduction or change |
| pH Control | Various additives | • |
| Enhanced Biological Treatment | Various additives | - |
| Solid/Colloidal Precipitation | Various additives | - |

Table 3. 2 Summary of parameters used to calculate flow rates of frozen manure components.

| Liquid Component | Time to Thaw (weeks) | Calculated Thaw Rate (m ³ /s) |
|------------------|-------------------------|---|
| Nutrient Brine | 1 | 3 x 10 ⁻³ |
| Nutrient Brine | 3 | 1 x 10 ⁻³ |
| Treated Water | 4 | 1 x 10 ⁻³ |
| Treated Water | 12 | 5 x 10 ⁻⁴ |

Table 3.3 Summary of expected results for nutrient extraction using thin-layeredfreezing at a 3000 head swine operation in Central Alberta.

| Component | Volume | | Nutrient | Mass (kg) | |
|----------------|--------|------------|----------|------------|------------|
| | (m³) | N | Р | ĸ | S |
| Raw Manure | 6130 | 5200 | 92 | 920 | 1840 |
| Nutrient Brine | 1840 | 3900 | 16 | 690 | 1380 |
| Treated Water | 3680 | 1300 | 12 | 230 | 460 |
| Solids | 613 | Negligible | 64 | Negligible | Negligible |

.

| Freezing Pit Component | Details | Quantity | Units | Unit Cost | Total Cost |
|--------------------------------------|-----------------------------------|----------|----------------|---------------------|------------|
| Earthwork | Excavation and bank forms | 140 | hours | 150 | 21000 |
| Geomembrane (Ag Liner) | 30 mil PVC Ag Liner | 3600 | m ² | 6 | 21600 |
| Liner Installation | Boots and Liner placement | 2 | days | 2000 | 4000 |
| Geotextile | 8 ounce/vard GT | 3600 | m ² | 1.2 | 4320 |
| Boots | For placement and effluent piping | 3000 | | 65 | 520 |
| <u> </u> | For pracement and embern piping | <u> </u> | | Subtotal = | |
| Raw Manure Storage | | | | <u>- 300101ai -</u> | 51440 |
| Component | Details | Quantity | · · · · · | Unit Cost | Total Cost |
| Earthwork | Excavation and bank forms | 140 | hours | 150 | 21000 |
| Geomembrane (Aq Liner) | 30 mil PVC Ag Liner | 3025 | m² | 6 | 18150 |
| Geotextile | 8 ounce/yard GT | 3025 | m² | 1.2 | 3630 |
| Boot | Raw manure transfer pipe | 1 | - | 65 | 65 |
| Liner Installation | Boot and liner placement | 2 | days | 2000 | 4000 |
| Washed Concrete Sand | Protection from agitator | 160 | m ³ | 16.8 | 2693 |
| Round Pea Gravel | Protection from agitator | 160 | m⁴ | 40.6 | 6492 |
| Sand Installation | Placement and levelling | 10 | hours | 150 | 1500 |
| Gravel Installation | Placement and levelling | 10 | hours | 150 | 1500 |
| Agitator | PTO Propeller Driven Agitator | 2 | - | 5000 | 10000 |
| 6" PVC Pipe | Raw manure transfer pipe | 10 | m | 8.01 | 80 |
| Pipe Installation | | 8 | hours | 20 | 160 |
| | | | | Subtotal = | 69270 |
| Effluent Collection System | | | | | |
| Component | Details | Quantity | | Unit Cost | Total Cost |
| 3" Single Slotted Piping PVC | | 220 | m | 9.74 | 2144 |
| 3" PVC Piping | | 110 | m | 8.01 | 881 |
| 3" PVC Ball Valve | | 3 | - | 217 | 651 |
| 3" PVC Swing Check Valve | | 1 | - | 36 | 36 |
| Pipe and Valve Installation | | 20 | hours | 20 | 400 |
| Collection Sump Tank | Culvert tank, ladder installation | 1 | | 10000 | 10000 |
| Earthwork | Collection sump installation | 10 | hours | 150 | 1500 |
| Pipe Insulation | | 20 | | 2 | 40 |
| Heat Tape | | _20 | m | 0.25 | 5 |
| Nutrient Storage Lagoon ¹ | Earthwork, liner, 20x20 m base | 1 | | 25396 | 25396 |
| Treated Water Lagoon ¹ | Earthwork, liner, 30x30 m base | 1 | - | 30235 | 30235 |
| Level Actuator | Red Lion Centrifugal Pump | 11 | - | 50 | 50 |
| Sump Pump | Purchase and installation | 11 | | 400 | 400 |
| Washed Concrete Sand | Drainage Layer | 405 | m` | 16.8 | 6818 |
| Rounded Pea Gravel | Drainage Layer | 608 | m ⁴ | 40.6 | 24648 |
| Sand Installation | Placement and levelling | 10 | hours | 150 | 1500 |
| Gravel Installation | Placement and levelling | 10 | hours | 150 | 1500 |
| | | | | Subtotal = | 106204 |

Table 3.4 Construction costs for new thin-layered freezing treatment system.

¹ Estimate based on cost for freezing pit construction

Table 3.4 continued.

| Manure Placement Equipm | ent | | | | |
|-----------------------------|---------------------------------|----------|---------|-----------------------------|------------|
| Component | Details | Quantity | | Unit Cost | Total Cost |
| 6" PVC Pipe | | 10 | m | 20.5 | 205 |
| 4" PVC Pipe | | 45 | m | 11.5 | 515 |
| 3" PVC Pipe | | 20 | m | 8.01 | 160 |
| 6" PVC Gate Valve | | 4 | - | 250 | 1000 |
| Pipe and Valve Installation | | 20 | hours | 20 | 400 |
| Centrifugal Pump | Flow rate = 1400 L/min | 2 | - | 1500 | 3000 |
| Pump Installation | | 8 | hours | 20 | 160 |
| Shed | 3 x 3 m shed | 1 | - | 400 | 400 |
| Shed Heater | | 1 1 | - | 50 | 50 |
| EC Meter | Meter and calibration solutions | 1 1 | - | 1000 | 1000 |
| Pipe Insulation | Per linear meter | 75 | m | 2 | 150 |
| Heat Tape | Per linear meter | 75 | m | 0.25 | 18.75 |
| Retractable Troughs | 18 gauge galvanized steel | 6 | - | 200 | 1200 |
| | | | | Subtotal = | 8259 |
| | | | Cons | struction Cost = | 235173 |
| | | | Constru | ction Cost/m ³ = | : 38 |

| Table 3.5 | Construction costs for developing thin-layered freezing system a | at |
|-----------|--|----|
| | an existing earthen manure storage operation. | |

| Freezing Pit | | | | | |
|-----------------------------|-------------------------------|-------------|---------|-----------------------------|------------|
| Component | Details | Quantity | Units | Unit Cost | Total Cos |
| Earthwork | Excavation and bank forms | 50 | hours | 150 | 7500 |
| Boot | Placement and effluent piping | 8 | - | <u>6</u> 5 | 520 |
| Boot Installation | | 16 | hours | 50 | 800 |
| Piping Installation | | 20 | hours | 20 | 400 |
| | | | | Subtotal = | 9220 |
| Raw Manure Storage | | · · · · · · | | | |
| Component | Details | Quantity | | Unit Cost | Total Cost |
| Earthwork | Excavation and bank forms | 50 | hours | 150 | 7500 |
| Boot | Raw manure transfer pipe | 1 | | 65 | 65 |
| Boot Installation | | 2 | hours | 50 | 100 |
| Washed Concrete Sand | Protection from agitator | 160 | m³ | 16.8 | 2693 |
| Round Pea Gravel | Protection from agitator | 160 | m⁴ | 40.6 | 6492 |
| Sand Installation | Placement and levelling | 5 | hours | 150 | 750 |
| Gravel Installation | Placement and levelling | 5 | hours | 150 | 750 |
| Agitator | PTO Propeller Driven Agitator | 2 | - | 5000 | 10000 |
| 6" PVC Pipe | Raw manure transfer pipe | 10 | m | 8.01 | 80 |
| Pipe Installation | | 8 | hours | 20 | 160 |
| | | | | Subtotal = | 28590 |
| Effluent Collection System | 1 | | | <u></u> | |
| Cost is the same as for new | | | | Subtotal = | 106204 |
| Manure Placement Equipn | nent | | | | |
| Cost is the same as for new | | | | Subtotal = | 8259 |
| | | | Cons | struction Cost = | 152273 |
| | | | Constru | ction Cost/m ³ = | 25 |

Table 3.6 Operating costs for thin-layered freezing system for the treatment of liquid swine manure.

