NOTE TO USERS

Page(s) not included in the original manuscript and are unavailable from the author or university. The manuscript was scanned as received.

p. 169

This reproduction is the best copy available.

UMI[®]

**EVALUATION OF LOSSES FROM ANAEROBIC BIOSOLIDS DUE TO
WINTER AND LATE FALL LAND APPLICATION**

**BY
KHALED EL-NAGGAR, B.SC (ENG.)**

**A THESIS PRESENTED TO
THE FACULTY OF GRADUATE STUDIES AND RESEARCH
IN PARTIAL FULLFILLMENT OF THE REQUIREMENTS
FOR THE DEGREE OF
MASTER OF APPLIED SCIENCE*
DEPARTMENT OF CIVIL AND ENVIRONMENTAL ENGINEERING
CARLETON UNIVERSITY
OTTAWA, ONTARIO
CANADA**

2004

* The Master of Applied Science program is a joint program with the University of Ottawa
administrated by the Ottawa-Carleton Institute for Civil Engineering

Library and
Archives Canada

Bibliothèque et
Archives Canada

Published Heritage
Branch

Direction du
Patrimoine de l'édition

395 Wellington Street
Ottawa ON K1A 0N4
Canada

395, rue Wellington
Ottawa ON K1A 0N4
Canada

Your file Votre référence

ISBN: 0-612-97404-9

Our file Notre référence

ISBN: 0-612-97404-9

The author has granted a non-exclusive license allowing the Library and Archives Canada to reproduce, loan, distribute or sell copies of this thesis in microform, paper or electronic formats.

L'auteur a accordé une licence non exclusive permettant à la Bibliothèque et Archives Canada de reproduire, prêter, distribuer ou vendre des copies de cette thèse sous la forme de microfiche/film, de reproduction sur papier ou sur format électronique.

The author retains ownership of the copyright in this thesis. Neither the thesis nor substantial extracts from it may be printed or otherwise reproduced without the author's permission.

L'auteur conserve la propriété du droit d'auteur qui protège cette thèse. Ni la thèse ni des extraits substantiels de celle-ci ne doivent être imprimés ou autrement reproduits sans son autorisation.

In compliance with the Canadian Privacy Act some supporting forms may have been removed from this thesis.

Conformément à la loi canadienne sur la protection de la vie privée, quelques formulaires secondaires ont été enlevés de cette thèse.

While these forms may be included in the document page count, their removal does not represent any loss of content from the thesis.

Bien que ces formulaires aient inclus dans la pagination, il n'y aura aucun contenu manquant.

Canada

ABSTRACT

Land application of biosolids has typically been avoided under adverse conditions (frozen ground, snow cover). In some years this can substantially constrain the land application period and result in either increased landfilling of biosolids or the need for extended storage capacity. The concern related to application on frozen ground or snow cover is the mobility of contaminants if placed on the surface on frozen soil, especially nutrient losses. Little is currently known about the fate of biosolids when land applied under such conditions (> 30% solids). To date, regulations were based on slurry-digested sludges. Interested in examining land application of high solids cakes, is its low mobility over the slurry-digested sludges. This could represent considerable savings for municipalities and would also make beneficial reuse of materials that are now often lost to landfills.

In this study the availability of contaminants in frozen biosolids cake was evaluated in batch tests. In addition, the potential for infiltration of contaminants into the subsurface during thawing of frozen soils as assessed in term of solute mobility. Chloride, fluoride, bromide, nitrate-N, nitrite-N, ammonia, sulfate, o-phosphate, and COD mobility were quantified. Pathogen indicator tests were also conducted. The study was divided into two parts. In the first part, a series of batch tests was performed to evaluate the mobility of contaminant from biosolids cake under frozen and thawed conditions. In the second part, column tests were performed to evaluate losses and infiltration into thawing soils and the potential for contaminant transport under these conditions.

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to my supervisor Dr. W. Parker for providing me with all the professional and personal support to complete my thesis work, giving me the freedom to work independently, helping whenever asked.

I would like to thank Dr. P. Van Geel for all his helpful guidance, many thanks to Dr. Christopher Burn, Department of Geography and Environmental Studies, for his help and guidance. Thanks for Dr. Prof. M.D. Gray, Saskatchewan University, for sending me the requested papers. Many thanks to Dr. Adrian Unc on his advice for the use of ceramic cup samplers. I would like also to thank all the technician stuff, Mrs. Richardson and Mrs. Kennedy, Mr. Conley, Mr. McMartin, and Mr. Trudel for their help during my experiments.

My special gratitude goes to my parents, my wife, and my friends. With my thesis behind me, hopefully, I will be able to fulfill the role of husband and father that I always wished to be.

TABLE OF CONTENTS

1. INTRODUCTION	1
1.1 OVERVIEW	1
1.2 OBJECTIVE	3
2. BACKGROUND	5
2.1 LAND APPLICATION OF BIOSOLIDS	5
2.1.1 BENEFICIAL USES	5
2.1.2 PREVIOUS RESEARCH AND STUDIES	6
2.1.2.1 SLURRY VERSUS DEWATERED BIOSOLIDS APPLICATION	6
2.1.2.2 NUTRIENT TRANSPORT	7
2.1.2.3 HEAVY METAL TRANSPORT	9
2.1.2.4 pH EFFECT ON HEAVY METALS TRANSPORT	10
2.1.2.5 WINTER APPLICATION OF BIOSOLIDS	11
2.1.3 ENVIRONMENTAL IMPACTS	14
2.2 REGULATIONS	15
2.2.1 OVERVIEW	15
2.2.2 RELATED	16
2.3 BIOSOLIDS DEWATERING	16
2.3.1 DEWATERING METHODS	16
2.3.2 DEWATERING CHALLENGES	17
2.4 NITROGEN LOSSES FROM BIOSOLIDS	18
2.4.1 EFFECT OF pH ON NITROGEN LOSSES	20
2.4.2 AMMONIA VOLATILIZATION	21
2.5 PATHOGENS IN BIOSOLIDS	21
2.5.1 EFFECT OF SOIL CHARACTERISTICS ON BACTERIAL TRANSPORT	23
2.5.2 EFFECT OF MOISTURE PROPERTIES ON BACTERIAL TRANSPORT	24
2.5.3 EFFECT OF TEMPERATURES ON BACTERIA	25
2.6 SNOWPACK	27
2.6.1 OVERVIEW	27
2.6.2 CHARACTERISTICS OF THE SNOWPACK	28
2.7 INFILTRATION	29
2.7.1 MELTWATER INFILTRATION	29
2.7.2 INFILTRATION INTO FROZEN SOIL	30
2.7.3 TEMPERATURE EFFECT ON INFILTRATION	33
2.7.4 MOISTURE CONTENT EFFECT ON INFILTRATION	34
2.7.5 INFILTRATION MATHEMATICAL MODELS	35
2.8 DISCUSSION AND CONCLUSION	36
2.9 CONSIDERATIONS FOR WINTER SPREADING OF BIOSOLIDS	38

3. EQUIPMENTS, MATERIALS, AND METHODOLOGY	41
3.1 BIOSOLIDS BATCH TEST	41
3.2 COLUMN EXPERIMENT	42
3.2.1 COLUMN CONSTRUCTION	42
3.2.2 COLUMN PREPARATION	44
3.3 MATERIALS, EQUIPEMENTS, AND METHOD RESULTS	50
3.3.1 REVIEW OF SNOW WATER EQUIVALENT CALCULATION	50
3.3.2 SNOW WATER EQUIVALENT CALCULATION	52
3.3.3 SNOW WATER EQUIVALENT RESULTS	53
3.4 BIOSOLIDS	53
3.4.1 BIOSOLIDS RESULTS	54
3.5 REVIEW OF SOIL COMPOSITION	56
3.5.1 SOIL IN COLUMNS	57
3.5.2 SOIL CLASSIFICATION METHODS	58
3.5.3 METHOD USED FOR GRAIN SIZE DISTRIBUTION	58
3.5.4 GRAIN SIZE DISTRIBUTION RESULTS	59
3.5.5 BULK DENSITY AND UNIT WEIGHT	60
3.5.6 BULK DENSITY AND UNIT WEIGHT RESULTS	60
3.5.7 PARTICLE DENSITY AND POROSITY	61
3.5.8 PARTICLE DENSITY AND POROSITY RESULTS	62
3.6 SOIL WATER SAMPLING AND SUCTION LYSIMETERS	63
3.6.1 METHODS FOR SAMPLING THE SOIL SOLUTIONS	65
3.7 METHOD OF TEMPERATURE MEASUREMENT	65
3.8 SOIL SATURATION AND TIME DOMAIN REFLECTOMETRY	66
3.8.1 METHOD USED FOR WATER MOVEMENT AND SATURATION MEASUREMENT	68
3.8.2 CALIBRATION OF TDR	69
3.8.3 TDR CALIBRATION RESULTS	70
3.9 REVIEW OF ION CHROMATOGRAPHY	73
3.9.1 DETERMINATION OF ANIONS BY ION CHROMATOGRAPHY	74
3.9.2 CALIBRATION OF ION CHROMATOGRAPHY	74
3.9.3 ION CHROMATOGRAPHY CALIBRATION CURVE RESULTS	75
3.10 AMMONIA MEASUREMENT	75
3.10.1 CALIBRATION OF AMMONIA	76
3.10.2 AMMONIA CALIBRATION RESULTS	76
3.11 CHEMICAL OXYGEN DEMAND (COD)	77
3.11.1 CALIBRATION OF COD	77
3.11.2 COD CALIBRATION RESULTS	77
3.12 REVIEW OF BACTERIOLOGICAL ANALYSIS	78
3.12.1 EXPERIMENTAL METHOD FOR BACTERIOLOGICAL ANALYSIS	79

4. BIOSOLIDS BATCH TEST	81
4.1 RESULTS AND DISCUSSION FOR THE BATCH TEST	81
4.1.1 QUALITATIVE OBSERVATIONS	81
4.1.2 ANION CONCENTRATIONS	82
4.1.3 AMMONIA CONCENTRATIONS	89
4.1.4 pH RESULTS	92
4.1.5 COD RESULTS	92
4.1.6 BACTERIOLOGICAL ANALYSIS	95
4.2 CONCLUSION FROM THE BATCH TEST	95
5. COLUMNS TEST	97
5.1 HYDROLOGICAL CONDITIONS	97
5.1.1 RESULTS OF TEMPERATURES AND MOISTURE CONTENTS	97
5.1.2 RESULTS OF WATER BUDGET	106
5.1.3 CONCLUSION	107
5.2 BIOSOLID SOLUTE LOSSES AND TRANSPORT	108
5.2.1 RESULTS OF THE COLUMN EFFLUENT CONCENTRATIONS	108
5.2.2 RESULTS OF MASS BALANCE CALCULATIONS	126
5.2.3 GENERAL DISCUSSION FOR BIOSOLIDS LOSSES AND TRANSPORT	134
5.3 ENVIRONMENTAL IMPACT	136
5.4 PATHOGEN INDICATORS	137
6. GENERAL CONCLUSION AND RECOMMENDATIONS	140
7. SUGGESTIONS AND FUTURE WORK	145
REFERENCES	148
APPENDIX A: RESULTS FOR CHAPTER 3 METHOD PREPARATION	159
APPENDIX B: RESULTS FOR CHAPTER 4 BATCH TESTS	169
APPENDIX C: RESULTS FOR CHAPTER 5 COLUMN TESTS	197

SYMBOLS AND NOMENCLATURE

ASTM	: American Standard Test Method
A	: Area
Al	: Aluminium
Br ⁻	: Bromide
C	: Carbon
<i>C</i>	: Velocity
CEC	: Cation Exchange Capacity
Cd	: Cadmium
Cl ⁻	: Chloride
COD	: Chemical Oxygen Demand
Cr	: Chromium
Cu	: Copper
EPA	: Environmental Protection Agency
F/T	: Freeze - Thaw
F ⁻	: Fluoride
FC	: Fecal Coliform
Fe	: Iron
HPO ₄ ³⁻	: Ortho-Phosphate
K	: Potassium
K ₂ Cr ₂ O ₇	: Dichromate Potassium
K _d	: Dielectric conductivity
L	: Length
<i>L</i>	: Line Length
<i>L_w</i>	: Measured Length
MF	: Membrane Filter
Mu	: Molybdenum
N	: Nitrogen
Na ₂ CO ₃	: Sodium Carbonate
NaHCO ₃	: Sodium Hydrogen Carbonate
NaOH	: Sulphuric Acid

NH_3	: Ammonia
$\text{NH}_3\text{-N}$	: Ammonia Nitrogen
NH_4Cl	: Ammonium Chloride
$\text{NH}_4\text{-N}$	: Ammonium Nitrogen
NH_4NO_3	: Ammonium Nitrate
Ni	: Nickel
$\text{NO}_2^- \text{-N}$	: Nitrite Nitrogen
NO_3^-	: Nitrate
$\text{NO}_3^- \text{-N}$	: Nitrate Nitrogen
ON	: Organic Nitrogen
P	: Phosphorus
pH	: Hydrogen Power
POTW	: Publicly-Owned Treatment Works
Q	: Fluid Volume
ROPEC	: Robert O. Pickard Environmental Centre
S_0	: Surface Saturation Moisture Content
SD	: Standard Deviation
S_1	: Average Soil Saturation
SO_4^{2-}	: Sulfate
S_p	: Soil Surface Saturation
SWE	: Snow Water Equivalent
t	: Time
t	: One Way Travel Time
t_0	: Infiltration Opportunity Time
T_1	: Average Temperature
TC	: Total Coliform
TDR	: Time Domain Reflectometry
TNTC	: Too Numbers To Count
TKN	: Total kejdehal Nitrogen
USCS	: Unified Soil Classification System
USEPA	: United State Environmental Protection Agency
V	: Velocity
V_a	: Volume of Air
V_p	: Propagation Velocity
V_s	: Volume of Soil Particles

V_t	:	Total Volume
V_v	:	
V_w	:	Volume of Water
W_{dish}	:	Weight of Dish
WMO	:	World Meteorological Organization
W_{sample}	:	Weight of Wet Sample and Dish
W_{total}	:	Weight of Dried Residue and Dish
Zn	:	Zinc
D_{eff}	:	Effective Diffusivity
ε	:	Porosity of Media
η	:	Porosity
θ	:	Volumetric Soil Moisture
ρ_w	:	Water Density
ω	:	Water Content
e	:	Void Ratio

LIST OF FIGURES

Figure	Page
2.1 Infiltration into Unlimited, Limited and Restricted Frozen Soils.	32
2.2 Infiltration plotted against the premelt moisture content of 30 cm soil layer of completely frozen soils.	35
3.1 Biosolids batch test processes.	41
3.2 Schematic presentation for column construction.	47
3.3 Photograph of the columns.	48
3.4 Photograph - Inserting the ceramic cup sampler into the sampling port.	48
3.5 Photograph - Snow water equivalent head for 3 days interval.	49
3.6 Photograph – Tubing and collection system for the infiltrated SWE.	49
3.7 Mean temperature index and daily snowmelt equations	51
3.8 Volumetric distributions of soil components.	56
3.9 Sieve soil analysis for the sandy Soil A and the mixed Soil B.	60
3.10 Schematic of the vacuum lysimeter.	64
3.11 Method of acquiring a waveform from the 1502 Tektronix TDR.	69
3.12 Percent saturation calibration curve for unfrozen Soil A.	71
3.13 Percent saturation calibration curve for frozen Soil A.	71
3.14 Percent saturation calibration curve for unfrozen Soil B.	72
3.15 Percent saturation calibration curve for frozen Soil B	72
3.16 Ammonia calibration curve.	76
3.17 COD calibration curve.	77
4.1 Average concentrations for fresh versus F/T samples with respect to time.	85
5.1 Temperature profile versus depth and time for the thawing Soil A and B	98
5.2 Moisture content for Soil A versus time and depth for frozen and thawed column A, B, and C.	100
5.3 Moisture content for Soil B versus time and depth for frozen and thawed column D, E, and F.	101
5.4 Gravity drainage equilibrium for different layer soil.	105
5.5 Chloride concentration versus time and depth	111
5.6 Fluoride concentration versus time and depth.	113
5.7 Bromide concentration versus time and depth.	114
5.8 Nitrite-N concentration versus time and depth.	116
5.9 Nitrate-N concentration versus time and depth.	117
5.10 Ammonia concentration versus time and depth.	119
5.11 Phosphate concentration versus time and depth.	122
5.12 Sulfate concentration versus time and depth.	123
5.13 COD concentration versus time and depth.	124
5.14 Total coliform count from Port 3 for Columns A, B, C, D, E, and F.	138

LIST OF TABLES

Table	Page
2.1 Reduction of pathogenic and indicator micro organisms in two different sludges by freeze-thaw conditioning.	26
3.1 Time and amount (mL) of equivalent snow water application.	46
3.2 Temperature and precipitation climate normal for 1971-2002.	52
3.3 Physical and chemical characteristics for biosolids.	54
3.4 Total Solids for January sampling.	55
3.5 Total Solids for February sampling.	55
3.6 Total Solids for March sampling.	55
3.7 Bulk Density for Soil A and B.	61
3.8 Particle density and Porosity for Soil A and B.	63
3.9 Dielectric constant of fluids and soils materials.	70
4.1 Anion concentrations in extracts after 1 day.	83
4.2 Anion concentrations in extracts after 14 days.	84
4.3 Anion concentrations in extracts after 28 days.	84
4.4 T-test at the 0.05 critical alpha levels for anions.	86
4.5 Fresh and frozen samples for ammonia concentrations based on 25 g calculations.	90
4.6 T-test at the 0.05 critical alpha levels for ammonia.	91
4.7 pH values for 25 g/100 mL biosolids samples.	92
4.8 Fresh and frozen COD concentrations based on 25 g/10 mL calculations.	93
4.9 T-test at the 0.05 critical alpha levels for COD.	94
5.1 Concentration of measured parameters before and after simulating snow water equivalent for the biosolids cake applied on the columns.	127
5.2 Mass Balance calculation for 49 g biosolids application, Col. B and E.	130
5.3 Mass Balance calculation for 1000 g biosolids application, Col. C and F.	131
5.4 Maximum solutes concentration compared to MCL.	136

1 INTRODUCTION

1.1 OVERVIEW

United States and Canada generated approximately 7.1 million tons of biosolids for use or disposal in 2000. This amount of biosolids generated from the wastewater treatment plants is expected to grow to 7.6 million tons in 2005 and to 8.2 million tons in 2010. It is anticipated that the percentage of biosolids directed to beneficial application will grow from 66 percent in 2005 and to 70 percent in 2010 (USEPA, 1997).

A major beneficial use of sewage biosolids has been the application to land as fertilizer. This has been widely practiced in North America, and Europe for many decades. Over the years, regulations and procedures have been developed to manage the application to protect human and animal health, and the environment. These regulations aim to maximize the benefits of biosolids land application, such as fertilization and soil conditioning while minimizing the risks associated with pathogen and nutrient contamination. Although many scientific studies have been performed to support the development and confirm the effectiveness of the application procedures, the public still has concerns that land application may be unsafe because it involves human and industrial waste.

In recent years, there has been an increase in the number of regulations regarding the beneficial use of biosolids. This has raised public awareness about the potential detrimental health and environmental effects associated with sewage biosolids, and interest in the impact and risks of biosolids land application. Studies have focused on the

following main issues: Surface and groundwater pollution, fate and effects of pathogens, fate and effects of heavy metals, and fate and effects of contaminants.

The land application of biosolids in northern climates is often restricted by cold weather. Here, we are concerned with the mobility of contaminants and nutrients that are released from frozen sewage biosolids if placed on the surface of frozen soil and subjected to winter weather conditions. It is long recognized that freezing the sewage sludge changes its physical properties (Vesilind and Martel, 1990). Sewage biosolids contain water that, when frozen, form ice crystals which do not allow impurities to be incorporated. These impurities migrate to the outside of the crystal and are loosely bound in this position (Vesilind and Martel, 1990).

To date, regulations have been based on biosolids slurries rather than high-solids cakes. In examining land application of high solids cakes, we are interested in contaminant mobility as compared to the slurry-digested sludges. Little is currently known about the chemicals and nitrogen availability of high solids cakes. Generally, most of the nitrogen present is in organic form where there is less likelihood of losses occurring. It is believed that the digested liquids that carry the dissolved chemicals in the high-solids cakes are generally in the range of 70% moisture content by weight. According to Gendebien et al., (1999) dewatered biosolids generally have lower ammonium-N contents than digested liquids due to losses occurring during the dewatering process. The same trend was reported for the available phosphorus.

When placed on a soil surface, biosolids cake, due to its sponge-like characteristics, works as mulch. Hence, it absorbs the impacts of meltwater drops and reduces the volume of surface runoff. In this condition, the movement of the losses from the

biosolids, due to rain or meltwater, will depend on soil physical and chemical properties, temperature, pore saturation, and moisture content of the soil.

In this study, winter application of high solids cake, subjected to winter weather conditions, was evaluated to ensure that the safety of biosolids beneficial use in terms of solute mobility is scientifically confirmed.

1.2 OBJECTIVE

This study focuses on the infiltration of contaminants upon thawing, into frozen soils that are released from the application of biosolids cake before the accumulation of a snowpack.

This thesis addresses the following questions regarding contaminant losses from the biosolids and infiltration into thawing soils. Can biosolids be safely applied for winter land application as for summer application? To what extent does temperature affect the mobility of the biosolids constituents from the cake? Can biosolids losses infiltrate into frozen soil (rather than running off) thus minimizing the detrimental effect of surface water contamination? To what extent does soil type, soil-moisture content and soil-ice content influence contaminant infiltration into soils from the winter application? Are frozen soils resistant to contaminant penetration? What biosolids management practices should be employed to minimize the amount of groundwater contamination?

To answer these questions, the study was divided into two parts. In the first part, a series of batch tests were performed to evaluate the availability of contaminants from biosolids cake under frozen and thawed conditions. In the second part, column tests were

performed to evaluate losses and infiltration into thawing soils and the potential for contaminant transport under these conditions.

In this study, a literature review is provided in Chapter 2. The literature review introduces and provides background on key concepts of land application. The impact of different parameters such as soil characteristics, moisture content, and temperature on the process of land application are also discussed in this chapter. The experimental testing program and methodology is presented in Chapter 3. This provides details on how the research and experiments were conducted. Chapters 4 and 5 present the test results and discussions for the biosolids batch tests and column tests, respectively. The research findings and conclusions are presented in Chapter 6. Future work and recommendations from this study are presented in Chapter 7. All measurements and calculations are attached in the Appendices, which have been placed in the back of this thesis.

2 BACKGROUND

2.1 LAND APPLICATION OF BIOSOLIDS

2.1.1 BENEFICIAL USES

Biosolids are primarily organic, accumulated solids that are separated from wastewater, and stabilized by treatment. The high organic content makes them effective fertilizers and good soil conditioners (USEPA, 1994). When applied to land, biosolids supply nutrients essential for plant growth, including nitrogen and phosphorous, as well as some essential micronutrients such as nickel, zinc, and copper. Biosolids cake can be spread on the soil surface or incorporated into soil. This beneficial practice is known as ultimate disposal by land application (USEPA, 1994).

Biosolids are applied to agricultural land, forests, rangelands, or on disturbed land in need of reclamation. This practice improves soil properties, such as structure and water holding capacity, which make conditions more favorable for root growth and increases the drought tolerance of vegetation (McFarland, 2000). Many investigations have shown that organic waste or biosolids, when applied to land, improve soil physical properties (Kladivko and Nelson, 1979; Guidi et al., 1983; Hall and Coker, 1983; Gendebien et al., 1999). As different qualities and forms of biosolids (such as liquid, dewatered sludge cake, in the form of aggregates, and dried and ground biosolids) are used in the above studies, the results are variable but the general trends are useful for similar treatments and management processes.

Biosolids can also serve as an alternative or partial substitute for expensive chemical fertilizers. Many researchers have reported that biosolids-treated-soil produced crop yields equivalent to or higher than those when commercial fertilizers alone were applied (Hinsley, 1976; Lamb, 1972). The nutrients in the biosolids offer several advantages over those in inorganic fertilizers because they are organic and are released more slowly to growing plants. These organic forms of nutrients are less water-soluble and, therefore, less likely to leach into groundwater or run off into surface waters.

2.1.2 PREVIOUS RESEARCH AND STUDIES

Land application of biosolids generated by certain wastewater treatment plants is performed in an environmentally safe and cost effective manner in many communities. Some studies have assessed the effect and influence of application rate of the nitrogen, phosphorus, heavy metals, and sediment content, on the quantity and the quality of runoff water and on infiltration from agriculture lands (USEPA, 1997).

2.1.2.1 SLURRY VERSUS DEWATERED BIOSOLIDS APPLICATION

Kelling (1977) found low runoff and sediment losses from areas treated with surface application of liquid biosolids relative to untreated areas. However, runoff phosphorus and nitrogen loads from treated plots increased compared to the control plots. Joshua (1996) investigated runoff and downward movement from a long-term of surface application of dewatered sludge. The application reduced runoff and increased surface retention of rainfall. Dunnigan and Dick (1980) found that the nitrogen and phosphorus

content of runoff were higher when the biosolids were surface applied as compared to when they were incorporated into the soil.

In many cases, surface application of liquid biosolids retarded infiltration but infiltration rate recovered or improved after the biosolids had dried or were incorporated into the soil (Sabey, 1973). On the other hand, Epstein (1975) showed that the addition of biosolids to soil increased saturated hydraulic conductivity initially, but that it decreased to that of the original soil after 50-80 days.

A study using a rainfall simulator showed that the surface application of dewatered biosolids increased soil infiltration and reduced runoff, sediment, and nutrient losses. This effect was more realized in a no-till treatment than when the biosolids were incorporated by traditional tillage (Burggeman and Mostaghimi, 1993).

In another study, it was demonstrated that a one time surface application of dewatered biosolids on semiarid rangelands increased soil infiltrability and decreased soil erosion. However, it was not clear if the increases in infiltration rate and the decreases in soil erosion were due to biosolids cover alone or due to changes of the soil properties (Aguilar and Loftin, 1991).

2.1.2.2 NUTRIENT TRANSPORT

Cooke et al. (2001) reported in their investigations of the movement of nitrate and phosphate through experimental soil columns, that the $\text{NH}_3\text{-N}$ that was leached from the sand soil were small; 0.06% for fresh and dried, and 0.63% for composted mesophillic anaerobic digested biosolids. $\text{NH}_3\text{-N}$ from a sandy loam soil were greater; 4% for fresh and dried, and 3% for the composted biosolids. Phosphate that was leached from the

sand soil were small; 0.02% for fresh and dried, and 0.002% for composted mesophilic anaerobic digested biosolids. Phosphate showed similar values for a sandy loam soil; 0.02 % for fresh and dried, and 0.02% for the composted biosolids. It was concluded that the extent of soil contamination is governed by the treatment processes of the sludge and the soil type.

Phosphorus retention mechanisms in many soils are considered to result in a low risk of phosphate leaching due to sorption onto soil colloids (James and Anderson, 1996). However, leaching is a concern in some sandy soils, especially at low pH due to increased phosphorus solubility. Some other studies reported that if phosphorus exceeds a critical level, loss may occur. For example, from the surface application of sewage sludges Heckrath et al. (1995) reported that the critical limit for sandy Broadbalk plots in England was about 60 mg available P per Kg soil, and any additional load could have detrimental effects on groundwater.

Hinsley (1976) reported that the surface application of 1.764 kg $\text{NH}_4\text{-N/ha}$ in a liquid sludge resulted in nitrate–nitrogen concentrations in soil leachates of more than 100 mg/L. These excessive nitrate-nitrogen levels in the leachate were expected because the plant available nitrogen added was ten to twenty times that required by the crops grown. This study concluded that a sludge application rate of 15 metric tones/ha would minimize the amounts of nitrate leached into the subsurface.

Soon and Bates (1978) concluded from their study that compared nitrate levels in soils treated using NH_4NO_3 and anaerobic digested sludge. They showed that minimal nitrate pollution of groundwater will occur if the amount of nitrogen applied in sludge was almost equivalent with the nitrogen required for the crop grown. Based on this study,

Steward and Beauchemin (1975) suggested that the sludge surface application rate should be based on the need for the specific crops, because the addition of sludge did not increase crop yield, but increased risk of nitrate nitrogen leaching into the groundwater.

2.1.2.3 HEAVY METAL TRANSPORT

Yangming and Corey (1993) studied the vertical and horizontal redistribution of Cd, Cu, and Zn in a silt loam plot where sludge had been surface applied. After eleven years, the fractions of Cd, Cu, and Zn remaining in the top 20 cm were found to be 66.7%, 67.5% and 68 % respectively. Some of the heavy metals, from 15.5% to 21%, moved outside the plot, and from 11.6% to 15.2% were found in the deeper layer of the 20-30 cm. No significant accumulations of these metals were found below a depth of 30 cm. Also, in a study conducted on a watershed, Dowdy (1991) observed that the concentration of metals from the biosolids were low in the runoff and in the soil water solution.

Laboratory and field studies showed that Cd, Cu, and Zn released from the land application of biosolids are present in or near the layer of incorporation in the soil (Emmerich et al., 1982; Williams, 1987). After different studies on the movement of biosolids based on Cd, Cu, and Zn in a cultivated plot, Yangming and Corey (1993) concluded that those metals do not enter groundwater directly but remain below the incorporation layer in cultivated plots.

In a study conducted by Surampalti and Lin (1997) to evaluate the long-term surface application of sewage sludge, it was concluded that the metal concentrations and the bacteriological levels in sludge, soil, and groundwater were below permissible limits.

The groundwater nitrate-nitrogen concentration showed a range of 1.9 to 5.2 mg/L, however in some instances the $\text{NH}_3\text{-N}$ concentration was slightly above 10 mg/L due to the excessive nutrient loading. The evaluation concluded that biosolids application is environmentally safe and the rate of application should follow the agronomic nitrogen rate based on crop needs.

2.1.2.4 pH EFFECT ON HEAVY METALS TRANSPORT

It was observed from a study conducted by Chaney (1987), that movement of metals from sludge-amended soil is related to soil pH. Maintaining a soil pH of 6.5 or higher immobilized most cationic heavy metals in soils. Boswell (1975) reported the movement of Zn to a depth of 30 cm and Cd, Cr, Cu to 15 cm after sludge application of 415 metric tonnes/ hectare to the soil. The soil pH dropped from 6.2 to 5 during the 2-year study and it was found that the heavy metals disappeared from the incorporation soil layer.

Hinsley (1971) reported the movements of Zn, Cd, Cr, Cu, and Ni to a depth of 75 to 115 cm through an agricultural soil after surface application of 336 metric tonnes/ hectare over four years. The soil pH dropped from 5.6 to 4.9 after two years. It was concluded that the lower pH values could have increased the solubility of the metals and contributed to heavy metal movement.

2.1.2.5 WINTER APPLICATION OF BIOSOLIDS

In Michigan, an investigation of a surface winter land application of secondary sludge onto frozen soil was conducted by O'Neil and Sharon (1995) to determine

nitrogen and phosphorus loading to surface water and ground water supplies during the thaw period. Runoff was not detected neither on the test plot nor the control plot. They suggested that precipitation either evaporates or infiltrates through the soils. Infiltration rates for different test locations were 0.99, 0.38, and 1.19 cm /min for the control plot and 0.66, 0.91, and 0.79 cm/min for the test plot. Also, samples of water collected from the vadose zone around the site and under windrows (storage area) did not show any increase in the concentration of total nitrogen in the groundwater. When the samples collected from the tested plot were compared to samples from control plots, it was concluded that winter land application is not detrimental to the environment. It can reduce storage facilities and the disposal of biosolids is an effective method for fields with the appropriate slope, soil and vegetative cover (O'Neil and Sharon, 1995).

In western Washington state, Sullivan et al. (2000) evaluated the impacts of soil hydrology on the winter spreading of biosolids onto unfrozen soils. Soil hydrologic groups were divided into four groups, two sandy soils with high leaching potential and very low runoff potential and moderate leaching potential with low runoff. The other two groups were clayey soil with low leaching potential, moderate and high runoff. They concluded from the nutrient loss results, that the sandy soils were best suited for the winter application due to their high permeability and infiltration opportunity.

Only a few studies reported on winter land application of biosolids, especially when soils are frozen. However, some winter application of manures have been performed. These studies are reported in the following paragraphs. Manure differs from biosolids in its chemical and physical composition, and treatment processes. However, the ultimate beneficial disposal is the same target as for biosolids. The behavior of

nutrients, and contaminant movement after release onto frozen soil and while thawing, is quite similar for the application of biosolids and manure, and can contribute to the same environmental effects.

Midgley and Dunklee (1945) in their study in Vermont, spread manure onto frozen, snow-covered ground in late December and early January. The field investigations were done at three different sites, having slopes of 8%, 10%, and 20% in different locations. They found that all frozen soils were impervious to water, and runoff occurred when the soil was frozen. The steepness of slope had relatively little impact on runoff losses. Spreading manure on frozen ground resulted in large losses of nitrogen in the runoff. In addition to the runoff losses, volatilization of ammonia contributed to large nitrogen losses from the manure.

In Wisconsin, Hensler et al. (1970) found that runoff losses from manure surface applied to frozen onto field-scale plots (silt loam soil) were variable. During the first year of observation, large losses of nitrogen and phosphorus were observed in the runoff. These losses were caused by a 2 cm rain that fell within 24 hours after manure application. In the following year, there was very little precipitation throughout the winter months. This resulted in minimal nutrient losses in runoff. Over the two-year study, average runoff losses of N, P, and K were 10%, 6%, and 8%, respectively.

In an other study in Wisconsin, Converse et al. (1976) compared in their study the nutrient runoff from the fall, winter and spring treatments of solid dairy manure on ten different plots (silt loam soil). No significant differences in nutrient losses were found between the three treatments over the three-year study period. However, they found that the amount of nutrients lost varies directly with the volume of runoff. They found that

runoff volume for treated winter plot and spring plots was larger than for fall plots, and the control plots had more runoff volume than the ones treated with manure. It was concluded that the differences were due to variations in infiltration rates. The infiltration rates appeared to be influenced by the number of earthworms present and by grass and mulch cover.

In New York, Klausner et al. (1976) investigated the effect of surface application rate and weather conditions in determining the amount of nutrients lost in runoff from winter-applied manure. Dairy manure was applied on frozen ground (silt loam soil) with three different spreading rates for three consecutive winters. The study demonstrated that excessive nutrient losses were detected when manure spreading happened during the thaw time. No significant nutrient losses were seen when manure was applied at 35 tones /hectare and covered with snow, which melted at a later time. These minimal losses were comparable to the nutrient losses of plots receiving no manure.

Young and Holt (1977) performed an experiment, in Minnesota, by surface applying solid dairy manure. They confirmed that winter application of manure could significantly reduce soil loss and showed reduction in runoff and nutrient loss reduction on plowed ground when compared to untreated manure plots. Soil loss decreased because the manure worked as mulch on the soil surface and absorbed the impact of raindrops, reducing the volume of the surface runoff. This study found that total nutrient and runoff losses were consistently less in the manured corn compared to the non-manured corn.

Steenhuis et al. (1981) found in laboratory and field experiments in Wisconsin that solid dairy manure spread on frozen ground without snow cover, did not contribute to loss of nitrogen because not all frozen soils are impermeable. They found that

temperature affected the soil permeability and thus the extent of the pores blocked by ice. Therefore, under some conditions, winter application of manure onto frozen ground may not cause more risk of contamination than fall application. It was also found that the first meltwater had the highest concentration of nitrogen. It is this first meltwater after spreading that largely determines the fate of manure nitrogen. If the water infiltrates, there will be very little loss of N. However, if the water runs off, the losses will be high.

In a laboratory experiment study performed in Alberta, it was found that the pollution potential of snowmelt runoff from composted manure applied on top of snow was significantly lower than the pollution potential from fresh manure (Qu et al., 1996). Also, in Ontario at Guelph University, Blais and Weil (1999) assessed the water quality after liquid manure applications on a clay soil. Manure was applied in the late fall on frozen ground, and in the spring on unfrozen ground. They concluded for these kinds of soil and from the weather conditions that neither late fall application nor spring application contribute to significant nitrogen contamination of surface runoff or subsurface drainage water.

2.1.3 ENVIRONMENTAL IMPACTS

Despite many positive impacts on the environment, land application can have negative impacts on water, soil, and air if not practiced correctly. The excessive applications of nitrogen, phosphorus, organic materials, accumulation of heavy metals, trace organic chemicals, and pathogenic microorganisms are major concerns in the land application of biosolids. These include possible surface runoff and groundwater contamination by rainfall or snowmelt. The importance of groundwater is that it is

considered one of the main sources of drinking water. When nitrate-nitrogen, for example, is ingested in high amounts by humans and animals, it could contribute to potential adverse ecologic or health effects. These effects are reported to include eutrophication, methemoglobinemia, cancer, and possibly others (Kenny, 1989).

2.2 REGULATIONS

2.2.1 OVERVIEW

Regulations defining land application of biosolids are slowly being developed in the European Union, United Kingdom and Canada, and are largely based on USEPA standards. The land application of biosolids in Ontario is regulated by the Environmental Protection Act, specifically Regulation 347, which is based on the regulations developed in the United States.

The US Environmental Protection Agency (USEPA) has a comprehensive regulation, commonly called the EPA Part 503 rule (EPA 40 CFR Part 503 and Chapter 173-308 WAC), to sustain the beneficial use of biosolids while protecting public health and the environment from other effects of harmful materials potentially present in biosolids. It sets limits for trace element concentrations in biosolids.

Canadian biosolids land application programs often cite the EPA Part 503 rule because of the extensive research on which it is based. The Ontario regulation covers a wider range of organic matter than the US EPA 503 rule. The EPA Part 503 rule is based on assessment of any risks that associate with the use of biosolids over a 10-year period.

2.2.2 RELATED REGULATIONS

The regulations relevant to late fall and winter application of biosolids are EPA 40 CFR Part 503 and Environmental Protection Act, regulation 347 in the US and Ontario respectively. Both regulations agree that for fields with a sustained slope of 3% or less, spreading may be allowed on partially saturated frozen soil. In such cases, the separation distances from surface watercourses should be doubled. Where surface runoff is expected as a result of snowmelt, biosolids may be applied at a maximum rate of 8 Mg/hectare per 5-year period, and a maximum nitrogen application rate of 135 kg/hectare for the same period of time. This application rate is regulated not only for winter application but for the other seasons, too.

2.3 BIOSOLIDS DEWATERING

Dewatering is the separation of water from solids to achieve a substantial reduction in volume and an increase in solids concentration. The removal of water from liquid residuals could achieve an end product with 10 to 40 percent total solids (Metcalf and Eddy, 1991).

2.3.1 DEWATERING METHODS

Typically, there are three types of dewatering processes: mechanical, natural, and thermal. Mechanical dewatering equipment includes screws, centrifuges, and belt presses; natural dewatering is usually accomplished in lagoons and on sludge drying beds.

Belt filters and plate and frame filter presses both operate by drawing the fluid to be dewatered through a filter fabric. Belt filters utilize permeable belts to compress and

shear the sludge to remove water. Plate and frame filter presses force the sludge into contact with a series of cavities lined with filter cloths that retain the solid matter while allowing the liquid filtrate to pass through them. Belt filter presses can achieve 20 to 32 percent solids content.

Centrifuges spin biosolids in a horizontal, cylindrical vessel at high speeds, with solids concentrating on the outside of the vessel. These solids are then collected. Centrifuging can result in 25 to 35 percent solids content.

Lagooning is another popular dewatering method. It owes its popularity to its simplicity and relatively low cost. A lagoon is a basin into which raw sludge is deposited. The sludge is dewatered naturally by evaporation induced by the sun and wind until it is free of moisture. The dry sludge is then removed and disposed of in a dedicated area or a sanitary landfill.

Sludge drying beds are similar to lagoons. A drying bed is built by layering sand. Sludge is placed on top of the sand in 8-12 inch layers and allowed to dry naturally. The sand acts as a sieve to strain the water away from the drying sludge. This process can produce solids content in primary sludge biosolids reaching 45 to 90 percent.

2.3.2 DEWATERING CHALLENGES

The choice of the right process of biosolids dewatering should be made on the basis of the desired quality for the selected mode of ultimate disposal. Various biosolids parameters have major influence on the chosen process and are regulated in any disposal permit. These include: Total solids content (TS), pathogen content, concentration of

hazardous organic compounds, and availability of suitable land for ultimate disposal (Metcalf and Eddy, 1991).

The concentration of nutrients and contaminants in biosolids vary over time and between different treatment plants, depending on the treatment process and the wastewater source (Smith, 1996). The form in which the biosolids are applied to the agricultural land also varies; including digested fresh solids, in liquid or solids forms, thermally treated, and chemically treated. Thus, the amount and form of nutrients present in different types of biosolids will vary, and as consequence may influence nutrient losses. For example, the greater the percentage of nitrogen present in the form of $\text{NH}_4\text{-N}$, the greater the potential for nitrate leaching (Shepherd, 1996; Smith and Wood, 1998). According to Gendebien et al. (1999), dewatered biosolids generally have lower $\text{NH}_4\text{-N}$ contents than in digested liquids due to losses occurring during dewatering processes. The same trend was reported for the available P, with dewatered biosolids having a range of P availability of 35-50% compared to inorganic P fertilizers, and the digested liquids are generally in the range of 70-100%.

2.4 NITROGEN LOSSES FROM BIOSOLIDS

Nitrogen (N) occurs in several chemical forms and oxidation states (Metcalf & Eddy, 1991). These forms vary greatly not only in their characteristics (from the proteins of solid organic matter to gaseous ammonia), but also in the way they behave. These forms are classified as either organic or inorganic N. Organic forms of N are found in compounds such as amino acids and protein. Inorganic forms include ammonium (NH_4^+), ammonia (NH_3), nitrate (NO_3^-), and nitrite (NO_2^-). Inorganic forms of N are the available

forms that plants and microorganisms can use or that can move in the soil as water moves through it. Most N in the soil is in the organic form. In biosolids, the majority of N added to the soil is in organic forms, and what is left (NH_4^+ and NO_3^-) is in inorganic forms (McFarland, 2000).

Many forms of N are present in the environment at any time, because N can change from one form to another. NO_3^- , NH_4^+ , and N_2 can be converted to organic N by plants and microorganisms; Organic N can be converted back to inorganic forms as the organic compounds decompose. N can also change between inorganic forms (for example, NH_3 to NH_4^+ or NO_3^- to N_2). These changes happen as nature attempts to establish equilibrium among the various forms as the environmental conditions change. N in the soil interacts with the atmosphere, soil particles, the water that surrounds or moves through the soil, microorganisms, and plants. If a new source of N is added to the soil, or changes in temperature and moisture conditions occur, N transformations can take place. Because environmental conditions are constantly changing, N transformations are constantly occurring. This continual movement of N from one form to another is known as the Nitrogen Cycle.

Most of the transformations and losses are caused by microbial action. All the processes are slowed down considerably when temperatures are low. For example, for each decrease of 10°C in the soil temperature, microbial action decreases by about half. At about 5°C , microbial action is fairly slow. Through much of the winter, the average soil temperature is at or below this temperature, therefore, mineralization, nitrification, and denitrification are very slow. The nutrients from the biosolids applications made

during the winter will essentially be stored in the ground and soil layers until temperatures increase.

Significant NO_3^- leaching will not occur from winter application because NO_3^- generally will not be formed. Only NO_3^- present in the soil at the time of application will be lost. Although this inactivity can also be the case in agricultural soils, winter sunshine can warm up the surface of the soil for some periods to permit some transformations. If there is no plant uptake, NO_3^- leaching most probably will occur.

2.4.1 EFFECT OF PH ON NITROGEN LOSSES

The possible loss of ammonia gas (NH_3) to the air depends on different equilibrium relationships in the soil. Ammonium NH_4^+ plays an important role in these relationships. Most of the NH_4^+ is bound to soil surface exchange sites, and what is left, is dissolved in soil solution that surround the soil particles. If some NH_4^+ is removed from the soil solution by the way of plant uptake or by other means, NH_4^+ will move from the soil surface sites into the soil solution to re-establish the equilibrium. The NH_4^+ in the soil solution also tries to maintain equilibrium with the NH_3 in the soil solution. If more NH_3 is needed to retain the equilibrium in the soil solution, NH_4^+ will transform to NH_3 . On the other hand, if more NH_4^+ is needed in the soil solution, NH_4^+ will move from the soil exchange sites. This equilibrium depends on the soil pH. At pH 6, only 0.1 percent of the total of these two is NH_3 and at pH 7, 1 percent is NH_3 , and at pH 9, the ratio is one to one (Sparks, 2003).