| Number of Layers Placed | 1 = 40 | | |
|---|--|---|---|
| Time to Place One Layer (hours | | | |
| Labor Charge (\$/hour | • | | |
| | /- 12 | | |
| Winter Operation | · · · · · · · · · · · · · · · · · · · | | |
| Task | Details | Time to Complete (hours) | Total Cost |
| Sealing Layer | 4 to 5 applications of water | 15 | 180 |
| Raw Manure Storage Agitation | Agitate for 30 min prior to placing and during | 20 | 240 |
| Layer Placement | Trough placement and retrieval, pump operation | 80 | 960 |
| Snow Saturation | Required for each snowfall, est. 20 x 2 hour | 40 | 480 |
| EC Reading | Once every 5 layers | 2 | 24 |
| | | Winter Cost = Operating Cost/m ³ = | |
| Spring/Summer Operation | | | |
| Spring/Summer Operation Task | Details | | 0.31 |
| Task | Details Weekly to prevent large buildup | Operating Cost/m ³ = | 0.31 |
| | | Operating Cost/m ³ = Time to Complete (hours) | 0.31 Total Cost |
| Task Raw Manure Storage Agitation | Weekly to prevent large buildup | Operating Cost/m ³ = Time to Complete (hours) 18 | 0.31 Total Cost 216 |
| Task Raw Manure Storage Agitation Biweekly EC Readings | Weekly to prevent large buildup 15 min per reading, until treated water flow | Operating Cost/m ³ = Time to Complete (hours) 18 2 | 0.31 Total Cost 216 24 |
| Task Raw Manure Storage Agitation Biweekly EC Readings Collection Sump Operation | Weekly to prevent large buildup 15 min per reading, until treated water flow Daily checks for pump working | Operating Cost/m ³ = Time to Complete (hours) 18 2 6 48 Summer/Spring Cost = | 0.31 Total Cost 216 24 72 576 888 |
| Task Raw Manure Storage Agitation Biweekly EC Readings Collection Sump Operation | Weekly to prevent large buildup 15 min per reading, until treated water flow Daily checks for pump working | Operating Cost/m ³ = Time to Complete (hours) 18 2 6 48 | 0.31 Total Cost 216 24 72 576 888 |
| Task Raw Manure Storage Agitation Biweekly EC Readings Collection Sump Operation | Weekly to prevent large buildup 15 min per reading, until treated water flow Daily checks for pump working | Operating Cost/m ³ = Time to Complete (hours) 18 2 6 48 Summer/Spring Cost = | 0.31 Total Cost 216 24 72 576 888 0.14 |

3.10 References

- Alberta Agriculture Food and Rural Development (AAFRD). 2000. Code of Practice for Responsible Livestock Development and Manure Management. Government of Alberta.
- Ashton, G.D. 1980. *Freshwater ice growth, motion and decay.* In: Dynamics of Snow and Ice Masses, S.C. Colbeck, Editor. New York: Academic Press, Harcourt Brace Javanovich Publishers. pp. 261-304.
- Boyd, D.W. 1976. Normal freezing and thawing degree-days from normal monthly temperatures. Canadian Geotechnical Journal. **13**(2): 176-180.
- Canadian Environmental Technology Advancement Corporation (CETAC) West. 1999. Inventory of environmental technologies for the hog industry: final report.
- Day, D.L., and Funk, T.L. 1998. Processing manure: physical, chemical, and biological treatment. In: Animal Waste Utilization: Effective Use of Manure as a Soil Resource, J.L. Hatfield and B.A. Stewart, Editors. Chelsea Michigan: Ann Arbor Press.
- Fertuck, L. 1968. Water purification by freezing in reservoirs, Work done during the season 1968-1969. Saskatchewan Research Council Report E 69-12, November 1968.
- Fleming, R., and Ford, M. 2002. Comparison of storage, treatment, utilization, and disposal systems for human and livestock wastes. Internal Research Report, Ridgetown College, University of Guelph, Ontario.
- Jones, J.R. 1999. Animal agriculture: waste management practices: Report to the Honorable Tom Harkin, Ranking Minority Member, Committee on Agriculture, Nutrition, and Forestry, U.S. Senate. United States General Accounting Office, Washington, D.C.
- Louey, K. 2003. Personal Communication, October 21, 2003. Nilex Incorporated: 9304-39 Avenue, Edmonton, Alberta.
- McGill, W.B. 1997. Sustainability of manure management: nutrient retention and cost-benefit analysis. Final Report CAESA Project RES-056-93.
- Miner, J.R., Humenik, F.J., and Overcash, M.R. 2000. Managing livestock wastes to preserve environmental quality. Iowa State University Press, Ames, Iowa.
- Natural Resources Conservation Service. 2004. Costs Associated with Development and Implementation of Comprehensive Nutrient Management Plans, Part I:Nutrient Management, Land Treatment, Manure and Wastewater Handling and Storage, and Recordkeeping. United States Department of

Agriculture: <u>http://www.nrcs.usda.gov/technical/land/pubs/cnmp1h.pdf</u>, accessed on August 21, 2004.

- Province of Alberta. 2000. Agricultural Operation Practices Act. Alberta Queen's Printer.
- Statistics Canada. 2003. Manure storage in Canada 2003: Vol. 1, No. 1. Catalogue no. 21-021-MIE. Agriculture Division, Ottawa.
- Westerman, P.W., and Zhang, R. 1995. Aerobic treatment of animal waste for odour control. In Proceedings '95, New Knowledge in Livestock Odour, pp. 218-222. Iowa Sate University, Ames, Iowa.
- Willoughby, A.R., Biggar, K., and Sego, D.C. 2001. Nutrient extraction from hog barn wash water using freeze separation. Internal Research Report, University of Alberta, Department of Civil and Environmental Engineering. Edmonton, Canada.
- Zering, K. 2000. *How to select an alternative manure treatment system*. Presented at the North Carolina Pork Conference, January 12. Fayetteville, North Carolina.
- Zhang, R., and Westerman, P.W. 1995. Solid-liquid separation for animal waste. In Proceedings '95, New Knowledge in Livestock Odour, pp. 228-233. Iowa Sate University, Ames, Iowa.

4 CONCLUSIONS

This study focused on the use of thin-layered freezing to treat liquid swine manure. Freeze separation technology could increase the economic value of treated manure by concentrating the nutrients into a smaller volume. To evaluate this premise, a pilot-scale field test was conducted to measure the efficiency of nutrient extraction and concentration using thin-layered freezing to treat liquid swine manure. The results of the pilot study were used to develop a full-scale system for intensive livestock operations in central Alberta. The conclusions reached from this research are presented below.

4.1 Field Test Results

The purpose of the field test was to validate the system design and to determine the percentage of nutrient extraction during field scale freeze separation. The efficacy of nutrient extraction was measured by determining the total mass of nutrients placed in the freezing pit and comparing it to the mass of nutrients contained in the thawed effluent over time. More than 75% of the nutrients (nitrogen, potassium, and sulfur) were recovered and concentrated to one-third the original volume. The increased concentration and decreased volume adds significant economic value to the treated effluent as it decreases the cost for transport and reduces the number of field applications required. A significant volume of treated water was produced, which may be a valuable resource for use as recycle or irrigation water on farms. The freezing and thawing process also removed the objectionable odour of liquid swine manure and provides a high source of phosphorous in the solids component. At the pilot scale thin-layered freezing was shown to be a viable component of sustainable manure management programs in cold regions that utilizes all components of the waste.

4.2 On-Farm Application Design and Limitations

A thin-layered freezing system was designed for a 3000 head swine operation in central Alberta, including both the technical components and operational procedures. The capital cost for constructing a new system was \$25/m³ of manure treated

compared to \$15/m³ treated for retrofitting an existing dual earthen manure storage system. The annual operating costs were found to be \$0.44/m³ of treated manure and the geometric rate of return for investing in thin-layered freezing was found to be 2.3% over a 5-year period.

Limitations of the full-scale operation include dealing with the large volumes of raw and treated fluid, the incorporation of covers to minimize odour and nutrient loss during storage, and the significant capital investment required. The benefits of introducing an economical and environmentally sustainable manure management system on a regional level must be weighed against the limitations and cost. Thinlayered freezing has shown the ability to recover and concentrate nutrients into a smaller volume, which translates into the production of a valuable product that can be used regionally. The key to overcoming the limitations is to view the system as a means of turning waste into a valuable and usable product.

4.3 Recommendations for Future Work

There are several components that require further research to support the development of thin-layered freezing as a viable treatment option for intensive livestock operations. Laboratory testing is required to validate the odour reducing capacity of freeze separation and to quantify the quality of treated water for use as animal drinking water, recycle water, or irrigation water. Further research into the effects of freezing on organic and inorganic nitrogen in treated manure is required to validate the observed low nitrate levels and high ammonium nitrogen in treated manure. Future testing is required to explore the effect of freezing and thawing on the consistency and friability of solids in manure and also to determine its value as compost additive. The treated water requires further testing to investigate its use as animal drinking water or irrigation water.

The results of this study show the potential of thin-layered freezing as an alternative means of treating and managing liquid swine manure at intensive livestock operations. Further work that supports the additional benefits of the process is necessary before the true economic worth of the technology is seen at the farm scale.

Appendix A: 2003 Field Test Recommendations

The objectives set out for the 2003 field test included proving out the design of the freezing pit and collection system, testing the viability of using a pit versus a subgrade holding tank for freeze separation, determination of the percent nutrient extraction during freeze separation, and determining the nutrient value of the effluent produced throughout the period of the test and the quality of thaw fluid for use as recycle water. Due to technical difficulties encountered during layer application the ability to assess the production of nutrient rich brine from the freeze separation process was impeded. As such, the percent nutrient extraction and the nutrient value of the effluent could not be determined nor could the quality of thaw water be assessed for use as recycle water. The technical difficulties encountered have allowed the design of the freeze separation system to be improved for testing in 2004. The recommendations based on the 2003 system focused on two components: system design and layout, and layer placement procedures.

In terms of system design and layout the collection piping and drainage layers should be maintained as well as the size of the pit used. Recommended changes to the system included: relocation of the collection sump to the outside of the pit with valving to isolate the sump from the pit during layer application, use of a continuous engineered geomembrane liner system, incorporation of a movable spigot system and variable speed pump for layer application, installation of sampling ports at the inlet to the pit and the exit of the collection sump, and incorporation of adequate access to the base of the pit and ice layers without creating preferential flow paths within the frozen layers. The incorporation of these design components will allow determination of the efficacy of freeze separation to isolate and concentrate the nutrients and solutes in liquid hog manure and investigate the quality of thaw water for its possible use as recycle water in hog operations.

In terms of layer placement both the system design and procedures used had several recommendations for improvement. Recommendations for improving the system used to place manure within the freezing pit included: the use of heat tape in placement and collection piping to prevent pipe freeze up, redesign of the manure

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placement piping to prevent migration of unfrozen manure beneath the frozen layers and collection system, and the use of a variable speed pump for manure placement to control the volume and placement of manure.

Three major recommendations were made to improve layer placement and prevent thermal erosion of frozen layers. First, a thin layer of ice must be developed over the entire drainage layer (sand, gravel and piping) to minimize the amount of manure entering the collection system during subsequent layer placement. Following development of the sealing layer, manure may be placed in the pit for freeze separation. Second, in the event of a snowfall, a thin layer of manure should be placed to melt the snow and allow proper formation of a frozen layer before subsequent manure placement. Finally, layers should not be placed if the manure is above 2 °C and preferably less then -1 °C. The daily high ambient air temperature should be less than 0 °C and the daily low should be less than -10 °C.

Appendix B: Manure Placement Procedures

- 1. Verify that any previously placed material has frozen completely by tapping the ice surface with a rod or shovel.
- 2. Verify that the collection system effluent valve is closed to prevent any fresh manure from entering the sump during layer application.
- 3. Record ambient air temperature and the temperature of the manure in the storage tank.
- 4. Circulate the manure in the storage tank for a minimum of 5 minutes to ensure adequate mixing of the solids prior to application. Circulation should not exceed 5 minutes as the heat generated from the pump may increase the temperature of manure.
- 5. Lower the layer dispenser into the pit and connect the flexible hosing to the dispenser and the layer placement piping. Position the dispenser to allow for even flow of manure over the previously frozen layer and for ease of handling during application.
- 6. Position a thermistor on top of the previously frozen layer.
- 7. Place a depth marker at the base of the ladder used to access the pit and ensure a 7.62 cm (3 inch) marking is visible from the upper embankment.
- 8. Exit the pit and turn on the pump to desired flow rate for application. The flow rate must to low enough to allow for dissipation of heat from the manure as it flows over the dispenser. Record the time at which manure flows from the dispenser and the flow rate used during application.
- 9. Allow manure to be placed to a maximum depth of 7.62 cm (3 inches). If the base is not level ensure the maximum depth placed at the lowest point is 7.62 cm. The first few layers may not cover the entire base of the pit due to sloping of the collection system for drainage.
- 10. Turn off the pump and leave all valves in the open position to allow gravity drainage of the piping. Record the length of time manure was applied for at the specified flow rate.
- 11. Remove the layer dispenser from the pit and disconnect the flexible hosing. Ensure the hose and all piping is completely drained of manure.
- 12. Photograph the pit prior to and after placement of the manure.

13. Layer placement should not be undertaken during or in anticipation of heavy snowfall events. In the event of heavy snowfall manure should be placed only after completion of snowfall and in an amount sufficient to saturate and liquefy the snow. Subsequent placement should not occur until the snow has been melted with manure and allowed to freeze.

Appendix C: Field Data

Manure Placement Data Flow Rate (L/min):

| FR1 = FR2 = | | FR3 = | 100 | MST = manure | SUI OUT LOUK | | |
|----------------|---------------------------|------------------------------------|------------------------|------------------------|-----------------------|-------------------------|-----------------|
| Date | Air Temp | Manure Temp | Time @FR1 | Time@FR2 | Time @ FR3 | Depth Placed | Volume (L |
| | -11 | -2 | 4 | 5 | 0 | 5.0 cm (max) | 505.6 |
| 23-Jan-04 | Comments: Wate | er indicator didn't work | c, will have to use fi | owrates. Snowing a | nd windy during pla | cement. Time of place | cement 9:30 a |
| | -16 | -2 | 0 | 11 | 0 | 5.0 cm (max) | 880 |
| | Comments: Heav | vy snowfall, SW wind. | Time of placement | t 9:44 a.m. Valve fro | oze, must leave in o | ppen position with hea | at tape on. |
| 24-Jan-04 | | | | | | | |
| | -21 | -2 | 0 | 20 | 0 | Snow cover | 1600 |
| | Comments: Sligh | t wind. Flumies. Mar | iure placement at 8 | :44 a.m. Saturated s | snow cover. | | |
| 25-Jan-04 | | | | | | | |
| | -31 | -2 | 23 | 0 | 0 | 2.5 cm (max) | 230 |
| | | cold, pipe froze off. P | | | | Flow rate was 10 L/m | in due to pipe |
| 26-Jan-04 | freezing off. Man | ure still ponding along | east side of pit. Ti | me of placement 1:0 | 00 p.m. | | |
| | -20 | -2 | 18 | 0 | 0 | Snow cover | 2160 |
| | Comments: Abou | t 15 cm of snow in ba | se of pit. Couldn't | reach west side of pi | it with hose. Placed | t manure with hose o | nly, not disper |
| 1-Feb-04 | Flow rate was 120 |) L/min. | | | | | |
| _ | -18 | -2 | 26 | 0 | 0 | Snow cover | 686 |
| | Comments: Manu | re placed at 11:00 a. | m. Covered snow | on west side. Filled s | storage tank with m | anure, temp at time of | of transfer +5. |
| 2-Feb-04 | Discovered 1* of f | luid in collection sump | o, probably from that | w two weeks prior. | | | |
| | -11 | 2.5 | 48 | 10 | 0 | 7.62 cm (max) | 2067 |
| | Comments: Place windy. | ed layer at 2:22 a.m. I | No fluid in collectio | n sump atter placem | ent. Downloaded d | ata from Jan 5 to Fet | 53. Sunnyan |
| 3-Feb-04 | | | | | | | |
| | -10 | 1.5 | 6 | 47 | 13 | 7.62 cm (max) | 5652 |
| 1-Feb-04 | Comments: Laye | r 1. Time of placement | n 11:00 a.m. Sunr | ny with SW who. This | stalled them istor a | in trastayer. | |
| | -18 | -1 | 0 | 0 | 31 | 7.62 cm (max) | 4133 |
| | | r 2. Time of placement | | | | iyers. Transferred m | anure from lift |
| 5-Feb-04 | station to storage. | manure temp @ 15 a | t time of transfer. | Sampled manure from | m tank transfer. | | _ |
| | -6 | 3 | 0 | 20 | 0 | 5.08 cm (max) | 1600 |
| | Comments: Layer | r 3. Time of placemer | nt 9:00 a.m. Placed | thermistor 6 in base | e of this layer. Plac | ed thermistor 3 at top | of pit. Drifted |
| 3-Feb-04 | snow in pit base, r | nax depth at edges 15 | 5 cm and 5 cm in c | entre. Saturated maj | jority of snow cover | : | |
| | -8 | -0.8 | 0 | 0 | 3 | Surface cover | 400 |
| | | npted to place manure | | | | orth side of membrar | ne. Melting |
| 5-Feb-04 | created gap betwe | en surface of frozen l | ayers and membra | ne, and manure flow | ed here. | | |
| | -11 | -1 | 0 | 0 | 8 | Snow cover | 1067 |
| | Comments: Time | of placement 09:00 a | .m. Saturated snow | w cover. Snow cover | r approximately 8 c | m. Returned at 3:30 | p.m to place |
| 2-Mar-04 | | morning placement no | | | | | |
| | -14 | -1 | 0 | 0 | 18 | 7.62 cm (max) | 2969 |
| | | | | | | | |
| | Comments: Laver | Time of placemer | nt 8:45 a.m. Snow | cover approximately | 2.5 cm. Placed the | ermistor #4 in this lay | er. Overcast a |