2.4.2 AMMONIA VOLATILIZATION

When a significant amount of NH_3 is present in the soil solution, some will volatilize into the air. When this happens, all the relationships try to re-establish equilibrium. More NH_4^+ transforms to NH_3 , and more NH_4^+ will disappear from the soil exchange sites. When the soil solution evaporates, the concentration of NH_3 increases and the possibility for loss increases. Most volatilization occurs within the first days of biosolids application. The amount of NH_3 that volatilizes depends on a number of environmental factors and biosolids management techniques. The important factors in addition to pH, are temperature and wind speed, and whether the biosolids are incorporated, injected, or surface applied. As much as 100 percent of the initial NH_3 and NH_4^+ in biosolids that are surface applied to agricultural soils can be lost by volatilization. Even if the biosolids have been mixed in with the soil, the majority of the NH_3 may volatilize if the soil has a high pH over 8.0.

2.5 PATHOGENS IN BIOSOLIDS

Sewage entering a wastewater treatment plant contains a wide variety of pathogenic organisms. The pathogens most present in biosolids are bacteria, viruses, protozoa and helminthes (Burge and Marsh, 1978). Once land applied, the major concern is the potential of these organisms to survive in soil for extended periods of time. Chemical and physical properties of the soil affect the survival of pathogens. During the wastewater treatment process, the following stabilization processes kill pathogens: high temperatures, chemical disinfectants, destruction of the microbial food source (volatile solids reduction) and drying of biosolids (McFarland, 2000). The risk to public health from the pathogens

on land to which biosolids are applied is dependent on the treatment process used, how the organism survives in the environment after application, and the exposure route or pathway to humans (McFarland, 2000).

Biosolids are treated by using different technologies to reduce pathogens to an acceptable level for the density counts of organisms. As it is difficult and expensive to measure pathogenic organisms within detection limits, indicator organisms are used to assess biosolids pathogenic content. If these indicators are present, other organisms may be present at lower levels. Salmonella, fecal coliforms and total coliforms are used as primary indicator organisms for pathogens reduction in biosolids due to their ability to resist and survive. Current USEPA fecal coliforms recommendations for body-contact recreation is less than 200 colonies/100 mL; for fishing and boating, less than 1000 colonies/ 100 mL; and for domestic water supply, for treatment, less than 2000 colonies/ 100 mL. The drinking water standard is less than 1 colony/ 100 ml (USEPA, 1997).

Pathogen survival and die off is influenced by many factors such as soil characteristics, temperature, pH, and moisture. Also, the presence of nutrients has a great impact on the pathogens survival. On the other hand, movement of bacteria to the subsurface may contribute to surface and groundwater contamination by bacterial leaching. When bacteria are applied to the soil through land application of biosolids, the rate and depth the bacteria reach is of great importance for biosolids application management.

2.5.1 EFFECT OF SOIL CHARACTERISTICS ON BACTERIAL TRANSPORT

Structure and particle size distribution affect the infiltration processes. Jang et al. (1983) showed that straining contributes to the removal of bacteria from leachate when the cell size of bacteria was bigger than the size of at least 5% of the soil particles. Peterson and Ward (1989) found that pore size also contribute to filtration removal and sedimentation of bacteria in pores, and consequently reducing permeability. Other studies performed by Abu-Ashour (1998) and Smith and Woods (1998) found that macropore flow represents a major pathway for bacterial contamination of effluent discharged from columns.

In 1998, Scott et al. found in their field study that preferential flow leads to a rapid transport of fecal coliform to tile lines at depth of 90 cm where dairy manure had been applied. Bacteria sorption is also affected by the different soil characteristics, which consequently affect the bacteria movement and transport. Bacteria adsorb more to the positive charge of organic matter attached on the mineral surface than to the negative surfaces (Scholl et al., 1990).

Crane and Moore (1986) found that a lower temperature increases bacterial survival time in the soil environment. However, Kibbey (1978) found that freezing and thawing conditions are detrimental and that extreme pHs are also detrimental to bacteria survival.

The fate of indicator bacteria after land application of anaerobically digested sewage sludge in an arid region was reported by Pepper et al. (1993). Not only the surface horizon was studied, but also the transport of these organisms in the unsaturated subsurface was also monitored. Immediately after application, the numbers of fecal

streptococci, total coliform, and fecal coliform were 6.1×10^6 , 1.3×10^8 , and $3.7 \times 10^8/\text{kg}$, respectively. After 5 weeks, fecal streptococci decreased to 9.6×10^3 , total coliforms increased to 4.6×10^8 , and the fecal coliform decreased to 4.6×10^4 . However, after these five weeks, soil temperatures decreased and was followed by a rainfall event, which increased the soil moisture and fecal coliform showed regrowth. After 84 days, total coliforms values increased to 9.8×10^6 , and fecal coliform to 4.5×10^6 . Fecal streptococci showed little regrowth. After seven months, the moisture was at its lowest value and none of the three bacteria indicators were detected, and it was concluded that cool and moist conditions in the field could help regrowth of these bacteria. In addition, the transport through the subsurface of fecal coliform indicator bacteria after land application of sewage sludge to cotton farms in Arizona was investigated. Soil core samples were taken in 50 cm intervals to a depth of 200 cm. None of the indicators were detected before the application. After the application, the study detected fecal coliforms at 200 cm on the day of sludge application. Seven weeks after, fecal coliforms were only detected from the top to a depth of 100 cm.

2.5.2 EFFECT OF MOISTURE PROPERTIES ON BACTERIAL TRANSPORT

As noted earlier, physical and moisture conditions such as soil water content, and temperature impact the bacterial transport. High moisture content contributes to bacterial leaching. McCoy (1979) found that bacteria generally move less than 1 meter under unsaturated conditions, and can move to 60 meters under saturated conditions. The effect of high and low pH has also been observed (Williams, 1987). It was founded that extreme

pH decreases bacterial survival. In a similar study by McFeters and Stuart (1972), die-off was minimized at a pH ranging from 6 to 7.

2.5.3 EFFECT OF TEMPERATURES ON BACTERIA

Above 40⁰ C, lower temperature is more favorable to bacterial survival than higher temperatures. Between 30⁰ C and 50⁰ C, die-off rate of fecal bacteria generally doubles with each 10⁰ C increase in temperature. Kibbey et al., (1978) found that survival rates of fecal bacteria increased with soil moisture content over different temperatures. In a column study, Kanwar (2002) found that the survival of fecal coliforms was at maximum when the soil was saturated. Freeze – thaw cycles were detrimental to bacterial survival, however, additional research is needed to clarify this effect (Kanwar, 2002).

Cold shock, freezing, and storage at low and subzero temperatures contribute to the death of bacteria. Studies have shown that cold shock can damage the cytoplasmic membranes and the DNA of bacteria, and can cause the deterioration of the outer membrane of gram-negative bacteria. Also, freezing and thawing has been shown to cause the same damage (MacLeod and Calcott, 1976; Mackey, 1984). When the cytoplasmic membrane is damaged, inorganic phosphate, amino acids and potassium and magnesium are released from the bacteria cell (Macleod and Calcott, 1976). Researchers have attributed microbial death and injury to one or both of these processes; cold shock and freeze/thaw. However, due to the surrounding medium and the species of bacteria, many of the cells injured by these processes can self-repair (Parker et al., 2000). Frozen storage also affects the death rate. Usually, the death of bacteria happens quickly, and initially the rates slow down with time until it remains constant (Mackey, 1984). Mazur

(1966) reported that the death rates are low or zero when storage is at temperatures of -70°C or below, and temperatures between -60°C and 0°C decrease the survival of most species with time. It was also reported that the decrease in survival depends on the species, the storage temperature, and the nature of the freezing medium.

Little research has reported the impact of freezing on sewage pathogenic microorganisms. Sanin et al. (1994) compared the survival of several microorganisms in frozen sludge (Table 2.1). They found that parasitic worms like ascaris eggs and single-celled organisms like fecal streptococci were the most resistant to the effect of freezing.

Table 2.1 Reduction of pathogenic and indicator microorganisms in two different sludges by freeze/thaw conditioning. (From Sanin et al.1994).

	Overall Log Reduction	
	Aerobically Digested sludge	Anaerobically Digested sludge
Fecal Coliforms	1.90	1.10
Fecal Streptococci	0.21	0.20
Salmonella	0.54	0.74
Helimth Ova	-0.06	-0.03
Protozoa Oocysts	> 8.00	> 8.00

Parker et al. (2000) examined whether bacteria would survive snowmaking and the freeze/thaw process that occurs in snow during winter and spring melt. It was found that bacteria able to grow at the temperature of the human body, which could be pathogens, survived the snowmaking process. Gram-negative coliforms were less affected by this process, with loss of two and three orders of magnitude for fecal coliforms and total coliforms, respectively. The gram positive and gram negative bacteria survived multiple Freeze-thaw cycles in the snow columns that were intended to replicate the melting process.

2.6 SNOWPACK

2.6.1 OVERVIEW

In most northern climates, snowfall and the resulting seasonal snowcover represent an important source of water. The time interval from the start to the end of the seasonal process may vary between years. Newly fallen snow has a density of about 10 percent of its water equivalent. But as the snow depth increases, settling and compaction increase the density, and the water equivalent can increase to 50 percent or greater (Anderson and Crawford, 1964). When the snowpack melts, the snowmelt recharges the groundwater and replenishes surface water storage.

When snow melts, the ice that composes the snow is converted into water. This water is called snowmelt. Since the conversion from ice to water requires the input of energy or heat, the process of snowmelt is strongly related to the flow and storage of energy into and through the snowpack. The sources of energy or heat to produce snowmelt are derived from short and long wave radiation, condensation of vapor, convection, air and ground conduction, and rainfall. The most important of these sources are convection, vapor condensation, and radiation (Anderson, 1973).

Convection from the air (sensible energy), vapor condensation (latent energy), and conduction from the ground are sources and sinks of energy that melt the ice portion of the snowpack, freeze the liquid water in the snow, and change the temperature of the snow. Male and Gray (1981) suggest that snowmelt is not homogeneous throughout the snowpack depth and point out that most of the melting occurs at the upper and lower interfaces of the snow.

Whenever sufficient energy is available, some snow (ice) will melt and form liquid water defined as snowmelt or meltwater. Since the physical structure of the snowpack is a porous matrix, this snowmelt will be held as liquid water, provided it does not refreeze between the snow grains and increase snow density and snow water content, Male and Gray (1981).

2.6.2 CHARACTERISTICS OF THE SNOWPACK

Before snowmelt infiltrates or runs off, the initial melt component is retained on snow crystal until they are thick enough to flow, coalesce and fill capillaries. The snowpack medium changes continuously as snow grains change in shape and size. Colbeck and Ray (1979) and Yosida (1973) have shown that as meltwater drains through the snowpack there exists a wetting front that is isothermal at 0°C . Anderson (1973) found that the delay to runoff caused by the snowpack might be much larger than for the vertical transit of water through the pack alone. Once the liquid water holding capacity of the snow is reached, the snow is said to be ripe. And due to the refreezing process, pack density increases. After the water holding capacity is reached, the density remains relatively constant with continued melt. Meltwater that exceeds the water holding capacity will continue to move down through the snowpack until the ground is reached. The amounts that infiltrate depend on soil characteristics, biosolids cover characteristics, soil moisture content, and whether or not the ground surface is completely or partially frozen. At this point runoff can happen. Here, the melting water released from the snowpack either runs off or infiltrates.

2.7 INFILTRATION

It is believed that chemicals added to a soil profile may dissolve and infiltrate with the flow of meltwater to the subsurface. The process of infiltration is defined as entry of water through the soil surface and entering the soil (McFarland, 2000). Infiltration can be considered as a three-step sequence: Surface entry, transmission through the soil, and depletion of storage capacity in the soil (Chou, 1964). Many terms define infiltration measurements. One of the common terms is “Cumulative infiltration”. It refers to the depth of moisture that has penetrated the surface after a particular amount of time or the total volume of water infiltrated per unit area of soil surface during a specified time period.

2.7.1 MELTWATER INFILTRATION

The subsurface conditions are important in evaluating snowmelt infiltration and runoff especially in the root zone, especially if fertilizer is applied. The soil layer functions as a reservoir, storing water when available to be used during periods when potential evapotranspiration exceeds current supply. In snow hydrology, presumably there will be essentially no direct runoff until the soil storage is filled to its field capacity, which is the total amount of water that can be held against gravity. The theoretical maximum capacity of the soil to withhold water permanently is determined by the difference between the permanent wilting point and the field capacity of the soil. For typical mountain soils, the maximum soil storage capacity ranges from approximately 10.2 to 20.3 cm (4 to 8 in.) of water in the zone from which stored water may be removed by transpiration or evaporation. After the field capacity of the soil has been reached,

excess water may pass through the soil under gravitational force and appear later as subsurface or base flow components of stream flow. The time delay of transitory storage in the soil is integrated in the total basin storage effect (Viessman and Lewis, 1995).

2.7.2 INFILTRATION INTO FROZEN SOIL

Partitioning of the snow water resources into runoff and soil water largely depends on the infiltrability of the frozen ground at the time of snowmelt (Viessman and Lewis, 1995). In Chapter two, studies on the application of biosolids onto different soil textures were addressed and confirmed that infiltration is an important process that contributes to nutrient and contaminant transport.

Infiltration of meltwater into frozen agricultural soil is a complicated process, as it involves coupled heat and mass flow with phase changes (Zhao and Gray, 1999). The process is affected by the thermal and hydro-physical properties of the soil, soil temperature, moisture content, and the quantity and rate of release of meltwater from the snowcover. Frozen soils do not change chemically in composition from the unfrozen except ice exists within soil voids. When soil temperatures decrease below the freezing point, not all water in a soil freezes. Ice-water surface tension and capillary forces depress pore water freezing temperatures adjacent to the soil grains (Most of the water that is in the unfrozen films on the soil surfaces is unfrozen because of osmotic freezing point depression). The result is a thin film of unfrozen water surrounding soil grains. This phenomenon is greater in fine grained soils such as clays and silts. Surface tension and freezing have less effect on soil moisture as the distance from soil grains increases. The unfrozen water content decreases as soil temperature decreases (Zhao and Gray, 1999).

Numerous approaches and empirical equations have been developed to represent the water infiltration into soils (Ampt, 1911; Horton, 1940; Philip, 1957). Despite the advancements made in understanding the process of infiltration into frozen soil, the methodologies and the procedures for proper implementation have not been fully described for frozen soil. Literature discussing frozen soils is much less extensive than for unfrozen soils. Komarov and Makarova (1973) suggest that a soil frozen to a depth of 150 mm or less absorbs meltwater, as does an unfrozen soil. And once a soil is frozen to a depth of 600 mm, freezing to greater depth has no further effect on the infiltration of meltwater.

Horton (1940) described three situations when meltwater reaches the ground. First, considering the case where the melt rate is less than the infiltration capacity of the soil. The meltwater directly enters the soil and a slush layer is not formed. The second case occurs when the infiltration capacity of the soil is greater than the meltwater rate, but the net capillary pull of the snowpack exceeds that of the soil aided by gravity. A slush layer forms and provides a supply of water that infiltrates the soil as rapidly as it enters the slush layer. The third case, is when the rate of meltwater exceeds the infiltration capacity. A slush layer forms and water infiltrates the soil at the infiltration capacity rate, and the excess water finds its way as surface runoff. As warm weather continues, the melt process is maintained until all the snowcover disappears.

Gray et al. (1986) proposed that frozen soils can be separated into three categories in terms of the snowmelt infiltration based on their surface entry condition (see Figure 2.1); unlimited, limited, and restricted. It can be seen that infiltration

increases with decreasing the soil surface saturation (S_p). Under unlimited conditions, the flow is predominant and most of the available meltwater is capable of infiltrating through the soils, containing a high percentage of large air-filled macropores. Restricted conditions are recognized when infiltration of meltwater is restricted by an ice/soil layer on the top surface, due to a freeze/thaw cycle with a pore saturation of $> 70-80\%$. The limited groups are identified where capillary flow is predominant and soil is governed by the snow water equivalent, soil moisture content, temperature at the beginning of snow ablation, and the infiltration opportunity time.

Zhao and Gray (1999) reported that in frozen mineral agricultural soils of the Canadian Prairies, where a frozen soil is of limited class and where capillary flow dominates, it does not become saturated throughout its wetted depth by infiltrating meltwater. This results from ice that blocks the interconnectivity of soil pores and the wetting front advances ahead of an overlying layer of high moisture.

Figure 2.1 Infiltration into Unlimited, Limited and Restricted Frozen Soils (Gray et al., 2001).

2.7.3 TEMPERATURE EFFECT ON INFILTRATION

In a completely frozen soil, meltwater always infiltrates against a freezing temperature and its ability to penetrate the soil is strongly related to the volume of air-filled pores, which is defined as effective porosity. Steenhuis et al. (1977) and Granger et al. (1984) investigated soil temperature at the wetting front versus effective porosity and showed that there is movement of water at low temperatures in soils with large effective porosities. Kamarov and Makarova (1973) and Steenhuis et al. (1977) suggested that the effect of temperature in frozen soils at the time of melt on infiltration may be secondary to those of effective porosity, which is inversely related to the ice content. Also, the entry of water in frozen soils is given mainly through large non-capillary pores.

Zhao and Gray (1997) suggested that during infiltration of meltwater into frozen soils, latent heat, released from freezing of percolating water, plays a very important role in the heat transfer process. This latent energy released is used to increase soil temperature and also to melt ice in the soil layers. Since the latent heat of fusion of water is two magnitudes higher than the specific heat of soil (Zhao and Gray, 1997), only small amounts of percolating water need to freeze in order to produce sufficient heat to increase the soil temperature of the frozen soil by several degrees. The colder the soil the larger the amount of water frozen and the greater the amount of latent heat released. As a result, the soil temperature at the wetting front is relatively constant regardless of the initial temperature.

Zhao and Gray (1997) demonstrated the relationship between the cumulative infiltration, after 24 hour and the initial temperature. As temperature decreased from -4° to -8° C, infiltration decreased by 25% in clay, by 20% in silty clay loam, by 19% in silt

loam, and by 41 percent in sandy loam. These changes in infiltration were less than the changes associated with initial soil saturation and surface saturation. In addition, the soil temperature at an advancing infiltration wetting front is relatively independent of the initial temperature of a frozen soil. The soil temperature increased faster for a colder soil, mainly because a larger amount of latent heat is supplied by the freezing of water in a colder soil. In a very cold soil the formation of ice may lead to significant blockage of pores by frozen water which will greatly impede water movement and infiltration.

2.7.4 MOISTURE CONTENT EFFECT ON INFILTRATION

Numerous researchers (Willis et al., 1964; Motovilov, 1979; Granger et al., 1984) reported the relationship between soil moisture content and infiltration in frozen soils. The degree of pore saturation that a shallow surface layer of soil reaches during snow cover ablation increases with increasing initial moisture content. It is seen from Figure 2.2 which shows snowmelt infiltration plotted against the average moisture/ice content of the 30 cm surface layer of a frozen soil, that an inverse relationship exists between soil moisture content and infiltration. This data, combined with the observation that the average depth to which infiltrating melt water penetrates into a frozen soil during snow cover ablation is of the order of 260 mm can be used to demonstrate the low infiltration characteristics of these soils. For example, a normal soil with porosity of 50 percent at an initial moisture content of 20 percent by volume, having a dry to medium wetness, would likely infiltrate on average less than about 47 mm of melt water or the water equivalent of a snow cover having a depth of 18.8 cm and a density of 250 kg/m^3 (Granger et al., 1984).

Figure 2.2 Infiltration plotted against the premelt moisture content of the 30 cm soil layer of completely frozen soils. (Granger et al., 1984)

2.7.5 INFILTRATION MATHEMATICAL MODELS

A general mathematical model was developed by Granger et al. (1984). The model predicts the degree of movement and penetration of water into frozen columns. The model is based on the measurements from 130 sites in different textured soils under various land uses.

$$INF = 5 (1 - \theta_p) SWE^{0.584} \quad (2.1)$$

In which INF is the amount of cumulative infiltration per unit length into the frozen soil (mm), θ_p is the degree of pore saturation (mm^3/mm^3), and SWE is the snow cover water equivalent in (mm).

Recently, a fully empirical model was described for simulating the process of infiltrability by Zhao and Gray (1999). They reported a parametric equation describing cumulative infiltration into frozen unsaturated soils of limited infiltrability, INF (mm) as:

$$INF = C S_0^{2.92} (1 - S_I)^{1.64} \left(\frac{273.15 - T_I}{273.15} \right)^{-0.45} t_0^{0.44} \quad (2.2)$$

Where $C = 2.10$ (Coefficient), S_0 = surface saturation moisture content at the soil surface ($\text{mm}^3 / \text{mm}^3$), S_1 = average soil saturation (water + ice) of 40 cm soil layer at the start of infiltration ($\text{mm}^3 / \text{mm}^3$). T_1 = average temperature of 0-40 cm soil layer at the start of infiltration (K), and t_0 = infiltration opportunity time (h).

2.8 DISCUSSION AND CONCLUSION

As opportunities for biosolids land filling, storage, and incineration are decreasing and becoming more costly, and as priority increases to beneficially recycle waste residuals whenever possible, directing biosolids for land application and for longer time is increasingly chosen as a safe ultimate disposal practice. However, in northern climates the process of land application in wintertime is still avoided, which minimizes the time available for land application. At the same time, little is known about the leaching properties of high-solid cakes. To assess the safety of practicing land application in winter months, the relevant studies performed on the ultimate disposal of different sludge and manure by-product were first reviewed. The relevant literature on the impact of soil moisture, temperature, and pH on nutrient losses from the applied sludges were also reviewed.

The current regulations are based on more than 25 years of research on land application of biosolids and an even longer record of beneficial use in North America. The scientific facts indicate that land-applying biosolids of the quality currently generated according to the regulations established by the USEPA will not result in significant detrimental health or environmental impacts, if precautions are taken.

The literature reveals that liquid and solid sludge have different impacts on nutrient runoff. The dry characteristics of dewatered sludge minimize the runoff from the snowpack when applied onto frozen soil, and increase the surface retention of meltwater. Thus soil infiltration may increase.

Of the several studies summarized in this report, it appears that there are variations in the findings. The variations from study to study stem primarily from the different physical properties of the soil when meltwater or raindrops reached the surface soils. Pore effects, temperature, and initial moisture content were also found to have a high impact on water infiltrability, whether the soil is completely frozen, partially frozen, or unfrozen.

Some studies reported that potential contaminant losses by surface runoff were not detected, and when compared to the control plots, no significant change was observed. Other studies reported that these losses contaminated the surface water, especially in the early stage. On the other hand, it was reported in other studies that all the lost materials infiltrated into the soil and no surface runoff was detected. This would suggest that mobilization of materials by raindrops or meltwater are highly dependent on the concentration of nutrients and contaminants in the biosolids, which vary both over time and between treatment plants and the type of sludge processing, example is pH and dewaterability.

When placed on a frozen surface, biosolids cake, due to its sponged-like characteristics, works as mulch. Hence, the cake absorbs the impacts of meltwater drops and reduces the volume of surface runoff.

High-solids cakes would allow for channelized meltwater or SWE to wash soluble pollutants and to some extent bacteria from the cake. Organic matter will remain in the

cake as colloid particles rather than leaching. Organic matter tends to leach but at a slow rate providing continuous nutrients to the plants.

In reaching the frozen soil surface, this flush (meltwater) would either infiltrate or runoff. The degree to which the flush infiltrates, will depend on soil physical and chemical properties, temperature, pore saturation, and moisture content of the soil. In addition, it is expected that meltwater carrying contaminants from the biosolids cake will infiltrate into sandy soil, but will not extend to lower depths when soils are completely frozen. It was also found that biosolids applied in freezing conditions had a higher fecal coliform mortality rate than spring-applied biosolids, and that freezing conditions are usually lethal to fecal bacteria.

Meeting the objective of this study, it was generally concluded from the previous studies that biosolids cake carries less water and less dissolved chemicals compared to the slurry sludges. Hence, surface water contamination from runoff is of less concern and current rates of land application could be safely applied into different time all over the year. Also, studies confirmed that sandy soils especially when frozen have high infiltrability to water. Hence, more biosolids could be directed for winter land application. And as management process, sandy soil is highly recommended for the practice. However, more researches are needed to investigate the process.

2.9 CONSIDERATIONS FOR WINTER SPREADING OF BIOSOLIDS

One of the goals of this literature review was to investigate the safety and the various risk factors associated with winter spreading of biosolids. Considerations should be taken before applying biosolids in winter from the generally following true points:

- The greatest impact is “weather”. Since this is a factor that is out of our control and cannot be accurately predicted, the risk of runoff from winter-spreading biosolids will be low some years and high in other years. Climate records may help to identify the areas where the risks are highest.
- Most but not all frozen soils are virtually impervious - there is a high likelihood that snowmelt and rainfall on biosolids-covered frozen ground will result in the runoff of constituents.
- The fate of the first meltwater or rainfall following winter biosolids spreading will usually determine the amount of biosolids runoff - if it percolates into the ground, the runoff amount will be relatively small or negligible. If the ground is frozen, the runoff amount can be relatively high.
- The amounts of nutrients and contaminants lost in runoff following the winter application are usually greater than the other seasons, though this is not always the case.
- Spreading biosolids in the winter provides no guarantee of pathogen die-off, though freezing conditions are usually lethal to fecal bacteria.
- The rate of volatilization of ammonia from biosolids is diminished for winter-spread of biosolids, especially if the cake is covered by snow.
- The risk of biosolids runoff is expected to be similar, whether the biosolids are spread on frozen bare ground or on snow-covered ground.

- Spreading biosolids in the winter can actually reduce the amount of runoff and soil erosion. It forms a mulch on the soil surface that slows down the flow of water. However, spreading biosolids onto a soil in the winter does not necessarily reduce the risk of runoff. Climate records may help to identify the areas where the risks are highest.
- In order to minimize the risk of nutrient and contaminant losses associated with runoff, sandy soils are characterized by their high water infiltrability, especially when frozen. And as management process for winter land application of biosolids, application to sandy soils is highly recommended.

3 METHODOLOGY, EQUIPMENT, AND MATERIALS

This section presents a discussion of the methods of analysis and equipment used in the study of winter applications of biosolids. The methods and measurements include snow water equivalent, soil physical properties, soil saturation, temperature, and soil water sampling that were used for analysis.

3.1 BIOSOLIDS BATCH TEST

Biosolids batch tests were performed to evaluate the mobility of contaminants from biosolids under frozen and thawed conditions. Fresh biosolids cake samples were collected in 3 different periods, January, February, and March (2002). Figure 3.1 summarizes the process.

Figure 3.1 Biosolids Batch test processes

To measure the effect of freezing on fresh samples, at each collection time, sterilized 250 ml polyethylene bottles were used for collecting triplicate 25 gram and 50 gram for

fresh samples preserved at 4°C . In order to analyze a trend, the samples were tested after 1 day, 14 days, and 28 days for each of the 3 months. Frozen samples were separated and tested with the same method used for the fresh samples, except that preservation was at -4°C .

At each time, the properties of the samples were analyzed. 3 samples from 25 g and 50 g of fresh sample were measured and 3 samples from 25 g and 50 g frozen sample. 100 ml of distilled water was added to each sample. The distilled water added to the samples was at room temperature (21°C), then the bottles were shaken on a mechanical shaker for one hour and centrifuged in a SORVAL™ rotor at 5000-rpm speed for 15 minutes, and filtered through a $0.45\text{-}\mu\text{m}$ -membrane filter. The filtrates were then analyzed using ion chromatography, ammonia selective electrode and chemical oxygen demand tests.

3.2 COLUMN EXPERIMENT

3.2.1 COLUMN CONSTRUCTION

The column experiment test consisted of 6 identical columns. Each column was constructed of 15.3 cm internal diameter PVC pipe and was 105 cm tall and a wall thickness of 0.5 cm. A square PVC plate of 25 x 25 cm was glued to the bottom end of the column with a PVC adhesive material. The main objective of the experiments was to sample percolating water from various depths while soils thawed. Tests were performed using 3 different accessories at different heights: wave guide rods connected directly to a Tektronix 1502B cable tester for volumetric moisture content measurement, thermocouple wires for temperature measurement, and ceramics cups for sampling soil solution. The column design is illustrated in Figure 3.2

For measuring the moisture profile along the column, vertical alignments of holes were drilled at 5 different soil depths (7, 17, 35, 55, and 85 cm). For temperature profile measurement, vertical aligned of holes were drilled to insert the thermocouple wires at 6 different soil depths (9.5, 19, 29, 39, 59, and 89 cm). Holes for the ceramic cups were also drilled at the required locations (A vertical alignment of ceramics cups model 1900 SOILMOISTURE®, with a 2 bar air entry value). The samplers had a 2 cm outside diameter and 6 cm length with an end PVC tube of 15 cm. The samplers were inserted at soil depths of 15, 60, and 95 cm, respectively. 10 cm long nipples (2.2 cm diameter) were screwed to each ceramic cup location to work as support for the sampling tube and to prevent bending and displacement when sampling with the handy suction pump.

The sides of the columns were wrapped with 2 layers of 1 cm REFLECTIX® insulation tape (made with 9% pure aluminum separated by a 5/16 inch dead air space) to limit warming from the sides, and thus simulate what would happen in the soil. The horizontal R-value reported by the manufacturer company for the insulation was $R = 9.8$. Figures 3.3 to 3.6 show photographs of the columns which were placed in the cold chamber.

Prior to packing the columns, the inside of the columns were rinsed with tap water. Then the two different soils, A and B, reported in section 3.3.3, were packed. Soils were filled at 20 cm layers each time and compacted by tapping the columns from outside with a thick metal bar.

To prevent the preferential flow from the top surface (flow between soil and column wall), at 3 cm depth from the surface a neoprene tube was welded with an adhesive around

the internal circumference of the columns, maintaining a good sealing and allowing the snow water equivalent to infiltrate in the soil rather than the edges.

3.2.2 COLUMN PREPARATION

Prior to the experiment, Ports 1 and 2 (see Figure 3.2) were temporarily sealed with tape and a small aluminum meshes were placed on all Port 3 to act as a drainage port and to prevent losses of soil particles (none of the ceramic cups were inserted). Two different soils were used for the columns experiment; Soil "A" (100 % sand) and Soil "B" (75% sand and 25% soil collected from the Carleton University area). Column A, Column B, and Column C contained the sandy Soil A while Column D, Column E, and Column F contained the mixed Soil B. Thermocouple wires and TDR rods were adjusted and fixed while filling the columns with soil.

The columns were packed with soil to a depth of 100 cm and then filled with 10 liters of distilled water. At the bottom, all Port 3 were opened to let the columns drain. The columns were allowed to gravity drain for 3 days to approach conditions found in nature. In order to simulate an unsaturated soil moisture profile, the mesh on Port 3 was removed and ceramics samplers were pushed horizontally inside the soil with a light clockwise motion. TDR moisture volume was recorded with the TDR rods, and then a negative pressure was applied on the ceramics cups until an unsaturated moisture profile was achieved.

After achieving an unsaturated moisture profile within the columns, the ceramic cups were removed (to prevent cracks from freezing temperature). A cold chamber was set to -10 ± 2 °C. The columns were allowed to cool until the thermocouple readings of the soil temperature reached -10 °C. At the same time, frozen biosolids cakes were weighed. Two

loads of cake were investigated; a load of 49 grams (14.7 grams dry weight basis, meeting regulations) for both Column B and E, and 1000 grams (300 grams dry weight basis) for both Column C and F were used for comparison. Columns A and D did not receive any biosolid cake and were monitored as controls.

The snow water equivalent was also calculated and cooled to 1 °C. Aluminum mesh was placed on the top of each column on which biosolids cakes were placed. Snow water equivalent (distilled water) was then added on a daily basis melt, a volume of 280 ml (1.5 cm) was assumed. The daily melt water was only added on the first three days. Then a large volume, equivalent to 3 days snowmelt (840 mL), was applied due to the poor volume of water sampled for analysis and the low hydraulic conductivity of the ceramics cups. This amount was applied every three days. Table 3.1 summarizes the time and location of sampling from Port 1, Port 2, and Port 3. The amount and time of the applied SWE was reported in the table. The length of the experiment was 21 days that was divided into seven periods, each period consisting of 840 mL of SWE.

For sampling, suction was applied by a hand pump until a value of 0.65 bars was achieved, then the tubes were attached to a continuous electrical suction pumps to draw the sample over night. At each application of water (840 ml), suction was maintained on Port 3 of the six columns until the moisture profile returned to its original profile. The volume of effluent from (Port 1 + Port 3) or (Port 2 + Port 3) was also measured and values are presented in Appendix C. TDR was used to record the moisture content for the columns. Samples extracted from the samplers were stored in sterilized bottle at 4°C and analyzed for pH, ion concentrations, ammonia, chemical oxygen demand, and bacteria indicator analysis.

Table 3.1 Time and amount (mL) of equivalent snow water application, * represents the volume of water applied, ● represents the location and day of sampling infiltrated water.

Time days	SWE Application (mL)	Sampling location		
		Port 1	Port 2	Port 3
1 (Start)	* (280)			
2	* (280)			
3	* (280)			●
4	* (840)	●		
5				
6				●
7	* (840)		●	
8				
9				●
10	* (840)	●		
11				
12				●
13	* (840)		●	
14				
15				●
16	* (840)	●		
17				
18				●
19	* (840)		●	
20				
21 (End)				●

Figure 3.2 Schematic presentation for column construction (measurements are in cm).

Figure 3.3 Photograph of the columns.

Figure 3.4 Photograph. - inserting the ceramic cup samplers into the sampling port.

Figure 3.5 Photograph - Snow water equivalent head for 3 days intervals (from the right-control, regulated application 14.7 g dry weight biosolids, and 300 g dry weight).

Figure 3.6 Photograph – tubing and collection system for the infiltrated SWE.

3.3 MATERIALS, EQUIPMENTS, AND METHOD RESULTS

3.3.1 REVIEW OF SNOW WATER EQUIVALENT CALCULATION

The method employed to measure rain, drizzle, freezing rain, freezing drizzle and hail uses the standard Canadian rain gauge. A cylindrical container 40 cm in height and 11.3 cm in diameter is used. The precipitation is collected in such a container funneled into a plastic graduated cylinder that serves as the measuring device. Snowfall precipitation is the measured depth of newly fallen snow, measured using a snow ruler. In this case, measurements are taken at several points that appear representative of the immediate area and then averaged.

At most typical stations, the water equivalent of snowfall is measured by dividing the measured depth by ten. In other stations it is determined once the snow fallen in the Nipher gauges has completely melted. The amount of snow determined by the latter method provides more accuracy for estimation of precipitation than using the ten to one rule. We find that, even at typical climate stations, the normal precipitation values do not always equal the observed rainfall plus one tenth of the snowfall.

Snowmelt rates are also determined using the temperature index methods (US Army Corps of Engineers). The equation reflects the extent of radiation and the vapor pressure of the air. The equation is also sensitive to air motion. Frequently it is the only adequate meteorological variable regularly on hand, (Viessman and Lewis, 1995). Various methods estimate the mean temperatures. One method is simply to average the maximum and minimum daily temperatures. Figure 3.7 shows temperature index equations in spring for snowmelt in clear and forested areas.

Figure 3.7 Mean temperature index and daily snowmelt equations for open and forest site. (After U.S. Army Corps of Engineers).

A degree hour or degree-day is an index of the amount of heat present for snowmelt or other purposes and has proved useful in point-snowmelt and runoff from snowmelt determinations (Viessman and Lewis, 1995). Another approach used is to calculate a degree-day factor, defined as the ratio of runoff or snow melt to the accumulated degree-days that produce the runoff or melt. In this case, the degree-day factor was found to vary seasonally and between basins. Thus, single representative values are used. Point degree-day factors for snow-covered basins range from 0.015 to 0.20 inches per degree per day when melting occurs. Gartska (1958) states that an average point value of 0.05 can be used to represent spring snowmelt. Linsley et al. (1975) states that mean degree-day factors are usually

between 0.06 and 0.15 inch/degree-day under conditions of continuous snowcover and at melting temperatures.

3.3.2 SNOW WATER EQUIVALENT CALCULATION

To simulate the desired field conditions, snow water equivalents were calculated using the climate normals. These are arithmetic calculations based on observed climate values for a given location over a specified time period. In order to eliminate variations, thirty years of observed climate data was analyzed from 1971 to 2002. This data was adapted from the Canadian Climate Normals for Ottawa Macdonald-Cartier station, Latitude 45° 19' N, Longitudes 75° 40' W, and Elevation 114 m. This station meets WMO standards for temperature and precipitation (Environment Canada, 2002). Rainfall, snowfall and precipitation amounts given in Table 3.2 represent the average accumulation for December, January, February, and March covering the winter period. "Precipitation" in Table 3.2 is the water equivalent at room temperature of all types of precipitation.

Table 3.2 Temperature and precipitation climate normals for 1971 - 2002 for Ottawa Macdonald - Cartier station.

	Dec.	Jan.	Feb.	March
Daily Mean Temperature (C°)	-7.1	-10.8	-8.7	-2.5
Standard Deviation	3.2	2.9	2.7	2.4
Daily Maximum Temp. (C°)	-3.0	-6.1	-4.1	2.2
Daily Minimum Temp. (C°)	-11.1	-15.3	-13.3	-7.1
Extreme Minimum Temp. (C°)	-34.4	-35.6	-36.1	-30.6
Rainfall (mm)	31.3	25.2	17.6	36.3
Snowfall (cm)	57.2	55.2	46.0	39.8
Precipitation (mm)	81.5	70.2	58.9	73.9
Mean Snow Depth (cm)	14	27	32	23
Median Snow Depth (cm)	13	27	32	23
Snow Depth at Month-end (cm)	24	30	30	7

3.3.3 SNOW WATER EQUIVALENT RESULTS

The volume of snow water equivalent was first calculated with the start of spring melt on the first of April. The summation of the cumulated precipitation for the four winter months December to March was computed from the climate normals.

The total height of snow for the four months was 28.5 cm. Rainfall precipitation was taken into account, assuming that the snow was ripe and no runoff occurred for the four winter months. Following the finding of the U.S. Army Corps of Engineers (Viessman and Lewis, 1995), the daily amount of snowmelt or equivalent snow water was predicted using the equation for an open site at 1°C (33.8 °Fahrenheit). The equation is available in Figure 2.3 and it computed a value of 0.588 inch /day (1.5 cm/day).

3.4 BIOSOLIDS

Dewatered anaerobically digested biosolids by-product was collected from Robert O. Pickard Environmental Centre (ROPEC) (located in Ottawa, Ontario) and used for this study. Centrifugation is the technology used for dewatering at ROPEC ($\geq 30\%$ solids content). Samples were collected from the centrifuge outlet and transported in sterilized polyethylene plastic containers to the laboratory for analysis. The sludge or biosolid characteristics for the period of January, February, and March of 2002 are given in Table 3.3.

Table 3.3 Physical and chemical characteristics for biosolids (Adapted from ROPEC, 2002).

Date	Total Solids (%)	Ammonia (mg/kg)	Total Phos. (mg/kg)	TKN (mg/kg)	Nitrate/Nitrite (mg/kg)
07-Jan-02	29	5,900	33,000	45,000	10
15-Jan-02	29	6,000	32,000	40,000	7
07-Feb-02	29	5,900	33,000	28,000	<1
15-Feb-02	29	5,200	28,000	33,000	<1
07-March-02	30	5,200	29,000	35,000	<1
MIN	29	5,200	28,000	28,000	1
AVG	29	5,640	31,000	36,200	4
MAX	30	6,000	33,000	45,000	10

3.4.1 BIOSOLID RESULTS

Total solids were measured for fresh biosolids samples collected from ROPEC. 10 random samples were placed in prepared evaporation dishes and weighted using a digital scale. The samples were dried overnight at 105⁰ C, and cooled to room temperature in desiccators and total solid was measured. Equation (3.1) presents the calculation method.

$$\% \text{ Total Solids} = \frac{W_{total} - W_{dish}}{W_{sample} - W_{dish}} \times 100 \quad (3.1)$$

Where:

W_{dish} = Weight of dish (g)

W_{sample} = Weight of wet sample and dish (g)

W_{total} = Weight of dried residue and dish (g)

Tables 3.4, 3.5, and 3.6 present the average and standard deviation for the collected samples of biosolids for January, February, and March respectively. Figures A-1, A-2, and A-3 illustrate the values and are available in Appendix A. No significant changes were observed for the three months. The average total solids content in the cake for the three months was 29.6 % ± 0.5.

Table 3.4 Total Solids for January sampling.

Sample No.	W_{dish}	W_{Cake}	W_{Sample}	W_{total}	% Total Solids
1	62.73	8.1	70.83	65.14	29.72
2	60.25	14.17	74.42	64.48	29.85
3	58.65	10.25	68.9	61.72	29.91
4	56.74	16.46	73.2	61.64	29.74
5	59.48	29.54	89.02	68.34	29.99
6	59.18	22.14	81.32	65.85	30.12
7	58.24	17.36	75.6	63.38	29.59
8	59.74	14.56	74.3	63.98	29.15
9	59.25	10.59	69.84	62.40	29.73
10	60.25	18.56	78.81	65.79	29.83
Mean					29.76
Std. Dev.					0.26

Table 3.5 Total Solids for February sampling.

Sample No.	W_{dish}	W_{Cake}	W_{Sample}	W_{total}	% Total Solids
1	57.12	34.26	91.38	67.32	29.78
2	59.49	45.29	104.78	73.08	30.01
3	57.08	25.26	82.34	64.66	29.99
4	59.36	33.15	92.51	69.31	30.03
5	56.74	15.25	71.99	61.24	29.48
6	59.18	19.26	78.44	64.92	29.78
7	59.25	25.45	84.7	66.75	29.48
8	60.25	13.25	73.5	64.18	29.69
9	56.74	17.59	74.33	62.02	30.01
10	57.12	18.36	75.48	62.59	29.77
Mean					29.80
Std. Dev.					0.21

Table 3.6 Total Solids for March sampling.

Sample No.	W_{dish}	W_{Cake}	W_{Sample}	W_{total}	% Total Solids
1	56.74	15.26	72	61.26	29.63
2	60.05	9.56	69.61	62.93	30.12
3	59.48	31.58	91.06	68.88	29.78
4	59.68	18.95	78.63	65.34	29.88
5	59.16	16.78	75.94	64.14	29.65
6	59.48	9.56	69.04	62.34	29.89
7	59.79	7.89	67.68	62.14	29.78
8	58.48	14.14	72.62	62.68	29.69
9	59.12	8.59	67.71	61.70	30.06
10	59.75	22.54	82.29	66.48	29.86
Mean					29.83
Std. Dev.					0.16

3.5 REVIEW OF SOIL COMPOSITION

Soil in the environment consists of three main phases; soil particles, water, and air (see Figure 3.8). The soil grains represent the solid phase. Void spaces include air and water and exist in between the soil particles. The voids may have other constituents in liquid or gases phases may also exist, like contaminants, these contaminants are usually not considered in typical mechanical composition (for texture) but they are considered in the chemical composition. The solid phase of the soil can include organic matter, ice, or frozen contaminants. The liquid phase may include immiscible contaminants such as oil or contaminants dissolved in soil moisture. Some liquid contamination usually adsorbed in the soil particles. Gases and water vapor that could be present in voids may include methane and volatile organics.

Figure 3.8 Volumetric distributions of soil components where V_a , V_w , V_s , V_v , and V_t represent volume of air, water, soil particles, voids, and all soil components.

3.5.1 SOIL IN COLUMNS

Two different soils, defined as Soil A and Soil B were used for this study. Soil A was a commercial sand used for construction (BURNSIDE SAND & GRAVEL LIMITED), beige in color and consisted of 100% sand. Soil B consisted of 75% sand (same as sand for Soil A) and 25% dark brown soil collected from a 1-meter depth of a construction site located at Carleton University. This ratio of 75% sand and 25% soil collected from the site was chosen because high clay content caused soil to be too tight and hence minimized infiltration. Debris from the soil was removed manually and soil was oven dried at 105°C. In order to have a homogenous mixture, dry soil was blended with sand in a concrete mixer for 30 minutes. Soil samples were analyzed for their grain size distribution, particle density, porosity, and void ratio to determine their physical properties.

Sand was chosen for these experiments for two reasons. First, because of the packing procedure, it is difficult to obtain uniform bulk densities across different water contents in finer textured soils, because the compressibility varies with water content. Second, finer textured soils may contain considerable amounts of frozen water when the temperature drops to below 0 °C. This would limit the infiltration process and is not recommended for winter spreading of biosolids as reviewed in Chapter two (Cooke et al., 2001; Sullivan et al., 2000; O'Neil and Sharon, 1995; Mostaghimi, 1993). In addition, clay can absorb water and due to the method used for sampling, which employed ceramic cups, the soil needed to have a low-tension force between particles.

3.5.2 SOIL CLASSIFICATION METHODS

Soil classification methods specify criteria for grouping soils based on visual observations and laboratory test results. The most common method used for engineering applications, and the method used for this thesis study, was the Unified Soil Classification System (USCS). ASTM D 2487-85 describes the standard test method for soil classification for engineering purposes (ASTM, 1998). ASTM D 422-63 (1990) describes mechanical methods for determining grain-size analysis of soils using sieve and hydrometer analysis (ASTM, 1998).

3.5.3 METHOD USED FOR GRAIN SIZE DISTRIBUTION

Grain size analysis was performed on each of the two soils using ASTM D 422-63 test method of Particle-Size Analysis of Soils. In this method the samples were oven-dried at 105⁰C, most residues and organic particles were visually identified and removed manually by shaking the soil on a #10 sieve and discarded. A rubber-tipped mortar was used to break up conglomerations of soil. The aim was to make sure that the soil sample consisted of individual particles. Each soil sample was mechanically analyzed to determine the grain-size distribution. The sieves used were #'s 10 (2 mm), 20 (850 µm), 30 (600 µm), 40 (425 µm), 100 (150 µm), and 200 (75 µm). Sieves were shaken using a mechanical shaker for 15 minutes. Soils were classified using the Unified Soil Classification System (USCS).

3.5.4 GRAIN SIZE DISTRIBUTION RESULTS

Tables A-4 and A-5 present the sieve analysis data for sandy Soil A and mixed Soil B, respectively and are available in Appendix A. Figure 3.9 illustrates the grain size distribution for the two different soils. The grain size distribution curves indicate that the soils were approximately similar. It was found from the distribution that the medium to fine grain was smaller than 12% coarse sand and bigger than 30% medium sand for the two soil. (Coarse is from sieve #4 to #10, Medium is from sieve #10 to #40, and Fine is from sieve #40 to #200, USCS). It was observed that Soil B retained 9.6% of the weight on sieve #200 and 4.8% of the weight on the pan, as Soil A retained 5.4% of the weight on sieve # 200 and 1.2% of the weight on the pan.

Based on the USCS, the results indicated that for the two soil types A and B more than 50% of the grains fraction was retained on sieve #200 and more than 50% of the grain fraction passes the sieve #4, and the soil were defined as either clean sand or sand with fines. The mixed Soil B contained approximately twice the percentage of fine soil retained on sieve #200 and four times what was retained on the pan, and from the beige color of Soil A and the dark brown color of Soil B, Soil A was clean sand and was classified as WELL GRADED SAND (SW). Soil B contained fines particles and was classified as CLAYEY SAND (SC). These characteristics could significantly affect the hydraulic properties, solute and pathogen percolation into the two soils.

Figure 3.9 Sieve soil analysis for the Soil A and the Soil B.

3.5.5 BULK DENSITY, UNIT WEIGHT

Dry bulk density of soil (ρ_b) [M/L^3] and unit weight (γ) [ML/T^2L^3] are measured parameters for the soil. Bulk density represents the soil mass per unit volume. It can be calculated on a wet basis or on a dry mass basis. Wet bulk density is the mass of soil and moisture per unit volume of a soil sample. Dry bulk density is the dry soil mass per unit volume of soils. Unit weight is the soil weight per unit volume of all soil components that are present. Soil particle density (ρ_p) [M/L^3] is also a measured soil characteristic. Particle density is defined as the soil particle mass per unit volume of soil particle. ASTM D 854 explains how to determine soil particle density (ASTM, 1998).

3.5.6 BULK DENSITY AND UNIT WEIGHT RESULTS

The method is described in soil analysis part 1, (ASTM, 1986). Bulk density of Soil A and B were calculated and then average and standard deviation were reported.

Table 3.7 presents the average and standard deviation for the bulk density of Soil A and Soil B. These results indicated that the Soil A had a higher bulk density than the mixed Soil B. This was attributed to the fact that sand has less pores space than fine textured soils.

Table 3.7 Bulk density for Soil A and Soil B.

Test No.	Volume (mL)	Dry Mass (g)	Bulk Density (g / mL)
Soil (A) 100% Sand			
1	100	178.6	1.786
2	200	358.2	1.791
3	250	449.1	1.796
4	250	448.7	1.795
Average			1.792
S.D			0.004
Soil (B) 25% from site + 75% Sand			
1	60	100.6	1.677
2	200	339.1	1.696
3	200	337.4	1.687
4	250	423	1.692
Average			1.687
S.D			0.008

3.5.7 PARTICLE DENSITY, POROSITY, AND VOID RATIO

Porosity (η) [L^3/L^3] and void ratio (e) [L^3/L^3] refer to the void spaces in a soil. Soil voids consist of the soil constituents not comprised of the soil particles. Porosity is the ratio of void volume per volume of all soil constituents. Equation (3.2) shows the calculation for porosity.

$$\eta = \frac{\text{Void Volume}}{\text{Total Volume}} = \frac{V_v}{V_t} \quad (3.2)$$

Porosity is related to dry bulk density and particle density by Equation (3.3).

$$\eta = 1 - \left(\frac{\rho_b}{\rho_p} \right) = \frac{e}{1+e} \quad (3.3)$$

Void ratio (e) [L^3/L^3] is the ratio of void volume [L^3] per soil-particle volume [L^3]. Equation (3.4) shows how to calculate void ratio.

$$e = \frac{\text{Void Volume}}{\text{Soil particle Volume}} = \frac{V_v}{V_s} = \frac{\eta}{1-\eta} \quad (3.4)$$

Equations (3.4) can be multiplied by 100 to represent porosity as a percentage.

3.5.8 PARTICLE DENSITY AND POROSITY RESULTS

Particle density of Soil A and B was calculated using ASTM D 854 and porosity (η) [L^3/L^3] was calculated using equation (3.2). Table 3.8 presents the calculated particle density and porosity for Soil A and Soil B. The average and standard deviation for porosity of different masses and volumes for Soil A and Soil B, respectively. These results indicated that the porosity of Soil A was less than what was found for Soil B. Porosity depends on both soil texture and structure. Soil B had smaller but more numerous pores than Soil A that had bigger particles than Soil B. Water can be held tighter in small pores than in large ones, and the mixed Soil B will hold more water than the Soil A.

Table 3.8 Particle density and porosity for Soil A and Soil B.

Test No.	Volume (cc)	Mass Dry (g)	Final Volume	Water displaced (cc)	Particle Density (g/cc)	Porosity (%)
Soil (A) 100% Sand						
1	150	115.4	190	40.00	2.885	38.09
2	200	86.8	230	30.00	2.883	37.88
3	100	121.8	142	42.00	2.886	37.75
4	125	43.6	140	15.00	2.886	37.80
Average					2.896	38.13
S.D					0.009	0.09
Soil (B) 25% from site+75% Sand						
1	150	110.6	190	40.00	2.765	39.36
2	200	90.5	233	33.00	2.742	39.18
3	100	86.2	132	32.00	2.694	39.37
4	150	120	195	45.00	2.667	39.55
Average					2.717	39.36
S.D					0.045	0.15

3.6 SOIL WATER SAMPLING AND SUCTION LYSIMETERS

Suction lysimeters are devices that are used for collecting pore-liquid samples from unsaturated and saturated soils and fractured rocks (ASTM D 4696-92 Standard Guide). The operating principles of the samplers are that soil water is held largely under a state of tension within the soil by capillary forces. And water is naturally attracted to soil particles and sticks to the surface of each particle and is held in the various sized capillary spaces or pores between the soil particles. When the soil is very wet, the large pores fill with water. This excess water has no direct surface contact with the soil and is held cohesively, one water molecule to another, and can move quite freely. As the soil dries out, the excess water first evaporates, as it requires less energy to break the cohesive bonds. The remaining water, held tightly inside the capillary spaces by adhesive qualities, requires more energy to be removed from the soil (Looney and Falta, 2000).

Suction lysimeters consist of a hollow porous tip attached to a vessel or a body tube. Samples are obtained by applying a vacuum to the sampler and collecting pore-liquid in the body tube (Figure 3.10). When suction greater than the soil pore-liquid capillary pressure is applied to the sampler, liquid moves into the sampler. Ceramic porous segments are hydrophilic, and the maximum pore sizes are small enough to allow menisci to withstand the entire range of sampling suctions. If the maximum pore sizes are too large, the menisci are not able to withstand the applied suction. When the menisci break down, hydraulic contact between the water in the porous tip and soils is lost, and soil air enters the sampler, leading to the release of suction.

The suction required to remove the moisture from the soil differs. For example, it is easier to remove water from a sandy soil with 10 % moisture, than it is to remove water from a clay soil with 30 % moisture. This is because the water in the clay soil is held in very small capillary spaces within the soil particles under higher soil suction, whereas the sandy soil holds water in large capillary spaces under lower soil suction.

Figure 3.10 Schematic of vacuum lysimeter (ASTM D 4696-92)

3.6.1 METHOD FOR SAMPLING THE SOIL SOLUTIONS

Suction lysimeters were used for sampling the infiltrated water in the column experiments. The sampler used was a model 1900 SOILMOISTURE[®] with a 2 bar air entry value and sampler consisting of a 2 cm outside diameter, 6 cm length with an end PVC tube of 15 cm, and neoprene tubing attached to the vacuum pump (see Figure 3.2 and 3.6).

Ceramic cup samplers were used for this experiment due to their ability to sample filtered water from the soil pores. In addition, sampling from the unsaturated zones was not feasible with other sampling methods.

The samplers used in this experiment allowed water to be removed from the soil by creating a vacuum or a negative pressure inside the sampler greater than the soil suction holding the water in the capillary spaces. This established a hydraulic gradient for the water to flow through the porous ceramic cup and into the sampler. This allowed the liquid water to flow to the ceramic cups due to the potential gradient where water will move from less negative potential to more negative potential.

3.7 METHOD OF TEMPERATURE MEASUREMENT

Temperatures profiles were measured with a thermocouple, a device consisting of a pair of wires of different metals “semiconductors” joined at both ends. One junction is at the temperature to be measured, the second at a fixed temperature. The electromotive force generated depends upon the temperature difference. Thermocouples (Type K OMEGA[®]) were used for the column experiments. Thermocouples were chosen for these measurements for their high sensitivity and flexibility over other measurement methods. Temperatures were observed to within a tenth of a degree Celsius.

3.8 SOIL SATURATION AND TIME DOMAIN REFLECTOMETRY

The time domain reflectometry (TDR) technology is relatively a new method which has been employed successfully in the geotechnical and mining industries for monitoring rock mass deformation and subsidence, and for measuring water level, water pressure, soil moisture content, and liquid content of frozen soil (Huang and Dowding 1994; O'Connor and Wade 1994). The TDR method was first applied to soil-water investigations by Topp et al. (1980). This method is based on the determination of the propagation velocity of an electromagnetic wave along a transmission line (that is, waveguides inserted in the soil). Because of the large difference in dielectric constants of the soil components, the TDR method is practically insensitive to soil solid-phase composition and texture. However, at contaminated sites, the dielectric constant (K_d) may vary as the contaminant volume changes.

A variety of TDR probes have been developed for different practical applications. Topp and Davis (1985) and Yokuda and Smith (1993) determined vertical moisture-content profiles using rods of different diameters at fixed intervals, which created impedance discontinuities. Ward et al. (1994) used horizontally installed probes. Ferre et al. (1994) developed a field multilevel probe consisting of two water-filled PVC access tubes (located in pilot holes) and a pair of target rods that are moved manually.

DeRyck et al. (1993) and Redman and DeRyck (1994) developed a multilevel TDR device that includes several horizontal rods attached every 2.5 cm to a PVC access tube at one end, and to a PVC support tube at the other. Each pair of rods forms a waveguide. A spring-loaded contact device moves through the access tube to provide measurements of

changes in the apparent dielectric constant, which are related to changes in the moisture content. This approach was also used to determine the changes in concentration of kerosene and tetrachloroethylene (TCE). However, this technique was used only in repacked flow cells.

Oliphant (1985) and Seyfreid (1991) reported that the (K_d) of frozen soil increases with the total soil water content. Specifically, two assumptions are understood to Topp's equation. The first assumption, termed the ice effect, is that the dielectric constant of ice is equal to that of air. The second assumption, termed the temperature effect, is that the dielectric constant of water is independent of temperature.

The assumption that the dielectric properties of soil water are similar to those of bulk water does not hold for sediments with high surface charge (Dirksen and Dasberg, 1993; Roth 1990; White et al. 1992). Significant deviations from a linear calibration curve occur at low water contents because of the effect of bound water. Herkelrath et al. (1991) recommended determining the TDR calibration between soil bulk dielectric constant and the soil water content for particular types of soils investigated.

The main advantages of the TDR method over other direct and indirect methods of soil-water content measurement are the accuracy (may be up to 1 or 2 percent of the volumetric water content), minimal calibration requirements, and that there are no radiation hazards as those associated with neutron-probe or gamma-attenuation techniques. TDR equipment (manufactured by Tektronix, Inc., and Campbell Scientific, Inc.) is available for telemetric and multiplexing enhancement of remote measurements in different geotechnical and environmental engineering applications (Huang and Dowding, 1994).

Mallants et al. (1996) showed that the success or failure of TDR for monitoring solute transport depends on the quality of the calibration procedure. These authors recommended calibrating TDR probes using undisturbed soils taken from a location adjacent to the measurement point.

Bilskie (1999) showed that the error of the TDR method for measuring water content is caused by reflection distortion, which is caused either by attenuation in the coaxial connecting cables, or by attenuation of the signal at the probe via free ions in the soil solution. These processes are not taken into account in mathematical models used for evaluation of the reflection waveform. The TDR measurements can be improved by taking into account the resistance of the coaxial cable and connectors.

3.8.1 METHOD USED FOR WATER MOVEMENT AND SATURATION MEASUREMENT

TDR was employed to investigate the saturation and the water movement of the soils in the columns. TDR waveforms were generated using a Tektronix 1502BC waveform generator. The saturation was analyzed using the WATTDR software (Redman, 2000) that permitted acquisition of TDR by converting the distance to time units in nanoseconds (ns) through the RS-232 interface module. The time units were used for displaying and plotting data on the PC (see Figure 3.11). The acquired waveforms were calibrated (dielectric constant versus percent saturation), and then calibration curves were used for the moisture content measurements.

Figure 3.11 Method of acquiring a waveform from the 1502 Tektronix TDR.
(Adapted from the WATTDR manual).

3.8.2 CALIBRATION OF TDR

Experimental values of K_d at different water contents were obtained by mixing known quantities of distilled water with determined volumes of soil samples to obtain a desired water content, starting at 10% saturation and going to 100% saturation. Actual values of K_d and the corresponding percent volumetric moisture contents of this study are shown in Figures 3.12 to 3.15. Calculations are given in Appendix A. Typical K_d values for water and ice are given in Table 3.10.

Samples of Soil A and Soil B were packed in polyethylene bottles that were about 2 cm longer than the TDR wave guide rods. The wave guides consisted of two parallel, 16 cm long rods of 6.35 mm diameter stainless steel rods. The waveguides were inserted into the soil with a separation of 2 cm and connected to the TDR via a coaxial cable. Soil water content was first measured at room temperature, and samples were then cooled to a

temperature of -4°C and measured again to provide values for room temperature and frozen soil.

Table 3.9 Dielectric constants of fluids and soils materials (CRC Handbook of chemistry and physics 1993; Von Hippel 1954)

Fluids	Dielectric Constant	Solids	Dielectric Constant
Water	78.5 -80.4	Ice	3.7-4.1
Ethanol	24.3	Fused quartz (SiO ₂)	3.78
Acetone	20.7	Sandy soil (dry)	2.55
Ammonia	16.9	Loamy soil (dry)	2.51
Benzene	2.29	PVC	2.89
CO ₂ (liquid)	1.6	Polyethylene	2.25
CO ₂ (gas)	1.001	Teflon	2.1
Air	1.0	Wood	1.9-1.95

3.8.3 TDR CALIBRATION RESULTS

Figures 3.12 and 3.13 present the measured values of K_d versus soil moisture content for Soil A. Figures 3.14 and 3.15 present this data for Soil B. Calculations and data for calibration are available in Appendix A. Results indicated that the dielectric constant (K_d) varied greatly for different moisture content. Also, K_d changed dramatically for the frozen and unfrozen conditions for the same soil. TDR detected values of 5.3 and 25.3 for unfrozen Soil A and 4.2 and 6.9 when it was frozen, for saturations of 10% and 100% respectively. For Soil B, values of 5.4 and 26.7 were observed for the unfrozen condition and 4.5 and 7.1 were observed for the frozen condition, for saturations of 10% and 100% respectively. These results indicate that K_d of the soils under frozen condition was dramatically smaller than K_d under thawed condition and respond like dry soil, which implicitly indicate that the dielectric constant of ice equals that of air and liquid water coexists with ice in soils at subfreezing temperatures (Topp et al., 1980).

Figure 3.12 Percent saturation calibration curve for unfrozen Soil A.

Figure 3.13 Percent saturation calibration curve for frozen Soil A.

Figure 3.14 Percent saturation calibration curve for unfrozen Soil B.

Figure 3.15 Percent saturation calibration curve for frozen Soil B.

3.9 REVIEW OF ION CHROMATOGRAPHY

Analytical ion exchange chromatography is described by USEPA method 300, part A. An IC system is typically equipped with an auto sampler, high-pressure pump, electronically activated 6-port sample valve, and an analytical column packed with high capacity ion exchange material. The anion exchange column is used to separate anions, the eluent, which is a dilute solution containing anions, is pumped through the column to replace virtually all of the exchangeable ions in the column with the eluent anion and to establish a constant signal from the detector. Then a small volume of a sample is injected, and the resin column takes up the ions by exchange with an equivalent number of eluent ions.

After injection the eluent ions, which continue to be pumped through the column, compete with the sample ions for the exchange sites of the resin and cause the sample ions to move along the column. Due to differing affinities for the resin exchange sites, the various ions species in the sample move at different rates down the column and are separated. The detector senses when sample ions are passing through the cell and gives a signal that is proportional to their concentration.

Anions that could be present in a sample include fluoride, nitrite and bromide (Karmarkov, 1996), however, these anions are not present in appreciable amounts in biosolids. Of these anions, fluoride, nitrite, and small organic anions, such as formate and acetate, will be masked by the chloride peak if chloride is present in the sample. Therefore, the accuracy in a chloride determination will not be affected by these anions (Karmarkov, 1996).

Similarly, the presence of bromide in the sample would not affect the determination of nitrate. Phosphate and sulfate are well resolved. The rapid IC method defined by the USEPA method 300, part A, provides accurate and fast determination of chloride, nitrate, phosphate, and sulfate in the biosolids effluent sample, and can effectively detect very small concentrations for the samples.

3.9.1 DETERMINATION OF ANIONS BY ION CHROMATOGRAPHY

In this study, analytical ion exchange chromatography was used for the inorganic anions analysis. The method complied with USEPA method 300, part A. Chemically suppressed ion chromatography on a Lachat Instrument QuickChem 5000 was used. The system was equipped with an auto sampler, high-pressure pump, electronically activated 6-port sample valve, and an analytical anion column (Lachat Instrument Division®, QS-A1-5.5 X 150 mm) packed with high capacity ion exchange material. Eluent solution was prepared by mixing 5 mL of Sodium Carbonate Na_2CO_3 and 5 mL of Sodium Hydrogen Carbonate NaHCO_3 to 1 liter of distilled water. A short retention time for the anions was obtained by adjusting the rate to 2 mL /min.

3.9.2 CALIBRATION OF ION CHROMATOGRAPHY

For the analytes of interest, calibration standards were prepared at five concentration levels by adding accurately measured volumes of a the stock standard mixture to obtain concentrations of 150, 150, 100, 50, 25, 25, and 20 mg/L of SO_4 , HPO_4^- , Br^- , Cl^- , $\text{NO}_3\text{-N}$, $\text{NO}_2\text{-N}$, and F, respectively. The stock standards were added to a volumetric flask and diluted to the different concentrations with deionized water to obtain a total volume of 20

ml. Using automatic injections of 1 mL of each calibration standard, peak height and area responses were tabulated against concentration.

The results were used to prepare the calibration curve for each analyte. Calibration curves were verified whenever the anion eluent was changed. These plots are presented in Figure A-4 to A-10 in Appendix A.

3.9.3 ION CALIBRATION CURVE RESULTS

The linear equations of the anion calibration curves were reported in Appendix A. The achieved R^2 values for F^- , Cl^- , NO_2^- -N, Br^- , NO_3^- -N, HPO_4^{2-} , SO_4^{2-} were 0.999, 0.999, 0.998, 0.999, 0.998, 0.998, 0.988, respectively. Calibration curves performed with the rapid IC method defined by the USEPA method 300, part A, provided accurate and fast determination of chloride, nitrate, phosphate, and sulfate in the biosolids effluent samples, and could effectively detect very small concentrations for the samples.

3.10 AMMONIA MEASUREMENT

Ammonia was measured using the ammonia selective electrode method. Readings were measured using a VWR SympHony™ 14002-794 NH_3 probe. The dissolved ammonia (NH_3 in the aqueous phase and NH_4^+) is converted to aqueous NH_3 by raising the pH above 11 with 10 N of NaOH. Readings were recorded in millivolts, and the concentrations of NH_3 were calculated from the calibration curve.

3.10.1 CALIBRATION OF AMMONIA

A series of standard solutions covering the concentrations of 1000, 100, 10, and 1 mg NH₃-N/L were prepared by making decimal dilutions of a stock concentration of NH₄Cl solution with water. The electrode was calibrated by placing 100 ml of each standard solution in a 100–mL beaker, and the electrode was immersed around 3 minutes until it was stabilized. Using a spreadsheet, the ammonia concentration in milligrams NH₃-N per liter was plotted on the log y-axis versus potential in millivolts on the x- axis.

3.10.2 AMMONIA CALIBRATION RESULT

Figure 3.16 presents the ammonia calibration curve. The mean value of the mV readings versus concentration of standard ammonium solution were plotted on a log scale. The linear equation was reported to be $y = 109.7e^{-0.0405x}$ achieving a good correlation coefficient of 0.999.

Figure 3.16 Ammonia calibration curve, (mV versus concentration Log scale).

3.11 CHEMICAL OXYGEN DEMAND (COD)

Chemical oxygen demand (COD) was measured to find the oxygen equivalent of the organic matter content of the biosolids sample and the samples extracted from the columns. The samples were filtered through a 0.45 μm - membrane filter and then analyzed using the closed reflux, colorimetric method (method 5200 D, Standard Method). A Milton Spectronic 20D[®] spectrophotometer was used and set at 600 nm wave length, then absorbance was read and calculated from the prepared COD calibration curve.

3.11.1 CALIBRATION OF COD

Five standards were prepared from potassium hydrogen phthalate solution with COD values that ranged from 20 to 900 $\text{mg O}_2/\text{L}$.

3.11.2 COD CALIBRATION RESULT

Figure 3.17 presents the measured absorbance versus oxygen content for the COD calibration curve. The linear equation was found to be $y = 3002.8x - 36$ achieving a good correlation coefficient (R^2) of 0.991.

Figure 3.17 COD calibration curve.

3.12 REVIEW OF BACTERIOLOGICAL ANALYSIS

Biosolids are treated using different technologies to reduce pathogen concentrations to an acceptable level. However, some pathogens remain in the final cake product. Few methods have been reported for estimating the concentration of bacteria in soil and soil solution. However, a wide range of approaches are possible. Estimating bacterial movement in unsaturated soil could be made by either collecting soil samples over different depths (Natsch et al., 1996), or collecting water drained from the unsaturated zone using sampling devices that allow collection of soil solution but restrict soil particles from entering the samples. While the soil sampling may give very detailed information on spatial distribution (horizontal and vertical), it can offer only a snapshot of bacterial movement through the vadose zone (Unc et al., 2000). Once the soil samples are collected, the profile is disturbed and no more sampling can be performed at the depth location.

Simmons and Baker (1993) have used zero tension samplers to collect soil solution. These samplers have proven useful for macropores systems with a saturated flux. However, for collection of soil solution in unsaturated conditions, porous cup suction samplers have been widely employed. Porous suction samplers have mostly been used for the purpose of evaluating the movement of solutes through soil (Wood, 1973; Hansen and Harris, 1974).

Krejzl et al. (1996) tested different methods for monitoring bacterial transport in the vadose zone, including the use of ceramic porous cup suction samplers. Only 6 % of the coliform bacteria actually went through the ceramic cup walls. However, these samples were collected over long periods of time using constant suction, which can lead to significant changes in the filtration characteristics of the cups. Based on the above study and

considerations, porous ceramic cups samplers could be reasonably used for qualitative measuring movement and variation of bacteria from the unsaturated zone.

3.12.1 EXPERIMENTAL METHOD FOR BACTERIOLOGICAL ANALYSIS

The objective of the bacteriological analysis was to evaluate the movement of pathogenic indicators with the infiltrating water in the soil columns. In this test only indicator organisms were used. If these indicators were present, other organisms may be present at lower levels. Bacterial analysis performed in this phase of the study included two different methods, one for the soil solution and the second for solid cakes. Total coliform (TC) and fecal coliform (FC) were reported.

Bacterial culture of the soil solution extracted by ceramics cups was performed using a membrane filter method. 100 ml of sample were filtered on Membrane Filters (MF) that were grid-marked, had a 47-mm diameter, and $0.45 \mu\text{m} + 0.02 \mu\text{m}$ pore size. For fecal coliform determination, filter papers were placed on petri dishes filled with commercially prepared M-FC Agar. The product was mixed and rehydrated in 1 L water containing 10 mL 1% rosolic acid in 0.2N NaOH solution and incubated at $45 \pm 0.5^{\circ}\text{C}$. For the total coliform density determination, filters were placed on a commercially prepared M-Endo media and incubated at $35 \pm 0.5^{\circ}\text{C}$. Incubation time for total and fecal coliforms was 24 hours. The results were reported as the density of colony forming units per 100 ml of sample.

Biosolids (cake form) samples that were analyzed for total and fecal coliforms using the above method required dilution prior to analysis. In this method 30.0 ± 0.1 g of cake sample was weighted in a sterile dish. The samples were then transferred to a sterile blender and 270 mL of distilled water was added to rinse any remaining sample into the blend. The

samples were blended at low speed for 2 minutes. This was the “blended” sample. A volume of 1 mL of the “blended” sample contained 0.1 g of the original sample.

Three individual filtrations were conducted using 10^{-5} , 10^{-4} , and 10^{-3} mL of the original sample. A volume of 1 mL of the “blended” sample contained 10^{-1} g of the original sample. A sterile pipette was used to transfer 11 mL of the blender contents to 99 mL of distilled water and shaken. Next, 1 mL of dilution “A” was transferred to 99 mL of distilled water, and mixed as before. This solution “B” contained 10^{-4} grams of the original sample per mL. Similarly, 1 mL of dilution “B” was transferred to 99 mL of sterile dilution water, and mixed as before. This solution “C” contained 10^{-5} g of the original sample per mL. Next, colony densities were calculated and converted to $\log_{10}$ values to compute the geometric mean. Geometric means for the samples were determined by averaging the $\log_{10}$ values of the colony densities, and then calculating the antilog of this value.

4 BIOSOLIDS BATCH TESTS

Laboratory batch tests were conducted to examine freeze-thaw (F/T) effects on the physical and chemical characteristics of biosolids cake. Moreover, they examined the potential for release or mobilization of trace inorganic and organic solutes from the biosolids cake samples.

4.1 RESULTS AND DISCUSSION FOR THE BATCH TEST

4.1.1 QUALITATIVE OBSERVATIONS

After shaking the samples for one hour and before centrifuging samples, it was visually observed that the frozen cake samples had more dense particles than were observed in the fresh (unfrozen) samples. Particles in the frozen cake samples settled more rapidly than in the fresh samples. It was also observed during filtering that the filtrate from frozen samples filtered easier and faster than that from fresh samples. For filtering approximately 50 mL of filtrate, filters were changed twice for the fresh samples with small particles because of filter pore blockages. For frozen samples, only one filter was used to filter the same volume. These findings suggest that freezing biosolids cake enhances dewaterability and separation of solid particles from the liquid phase.

Another interesting finding was that freeze-thaw samples had a “coffee ground” appearance and were easily dewatered by gravity drainage, as compared to the fresh cake samples. When stirring of the fresh cake slurries was stopped, small solid particles remained suspended in the liquid phase and the slurry had a homogenous consistency. Particles did not separate or settle down as was observed with the freeze-thaw samples.

This trend has been seen in freeze-assisted sand drying beds (McFarland, 2000). Sludge with a jelly consistency was found to change to a granular-type material that was easily drained when subjected to freezing. The solid particles compressed into large, discrete conglomerates surrounded by ice crystals. These findings indicate that the thawed cake samples consist of more concentrated solid and organic matter particles, hence thawed samples will be very rich in nutrient value. For the fresh sample of biosolids, more of its solid particles will be suspended and available in the free water, and if the meltwater runs off the land this will contribute to the losses of more colloids particles within the meltwater when applied in natural conditions.

4.1.2 ANION CONCENTRATIONS

The impact of both time and freeze-thaw effect on the chemical concentrations of water extracts were evaluated in this experiment to examine the potential for release or mobilization of the solutes. The concentrations of the seven ions were determined for cake samples that were collected in three consecutive winter months, January, February, and March. Tables 4.1, 4.2, and 4.3 summarize the average and standard deviation of measured concentrations for fresh and frozen samples (25 g/100 mL) after 1, 14, and 28 days respectively. For detailed results of replicate concentrations for 25 and 50 grams of cake diluted in 100 mL of distilled water refer to Appendix B, Tables B.1 to B.6.

It is observed from Table 4.1 for the initial fresh samples that some of the ions remained almost constant for the period between January to March; standard deviations were 0.039, 0.022, 0.005, and 0.023 for fluoride, nitrite-N, bromide, and nitrate-N respectively whereas chloride, phosphate, and sulfate varied more (standard deviation of 0.755, 0.547, and 1.099, respectively). Changes in the influent characteristics entering the

wastewater treatment plant could be the reason for this variation. In addition, it is observed that initial fresh samples contained higher concentrations of chloride, phosphate and sulfate than fluoride, bromide, nitrite-N and nitrate-N. The comparison between the analysis of the fresh sample and the frozen sample in Table 4.1 to Table 4.3 shows that in general the concentrations of anions in the fresh samples were lower than those observed in the frozen samples. It can also be observed from the average extract concentrations for fresh and freeze-thaw samples in Tables 4.1, 4.2, and 4.3 that some of the extract concentrations remained relatively constant with time such as fluoride and bromide, and some other concentrations changed with time of storage such as nitrate-N, nitrite-N, sulfate, and phosphate.

Table 4.1 Anion concentrations in extracts after 1 day.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 25 g/100 mL</u>						
		F ⁻	Cl ⁻	NO ₂ ⁻ -N	Br ⁻	NO ₃ ⁻ -N	HPO ₄ ²⁻	SO ₄ ²⁻
<u>Fresh</u>	January	0.259	2.591	0.107	0.227	0.169	1.065	5.472
	February	0.333	3.635	0.082	0.231	0.130	2.155	3.427
	March	0.320	4.058	0.126	0.222	0.127	1.691	5.145
	Average	0.304	3.428	0.105	0.227	0.142	1.637	4.681
	Std. Dev.	0.039	0.755	0.022	0.005	0.023	0.547	1.099
<u>Frozen</u>	January	0.309	3.694	0.128	0.298	0.164	2.081	5.918
	February	0.471	5.447	0.134	0.306	0.127	4.030	6.000
	March	0.396	4.855	0.147	0.317	0.196	3.400	5.482
	Average	0.392	4.665	0.136	0.307	0.163	3.171	5.800
	Std. Dev.	0.081	0.892	0.010	0.010	0.034	0.995	0.278

Table 4.2 Anion concentrations in extracts after 14 days.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 25 g/100 mL</u>						
		F ⁻	Cl ⁻	NO ₂ ⁻ -N	Br ⁻	NO ₃ ⁻ -N	HPO ₄ ²⁻	SO ₄ ²⁻
<u>Fresh</u>	January	0.263	3.111	0.098	0.212	0.159	1.112	3.257
	February	0.339	3.245	0.121	0.222	0.173	2.813	4.569
	March	0.331	2.997	0.108	0.235	0.160	2.154	4.222
	Average	0.311	3.118	0.109	0.223	0.164	2.027	4.016
	Std. Dev.	0.042	0.124	0.012	0.011	0.008	0.858	0.680
<u>Frozen</u>	January	0.315	4.452	0.131	0.306	0.358	3.053	5.725
	February	0.481	3.494	0.137	0.314	0.382	4.998	5.463
	March	0.412	5.610	0.151	0.321	0.356	4.506	5.022
	Average	0.403	4.519	0.140	0.314	0.365	4.186	5.403
	Std. Dev.	0.083	1.060	0.010	0.008	0.014	1.011	0.355

Table 4.3 Anion concentrations in extracts after 28 days.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 25 g/100 mL</u>						
		F ⁻	Cl ⁻	NO ₂ ⁻ -N	Br ⁻	NO ₃ ⁻ -N	HPO ₄ ²⁻	SO ₄ ²⁻
<u>Fresh</u>	January	0.256	3.877	0.105	0.248	0.168	1.224	3.003
	February	0.335	3.210	0.094	0.263	0.162	2.727	3.903
	March	0.311	2.473	0.070	0.255	0.165	2.598	3.690
	Average	0.301	3.187	0.090	0.255	0.165	2.183	3.532
	Std. Dev.	0.041	0.702	0.018	0.007	0.003	0.833	0.470
<u>Frozen</u>	January	0.321	4.377	0.133	0.311	0.311	3.120	5.803
	February	0.485	5.673	0.141	0.318	0.327	5.626	5.642
	March	0.410	4.089	0.151	0.325	0.373	4.772	5.143
	Average	0.406	4.713	0.142	0.318	0.337	4.506	5.529
	Std. Dev.	0.082	0.844	0.009	0.007	0.032	1.274	0.344

Figure 4.1 was presented to illustrate the general trend of the results presented in Tables 4.1 to 4.3. It is clearly observed that the extract concentrations of the anions from the frozen samples were higher than those measured from the fresh samples at all time.

Figure 4.1 Average anion concentrations for fresh versus freeze-thaw samples with respect to time.

A t-test was performed on the data from the fresh and freeze-thaw samples to determine if there was a significant difference between the concentrations for fresh and freeze-thaw conditions. A significance level of 0.05 was employed. Table 4.4 shows the results of the t-test analysis performed on the data. All detailed calculations are presented at the end of Appendix B.

Table 4.4 T-statistic significance at the 0.05 critical alpha levels from fresh and freeze/thaw samples

Parameter Fresh versus Frozen	Statistically significant P (T<=t) two-tail
	<u>25 gr/100mL</u>
Fluoride (1 Day)	No significant difference
Fluoride (14 Days)	No significant difference
Fluoride (28 Days)	No significant difference
Chloride (1 Day)	No significant difference
Chloride (14 Days)	No significant difference
Chloride (28 Days)	No significant difference
Nitrite-N (1 Day)	No significant difference
Nitrite-N (14 Days)	No significant difference
Nitrite-N (28 Days)	No significant difference
Bromide (1 Day)	Significant difference
Bromide (14 Days)	Significant difference
Bromide (28 Days)	Significant difference
Nitrate-N (1 Day)	No significant difference
Nitrate-N (14 Days)	Significant difference
Nitrate-N (28 Days)	Significant difference
Phosphate (1 Day)	Significant difference
Phosphate (14 Days)	Significant difference
Phosphate (28 Days)	Significant difference
Sulfate (1 Day)	No significant difference
Sulfate (14 Days)	No significant difference
Sulfate (28 Days)	Significant difference

The t-statistic for the fresh and freeze-thaw samples presented in Table 4.4 indicates that the concentrations of fluoride, chloride and nitrite-N in the fresh and freeze-thaw samples in the 25 g /100 mL solutions were not statistically different at any time

(5% level) based on t-test. The difference between the concentrations of bromide and phosphate for all times for the 25 g /100 mL solutions were statistically significant (at the 5% level). Bromide and phosphate increased by 35 % and 93%, respectively from the fresh conditions to the freeze-thaw conditions. The results suggest that under freeze-thaw conditions the mobility of bromide and phosphate from the cake when subjected to wash off will be greater than that from the fresh cake.

After freeze-thaw, sulfate concentrations in the extract did not change significantly. However, it is seen in Figure 4.1 that with increasing time, the concentration of sulfate in the fresh samples decreased to a greater extent than in the frozen sample where no change was realized with time (after 28 days). This significant difference after 28 days suggests that some biochemical reactions occurred in the fresh sample and the subzero temperature preserved the samples from reactions. Sulfate in the fresh sample was likely reduced to sulfide. No differences in concentrations for Nitrite-N were observed. However, concentrations of nitrate-N increased in the 28 day in frozen samples. Increases in nitrate-N suggest that some nitrification occurred when the samples were stored over the 28 days period of time. The 93% increase in phosphate concentration in the frozen samples suggests that some microorganisms died off and contributed to this observed increases in the solution. These observed increases of phosphate were also seen in the literature review when microorganisms died off (Macleod and Calcott, 1976).

It is believed that water in biosolids is present as free water, interstitial, vicinal water, and water of hydration (Vesilind, 1974). The easiest water to remove from wet biosolids is free water, which is usually eliminated by gravity. The interstitial water

trapped in the crevices, interstitial spaces of the flocs, and organisms, requires destruction or compression of floc structures using sufficient mechanical energy to squeeze out the water. In this study, 70% weight of biosolids cakes was water, and the higher centrifugation forces exerted on the cake samples removed the excessive interstitial water which remaining in the cake. Centrifugation, however cannot remove vicinal water defined as the multiple layers of water molecules held tightly to the particles surface by hydrogen bonding and even the water chemically bound to particles or water of hydration as defined by Vesilind (1974).

Based on the above observation, the changes in ion concentrations with respect to time and temperature are ascribed to the following process: Biosolids collected from ROPEC contained residual interstitial water, vicinal and water of hydration. The ions analyzed in this experiment were soluble in the liquid phase. When freeze-thaw was applied to the samples and temperature dropped below the melting point, some of this water froze as pure ice resulting in expulsion and rejection of the ions from the ice to the unfrozen water that remained in the cake. This process resulted in the anion concentrations presented in Figure 4.1. By adding the deionized water (100 ml) to the frozen sample and shaking for one hour, the easily available ions entrapped in the cake were released in the free water and thus the concentration increased.

Several authors described this process that begins when snowflakes respond to freezing and thawing cycles by metamorphosing (when ice crystals enlarge and round) (Colbeck, 1981; Schondorf and Herrmann, 1987). The ice crystal lattices do not allow impurities to be incorporated, so the impurities migrate to the outside of the crystal. They are loosely bound in this position and thus exposed for wash off by passing meltwater.

In addition, the comparison between the analysis of the fresh sample and the frozen sample showed that in general the concentration of anions were lower in the fresh sample than those in the frozen samples especially for anions such as SO_4^{-2} and HPO_4^{-2} . In addition, concentrations of some anions did not change. This may be explained by the fact that aerobic bacteria are active in the fresh sample and are dormant in the frozen sample, accordingly active bacteria consumes some anions as nutrients during their growth in the fresh samples (Schlegal, 1995).

It was concluded that the effect of freeze-thaw on biosolids cake was to contribute to the release of more solutes into solution. In addition, bacteria that were dormant in the frozen samples did not consume the ions as nutrients for their needs, and cake samples, rich in nutrients, were preserved under frozen condition. With increasing time, active bacteria in the fresh samples may have consumed the nutrients of the fresh cake samples which decreased the anions concentrations.

4.1.3 AMMONIA CONCENTRATIONS

The samples collected from the cake extracts were also analyzed for ammonia. Table 4.5 presents the ammonia concentrations of the fresh and frozen samples that were measured in the extracts for 25 g / 100 mL tests. Data for the 50 g / 100 mL tests are given in Table B.10 of Appendix B. In the analysis, all the ammonium was converted to ammonia in the aqueous phase by raising the pH above 12. Concentrations were calculated from the calibration curve and means were reported.

Table 4.5 Fresh and frozen samples for ammonia concentrations based on 25 g calculations.

Ammonia (mg/L) for 25 g / 100 ml					
		Concentration mg/L (range)	Mean	Concentration mg/Kg (range)	Mean
<u>FRESH SAMPLES</u>					
January					
1 day		760 - 763	762	3040 - 3052	3046
14 days		687 - 693	690	2748 - 2772	2760
28 days		671 - 673	672	2684 - 2692	2688
February					
1 day		611 - 618	615	2444 - 2472	2458
14 days		566 - 575	571	2264 - 2300	2282
28 days		548 - 557	553	2192 - 2228	2210
March					
1 day		596 - 601	599	2384 - 2404	2394
14 days		526 - 541	534	2104 - 2164	2134
28 days		520 - 530	525	2080 - 2120	2100
<u>F/T SAMPLES</u>					
January					
1 day		763 - 766	765	3052 - 3064	3058
14 days		770 - 773	772	3080 - 3092	3086
28 days		788 - 792	790	3152 - 3168	3160
February					
1 day		611 - 621	616	2444 - 2484	2464
14 days		613 - 618	616	2452 - 2472	2462
28 days		628 - 634	631	2512 - 2536	2524
March					
1 day		604 - 608	606	2416 - 2432	2424
14 days		626 - 631	629	2504 - 2524	2514
28 days		647 - 649	648	2588 - 2596	2592

In January, higher extract concentrations (762 mg/L) were observed than in February (615 mg/L) and March (599 mg/L). Variation of the temperature from January to March before sampling could be the reason for this variation permitting some ammonia to volatilize, or because of changes in influent characteristics entering the wastewater treatment plant. Ammonia in the fresh sample extracts slightly decreased over time. The concentration in the fresh samples of the January month changed from 762

mg/L to 672 mg/L (13.5 % reduction) after 28 days while in February, the concentration decreased from 615 mg/L to 553 mg/L (11 % reduction) after 28 days and for March, the concentration decreased from 599 mg/L to 525 mg/L (14 % reduction) after 28 days.

Frozen samples when stored, increased very slightly the ammonia concentration (Table 4.5). In January, the concentration increased by 10.5% after 14 days and by 15% after 28 days. In February, the concentration increased by 7.5% after 14 days and by 12.5% after 28 days. In March, the concentration increased by 15% after 14 days and by 19% after 28 days.

The t-statistic comparing the fresh and freeze-thaw samples given in Table 4.6 indicates the significance of this concentration change for ammonia. The differences between the concentrations of ammonia in fresh versus frozen samples measured over time for the 25g / 100 mL solutions were statistically significant (5% level) based on t-test.

Table 4.6 T-statistic significance at the 0.05 critical alpha levels for ammonia.

Parameter Fresh versus Frozen	Statistically significant P (T<=t) two-tail
	<u>25 g/100mL</u>
Ammonia (1 Day)	0.021
Ammonia (14 Days)	0.001
Ammonia (28 Days)	0.000

concentrations increase in the sample extracts indicating that freeze-thaw has the same effect for ammonia as for the other anions. The ice crystals lattice did not allow ammonia to be incorporated, so the ammonia migrated to the outside of the crystal, and thus was exposed for wash off from the solid part of the cake.

4.1.4 PH RESULTS

Table 4.7 summarizes the pH values from fresh and frozen samples that were measured in the extracts for 25 g / 100 mL tests. pH was measured to determine if it could explain the loss of NH_3 . The results obtained showed that pH did not appreciably change with state and time. The pH throughout the 28 days was 7.9 ± 0.24 under fresh conditions and 8.1 ± 0.11 under freeze-thaw conditions. These results would indicate that ammonia was not volatilized.

Table 4.7 pH values for 25 g /100 mL biosolid samples.