Effluent Collection Data

| Date | Daily Volume | Cumulative Volume | Sample ID | EC | pН |
|-----------|--------------|-------------------|------------------|---------|-----|
| | (L) | (L) | | (mS/cm) | |
| 17-Feb-04 | 151 | 151 | CS-Top-170204 | 31.1 | 7.8 |
| 17-Feb-04 | 757 | 908 | CS-Bottom-170204 | 33.5 | 7.5 |
| 18-Feb-04 | 151 | 1059 | CS1-180204 | 39.9 | 7.7 |
| 18-Feb-04 | 453 | 1512 | CS2-180204 | 37.4 | 7.6 |
| 18-Feb-04 | 304 | 1816 | CS3-180204 | 34.4 | 7.7 |
| 19-Feb-04 | 742 | 2558 | CS-190204 | 35.4 | 7.6 |
| 20-Feb-04 | 565 | 3123 | CS-200204 | 29.0 | 7.7 |
| 21-Feb-04 | 459 | 3582 | CS-2100204 | 23.7 | 7.5 |
| 22-Feb-04 | 778 | 4360 | CS-220204 | 26.2 | 7.7 |
| 23-Feb-04 | 636 | 4996 | CS-230204 | 16.7 | 7.8 |
| 24-Feb-04 | 353 | 5350 | CS-240204 | 20.5 | 7.6 |
| 25-Feb-04 | 212 | 5562 | CS-250204 | 22.9 | 7.6 |
| 26-Feb-04 | 212 | 5774 | CS-260204 | 17.1 | 7.9 |
| 27-Feb-04 | 212 | 5986 | CS-270204 | 17.7 | 7.8 |
| 28-Feb-04 | 141 | 6127 | CS-280204 | 15.9 | 7.7 |
| 1-Mar-04 | 35 | 6162 | CS-010304 | 24.6 | 7.9 |
| 8-Mar-04 | 601 | 6762 | CS-080304 | 21.4 | 7.7 |
| 10-Mar-04 | 1272 | 8035 | CS-100304 | 23.3 | 7.6 |
| 11-Mar-04 | 459 | 8494 | CS-110304 | 22.2 | 7.7 |
| 13-Mar-04 | 1590 | 10085 | CS-130304 | 17.0 | 7.6 |
| 14-Mar-04 | 424 | 10509 | CS-140304 | 16.6 | 7.6 |
| 15-Mar-04 | 212 | 10721 | CS-150304 | 17.4 | 7.6 |
| 16-Mar-04 | 177 | 10898 | CS-160304 | 10.6 | 7.5 |
| 17-Mar-04 | 212 | 11110 | CS-170304 | 11.9 | 7.7 |
| 19-Mar-04 | 1696 | 12806 | CS-190304 | 7.3 | 7.6 |
| 22-Mar-04 | 1944 | 14750 | CS-220304 | 6.0 | 7.7 |
| 25-Mar-04 | 7528 | 22278 | CS-250304 | 3.5 | 7.9 |
| 29-Mar-04 | 5195 | 27473 | CS-290304 | 2.4 | 7.9 |

Appendix D: Analytical Methods and Standards

Anions

Sulfate and Chloride

EPA Method 300.0. Determination of Inorganic Anions by Ion Chromatography. Revision 2.1. John D. Pfaff. United States Environmental Protection Agency Enviromental Monitoring Systems Laboratory, Cincinnati, OH 45268. Office of Research and Development. Revised August 1993

Instrument Used: Dionex 2000i/SP Ion Chromatograph (prior to 2000) After 2000: Dionex DX600 Ion Chromatograph

Cations

Stainton, M. P., M. J. Capel, and F. A. J. Armstrong. 1977. The Chemical Analysis of Freshwater. 2nd ed. Fish. Environ. Can. Misc. Spec. Publ. 25: 180 p. (Available from the Freshwater Institute, Winnipeg, Man.). pp. 147-160

Instrument Used: Perkin Elmer 3300 Atomic Absorption Spectrometer

Dissolved Organic Carbon (DOC)

Total Organic Carbon (5310)/Combustion Infrared Method. In: Standard Methods for the Examination of Water and Wastewater. 18th Ed. (1992). Greenberg, A.E., L. S. Clesceri, and A. D. Eaton, editors. American Public Health Association, American Water Works Association and Water Environment Federation, publishers.

Instrument Used: Ionics Model 1505 Programmable Carbon Analyzer

Nitrogen

Ammonium (NH4+) by FLOW-INJECTION ANALYSIS Automated Berthelot Reaction1: TechniconTM AutoanalyzerTM II Method #696-82W (Pre-1994) TechniconTM AutoanalyzerTM II Method #154-71W/B (1994 to date)

Nitrite+Nitrate (NO2- + NO3-) by FLOW-INJECTION ANALYSIS

Automated Cu/Cd Reduction2: TechniconTM AutoanalyzerTM II Method #158-71W/Preliminary (1994 to *date*)

Total Dissolved Nitrogen (TDN) by FLOW-INJECTION ANALYSIS

Stainton, M. P., M. J. Capel, and F. A. J. Armstrong. 1977. The Chemical Analysis of Freshwater. 2nd ed. Fish. Environ. Can. Misc. Spec. Publ. 25: 180 p. (Available from the Freshwater Institute, Winnipeg, Man.). P.91.

TechniconTM AutoanalyzerTM II

Total Nitrogen (TN) by FLOW-INJECTION ANALYSIS

Ameel, J.J., R.P. Axler and C.J. Owen. Persulfate digestion for Determination of Total Nitrogen and Phosphorus in Low-Nutrient Water. American Environmental Laboratory (AEL), 10/93, Feature Article.

Automated Cu/Cd Reduction2: TechniconTM AutoanalyzerTM II Method #158-71W/Preliminary. With modifications based on U.S. EPA Method 353.2

Total Phosphorous

Menzel, D. W., and N. Corwin. 1965. The measurement of total phosphorus in seawater based on the liberation of organically bound fractions by persulfate oxidation. Limnol. Oceanogr. 10: 280-282.

As modified by: Prepas, E. E., and F. H. Rigler.1982.Improvements in quantifying the phosphorus concentration in lake water. Can. J. Fish. Aquat. Sci. 39: 822-829.

Varian Cary 50 UV-Vis Spectrophotometer at 885nm

Additional Analytical References

- 1. i. Van Slyke, D.D. and Hillen, A.J., BioChem., 102, p.499, 1933
 - ii. Kallman, S., Presentation at Div. I Meeting of ASTM Committee E-3, April, 1967, San Diego, California.
 - iii. Bolleter, W.T., Bushman, C.J. and Tidwell, P.N., Anal. Chem., 33 p.592, 1961.
 - iv. Tellow, J.A. and Wilson, A.L., Analyst, 89, p.453,1964
 - v. Tarugi, A. and Lenci, F., Boll Chim. Farm., 50, p.907, 1912.
 - vi. FWPCA Methods of Chem. Anal. Of Water & Wastewater, November 1969, p.137.
- i. Armstrong, F.A.J., Sterns, C.R. and Strickland, J.D.H., 1967, Deep-Sea Res., 14, pp.381-389, "The Measurement of Upwelling and Subsequent Biological Processes by Means of the Technicon auto-Analyzer and Associated Equipment".

- ii. Grasshoff, K., Technicon International Congress, June 1969.
- iii. Federal Water Pollution Control Administration Methods for Chemical Analysis of Water and Wastes, November 1969.
- 3. i. Standard Methods for the Examination of Water and Wastewater, 12th Ed., 1965,p.205.
 - ii. Kamphake, L.J., Hannah, S.A. and Cohen, J.M., Automated Analysis for Nitrate by Hydrazine Reduction, Water Research, Vol. 1, 1967, p.206.
 - iii. Federal Water Pollution Control Administration Methods for Chemical Analysis of Water and Wastes, November 1969.
- 4. i. Van Slyke, D.D. and Hillen, A.J., BioChem., 102, p.499, 1933
 - ii. Ferrari, A., N.Y. Acad. Sci., Vol. 97, Art. 2, p.792-800.