<u>pH for 25 g / 100 mL</u>		
	<u>Fresh</u>	<u>Freeze-thaw</u>
January		
1 day	8.07 - 8.08	8.17 - 8.17
14 days	7.65 - 7.65	8.15 - 8.14
28 days	8.07 - 8.09	8.14 - 8.14
February		
1 day	8.09 - 8.09	8.09 - 8.08
14 days	8.13 - 8.11	8.16 - 8.15
28 days	8.12 - 8.12	8.16 - 8.16
March		
1 day	7.73 - 7.74	7.93 - 7.95
14 days	7.96 - 7.95	8.02 - 8.06
28 days	8.04 - 8.07	8.09 - 8.11

4.1.5 COD RESULTS

The extract samples were also analyzed for COD. Table 4.8 shows soluble COD values recorded over the 3 months for fresh and frozen samples that were measured in the extracts for 25 g / 100 mL tests. It is observed that COD concentrations in the extracts decreased with time. After one day of storage of fresh cake, 6046 mg/Kg soluble COD was produced for January, 6464 mg/kg for February, and 6004 mg/kg for March. After

14 days of storage, the COD values of the fresh cake extracts were approximately the same as after one day of storage. However, after 28 days of storage of fresh cake reductions of 19%, 14%, and 16.5% were observed for January, February, and March, respectively.

Table 4.8 Fresh and freeze/thaw COD concentrations based on 25 g/100 mL calculations.

	<u>COD (mg/L) for 25 g / 100 mL</u>			
	Concentration mg O ₂ /L (range)	Concentration mg/Kg (range)	Mean mg/Kg	
<u>FRESH SAMPLES</u>				
January				
1 day	1492 - 1531	5968 - 6124	6046	
14 days	1501 - 1516	6004 - 6064	6034	
28 days	1273 - 1261	5092 - 5044	5068	
February				
1 day	1607 - 1625	6428 - 6500	6464	
14 days	1495 - 1507	5980 - 6028	6004	
28 days	1432 - 1396	5728 - 5584	5656	
March				
1 day	1510 - 1492	6040 - 5968	6004	
14 days	1429 - 1390	5716 - 5560	5638	
28 days	1297 - 1282	5188 - 5128	5158	
<u>F/T SAMPLES</u>				
January				
1 day	1180 - 1162	4720 - 4648	4684	
14 days	1147 - 1108	4588 - 4432	4510	
28 days	895 - 862	3580 - 3448	3514	
February				
1 day	1219 - 1192	4876 - 4768	4822	
14 days	1171 - 1198	4684 - 4792	4738	
28 days	1129 - 1183	4516 - 4732	4624	
March				
1 day	1228 - 1267	4912 - 5068	4990	
14 days	1183 - 1192	4732 - 4768	4750	
28 days	1048 - 1036	4192 - 4144	4168	

The results of the t-test comparing the fresh and freeze-thaw samples presented in Table 4.9 indicated the significance of the changes at the different times. The differences

between the concentrations of the raw and freeze-thaw samples measured at different times for the 25 g / 100 mL solutions were statistically significant (5% level) based on t-tests.

Table 4.9 T-statistic significance at the 0.05 critical alpha levels.

Parameter Fresh versus Frozen	Statistically significant P(T<=t) two-tail
	<u>25 g/100mL</u>
COD (1 Day)	0.000
COD (14 Days)	0.000
COD (28 Days)	0.000

The extracts from the freeze-thaw samples were consistently lower than those of the fresh samples. In January, the COD concentration decreased 23% from 6046 to 4684 mg/Kg. After 14 and 28 days, the differences between the fresh and the freeze-thaw samples were 25% and 31%, respectively. In February, the COD concentration decreased by 25.5% from 6464 to 4822 mg/Kg. After 14 and 28 days, differences between the fresh and the freeze-thaw samples were 21% and 18% respectively. In March, the COD concentration decreased by 17% from 6004 to 4990 mg/Kg. After 14 and 28 days, the differences between the fresh and the freeze-thaw samples were 16% and 19%, respectively.

The high COD in the fresh sample compared to the frozen samples may be accounted for by the difference in the bacteria activity in fresh and frozen samples. Biomass was still active in the fresh sample. It is believed that most of the COD in the cake sample were in solid forms, bacteria secrete extra-cellular enzymes that break down the large solids particulates into reduced and soluble forms that they can use as nutrient sources. Whereas in the frozen samples active bacteria were dormant and were a smaller

portion of the solid, and solids particles were reduced to less soluble forms (Schlegel, 1995).

4.1.6 BACTERIOLOGICAL ANALYSIS

Total coliform (TC) and fecal coliform (FC) counts were not performed for January, February and March. A sample of cake was collected in April from the wastewater treatment plant and investigated under freeze-thaw condition. The plate count results and associated geometric means are presented in Tables B.13 and B.14 of Appendix B.

It was found that freeze-thaw was lethal to fecal coliform and that the subfreezing temperature inactivated all the fecal coliform in the samples. The geometric mean decreased from 8.11×10^4 to below the detection limit for the fecal coliform. In addition, the total coliform concentrations measured in the fresh samples had a geometric mean of 1.89×10^6 CFU/g, and after the freeze-thaw process, the total coliform concentration was slightly reduced to 7.89×10^5 CFU/g. These findings suggest that storing or spreading biosolids under frozen weather condition will substantially reduce the fecal coliform concentrations, however the total coliform would resist and survive frozen weather conditions.

4.2 CONCLUSION FROM THE BATCH TESTS

The ability of biological and colloidal systems to hold a quantity of water with great forces of attraction has been long recognized. This attraction, when exhibited towards water, results in hydration of the particles and produces a phenomenon known as

water binding (Kantardjieff et al., 2000). Based on the above results, it was concluded from the changes in ion concentrations with respect to time and temperature that chloride, fluoride, bromide, nitrite, and nitrate were released to a greater extent than sulfate and phosphate during freeze-thaw. Ammonia was also released to a greater extent. The ice crystal lattice formed in the cake does not allow anions to be incorporated, so the ions migrate to the outside of the crystal. They are loosely bound in this position and thus exposed for wash off by passing meltwater. Also, it was found that freezing biosolids cake enhances dewaterability and separation of solid particles from the liquid phase. In addition, dormant bacteria in the frozen samples did not consume the available anions. The cake samples were preserved under frozen condition. With increasing time, active bacteria in the fresh samples consumed the nutrients of the fresh cake samples which decreased the anion concentrations. The low soluble COD in the frozen sample compared to the fresh samples were attributed to the difference in the bacteria activity in fresh and frozen samples. This would result in less solute in the frozen samples hence less contaminated water. It was also found from the bacteria indicator tests that subfreezing temperature would substantially reduce fecal coliform concentrations in frozen weather conditions.

Although, the ion concentrations after freeze-thaw of the samples were small, phosphate concentrations were significantly large, as a result consideration and better land application management practices should be assigned for phosphate.

5 COLUMNS TEST

6 SUMMARY

This part of the study was performed to evaluate the potential infiltration of the biosolids losses into the subsurface in terms of mobile solutes. It is believed that chemicals added to a soil from the surface may infiltrate and leave the zone of incorporation by leaching. The main objective of the experimentation was to sample percolating water from various depths while soils thawed. Three different monitoring devices were employed at different heights. These included wave-guide rods (TDR) for percent saturation measurement, thermocouple wires for temperature measurement, and ceramic cups for sampling the soil solution.

6.1 HYDROLOGICAL CONDITIONS

Initially moisture and water movement in the columns were investigated, and then water budgets were performed and data on concentrations of contaminants were employed to evaluate the losses of the chemicals released from the applied biosolids cake. The length of the test was 21 days and it was divided into 7 periods. Water was collected in alternating sampling events with samples collected from Port 1 and Port 3 in the first period, and from Ports 2 and Ports 3 in the alternate period. At the end of the experiment, samples of biosolids from the top of the column were analyzed to facilitate mass balance calculations.

6.1.1 RESULTS OF TEMPERATURES AND MOISTURE CONTENTS

During the experiment, temperatures were recorded to determine whether the soil was frozen in order to ensure that the appropriate calibration curve was employed for the TDR readings. Figure 5.1 illustrates the temperature profiles for Soils A and B respectively as a function of depth and time on the first day of the test. Similar temperature profiles were seen for the other 4 columns of the two soils. For the mixed Soil B, the temperature increased slightly more slower than in the sandy Soil A. As the water-holding-capacity of the soil increases, more energy is required to raise its temperature. This was attributed to the fact that water-holding capacity is controlled primarily by soil texture and organic matter. Soils with smaller (silt and clay) particles such as Soil B have a larger surface area than those with larger sand particles (Sand A), and a large surface area allows a soil to hold more water. In other words, a soil with a higher percentage of silt and clay particles has a higher water-holding capacity.

Figure 5.1 Temperature profile versus depth and time for the thawing Soil A and Soil B

It is believed that the flow of heat in porous and fractured rocks is dominated by thermal convection and thermal conduction (Looney and Falta, 2000). In this study the temperature started to increase in the columns after 3 hours of observation and this was likely due to heat input from the perimeter of the columns. The results differ from that which would be expected from the field for several reasons. In the field lateral heat transfer is likely minimized. Moreover, in natural conditions when the temperature starts to rise and the snowpack starts to melt, the soil is still covered with snow that behaves as a heat insulator (Perfect and Williams, 1980). At 0° (273 K) the thermal conductivity of snow is 0.049 W/mK. This relatively small thermal conductivity under natural conditions will not allow the soil to thaw quickly from the top. In the experimental columns, the energy from the room was directly transferred to the soil surfaces of the columns, thereby increasing the heat transfer rate as compared to natural conditions.

The raw data employed to determine percent saturation for the six columns are presented in Appendix C, Tables C-1 to C-12. These tables present the percent saturation data as a function of depth and time. Figures 5.2 and 5.3 illustrate the percent saturation as a function of depth and time for Soil A and Soil B, respectively. The percent saturation curves of the frozen columns were plotted on the left side of the figure and the percent saturation curves of the thawed columns were plotted on the right side of the figure. It is generally observed that the percent saturation curves were reproducible. The initial percent saturation values at the surface of the frozen columns A, B, and C were 21.1%, 22%, and 21.4% respectively. The values on the top surface 5 minutes after the addition of water ($t = 5$ min.) were 58.4%, 56.7% and 44.7% for Columns A, B and C, respectively. The observed variation was due to the biosolid cakes that adsorbed a portion

of the SWE (49 g and 1000 g of biosolids were applied on Column B and C, respectively).

Figure 5.2 Saturation for Soil A versus time and depth for frozen and thawed Column A, B, and C.

Figure 5.3 Saturation for Soil B versus time and depth for frozen and thawed Column D, E, and F.

It was observed from the temperature profile of Soil A in Figure 5.1 and the percent saturation in Figure 5.2 that at -7 cm depth and at time = 0, the temperature of Column A was -10°C and that the percent saturation (ice + liquid water) at -7 cm was at 21.1%, and at the lower bottom of the soil (-85 cm), the percent saturation was 67.6%.

At a time of 5 minutes after pulsing the column with 280 ml of SWE, the temperature at -7 cm from the surface changed from -10°C to -9.3°C . The response from the TDR indicated a value for K_d of 11.8. When this K_d value was employed in the frozen calibration equation, it indicated a percent saturation of 225% which was clearly unrealistic. When the same value was employed in the unfrozen calibration curve it indicated a percent saturation of 58.4%. This was considered to be more realistic. Hence, this dielectric constant was interpreted to be applicable. This approach assumes that the water content is predominantly in the liquid form.

The same pattern was observed for the other columns. The percent saturation at depth of -7 cm changed from 22% to 56.4% for Column B, and from 21.4% to 44.7% for

Column C after 5 minutes, and no change in percent saturation was observed for the lower depths in all the columns.

At a time of 45 minutes and at a depth of -7 cm the temperature of Column A was -6.2°C and percent saturation decreased to 51.6%. The percent saturation decreased from 56.4% to 50.4% in Column B, and from 44.7% to 40.1% in Column C. No change in percent saturation was observed for the lower depths (-85 cm) in all the columns. Here, with increasing time, the moisture profile curve begun to move back to its initial condition, and the temperature rose to above 0°C .

When the second dosing of SWE with a volume of 840 mL was applied on the fourth day, the soil was completely thawed. The graphs on the right side of Figures 5.2 and 5.3 present the moisture curves in a thawed conditions for Soil A and B, respectively. It was observed from Figures 5.2 that the differences in percent saturation at the same time were larger than those observed when the soils were in frozen state and that the pulse of water moved faster in the soil than what was observed when the soils were frozen.

At $t = 5$ minutes, the infiltrated water infiltrated to -35 cm. And percent saturation changed from 25.1% to 74% in Column A, from 23.9% to 73.8% in Column B, and from 25.9% to 72.4% in Column C. No change was seen for the lower depths (-85 cm) in all of the columns. The larger values of the percent saturation when the columns were thawed were due to the fact that on the fourth day a larger amount of water was applied. These larger amounts of water increased the initial saturation at $t = 0$. In addition it was found that the water infiltrated into a partially saturated frozen soil but to lower depths than into a thawed soil.

The same behavior was seen for the mixed Soil B in the other three columns (D, E, and F) with the exception that the initial saturation in Soil B was higher than what was found in Soil A. In Figure 5.3 the average percent saturation of the frozen soil of Columns D, E, and F were 23.4% at $t = 0$, 57.3% at $t = 5$ min. and 51.4% at $t = 45$ minutes, and with increasing depth (-85 cm) no significant change of percent saturation were realized. When the columns were completely thawed, the average percent saturation were 28.1%, 79.8%, and 60.4% at $t = 0$, 5 and 45 minutes, respectively. The results also indicate that the SWE could infiltrate in a partially saturated soil in a frozen condition.

To evaluate the accuracy of the TDR readings, the amount of water in each segment (Figure 5.2) at each time increment for each column was calculated. At each time, the difference of the percent saturation relative to the initial saturation at time equal zero was calculated by subtracting the area under the two curves. Then, the distances between the TDR probes were multiplied by the cross sectional area, the porosity and the percent saturation of the soil. These calculations are available in Table C-19 to C-24 of Appendix C.

In Column A the total volume of water was 2134 mL at $t = 0$. After adding 280 mL, the total volume was estimated to increase to 2445 mL after 5 minutes via the TDR. This showed a difference of 311 mL, which was 31 mL more than the SWE (280 mL). This was likely due to the fact that the probe was not inserted at the top of the column and it was therefore assumed that the soil percent saturation was constant from zero cm to -7 cm. The differences in total volume at the different sampling times for the top sector of Column A were 312, 289, 305, 300, 258 mL after 5 min., 45 min., 3 hours, 5 hours, and 10 hours, respectively. These differences indicate the accuracy of the TDR

measurement (285 ± 27 mL). It was also seen that the water infiltrated to the second TDR segment (-17 cm) after 5 minutes and moved down the soil column with a very slow rate. Similar moisture profiles were observed in Column B and C, respectively.

The same pattern was seen for columns D, E, and F that contained Soil B with the exception that higher initial volumes were found in Soil B where the initial saturation was 2416 mL. The differences in water volume after applying 280 mL of SWE, were 341, 294, 312, 347, 284 mL after 5 min., 45 min., 3 hours, 5 hours, and 10 hours, respectively. This indicated that at subzero temperatures, the infiltrated meltwater does not freeze and water can percolate in a partially saturated frozen soil, (Kamarov and Makarova, 1973; Steenhuis et al., 1981; Granger et al., 1984; Gray et al., 1986; Sullivan et al., 2000; Zhao et al., 2001).

In this experiment, the initial water content in Soil B was higher than in Soil A. The percent saturation in the columns before adding the SWE at time = 0, agrees with the literature. Figure 5.4 illustrates typical moisture curves profile for gravity drainage where saturation (S_w) varied from 1 to partial saturation with respect to depth (h) (Looney and Falta, 2000).

Figure 5.4 Gravity drainage equilibrium for different layer soil, *Looney and Falta 2000*[©]

This was attributed to the fact that soil texture greatly influences water content in the columns. The sandy soil was quickly recharged with soil moisture but was unable to hold as much water as the soils with heavier textures as Soil B. Because soils such as Soil B has narrower pore spacing that holds water more tightly.

6.1.2 RESULTS OF WATER BUDGET

The volumes of simulated SWE that were applied onto the surfaces of the six columns were presented in Table 3.1 of Section 3.2.2. The volumes of effluent that were extracted from the three ports of the six columns are reported in Table C-43 to C-54 of Appendix C. During each sample event, approximately 100 ± 20 ml were extracted from Port 1 and Port 2, and 700 ± 50 mL were extracted from Port 3.

The water budgets presented in Tables C-43 to C-54 indicate acceptable water mass balances with recoveries ranging from 89% to 96.5%. No losses of water from the columns were observed in the experiment. The water budget was calculated in terms of the differences between the volumes of meltwater applied and the volumes of effluent extracted by the ceramic cups. The slight variations in water budget were due to some water that remained in the soil by surface tension forces and due to some amount of water that remained in the tubes of the sampling system.

It was also observed in Columns C and F (1 kg cake) for the first 840 mL SWE application, that 756 mL (90%) and 749 mL (89%) were recovered, respectively. These volumes were much lower than the volumes recovered from the other four columns were 803 mL (95.5%), 810 mL (96.5%), 806 mL (96%) and 791 mL (94%) for columns A, B,

D, and E respectively. This indicates that approximately 90 mL (10.5%) of the snow water equivalent was absorbed in the 1 kg of cake and suggests that biosolids cakes can behave as mulch. Cakes absorb water due to their low moisture content ($\geq 30\%$ solids).

It was also observed that the total volume of water stored in the Soil A was less than the volume stored in the Soil B. Average volumes collected from Soil A and B were 5571 mL and 5488 mL, respectively. During the experiment, it was easier to remove water from the Soil A than from the Soil B. This was attributed to the fact that the water in the Soil B was held in very small capillary spaces within the soil particles under higher soil suction, whereas the Soil A holds water in large capillary spaces under lower soil suction (Looney and Falta, 2000).

6.1.3 CONCLUSION

It was concluded from the experiments that no significant differences were observed between Soil A and Soil B for water movement. However water infiltrated slightly quicker in the Soil A than in the Soil B and due to the gravitational forces the infiltrated water could move into the partially saturated frozen soil having porosity from approximately 0.38 to 0.39. The water moved to only a depth of 17 cm when the temperature was below the melting point. In addition, it was concluded that the cover of the biosolids cake behaved as mulch and absorbed a portion of the meltwater volume. For a natural field condition this would suggest that when the snow cover starts to melt and due to the characteristics of the biosolids cake ($\leq 30\%$ solids), the initial volume of melted water will be adsorbed in the cake. In the meantime when the temperature of the soil reaches 0°C the frozen surface of the soil will start to thaw and the meltwater will

infiltrate rather than running off to the surface water. Meeting the objectives of the winter land application practices, spreading high-solid cakes will minimize the detrimental effect of surface contamination from the solutes lost with the slow rate of release of meltwater in springtime.

6.2 BIOSOLID SOLUTE LOSSES AND TRANSPORT

Contaminants can be present in soils in all three phases liquid, solid, and gaseous. The main processes that affect contaminants in the liquid phase are advection, dispersion, sorption-desorption, ion exchange, and decay reactions (Looney and Falta, 2000). In this part of the experiment, monitoring of contaminants transported with the meltwater was accomplished by measuring the chemical concentrations in the soil solution extracted from the columns at different times and depths.

The extracted solutions were analyzed for chloride, fluoride, bromide, nitrite-N, nitrate-N, ammonia, phosphate, sulfate, COD, and bacterial indicator organisms. The average concentrations were multiplied by the water volumes for mass balance calculations. The concentrations were compared with the maximum concentration limits for drinking water set by EPA.

6.2.1 RESULTS OF THE COLUMN EFFLUENT CONCENTRATIONS

Figures 5.5 to 5.13, previously introduced, show the concentrations that were extracted from the six columns for chloride, fluoride, bromide, nitrite-N, nitrate-N, ammonia, phosphate, sulfate, and COD, respectively. Each plot represents the concentration of the solutes in the soil solution versus time and depth. Port 1, Port 2, and Port 3 correspond to depths of -10 cm, -60 cm, and -95 cm respectively. Columns that

contained Soil A were plotted on the left side of the figures and columns that contained Soil B were plotted on the right side of the figures. Numerical results for the volumes and chemical concentrations extracted from Ports 1, 2, and 3 are presented in Tables C-98 to C-138 in Appendix C.

It can be generally observed from the plots of Figures 5.5 to 5.13 that the magnitude and peaking patterns of the solute concentrations, represented from the curves, had different aspects. It can be also observed from the control Columns A and D of the plots that small concentrations of some ions were present in the unamended soils. Chloride was only detected from Port 3 for the unamended Soil A and the Soil B. Phosphate and sulfate were found in the extracts from the unamended Soil B. Ammonia, bromide, nitrite-N, and nitrate-N were not detected in the control columns. The results suggest that when the columns were filled with tap water and then with distilled water to stabilize the soils columns, some ions remained in the soil and were not completely washed from the soils.

It is also indicated from the results that for the two soil types, the columns loaded with biosolids yielded higher concentrations of solutes than the control columns. Solute concentrations from Columns C and F which were loaded with 1000 g of biosolids were significantly higher than that found from Columns B and E that were loaded with 49 g of biosolids (Figures 5.5 to 5.13). This indicates that an increase in biosolids application rates, increases the correspondingly solute concentrations.

From Figure 5.5 it is observed for Column B, where 49 grams of frozen cake were applied, that the initial chloride concentration from Port 3 on day 3 was approximately the same as for the control Column A. It is also observed that the peak 0.282 mg/L of

chloride was detected from Port 1 at day 4 and with time the concentration decreased to 0.03 mg/L after 16 days. On day 7, 0.387 mg/L was detected from Port 2 and with time the concentration decreased to 0.02 mg/L. From Port 3, 0.395 mg/L appeared on day 9, and with time the concentration decreased to 0.03 mg/L. And it is observed that the peak concentration appeared first from Port 1 on day 4, then on the curve of Port 2 on day 7, and later at the bottom Port 3 on day 9. In addition, for the concentrations of Port 3 it is observed that a sudden increase appeared on day 9. Chloride was 0.064 mg/L on day 6, 0.395 mg/L on day 9, and decreased again to 0.140 mg/L on day 12. After day 12 concentrations started to decrease from the three Ports 1, 2, and 3. This peaking pattern was seen for Soil B in Column E but with slightly lower concentrations. The peak concentrations were 0.252 mg/L, 0.382 mg/L, and 0.353 mg/L from Port 1, Port 2, and Port 3 respectively.

Concentrations of chloride from both soils, in Column C and F, were significantly higher compared to the peak concentration in Column B and E. The peak concentrations from Port 3 were 8.233 mg/L and 8.604 mg/L for Column C and F respectively. However the same peaking pattern was seen as for Column B and E. And it was suggested from the results presented from the three curves of Port 1, 2, and 3 for each Column B, C, E, and F that at the start of the experiment the chloride concentrations in the soil solution was high on the top of the column and by applying more water, the peak concentration appeared at Port 2, and later at the bottom Port 3.

The peak concentration from Port 3 appeared after three meltwater pulses ($3 \times 840 \text{ mL} = 2520 \text{ mL}$). This indicates that the larger amount of chloride in the cake was flushed by the first meltwater. The initial volumes of water in Column B and E were

2111 mL and 2416 mL, respectively, and 2015 mL and 2318 mL in Column C and F, respectively. The appearance of chloride peaks from Port 3 after applying 2520 mL of water indicates that the flow of water displaced these initial volumes of water from the soil column and that chloride was passively transported with the flow by advection. The results also suggest that leaching of chloride anions was independent of the soil type and

Figure 5.5 Chloride concentration versus time and depth. chloride was easily leached from the cake and the soil. In addition, it was found that an increase of the amount of biosolids cake would result in more leaching of chloride anions.

Similarly, in Figures 5.6 and 5.7 where fluoride and bromide were investigated, it is observed that the appearance and depletion pattern of the concentrations was the same as what was seen for chloride. At the start of the experiment, fluoride and bromide in the soil solution were high on the top of the column and by applying more water, the peak concentration appeared at Port 2, and later at the bottom Port 3. Peaks of fluoride and bromide also appeared on day 9 from Port 3. As well as for chloride, no significant differences were seen between the two soils A and B, but losses from Columns C and F were higher than from Columns B and E. The peak concentrations of fluoride were 0.832 mg/L and 0.902 mg/L in Column C and F and 0.036 mg/L and 0.032 mg/L in Column B and E. It is suggested from the results that fluoride and bromide anions were transported through the soil by advection.

SOIL A (100% sand)**Col. A**

Fluoride

Below detection

SOIL B (75% sand, 25% from site)**Col. D**

Fluoride

Below detection

Figure 5.6 Fluoride concentration versus time and depth.

Figure 5.7 Bromide concentration versus time and depth.

Figures 5.8 and 5.9 present nitrite-N and nitrate-N concentrations, respectively. It is seen that increasing the rate of biosolids cake resulted in higher concentrations of nitrite-N and nitrate-N in the soil solution. No significant leaching difference were seen between the two soil types for the leaching of nitrite-N, and there is some evidence to suggest that nitrite-N leached faster from the 49 g biosolid loaded columns. Nitrite-N peaked from Column B and E on day 9 and on day 12 from Column C and F. It is also observed, from the 49 g biosolids loaded columns, that the peak concentrations of nitrite-N from Port 1 and Port 2 on day 4 and 7, respectively were larger than that from Port 3 on day 9, but in the 1000 g biosolids loaded columns the nitrite-N concentration from Port 1 and Port 2 were smaller than that extracted from Port 3. These observations also indicate that the nitrite-N anions were transported by advection from the two soil types.

On the other hand when nitrate-N was investigated, significant differences were observed between the movement of nitrate-N from the two different soils. From Port 3 it is seen that on day 9 larger concentrations were extracted from Column B and C than that extracted from Column E and F. It is also observed, from the 49 g biosolids loaded

columns, that in Soil B the concentration of nitrate-N from Port 1 and Port 2 on day 4 and 7, respectively were larger than that from Port 3 on day 9 while for the 1000 g biosolids loaded columns, larger concentrations were also seen from Port 1 and Port 2 than from Port 3. Referring to the water volumes calculations approach that were seen when chloride was investigated, and the peaking pattern of nitrite-N and nitrate-N that first appeared on the top of the columns and then at Port 2 and later at the bottom Port 3 on Day 9, there is some evidence to suggest that the larger amount of nitrite-N and nitrate-N in the cake were flushed from the first meltwater and were transported by advection.

Figure 5.8 Nitrite-N concentration versus time and depth.

Figure 5.9 Nitrate-N concentration versus time and depth.

After day 12 until the end of the experiment, the concentration magnitudes of nitrate-N from Port 1 and Port 2, as indicated from the tails of the curves, were slightly above the concentrations of Port 3. This was not seen for chloride, fluoride and bromide. And it was suggested that during the experiment some of the biosolids organic nitrogen were biodegraded and were continuously washed from the cake and nitrification took place. Organic materials usually contain proteins in variable amounts. When proteins were broken down, either by bacterial decomposition or as a waste product of protein metabolism, ammonia was formed. Bacterial action converted ammonia into nitrite-N, which in turn might be converted to nitrate-N.

The ammonia of the soil solutions that were presented in Figures 5.10 indicate that increasing the rate of biosolids cake would result in higher concentrations of ammonia in the soil solution. From Port 3, the peak concentrations of ammonia from Column B and E were 18 mg/L and 12 mg/L, respectively, and 105 mg/L and 95 mg/L for Column C and F, respectively. The results also show that ammonia that was washed

from the first meltwater application appeared earlier and with larger concentrations from Soil A than from Soil B. On day 7, for the 49 g biosolids loaded columns B and E, 36 mg/L and 29 mg/L were observed for Soil A and Soil B, respectively, and 120 mg/L and 177 mg/L for the 1000 g biosolids loaded columns C and F. On day 9, 18 and 12 mg/L were detected from Port 2 for the two Soils A and B, respectively. In Columns B and E and after day 12, it was observed that ammonia concentrations were larger from the solutions extracted from the top and the middle Port 1 and Port 2 than that from the bottom Port 3. In Columns C and F, ammonia peaked also on day 9 from Port 3 but it is seen that the width of the peaks were larger than that of the columns loaded with 49 g of

Figure 5.10 Ammonia concentration versus time and depth.

biosolids. Concentrations significantly decreased on day 15 for Soil A in Column C and in Column F. The concentration also started to decrease after day 9 but the concentration presented by the curve had longer tail. In addition it is seen that concentration in the top and the middle of the Column C (Port 1 and 2) declined faster compared to that of Column F. The results are indicative that increasing the rate of biosolids would yield more ammonia in the soil solution. The larger ammonia concentrations were washed from the first meltwater application, transported by advection and appeared on day 9 as was seen for the chloride anions. The large concentration found from the top and the middle of the columns after day 12 compared to that of the concentration extracted from the bottom (Port 3) suggest that during the experiment the available proteins of the biosolids cakes were broken down and ammonia were continuously leached from the columns.

The concentrations of o-phosphate in the soil solutions that are presented in Figure 5.11 also indicate that increasing the rate of biosolids cake would result in higher concentrations of o-phosphate in the soil solution. The peaking concentrations from Port 3 of o-phosphate from Column B and E were 0.067 mg/L and 0.047 mg/L respectively,

and 3.545 mg/L and 2.833 mg/L for Column C and F respectively. It is also observed for the two different soils that the peak concentrations extracted from Port 3 were retarded compared to the peaking pattern of chloride and appeared on day 12. Only the values in Column C peaked on day 9, however large concentrations were also seen after 3 days and on day 12. This is indicative of less rapid leaching of phosphate from Soil B that contained appreciable particles of silt and clay. It is observed after day 12 to the end of the experiment that the concentrations of phosphate with respect to depth and time did not decline quickly and took longer time to deplete from the columns. The results suggest that phosphate anions might be retained in the columns and especially the columns that contained the mixed Soil B.

Figure 5.12 presents the sulfate concentrations and it can be seen that the appearance and depletion pattern of the concentrations indicated a similarity as what was seen for chloride. At the start of the experiment, sulfate concentrations in the soil solution were high on the top of the column and by applying more water, the peak concentration appeared at Port 2, and later at the bottom Port 3. No significant differences were seen between the two soils except that the mixed Soil B initially contained a small amount of sulfate. Losses from Column C and F were higher than that from Columns B and E. The peak concentrations of sulfate were 2.813 mg/L and 3.314 mg/L in Column C and F and 0.143 mg/L and 0.179 mg/L in Column B and E. It is suggested from the results that the sulfate anions were leached from the soil by advection and that increasing the application rate of biosolids cake would result in more concentrations of sulfate into the soil solution.

From Figure 5.13 when the soluble COD was investigated, it is seen from the graphs of the control columns A and D that the concentrations were equally distributed in

Column A with respect to depth and time, and in Column D two peak values were seen from Port 3 on day 3 and day 15. In fact the magnitude of the concentrations presented for Soil A were insignificant because no peaks were seen and concentration are equally distributed in the column A. These small observed concentrations that varied from 20 to 45 mg/L were due to the change in the sample color and the absorbance recorded with the spectrophotometer during the laboratory analysis. The two peaks seen in control Column D at day 3 and 15 suggest that some soluble COD was easily extracted from the soil before applying the meltwater and it was due to the freezing effect as seen in the batch test (Section 4.1.2). From the 49 g and 1000 g biosolids loaded columns and for the same times and depths, insignificant soluble COD were seen in Column B compared to that of

Figure 5.11 Phosphate concentration versus time and depth.

Figure 5.12 Sulfate concentration versus time and depth.

Figure 5.13 COD concentration versus time and depth.

the Column E. However, it is seen in the soil solutions of the 1000 g biosolids loaded columns that large peak concentrations appeared from Port 3, the COD was 456 mg/L for Column C and 529 mg/L for Column F and it was suggested that this COD were due to active biomass in the biosolids cake and bacteria secrete extra-cellular enzymes that broke down the large solids particulates into reduced soluble forms and appeared in the columns whenever the SWE was applied during the experiment.

From the graphs presented in Figures 5.5 to 5.13, there is evidence to suggest that the largest portion of the solutes was flushed from the biosolids cakes from the first

volumes applied. In addition, chloride, fluoride, bromide, and sulfate were rapidly leached from the cake and the soil with the applied water. This was indicated by the high fractions of these ions that were seen in the soil solutions extracted from the columns on day 9. Nitrite-N and nitrate-N were also rapidly leached by advection. The peaking pattern that was seen from Port 3 for ammonia, o-phosphate, and the soluble COD were relatively retarded compared to that of the chloride and the peaks appeared on day 12. These results showed that soils with less sand and with either more silt, clay, and organic matter adsorbed a greater amount of anions (It is believed from the dark brown color of the soil collected from the site that this soil contains iron oxides and organic matter on the sand particle surfaces). And this pattern is consistent with the strong interaction of these ions with the soils, especially the mixed Soil B containing hydrous oxide, in addition the coarser the soil, the faster the rate, and the finer the soil, the slower the rate. The literature indicates that the lower the CEC, the lower the retention properties of a soil, and the faster the anions will leach through the soil. Therefore Soil A, since it has a very low CEC (Kim, 1998) would be a poor soil type as solutes will readily leach from such a soil, thereby contributing to pollution if the rate of cake applied is significantly large. This could explain the retarded movements of these solutes in Soil B that had small amounts of silt and clay (Bolt, 1976).

6.2.2 RESULTS OF MASS BALANCE CALCULATIONS

Mass balances were performed to quantify the contaminants entering and leaving the columns, to determine if solutes were retained in the columns. At the end of the experiment, 25 g of cake from the top of the columns were analyzed to determine what

was retained in the biosolid cakes. The mass of solutes from the soil solutions and the mass of solutes extracted from the cakes applied on the top of the columns were measured. The percent recovery was also reported. The mass in the soil solution was the product of the water volumes extracted from the ports and their measured concentrations. The masses in the biosolids cake before and after the run were the easily available ions that were determined by extracts, and the reported masses were the product of the measured concentrations for the frozen cake and the mass applied to the columns (49 g or 1000 g).

Table 5.1 indicates the fractions of the solutes that remained in the cakes. In this analysis 25 g of cake were collected from the top of each column and analyzed using same method employed in the batch tests. The final concentrations that remained in the cake at the end of SWE simulations were compared with the initial concentrations of the cake. The cake that was applied on the columns was the same cake as the frozen samples presented for March (Section 4.1). It was observed that some solutes were washed to a great extent from the cake and other solutes remained with considerably significant concentrations. Compared to the original mass before simulating the meltwater, it was found from the average values that 2.5%, 5.3%, 4.1%, and 6.3% of chloride, fluoride, bromide, and nitrite-N respectively remained in the cakes (SD by mass = 0.8, 1.4, 1.5, 3.2, respectively for the columns). The fraction of the average ammonia, phosphate, sulfate and COD that remained in the cake by mass were 32.9%, 24.8%, 29.2%, 33.8 % respectively (SD by mass = 15.6, 2.2, 2.7, 10.3 respectively for the columns). The results suggest that the volume of SWE employed in this study washed out most of the solutes from the cake.

Table 5.1 Concentration of measured parameters before and after simulating snow water equivalent for the biosolids cake applied on the columns.

	Initial Conc.	Final Concentration remaining in the cake at the end of SWE simulation 25 g cake /100mL							
	mg/L	Column B		Column C		Column E		Column F	
	Frozen 28 days	Conc. mg/L	% Remaining	Conc. mg/L	% Remaining	Conc. mg/L	% Remaining	Conc. mg/L	% Remaining
Cl ⁻	4.089	0.086	2.1	0.102	2.5	0.142	3.5	0.068	1.7
F	0.410	0.014	3.4	0.027	6.6	0.024	5.9	0.021	5.1
Br ⁻	0.325	0.019	5.8	0.005	1.5	0.016	4.9	0.013	4.0
NO ₂ -N	0.151	0.014	9.3	0.009	6.0	0.003	2.0	0.012	7.9
NO ₃ -N	0.373	0.052	13.9	0.067	18.0	0.041	11.0	0.082	22.0
NH ₃	648	178	27.5	210	32.4	112	17.3	351	54.2
HPO ₄ ⁻	4.772	1.05	22.0	1.288	27.0	1.145	24.0	1.241	26.0
SO ₄ ⁻	5.143	1.337	26.0	1.001	19.5	1.237	24.1	1.183	23.0
COD	1042	316	30.3	512	49.1	303	29.1	277	26.6

The relatively large percentage found for ammonia, COD, phosphate, and sulfate compared to that of chloride suggest that due to the active bacteria in the cake. A large amount of ammonia was generated due to the breakdown of organic nitrogen compounds from the organic materials by the microorganisms to the mineral nitrogen as indicated in the equations below (Schlegal, 1995).

a. Aminization – Heterotrophic

b. Ammonification (Mineralization)

The amines and amino acid so released are further utilized by still other groups of heterotrophs with the release of ammoniacal compounds, and this step is termed as ammonification. As was seen from Table 5.1 the average fraction of nitrate-N that was retained was 16.2%. Nitrate-N was expected to leach as chloride anions but this percentage in the cake was larger than the chloride fraction indicating that the ammonia so released may have been converted to nitrites-N and nitrates-N by the process of nitrification. Organic matter that was in solid form was also broken down by the active microorganisms, thus increasing the available soluble COD in the cake. The phosphate that remained in the cake might be due to the presence of Al and Fe that react with phosphate anions. These ions combine with phosphate to form insoluble compounds of Al and Fe. The relatively large fraction of sulfate found in the cake might be due to the continuous mineralization of organic sulfur as indicated in the equation (Schlegel, 1995).

Tables 5.2 and 5.3 present the results of the mass balance calculations for 49 g and 1000 g of biosolids respectively. It was observed that the masses of solutes were not completely recovered in the soil solution and the residual cake. The general trend indicates that the recoveries of the ions in Soil A were larger than which was observed from Soil B. At the same time, there is evidence from the results that in the soil columns the solute masses retained in Soil A were lower than those observed in Soil B. When the different concentrations were ranked for the 49 g application rates (Column B and E), it was observed that chloride, fluoride, bromide, and nitrate-N were weakly retained in the columns. Ammonia, nitrite-N, phosphate, sulfate, and COD were largely retained in the

columns as compared to the other ions. There was no significant difference (diff. <10%) in total concentrations of chloride, fluoride and bromide between the two Soil A and B. However, the mass balances indicated that Soil B retained more ammonia, nitrite-N, nitrate- N, phosphate, sulfate, and COD (diff. >10%). This would indicate partial retention of solute in the columns and these results show that soils with less sand and with more silt, clay, and organic matter as Soil B retained a greater amount of solutes. These results also indicate the high leaching potential of chloride, fluoride and bromide to the subsurface water as compared to the others solutes analyzed.

Correspondingly, for the 1000 g application rate (Column C and F), the solutes analyzed followed the same pattern. Chloride, fluoride, bromide, and nitrate-N were weakly retained in the columns. Ammonia, nitrite-N, phosphate, and sulfate, were largely retained in the columns as compared to the other ions

Table 5.2 Mass Balance calculation for columns loaded with 49 g biosolids application (Columns B and E)

	SOIL A (100% sand)						SOIL B (75% Sand + 25% from the site)					
	Column B (Load 49 g of cake)						Column E (Load 49 g of cake)					
	<u>1</u>	<u>2</u>	<u>Σ</u>				<u>1</u>	<u>2</u>	<u>Σ</u>			
Initial mass in cake (mg)	Final mass in cake (mg)	Mass in soil solution (mg)	Cake + Soil Solution (mg)	% Recovery	% Remained In column		Final mass in cake (mg)	Mass in soil solution (mg)	Cake + Soil Solution (mg)	% Recovery	% Remained In column	
Cl⁻	0.801	0.017	0.598	0.615	76.8	23.2	0.028	0.539	0.567	70.8	29.2	
F⁻	0.080	0.003	0.056	0.059	73.4	26.6	0.005	0.052	0.057	70.9	29.1	
Br⁻	0.064	0.004	0.049	0.053	82.4	17.6	0.003	0.046	0.049	76.8	23.2	
NO₂⁻N	0.030	0.003	0.016	0.019	62.5	37.5	0.001	0.013	0.014	45.3	54.7	
NO₃⁻N	0.073	0.010	0.051	0.061	83.8	16.2	0.008	0.037	0.045	61.7	38.3	
NH₃	127	35	29	64	50.3	49.7	22	27	49	38.5	61.4	
HPO₄²⁻	0.935	0.206	0.208	0.414	44.3	55.7	0.224	0.405	0.629	67.3	32.7	
SO₄²⁻	1.008	0.262	0.285	0.547	54.3	45.7	0.242	0.277	0.519	51.5	48.5	
COD	204	62	35	97	47.5	52.5	59	194	253	124	-24	

Table 5.3 Mass Balance calculation for columns loaded with 1000 g biosolids application (Columns C and F).

	SOIL A (100% sand)						SOIL B (75% Sand + 25% from the site)					
	Column C (Load 1000 g of cake)						Column F (Load 1000 g of cake)					
	<u>1</u> Final mass in cake (mg)	<u>2</u> Mass in soil solution (mg)	<u>Σ</u> Cake + Soil Solution (mg)	<u>%</u> Recovery	<u>%</u> Remained In column		<u>1</u> Final mass in cake (mg)	<u>2</u> Mass in soil solution (mg)	<u>Σ</u> Cake + Soil Solution (mg)	<u>%</u> Recovery	<u>%</u> Remained In column	
Initial mass in cake (mg)												
Cl⁻	16.356	0.408	11.758	12.166	74.4	25.6	0.068	11.593	11.661	71.3	28.7	
F⁻	1.64	0.108	1.197	1.305	79.6	20.4	0.021	1.288	1.309	79.8	20.2	
Br⁻	1.3	0.020	0.973	0.993	76.4	23.6	0.013	0.896	0.909	69.9	30.1	
NO₂⁻-N	0.604	0.036	0.265	0.301	49.8	50.2	0.012	0.276	0.288	47.7	52.3	
NO₃⁻-N	1.492	0.268	0.753	1.021	68.4	31.6	0.082	0.695	0.777	52.1	47.9	
NH₃	2592	840	228	1068	41.2	58.8	351	267	618	23.8	76.2	
HPO₄²⁻	19.088	5.152	8.796	13.948	73.1	26.9	1.241	8.037	9.278	48.6	51.4	
SO₄²⁻	20.572	4.004	5.457	9.461	46.0	54.0	1.183	7.04	8.223	40	60	
COD	4168	2048	1168	3216	77.2	22.8	1108	823	1931	46	54	

When ammonia was investigated it was found that larger concentrations were retained in the soil columns. The fractions of ammonia that were retained in the soils were 49.7% and 58.8% for Columns B and C respectively, and 61.4% and 76.2% for Columns E and F respectively. Results showed that increasing the rate of biosolids application would result in increased concentrations of ammonia in the soil and the soil solution. It was also observed that percentage of ammonia within the soil was larger in Soil B than in Soil A. The results suggest that the cationic nature of NH_4^+ permits its absorption and retention by soil colloidal material. It is necessary that the soils have a sufficiently high exchange capacity to retain the added ammonium or it will be removed in percolating water. Columns B and C that contained sandy soils with low exchange capacities permitted appreciable movement of ammonium from the soil. However, this was not seen in large concentrations as chloride (50.3 % and 41.2 % for Columns B and C, respectively). It was suggested from the large percent recovery of nitrate-N that ammonium was nitrified to nitrate-N and within limits of the soil characteristics, it largely moved with the soil water.

The fractions of Nitrite-N that were retained in the soils were 37.5% and 50.2% for Columns B and C, respectively, and 54.7% and 52.3% for Columns E and F, respectively. The fractions of nitrate-N that were retained in the soils were 16.2% and 31.6% for Columns B and C respectively, and 38.3% and 47.9% for Columns E and F, respectively. Although nitrate-N is an anion that can readily leach through the soil profile, Soil B will also retain more nitrate-N than soils without much silt and clay. In addition, such soils that are characterized by their large physical surfaces area could retain more microorganisms and the excess nitrate-N could result due to nitrification.

These results suggest that nitrate-N can be a more significant groundwater pollutant when applied to a sandy soil than when applied to a mixed or loamy soil since nitrate-N will leach from the soil into the groundwater at a faster rate.

COD concentrations in the soil were larger in the 1000 g loaded columns than in the 49 g loaded column. This was likely due to the significant large amount of cake and number of microorganisms of the 1000 g cake that decomposed the organic matter compared to that of the 49 g biosolids cake. The negative COD value (-24%) found in Column E indicated in Table 5.2 was due to the very low soluble organic matter that was released from the cake and might be due to the initial COD of the soil. When the cumulative mass 859 mg of the soil solution in the control Column D was subtracted from the 1053 mg found for Column E (Table C-133 and C-134), 194 mg was resulted. This was not seen for the COD in Column F because the 823 mg measured from the soil solution were relatively large and not expected to interfere with the COD of the amended Column F.

The fractions of o-phosphate that were retained were 55.7% and 26.9% for Columns B and C respectively, and 32.7% and 51.4% for Columns E and F, respectively. The larger fraction of phosphate that was retained in Column F compared to that of Column E was likely due to the reaction of phosphates with silicate clays. Soil clays are composed of layers of silica and alumina combined to form aluminosilicate sheets. Phosphate ions may combine directly with these clays by replacing a hydroxyl group on the Al atom. Also, Fe and Al ions combine with phosphate to form insoluble compounds. The resulting compounds may be precipitated from the solution or absorb on the surface of iron and aluminum oxides or on clay particles or even on quartz sand grains.

The fractions of sulfate that were retained were 45.7% and 54% for Columns B and C, respectively, and 48.5% and 60% for Columns E and F, respectively. No significant percentage differences were observed between the columns. It was reported that organic matter as a competing ligand may decrease sulfate adsorption. In addition, organic acids such as citrate and oxalate may solubilize Fe and Al (Craddock and Adams, 1988). The latter was not expected to occur since the soil pH of this experiment was 7 ± 0.6 . The first factors may explain the reduction in sulfate retention with increasing organic matter concentration in soil. However, the role of soil organic matter in affecting sulfate retention may be easily confounded with other factors such as surface area and presence of Fe and Al oxides which may increase organic matter as well as sulfate retention.

5.2.3 GENERAL DISCUSSION FOR BIOSOLIDS LOSSES AND TRANSPORT

The leaching results of the solutes (easily leached) shown in Figures 5.5 to 5.13 and the calculated mass balance were generally compared with the literature and the substantial behaviour of the solutes were suggested due to the following:

- 1) Biosolids cake and soil contain colloidal and mineral particles at their surfaces. The greater the negative charge density of the minerals, the greater the exclusion and repulsion of anions from the water near their surface (Bolt and Bruggenwert, 1976). From the experiment result anions were retained less in sands than in soils that had an appreciable amount of silt and clay, such as Soil B. This can be a significant source of groundwater pollution when biosolids are applied to a sandy soil than when applied to

a mixed or loamy soil. Hence, excessive rates of cake addition should be avoided for sandy soils as compared to loamy or mixed soils due to their low anion retention capacity.

- 2) Solubility of the salts containing the ion; while all nitrates form water soluble salts with all cations, other anions such as SO_4^{2-} , PO_4^{3-} , form sparingly soluble salts with some cations, for instance, PO_4^{3-} forms sparingly soluble salts with Ca^{++} , Al^{+++} , Fe^{+++} . Sparingly soluble salts are more difficult to leach than soluble salts.
- 3) Bolt (1976) reported a decreasing order of preferential adsorption among the following anions: $\text{SiO}_4^{4-} > \text{PO}_4^{3-} > \text{SO}_4^{2-} > \text{NO}_3^- > \text{Cl}^-$. This lyotropic series (The relative ability of ions to replace one another) indicates that SiO_4^{4-} and PO_4^{3-} ions are highly adsorbed, whereas SO_4^{2-} and NO_3^- ions are adsorbed in considerably lower concentration or are often not adsorbed. At neutral soil condition of $\text{pH} > 6$, adsorption of SO_4^{2-} and Cl^- ions is very small. These two ions are generally subjected to repulsion (Bolt and Bruggenwert, 1976). The pH of the soil solutions in this experiment was 7 ± 0.6 . Also, in the column experiments, chloride, bromide and fluoride that belong to the same group (7A) appeared early (on Day 9) in the soil solutions as compared to the phosphate that appeared later (on Day 12) and was retarded. The time to peak appearance of the anions and their percentage mass distribution in the soil solutions and in the soil reported above agree with the finding of Bolt, and Bruggenwert 1976.

5.3 ENVIRONMENTAL IMPACT

The presence of solutes in the soil solution does not necessarily indicate that water is contaminated and poses a health risk. These concentrations apparently indicate a contaminated soil water solution for a short time but not necessarily the case for a five year rate application. Also, judgment of soil water contamination depends on the crop species and the process of nutrient uptake. EPA has set Maximum Concentration Limit (MCL) standard for approximately 90 contaminants for drinking water (note that the limits for aquatic life and livestock were tolerated and slightly deviate from the MCL for drinking water. In addition most of the Canadian guidelines were derived from American studies, hence the maximum concentration limits set by the EPA were used in this study for comparison). Table 5.4 indicates the maximum concentration of the solutes detected from the soil solution versus the MCL.

Table 5.4 Maximum solutes concentration compared to MCL.

	MCL (mg/L)	Maximum detected concentration (mg/L)			
		Col. B	Col. C	Col. E	Col. F
Cl ⁻	250	0.395	8.233	0.353	8.604
F	2	0.036	0.832	0.032	0.902
Br ⁻	0.56	0.037	0.676*	0.034	0.644*
NO ₂ -N	1	0.010	0.168	0.009	0.184
NO ₃ -N	10	0.031	0.460	0.025	0.332
NH ₃	0.5	48*	126*	51*	185*
HPO ₄ ⁻	0.10	0.146*	3.545*	3.095*	3.674*
SO ₄ ⁻	250	0.154	2.813	1.119	3.617

* Concentrations exceeded the MCL.

Judging from the concentration of solutes, insignificant differences were observed for the sandy Soil A and the mixed Soil B. For the current sludge regulated rate (49 g) set

by the EPA that was applied to Column B and E, it was observed that most of the solute concentrations were below the MCL, only ammonia and phosphate exceeded the limit. When the rate of biosolid application was increased to 1000 g, ammonia, phosphate, and bromide exceeded the limit. Chloride, fluoride, nitrate-N and sulfate were detected below the MCL for the two application rates (49 g and 1000 g). This indicates that the contaminants flushed from the biosolid cakes are insignificant and are too low to cause soil water contamination. Though the biosolids had significant concentrations of ammonia and phosphate that exceeded the MCL. These inorganic anions that were measured in this experiment are significant groundwater pollutants and winter application of biosolids should be approached with caution and be managed for crops nutrient needs.

5.4 PATHOGEN INDICATORS

Pathogen indicator tests were performed on the soil solutions extracted with the ceramic cups from the six columns. The test was only performed for the samples extracted from Port 3, due to the small available volumes extracted from Port 1 and Port 2 that were directed for anion and COD analysis.

Figure 5.14 presents the total coliform densities with respect to time for the six columns. It was generally observed that after an induction period, all the coliform were filtered in the early stage of the experiment from both Soil A and B, and did not reach the lower depths. With time bacteria started to percolate down the columns and appeared in the column effluents. The total coliform concentrations that were leached from the sandy Soil A were greater than those from the mixed Soil B. In Column B, on day 12, the concentration was 6 CFU/100 ml. The concentration increased to 15 CFU/100 ml on day

15 and then decreased to 6 CFU/100 ml on day 21. Compared to Soil B, the peak concentration that was leached from Column E was 14 CFU/100 ml and appeared on day 18, and the concentration decreased to 1 CFU/100 ml on day 21.

When the concentrations of the 49 g loaded Column B were compared to that of Column C, it was realized that on Day 15 to Day 21 the Column C had larger concentrations than was measured in Column B. 15 CFU/100 ml, 4 CFU/100 ml and 6 CFU/100 ml were measured for Column B and 27 CFU/100 ml, 55 CFU/100 ml, and 67 CFU/100 ml for Column C and it was suggested from the results that increasing the rate of biosolids would yield more bacteria into the soil solution. The concentrations measured in Column C were greater than those in Column F and appeared earlier on day 9. In comparison with Column F, the concentration appeared later (on day 12) and at lower density suggesting that bacteria can be more easily leached from the Soil A than from the Soil B.

Figure 5.14 CFU/100mL total coliform count from Port 3 for Columns A, B, C, D, E, and F.

The results from the bacteria indicator test suggest that bacterial movement was retarded relative to the solute tracers in the two soils. The higher specific surface area enhanced retention through filtration and bacteria sorbed onto the colloidal clay particles more than to the sand particles. The interactions of these organisms with soil are more complex and less well understood than the reactions of the anions. Bacteria are approximately the size of fine silt and coarse clay, and viruses the size of very fine clay (MacLeod and Calcott, 1976). Viruses, which are small enough to move easily through soil pores, are retained primarily by chemical or physical adsorption to clay or oxide surfaces. Bacteria, which are intermediate in size, are retained by both entrapment and adsorption. These retained organisms are not necessarily inactivated, and may even be protected from inactivation. This is especially true when retention occurs by adsorption. Retention slows the movement of bacteria and viruses through the soil, but may also prolong their survival and with meltwater recharge, they leached later to lower depth (Ashour, 1998). The EPA standard for drinking water is 1 CFU/100 ml of total coliform bacteria, and compared to the groundwater quality standards, concentration exceeded the limits. Fecal coliform were not observed in the column effluents. This observation is supported by Kanwar (2002) who reported that freezing the biosolids would have a lethal effect on bacteria and that all the fecal coliform were inactivated or died.

6 GENERAL CONCLUSION AND RECOMMENDATIONS

Land application of biosolids has been recently restricted during the cold weather season. This can result in a restricted time for land application, an increased need for storage facilities and a diversion of biosolids to landfilling and incineration. To date land application of biosolids is based on slurry sludge. Little is known about high solids cake when applied under frozen conditions. Cake sludge over slurry sludge is characterized by its low water content and its high nutrient values.

In this study, land application of high solids cake, subjected to winter weather conditions, was evaluated in terms of solute mobility to ensure that the safety of biosolids beneficial use is scientifically confirmed. Not all the current biosolids constituents were evaluated. In this study chloride, fluoride, bromide, nitrogen, phosphate, sulfate, and COD were evaluated, and the study was divided into two parts. In the first part, a series of batch tests were performed to evaluate the availability of contaminants from biosolids cake under frozen and thawed conditions. In the second part, soil column tests were performed to evaluate losses and infiltration into thawing soils and the potential for contaminant transport under these conditions. Anaerobic dewatered biosolid collected from ROPEC was used for this study and two rates of frozen cake loading were assessed; 49 g and 1000 g were applied and evaluated on 183.85 cm² surface area.

The batch test performed on the biosolids cake samples indicated that physical changes occurred when the samples of cake were frozen. It was visually observed that the frozen cake samples had more dense particles than were observed in the fresh (unfrozen) samples. Particles in the frozen cake samples settled more rapidly than in the fresh

samples. It was also observed during filtering that the filtrate from frozen samples filtered easier and faster than that from fresh samples. These findings suggest that freezing biosolids cake enhances dewaterability and separation of solid particles from the liquid phase

Through the batch test the effect of freeze/thaw on biosolids cake was to contribute to the release of more solutes into solution. Freeze/thaw increased the concentration of all the anions in the extract. Chloride, fluoride, bromide, nitrite-N, and nitrate-N were released to a greater extent than sulfate and phosphate. Ammonia was also released to a greater extent. However, t-test indicated that under freeze/thaw conditions the mobility of fluoride, chloride, and nitrite-N from the cake when subjected to wash off will not move to greater extent compared to the fresh cake. Also, no significant change ($P < 0.05$) of these anions in the extract from the water either washing the cake under fresh conditions or washing the cake under freeze/thaw conditions will differ in concentrations.

Although, the ion concentrations after freeze/thaw the samples were small, phosphate concentrations were significantly large, as a result consideration and better land application management practices should be assigned for phosphate.

Bacteria tests indicate that subfreezing temperature would substantially inactivate the fecal coliform and that total coliform will survive and possibly regrow in cold weather conditions. Though, more time and natural conditions are needed to evaluate the process.

In the columns test, the potential infiltration and movement of the meltwater into the frozen soils columns was observed to penetrate to smaller depths compared to that of the melted soil. In the frozen Soil A and Soil B, it was observed that the pulse moved

very slow through the column with respect to time. Results suggested that at subzero temperatures, the infiltrated meltwater does not freeze and water can percolate in a partially saturated frozen soil having porosity from approximately 0.38 to 0.39, and with decreasing initial saturation of the soil at the surface, water could infiltrate to greater extent.

Through the column test it was seen that biosolid cakes behave as mulch. Cakes absorbed water due to its low moisture content ($\geq 30\%$ solids) and for a natural land application this indicates that biosolids cake will minimize the runoff water and will provide the soil with a continuous slow rate of water.

Biosolids cake cover behaved as mulch and absorbed the first meltwater volume due to its low moisture content ($\geq 30\%$ solids) and provided the partially frozen soil with a continuous slow rate of water. This first water volume carries most of the solutes washed from the cake and will infiltrate rather than run off to surface water. Meeting the objectives of the winter land application practices, spreading high-solid cakes will minimize the detrimental effect of surface contamination and will permit the dissolved solutes to slowly soak into the partially frozen ground with the meltwater.

No significance differences were found for the losses in the two soil types. However more ions were recovered from the sandy Soil A and more ions were retained in the mixed Soil B. Chloride, fluoride, and bromide were rapidly leached from the cake and the soil. And more of these ions were detected in the soil solutions extracted from the columns, indicating a high mobility within the flow. Nitrite-N and nitrate-N were also highly detected in the soil solution, however some nitrate-N was stabilized in the column by the addition of organic matter from the biosolids cake. Ammonia, phosphate, sulfate

and the organic matter indicated by the COD were relatively retarded. This pattern of retarded nutrient movement is consistent with the strong interaction of these ions with the soils especially the mixed Soil B. And it was concluded that a mixture of soil, containing a large percentage of sand and a low percentage of clay particles, will retain larger concentration of solutes in the root zone that will be later available for the plant needs. Observations from the column tests allow it to be inferred that some of the pathogens sorbed more onto the colloidal clay particles than onto the sand particles, but regardless they are poorly attached to the soil and travel significant distances under flow conditions.

Judging from the concentration of solutes in the soil solutions, insignificant differences of concentration in the solution were observed for the sandy Soil A and the mixed Soil B. For the loaded rate (49 g) that has been applied to Column B and E, it is observed that all the solutes are below the MCL, only ammonia and phosphate exceeded the limit. When the load of biosolids application was increased to 1000 g, ammonia, phosphate, and bromide exceeded the limit. Chloride, fluoride, nitrate-N and sulfate were detected below the MCL for the two loads (49 g and 1000 g). This indicates that the losses concentration flushed from the current regulated rates of biosolids cake are insignificant and are too low to cause soil water contamination, though significant concentration of ammonia and phosphate were found and exceeded the MCL. As a safety environmental concern, these inorganic anions that were measured in this experiment are significant groundwater pollutants and winter application of biosolids should be approached with caution and be managed for crops nutrient needs.

This study failed to simulate a field thermal regime. In natural conditions and for a similar depth of frozen soil, the thaw period is closer to a month. For this laboratory

study the soil in the column thawed in one day, Hence, a field study is recommended and need to be carried out.

Summarizing preliminary recommendations for spreading biosolids under frozen condition are as follows:

- Directing biosolids for land application under frozen conditions and following the current rate of biosolids regulated by the EPA, winter spreading of high solids cake can be acceptable. Climate records may help to identify the areas where the risks of surface runoff are lowest.
- The amounts of nutrients and contaminants lost in runoff following the winter application are usually greater than the other seasons, though this is not the case when sand or mixed sand are used as a thin layer on top of a clay soil before adding biosolids and eliminating the potential for runoff.
- Applying biosolids at rate more than 8 ton/ha once every 5 years resulted in more bromide, ammonia and phosphate leaching and exceeding the MCL especially in the soil solution. Hence, based on the current regulations, strategy for optimizing biosolids application under frozen condition should consider the long-term effects on soil phosphorus and possible hazards for pH if crops nutrient needs are not properly estimated.
- As precaution, spreading biosolids in the winter provides no guarantee of pathogen die-off. It is found that freezing conditions are usually lethal to fecal bacteria and inactivated most of the bacteria. However, before land application, thermophilic treatment should be considered for the other pathogens.

7 SUGGESTIONS AND FUTURE WORK

Research work leading to the achievement of more information on the transport of biosolids cake in soils and on the characterization of the dynamics of biosolids transport through numerical modeling should continue. In order to generate a wider database on biosolids cake constituent transport in thawing soils that could serve as an adequate basis for biosolids land application management and for developing a numerical modeling, the following changes and/or additions should be made to this preliminary study.

1. In addition to the laboratory experiments and analysis presented here, actual field experiments using relatively virgin plots and sites with installed lysimeters or tile drains should be used in case of refreezing soils. Biosolids cake should be applied in the same manner as in real farms and be allowed to infiltrate naturally the way it does during a thawing season and with actual regulation in term of rate and method of application. The groundwater quality must be monitored at regular time intervals by collecting groundwater and tile drain samples over several months that will allow potential nitrification and other biodegradation processes to occur. The groundwater samples must be analyzed for the major chemical constituents that may pose a threat to groundwater quality such as nitrates and orthophosphates. A sufficient number of TDR probes and Neutron probes should be installed within the frozen and unfrozen soil layers, and both moisture and electrical conductivity fluctuations should be monitored using a TDR cable tester. Temperature must be also monitored. The termination of the experiment will be

determined through occasional checks on both the water quality of the groundwater samples or tile drain samples and on TDR readings. Replications of this type of experiment will need to be done throughout the thawing and part of the growing seasons.

2. To generate a more comprehensive evaluation of groundwater quality beneath biosolids amended areas, groundwater samples should be analyzed not only for the inorganic, organic and indicator bacteria tests as performed in this preliminary study but also for total nitrogen, total phosphorus, and heavy metals. Different biosolid by-products from different treatment plants such as anaerobic, aerobic, and ATAD system need to be tested with the bulk electrical conductivity of the groundwater and tile drain samples being determined and compared with TDR readings.
3. To mathematically characterize the behaviour of biosolids transports into thawing soils and to enable numerical modeling using field scale, hydrologic data such as snowmelt, rainfall, evapotranspiration, heat exchange and other hydrologic data, that may be used to calculate infiltration and runoff along with crop and soil management data must be collected. The spatial distribution of all parameters, TDR, Neutron probes, temperature distribution, tile drain, and lysimeters layout should be planned to allow for two or three-dimensional numerical modeling of biosolids cake transport. The resulting break through curve can then be simulated. Numerical contaminant transport models and other potentially applicable numerical models also can be explored for simulating biosolid cake transport based on the data gathered.

These suggestions methods and experiment algorithms could be used not only for biosolids cake, but also for composted biosolids and manure by-product, leading to an optimum land application management system and a safe sustainable environment

REFERENCES

- Ashour A. (1998). Movement of bacteria in unsaturated soils columns with macropores. ASAE. Vol.4: 1043-1050.
- Adrian, U., and Michael, J.G. (2000). Development of a protocol for sampling fecal coliform bacteria originating from manure in the vadose zone. *Water Quality Resources Journal*. Vol.35: 23-38.
- Aguilar, R.S., and Loftin, R.. (1991). Sewage sludge application in semiarid grasslands: Effect on runoff and surface water quality. In 36th annual New Mexico water conference. Report no.265: 101-111.
- Anderson, E.A., and Crawford, N.H. (1964). The synthesis of continuous snowmelt runoff hydrographs on a digital computer. Department of Civil Engineering, Stanford University. Technical report No. 36.
- Anderson, E.A. (1973). National weather service river forecast system- Snow accumulation and ablation model. National Oceanic and Atmospheric Administration. Silver spring, Maryland. NOAA technical memorandum.
- Bilskie, J. (1999). Reducing Measurement Errors of Selected Soil Water Sensors. in Proceedings of the int. workshop Characterization and Measurement of the Hydraulic Properties of Unsaturated Porous Media, van Genuchten, M.Th., F. J. Leij and L. Wu (Eds.), Proc. Int. Workshop, "Characterization and Measurement of the Hydraulic Properties of Unsaturated Porous Media." University of California, Riverside, CA.
- Blais, P.A., and Weil, C. (1999). Nitrogen losses in surface and drainage waters from spring and late fall liquid manure applications on a flat clay soil. *Canadian Journal of Soil Science*. Vol. 4: 656-662.
- Bolt, G.H. and Bruggenwert, M.G.M. (1976). Adsorption of anions by soils. In: *Soil Chemistry. A Basic Elements*. Elsevier Science, Amsterdam, pages 91-95
- Boswell, F.C. (1975). Municipal sewage sludge and selected element application to soil, effect of soil and fescue. *Journal of Environmental Quality*. Vol.4: 267-273.
- Bouwer, S., Lance, S.C., and Riggs, M.S. (1974). High-rate land treatment II: Water quality and economic aspects of the flushing Meadows project. *JWPCF*. Vol.46: 844-853.
- Burge, W.D., and Marsh, P.B. (1978). Infectious disease hazards of landspreading sewage wastes. *Journal of Environmental Quality*. Vol.7: 1-9.

- Burggeman, A.C., and Mostaghimi, S. (1993). Sludge application effects on runoff, infiltration and water quality. Water Resources Bulletin. American Water Resources Association.
- Burn, CR. (1990). Snowmelt infiltration into frozen soil at sites in the discontinuous permafrost zone near Mayo, Yukon territory. In northern hydrology, Canadian perspectives, Prowse, Ommanney. NHRI science. Report no. 1, National Hydrology Research Institute, inland waters directorate, Environment Canada, Saskatoon, page 455-459.
- Chou, V. (1964). Handbook of applied hydrology: A compendium of water reservoir technology. McGraw Hill Book Company. New York.
- Chaney, R.L. (1987). Residual effects of sewage sludge on soybean, accumulation of heavy metals. Journal of Environmental Quality. Vol.16: 113-118.
- Colbeck, S.C., and Ray, M. (1979). Proceedings of snow cover runoff, U.S. Army Cold Regions Research and Engineering Laboratory, CRREL report: 26-28.
- Colbeck, S.C., (1981). A simulation of the enrichment of atmospheric pollutants in snow covers runoff. Water Resources Research Vol.17(5): 1383-1388.
- Converse, J.C., Bubenzer, G.D., and Paulson, W.H. (1976). Nutrient losses in surface runoff from winter spread manure. Trans. ASAE 19:517-519
- Cooke, C.M., Lindsey, G. and Fiona, A.N. (2001). Effect of drying and composting biosolids on the movements of nitrate and phosphate through repacked soil columns under steady-state hydrological conditions. Journal of Environmental Quality. Vol.13: 738-756
- Corps Engineering, snow hydrology, NTIS Pb. 151 660, north pacific division, corps of engineers, Portland, Or, June 1956.
- Crane S.R., Moore J.A., 1986. Modelling enteric bacterial die-off: A review. Water, Air and Soil Pollution, Vol.27: 411-439.
- Dasberg, S., and Hopmans, J.W. (1992). Time domain reflectometry calibration for uniformly and no uniformly wetted sandy and clayey loam soils. Soil Science Society of American Journal, Vol.56: 1341-1345.
- DeRyck, S.M., J.D. Redman, and A.P. Annan. (1993). Geophysical Monitoring of a Controlled Kerosene Spills. in Proceedings of the Symposium on the Application of Geophysics to Engineering and Environmental Problems, San Diego, CA: 5-20.
- Dirksen, C.E., and S. Dasberg. (1993). Four Component Mixing Model for Improved Calibration of TDR Soil Water Content Measurements. Soil Sci. Soc. Amer. J., Vol.57: 660-667.

- Dowdy, R.H. (1991). Management of agricultural land receiving wastewater sludge, soil management for sustainability, soil and water conservation society publications. Ankeny, IA, 85-101.
- Dunnigan, E.P., and R.P. Dick. (1980). Nutrient and coliform losses in runoff from fertilized and sewage sludge-treated soil. *Journal of Environmental Quality*. Vol.9:243-250.
- Emmerich, W.E., and Lund, L.J. and Chang, A.C. Solid phase forms of heavy metals in sewage sludge treated soils. (1982). *Journal of Environmental Quality*. Vol.11: 178-191.
- Epstein, E. (1975). Effect of sewage sludge on some soil physical properties. *Journal of Environmental Quality*. Vol.4: 139-142.
- Ferre, P.A., D.L. Rudolph, and R.G. Kachanoski. (1994). A Multilevel Waveguide for Profiling Water Content using Time Domain Reflectometry. in *Proceedings of the Symposium and workshop on Time Domain Reflectometry in Environment, Infrastructure and Mining Applications*, Special Pub. SP-19-94 Northwestern University, Evanston IL.
- Ferre, P.A., D.L. Rudolph, and R.G. Kachanoski. (1998) Water Content of a Profiling Time Domain Reflectometry Probe. *Soil Sciences Society of American Journal*. Vol.62: 865-873.
- Geankoplis, J. (1972). Mass transport phenomena, Holt, Rinehart and Winston, Inc., N.Y.
- Gendebien, A.H., Cartmell, E., and Chamber, B.J. (1999). Beneficial effect of biosolids on soil quality and fertility. SAC/SEPA. Agriculture and waste management for sustainable future. Conference paper.
- Gray, D.M, Ganger and P. G. Landine. 1985. Simulating infiltration into frozen prairies soils in stream flow models. *Canadian journal of earth sciences*. Vol.22: 464-474.
- Gray, D.M, Granger, R.J., and Nicholaichuk, W. (1986). Snowmelt infiltration to cracked, uncracked and subsoiled frozen soils. In *proceedings of the Symposium on Snow Management for Agriculture*, publication No.120: 219-319.
- Gray, D.M., Granger, R.J., and Landine, P.G. (1986). Modeling snowmelt infiltration and runoff in prairie environment. In *proceeding: Cold Regions Hydrology Symposium*. American Water Resources Association, July 1986, page 427-438.
- Gray, D.M, Toth, B., Zaho, L., and Granger, R.J. (2001). Estimating areal snowmelt infiltration into frozen soils. *Hydrological process*. Vol.15: 3095-3111.

- Granger, R.J., Gray, D.M., and Dyck, G.E. (1984). Snowmelt infiltration to frozen prairie soils. *Canadian Journal of Earth. Vol.21: 669-677.*
- Gupta, S.C., Dowdy, R.H., and Larson. (1977). Hydraulic and thermal properties of a sandy soil as influenced by incorporation of sewage sludge. *Soil Sciences Society of American Journal. Vol.41: 601-605.*
- Heckrath, G., and Brooks, P.C. (1995). Phosphorus leaching from soils containing different phosphorus concentrations in the Broadbalk experiment. *Journal of Environmental Quality. Vol.24: 904-910.*
- Herkelrath, W.N. and S.P. Murphy. (1991). Automatic, Real Time Monitoring of Soil Moisture in a Remote Field Area With Time Domain Reflectometry. *Water Resour. Res., Vol.27(5).*
- Hensen, E.A., and Harris, A.F. (1974). Validity of soil-water samples collected with porous ceramic cups. *Soil Science Society of American Journal. Vol.39: 528-536.*
- Hensler, R.F., Olsen, R.J., Witzel, S.A., Attoe, O.J., Paulson, W.H, and Johannes, R.F. (1970). Effect of method of manure handling on crop yields, nutrient recovery and runoff losses. *Trans.of ASAE. 726-731.*
- Herkelrath, W.N., Hamburg, P. and Murphy. (1991). Automatic real time monitoring of soil moisture in a remote field area with time domain reflectometry. *Water Resource Researches. Vol.27: 857-864.*
- Hern, S.C., and Melancon, S.M. (1986). Vadose zone modeling of organic pollutants. Lewis publishers, INC.
- Hinsley, T.D. (1971). Agricultural benefits and environmental changes resulting from the use of digested sewage sludge on field crop. A report on a solid waste demonstration project, US EPA, Washington, D.C.
- Hinsley, T.D. (1976). Soybean yield responses and assimilation of Zn and Cd from sewage sludge amended soil. *Journal of Water Pollution Control Federation, Vol.48: 2137-2152.*
- Horton, R.E. (1940). Phenomena of the contact zone between the ground surface and a layer of melting snow. *Transaction of meetings of international commission of snow and glaciers, Edinburgh, International association of hydrology bulletin 23.*
- Huang, F.C. and C.H. Dowding. (1994). Telemetric and Multiplexing Enhancement of Time Domain Reflectometry Measurements. in the *Proceedings, Symposium and Workshop on Time Domain Reflectometry in Environmental, Infrastructure, and Mining Applications, Northwestern University, U.S. Bureau of Mines Special Publication SP-19-94.*

- Jacobsen, O.H., and Schjonning, P. (1993). A laboratory calibration of time domain reflectometry for soil water measurement including effects of bulk density and textures. *Journal of Hydrology*. Vol.151: 147-157.
- James, D.W., and Anderson, G.L. (1996). Phosphorus mobility in calcareous soils under heavy manuring. *Journal of environmental quality*. Vol.25: 770-775.
- Jang, L.K., Chang, P.W. Findley, and Yen, T.F. (1983). Selection of bacteria with favorable transport properties through porous rock for the application of microbial enhanced oil recovery. *Applied Environmental Microbiology*. Vol.46:1066-1072.
- Joshua, W.D. (1996). Change in soil physical properties due to biosolids application to agricultural lands. *Biosolids research in NSW agriculture*, 28-33.
- Johannessen, M.A. and Henriksen. (1978). Chemistry of snow meltwater, changes in concentration during melting. *Water Resources Research*. Vol.14 (4): 615-619.
- Kane, D.L., and Stein, J. (1983). Water movement into seasonally frozen soils. *Water Resources Research*. Vol.9: 1547-1557.
- Kanwar, R.S. (2000). The effect of swine manure application on bacterial quality of leachate from intact soil columns. ASAE Paper No. 00-2053. St. Joseph, MI: ASAE
- Karmarkov, S.V. (1996). Optimized ion chromatography for rapid determination of chloride, nitrate, phosphate and sulfate in discharge waters, American Environmental Laboratory.
- Kenny, D.R. (1989). Source of nitrate to groundwater in nitrogen management and groundwater protection. Elsevier Science Publishers, Amsterdam, pages 23-34.
- Kelling, K.A. (1977). Effect of wastewater sludge on soil moisture relationship and surface runoff. *Journal of Water Pollution*. Vol.59: 1698-1703.
- Kibbey, H. J., C. Hagedorn, and E. L. McCoy. (1978). Use of fecal streptococci as indicators of pollution in soil. *Applied Environ. Microbiology*. Vol. 35(4): 711-717.
- Kim, H.T. (1998). Principles of soil chemistry, third edition, revised and expanded.
- Klausner, S.D., Zwerman P.J., and Ellis D.F. (1976). Nitrogen and phosphorus losses from winter disposal of manure. *Journal of Environmental Quality*. Vol.5: 47-49.
- Kladiviko, E.J. (1979). Change in soil properties from application of anaerobic sludge. *Journal of Water Pollution*. Vol.51: 325-332.

- Komarov, V.D., and Makarova, T.T. (1973). Effect of the ice, cementation and freezing depth of the soil on meltwater infiltration in a basin. *Soviet Hydrology: Selected papers*. Issue No.3: 243-249.
- Krejsl, J., Harrison, R., and Henry, C. (1994). Comparison of lysimeter types in collecting microbial constituents from sewage effluent. *Soil Science Society of American Journal*. Vol.58: 131-133.
- Lamb, C. (1972). Sewage sludge produces hay. *Environmental Action Bul.* Vol.3: 4-5.
- Linsley, R.K., Kohler, M.A. and Paulhus, J.L. (1975). Hydrology for engineers. New York: McGraw-Hill.
- Looney, B. and R. Falta. (2000). Vadose Zone Science and Technology Solutions. Battelle Press, OH.
- Mackey, B.M. (1984). Lethal and sublethal effects of refrigeration, freezing and freeze-drying on microorganisms. *Academic press*, pages: 45-75.
- MacLeod, R.A., and Calcott, P.H. (1976). Cold shock and freezing damage to microbes. 26th symposium of the Society for General Microbiology. New York: Cambridge University press, pages 81–109.
- Male, D.H., and Gray, D.M. (1981). Snowcover ablation and runoff principles process, management and use, *Pergamon press*, pages 360-436.
- Mallants, D., M. Vanclooster, N. Toride, J. Vanderborght, M.Th. van Genuchten, and J. Feyen. (1996). Comparison of Three Methods to Calibrate TDR for Monitoring Solute Movement in Undisturbed Soil. *Soil Sci. Soc. Am. J.*, Vol.60(3): 747–754.
- Mazur, P. (1966). Physical and chemical basis of injury in single-celled microorganism subjected to freezing and thawing. In *Cryobiology* H.T. Merman. Edition: Academic press, New York. pages: 214-315.
- McFarland, J. (2000). Biosolids Engineering. McGraw-Hill Professional
- McFeters, G. A., and D. G. Stuart. (1972). Survival of coliform bacteria in natural waters: Field and laboratory studies with membrane filter chambers. *Applied Microbiology*. Vol.24(5):805–811.
- Metcalf and Eddy, Inc. (1991). *Wastewater Engineering. Treatment, Disposal, and Reuse*, 3d ed., McGraw-Hill, New York.
- Method for the determination of metal and inorganic chemicals in environmental samples EPA, Ohio, (1996).

- Midgley, A.R., and Dunklee, D.E. (1945). Fertility runoff losses from manure spread during the winter. University of Vermont, agriculture experiences station. Bulletin 523. Pages 1-19.
- Miroslav, K., and Donald, R. (1994). Soil Hydrology- Geoecology textbook.
- Motovilov, Y.G. (1979). Simulation of meltwater losses through infiltration into soil. Soviet hydrology selected papers. Vol.18 (3): 217-221.
- Mostaghimi, S., T.M. Younos, and U.S. Tim. (1992). Effects of sludge and chemical fertilizer application on runoff water quality. Water Resour. Bull. Vol.28:545-552.
- Natsh, A., Keel, C., Troxler, J., Zala, M., and Von Abertini, N. (1996). Importance of preferential flows and soil management in vertical transport of a bicontrol strain of *Pseudomona fluorescens* in structured fields. Journal of Applied Environment Microbiology. Vol.62: 33-40.
- O'Connor, K.M. and L.V. Wade. (1994). Applications of Time Domain Reflectometry in the Mining Industry. in Proceedings of the Symposium and Workshop on Time Domain Reflectometry in Environmental, Infrastructure, and Mining Applications held at Northwestern University, Evanston, Illinois, September 17-19, 1994 USBM special publication SP 19-94 :494-506.
- Ontario Ministry of Environment. (1996). Guidelines for the utilization of biosolids and other wastes on agricultural land.
- O'Neil, D.C., and Sharon, L.B. (1995). Assessing the impact of winter land application of secondary sludge on water quality. International environmental conference proceedings. TAPPI: 735-750.
- Parker, Louise, V., Melinda, L., Martel, C.J., and Charles, M. (2000). Bacterial survival in snow made from wastewater engineering research and development center, Hanover. Cold regions research and engineering lab. (CRREL) report TR-00-9, pages 1-15.
- Patterson, D.E., and Smith, M.W. (1981). The measurement of unfrozen water content by time domain reflectometry: Results from laboratory tests. Canadian Geotechnical Journal. Vol.18: 131-144.
- Peterson, T.C., and Ward, R.C. (1989). Development of a bacterial transport model for coarse soils. Water Resources Bulletin, Vol.25:349-357.
- Pepin, S., and Hook, W.R. (1995). Temperature dependent measurement errors in time domain reflectometry determination of soil water. Soil Science Society of American Journal. Vol.59: 38-43

- Pepper, I.L., Straub, T.M, and C.P. Gerba. 1993. Hazards from pathogenic microorganisms in land-disposed sewage sludge. *Reviews of environmental contamination and toxicology*. Vol.132: 55-91.
- Pfaff, J.D. (1993). Determination of inorganic anions by ion chromatography, US EPA Method 300 SW-846 Ch.5, EMSL, office of research and development.
- Popov, E.G. (1972). Snowmelt runoff forecasts. Theoretical problems in the role of snow and ice hydrology. *Proceedings of the Banff symposium*. Banf, Alberta, Canada, UNESCO-WMO. Vol.2: 829-839.
- Qu, G., Leonard J., Feddes J., and McGill, W. (1996). Effects of fresh and composted manure on runoff water quality when applied on snow. *Canadian Society of Agricultural Engineering*. Paper no. 96-205.
- Roth, K., Shulin, R., and Fluhler, H. (1990). Calibration of time domain reflectometry for water content measurement using a composite dielectric approach. *Water Resources Researches*. Vol.26: 2267-2273.
- Sabey, B.R. (1973). Denver sewage sludge recycled on the land. *Compost Science*. Vol.14: 4-7.
- Sanin, F. D., P. A. Vesilind, and C. J. Martel. (1994). Pathogen reduction capability of freeze/thaw sludge conditioning: *Water Research*. 28 2393–2398. Ward, A.L., R.G. Kachanoski and D.E. Elrick. "Laboratory Measurement of Solute Transport Using Time Domain Reflectometry." *Soil Sci. Soc. Am J.*, Vol.58: 1031 1039.
- Schlegel, H.G. (1995). General microbiology, Cambridge University press, Cambridge.
- Schndorf, T. and Herrmann, R. (1987). Transport and chemodynamics of organic micropollutants and ions during snowmelt. *Nordic hydrology*. Vol.18: 254-278.
- Scholl, M.A., A.L. Mills, J.S. Herman, and G.M. Hornberger. 1990. The influence of mineralogy and solution chemistry on the attachment of bacteria to representative aquifer minerals. *Journal of Contaminant Hydrology*. 6:321-336.
- Scott, T.M., J. Lukasik, and S.R. Farrah. 2002. Improved method for recovery of bacteriophage from large volumes of water using negatively charged microporous filters. *Canadian Journal of Microbiology*. 48: 305-310.
- Shepher, M.A. (1996). Factors affecting nitrate leaching from sewage sludges applied to a sandy soil in arable agriculture. *Agricultural ecosystems environment*. Vol.58: 171-185.
- Simmons, K., and Baker, D. (1993). A zero-tension sampler for the collection of soil water in macropores systems. *Journal of Environmental Quality*. Vol.22: 207-212.

- Sims, J.T., and Simard, R.R. (1998). Phosphorus loss in agricultural drainage: Historical perspective and current research. *Journal of Environmental Quality*. Vol.27 (2): 277-293.
- Smith, S.R. (1996). Agricultural recycling of sewage sludge and the environment. CAB international, Wallingford, Oxford. Page 1-8.
- Smith, S.R., and Woods, V. (1998). Nitrate dynamics in biosolids treated soils, influence of biosolids type and soil type. *Bioresources Technology*. Vol.66: 139-149.
- Soon, T.K., and Bates, T.E. (1978). Land application of chemically treated sewage sludge. Effect on crop yield and nitrogen availability. *Journal of Environmental Quality*. Vol.7: 264-269.
- Sparks, D.L. (2003). Environmental soil chemistry, second edition, Elsevier Science press (USA).
- Standard Methods for the Examination of Water and Wastewater, 19th edition (1995). American Public Health Association, NW, Washington, DC.
- Steenhuis, T.S., Bubenzer, G.D., Converse, J.C., and Watler, M.F. (1981). Winter-spread manure nitrogen loss. *Transaction of American Society of Agricultural Engineers*. Pages 436-449.
- Steven, A.G. (2000). Physical and chemical factors affecting contaminant hydrology in cold environments ERDC/CRREL / Cold regions research and engineering laboratory, TR-00-21.
- Stewart, N.E., and Beauchemin, E.G. (1975). Nitrate nitrogen distribution in corn land following applications of digested sewage sludge, *Journal of Soil Science*. Vol. 55: 287-298
- Surampalti, R.Y, and Lin, K.L., (1997). Impact of long-term land application of biosolids on groundwater quality and surface soils. *Journal of Environmental System*. Vol.26: 305-324.
- Sullivan, D.M., Cogger, C.G. Bary, A.I. and Fransen, S.C. (2000). Timing of dairy manure applications to perennial grass on well-drained and poorly-drained soils *Journal of Soil and Water Conservation*. Vol.55:147-152.
- Steenhuis, T.S., Budenzer, G.D., and Walter, M.F. (1977). Water movement and infiltration in a frozen soil. Theoretical and Experimental Consideration. *Transaction of American Society of Agricultural Engineers*, paper No.77-2545, presented at the 1977 winter meeting of the American society of agricultural engineers, 31 pages.

- Tao, Y.X., and Gray, D.M. (1994). Prediction of snowmelt infiltration into frozen soils. Numerical heat transfer. Vol.26 (A): 643-665.
- Topp, G.C., Davis, J.L., and Annan, A.P. (1980). Electromagnetic determination of soil water content: measurements in coaxial transmission lines. Water Resources Research. Vol.16: 574-582.
- Topp, G.C., and J.L. Davis. (1985). Measurement of Soil Water Content Using Time Domain Reflectometry (TDR): A Field Evaluation. Soil Sci. Soc. Am. J., Vol.49:19-24.
- USEPA. (1994). A plain English guide to the EPA part 503 biosolids rule. U.S. Environmental Protection Agency, Washington, D.C.
- USEPA. (1995). Biosolids management handbook for small publicly owned treatment works. U.S. Environmental Protection Agency, Washington, D.C.
- USEPA. (1997). Biosolids management, use and disposal. Office of water.
- Vesilind, P. A. (1974). Treatment and disposal of wastewater sludge. Ann Arbor science publishers Inc.
- Vesilind, P.A., and Martel, C.J. (1990). Freezing of water and wastewater sludges. American society of civil engineering. Journal of Environmental Engineering. Vol. 116: 854-862
- Viessman, W., and Lewis, L. (1995). Introduction to hydrology. 4th edition. Harper Collins. New York,.
- White, I., S.J. Zegelin, G.C. Topp. (1994) A. Fish. Effect of Bulk Electrical Conductivity on TDR Measurement of Water Content in Porous Media. in Proceedings of the Symposium and Workshop on Time Domain Reflectometry in Environmental, Infrastructure, and Mining Applications, Northwestern University, Evanston, IL, September 17–19, 1994: 294–308, and USBM special publication SP.19–94.
- Wild, A. (1996). Soil and the environment, Cambridge University press, Cambridge
- Williams, D.E. (1987). Metal movement in sludge amended soils: A nine-year study, Journal of Soil Science. Vol.143: 124-131.
- Willis, W.D., Parkinson, H.L., and Carlson, C.W. (1964). Water table changes and soil moisture loss under frozen conditions. Canadian Journal of Soil Science, Vol.98: 244-248.

- Wood, W. (1973). A technique using ceramic porous cups for water sampling at any depth in the unsaturated zone. *Water Resources Researches*. Vol.9: 486-488.
- Yangming, and Corey, R.B. (1993). Redistribution of sludge-borne cadmium, copper, and zinc in a cultivated plot. *Journal of Environmental Quality*. Vol.22: 1-8.
- Yokuda, E., and R. Smith. "A New Probe for In Situ TDR Moisture Measurement." Paper #93-106. Idaho, National Engineering Laboratory, Idaho Falls, ID (1993).
- Yosida, Z. (1973). Infiltration of thaw water into a dry snow cover, low temperature science, series A.
- Young, R.A. and Holt, R.F. (1977). Winter applied manure: Effects on annual runoff, erosion, and nutrient movement. *Journal of Soil and Water Conservation*. Vol.35: 219-222.
- Zapf-Gilje, R., Russell, S.O., and Mavinic, D.S. (1986). Concentration of impurities during melting snow made from secondary sewage effluent. *Water Science and Technology*. Vol.18: 151-156.
- Zibilske, L. M., and R. W. Weaver. (1978). Effect of environmental factors on survival of *Salmonella typhimurium* in soil. *J.Environ. Quality* 7(4): 593-597.
- Zhao, L., and Gray D.M. (1997). A parametric expression for estimating infiltration into frozen soils. *Hydrological process*. Vol.11: 1761-1775.
- Zhao, L., and Gray, D.M. (1999). Estimating snowmelt infiltration into frozen soils, *Hydrological process*. Vol.15: 1827-1842.

Appendix A
Results for Chapter 3
Method Preparation

Figure A-1 Total solids in the biosolids cake samples for January.

Figure A-2 Total solids in the biosolids cake samples for February.

Figure A-3 Total solids in the biosolids cake samples for March.

Table A-1 Sieve Analysis Sample Data for Soil A.