Limnology Laboratory Standards used for Raw Manure and Effluent Sample Analysis

| | | | | i | |
|--|---|--|--|-----------------------------------|------------------------------------|
| alibration Stan | dards: Raw Dat | and a second | | antenestan des commercials | |
| | (7) | | TDN analyzed in | | <u>(7)</u> |
| NH4+(| | | | TDN (| |
| Conc'n | Peak Height | Conc'n | Peak Height | Conc'n | Peak Height |
| 1005 | 78.4 | 2000 | 71.6 | 2000 | 71. |
| 1005 | 76.4 | 2000 | 72.4 | 2000 | 72 |
| 503 | 38.0 | 1000 | 38.8 | 1000 | 38. |
| 503 | 37.0 | 1000 | 39.3 | 1000 | 39. |
| 251 | 18.7 | 500 | 19.6 | 500 | 19. |
| 251 | 18.0 | 500 | 19.9 | 500 | 19 |
| | | 200 | 8.20 | 200 | 8.2 |
| | | 200 | 8.27 | 200 | 8.2 |
| | | | | | |
| | | | | | |
| | | | | | |
| TP (µ | | DOC | | K (mg | |
| Conc'n | Absorption | Conc'n | Peak Area | Conc'n | Absorbance |
| Conc'n 500 | Absorption 0.727 | Conc'n 50.0 | Peak Area 28,614 | Conc'n 2.00 | Absorbance 0.34 |
| Conc'n 500 500 | Absorption 0.727 0.727 | Conc'n 50.0 25.0 | Peak Area 28,614 14,557 | Conc'n 2.00_ 1.50_ | Absorbance 0.34 0.25 |
| Conc'n 500 | Absorption 0.727 0.727 0.357 | Conc'n 50.0 25.0 10.0 | Peak Area 28,614 14,557 6,087 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 500 | Absorption 0.727 0.727 | Conc'n 50.0 25.0 | Peak Area 28,614 14,557 | Conc'n 2.00_ 1.50_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 500 250 | Absorption 0.727 0.727 0.357 | Conc'n 50.0 25.0 10.0 | Peak Area 28,614 14,557 6,087 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 500 250 250 | Absorption 0.727 0.727 0.357 0.361 | Conc'n 50.0 25.0 10.0 5.00 | Peak Area 28,614 14,557 6,087 2,882 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 500 250 250 100 | Absorption 0.727 0.727 0.357 0.361 0.147 | Conc'n 50.0 25.0 10.0 5.00 1.00 | Peak Area 28,614 14,557 6,087 2,882 830 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 250 250 100 100 | Absorption 0.727 0.727 0.357 0.361 0.147 0.147 | Conc'n 50.0 25.0 10.0 5.00 1.00 | Peak Area 28,614 14,557 6,087 2,882 830 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance 0.34 0.25 0.17 |
| Conc'n 500 500 250 250 100 100 50 | Absorption 0.727 0.727 0.357 0.361 0.147 0.147 0.073 | Conc'n 50.0 25.0 10.0 5.00 1.00 | Peak Area 28,614 14,557 6,087 2,882 830 | Conc'n 2.00_ 1.50_ 1.00_ | Absorbance |

Appendix E: Analytical Data

Nutrient Extractions: 2004

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Note 1: Results include predilution factor(s); thus concentrations given (in mg/L) are for raw samp

| Note 2: | Discrepancies between TN & NH4 (i.e. TN <nh4, highlighted="" samples)<="" th=""></nh4,> |
|---------|---|
| | may be due to differences in dilutions used (1/2000 for NH4, & 1/1000 for TN) |

| | Predilutions Used>>>>> | 1/2000 | 1/1000 | 1/1000 |
|---|----------------------------|----------------|-----------|------------|
| Alexandre de la composition de la compo | and the state of the state | •• NH4+ (mg/L) | TN (mg/L) | TDN (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 2,117 | 2,389 | 2,092 |
| 5-Feb-04 | MST2-Raw Manure | 520 | 3,986 | 3,608 |
| 13-Feb-04 | CS-Leakage-130204 | 3,657 | 4,391 | 3,987 |
| 17-Feb-04 | CS-Bottom-170204 | 4,570 | 4,814 | 4,538 |
| 18-Feb-04 | CS1-180204 | 4,993 | 5.527 | 5,117 |
| 19-Feb-04 | CS-190204 | 4,104 | 4,563 | 4,172 |
| 20-Feb-04 | CS-200204 | 3,707 | 4,059 | 3,650 |
| 21-Feb-04 | CS-2100204 | 3,348 | 3,482 | 3,414 |
| 22-Feb-04 | CS-220204 | 3,205 | 3,823 | 3,581 |
| 23-Feb-04 | CS-230204 | 3,223 | 3,268 | 2,729 |
| 24-Feb-04 | CS-240204 | 3,372 | 3,051 | 2,850 |
| 25-Feb-04 | CS-250204 | 3,712 | 2,777 | 2,650 |
| 26-Feb-04 | CS-260204 | 2,190 | 2,391 | 2,110 |
| 27-Feb-04 | CS-270204 | 2,823 | 2,987 | 2,669 |
| 28-Feb-04 | CS-280204 | 2,916 | 2,770 | 2,623 |
| 1-Mar-04 | CS-010304 | 2,911 | 3,279 | 2,943 |
| 8-Mar-04 | CS-080304 | 2,353 | 2,654 | 2,463 |
| 10-Mar-04 | CS-100304 | 2,659 | 3,111 | 2,706 |
| 11-Mar-04 | CS-110304 | 2,567 | 2,935 | 2,643 |
| 13-Mar-04 | CS-130304 | 1,822 | 2,155 | 1,853 |
| 14-Mar-04 | CS-140304 | 1,611 | 2,280 | 1,991 |
| 15-Mar-04 | CS-150304 | 1,709 | 2,018 | 1,754 |
| 16-Mar-04 | CS-160304 | 1,480 | 1,934 | 1,627 |
| 17-Mar-04 | CS-170304 | 1,542 | 2,050 | 1,989 |
| 19-Mar-04 | CS-190304 | 841 | 1,413 | 1,108 |
| 22-Mar-04 | CS-220304 | 615 | 848 | 633 |
| 25-Mar-04 | CS-250304 | 367 | 540 | 483 |
| 29-Mar-04 | CS-290304 | 227 | 364 | 245 |

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| ······································ | Predilutions Used>>>>> | 1/1000 | 1/100 | 1/1000 | Raw |
|--|------------------------|-----------|------------|----------|------------------|
| | lan da shandar e de | TP (mg/L) | DOC (mg/L) | K (mg/L) | [NO2+NO3] (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 235 | 2,791 | 1,140 | 0.00 |
| 5-Feb-04 | MST2-Raw Manure | 877 | 2,844 | 1,380 | 0.00 |
| 13-Feb-04 | CS-Leakage-130204 | 201 | 4,388 | 2,040 | 0.00 |
| 17-Feb-04 | CS-Bottom-170204 | 283 | 4,040 | 2,200 | 0.00 |
| 18-Feb-04 | CS1-180204 | 305 | 4,787 | 2,390 | 0.00 |
| 19-Feb-04 | CS-190204 | 95 | 4,464 | 2,080 | 0.00 |
| 20-Feb-04 | CS-200204 | 93 | 5,329 | 1,810 | 0.00 |
| 21-Feb-04 | CS-2100204 | 78 | 5,457 | 1,680 | 0.00 |
| 22-Feb-04 | CS-220204 | 99 | 5,672 | 1,810 | 0.00 |
| 23-Feb-04 | CS-230204 | 81 | 4,487 | 1,640 | 0.00 |
| 24-Feb-04 | CS-240204 | 62 | 4,793 | 1,480 | 0.00 |
| 25-Feb-04 | CS-250204 | 47 | 3,977 | 1,440 | 0.00 |
| 26-Feb-04 | CS-260204 | 67 | 3,761 | 1,180 | 0.00 |
| 27-Feb-04 | CS-270204 | 77 | 3,760 | 1,420 | 0.00 |
| 28-Feb-04 | CS-280204 | 48 | 4,303 | 1,330 | 0.00 |
| 1-Mar-04 | CS-010304 | 49 | 5,139 | 1,650 | 0.00 |
| 8-Mar-04 | CS-080304 | 239 | 4,251 | 1,330 | 0.00 |
| 10-Mar-04 | CS-100304 | 167 | 4,949 | 1,940 | 0.00 |
| 11-Mar-04 | CS-110304 | 106 | 4,870 | 1,410 | 0.00 |
| 13-Mar-04 | CS-130304 | 64 | 3,466 | 1,060 | 0.00 |
| 14-Mar-04 | CS-140304 | 64 | 3,340 | 1,050 | 0.00 |
| 15-Mar-04 | CS-150304 | 57 | 3,353 | 971 | 0.00 |
| 16-Mar-04 | CS-160304 | 51 | 3,194 | 931 | 0.00 |
| 17-Mar-04 | CS-170304 | 51 | 3,104 | 971 | 0.00 |
| 19-Mar-04 | CS-190304 | 48 | 1,862 | 621 | 0.00 |
| 22-Mar-04 | CS-220304 | 41 | 1,267 | 391 | 0.00 |
| 25-Mar-04 | CS-250304 | 34 | 788 | 250 | 0.00 |
| 29-Mar-04 | CS-290304 | 29 | 508 | 150 | 0.00 |