Sieve No.	Sieve Opening (mm)	Wt. Retained (g)	Percent Retained	Cumulative % Retained	Percent Finer
10	2.000	4.4	0.51	0.51	99.49
20	0.850	52.6	6.12	6.64	93.36
30	0.600	99.3	11.56	18.20	81.80
40	0.425	212.3	24.72	42.92	57.08
100	0.150	433.3	50.45	93.36	6.64
200	0.075	46.7	5.44	98.80	1.20
Pan	-	10.3	1.20	100.00	0.00
		Σ 858.9			

Oven dry weight of sample: 859.38 g

Sum of weight retained: **858.9**Soil Loss: **0.48** g**Table A-2** Sieve Analysis Sample Data for Soil B

Sieve No.	Sieve Opening (mm)	Wt. Retained (g)	Percent Retained	Cumulative % Retained	Percent Finer
10	2.000	6.1	0.64	0.64	99.36
20	0.850	67.5	7.07	7.71	92.29
30	0.600	94.7	9.91	17.62	82.38
40	0.425	183.3	19.19	36.81	63.19
100	0.150	465.9	48.78	85.58	14.42
200	0.075	91.6	9.59	95.17	4.83
Pan	-	46.1	4.83	100.00	0.00
		Σ 955.2			

Oven dry weight of sample: **955.56** gSum of weight retained: **955.2** gSoil Loss: **0.36** g**Table A-3** Saturated hydraulic conductivity for Soil A.

Cumulative time (Sec.)	Volume (mL)	Area (cm ²)	h (cm)	L (cm)	K (cm/sec)
60	150	44.18	130	23	0.01001
120	340	44.18	130	23	0.01135
180	510	44.18	130	23	0.01135
360	1000	44.18	130	23	0.01112
600	1640	44.18	130	23	0.01095
Average					0.011
Std. Dev.					0.001

Table A-6 Saturated hydraulic conductivity for Soil B.

Cumulative time (Sec.)	Volume (mL)	Area (cm²)	h (cm)	L (cm)	K (cm/sec)
60	70	44.18	130	23	0.00467
120	132	44.18	130	23	0.00441
180	195	44.18	130	23	0.00434
300	320	44.18	130	23	0.00427
600	635	44.18	130	23	0.00424
Average					0.004
Std. Dev.					0.000

Calibration Curve Calculations for TDR

Volume = Area x Length

Diameter of Bottles = 6.5 cm, Then Radius = 3.25 cm

Length = 15.3 cm the same as column diameter.

Volume = $\pi (r)^2 \times 15.3 \text{ cm} = 507.7 \text{ cm}^3 \rightarrow$ to convert to liter multiply by 0.001
 $= 507.7 \text{ cm}^3 \times 0.001 = 0.5077 \text{ liter} = 507.70 \text{ mL}$

Since

Soil A (Sand) Porosity = 0.3813

Then

$507.7 \times 0.3813 = 193.58 \text{ mL} = \text{Volume added for 100\% saturation}$

Soil B Porosity = 0.3936

Then $507.70 \times 0.3936 = 199.83 \text{ mL} = \text{Volume added for 100\% saturation}$

Soil A		Porosity = 0.3813				Volume = 193.6 mL at 100%					
mL added		19.4	38.7	58.1	77.4	96.8	116.1	135.5	154.9	174.2	193.6
		10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
Frozen											
T1		28.87	28.87	28.87	28.87	28.86	28.87	28.86	28.87	28.87	28.86
T2		29.89	29.97	29.95	29.99	29.94	29.98	30.00	30.05	30.09	30.13
Delta T		1.02	1.10	1.08	1.12	1.08	1.11	1.14	1.18	1.22	1.27
K _d		4.2	4.8	5.4	5.9	5.9	5.9	5.9	6.0	6.5	6.9
Unfrozen											
T1		28.87	28.87	28.87	28.87	28.86	28.87	28.86	28.87	28.87	28.86
T2		30.01	30.10	30.22	30.28	30.41	30.43	30.60	30.78	30.87	31.22
Delta T		1.14	1.23	1.35	1.41	1.55	1.56	1.74	1.91	2.00	2.36
K _d		5.3	7.0	7.9	9.2	9.6	11.2	13.7	15.7	18.6	25.3

Soil B		Porosity = 0.3936				Volume = 199.8 mL at 100%					
mL added		20.0	40.0	59.9	79.9	99.9	119.9	139.9	159.9	179.8	199.8
		10%	20%	30%	40%	50%	60%	70%	80%	90%	100%
Frozen											
T1		28.88	28.88	28.88	28.9	28.88	28.87	28.87	28.86	28.87	28.87
T2		29.94	30.01	30.05	30.1	30.04	30.06	30.09	30.11	30.14	30.23
Delta T		1.06	1.13	1.17	1.17	1.16	1.19	1.22	1.25	1.27	1.36
K _d		4.5	5.1	5.4	5.5	5.6	5.7	6	6.3	6.4	7.1
Unfrozen											
T1		28.88	28.88	28.88	28.9	28.88	28.87	28.87	28.86	28.87	28.87
T2		30.03	30.22	30.36	30.5	30.51	30.62	30.81	30.94	31	31.44
Delta T		1.15	1.34	1.48	1.57	1.63	1.75	1.94	2.08	2.13	2.57
K _d		5.4	7.2	8.8	10.1	10.9	12.3	15	17.2	18.2	26.7

Figure A-4 IC Calibration Curve for Fluoride

Figure A-5 IC Calibration Curve for Chloride

Figure A-6 IC Calibration Curve for Nitrite-N.

Figure A-7 IC Calibration Curve for Bromide.

Figure A-8 IC Calibration Curve for Nitrate-N. **Figure A-9** IC Calibration Curve for O-Phosphate.

Figure A-10 IC Calibration Curve for Sulfate.

Table A-7 Standard Solution Concentration for the Calibration Curve preparation.

Standard Solution Concentration mg/L		Percent (%) Concentration for 20 ml						
		100	80	60	30	20	10	2
Fluoride	20	0.200	0.250	0.333	0.667	1.000	2.000	10.000
Chloride	50	0.500	0.625	0.833	1.667	2.500	5.000	25.000
Nitrite	25	0.250	0.313	0.417	0.833	1.250	2.500	12.500
Bromide	100	1.000	1.250	1.667	3.333	5.000	10.000	50.000
Nitrate	25	0.250	0.313	0.417	0.833	1.250	2.500	12.500
O-Phosphate-p	150	1.500	1.875	2.500	5.000	7.500	15.000	75.000
Sulfate	150	1.500	1.875	2.500	5.000	7.500	15.000	75.000

Table A-11 Absorbance value for COD Calibration Curve.

COD (mg O ₂ /L)	Distilled water Volume (mL)	Tube No.	Absorbance	Distilled Water (mL)	COD Standard
BLANK	2.5	A	0.011	2.5	0

Potassium Hydrogen Phthalate Standard (mg O ₂ /L)*	Standard Volume (mL)	Tube No.	Absorbance	Distilled Water (mL)	COD Standard
200	2.5	B	0.080	2.0	0.5
400	2.5	C	0.161	1.5	1.0
600	2.5	D	0.223	1.0	1.5
800	2.5	E	0.273	0.5	2.0
1,000	2.5	F	0.334	0.0	2.5

Standards prepared from 10,000 mg O₂/L Potassium Hydrogen Phthalate Standard

Land Application Rate Calculation

Regulations:

Apply 8-tons/ hectare for a 5-year Period (dry weight basis).

1- Calculate cross-section area of columns.

Area= $3.14 (15.3)^2 / 4 = 183.85 \text{ cm}^2$ where 15.3 is the diameter.

2- Convert the area of 8tons/hectare to gr/cm².

= 8 000 000 / 100 000 000 cm².

3- Regulated load.

$183.85 \times 8\,000\,000 / 100\,000\,000 = 14.70 \text{ g}$

14.70 g is the amount of biosolids that could be applied based on dry weight for a cross-section area of 183.85 cm² (columns cross section area).

Since the cake biosolids contain 30% solids and 70% liquid.

14.7 gr. dry weight (0% liquid) = $14.7 \times 3.3333 = 49 \text{ gr.}$ of fresh biosolids.

Load of frozen biosolids cake applied on each column

		Based on fresh weight (g)	Based on dry weight (g)
Column A	Control	0	0
Column B	Regulated	49	14.7
Column C	Excessive	1000	300
Column D	Control	0	0
Column E	Regulated	49	14.7
Column F	Excessive	1000	300

APPENDIX B
RESULTS FOR CHAPTER 4
BIOSOLIDS BATCH TESTS

Table B-1 Anion Concentration for January.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	25gr/100mL	Fluoride	0.259	0.259	0.260	0.259
Fresh 14 days	25gr/100mL	Fluoride	0.263	0.262	0.263	0.263
Fresh 28 days	25gr/100mL	Fluoride	0.255	0.256	0.256	0.256
Frozen 1 day	25gr/100mL	Fluoride	0.309	0.309	0.309	0.309
Frozen 14 days	25gr/100mL	Fluoride	0.314	0.316	0.314	0.315
Frozen 28 days	25gr/100mL	Fluoride	0.321	0.321	0.321	0.321
Fresh 1 day	25gr/100mL	Chloride	2.592	2.591	2.591	2.591
Fresh 14 days	25gr/100mL	Chloride	3.111	3.111	3.110	3.111
Fresh 28 days	25gr/100mL	Chloride	3.876	3.876	3.878	3.877
Frozen 1 day	25gr/100mL	Chloride	3.694	3.694	3.694	3.694
Frozen 14 days	25gr/100mL	Chloride	4.453	4.451	4.452	4.452
Frozen 28 days	25gr/100mL	Chloride	4.378	4.376	4.377	4.377
Fresh 1 day	25gr/100mL	Nitrite-N	0.107	0.107	0.107	0.107
Fresh 14 days	25gr/100mL	Nitrite-N	0.098	0.098	0.097	0.098
Fresh 28 days	25gr/100mL	Nitrite-N	0.105	0.105	0.106	0.105
Frozen 1 day	25gr/100mL	Nitrite-N	0.127	0.129	0.127	0.128
Frozen 14 days	25gr/100mL	Nitrite-N	0.130	0.131	0.131	0.131
Frozen 28 days	25gr/100mL	Nitrite-N	0.133	0.133	0.133	0.133
Fresh 1 day	25gr/100mL	Bromide	0.226	0.226	0.228	0.227
Fresh 14 days	25gr/100mL	Bromide	0.212	0.212	0.213	0.212
Fresh 28 days	25gr/100mL	Bromide	0.248	0.248	0.249	0.248
Frozen 1 day	25gr/100mL	Bromide	0.298	0.298	0.299	0.298
Frozen 14 days	25gr/100mL	Bromide	0.306	0.306	0.306	0.306
Frozen 28 days	25gr/100mL	Bromide	0.311	0.310	0.312	0.311
Fresh 1 day	25gr/100mL	Nitrate-N	0.168	0.169	0.169	0.169
Fresh 14 days	25gr/100mL	Nitrate-N	0.159	0.159	0.159	0.159
Fresh 28 days	25gr/100mL	Nitrate-N	0.168	0.168	0.169	0.168
Frozen 1 day	25gr/100mL	Nitrate-N	0.164	0.164	0.165	0.164
Frozen 14 days	25gr/100mL	Nitrate-N	0.358	0.357	0.358	0.358
Frozen 28 days	25gr/100mL	Nitrate-N	0.311	0.311	0.312	0.311
Fresh 1 day	25gr/100mL	O-Phosphate	1.065	1.065	1.065	1.065
Fresh 14 days	25gr/100mL	O-Phosphate	1.112	1.112	1.112	1.112
Fresh 28 days	25gr/100mL	O-Phosphate	1.225	1.224	1.224	1.224
Frozen 1 day	25gr/100mL	O-Phosphate	2.081	2.081	2.082	2.081
Frozen 14 days	25gr/100mL	O-Phosphate	3.051	3.055	3.053	3.053
Frozen 28 days	25gr/100mL	O-Phosphate	3.121	3.119	3.119	3.120
Fresh 1 day	25gr/100mL	Sulfate	5.472	5.473	5.472	5.472
Fresh 14 days	25gr/100mL	Sulfate	3.256	3.257	3.257	3.257
Fresh 28 days	25gr/100mL	Sulfate	3.001	3.000	3.009	3.003
Frozen 1 day	25gr/100mL	Sulfate	5.917	5.918	5.918	5.918
Frozen 14 days	25gr/100mL	Sulfate	5.725	5.726	5.725	5.725
Frozen 28 days	25gr/100mL	Sulfate	5.801	5.801	5.806	5.803

Table B-2 Anion Concentration for February.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	25gr/100mL	Fluoride	0.333	0.332	0.333	0.333
Fresh 14 days	25gr/100mL	Fluoride	0.325	0.360	0.333	0.339
Fresh 28 days	25gr/100mL	Fluoride	0.336	0.335	0.335	0.335
Frozen 1 day	25gr/100mL	Fluoride	0.471	0.471	0.470	0.471
Frozen 14 days	25gr/100mL	Fluoride	0.480	0.481	0.481	0.481
Frozen 28 days	25gr/100mL	Fluoride	0.485	0.486	0.485	0.485
Fresh 1 day	25gr/100mL	Chloride	3.635	3.635	3.635	3.635
Fresh 14 days	25gr/100mL	Chloride	3.245	3.245	3.246	3.245
Fresh 28 days	25gr/100mL	Chloride	3.210	3.209	3.210	3.210
Frozen 1 day	25gr/100mL	Chloride	5.447	5.447	5.448	5.447
Frozen 14 days	25gr/100mL	Chloride	3.494	3.494	3.495	3.494
Frozen 28 days	25gr/100mL	Chloride	4.674	4.673	7.673	5.673
Fresh 1 day	25gr/100mL	Nitrite-N	0.082	0.082	0.083	0.082
Fresh 14 days	25gr/100mL	Nitrite-N	0.120	0.121	0.121	0.121
Fresh 28 days	25gr/100mL	Nitrite-N	0.093	0.094	0.094	0.094
Frozen 1 day	25gr/100mL	Nitrite-N	0.134	0.134	0.133	0.134
Frozen 14 days	25gr/100mL	Nitrite-N	0.137	0.138	0.137	0.137
Frozen 28 days	25gr/100mL	Nitrite-N	0.141	0.141	0.142	0.141
Fresh 1 day	25gr/100mL	Bromide	0.231	0.231	0.232	0.231
Fresh 14 days	25gr/100mL	Bromide	0.222	0.223	0.222	0.222
Fresh 28 days	25gr/100mL	Bromide	0.263	0.263	0.263	0.263
Frozen 1 day	25gr/100mL	Bromide	0.307	0.306	0.306	0.306
Frozen 14 days	25gr/100mL	Bromide	0.314	0.314	0.314	0.314
Frozen 28 days	25gr/100mL	Bromide	0.318	0.319	0.317	0.318
Fresh 1 day	25gr/100mL	Nitrate-N	0.130	0.130	0.131	0.130
Fresh 14 days	25gr/100mL	Nitrate-N	0.173	0.172	0.173	0.173
Fresh 28 days	25gr/100mL	Nitrate-N	0.162	0.162	0.163	0.162
Frozen 1 day	25gr/100mL	Nitrate-N	0.127	0.127	0.128	0.127
Frozen 14 days	25gr/100mL	Nitrate-N	0.382	0.382	0.382	0.382
Frozen 28 days	25gr/100mL	Nitrate-N	0.326	0.327	0.327	0.327
Fresh 1 day	25gr/100mL	O-Phosphate	2.156	2.155	2.155	2.155
Fresh 14 days	25gr/100mL	O-Phosphate	2.813	2.813	2.814	2.813
Fresh 28 days	25gr/100mL	O-Phosphate	2.726	2.726	2.728	2.727
Frozen 1 day	25gr/100mL	O-Phosphate	4.032	4.030	4.030	4.030
Frozen 14 days	25gr/100mL	O-Phosphate	4.999	4.997	4.998	4.998
Frozen 28 days	25gr/100mL	O-Phosphate	5.627	5.627	5.625	5.626
Fresh 1 day	25gr/100mL	Sulfate	3.426	3.426	3.428	3.427
Fresh 14 days	25gr/100mL	Sulfate	4.569	4.569	4.570	4.569
Fresh 28 days	25gr/100mL	Sulfate	3.905	3.902	3.902	3.903
Frozen 1 day	25gr/100mL	Sulfate	6.001	5.999	6.000	6.000
Frozen 14 days	25gr/100mL	Sulfate	5.463	5.463	5.464	5.463
Frozen 28 days	25gr/100mL	Sulfate	5.642	5.642	5.643	5.642

Table B-3 Anion Concentration for March.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	25gr/100mL	Fluoride	0.320	0.320	0.321	0.320
Fresh 14 days	25gr/100mL	Fluoride	0.331	0.331	0.331	0.331
Fresh 28 days	25gr/100mL	Fluoride	0.312	0.310	0.312	0.311
Frozen 1 day	25gr/100mL	Fluoride	0.396	0.396	0.396	0.396
Frozen 14 days	25gr/100mL	Fluoride	0.412	0.411	0.412	0.412
Frozen 28 days	25gr/100mL	Fluoride	0.410	0.410	0.411	0.410
Fresh 1 day	25gr/100mL	Chloride	4.057	4.058	4.058	4.058
Fresh 14 days	25gr/100mL	Chloride	2.998	2.997	2.997	2.997
Fresh 28 days	25gr/100mL	Chloride	2.472	2.475	2.473	2.473
Frozen 1 day	25gr/100mL	Chloride	4.855	4.855	4.855	4.855
Frozen 14 days	25gr/100mL	Chloride	5.609	5.611	5.610	5.610
Frozen 28 days	25gr/100mL	Chloride	4.088	4.090	4.090	4.089
Fresh 1 day	25gr/100mL	Nitrite-N	0.126	0.126	0.126	0.126
Fresh 14 days	25gr/100mL	Nitrite-N	0.107	0.108	0.108	0.108
Fresh 28 days	25gr/100mL	Nitrite-N	0.070	0.070	0.070	0.070
Frozen 1 day	25gr/100mL	Nitrite-N	0.148	0.147	0.146	0.147
Frozen 14 days	25gr/100mL	Nitrite-N	0.152	0.151	0.151	0.151
Frozen 28 days	25gr/100mL	Nitrite-N	0.150	0.151	0.151	0.151
Fresh 1 day	25gr/100mL	Bromide	0.221	0.221	0.223	0.222
Fresh 14 days	25gr/100mL	Bromide	0.235	0.235	0.235	0.235
Fresh 28 days	25gr/100mL	Bromide	0.255	0.255	0.255	0.255
Frozen 1 day	25gr/100mL	Bromide	0.317	0.317	0.318	0.317
Frozen 14 days	25gr/100mL	Bromide	0.321	0.321	0.321	0.321
Frozen 28 days	25gr/100mL	Bromide	0.325	0.325	0.324	0.325
Fresh 1 day	25gr/100mL	Nitrate-N	0.126	0.127	0.127	0.127
Fresh 14 days	25gr/100mL	Nitrate-N	0.160	0.161	0.160	0.160
Fresh 28 days	25gr/100mL	Nitrate-N	0.164	0.165	0.165	0.165
Frozen 1 day	25gr/100mL	Nitrate-N	0.195	0.196	0.197	0.196
Frozen 14 days	25gr/100mL	Nitrate-N	0.355	0.356	0.356	0.356
Frozen 28 days	25gr/100mL	Nitrate-N	0.375	0.369	0.376	0.373
Fresh 1 day	25gr/100mL	O-Phosphate	1.690	1.691	1.691	1.691
Fresh 14 days	25gr/100mL	O-Phosphate	2.154	2.155	2.154	2.154
Fresh 28 days	25gr/100mL	O-Phosphate	2.598	2.598	2.598	2.598
Frozen 1 day	25gr/100mL	O-Phosphate	3.399	3.401	3.401	3.400
Frozen 14 days	25gr/100mL	O-Phosphate	4.504	4.507	4.507	4.506
Frozen 28 days	25gr/100mL	O-Phosphate	4.770	4.773	4.773	4.772
Fresh 1 day	25gr/100mL	Sulfate	5.144	5.145	5.145	5.145
Fresh 14 days	25gr/100mL	Sulfate	4.222	4.222	4.223	4.222
Fresh 28 days	25gr/100mL	Sulfate	3.669	3.701	3.700	3.690
Frozen 1 day	25gr/100mL	Sulfate	5.482	5.483	5.482	5.482
Frozen 14 days	25gr/100mL	Sulfate	5.023	5.022	5.022	5.022
Frozen 28 days	25gr/100mL	Sulfate	5.144	5.142	5.144	5.143

Table B-4 Anion Concentration for January.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	50 gr/100mL	Fluoride	0.488	0.488	0.488	0.488
Fresh 14 days	50 gr/100mL	Fluoride	0.519	0.519	0.520	0.519
Fresh 28 days	50 gr/100mL	Fluoride	0.508	0.508	0.508	0.508
Frozen 1 day	50 gr/100mL	Fluoride	0.799	0.798	0.798	0.798
Frozen 14 days	50 gr/100mL	Fluoride	0.801	0.800	0.802	0.801
Frozen 28 days	50 gr/100mL	Fluoride	0.815	0.814	0.814	0.814
Fresh 1 day	50 gr/100mL	Chloride	5.222	5.223	5.221	5.222
Fresh 14 days	50 gr/100mL	Chloride	6.093	6.095	6.095	6.094
Fresh 28 days	50 gr/100mL	Chloride	8.168	8.166	8.166	8.167
Frozen 1 day	50 gr/100mL	Chloride	7.341	7.342	7.342	7.342
Frozen 14 days	50 gr/100mL	Chloride	8.874	8.875	8.876	8.875
Frozen 28 days	50 gr/100mL	Chloride	8.825	8.825	8.825	8.825
Fresh 1 day	50 gr/100mL	Nitrite-N	0.190	0.190	0.190	0.190
Fresh 14 days	50 gr/100mL	Nitrite-N	0.184	0.184	0.182	0.184
Fresh 28 days	50 gr/100mL	Nitrite-N	0.179	0.179	0.181	0.180
Frozen 1 day	50 gr/100mL	Nitrite-N	0.214	0.214	0.213	0.214
Frozen 14 days	50 gr/100mL	Nitrite-N	0.222	0.221	0.221	0.221
Frozen 28 days	50 gr/100mL	Nitrite-N	0.213	0.213	0.214	0.213
Fresh 1 day	50 gr/100mL	Bromide	0.441	0.441	0.440	0.441
Fresh 14 days	50 gr/100mL	Bromide	0.471	0.471	0.471	0.471
Fresh 28 days	50 gr/100mL	Bromide	0.521	0.521	0.521	0.521
Frozen 1 day	50 gr/100mL	Bromide	0.586	0.586	0.585	0.586
Frozen 14 days	50 gr/100mL	Bromide	0.602	0.601	0.601	0.601
Frozen 28 days	50 gr/100mL	Bromide	0.614	0.615	0.615	0.615
Fresh 1 day	50 gr/100mL	Nitrate-N	0.262	0.262	0.264	0.263
Fresh 14 days	50 gr/100mL	Nitrate-N	0.248	0.248	0.248	0.248
Fresh 28 days	50 gr/100mL	Nitrate-N	0.293	0.295	0.295	0.294
Frozen 1 day	50 gr/100mL	Nitrate-N	0.233	0.234	0.234	0.234
Frozen 14 days	50 gr/100mL	Nitrate-N	0.601	0.601	0.602	0.601
Frozen 28 days	50 gr/100mL	Nitrate-N	0.504	0.508	0.505	0.506
Fresh 1 day	50 gr/100mL	O-Phosphate	1.981	1.980	1.981	1.981
Fresh 14 days	50 gr/100mL	O-Phosphate	1.957	1.957	1.957	1.957
Fresh 28 days	50 gr/100mL	O-Phosphate	2.205	2.205	2.205	2.205
Frozen 1 day	50 gr/100mL	O-Phosphate	3.653	3.653	3.655	3.654
Frozen 14 days	50 gr/100mL	O-Phosphate	5.465	5.472	5.468	5.468
Frozen 28 days	50 gr/100mL	O-Phosphate	5.693	5.689	5.689	5.690
Fresh 1 day	50 gr/100mL	Sulfate	11.765	11.765	11.766	11.765
Fresh 14 days	50 gr/100mL	Sulfate	8.506	8.491	8.510	8.502
Fresh 28 days	50 gr/100mL	Sulfate	6.473	6.473	6.474	6.473
Frozen 1 day	50 gr/100mL	Sulfate	11.426	11.426	11.426	11.426
Frozen 14 days	50 gr/100mL	Sulfate	10.438	10.439	10.439	10.439
Frozen 28 days	50 gr/100mL	Sulfate	10.180	10.182	10.182	10.181

Table B-5 Anion Concentration for February.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	50 gr/100mL	Fluoride	0.638	0.638	0.638	0.638
Fresh 14 days	50 gr/100mL	Fluoride	0.648	0.648	0.648	0.648
Fresh 28 days	50 gr/100mL	Fluoride	0.652	0.652	0.651	0.652
Frozen 1 day	50 gr/100mL	Fluoride	0.824	0.825	0.824	0.824
Frozen 14 days	50 gr/100mL	Fluoride	0.820	0.821	0.821	0.821
Frozen 28 days	50 gr/100mL	Fluoride	0.837	0.836	0.836	0.836
Fresh 1 day	50 gr/100mL	Chloride	7.261	7.260	7.260	7.260
Fresh 14 days	50 gr/100mL	Chloride	6.478	6.480	6.480	6.479
Fresh 28 days	50 gr/100mL	Chloride	6.512	6.512	6.512	6.512
Frozen 1 day	50 gr/100mL	Chloride	10.995	10.996	10.996	10.996
Frozen 14 days	50 gr/100mL	Chloride	7.012	7.012	7.011	7.012
Frozen 28 days	50 gr/100mL	Chloride	9.342	9.341	9.340	9.341
Fresh 1 day	50 gr/100mL	Nitrite-N	0.149	0.149	0.151	0.150
Fresh 14 days	50 gr/100mL	Nitrite-N	0.209	0.211	0.211	0.210
Fresh 28 days	50 gr/100mL	Nitrite-N	0.177	0.179	0.179	0.178
Frozen 1 day	50 gr/100mL	Nitrite-N	0.228	0.227	0.226	0.227
Frozen 14 days	50 gr/100mL	Nitrite-N	0.238	0.237	0.237	0.237
Frozen 28 days	50 gr/100mL	Nitrite-N	0.231	0.232	0.232	0.232
Fresh 1 day	50 gr/100mL	Bromide	0.491	0.491	0.491	0.491
Fresh 14 days	50 gr/100mL	Bromide	0.461	0.462	0.462	0.462
Fresh 28 days	50 gr/100mL	Bromide	0.519	0.519	0.518	0.519
Frozen 1 day	50 gr/100mL	Bromide	0.613	0.612	0.612	0.612
Frozen 14 days	50 gr/100mL	Bromide	0.610	0.611	0.610	0.610
Frozen 28 days	50 gr/100mL	Bromide	0.621	0.620	0.620	0.620
Fresh 1 day	50 gr/100mL	Nitrate-N	0.195	0.196	0.198	0.196
Fresh 14 days	50 gr/100mL	Nitrate-N	0.214	0.215	0.215	0.214
Fresh 28 days	50 gr/100mL	Nitrate-N	0.308	0.309	0.311	0.309
Frozen 1 day	50 gr/100mL	Nitrate-N	0.240	0.241	0.243	0.241
Frozen 14 days	50 gr/100mL	Nitrate-N	0.542	0.542	0.541	0.542
Frozen 28 days	50 gr/100mL	Nitrate-N	0.571	0.572	0.571	0.571
Fresh 1 day	50 gr/100mL	O-Phosphate	3.902	3.901	3.901	3.901
Fresh 14 days	50 gr/100mL	O-Phosphate	4.838	4.838	4.840	4.839
Fresh 28 days	50 gr/100mL	O-Phosphate	5.316	5.316	5.320	5.317
Frozen 1 day	50 gr/100mL	O-Phosphate	11.376	11.376	11.377	11.376
Frozen 14 days	50 gr/100mL	O-Phosphate	12.968	12.968	12.970	12.969
Frozen 28 days	50 gr/100mL	O-Phosphate	14.122	14.122	14.124	14.123
Fresh 1 day	50 gr/100mL	Sulfate	5.749	5.748	5.749	5.749
Fresh 14 days	50 gr/100mL	Sulfate	9.646	9.645	9.645	9.645
Fresh 28 days	50 gr/100mL	Sulfate	6.182	6.182	6.123	6.162
Frozen 1 day	50 gr/100mL	Sulfate	12.110	12.110	12.110	12.110
Frozen 14 days	50 gr/100mL	Sulfate	9.833	9.832	9.833	9.833
Frozen 28 days	50 gr/100mL	Sulfate	10.923	10.924	10.924	10.924

Table B-6 Anion Concentration for March.

Condition	Dilution (g /mL)	Anion	Replicate Number (mg/L)			Average
			Rep. #1	Rep. #2	Rep. #3	
Fresh 1 day	50 gr/100mL	Fluoride	0.599	0.598	0.598	0.598
Fresh 14 days	50 gr/100mL	Fluoride	0.659	0.659	0.660	0.659
Fresh 28 days	50 gr/100mL	Fluoride	0.544	0.543	0.543	0.543
Frozen 1 day	50 gr/100mL	Fluoride	0.781	0.782	0.782	0.782
Frozen 14 days	50 gr/100mL	Fluoride	0.797	0.798	0.797	0.797
Frozen 28 days	50 gr/100mL	Fluoride	0.807	0.807	0.807	0.807
Fresh 1 day	50 gr/100mL	Chloride	8.188	8.187	8.188	8.188
Fresh 14 days	50 gr/100mL	Chloride	5.987	5.987	5.988	5.987
Fresh 28 days	50 gr/100mL	Chloride	5.033	5.030	5.031	5.031
Frozen 1 day	50 gr/100mL	Chloride	9.615	6.614	6.614	7.614
Frozen 14 days	50 gr/100mL	Chloride	11.314	11.314	11.314	11.314
Frozen 28 days	50 gr/100mL	Chloride	8.156	8.156	8.156	8.156
Fresh 1 day	50 gr/100mL	Nitrite-N	0.215	0.215	0.215	0.215
Fresh 14 days	50 gr/100mL	Nitrite-N	0.201	0.203	0.203	0.203
Fresh 28 days	50 gr/100mL	Nitrite-N	0.123	0.123	0.123	0.123
Frozen 1 day	50 gr/100mL	Nitrite-N	0.236	0.236	0.236	0.236
Frozen 14 days	50 gr/100mL	Nitrite-N	0.251	0.253	0.252	0.252
Frozen 28 days	50 gr/100mL	Nitrite-N	0.244	0.243	0.243	0.243
Fresh 1 day	50 gr/100mL	Bromide	0.477	0.476	0.476	0.476
Fresh 14 days	50 gr/100mL	Bromide	0.429	0.428	0.429	0.429
Fresh 28 days	50 gr/100mL	Bromide	0.499	0.499	0.500	0.499
Frozen 1 day	50 gr/100mL	Bromide	0.624	0.624	0.623	0.624
Frozen 14 days	50 gr/100mL	Bromide	0.618	0.618	0.618	0.618
Frozen 28 days	50 gr/100mL	Bromide	0.643	0.644	0.644	0.644
Fresh 1 day	50 gr/100mL	Nitrate-N	0.182	0.184	0.184	0.183
Fresh 14 days	50 gr/100mL	Nitrate-N	0.283	0.283	0.282	0.283
Fresh 28 days	50 gr/100mL	Nitrate-N	0.291	0.291	0.291	0.291
Frozen 1 day	50 gr/100mL	Nitrate-N	0.314	0.315	0.315	0.315
Frozen 14 days	50 gr/100mL	Nitrate-N	0.650	0.650	0.651	0.650
Frozen 28 days	50 gr/100mL	Nitrate-N	0.533	0.534	0.534	0.534
Fresh 1 day	50 gr/100mL	O-Phosphate	2.958	2.959	2.959	2.959
Fresh 14 days	50 gr/100mL	O-Phosphate	3.963	3.965	3.963	3.964
Fresh 28 days	50 gr/100mL	O-Phosphate	4.911	4.910	4.910	4.910
Frozen 1 day	50 gr/100mL	O-Phosphate	11.001	10.999	10.998	10.999
Frozen 14 days	50 gr/100mL	O-Phosphate	13.359	13.357	13.356	13.357
Frozen 28 days	50 gr/100mL	O-Phosphate	14.829	14.827	14.827	14.828
Fresh 1 day	50 gr/100mL	Sulfate	11.090	11.090	11.090	11.090
Fresh 14 days	50 gr/100mL	Sulfate	7.557	7.558	7.558	7.558
Fresh 28 days	50 gr/100mL	Sulfate	6.523	6.521	6.521	6.522
Frozen 1 day	50 gr/100mL	Sulfate	10.756	10.756	10.757	10.756
Frozen 14 days	50 gr/100mL	Sulfate	9.324	9.324	9.324	9.324
Frozen 28 days	50 gr/100mL	Sulfate	9.595	9.597	9.609	9.600

Table B-7 Fresh versus Frozen samples after 1 day for Jan – Feb – March.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 50 gr/100 mL</u>						
		F	Cl ⁻	NO ₂ -N	Br ⁻	NO ₃ -N	HPO ₄ ⁻	SO ₄ ⁻
Fresh 1 day	January	0.488	5.222	0.190	0.441	0.263	1.981	11.765
Fresh 1 day	February	0.638	7.260	0.150	0.491	0.196	3.901	5.749
Fresh 1 day	March	0.598	8.188	0.215	0.476	0.183	2.959	11.090
Average		0.575	6.890	0.185	0.469	0.214	2.947	9.535
Sum of Square		0.012	4.603	0.002	0.001	0.004	1.844	21.729
Std. Dev.		0.078	1.517	0.033	0.026	0.043	0.960	3.296
Frozen 1 day	January	0.798	7.342	0.214	0.586	0.234	3.654	11.426
Frozen 1 day	February	0.824	10.996	0.227	0.612	0.241	11.376	12.110
Frozen 1 day	March	0.782	7.614	0.236	0.624	0.315	10.999	10.756
Average		0.801	8.651	0.226	0.607	0.263	8.676	11.431
Sum of Square		0.001	8.286	0.000	0.001	0.004	37.912	0.916
Std. Dev.		0.022	2.035	0.011	0.020	0.045	4.354	0.677

Table B-8 Fresh versus Frozen samples after 14 days for Jan – Feb – March.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 50 gr/100 mL</u>						
		F	Cl ⁻	NO ₂ -N	Br ⁻	NO ₃ -N	HPO ₄ ⁻	SO ₄ ⁻
Fresh 14 days	January	0.519	6.094	0.184	0.471	0.248	1.957	8.502
Fresh 14 days	February	0.648	6.479	0.210	0.462	0.215	4.839	9.645
Fresh 14 days	March	0.659	5.987	0.203	0.429	0.283	3.964	7.558
Average		0.609	6.187	0.199	0.454	0.249	3.587	8.568
Sum of Square		0.012	0.134	0.000	0.001	0.002	4.366	2.186
Std. Dev.		0.078	0.259	0.014	0.022	0.034	1.478	1.045
Frozen 14 days	January	0.801	8.875	0.221	0.601	0.601	5.468	10.439
Frozen 14 days	February	0.821	7.012	0.237	0.610	0.542	12.969	9.833
Frozen 14 days	March	0.797	11.314	0.252	0.618	0.650	13.357	9.324
Average		0.806	9.067	0.237	0.610	0.598	10.598	9.865
Sum of Square		0.000	9.310	0.000	0.000	0.006	39.549	0.623
Std. Dev.		0.013	2.158	0.015	0.008	0.054	4.447	0.558

Table B-9 Fresh versus Frozen samples after 28 days for Jan – Feb – March.

<u>Sample State</u>	<u>Sample Date</u>	<u>Concentration mg/L, for 50 gr/100 mL</u>						
		F	Cl ⁻	NO ₂ -N	Br ⁻	NO ₃ -N	HPO ₄ ⁻	SO ₄ ⁻
Fresh 28 days	January	0.508	8.167	0.180	0.521	0.294	2.205	6.473
Fresh 28 days	February	0.652	6.512	0.178	0.519	0.309	5.317	6.162
Fresh 28 days	March	0.543	5.031	0.123	0.499	0.291	4.910	6.522
Average		0.568	6.570	0.160	0.513	0.298	4.144	6.386
Sum of Square		0.011	4.920	0.002	0.000	0.000	5.723	0.076
Std. Dev.		0.075	1.568	0.032	0.012	0.010	1.692	0.195
Frozen 28 days	January	0.814	8.825	0.213	0.615	0.506	5.690	10.181
Frozen 28 days	February	0.836	9.341	0.232	0.620	0.571	14.123	10.924
Frozen 28 days	March	0.807	8.156	0.243	0.644	0.534	14.828	9.600
Average		0.819	8.774	0.229	0.626	0.537	11.547	10.235
Sum of Square		0.000	0.706	0.000	0.000	0.002	51.699	0.880
Std. Dev.		0.015	0.594	0.015	0.015	0.033	5.084	0.663

Figure B-1 Fluoride concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-2 Fluoride concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-3 Chloride concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-4 Chloride concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-5 Nitrite-N concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-6 Nitrite-N concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-7 Bromide concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-8 Bromide concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-9 Nitrate-N concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-10 Nitrate-N concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-11 Phosphate-P concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-12 Phosphate-P concentration for 1 kg of biosolids based on 50 g calculations.

Figure B-13 Sulfate concentration for 1 kg of biosolids based on 25 g calculations.

Figure B-14 Sulfate concentration for 1 kg of biosolids based on 50 g calculations.

Table B.10 Fresh versus Frozen concentrations for ammonia based on 50 g calculations.

<u>Ammonia (mg/L) for 50 g / 100 ml</u>				
	Concentration mg/L (range)	Concentration Mg/Kg (range)	Mean	
<u>FRESH SAMPLE</u>				
January	1507 - 1526	3014 - 3052	3033	
Fresh 1 day	1351 - 1362	2702 - 2724	2713	
Fresh 14 days	1298 - 1324	2596 - 2648	2622	
Fresh 28 days				
February	1231 - 1241	2462 - 2482	2472	
Fresh 1 day	1135 - 1168	2270 - 2336	2303	
Fresh 14 days	1104 - 1135	2208 - 2270	2239	
Fresh 28 days				
March	1154 - 1182	2308 - 2364	2336	
Fresh 1 day	1077 - 1099	2154 - 2198	2176	
Fresh 14 days	1055 - 1095	2110 - 2190	2150	
Fresh 28 days				
<u>FROZEN SAMPLE</u>				
January	1495 - 1551	2990 - 3102	3046	
Frozen 1 day	1513 - 1544	3026 - 3088	3057	
Frozen 14 days	1551 - 1576	3102 - 3152	3127	
Frozen 28 days				
February	1201 - 1236	2402 - 2472	2437	
Frozen 1 day	1216 - 1236	2432 - 2472	2452	
Frozen 14 days	1231 - 1287	2462 - 2574	2518	
Frozen 28 days				
March	1211 - 1241	2422 - 2482	2452	
Frozen 1 day	1266 - 1287	2532 - 2574	2553	
Frozen 14 days	1287 - 1303	2574 - 2606	2590	
Frozen 28 days	1507 - 1526	3014 - 3052	3033	

Table B-11 pH concentrations for 50 g biosolids calculations.

<u>pH for 50 g / 100 mL</u>		
	<u>Fresh</u>	<u>F/T</u>
January		
1 day	8.09 - 8.11	8.09 - 8.09
14 days	7.73 - 7.73	8.14 - 8.13
28 days	8.07 - 8.05	8.14 - 8.12
February		
1 day	8.08 - 8.06	8.08 - 8.10
14 days	8.09 - 8.10	8.15 - 8.14
28 days	8.11 - 8.11	8.14 - 8.14
March		
1 day	8.06 - 8.06	8.07 - 8.08
14 days	7.96 - 7.98	8.13 - 8.14
28 days	8.03 - 8.04	8.16 - 8.16

Table B-12 Average and Standard Deviation for COD Concentrations.

State	Jan.	Feb.	March	Avg.	Std. Dev.
Fresh 1 day	1512	1616	1501	1543	63
Frozen 1 day	1171	1206	1248	1208	39
Fresh 14 days	1509	1501	1410	1473	55
Frozen 14 days	1128	1185	1188	1167	34
Fresh 28 days	1267	1414	1290	1324	79
Frozen 28 days	878	1156	1042	1025	140

Table B-13 Fecal coliform calculation before and after freezing.

Fresh Samples								
Sample NO.	% Solids	10 ⁻⁵ mL	10 ⁻⁴ mL	10 ⁻³ mL	CFU/ g	log ₁₀	Average	Geometric Mean
1	30	0	4	16	6.01E+04	4.78E+00	4.9	8.11E+04
2	30	1	8	23	9.61E+04	4.98E+00		
3	30	1	6	21	8.41E+04	4.92E+00		
4	30	2	10	23	1.05E+05	5.02E+00		
5	30	0	5	18	6.91E+04	4.84E+00		
6	30	0	6	19	7.51E+04	4.88E+00		
7	30	1	7	21	8.71E+04	4.94E+00		

Frozen Samples								
Sample NO.	% Solids	10 ⁻⁵ mL	10 ⁻⁴ mL	10 ⁻³ mL	CFU/ g	log ₁₀	Average	Geometric Mean
1	30	0	0	0	0			0
2	30	0	0	0	0			
3	30	0	0	0	0			
4	30	0	0	1	0			
5	30	0	0	0	0			
6	30	0	0	0	0			
7	30	0	0	0	0			

Table B-14 Total coliform calculation before and after freezing (Fresh versus F/T)

Fresh Samples								
Sample NO.	% Solids	10 ⁻⁵ mL	10 ⁻⁴ mL	10 ⁻³ mL	CFU/ g	log ₁₀	Average	Geometric Mean
1	30	17	55	TNTC*	2.18E+06	6.34E+00	6.3	1.89E+06
2	30	8	41	TNTC	1.48E+06	6.17E+00		
3	30	15	52	TNTC	2.03E+06	6.31E+00		
4	30	15	49	TNTC	1.94E+06	6.29E+00		
5	30	21	59	TNTC	2.42E+06	6.38E+00		
6	30	10	44	TNTC	1.64E+06	6.21E+00		
7	30	12	45	TNTC	1.73E+06	6.24E+00		

Frozen Samples								
Sample NO.	% Solids	10 ⁻⁵ mL	10 ⁻⁴ mL	10 ⁻³ mL	CFU/ g	log ₁₀	Average	Geometric Mean
1	30	5	14	TNTC	5.76E+05	5.76E+00	5.9	7.89E+05
2	30	9	26	TNTC	1.06E+06	6.03E+00		
3	30	5	16	TNTC	6.36E+05	5.80E+00		
4	30	4	17	TNTC	6.36E+05	5.80E+00		
5	30	8	21	TNTC	8.79E+05	5.94E+00		
6	30	7	27	TNTC	1.03E+06	6.01E+00		
7	30	6	22	TNTC	8.48E+05	5.93E+00		

*TNTC: Too Number To Count

Statistical data for 25 grams samples

t-Test: Paired Two Sample for Means

Fluoride (1 Day)	Variable 1	Variable 2
Mean	0.304	0.392
Variance	0.002	0.007
Observations	3	3
Pooled Variance	0.951	
Hyp. Mean Diff.	0.000	
Df	2.000	
t Stat	-3.371	
P(T<=t) one-tail	0.039	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.078	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Chloride (1 Day)	Variable 1	Variable 2
Mean	3.428	4.665
Variance	0.570	0.795
Observations	3	3
Pooled Variance	0.813	
Hyp. Mean Diff.	0	
df	2	
t Stat	-4.116	
P(T<=t) one-tail	0.027	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.054	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Fluoride (14 Days)	Variable 1	Variable 2
Mean	0.311	0.403
Variance	0.002	0.007
Observations	3	3
Pooled Variance	0.946	
Hyp. Mean Diff.	0.000	
df	2.000	
t Stat	-3.456	
P(T<=t) one-tail	0.037	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.074	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Chloride (14 Days)	Variable 1	Variable 2
Mean	3.118	4.519
Variance	0.015	1.123
Observations	3	3
Pooled Variance	-0.995	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.051	
P(T<=t) one-tail	0.088	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.177	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Fluoride (28 Days)	Variable 1	Variable 2
Mean	0.301	0.405
Variance	0.002	0.007
Observations	3	3
Pooled Variance	0.985	
Hyp. Mean Diff.	0.000	
df	2.000	
t Stat	-4.237	
P(T<=t) one-tail	0.026	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.051	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Chloride (28 Days)	Variable 1	Variable 2
Mean	3.187	4.713
Variance	0.493	0.712
Observations	3	3
Pooled Variance	0.199	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.685	
P(T<=t) one-tail	0.058	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.115	
t Critical two-tail	4.303	

Statistical data for 25 grams samples

t-Test: Paired Two Sample for Means		
Nitrite-N (1 Day)	Variable 1	Variable 2
Mean	0.105	0.136
Variance	0.000	0.000
Observations	3	3
Pooled Variance	0.609	
Hyp. Mean Diff.	0	
df	2	
t Stat	-3.032	
P(T<=t) one-tail	0.047	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.094	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Bromide (1 Day)	Variable 1	Variable 2
Mean	0.227	0.307
Variance	0.000	0.000
Observations	3	3
Pooled Variance	-0.628	
Hyp. Mean Diff.	0	
df	2	
t Stat	-10.821	
P(T<=t) one-tail	0.004	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.008	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Nitrite-N (14 Days)	Variable 1	Variable 2
Mean	0.109	0.140
Variance	0.000	0.000
Observations	3	3
Pooled Variance	0.220	
Hyp. Mean Diff.	0	
df	2	
t Stat	-3.891	
P(T<=t) one-tail	0.030	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.060	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Bromide (14 Days)	Variable 1	Variable 2
Mean	0.223	0.314
Variance	0.000	0.000
Observations	3	3
Pooled Variance	0.994	
Hyp. Mean Diff.	0	
df	2	
t Stat	-37.720	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.001	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Nitrite-N (28 Days)	Variable 1	Variable 2
Mean	0.090	0.142
Variance	0.000	0.000
Observations	3	3
Pooled Variance	-0.989	
Hyp. Mean Diff.	0	
df	2	
t Stat	-3.354	
P(T<=t) one-tail	0.039	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.079	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Bromide (28 Days)	Variable 1	Variable 2
Mean	0.255	0.318
Variance	0.000	0.000
Observations	3	3
Pooled Variance	0.466	
Hyp. Mean Diff.	0	
df	2	
t Stat	-14.462	
P(T<=t) one-tail	0.002	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.005	
t Critical two-tail	4.303	

Statistical data for 25 grams samples

t-Test: Paired Two Sample for Means		
Nitrate-N (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.142	0.162
Variance	0.001	0.001
Observations	3	3
Pooled Variance	-0.022	
Hyp. Mean Diff.	0	
df	2	
t Stat	-0.835	
P(T<=t) one-tail	0.246	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.491	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Nitrate-N (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.164	0.365
Variance	0.000	0.000
Observations	3	3
Pooled Variance	0.991	
Hyp. Mean Diff.	0	
df	2	
t Stat	-51.231	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.000	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Nitrate-N (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.165	0.337
Variance	0.000	0.001
Observations	3	3
Pooled Variance	-0.249	
Hyp. Mean Diff.	0	
df	2	
t Stat	-9.011	
P(T<=t) one-tail	0.006	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.012	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
O-phosphate-p (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	1.637	3.170
Variance	0.299	0.989
Observations	3	3
Pooled Variance	0.993	
Hyp. Mean Diff.	0	
df	2	
t Stat	-5.829	
P(T<=t) one-tail	0.014	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.028	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
O-phosphate-p (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	2.026	4.186
Variance	0.736	1.023
Observations	3	3
Pooled Variance	0.989	
Hyp. Mean Diff.	0	
df	2	
t Stat	-18.094	
P(T<=t) one-tail	0.002	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.003	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
O-phosphate-p (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	2.183	4.506
Variance	0.694	1.623
Observations	3	3
Pooled Variance	0.965	
Hyp. Mean Diff.	0	
df	2	
t Stat	-7.770	
P(T<=t) one-tail	0.008	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.016	
t Critical two-tail	4.303	

Statistical data for 25 grams samples

t-Test: Paired Two Sample for Means		
Sulfate (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	4.681	5.800
Variance	1.207	0.078
Observations	3	3
Pooled Variance	-0.499	
Hyp. Mean Diff.	0	
df	2	
t Stat	-1.537	
P(T<=t) one-tail	0.132	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.264	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Ammonia (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	658	662
Variance	6466	6316
Observations	6	6
Pooled Variance	0.999	
Hyp. Mean Diff.	0	
df	5	
t Stat	-3.303	
P(T<=t) one-tail	0.011	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.021	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means		
Sulfate (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	4.016	5.403
Variance	0.462	0.126
Observations	3	3
Pooled Variance	-0.600	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.564	
P(T<=t) one-tail	0.062	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.124	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Ammonia (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	598	672
Variance	5386	6000
Observations	6	6
Pooled Variance	0.95327609	
Hyp. Mean Diff.	0	
df	5	
t Stat	-7.727	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.001	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means		
Sulfate (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	3.532	5.529
Variance	0.221	0.118
Observations	3	3
Pooled Variance	-0.507	
Hyp. Mean Diff.	0	
df	2	
t Stat	-4.875	
P(T<=t) one-tail	0.020	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.040	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means		
Ammonia (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	583	690
Variance	4905	6103
Observations	6	6
Pooled Variance	0.962	
Hyp. Mean Diff.	0	
df	5	
t Stat	-11.869	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.000	
t Critical two-tail	2.571	

Statistical data for 25 grams samples

t-Test: Paired Two Sample for Means

COD (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	1543	1208
Variance	3451	1432
Observations	6	6
Pooled Variance	-0.207	
Hyp. Mean Diff.	0	
df	5	
t Stat	10.768	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.000	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means

COD (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	1473	1167
Variance	2620	1148
Observations	6	6
Pooled Variance	-0.546	
Hyp. Mean Diff.	0	
df	5	
t Stat	9.979	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.000	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means

COD (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	1324	1026
Variance	5182	15980
Observations	6	6
Pooled Variance	0.845	
Hyp. Mean Diff.	0	
df	5	
t Stat	9.592	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.000	
t Critical two-tail	2.571	

Statistical data for 50 grams samples

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Fluoride (1 Day)	Variable 1	Variable 2	Chloride (1 Day)	Variable 1	Variable 2
Mean	0.575	0.801	Mean	6.890	8.651
Variance	0.006	0.000	Variance	2.302	4.144
Observations	3	3	Observations	3	3
Pooled Variance	0.387		Pooled Variance	0.276	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
Df	2		df	2	
t Stat	-5.439		t Stat	-1.401	
P(T<=t) one-tail	0.016		P(T<=t) one-tail	0.148	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.032		P(T<=t) two-tail	0.296	
t Critical two-tail	4.303		t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Fluoride (14 Days)	Variable 1	Variable 2	Chloride (14 Days)	Variable 1	Variable 2
Mean	0.609	0.806	Mean	6.187	9.067
Variance	0.006	0.000	Variance	0.067	4.654
Observations	3	3	Observations	3	3
Pooled Variance	0.292		Pooled Variance	-0.924	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
df	2		df	2	
t Stat	-4.559		t Stat	-2.080	
P(T<=t) one-tail	0.022		P(T<=t) one-tail	0.087	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.045		P(T<=t) two-tail	0.173	
t Critical two-tail	4.303		t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Fluoride (28 Days)	Variable 1	Variable 2	Chloride (28 Days)	Variable 1	Variable 2
Mean	0.568	0.819	Mean	6.570	8.774
Variance	0.006	0.000	Variance	2.461	0.353
Observations	3	3	Observations	3	3
Pooled Variance	0.892		Pooled Variance	0.536	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
df	2		df	2	
t Stat	-7.024		t Stat	-2.834	
P(T<=t) one-tail	0.010		P(T<=t) one-tail	0.053	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.020		P(T<=t) two-tail	0.105	
t Critical two-tail	4.303		t Critical two-tail	4.303	

Statistical data for 50 grams samples

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Nitrite-N (1 Day)	Variable 1	Variable 2	Bromide (1 Day)	Variable 1	Variable 2
Mean	0.185	0.226	Mean	0.469	0.607
Variance	0.001	0.000	Variance	0.001	0.000
Observations	3	3	Observations	3	3
Pooled Variance	0.283		Pooled Variance	0.819	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
df	2		df	2	
t Stat	-2.236		t Stat	-16.152	
P(T<=t) one-tail	0.077		P(T<=t) one-tail	0.002	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.155		P(T<=t) two-tail	0.004	
t Critical two-tail	4.303		t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Nitrite-N (14 Days)	Variable 1	Variable 2	Bromide (14 Days)	Variable 1	Variable 2
Mean	0.199	0.237	Mean	0.454	0.610
Variance	0.000	0.000	Variance	0.000	0.000
Observations	3	3	Observations	3	3
Pooled Variance	0.719		Pooled Variance	-0.938	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
df	2		df	2	
t Stat	-5.923		t Stat	-8.917	
P(T<=t) one-tail	0.014		P(T<=t) one-tail	0.006	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.057		P(T<=t) two-tail	0.012	
t Critical two-tail	4.303		t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means			t-Test: Paired Two Sample for Means		
Nitrite-N (28 Days)	Variable 1	Variable 2	Bromide (28 Days)	Variable 1	Variable 2
Mean	0.160	0.229	Mean	0.513	0.626
Variance	0.001	0.000	Variance	0.000	0.000
Observations	3	3	Observations	3	3
Pooled Variance	-0.799		Pooled Variance	-0.997	
Hyp. Mean Diff.	0		Hyp. Mean Diff.	0	
df	2		df	2	
t Stat	-2.633		t Stat	-7.100	
P(T<=t) one-tail	0.060		P(T<=t) one-tail	0.010	
t Critical one-tail	2.920		t Critical one-tail	2.920	
P(T<=t) two-tail	0.119		P(T<=t) two-tail	0.019	
t Critical two-tail	4.303		t Critical two-tail	4.303	

Statistical data for 50 grams samples

t-Test: Paired Two Sample for Means

Nitrate-N (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.214	0.263
Variance	0.002	0.002
Observations	3	3
Pooled Variance	-0.684	
Hyp. Mean Diff.	0	
df	2	
t Stat	-1.060	
P(T<=t) one-tail	0.200	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.400	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Nitrate-N (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.249	0.598
Variance	0.001	0.003
Observations	3	3
Pooled Variance	0.998	
Hyp. Mean Diff.	0	
df	2	
t Stat	-29.781	
P(T<=t) one-tail	0.001	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.001	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Nitrate-N (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	0.298	0.537
Variance	0.000	0.001
Observations	3	3
Pooled Variance	0.825	
Hyp. Mean Diff.	0	
df	2	
t Stat	-16.402	
P(T<=t) one-tail	0.002	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.000	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

O-phosphate-p (1 Day)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	2.947	8.676
Variance	0.922	18.953
Observations	3	3
Pooled Variance	0.892	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.816	
P(T<=t) one-tail	0.053	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.106	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

O-phosphate-p (14 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	2.947	8.676
Variance	0.922	18.953
Observations	3	3
Pooled Variance	0.892	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.816	
P(T<=t) one-tail	0.053	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.106	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

O-phosphate-p (28 Days)	<u>Variable 1</u>	<u>Variable 2</u>
Mean	4.144	11.547
Variance	2.861	25.853
Observations	3	3
Pooled Variance	0.982	
Hyp. Mean Diff.	0	
df	2	
t Stat	-3.729	
P(T<=t) one-tail	0.032	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.065	
t Critical two-tail	4.303	

Statistical data for 50 grams samples

t-Test: Paired Two Sample for Means

Sulfate (1 Day)	Variable 1	Variable 2
Mean	9.535	11.431
Variance	10.862	0.458
Observations	3	3
Pooled Variance	-0.814	
Hyp. Mean Diff.	0	
df	2	
t Stat	-0.849	
P(T<=t) one-tail	0.243	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.485	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Ammonia (1 Day)	Variable 1	Variable 2
Mean	1307	1323
Variance	27425	24658
Observations	6	6
Pooled Variance	0.975	
Hyp. Mean Diff.	0	
df	5	
t Stat	-1.029	
P(T<=t) one-tail	0.175	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.351	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means

Sulfate (14 Days)	Variable 1	Variable 2
Mean	8.568	9.865
Variance	1.092	0.312
Observations	3	3
Pooled Variance	0.406	
Hyp. Mean Diff.	0	
df	2	
t Stat	-2.330	
P(T<=t) one-tail	0.073	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.145	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Ammonia (14 Days)	Variable 1	Variable 2
Mean	1199	1344
Variance	15923	21188
Observations	6	6
Pooled Variance	0.926	
Hyp. Mean Diff.	0	
df	5	
t Stat	-6.386	
P(T<=t) one-tail	0.001	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.001	
t Critical two-tail	2.571	

t-Test: Paired Two Sample for Means

Sulfate (28 Days)	Variable 1	Variable 2
Mean	6.386	10.235
Variance	0.038	0.440
Observations	3	3
Pooled Variance	-0.947	
Hyp. Mean Diff.	0	
df	2	
t Stat	-7.836	
P(T<=t) one-tail	0.008	
t Critical one-tail	2.920	
P(T<=t) two-tail	0.016	
t Critical two-tail	4.303	

t-Test: Paired Two Sample for Means

Ammonia (28 Days)	Variable 1	Variable 2
Mean	1169	1373
Variance	12904	22550
Observations	6	6
Pooled Variance	0.956	
Hyp. Mean Diff.	0	
df	5	
t Stat	-9.397	
P(T<=t) one-tail	0.000	
t Critical one-tail	2.015	
P(T<=t) two-tail	0.000	
t Critical two-tail	2.571	

APPENDIX C
RESULTS FOR CHAPTER 5
COLUMNS TEST

Table C-1 Change in percent moisture content respect to time for thawing Soil A-Column A.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	<i>4.9</i>	<i>21.1</i>	<i>5.0</i>	<i>25.2</i>	<i>5.1</i>	<i>28.4</i>	<i>5.2</i>	<i>35.6</i>	<i>6.0</i>	<i>67.6</i>
T = 5 min.	*	11.8 58.4	<i>5.0</i>	<i>25.2</i>	<i>5.1</i>	<i>28.4</i>	<i>5.2</i>	<i>35.6</i>	<i>6.0</i>	<i>67.6</i>
T = 45 min.	*	10.6 51.6	<i>5.1</i>	<i>28.7</i>	<i>5.1</i>	<i>28.4</i>	<i>5.2</i>	<i>35.6</i>	<i>6.0</i>	<i>67.6</i>
T = 3 hrs	*	9.2 42.6	*	8.3 36.3	<i>5.1</i>	<i>28.6</i>	<i>5.2</i>	<i>35.8</i>	<i>6.0</i>	<i>68.5</i>
T = 5h: 20 min.	*	9.1 42.0	*	8.3 36.3	<i>5.1</i>	<i>28.6</i>	<i>5.2</i>	<i>35.8</i>	<i>6.0</i>	<i>68.5</i>
T = 10h: 30 min.	*	7.1 25.6	*	9.7 45.9	<i>5.1</i>	<i>28.6</i>	<i>5.2</i>	<i>35.8</i>	<i>6.0</i>	<i>68.5</i>
T = 3 days		9.1 42.0		7.7 31.2	7.6	30.1	7.6	30.0	13.4	66.8

Italic number indicated K_d value from the frozen calibration curve

Table C-2 Change in percent moisture content respect to time for thawing Soil A-Column B.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	<i>4.9</i>	<i>22.0</i>	<i>5.0</i>	<i>24.6</i>	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 5 min.	11.5	56.7	<i>5.0</i>	<i>24.6</i>	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 45 min.	10.4	50.4	<i>5.1</i>	<i>27.8</i>	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 3 hrs	9.3	43.1	8.4	36.8	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 5h: 20 min.	9.2	42.6	8.4	36.7	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 10h: 30 min.	7.7	31.2	8.6	38.5	<i>5.1</i>	<i>28.9</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>66.4</i>
T = 3 days	9.2	42.6	7.3	27.6	7.5	29.4	8.2	35.1	13.7	68.1

Italic number indicated K_d value from the frozen calibration curve

Table C-3 Change in percent moisture content respect to time for thawing Soil A-Column C.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	4.9	21.4	5.0	25.1	5.1	29.6	5.1	31.4	5.8	61.5
T = 5 min.	9.5	44.7	5.0	25.1	5.1	29.6	5.1	31.4	5.8	61.5
T = 45 min.	8.8	40.1	5.1	28.6	5.1	29.6	5.1	31.4	5.8	61.5
T = 3 hrs	8.6	38.2	7.7	31.0	5.1	30.1	5.1	31.4	5.8	61.5
T = 5h: 20 min.	8.6	38.1	7.7	31.0	5.1	30.1	5.1	31.4	5.8	61.5
T = 10h: 30 min.	7.1	26.3	7.0	25.1	5.1	30.1	5.3	37.5	5.9	65.1
T = 3 days	8.6	38.1	7.0	25.1	7.6	30.1	8.4	36.6	13.3	66.3

Italic number indicated K_d value from the frozen calibration curve

Table C-4 Change in percent moisture content respect to time for thawing Soil B-Column D.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	<i>5.0</i>	<i>23.4</i>	<i>5.1</i>	<i>28.0</i>	<i>5.2</i>	<i>31.5</i>	<i>5.4</i>	<i>39.5</i>	<i>6.2</i>	<i>73.7</i>
T = 5 min.	13.9	63.7	<i>5.1</i>	<i>28.0</i>	<i>5.2</i>	<i>31.5</i>	<i>5.4</i>	<i>39.5</i>	<i>6.2</i>	<i>73.7</i>
T = 45 min.	12.3	56.2	<i>5.2</i>	<i>31.3</i>	<i>5.2</i>	<i>30.9</i>	<i>5.3</i>	<i>38.8</i>	<i>6.2</i>	<i>73.7</i>
T = 3 hrs	10.5	46.4	9.4	39.6	<i>5.2</i>	<i>31.2</i>	<i>5.3</i>	<i>39.0</i>	<i>6.2</i>	<i>74.7</i>
T = 5h: 20 min.	10.4	45.8	9.4	39.6	<i>5.2</i>	<i>31.2</i>	<i>5.3</i>	<i>39.0</i>	<i>6.2</i>	<i>74.7</i>
T = 10h: 30 min.	7.9	28.2	11.3	50.5	<i>5.2</i>	<i>31.5</i>	<i>5.4</i>	<i>39.4</i>	<i>6.2</i>	<i>75.4</i>
T = 3 days	10.4	45.8	8.6	34.0	8.5	32.8	8.6	33.4	16.1	72.8

Italic number indicated K_d value from the frozen calibration curve

Table C-5 Change in percent moisture content respect to time for thawing Soil B-Column E.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	5.0	23.5	5.1	26.3	5.2	30.9	5.3	36.9	6.1	71.0
T = 5 min.	13.3	60.7	5.1	26.3	5.2	30.9	5.3	36.9	6.1	71.0
T = 45 min.	11.9	53.9	5.1	29.7	5.2	30.9	5.3	36.9	6.1	71.0
T = 3 hrs	10.5	46.1	9.4	39.4	5.2	30.9	5.3	36.9	6.1	71.0
T = 5h: 20 min.	10.4	45.6	9.4	39.3	5.2	30.9	5.3	36.9	6.1	71.0
T = 10h: 30 min.	8.6	33.4	9.7	41.2	5.2	30.9	5.3	36.9	6.1	71.0
T = 3 days	10.4	45.6	8.0	29.5	8.3	31.5	9.1	37.6	16.1	72.9

Italic number indicated K_d value from the frozen calibration curve

Table C-6 Change in percent moisture content respect to time for thawing Soil B-Column F.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	<i>5.0</i>	<i>23.3</i>	<i>5.1</i>	<i>27.4</i>	<i>5.2</i>	<i>32.3</i>	<i>5.2</i>	<i>34.2</i>	<i>6.0</i>	<i>67.0</i>
T = 5 min.	10.7	47.4	<i>5.1</i>	<i>26.6</i>	<i>5.2</i>	<i>31.4</i>	<i>5.2</i>	<i>33.3</i>	<i>6.0</i>	<i>65.2</i>
T = 45 min.	10.2	44.1	<i>5.2</i>	<i>31.5</i>	<i>5.2</i>	<i>32.6</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>67.7</i>
T = 3 hrs	9.8	41.6	8.6	33.8	<i>5.2</i>	<i>32.8</i>	<i>5.2</i>	<i>34.2</i>	<i>6.0</i>	<i>67.0</i>
T = 5h: 20 min.	9.8	41.9	8.6	34.1	<i>5.2</i>	<i>33.1</i>	<i>5.2</i>	<i>34.5</i>	<i>6.0</i>	<i>67.7</i>
T = 10h: 30 min.	7.9	28.7	7.8	27.4	<i>5.7</i>	<i>32.8</i>	<i>5.4</i>	<i>40.9</i>	<i>6.1</i>	<i>71.0</i>
T = 3 days	9.8	41.9	7.8	27.4	8.5	32.8	9.5	39.9	16.0	72.3

Italic number indicated K_d value from the frozen calibration curve

Table C-7 Change in percent moisture content respect to time after thawing for Soil A-Column A.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	7.0	25.1	7.9	32.9	7.5	29.6	7.5	29.6	13.2	65.8
T = 5 min.	15.0	74.0	16.0	78.0	7.5	29.6	7.5	29.6	13.2	65.8
T = 45 min.	11.9	59.1	15.5	76.1	8.9	40.2	7.5	29.6	13.2	65.8
T = 3 hrs	9.0	41.0	10.2	49.2	11.3	55.6	8.0	33.4	13.2	65.8
T = 5h: 20 min.	7.9	32.6	9.1	41.7	10.8	53.2	9.3	43.5	13.3	66.1
T = 10h: 30 min.	7.4	28.7	8.0	33.5	8.1	34.3	10.3	49.6	19.0	89.2
T = 3 days	7.9	32.6	7.7	31.3	7.6	30.4	7.5	29.8	13.3	66.1

Table C-8 Change in percent moisture content respect to time after thawing for Soil A-Column B.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	6.9	23.9	7.8	32.4	7.5	29.8	7.5	29.4	13.5	67.4
T = 5 min.	15.0	73.8	16.0	78.2	7.5	29.8	7.5	29.4	13.5	67.4
T = 45 min.	12.1	60.4	15.3	75.3	8.8	39.8	7.5	29.4	13.5	67.4
T = 3 hrs	9.2	42.5	10.1	48.9	11.1	54.4	8.0	33.8	13.5	67.4
T = 5h: 20 min.	7.9	33.1	9.2	42.4	10.1	48.4	9.3	43.0	14.0	69.4
T = 10h: 30 min.	7.4	28.7	7.8	32.4	8.0	33.6	10.1	48.6	19.3	90.1
T = 3 days	7.9	33.1	7.7	31.3	7.6	30.4	7.5	29.8	13.2	66.0

Table C-9 Change in percent moisture content respect to time after thawing for Soil A-Column C

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	7.1	25.9	7.7	31.4	7.4	28.4	7.4	28.6	12.5	62.5
T = 5 min.	14.6	72.4	17.4	83.6	7.4	28.4	7.4	28.6	12.5	62.5
T = 45 min.	11.9	59.1	15.1	74.3	8.8	39.5	7.4	28.6	12.5	62.5
T = 3 hrs	8.9	40.5	9.9	47.6	10.7	52.4	7.9	32.8	12.5	62.5
T = 5h: 20 min.	7.7	30.9	9.1	41.9	10.3	49.7	8.9	40.4	13.3	66.4
T = 10h: 30 min.	7.4	29.1	8.0	33.5	8.1	34.3	10.3	49.6	19.0	89.2
T = 3 days	7.7	30.9	7.7	31.3	7.6	30.4	7.5	29.8	13.3	66.1

Table C-10 Change in percent moisture content respect to time after thawing for Soil B-Column D.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	8.0	28.9	8.8	35.1	8.3	31.2	8.3	31.9	15.1	68.7
T = 5 min.	18.2	80.2	18.0	79.5	8.3	31.2	8.3	31.9	15.1	68.7
T = 45 min.	13.2	60.4	17.4	77.6	9.4	39.4	8.3	31.9	15.1	68.7
T = 3 hrs	10.4	45.8	12.8	58.4	12.5	57.1	8.6	33.6	15.1	68.7
T = 5h : 20 min.	8.9	35.6	10.1	43.5	12.0	54.6	9.4	39.1	15.2	69.0
T = 10h: 30 min.	8.0	29.5	8.8	35.1	8.3	31.9	12.4	56.3	22.3	92.8
T = 3 days	8.9	35.6	8.8	34.9	8.3	31.3	8.4	32.0	15.1	68.6

Table C-11 Change in percent moisture content respect to time after thawing for Soil B-Column E.

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	7.8	27.4	8.6	34.1	8.2	30.8	8.2	30.7	14.3	65.4
T = 5 min.	17.9	79.4	17.8	79.1	8.2	30.8	8.2	30.7	14.3	65.4
T = 45 min.	13.1	59.8	17.2	76.8	9.3	38.4	8.2	30.7	14.3	65.5
T = 3 hrs	10.2	44.2	12.9	58.9	12.5	56.8	8.3	31.4	14.3	65.5
T = 5h: 20 min.	8.3	31.2	9.9	42.5	11.9	53.8	9.5	40.1	15.0	68.4
T = 10h: 30 min.	7.9	28.1	9.0	36.4	8.4	32.0	12.0	54.4	21.1	89.4
T = 3 days	8.3	31.2	8.6	33.9	8.2	31.0	8.3	31.4	14.6	66.7

Table C-12 Change in percent moisture content respect to time after thawing for Soil B-Column F

Depth (cm)	-7.0		-16.9		-35.2		-55.2		-85.2	
Cumulative time	% Volumetric Moisture Content									
	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC	K _d	%MC
T = 0	7.8	27.9	8.8	34.9	8.4	32.1	8.4	32.1	14.9	67.9
T = 5 min.	18.0	79.8	17.6	78.2	8.4	32.1	8.4	32.1	14.9	67.9
T = 45 min.	13.4	61.1	17.6	78.4	9.3	38.7	8.4	32.0	14.9	67.9
T = 3 hrs	10.5	46.1	12.8	58.6	12.5	56.9	8.6	33.8	14.9	68.0
T = 5h: 20 min.	8.8	34.9	10.2	44.5	12.7	58.2	9.3	38.5	15.2	69.2
T = 10h: 30 min.	8.0	29.5	8.7	34.7	8.4	32.1	12.2	55.4	22.8	94.3
T = 3 days	8.8	34.9	8.7	34.7	8.3	31.3	8.4	32.4	14.9	67.9

Table C-13 Temperature profile for thawing soil in column A

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C ⁰)					
Time 0	-10.3	-9.8	-9.7	-9.5	-9.2	-9
Time 5 min.	-9.3	-9.3	-9.4	-9.3	-9.3	-9.1
Time 45 min.	-6.2	-8.8	-9.2	-9	-9.2	-8.7
Time 3h	-3.1	-6.1	-7.2	-7.2	-7.3	-6.8
Time 5h: 20 min.	-1.1	-3.2	-4.4	-4.6	-5.1	-4.5
Time 10h: 30 min.	-0.6	-0.7	-1.3	-1.6	-2.1	-1.3
Time 12h: 30 min.	-0.1	-0.2	-0.4	-0.7	-1.2	-0.7
Time 24h: 30 min	1.1	1	1.2	1.1	1.1	1.1

Table C-14 Temperature profile for thawing soil in column B

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C ⁰)					
Time 0	-10.3	-9.8	-9.7	-9.6	-9.2	-9.1
Time 5 min.	-9.3	-9.3	-9.4	-9.3	-9.3	-9.1
Time 45 min.	-6.3	-8.8	-9.2	-9	-9.2	-8.7
Time 3h	-3.1	-6.1	-7.2	-7.3	-7.3	-6.9
Time 5h: 20 min.	-1.2	-3.2	-4.4	-4.6	-5.1	-4.5
Time 10h: 30 min.	-0.4	-0.7	-1.3	-1.8	-2.1	-1.4
Time 12h: 30 min.	-0.2	-0.2	-0.5	-0.7	-1.1	-0.7
Time 24h: 30 min	1.1	1.2	1	1	1.1	1.1

Table C-15 Temperature profile for thawing soil in column C

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C ⁰)					
Time 0	-10.5	-9.8	-9.7	-9.5	-9.2	-9
Time 5 min.	-9.3	-9.3	-9.4	-9.4	-9.3	-9.1
Time 45 min.	-6.2	-8.8	-9.2	-9	-9.2	-8.7
Time 3h	-3	-6.1	-7.2	-7.3	-7.4	-6.9
Time 5h: 20 min.	-1	-3.2	-4.5	-4.6	-5.1	-4.6
Time 10h: 30 min.	-0.5	-0.7	-1.3	-1.6	-2.1	-1.3
Time 12h: 30 min.	-0.3	-0.4	-0.8	-1.1	-1.1	-0.8
Time 24h: 30 min	1.1	1	1	0.9	1.1	1

Table C-16 Temperature profile for thawing soil in column D

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C⁰)					
Time 0	-9	-9.3	-10.1	-9.6	-10.3	-8.7
Time 5 min.	-9.3	-9.4	-9.4	-9.5	-9.5	-9.1
Time 45 min.	-7.5	-8.9	-9.4	-9.3	-9.4	-8.7
Time 3h	-4.5	-7.2	-7.5	-7.8	-7.8	-7.1
Time 5h: 20 min.	-2.1	-3.6	-4.5	-5.1	-5.6	-4.3
Time 10h: 30 min.	-0.5	-1.2	-1.7	-2.3	-2.7	-1.6
Time 12h: 30 min.	-0.2	-0.3	-0.5	-0.7	-1.2	-0.2
Time 24h: 30 min	0.9	1.1	1.1	1	1	1

Table C-17 Temperature profile for thawing soil in column E

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C⁰)					
Time 0	-9.1	-9.3	-10.2	-9.6	-10.3	-8.9
Time 5 min.	-9.3	-9.4	-9.5	-9.5	-9.5	-9.2
Time 45 min.	-7.5	-8.9	-9.4	-9.4	-9.4	-8.7
Time 3h	-4.6	-7.2	-7.6	-7.8	-7.8	-7.1
Time 5h: 20 min.	-2.1	-3.6	-4.5	-5.1	-5.6	-4.3
Time 10h: 30 min.	-0.5	-1.2	-1.7	-2.3	-2.7	-1.8
Time 12h: 30 min.	-0.2	-0.3	-0.7	-0.7	-1.2	-0.3
Time 24h: 30 min	0.9	1.1	1.1	1.1	1	1

Table C-18 Temperature profile for thawing soil in column F

Depth (cm)	-9.4	-19.2	-29.2	-39.2	-59.2	-89.2
Cumulative time	Temperature (C⁰)					
Time 0	-10.6	-9.8	-9.8	-9.5	-9.2	-9.2
Time 5 min.	-9.3	-9.3	-9.4	-9.3	-9.3	-9.1
Time 45 min.	-6.2	-8.8	-9.2	-9	-9.2	-8.7
Time 3h	-3.1	-6.1	-7.2	-7.2	-7.3	-6.7
Time 5h: 20 min.	-1.1	-3.2	-4.4	-4.6	-5.2	-4.5
Time 10h: 30 min.	-0.5	-0.8	-1.5	-1.6	-2.1	-1.3
Time 12h: 30 min.	-0.3	-0.5	-0.8	-1.2	-1.2	-0.3
Time 24h: 30 min	1	1	0.9	0.9	1	1

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	21.1	21.1	103
	17cm	9.9 cm	184	0.38	21.1	25.2	160
	35cm	18.3 cm	184	0.38	25.2	28.4	342
	55cm	20 cm	184	0.38	28.4	35.6	447
	85cm	30 cm	184	0.38	35.6	67.6	1082
							2134
T= 5 min	7 cm	7 cm	184	0.38	58.4	58.4	286
	17cm	9.9 cm	184	0.38	58.4	25.2	289
	35cm	18.3 cm	184	0.38	25.2	28.4	342
	55cm	20 cm	184	0.38	28.4	35.6	447
	85cm	30 cm	184	0.38	35.6	67.6	1082
							2445
T=45 min	7 cm	7 cm	184	0.38	51.6	51.6	252
	17cm	9.9 cm	184	0.38	51.6	28.7	278
	35cm	18.3 cm	184	0.38	28.7	28.4	365
	55cm	20 cm	184	0.38	28.4	35.6	447
	85cm	30 cm	184	0.38	35.6	67.6	1082
							2423
T= 3 h	7 cm	7 cm	184	0.38	42.6	42.6	208
	17cm	9.9 cm	184	0.38	42.6	36.3	273
	35cm	18.3 cm	184	0.38	36.3	28.6	415
	55cm	20 cm	184	0.38	28.6	35.8	450
	85cm	30 cm	184	0.38	35.8	68.5	1093
							2439
T= 5h: 20	7 cm	7 cm	184	0.38	42.0	42.0	205
	17cm	9.9 cm	184	0.38	42.0	36.3	271
	35cm	18.3 cm	184	0.38	36.3	28.6	415
	55cm	20 cm	184	0.38	28.6	35.8	450
	85cm	30 cm	184	0.38	35.8	68.5	1093
							2434
T= 10h: 30	7 cm	7 cm	184	0.38	25.6	25.6	125
	17cm	9.9 cm	184	0.38	25.6	45.9	247
	35cm	18.3 cm	184	0.38	45.9	28.6	476
	55cm	20 cm	184	0.38	28.6	35.8	450
	85cm	30 cm	184	0.38	35.8	68.5	1093
							2392
T= 3 days	7 cm	7 cm	184	0.38	22.3	22.3	109
	17cm	9.9 cm	184	0.38	22.3	31.2	185
	35cm	18.3 cm	184	0.38	31.2	30.1	392
	55cm	20 cm	184	0.38	30.1	30.0	420
	85cm	30 cm	184	0.38	30.0	66.8	1014
							2121

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	22.0	22.0	108
	17cm	9.9 cm	184	0.38	22.0	24.6	161
	35cm	18.3 cm	184	0.38	24.6	28.9	342
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2111
T= 5 min	7 cm	7 cm	184	0.38	56.7	56.7	277
	17cm	9.9 cm	184	0.38	56.7	24.6	281
	35cm	18.3 cm	184	0.38	24.6	28.9	342
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2401
T=45 min	7 cm	7 cm	184	0.38	50.4	50.4	246
	17cm	9.9 cm	184	0.38	50.4	27.8	270
	35cm	18.3 cm	184	0.38	27.8	28.9	362
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2380
T= 3 h	7 cm	7 cm	184	0.38	43.1	43.1	211
	17cm	9.9 cm	184	0.38	43.1	36.8	276
	35cm	18.3 cm	184	0.38	36.8	28.9	420
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2407
T= 5h: 20	7 cm	7 cm	184	0.38	42.6	42.6	208
	17cm	9.9 cm	184	0.38	42.6	36.7	274
	35cm	18.3 cm	184	0.38	36.7	28.9	419
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2402
T= 10h: 30	7 cm	7 cm	184	0.38	31.2	31.2	153
	17cm	9.9 cm	184	0.38	31.2	38.5	241
	35cm	18.3 cm	184	0.38	38.5	28.9	431
	55cm	20 cm	184	0.38	28.9	34.5	443
	85cm	30 cm	184	0.38	34.5	66.4	1057
							2325
T= 3 days	7 cm	7 cm	184	0.38	21.3	21.3	104
	17cm	9.9 cm	184	0.38	21.3	27.6	169
	35cm	18.3 cm	184	0.38	27.6	29.4	364
	55cm	20 cm	184	0.38	29.4	35.1	451
	85cm	30 cm	184	0.38	35.1	68.1	1082
							2170

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	21.4	21.4	105
	17cm	9.9 cm	184	0.38	21.4	25.1	161
	35cm	18.3 cm	184	0.38	25.1	29.6	350
	55cm	20 cm	184	0.38	29.6	31.4	426
	85cm	30 cm	184	0.38	31.4	61.5	974
							2015
T= 5 min	7 cm	7 cm	184	0.38	44.7	44.7	219
	17cm	9.9 cm	184	0.38	44.7	25.1	241
	35cm	18.3 cm	184	0.38	25.1	29.6	350
	55cm	20 cm	184	0.38	29.6	31.4	426
	85cm	30 cm	184	0.38	31.4	61.5	974
							2209
T=45 min	7 cm	7 cm	184	0.38	40.1	40.1	196
	17cm	9.9 cm	184	0.38	40.1	28.6	238
	35cm	18.3 cm	184	0.38	28.6	29.6	372
	55cm	20 cm	184	0.38	29.6	31.4	426
	85cm	30 cm	184	0.38	31.4	61.5	974
							2205
T= 3 h	7 cm	7 cm	184	0.38	38.2	38.2	187
	17cm	9.9 cm	184	0.38	38.2	31.0	239
	35cm	18.3 cm	184	0.38	31.0	30.1	391
	55cm	20 cm	184	0.38	30.1	31.4	430
	85cm	30 cm	184	0.38	31.4	61.5	974
							2220
T= 5h: 20	7 cm	7 cm	184	0.38	38.1	38.1	186
	17cm	9.9 cm	184	0.38	38.1	31.0	239
	35cm	18.3 cm	184	0.38	31.0	30.1	391
	55cm	20 cm	184	0.38	30.1	31.4	430
	85cm	30 cm	184	0.38	31.4	61.5	974
							2219
T= 10h: 30	7 cm	7 cm	184	0.38	26.3	26.3	129
	17cm	9.9 cm	184	0.38	26.3	25.1	178
	35cm	18.3 cm	184	0.38	25.1	30.1	353
	55cm	20 cm	184	0.38	30.1	37.5	472
	85cm	30 cm	184	0.38	37.5	65.1	1075
							2207
T= 3 days	7 cm	7 cm	184	0.38	21.3	21.3	104
	17cm	9.9 cm	184	0.38	21.3	25.1	160
	35cm	18.3 cm	184	0.38	25.1	30.1	353
	55cm	20 cm	184	0.38	30.1	36.6	466
	85cm	30 cm	184	0.38	36.6	66.3	1078
							2162

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	23.4	23.4	117
	17cm	9.9 cm	184	0.39	23.4	28.0	182
	35cm	18.3 cm	184	0.39	28.0	31.5	390
	55cm	20 cm	184	0.39	31.5	39.5	509
	85cm	30 cm	184	0.39	39.5	73.7	1218
							2416
T= 5 min	7 cm	7 cm	184	0.39	63.7	63.7	320
	17cm	9.9 cm	184	0.39	63.7	27.5	324
	35cm	18.3 cm	184	0.39	27.5	31.5	387
	55cm	20 cm	184	0.39	31.5	39.5	509
	85cm	30 cm	184	0.39	39.5	73.7	1217
							2757
T=45 min	7 cm	7 cm	184	0.39	56.2	56.2	282
	17cm	9.9 cm	184	0.39	56.2	31.3	311
	35cm	18.3 cm	184	0.39	31.3	30.9	408
	55cm	20 cm	184	0.39	30.9	38.8	500
	85cm	30 cm	184	0.39	38.8	73.7	1210
							2710
T= 3 h	7 cm	7 cm	184	0.39	46.4	46.4	233
	17cm	9.9 cm	184	0.39	46.4	39.6	305
	35cm	18.3 cm	184	0.39	39.6	31.2	464
	55cm	20 cm	184	0.39	31.2	39.0	503
	85cm	30 cm	184	0.39	39.0	74.7	1223
							2728
T= 5h: 20	7 cm	7 cm	184	0.39	45.8	45.8	230
	17cm	9.9 cm	184	0.39	45.8	39.6	303
	35cm	18.3 cm	184	0.39	39.6	31.2	464
	55cm	20 cm	184	0.39	31.2	39.0	503
	85cm	30 cm	184	0.39	39.0	74.7	1223
							2723
T= 10h: 30	7 cm	7 cm	184	0.39	28.2	28.2	141
	17cm	9.9 cm	184	0.39	28.2	50.5	279
	35cm	18.3 cm	184	0.39	50.5	31.5	538
	55cm	20 cm	184	0.39	31.5	39.4	508
	85cm	30 cm	184	0.39	39.4	75.4	1234
							2700
T= 3 days	7 cm	7 cm	184	0.39	24.3	24.3	122
	17cm	9.9 cm	184	0.39	24.3	34.0	207
	35cm	18.3 cm	184	0.39	34.0	32.8	439
	55cm	20 cm	184	0.39	32.8	33.4	475
	85cm	30 cm	184	0.39	33.4	72.8	1142
							2385

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	23.5	23.5	118
	17cm	9.9 cm	184	0.39	23.5	26.3	177
	35cm	18.3 cm	184	0.39	26.3	30.9	376
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2318
T= 5 min	7 cm	7 cm	184	0.39	60.7	60.7	305
	17cm	9.9 cm	184	0.39	60.7	26.3	309
	35cm	18.3 cm	184	0.39	26.3	30.9	376
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2637
T=45 min	7 cm	7 cm	184	0.39	53.9	53.9	271
	17cm	9.9 cm	184	0.39	53.9	29.7	297
	35cm	18.3 cm	184	0.39	29.7	30.9	398
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2613
T= 3 h	7 cm	7 cm	184	0.39	46.1	46.1	231
	17cm	9.9 cm	184	0.39	46.1	39.4	303
	35cm	18.3 cm	184	0.39	39.4	30.9	461
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2644
T= 5h: 20	7 cm	7 cm	184	0.39	45.6	45.6	229
	17cm	9.9 cm	184	0.39	45.6	39.3	301
	35cm	18.3 cm	184	0.39	39.3	30.9	461
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2638
T= 10h: 30	7 cm	7 cm	184	0.39	33.4	33.4	168
	17cm	9.9 cm	184	0.39	33.4	41.2	265
	35cm	18.3 cm	184	0.39	41.2	30.9	473
	55cm	20 cm	184	0.39	30.9	36.9	486
	85cm	30 cm	184	0.39	36.9	71.0	1161
							2553
T= 3 days	7 cm	7 cm	184	0.39	22.8	22.8	114
	17cm	9.9 cm	184	0.39	22.8	29.5	186
	35cm	18.3 cm	184	0.39	29.5	31.5	400
	55cm	20 cm	184	0.39	31.5	37.6	495
	85cm	30 cm	184	0.39	37.6	72.9	1188
							2383

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	23.3	23.3	117
	17cm	9.9 cm	184	0.39	23.3	27.4	180
	35cm	18.3 cm	184	0.39	27.4	32.3	391
	55cm	20 cm	184	0.39	32.3	34.2	477
	85cm	30 cm	184	0.39	34.2	67.0	1089
							2254
T= 5 min	7 cm	7 cm	184	0.39	47.4	47.4	238
	17cm	9.9 cm	184	0.39	47.4	26.6	263
	35cm	18.3 cm	184	0.39	26.6	31.4	380
	55cm	20 cm	184	0.39	31.4	33.3	464
	85cm	30 cm	184	0.39	33.3	65.2	1059
							2404
T=45 min	7 cm	7 cm	184	0.39	44.1	44.1	221
	17cm	9.9 cm	184	0.39	44.1	31.5	268
	35cm	18.3 cm	184	0.39	31.5	32.6	420
	55cm	20 cm	184	0.39	32.6	34.5	481
	85cm	30 cm	184	0.39	34.5	67.7	1099
							2490
T= 3 h	7 cm	7 cm	184	0.39	41.6	41.6	209
	17cm	9.9 cm	184	0.39	41.6	33.8	268
	35cm	18.3 cm	184	0.39	33.8	32.8	437
	55cm	20 cm	184	0.39	32.8	34.2	481
	85cm	30 cm	184	0.39	34.2	67.0	1089
							2483
T= 5h: 20	7 cm	7 cm	184	0.39	41.9	41.9	210
	17cm	9.9 cm	184	0.39	41.9	34.1	270
	35cm	18.3 cm	184	0.39	34.1	33.1	441
	55cm	20 cm	184	0.39	33.1	34.5	485
	85cm	30 cm	184	0.39	34.5	67.7	1099
							2505
T= 10h: 30	7 cm	7 cm	184	0.39	28.7	28.7	144
	17cm	9.9 cm	184	0.39	28.7	27.4	199
	35cm	18.3 cm	184	0.39	27.4	32.8	395
	55cm	20 cm	184	0.39	32.8	40.9	528
	85cm	30 cm	184	0.39	40.9	71.0	1203
							2469
T= 3 days	7 cm	7 cm	184	0.39	21.9	21.9	110
	17cm	9.9 cm	184	0.39	21.9	27.4	175
	35cm	18.3 cm	184	0.39	27.4	32.8	395
	55cm	20 cm	184	0.39	32.8	39.9	521
	85cm	30 cm	184	0.39	39.9	72.3	1206
							2407