| | Predilutions Used>>>>> | Raw | 10X | 10X | 10X |
|-----------|------------------------|------------|------------------|------------|------------|
| Date | Sample ID | SO4 (mg/L) | SO4 Anion (mg/L) | NO2 (mg/L) | NO3 (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 8.17 | 2.98 | 496 | 0.00 |
| 5-Feb-04 | MST2-Raw Manure | 4.18 | 2.16 | 710 | 0.00 |
| 13-Feb-04 | CS-Leakage-130204 | 6.83 | 8.64 | 913 | 0.00 |
| 17-Feb-04 | CS-Bottom-170204 | 7.91 | 4.18 | 1282 | 0.00 |
| 18-Feb-04 | CS1-180204 | 4.80 | 1.00 | 1375 | 2.22 |
| 19-Feb-04 | CS-190204 | 0.50 | 1.82 | 1311 | 0.00 |
| 20-Feb-04 | _CS-200204 | 1.37 | 1.85 | 1094 | 0.00 |
| 21-Feb-04 | CS-2100204 | 4.60 | 3.69 | 466 | 0.00 |
| 22-Feb-04 | CS-220204 | 6.24 | 4.60 | 1056 | _0.00 |
| 23-Feb-04 | CS-230204 | 2.11 | 0.50 | 883 | 3.12 |
| 24-Feb-04 | CS-240204 | 2.13 | 30.96 | 844 | 0.00 |
| 25-Feb-04 | CS-250204 | 3.94 | 1.58 | 898 | 0.63 |
| 26-Feb-04 | CS-260204 | 2.35 | 1.99 | 611 | 0.00 |
| 27-Feb-04 | CS-270204 | 1.70 | 0.00 | 608 | 0.00 |
| 28-Feb-04 | CS-280204 | 0.83 | 0.85 | 826 | 0.00 |
| 1-Mar-04 | CS-010304 | 0.89 | 3.88 | 896 | 2.57 |
| 8-Mar-04 | CS-080304 | 2.45 | 2.51 | 736 | 1.06 |
| 10-Mar-04 | CS-100304 | 0.47 | 1.40 | 772 | 1.86 |
| 11-Mar-04 | CS-110304 | 1.73 | 1.08 | 755 | 0.00 |
| 13-Mar-04 | CS-130304 | 12.53 | 1.84 | 553 | 0.00 |
| 14-Mar-04 | CS-140304 | 1.56 | 0.00 | 489 | 0.00 |
| 15-Mar-04 | CS-150304 | 1.83 | 1.52 | 484 | 4.14 |
| 16-Mar-04 | CS-160304 | 0.56 | 0.00 | 452 | 0.00 |
| 17-Mar-04 | CS-170304 | 0.00 | 5.37 | 456 | 0.00 |
| 19-Mar-04 | CS-190304 | 1.87 | 0.00 | 239 | 0.00 |
| 22-Mar-04 | CS-220304 | 1.75 | 0.00 | 150 | 0.00 |
| 25-Mar-04 | CS-250304 | 2.34 | 2.23 | 87 | 0.00 |
| 29-Mar-04 | CS-290304 | 5.17 | 3.68 | 53 | 0.00 |

| | Predilutions Used>>>>> | 10X | 10X | 10X |
|-----------|------------------------|------------|------------|-----------|
| | | | | |
| 😤 Date | Sample ID | Br- (mg/L) | Cl- (mg/L) | F- (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 0.00 | 43.78 | 0.00 |
| 5-Feb-04 | MST2-Raw Manure | 29.90 | 321.44 | 0.00 |
| 13-Feb-04 | CS-Leakage-130204 | 14.89 | 206.47 | 11.20 |
| 17-Feb-04 | CS-Bottom-170204 | 21.75 | 321.23 | 4.46 |
| 18-Feb-04 | CS1-180204 | 12.58 | 418.25 | 0.00 |
| 19-Feb-04 | CS-190204 | 16.41 | 378.92 | 0.00 |
| 20-Feb-04 | CS-200204 | 17.54 | 317.29 | 0.00 |
| 21-Feb-04 | CS-2100204 | 13.02 | 36.48 | 0.00 |
| 22-Feb-04 | CS-220204 | 14.15 | 110.53 | 0.00 |
| 23-Feb-04 | CS-230204 | 4.36 | 31.07 | 5.85 |
| 24-Feb-04 | CS-240204 | 15.24 | 108.51 | 0.00 |
| 25-Feb-04 | CS-250204 | 9.75 | 37.32 | 0.00 |
| 26-Feb-04 | CS-260204 | 12.32 | 61.25 | 3.79 |
| 27-Feb-04 | CS-270204 | 16.90 | 0.00 | 0.00 |
| 28-Feb-04 | CS-280204 | 22.56 | 59.62 | 0.00 |
| 1-Mar-04 | CS-010304 | 14.78 | 169.09 | 0.00 |
| 8-Mar-04 | CS-080304 | 10.69 | 14.14 | 7.37 |
| 10-Mar-04 | CS-100304 | 11.87 | 25.46 | 1.98 |
| 11-Mar-04 | CS-110304 | 16.97 | 121.38 | 2.18 |
| 13-Mar-04 | CS-130304 | 13.96 | 19.13 | 0.00 |
| 14-Mar-04 | CS-140304 | 9.21 | 0.55 | 0.85 |
| 15-Mar-04 | CS-150304 | 14.49 | 0.00 | 0.00 |
| 16-Mar-04 | CS-160304 | 16.50 | 0.00 | 0.00 |
| 17-Mar-04 | CS-170304 | 0.00 | 0.87 | 0.00 |
| 19-Mar-04 | CS-190304 | 0.00 | 0.92 | 0.00 |
| 22-Mar-04 | CS-220304 | 0.00 | 0.00 | 0.00 |
| 25-Mar-04 | CS-250304 | 0.00 | 0.00 | 0.00 |
| 29-Mar-04 | CS-290304 | 0.00 | 0.00 | 0.00 |

| | Predilutions Used>>>>> | 10X | 10X | 10X |
|-----------|------------------------|------------|------------|-----------|
| Date | Sample ID | PO4 (mg/L) | NH4 (mg/L) | Ca (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 341.73 | 2118 | 83 |
| 5-Feb-04 | MST2-Raw Manure | 488.06 | 3340 | 220 |
| 13-Feb-04 | CS-Leakage-130204 | 234.68 | 3333 | 22 |
| 17-Feb-04 | CS-Bottom-170204 | 300.77 | 4090 | 103 |
| 18-Feb-04 | CS1-180204 | 378.68 | 4377 | 133 |
| 19-Feb-04 | CS-190204 | 61.87 | 3840 | 248 |
| 20-Feb-04 | CS-200204 | 51.06 | 3417 | 179 |
| 21-Feb-04 | CS-2100204 | 0.00 | 1701 | 81 |
| 22-Feb-04 | CS-220204 | 2.15 | 3209 | 124 |
| 23-Feb-04 | CS-230204 | 0.00 | 2655 | 108 |
| 24-Feb-04 | CS-240204 | 9.14 | 2797 | 114 |
| 25-Feb-04 | CS-250204 | 0.00 | 2655 | 115 |
| 26-Feb-04 | CS-260204 | 10.90 | 2155 | 113 |
| 27-Feb-04 | CS-270204 | 0.00 | 2156 | 96 |
| 28-Feb-04 | CS-280204 | 0.00 | 2559 | 115 |
| 1-Mar-04 | CS-010304 | 0.00 | 2983 | 122 |
| 8-Mar-04 | CS-080304 | 7.24 | 2397 | 89 |
| 10-Mar-04 | CS-100304 | 74.46 | 2549 | 79 |
| 11-Mar-04 | CS-110304 | 89.91 | 2565 | 83 |
| 13-Mar-04 | CS-130304 | 0.00 | 1992 | 76 |
| 14-Mar-04 | CS-140304 | 0.00 | 1850 | 68 |
| 15-Mar-04 | CS-150304 | 23.14 | 1845 | 71 |
| 16-Mar-04 | CS-160304 | 0.00 | 1771 | 70 |
| 17-Mar-04 | CS-170304 | 0.00 | 1771 | 72 |
| 19-Mar-04 | CS-190304 | 0.00 | 1071 | 47 |
| 22-Mar-04 | CS-220304 | 36.00 | 771 | 33 |
| 25-Mar-04 | CS-250304 | 29.58 | 490 | 22 |
| 29-Mar-04 | CS-290304 | 35.46 | 332 | 19 |

| | Predilutions Used>>>>> | 10X | 10X | 10X |
|-----------|------------------------|-----------|-----------|-----------|
| | | | | |
| 🛪 Date 😣 | Sample ID | Mg (mg/L) | K+ (mg/L) | Na (mg/L) |
| 5-Feb-04 | MST1-Raw Manure | 3.46 | 1173 | 381 |
| 5-Feb-04 | MST2-Raw Manure | 16.80 | 1692 | 484 |
| 13-Feb-04 | CS-Leakage-130204 | 0.00 | 1777 | 639 |
| 17-Feb-04 | CS-Bottom-170204 | 6.94 | 2460 | 875 |
| 18-Feb-04 | CS1-180204 | 7.07 | 2597 | 923 |
| 19-Feb-04 | CS-190204 | 35.16 | 2235 | 872 |
| 20-Feb-04 | CS-200204 | 29.88 | 1941 | 745 |
| 21-Feb-04 | CS-2100204 | 13.05 | 909 | 351 |
| 22-Feb-04 | CS-220204 | 12.68 | 1930 | 721 |
| 23-Feb-04 | CS-230204 | 12.38 | 1563 | 582 |
| 24-Feb-04 | CS-240204 | 18.03 | 1602 | 602 |
| 25-Feb-04 | CS-250204 | 23.95 | 1511 | 575 |
| 26-Feb-04 | CS-260204 | 12.25 | 1234 | 452 |
| 27-Feb-04 | CS-270204 | 9.68 | 1240 | 446 |
| 28-Feb-04 | CS-280204 | 23.18 | 1445 | 541 |
| 1-Mar-04 | CS-010304 | 21.53 | 1694 | 641 |
| 8-Mar-04 | CS-080304 | 4.31 | 1457 | 517 |
| 10-Mar-04 | CS-100304 | 3.89 | 1528 | 537 |
| 11-Mar-04 | CS-110304 | 7.91 | 1523 | 540 |
| 13-Mar-04 | CS-130304 | 10.97 | 1172 | 423 |
| 14-Mar-04 | CS-140304 | 8.95 | 1046 | 379 |
| 15-Mar-04 | CS-150304 | 8.20 | 1032 | 376 |
| 16-Mar-04 | CS-160304 | 10.38 | 989 | 359 |
| 17-Mar-04 | CS-170304 | 9.78 | 993 | 363 |
| 19-Mar-04 | CS-190304 | 7.88 | 604 | 217 |
| 22-Mar-04 | CS-220304 | 6.65 | 415 | 149 |
| 25-Mar-04 | CS-250304 | 5.86 | 249 | 89 |
| 29-Mar-04 | CS-290304 | 6.27 | 171 | 57 |

Appendix F: Design Standards for Manure Storage Facilities

The design standards summarized here are based on the 2000 Code of Practice for Responsible Livestock Development and Manure Management. The Code defines acceptable standards and practices for the design and operation of earthen manure storages in Alberta. It includes covers, siting, site investigation and design criteria for earthen manure storages. A brief summary has been included for the purposes of this research and for illustrative purposes.

Siting criteria require the bottom elevation of the liquid manure storage facility be constructed a minimum of 1 m above the seasonal high water table. In areas subject to flooding, the earthen manure storage must be 1 m above the 1:50 year floodplain, or 1 m above the highest known flood elevation. The erosion control measures must be designed for a 1:50 year flood event.

Manure storage facilities may be earthen or a tank structure. Earthen facilities must be designed and constructed to minimize odour nuisance and protect ground and surface waters. Nine months of storage volume is required and must be sufficient to store all of the manure, wash water and water spillage produced. The material stability and method used to empty the storage facility will determine the side slopes, which will be no steeper than 1.5:1. A run-off control system is required to prevent the flow of surface water into the storage facility.

To prevent the loss of nutrients from earthen manure storage structures a minimum of 10 m of natural uniform material with a hydraulic conductivity less than 1×10^{-6} cm/s must be between the bottom and sides of the storage and above the uppermost identified groundwater source. A geosynthetic material that provides an equivalent or greater protection may be utilized provided it is designed and constructed by and approved by a professional engineer. Seepage monitoring may be required in addition to the liner requirements.

A site investigation should be undertaken to properly design and construct the storage facility. Useful data sources include but are not limited to: soil survey data, surficial geology maps, water well logs, hydrogeological maps, and aerial

photography. In addition, the operational limitations and maximum volume requirements over the lifetime of the facility should also be examined to ensure storage requirements are met.

Appendix G: Thin Layered Freezing System Design Spreadsheets

Volume Requirements

Assumptions:

Volumetric moisture content of manure is 90% 12 months of manure must be placed in one winter Assume nutrient rich brine is approximately 30% of volume to be treated Assume treated water is approximately 60% of volume to be treated Assume treated solids are 10% of volume to be treated

| Layer Depth (m) = | 0.075 |
|-----------------------------|-------|
| Max # of freezing days = | 90 |
| Maximum ice depth (m) = | 3 |
| Freeboard required (m) = | 0.5 |
| Pit Side Slope (Rise:Run) = | 1 |

| Swine Units | 3000 |
|-------------|------|

| Manure Production | (L/day) |
|-------------------|-----------------|
| Nursery | - |
| Growing | 6.3 |
| Finish | 5.6 |
| Gestating | 5.6 |
| | (Sutton et al.) |

Volume of Manure Requiring Treatment (Litres): 6132000 L

V_{treat}, Volume of Manure Requiring Treatment (m³): 6132 m³

9 Months Storage Volume (Litres): 4536000 L

9 Months Storage Volume (m³): 4536 m³

Freezing Pit Design

Freezing Pit Dimensions

Base Area $(m^2) = 2044$ Base Length, L (m) = 45Base Width, W (m) = 45

Volume of One Layer (m³) = 153

Number of Layers Placed = 40

Note:

ax. Volume Capacity of Pit (m3) = 6946

D_{ce} = depth of frozen manure L = pit length W = pit width V_{frozen-max} = maximum volume of frozen manure V_{treat} = volume of manure to treat

Maximum Volume Capacity Determination

 $V_{\text{frozen-max}} = (D_{\text{ce}}^2 \times L) + (D_{\text{ce}}^2 \times W) + (L \times W \times D_{\text{ce}})$

Base Area = V_{treet}/D_{ree}

Dimensions of freezing pit are calculated based on rectangular volume without considering volume of manure placed along side slopes. This allows for 9% volume expansion during freezing and snow and rainfall accumulation within the pit.

Liquid Storage Requirements

| Component | % of V _{treat} | Storage Volume (m ³) |
|---------------------|-------------------------|----------------------------------|
| Nutrient Rich Brine | 30 | 1840 |
| Treated Water | 60 | 3679 |
| Solids | 10 | 613 |
| | | 6132 |

Nutrient rich brine and treated water volumes >> capital available for field erected tank storage Require lagoon storage of both fluids

<u>Treated Water Lagoon</u> Depth of Lagoons (m) = 4 Side Slopes (1:1) Base Area Required (m²) = 920 Length, Width (m) = 30

Collection Sump (CS) Dimensions

Must be minimum of 5 m high to allow for gravity drainage of effluent from base of freezing pit Allow for 1 m of fluid level in CS before pump turns on Diameter chosen by designer

| CS Height (m) = 5 | Time to Empty CS | |
|--|--------------------------------|-----|
| CS Diameter (m) = 2.0 | Flow Rate (L/min) = | 130 |
| Max fluid level (m) = 1.00 | Time to Empty (min) = | 24 |
| Max Volume in tank (m ³) = 3 | Time to Empty (hours) = | 0.4 |
| | Effluent Inflow Rate (L/min) = | 91 |

Raw Manure Storage

Require 9 months storage, manure produced during winter is placed in freezing pit after allowing it to cool in raw manure storage lagoon Lagoon requires agitator to ensure solids are mixed prior to layer placement

Depth of liquid in lagoon (m) = 3

| Base Area (m ²) = | 1512 |
|-------------------------------|------|
| Length (m) = | 39 |
| Width (m) = | 39 |

Protection Layer

| Component | Area (m ²) | Depth (m) | Quantity (m ³) |
|----------------------|------------------------|-----------|----------------------------|
| 3/8 Round Pea Gravel | 1600 | 0.1 | 160 |
| Washed Concrete Sand | 1600 | 0.1 | 160 |

Effluent Collection and Storage

Thaw Rate Determination: -Based on experience with thawing in pilot test

-Nutrient Brine:

>max thawing rate based on 30% of total treated manure thawing in 1 week >min thawing rate based on 30% of total treated manure thawing in 3 weeks

-Treated Water:

>max thawing rate based on 60% of total treated manure thawing in 1 month >min thawing rate based on 60% of total treated manure thawing in 3 months

Flow Calculations: Q_{max, effluent} ≃ Max Thaw Rate/Number of Pipes

Q_{max, header} = 0.5Max Thaw Rate

Nutrient Thawing

Use Manning equation to size effluent collection pipes Max Thaw Rate $(m^3/s) = 3.04E-03$ Min Thaw Rate $(m^3/s) = 1.01E-03$ Number of Pipes = 10 Coefficient of Roughness (n) = 0.011 Slope of energy grade line (S) = 0.01 $Q_{max, effluent} (m^3/s) = 3.04E-04$ $Q_{min, effluent} (m^3/s) = 1.01E-04$ Max Pipe Radius, $r_{max} (m) = 0.0162$ Min Pipe Radius, $r_{min} (m) = 0.0108$ $D_{max} (m) = 0.0325$ $D_{min} (m) = 0.0215$

Treated Water Thawing

Use Manning equation to size effluent collection pipes Max Thaw Rate $(m^3/s) = 1.42E-03$ Min Thaw Rate $(m^3/s) = 4.73E-04$ Number of Pipes = 10 Coefficient of Roughness (n) = 0.011Slope of energy grade line (S) = 0.01 $Q_{max, effluent} (m^3/s) = 1.42E-04$ $Q_{min, effluent} (m^3/s) = 4.73E-05$ Max Pipe Radius, $r_{max} (m) = 0.0122$ Min Pipe Radius, $r_{min} (m) = 0.0081$ $D_{max} (m) = 0.024$ $D_{min} (m) = 0.016$ Manning Equation: $Q = (1/n)AR^{2/3}S^{1/2}$ Where: R = Hydraulic radius (m): A = Cross sectional area of pipe; S = Slope; n = Manning n; r = pipe radius

Rearrange to solve for radius of pipe: $r^{8/3} = (nQ)/(1.979S^{1/2})$

> Header Pipe Sizing Use 2 header pipes, one at each end of pit $Q_{max, header} (m^3/s) = 1.52E-03$ $Q_{min, header} (m^3/s) = 5.07E-04$ Coefficient of Roughness (n) = 0.011 Slope of energy grade line (S) = 0.01 Max Pipe Radius, $r_{max} (m) = 0.030$ Min Pipe Radius, $r_{mn} (m) = 0.020$ $D_{max} (m) = 0.0594$ $D_{min} (m) = 0.0393$

Header Pipe Sizing Use 2 header pipes, one at each end of pit $Q_{max, header} (m^3/s) = 7.10E-04$ $Q_{min, header} (m^3/s) = 2.37E-04$ Coefficient of Roughness (n) = 0.011 Slope of energy grade line (S) = 0.01 Max Pipe Radius, $r_{max} (m) = 0.022$ Min Pipe Radius, $r_{min} (m) = 0.015$ $D_{max} (m) = 0.045$ $D_{min} (m) = 0.030$

Effluent Collection and Storage

Pipe Selection

Based on max effluent flow during nutrient brine thawing Increase size by 20% as factor of safety, estimated thawing time is approximate

Single Slotted Effluent Collection Pipes

Number = 10 Diameter (m) = 0.04 Diameter (mm) = 40 Standard Pipe Size, ID (inches) = 2

Header Collection Pipes

Number = 2 Diameter (m) = 0.06 Diameter (mm) = 60 Standard Pipe Size, ID (inches) = 3

Drainage Layer

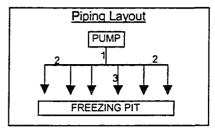
| Component | Area (m ²) | Depth (m) | Quantity (m ³) |
|----------------------|------------------------|-----------|----------------------------|
| 3/8 Round Pea Gravel | 2025 | 0.3 | 607.5 |
| Washed Concrete Sand | 2025 | 0.2 | 405 |

Manure Placement

Hazen Williams Equation:

$$Q = 0.278 \text{CD}^{2.63} \text{S}^{0.54}$$

Rearrange to solve for diameter of pipe:
 $D = [Q/(0.278 \text{CS}^{0.54})]^{1/2.63}$



Q = Flow rate

C = Roughness coefficient

D = Pipe diameter

S = Slope

Assumptions:

Maximum layer placement would be 1 layer/day for December, January, February, and March Design of pump and pipes based on placing one layer in 2 hours. Used Hazen Williams equation, circular pressure conduit flowing full

| Roughness Coefficient, C = | 140 | |
|--|------|----------------------|
| Number of Pumps = | 2 | |
| Required Single Pump Flow Rate (L/min) = | 1278 | $(m^{3}/s) = 0.0213$ |
| Total Flow Rate (L/min) = | | |
| Volume Placed per Layer $(m^3) =$ | 153 | |
| Time to Place One Layer (min.) = | 120 | |
| Time to Place One Layer (hour) = | 2 | |

| Pipe Number | Quantity | Q (m³/s) | Slope, S | D (m) |
|-------------|----------|----------|----------|-------|
| 1 | 1 | 0.0213 | 0.02 | 0.128 |
| 2 | 2 | 0.0106 | 0.02 | 0.099 |
| 3 | 6 | 0.0035 | 0.02 | 0.065 |

| Selected Pipe Size | | | |
|--------------------|--------|--|--|
| Pipe 1 = | 150 mm | | |
| Pipe 2 = | 100 mm | | |
| Pipe 3 = | 75 mm | | |

Appendix H: Operational Procedures

Manure Placement

- 1. Verify that any previously placed material has frozen completely.
- 2. Verify that the forecasted overnight low is less than -10 °C and the maximum daily temperature is less than 0 °C. Manure placement may be undertaken if the overnight low is between 0 °C and -10 °C, however the layer thickness should be decreased to allow the manure to freeze completely in 24 hours.
- 3. Verify that the collection system effluent value is closed to prevent any fresh manure from entering the sump during layer application.
- 4. Turn on the raw manure storage agitators for a minimum of 30 minutes to ensure adequate mixing of the solids prior to application. Agitation should proceed during layer placement to maintain a consistent solids content.
- 5. Lower the manure placement troughs onto the surface of the ice in the freezing pit.
- 6. Open the placement pump shutoff valves and turn on the pump to the appropriate flow rate for application of a 8 cm layer in 2 hours.
- 7. Allow manure to be placed to a maximum depth of 8 cm (3 inches). If the base is not level ensure the maximum depth placed at the lowest point is 7.62 cm. The first few layers may not cover the entire base of the pit due to sloping of the collection system for drainage.
- A sample of the raw manure should be taken during layer placement to measure the electrical conductivity (EC). Comparison of the raw manure EC and effluent EC will determine the cutoff between nutrient rich brine and treated water.
- 9. Turn off the pump and leave all valves in the open position to allow gravity drainage of the piping. Record the length of time manure was applied for at the specified flow rate.
- 10. Remove the manure placement troughs from the ice surface and close the placement pump isolation valves.
- 11. Layer placement should not be undertaken during or in anticipation of heavy snowfall events. In the event of heavy snowfall manure should be placed only after completion of snowfall and in an amount sufficient to saturate and liquefy the snow. Subsequent placement should not occur until the snow has been melted with manure and allowed to freeze.

Appendix I: Nutrient Extraction Calculation

Mass of Nutrients Extracted

Assumptions:

Raw manure concentration based on laboratory testing (Willoughby et al., 2001) Nurient Brine: 75% of the nutrients were recovered and concentrated to one-third the original volume Treated Water: 25% of nutrients remaining in 2/3 of total volume treated Solids: 70% of P placed remains in solids and concentration of K, S, N are negligible

| | | | Mass of Nutrient (kg) | | g) | |
|---------------------|-------------------------|----------------------------------|-----------------------|------|------|------|
| Component | % of V _{treat} | Storage Volume (m ³) | TN | TP | S | К |
| Nutrient Rich Brine | 30 | 1840 | 3909.2 | neg. | 1380 | 690 |
| Treated Water | 60 | 3679 | 1303 | neg. | 460 | 230 |
| Solids | 10 | 613 | neg. | 64.4 | neg. | neg. |
| | | 6132 | | | | |

| Raw Manure Conc | entration (mg/L) | Mass (kg) |
|-----------------|------------------|-----------|
| TN | 850 | 5212 |
| TP | 15 | 92 |
| S | 300 | 1840 |
| К | 150 | 920 |

Mass of Nutrient Produced = $(1000 \text{ L/m}^3) \times (\text{kg/1E6 mg}) \times (\% \text{ Nutrients Concentrated}) \times (Volume Liquid Treated m³) \times [Raw Manure, mg/L]$

Appendix J: Rate of Return Calculation

Rate of Return (ROR)

Compound rate of return used $ROR = (Capital/Return)^{(1/n)}$ n = 5Captial = 235173 Return = 2600 ROR = 2.5

Costs include: investments to modify or replace existing systems Not Included: recurring expenses such as electricity and utilities, repairs and maintenance, professional oversight