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	25.1	25.1	123
	17cm	9.9 cm	184	0.38	25.1	32.9	201
	35cm	18.3 cm	184	0.38	32.9	29.6	400
	55cm	20 cm	184	0.38	29.6	29.6	414
	85cm	30 cm	184	0.38	29.6	65.8	1000
							2138
T= 5 min	7 cm	7 cm	184	0.38	74.0	74.0	362
	17cm	9.9 cm	184	0.38	74.0	78.0	526
	35cm	18.3 cm	184	0.38	78.0	29.6	688
	55cm	20 cm	184	0.38	29.6	29.6	414
	85cm	30 cm	184	0.38	29.6	65.8	1000
							2990
T=45 min	7 cm	7 cm	184	0.38	59.1	59.1	289
	17cm	9.9 cm	184	0.38	59.1	76.1	468
	35cm	18.3 cm	184	0.38	76.1	40.2	743
	55cm	20 cm	184	0.38	40.2	29.6	488
	85cm	30 cm	184	0.38	29.6	65.8	1000
							2988
T= 3 h	7 cm	7 cm	184	0.38	41.0	41.0	201
	17cm	9.9 cm	184	0.38	41.0	49.2	312
	35cm	18.3 cm	184	0.38	49.2	55.6	670
	55cm	20 cm	184	0.38	55.6	33.4	622
	85cm	30 cm	184	0.38	33.4	65.8	1040
							2845
T= 5h: 20	7 cm	7 cm	184	0.38	32.6	32.6	159
	17cm	9.9 cm	184	0.38	32.6	41.7	257
	35cm	18.3 cm	184	0.38	41.7	53.2	607
	55cm	20 cm	184	0.38	53.2	43.5	676
	85cm	30 cm	184	0.38	43.5	66.1	1149
							2848
T= 10h: 30	7 cm	7 cm	184	0.38	28.7	28.7	140
	17cm	9.9 cm	184	0.38	28.7	33.5	215
	35cm	18.3 cm	184	0.38	33.5	34.3	433
	55cm	20 cm	184	0.38	34.3	49.6	586
	85cm	30 cm	184	0.38	49.6	89.2	1455
							2829
T= 3 days	7 cm	7 cm	184	0.38	25.7	25.7	126
	17cm	9.9 cm	184	0.38	25.7	31.3	197
	35cm	18.3 cm	184	0.38	31.3	30.4	394
	55cm	20 cm	184	0.38	30.4	29.8	421
	85cm	30 cm	184	0.38	29.8	66.1	1005
							2143

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	23.9	23.9	117
	17cm	9.9 cm	184	0.38	23.9	32.4	195
	35cm	18.3 cm	184	0.38	32.4	29.8	398
	55cm	20 cm	184	0.38	29.8	29.4	414
	85cm	30 cm	184	0.38	29.4	67.4	1014
							2138
T= 5 min	7 cm	7 cm	184	0.38	74.0	73.8	361
	17cm	9.9 cm	184	0.38	73.8	78.2	526
	35cm	18.3 cm	184	0.38	78.2	29.8	690
	55cm	20 cm	184	0.38	29.8	29.4	414
	85cm	30 cm	184	0.38	29.4	67.4	1014
							3005
T=45 min	7 cm	7 cm	184	0.38	59.1	60.4	292
	17cm	9.9 cm	184	0.38	60.4	75.3	469
	35cm	18.3 cm	184	0.38	75.3	39.8	736
	55cm	20 cm	184	0.38	39.8	29.4	483
	85cm	30 cm	184	0.38	29.4	67.4	1014
							2994
T= 3 h	7 cm	7 cm	184	0.38	41.0	42.5	204
	17cm	9.9 cm	184	0.38	42.5	48.9	316
	35cm	18.3 cm	184	0.38	48.9	54.4	660
	55cm	20 cm	184	0.38	54.4	33.8	616
	85cm	30 cm	184	0.38	33.8	67.4	1061
							2857
T= 5h: 20	7 cm	7 cm	184	0.38	32.6	33.1	161
	17cm	9.9 cm	184	0.38	33.1	42.4	261
	35cm	18.3 cm	184	0.38	42.4	48.4	580
	55cm	20 cm	184	0.38	48.4	43.0	639
	85cm	30 cm	184	0.38	43.0	69.4	1178
							2819
T= 10h: 30	7 cm	7 cm	184	0.38	28.7	28.7	140
	17cm	9.9 cm	184	0.38	28.7	32.4	211
	35cm	18.3 cm	184	0.38	32.4	33.6	422
	55cm	20 cm	184	0.38	33.6	48.6	574
	85cm	30 cm	184	0.38	48.6	90.1	1454
							2801
T= 3 days	7 cm	7 cm	184	0.38	25.7	25.7	126
	17cm	9.9 cm	184	0.38	25.7	31.3	197
	35cm	18.3 cm	184	0.38	31.3	30.4	394
	55cm	20 cm	184	0.38	30.4	29.8	421
	85cm	30 cm	184	0.38	29.8	66.0	1004
							2142

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	25.9	25.9	127
	17cm	9.9 cm	184	0.38	25.9	31.4	198
	35cm	18.3 cm	184	0.38	31.4	28.4	382
	55cm	20 cm	184	0.38	28.4	28.6	398
	85cm	30 cm	184	0.38	28.6	62.5	955
							2060
T= 5 min	7 cm	7 cm	184	0.38	72.4	72.4	289
	17cm	9.9 cm	184	0.38	72.4	83.6	461
	35cm	18.3 cm	184	0.38	83.6	28.4	727
	55cm	20 cm	184	0.38	28.4	28.6	476
	85cm	30 cm	184	0.38	28.6	62.5	955
							2908
T=45 min	7 cm	7 cm	184	0.38	59.1	59.1	289
	17cm	9.9 cm	184	0.38	59.1	74.3	461
	35cm	18.3 cm	184	0.38	74.3	39.5	727
	55cm	20 cm	184	0.38	39.5	28.6	476
	85cm	30 cm	184	0.38	28.6	62.5	955
							2908
T= 3 h	7 cm	7 cm	184	0.38	40.5	40.5	198
	17cm	9.9 cm	184	0.38	40.5	47.6	305
	35cm	18.3 cm	184	0.38	47.6	52.4	639
	55cm	20 cm	184	0.38	52.4	32.8	595
	85cm	30 cm	184	0.38	32.8	62.5	999
							2736
T= 5h: 20	7 cm	7 cm	184	0.38	32.6	30.9	155
	17cm	9.9 cm	184	0.38	30.9	41.9	252
	35cm	18.3 cm	184	0.38	41.9	49.7	586
	55cm	20 cm	184	0.38	49.7	40.4	629
	85cm	30 cm	184	0.38	40.4	66.4	1119
							2741
T= 10h: 30	7 cm	7 cm	184	0.38	29.1	29.1	142
	17cm	9.9 cm	184	0.38	29.1	33.5	216
	35cm	18.3 cm	184	0.38	33.5	34.3	433
	55cm	20 cm	184	0.38	34.3	49.6	586
	85cm	30 cm	184	0.38	49.6	89.2	1455
							2832
T= 3 days	7 cm	7 cm	184	0.38	25.7	25.7	126
	17cm	9.9 cm	184	0.38	25.7	31.3	197
	35cm	18.3 cm	184	0.38	31.3	30.4	394
	55cm	20 cm	184	0.38	30.4	29.8	421
	85cm	30 cm	184	0.38	29.8	66.1	1005
							2143

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	28.9	28.9	145
	17cm	9.9 cm	184	0.39	28.9	35.1	227
	35cm	18.3 cm	184	0.39	35.1	31.2	435
	55cm	20 cm	184	0.39	31.2	31.9	452
	85cm	30 cm	184	0.39	31.9	68.7	1082
							2341
T= 5 min	7 cm	7 cm	184	0.39	80.2	80.2	403
	17cm	9.9 cm	184	0.39	80.2	79.5	567
	35cm	18.3 cm	184	0.39	79.5	31.2	726
	55cm	20 cm	184	0.39	31.2	31.9	452
	85cm	30 cm	184	0.39	31.9	68.7	1082
							3230
T=45 min	7 cm	7 cm	184	0.39	60.4	60.4	303
	17cm	9.9 cm	184	0.39	60.4	77.6	490
	35cm	18.3 cm	184	0.39	77.6	39.4	768
	55cm	20 cm	184	0.39	39.4	31.9	511
	85cm	30 cm	184	0.39	31.9	68.7	1082
							3154
T= 3 h	7 cm	7 cm	184	0.39	45.8	45.8	230
	17cm	9.9 cm	184	0.39	45.8	58.4	370
	35cm	18.3 cm	184	0.39	58.4	57.1	758
	55cm	20 cm	184	0.39	57.1	33.6	650
	85cm	30 cm	184	0.39	33.6	68.7	1100
							3108
T= 5h: 20	7 cm	7 cm	184	0.39	32.6	35.6	171
	17cm	9.9 cm	184	0.39	35.6	43.5	281
	35cm	18.3 cm	184	0.39	43.5	54.6	644
	55cm	20 cm	184	0.39	54.6	39.1	672
	85cm	30 cm	184	0.39	39.1	69.0	1163
							2931
T= 10h: 30	7 cm	7 cm	184	0.39	29.5	29.5	148
	17cm	9.9 cm	184	0.39	29.5	35.1	229
	35cm	18.3 cm	184	0.39	35.1	31.9	440
	55cm	20 cm	184	0.39	31.9	56.3	632
	85cm	30 cm	184	0.39	56.3	92.8	1604
							3053
T= 3 days	7 cm	7 cm	184	0.39	28.7	28.7	144
	17cm	9.9 cm	184	0.39	28.7	34.9	226
	35cm	18.3 cm	184	0.39	34.9	31.3	434
	55cm	20 cm	184	0.39	31.3	32.0	454
	85cm	30 cm	184	0.39	32.0	68.6	1082
							2340

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	27.4	27.4	134
	17cm	9.9 cm	184	0.39	27.4	34.1	213
	35cm	18.3 cm	184	0.39	34.1	30.8	415
	55cm	20 cm	184	0.39	30.8	30.7	430
	85cm	30 cm	184	0.39	30.7	65.4	1007
							2199
T= 5 min	7 cm	7 cm	184	0.39	80.2	79.4	390
	17cm	9.9 cm	184	0.39	79.4	79.1	548
	35cm	18.3 cm	184	0.39	79.1	30.8	703
	55cm	20 cm	184	0.39	30.8	30.7	430
	85cm	30 cm	184	0.39	30.7	65.4	1007
							3078
T=45 min	7 cm	7 cm	184	0.39	60.4	59.8	294
	17cm	9.9 cm	184	0.39	59.8	76.8	472
	35cm	18.3 cm	184	0.39	76.8	38.4	736
	55cm	20 cm	184	0.39	38.4	30.7	483
	85cm	30 cm	184	0.39	30.7	65.5	1008
							2994
T= 3 h	7 cm	7 cm	184	0.39	45.8	44.2	220
	17cm	9.9 cm	184	0.39	44.2	58.9	357
	35cm	18.3 cm	184	0.39	58.9	56.8	740
	55cm	20 cm	184	0.39	56.8	31.4	616
	85cm	30 cm	184	0.39	31.4	65.5	1015
							2948
T= 5h: 20	7 cm	7 cm	184	0.39	31.2	31.2	153
	17cm	9.9 cm	184	0.39	31.2	42.5	255
	35cm	18.3 cm	184	0.39	42.5	53.8	616
	55cm	20 cm	184	0.39	53.8	40.1	656
	85cm	30 cm	184	0.39	40.1	68.4	1137
							2816
T= 10h: 30	7 cm	7 cm	184	0.39	28.1	28.1	137
	17cm	9.9 cm	184	0.39	28.1	36.4	223
	35cm	18.3 cm	184	0.39	36.4	32.0	437
	55cm	20 cm	184	0.39	32.0	54.4	604
	85cm	30 cm	184	0.39	54.4	89.4	1507
							2908
T= 3 days	7 cm	7 cm	184	0.39	27.9	27.9	136
	17cm	9.9 cm	184	0.39	27.9	33.9	214
	35cm	18.3 cm	184	0.39	33.9	31.0	415
	55cm	20 cm	184	0.39	31.0	31.4	436
	85cm	30 cm	184	0.39	31.4	66.7	1028
							2229

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	27.9	27.9	136
	17cm	9.9 cm	184	0.39	27.9	34.9	217
	35cm	18.3 cm	184	0.39	34.9	32.1	428
	55cm	20 cm	184	0.39	32.1	32.1	449
	85cm	30 cm	184	0.39	32.1	67.9	1048
							2278
T= 5 min	7 cm	7 cm	184	0.39	79.8	79.8	390
	17cm	9.9 cm	184	0.39	79.8	78.2	546
	35cm	18.3 cm	184	0.39	78.2	32.1	705
	55cm	20 cm	184	0.39	32.1	32.1	449
	85cm	30 cm	184	0.39	32.1	67.9	1048
							3138
T=45 min	7 cm	7 cm	184	0.39	61.1	61.1	299
	17cm	9.9 cm	184	0.39	61.1	78.4	482
	35cm	18.3 cm	184	0.39	78.4	38.7	749
	55cm	20 cm	184	0.39	38.7	32.0	494
	85cm	30 cm	184	0.39	32.0	67.9	1047
							3071
T= 3 h	7 cm	7 cm	184	0.39	46.1	46.1	225
	17cm	9.9 cm	184	0.39	46.1	58.6	362
	35cm	18.3 cm	184	0.39	58.6	56.9	738
	55cm	20 cm	184	0.39	56.9	33.8	634
	85cm	30 cm	184	0.39	33.8	68.0	1067
							3026
T= 5h: 20	7 cm	7 cm	184	0.39	34.9	34.9	171
	17cm	9.9 cm	184	0.39	34.9	44.5	275
	35cm	18.3 cm	184	0.39	44.5	58.2	657
	55cm	20 cm	184	0.39	58.2	38.5	676
	85cm	30 cm	184	0.39	38.5	69.2	1129
							2908
T= 10h: 30	7 cm	7 cm	184	0.39	29.5	29.5	144
	17cm	9.9 cm	184	0.39	29.5	34.7	222
	35cm	18.3 cm	184	0.39	34.7	32.1	427
	55cm	20 cm	184	0.39	32.1	55.4	611
	85cm	30 cm	184	0.39	55.4	94.3	1569
							2973
T= 3 days	7 cm	7 cm	184	0.39	28.6	28.6	140
	17cm	9.9 cm	184	0.39	28.6	34.7	219
	35cm	18.3 cm	184	0.39	34.7	31.3	422
	55cm	20 cm	184	0.39	31.3	32.4	445
	85cm	30 cm	184	0.39	32.4	67.9	1051
							2277

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	25.6	25.6	125
	17cm	9.9 cm	184	0.38	25.6	33.6	205
	35cm	18.3 cm	184	0.38	33.6	30.2	408
	55cm	20 cm	184	0.38	30.2	30.2	422
	85cm	30 cm	184	0.38	30.2	67.1	1020
							2179
T= 5 min	7 cm	7 cm	184	0.38	75.5	75.5	369
	17cm	9.9 cm	184	0.38	75.5	79.6	536
	35cm	18.3 cm	184	0.38	79.6	30.2	702
	55cm	20 cm	184	0.38	30.2	30.2	422
	85cm	30 cm	184	0.38	30.2	67.1	1020
							3049
T=45 min	7 cm	7 cm	184	0.38	60.3	60.3	295
	17cm	9.9 cm	184	0.38	60.3	77.6	477
	35cm	18.3 cm	184	0.38	77.6	41.0	758
	55cm	20 cm	184	0.38	41.0	30.2	497
	85cm	30 cm	184	0.38	30.2	67.1	1020
							3047
T= 3 h	7 cm	7 cm	184	0.38	41.8	41.8	205
	17cm	9.9 cm	184	0.38	41.8	50.2	318
	35cm	18.3 cm	184	0.38	50.2	56.7	683
	55cm	20 cm	184	0.38	56.7	34.1	634
	85cm	30 cm	184	0.38	34.1	67.1	1060
							2901
T= 5h: 20	7 cm	7 cm	184	0.38	33.3	33.3	163
	17cm	9.9 cm	184	0.38	33.3	42.5	262
	35cm	18.3 cm	184	0.38	42.5	54.3	619
	55cm	20 cm	184	0.38	54.3	44.4	689
	85cm	30 cm	184	0.38	44.4	67.4	1172
							2904
T= 10h: 30	7 cm	7 cm	184	0.38	29.3	29.3	143
	17cm	9.9 cm	184	0.38	29.3	34.2	219
	35cm	18.3 cm	184	0.38	34.2	35.0	442
	55cm	20 cm	184	0.38	35.0	50.6	598
	85cm	30 cm	184	0.38	50.6	91.0	1484
							2886
T= 3 days	7 cm	7 cm	184	0.38	26.2	26.2	128
	17cm	9.9 cm	184	0.38	26.2	31.9	201
	35cm	18.3 cm	184	0.38	31.9	31.0	402
	55cm	20 cm	184	0.38	31.0	30.4	429
	85cm	30 cm	184	0.38	30.4	67.4	1025
							2186

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	23.9	23.9	117
	17cm	9.9 cm	184	0.38	23.9	32.4	195
	35cm	18.3 cm	184	0.38	32.4	29.8	398
	55cm	20 cm	184	0.38	29.8	29.4	414
	85cm	30 cm	184	0.38	29.4	67.4	1014
							2137
T= 5 min	7 cm	7 cm	184	0.38	77.0	76.8	376
	17cm	9.9 cm	184	0.38	76.8	82.1	550
	35cm	18.3 cm	184	0.38	82.1	29.8	715
	55cm	20 cm	184	0.38	29.8	29.4	414
	85cm	30 cm	184	0.38	29.4	67.4	1014
							3069
T=45 min	7 cm	7 cm	184	0.38	60.3	61.6	298
	17cm	9.9 cm	184	0.38	61.6	76.8	479
	35cm	18.3 cm	184	0.38	76.8	39.8	745
	55cm	20 cm	184	0.38	39.8	29.4	483
	85cm	30 cm	184	0.38	29.4	67.4	1014
							3020
T= 3 h	7 cm	7 cm	184	0.38	43.1	44.6	214
	17cm	9.9 cm	184	0.38	44.6	51.3	332
	35cm	18.3 cm	184	0.38	51.3	57.1	693
	55cm	20 cm	184	0.38	57.1	35.5	647
	85cm	30 cm	184	0.38	35.5	70.8	1114
							3000
T= 5h: 20	7 cm	7 cm	184	0.38	33.9	34.4	167
	17cm	9.9 cm	184	0.38	34.4	44.1	272
	35cm	18.3 cm	184	0.38	44.1	50.3	604
	55cm	20 cm	184	0.38	50.3	44.7	664
	85cm	30 cm	184	0.38	44.7	72.2	1225
							2931
T= 10h: 30	7 cm	7 cm	184	0.38	30.1	30.1	147
	17cm	9.9 cm	184	0.38	30.1	34.0	222
	35cm	18.3 cm	184	0.38	34.0	35.3	443
	55cm	20 cm	184	0.38	35.3	51.0	603
	85cm	30 cm	184	0.38	51.0	94.6	1526
							2941
T= 3 days	7 cm	7 cm	184	0.38	27.0	27.0	132
	17cm	9.9 cm	184	0.38	27.0	32.9	207
	35cm	18.3 cm	184	0.38	32.9	31.9	414
	55cm	20 cm	184	0.38	31.9	31.3	442
	85cm	30 cm	184	0.38	31.3	69.3	1054
							2249

Table C-33 Column C -Third Simulation Calculation.

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	25.9	25.9	127
	17cm	9.9 cm	184	0.38	25.9	31.4	198
	35cm	18.3 cm	184	0.38	31.4	28.4	382
	55cm	20 cm	184	0.38	28.4	28.6	398
	85cm	30 cm	184	0.38	28.6	62.5	955
							2060
T= 5 min	7 cm	7 cm	184	0.38	74.6	74.6	365
	17cm	9.9 cm	184	0.38	74.6	86.9	558
	35cm	18.3 cm	184	0.38	86.9	29.5	745
	55cm	20 cm	184	0.38	28.4	28.6	398
	85cm	30 cm	184	0.38	28.6	62.5	955
							3021
T=45 min	7 cm	7 cm	184	0.38	61.5	61.5	301
	17cm	9.9 cm	184	0.38	61.5	76.5	477
	35cm	18.3 cm	184	0.38	76.5	41.1	752
	55cm	20 cm	184	0.38	41.1	29.7	495
	85cm	30 cm	184	0.38	28.6	62.5	955
							2979
T= 3 h	7 cm	7 cm	184	0.38	40.9	40.9	200
	17cm	9.9 cm	184	0.38	40.9	48.1	308
	35cm	18.3 cm	184	0.38	48.1	52.9	646
	55cm	20 cm	184	0.38	52.9	33.1	601
	85cm	30 cm	184	0.38	33.1	63.1	1009
							2763
T= 5h: 20	7 cm	7 cm	184	0.38	33.9	33.9	166
	17cm	9.9 cm	184	0.38	33.9	43.2	266
	35cm	18.3 cm	184	0.38	43.2	51.7	606
	55cm	20 cm	184	0.38	51.7	42.4	657
	85cm	30 cm	184	0.38	42.4	69.1	1168
							2864
T= 10h: 30	7 cm	7 cm	184	0.38	29.7	29.7	145
	17cm	9.9 cm	184	0.38	29.7	34.8	223
	35cm	18.3 cm	184	0.38	34.8	35.0	446
	55cm	20 cm	184	0.38	35.0	51.1	601
	85cm	30 cm	184	0.38	51.1	91.0	1489
							2905
T= 3 days	7 cm	7 cm	184	0.38	27.0	27.0	132
	17cm	9.9 cm	184	0.38	27.0	32.2	205
	35cm	18.3 cm	184	0.38	32.2	31.6	408
	55cm	20 cm	184	0.38	31.6	30.7	435
	85cm	30 cm	184	0.38	30.7	68.1	1035
							2216

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	28.9	28.9	145
	17cm	9.9 cm	184	0.39	28.9	35.1	227
	35cm	18.3 cm	184	0.39	35.1	31.2	435
	55cm	20 cm	184	0.39	31.2	31.9	452
	85cm	30 cm	184	0.39	31.9	68.7	1082
							2342
T= 5 min	7 cm	7 cm	184	0.39	84.2	84.2	423
	17cm	9.9 cm	184	0.39	84.2	83.5	595
	35cm	18.3 cm	184	0.39	83.5	32.8	763
	55cm	20 cm	184	0.39	32.8	31.9	464
	85cm	30 cm	184	0.39	31.9	68.7	1082
							3326
T=45 min	7 cm	7 cm	184	0.39	61.6	61.6	309
	17cm	9.9 cm	184	0.39	61.6	79.2	500
	35cm	18.3 cm	184	0.39	79.2	40.2	783
	55cm	20 cm	184	0.39	40.2	32.5	521
	85cm	30 cm	184	0.39	32.5	68.7	1089
							3202
T= 3 h	7 cm	7 cm	184	0.39	46.9	46.9	236
	17cm	9.9 cm	184	0.39	46.9	59.9	379
	35cm	18.3 cm	184	0.39	59.9	58.5	777
	55cm	20 cm	184	0.39	58.5	34.4	667
	85cm	30 cm	184	0.39	34.4	70.4	1128
							3186
T= 5h: 20	7 cm	7 cm	184	0.39	33.2	36.3	174
	17cm	9.9 cm	184	0.39	36.3	44.3	286
	35cm	18.3 cm	184	0.39	44.3	55.6	656
	55cm	20 cm	184	0.39	55.6	39.8	685
	85cm	30 cm	184	0.39	39.8	70.3	1185
							2986
T= 10h: 30	7 cm	7 cm	184	0.39	30.7	30.7	154
	17cm	9.9 cm	184	0.39	30.7	36.5	238
	35cm	18.3 cm	184	0.39	36.5	33.2	457
	55cm	20 cm	184	0.39	33.2	58.6	658
	85cm	30 cm	184	0.39	58.6	96.5	1668
							3175
T= 3 days	7 cm	7 cm	184	0.39	29.6	29.6	148
	17cm	9.9 cm	184	0.39	29.6	35.9	233
	35cm	18.3 cm	184	0.39	35.9	32.2	447
	55cm	20 cm	184	0.39	32.2	33.0	467
	85cm	30 cm	184	0.39	33.0	70.7	1114
							2410

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	27.4	27.4	134
	17cm	9.9 cm	184	0.39	27.4	34.1	213
	35cm	18.3 cm	184	0.39	34.1	30.8	415
	55cm	20 cm	184	0.39	30.8	30.7	430
	85cm	30 cm	184	0.39	30.7	65.4	1007
							2198
T= 5 min	7 cm	7 cm	184	0.39	82.6	81.8	402
	17cm	9.9 cm	184	0.39	81.8	81.5	565
	35cm	18.3 cm	184	0.39	81.5	31.7	724
	55cm	20 cm	184	0.39	31.7	30.7	436
	85cm	30 cm	184	0.39	30.7	65.4	1007
							3133
T=45 min	7 cm	7 cm	184	0.39	61.6	61.0	300
	17cm	9.9 cm	184	0.39	61.0	78.3	482
	35cm	18.3 cm	184	0.39	78.3	40.3	759
	55cm	20 cm	184	0.39	40.3	31.3	500
	85cm	30 cm	184	0.39	31.3	65.5	1015
							3055
T= 3 h	7 cm	7 cm	184	0.39	48.1	46.4	231
	17cm	9.9 cm	184	0.39	46.4	61.8	374
	35cm	18.3 cm	184	0.39	61.8	59.6	777
	55cm	20 cm	184	0.39	59.6	33.0	647
	85cm	30 cm	184	0.39	33.0	68.8	1066
							3095
T= 5h: 20	7 cm	7 cm	184	0.39	33.1	33.1	162
	17cm	9.9 cm	184	0.39	33.1	45.1	270
	35cm	18.3 cm	184	0.39	45.1	57.0	653
	55cm	20 cm	184	0.39	57.0	42.5	695
	85cm	30 cm	184	0.39	42.5	72.5	1205
							2985
T= 10h: 30	7 cm	7 cm	184	0.39	29.2	29.2	143
	17cm	9.9 cm	184	0.39	29.2	37.9	232
	35cm	18.3 cm	184	0.39	37.9	33.3	455
	55cm	20 cm	184	0.39	33.3	56.6	628
	85cm	30 cm	184	0.39	56.6	93.0	1567
							3025
T= 3 days	7 cm	7 cm	184	0.39	28.2	28.2	138
	17cm	9.9 cm	184	0.39	28.2	34.2	216
	35cm	18.3 cm	184	0.39	34.2	31.3	419
	55cm	20 cm	184	0.39	31.3	31.7	440
	85cm	30 cm	184	0.39	31.7	67.4	1038
							2251

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	27.9	27.9	140
	17cm	9.9 cm	184	0.39	27.9	34.9	223
	35cm	18.3 cm	184	0.39	34.9	32.1	440
	55cm	20 cm	184	0.39	32.1	32.1	460
	85cm	30 cm	184	0.39	32.1	67.9	1076
							2338
T= 5 min	7 cm	7 cm	184	0.39	83.0	83.0	417
	17cm	9.9 cm	184	0.39	83.0	81.3	583
	35cm	18.3 cm	184	0.39	81.3	33.4	753
	55cm	20 cm	184	0.39	33.4	32.1	470
	85cm	30 cm	184	0.39	32.1	67.9	1076
							3297
T=45 min	7 cm	7 cm	184	0.39	64.8	64.8	325
	17cm	9.9 cm	184	0.39	64.8	83.1	525
	35cm	18.3 cm	184	0.39	83.1	41.0	814
	55cm	20 cm	184	0.39	41.0	33.9	537
	85cm	30 cm	184	0.39	33.9	67.9	1095
							3297
T= 3 h	7 cm	7 cm	184	0.39	47.0	47.0	236
	17cm	9.9 cm	184	0.39	47.0	59.8	379
	35cm	18.3 cm	184	0.39	59.8	58.0	773
	55cm	20 cm	184	0.39	58.0	34.5	663
	85cm	30 cm	184	0.39	34.5	69.4	1117
							3168
T= 5h: 20	7 cm	7 cm	184	0.39	37.3	37.3	187
	17cm	9.9 cm	184	0.39	37.3	47.6	302
	35cm	18.3 cm	184	0.39	47.6	62.3	721
	55cm	20 cm	184	0.39	62.3	41.2	742
	85cm	30 cm	184	0.39	41.2	70.6	1202
							3154
T= 10h: 30	7 cm	7 cm	184	0.39	32.2	32.2	161
	17cm	9.9 cm	184	0.39	32.2	37.8	248
	35cm	18.3 cm	184	0.39	37.8	35.0	478
	55cm	20 cm	184	0.39	35.0	60.4	684
	85cm	30 cm	184	0.39	60.4	102.8	1755
							3326
T= 3 days	7 cm	7 cm	184	0.39	29.2	29.2	146
	17cm	9.9 cm	184	0.39	29.2	35.4	229
	35cm	18.3 cm	184	0.39	35.4	31.9	442
	55cm	20 cm	184	0.39	31.9	33.0	466
	85cm	30 cm	184	0.39	33.0	69.3	1100
							2383

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	26.1	26.1	128
	17cm	9.9 cm	184	0.38	26.1	34.2	209
	35cm	18.3 cm	184	0.38	34.2	30.8	416
	55cm	20 cm	184	0.38	30.8	30.8	430
	85cm	30 cm	184	0.38	30.8	68.5	1040
							2223
T= 5 min	7 cm	7 cm	184	0.38	77.0	77.0	377
	17cm	9.9 cm	184	0.38	77.0	81.2	547
	35cm	18.3 cm	184	0.38	81.2	30.8	716
	55cm	20 cm	184	0.38	30.8	30.8	430
	85cm	30 cm	184	0.38	30.8	68.5	1040
							3110
T=45 min	7 cm	7 cm	184	0.38	61.5	61.5	301
	17cm	9.9 cm	184	0.38	61.5	79.2	486
	35cm	18.3 cm	184	0.38	79.2	41.8	773
	55cm	20 cm	184	0.38	41.8	30.8	507
	85cm	30 cm	184	0.38	30.8	68.5	1040
							3108
T= 3 h	7 cm	7 cm	184	0.38	42.7	42.7	209
	17cm	9.9 cm	184	0.38	42.7	51.2	325
	35cm	18.3 cm	184	0.38	51.2	57.8	697
	55cm	20 cm	184	0.38	57.8	34.7	647
	85cm	30 cm	184	0.38	34.7	68.5	1082
							2959
T= 5h: 20	7 cm	7 cm	184	0.38	33.9	33.9	166
	17cm	9.9 cm	184	0.38	33.9	43.4	267
	35cm	18.3 cm	184	0.38	43.4	55.3	631
	55cm	20 cm	184	0.38	55.3	45.3	703
	85cm	30 cm	184	0.38	45.3	68.8	1195
							2962
T= 10h: 30	7 cm	7 cm	184	0.38	29.9	29.9	146
	17cm	9.9 cm	184	0.38	29.9	34.9	224
	35cm	18.3 cm	184	0.38	34.9	35.7	451
	55cm	20 cm	184	0.38	35.7	51.6	610
	85cm	30 cm	184	0.38	51.6	92.8	1513
							2944
T= 3 days	7 cm	7 cm	184	0.38	26.7	26.7	131
	17cm	9.9 cm	184	0.38	26.7	32.6	205
	35cm	18.3 cm	184	0.38	32.6	31.6	410
	55cm	20 cm	184	0.38	31.6	31.0	438
	85cm	30 cm	184	0.38	31.0	68.8	1046
							2229

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	23.4	23.4	114
	17cm	9.9 cm	184	0.38	23.4	33.1	195
	35cm	18.3 cm	184	0.38	33.1	29.4	400
	55cm	20 cm	184	0.38	29.4	28.9	407
	85cm	30 cm	184	0.38	28.9	66.4	999
							2115
T= 5 min	7 cm	7 cm	184	0.38	80.0	79.8	391
	17cm	9.9 cm	184	0.38	76.8	86.2	564
	35cm	18.3 cm	184	0.38	82.1	29.8	715
	55cm	20 cm	184	0.38	29.8	29.4	414
	85cm	30 cm	184	0.38	29.4	67.4	1014
							3098
T=45 min	7 cm	7 cm	184	0.38	61.5	62.8	304
	17cm	9.9 cm	184	0.38	62.8	78.3	488
	35cm	18.3 cm	184	0.38	78.3	39.8	755
	55cm	20 cm	184	0.38	39.8	29.4	483
	85cm	30 cm	184	0.38	29.4	67.4	1014
							3045
T= 3 h	7 cm	7 cm	184	0.38	45.2	46.9	225
	17cm	9.9 cm	184	0.38	46.9	53.9	348
	35cm	18.3 cm	184	0.38	53.9	60.0	728
	55cm	20 cm	184	0.38	60.0	37.3	679
	85cm	30 cm	184	0.38	37.3	74.3	1169
							3150
T= 5h: 20	7 cm	7 cm	184	0.38	35.3	35.8	174
	17cm	9.9 cm	184	0.38	35.8	45.9	282
	35cm	18.3 cm	184	0.38	45.9	52.3	628
	55cm	20 cm	184	0.38	52.3	46.5	691
	85cm	30 cm	184	0.38	46.5	75.1	1274
							3049
T= 10h: 30	7 cm	7 cm	184	0.38	31.6	31.6	155
	17cm	9.9 cm	184	0.38	31.6	35.7	233
	35cm	18.3 cm	184	0.38	35.7	37.0	465
	55cm	20 cm	184	0.38	37.0	53.6	633
	85cm	30 cm	184	0.38	53.6	99.3	1603
							3089
T= 3 days	7 cm	7 cm	184	0.38	27.8	27.8	136
	17cm	9.9 cm	184	0.38	27.8	34.5	215
	35cm	18.3 cm	184	0.38	34.5	33.5	435
	55cm	20 cm	184	0.38	33.5	32.9	464
	85cm	30 cm	184	0.38	32.9	72.8	1107
							2357

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.38	26.8	26.8	131
	17cm	9.9 cm	184	0.38	26.8	31.0	200
	35cm	18.3 cm	184	0.38	31.0	29.4	386
	55cm	20 cm	184	0.38	29.4	28.6	405
	85cm	30 cm	184	0.38	28.6	63.8	968
							2091
T= 5 min	7 cm	7 cm	184	0.38	76.8	76.8	376
	17cm	9.9 cm	184	0.38	76.8	86.9	566
	35cm	18.3 cm	184	0.38	90.4	30.7	774
	55cm	20 cm	184	0.38	28.4	28.6	398
	85cm	30 cm	184	0.38	28.6	62.5	955
							3069
T=45 min	7 cm	7 cm	184	0.38	63.9	63.9	313
	17cm	9.9 cm	184	0.38	63.9	78.8	494
	35cm	18.3 cm	184	0.38	78.8	42.7	777
	55cm	20 cm	184	0.38	42.7	30.9	515
	85cm	30 cm	184	0.38	28.6	62.5	955
							3053
T= 3 h	7 cm	7 cm	184	0.38	41.3	41.3	202
	17cm	9.9 cm	184	0.38	41.3	48.6	311
	35cm	18.3 cm	184	0.38	48.6	53.5	652
	55cm	20 cm	184	0.38	53.5	33.5	607
	85cm	30 cm	184	0.38	33.5	63.8	1019
							2791
T= 5h: 20	7 cm	7 cm	184	0.38	35.3	33.9	169
	17cm	9.9 cm	184	0.38	33.9	44.5	271
	35cm	18.3 cm	184	0.38	44.5	53.8	628
	55cm	20 cm	184	0.38	53.8	42.4	672
	85cm	30 cm	184	0.38	44.5	71.8	1219
							2959
T= 10h: 30	7 cm	7 cm	184	0.38	30.3	30.3	148
	17cm	9.9 cm	184	0.38	30.3	34.8	225
	35cm	18.3 cm	184	0.38	36.2	35.7	460
	55cm	20 cm	184	0.38	35.7	52.6	617
	85cm	30 cm	184	0.38	52.6	92.8	1524
							2974
T= 3 days	7 cm	7 cm	184	0.38	28.3	27.0	135
	17cm	9.9 cm	184	0.38	27.0	33.2	208
	35cm	18.3 cm	184	0.38	33.2	32.9	422
	55cm	20 cm	184	0.38	32.9	31.6	451
	85cm	30 cm	184	0.38	31.6	70.1	1066
							2283

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	28.4	28.4	143
	17cm	9.9 cm	184	0.39	28.4	34.2	222
	35cm	18.3 cm	184	0.39	34.2	31.2	429
	55cm	20 cm	184	0.39	31.2	32.5	457
	85cm	30 cm	184	0.39	32.5	66.5	1065
							2315
T= 5 min	7 cm	7 cm	184	0.39	83.4	83.4	419
	17cm	9.9 cm	184	0.39	83.4	81.9	587
	35cm	18.3 cm	184	0.39	81.9	31.2	742
	55cm	20 cm	184	0.39	31.2	31.9	452
	85cm	30 cm	184	0.39	32.5	66.5	1065
							3265
T=45 min	7 cm	7 cm	184	0.39	62.2	62.2	312
	17cm	9.9 cm	184	0.39	61.2	80.7	504
	35cm	18.3 cm	184	0.39	80.7	40.6	796
	55cm	20 cm	184	0.39	40.6	32.4	523
	85cm	30 cm	184	0.39	32.4	66.5	1064
							3199
T= 3 h	7 cm	7 cm	184	0.39	47.6	47.6	239
	17cm	9.9 cm	184	0.39	47.6	60.1	382
	35cm	18.3 cm	184	0.39	60.1	58.5	778
	55cm	20 cm	184	0.39	58.5	34.1	664
	85cm	30 cm	184	0.39	34.1	71.0	1130
							3194
T= 5h: 20	7 cm	7 cm	184	0.39	33.2	36.3	174
	17cm	9.9 cm	184	0.39	36.3	44.3	286
	35cm	18.3 cm	184	0.39	44.3	55.2	653
	55cm	20 cm	184	0.39	55.2	40.3	685
	85cm	30 cm	184	0.39	40.3	71.1	1198
							2996
T= 10h: 30	7 cm	7 cm	184	0.39	31.5	31.5	158
	17cm	9.9 cm	184	0.39	31.5	36.5	241
	35cm	18.3 cm	184	0.39	36.5	33.2	457
	55cm	20 cm	184	0.39	33.2	58.6	658
	85cm	30 cm	184	0.39	58.6	96.5	1668
							3182
T= 3 days	7 cm	7 cm	184	0.39	29.6	29.6	148
	17cm	9.9 cm	184	0.39	29.6	34.2	226
	35cm	18.3 cm	184	0.39	34.2	32.2	436
	55cm	20 cm	184	0.39	32.2	34.1	476
	85cm	30 cm	184	0.39	34.1	67.2	1090
							2376

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	27.9	27.9	140
	17cm	9.9 cm	184	0.39	27.9	34.9	223
	35cm	18.3 cm	184	0.39	34.9	32.1	440
	55cm	20 cm	184	0.39	33.2	33.2	476
	85cm	30 cm	184	0.39	33.2	68.1	1090
							2368
T= 5 min	7 cm	7 cm	184	0.39	86.3	86.3	433
	17cm	9.9 cm	184	0.39	86.3	84.6	607
	35cm	18.3 cm	184	0.39	84.6	34.7	783
	55cm	20 cm	184	0.39	33.2	33.2	476
	85cm	30 cm	184	0.39	33.2	68.1	1090
							3388
T=45 min	7 cm	7 cm	184	0.39	68.7	68.7	345
	17cm	9.9 cm	184	0.39	68.7	88.1	556
	35cm	18.3 cm	184	0.39	88.1	43.5	863
	55cm	20 cm	184	0.39	43.5	36.0	570
	85cm	30 cm	184	0.39	33.2	68.4	1093
							3426
T= 3 h	7 cm	7 cm	184	0.39	48.0	48.0	241
	17cm	9.9 cm	184	0.39	48.0	61.0	387
	35cm	18.3 cm	184	0.39	61.0	59.2	788
	55cm	20 cm	184	0.39	59.2	35.2	677
	85cm	30 cm	184	0.39	35.2	70.7	1139
							3232
T= 5h: 20	7 cm	7 cm	184	0.39	40.0	40.0	201
	17cm	9.9 cm	184	0.39	40.0	50.9	323
	35cm	18.3 cm	184	0.39	50.9	66.6	771
	55cm	20 cm	184	0.39	66.6	44.1	794
	85cm	30 cm	184	0.39	44.1	72.0	1248
							3337
T= 10h: 30	7 cm	7 cm	184	0.39	35.0	35.0	176
	17cm	9.9 cm	184	0.39	35.0	41.2	271
	35cm	18.3 cm	184	0.39	41.2	38.1	521
	55cm	20 cm	184	0.39	38.1	65.8	745
	85cm	30 cm	184	0.39	65.8	112.0	1913
							3626
T= 3 days	7 cm	7 cm	184	0.39	29.8	29.8	149
	17cm	9.9 cm	184	0.39	29.8	36.1	234
	35cm	18.3 cm	184	0.39	36.1	32.6	451
	55cm	20 cm	184	0.39	32.6	33.7	475
	85cm	30 cm	184	0.39	33.7	70.6	1122
							2431

	Depth	Difference	πr^2	θ	TDR UP	TDR Down	Volume (mL)
T = 0	7 cm	7 cm	184	0.39	26.3	26.3	129
	17cm	9.9 cm	184	0.39	26.3	34.1	209
	35cm	18.3 cm	184	0.39	34.1	30.8	415
	55cm	20 cm	184	0.39	35.8	35.8	500
	85cm	30 cm	184	0.39	35.8	66.3	1070
							2323
T= 5 min	7 cm	7 cm	184	0.39	85.1	84.2	414
	17cm	9.9 cm	184	0.39	84.2	83.9	582
	35cm	18.3 cm	184	0.39	83.9	32.7	745
	55cm	20 cm	184	0.39	35.8	35.8	500
	85cm	30 cm	184	0.39	35.8	66.3	1070
							3311
T=45 min	7 cm	7 cm	184	0.39	62.8	62.2	306
	17cm	9.9 cm	184	0.39	62.2	79.9	491
	35cm	18.3 cm	184	0.39	79.9	40.3	769
	55cm	20 cm	184	0.39	42.3	35.8	546
	85cm	30 cm	184	0.39	35.8	66.3	1070
							3182
T= 3 h	7 cm	7 cm	184	0.39	50.5	48.7	243
	17cm	9.9 cm	184	0.39	48.7	64.9	393
	35cm	18.3 cm	184	0.39	64.9	62.6	815
	55cm	20 cm	184	0.39	62.6	36.0	689
	85cm	30 cm	184	0.39	36.0	72.2	1134
							3274
T= 5h: 20	7 cm	7 cm	184	0.39	35.1	35.1	171
	17cm	9.9 cm	184	0.39	35.1	47.8	286
	35cm	18.3 cm	184	0.39	47.8	60.4	692
	55cm	20 cm	184	0.39	60.4	45.1	737
	85cm	30 cm	184	0.39	45.1	76.9	1278
							3164
T= 10h: 30	7 cm	7 cm	184	0.39	30.4	30.4	149
	17cm	9.9 cm	184	0.39	30.4	39.4	241
	35cm	18.3 cm	184	0.39	39.4	34.6	473
	55cm	20 cm	184	0.39	34.6	58.8	653
	85cm	30 cm	184	0.39	58.8	96.7	1630
							3146
T= 3 days	7 cm	7 cm	184	0.39	28.5	28.5	139
	17cm	9.9 cm	184	0.39	28.5	34.6	218
	35cm	18.3 cm	184	0.39	34.6	31.6	423
	55cm	20 cm	184	0.39	31.6	32.0	445
	85cm	30 cm	184	0.39	32.0	68.0	1049
							2274

Table C-43 Input and output volume of SWE from Column A.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				803
4	840	133		
5				
6				675
7	840		93	
8				
9				700
10	840	124		
11				
12				688
13	840		89	
14				
15				693
16	840	128		
17				
18				683
19	840		87	
20				
21 (End)				706

Table C-44 SWE mass balance and percent recovery for Column A.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1st	840	803	37	95.6
2nd	840	808	32	96.2
3rd	840	793	47	94.4
4th	840	812	28	96.7
5th	840	782	58	93.1
6th	840	811	29	96.5
7th	840	793	47	94.4
Σ	5880	5602	278	

Table C-45 Input and output volume of SWE from Column B.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				810
4	840	121		
5				
6				675
7	840		84	
8				
9				718
10	840	119		
11				
12				669
13	840		82	
14				
15				718
16	840	108		
17				
18				687
19	840		75	
20				
21 (End)				700

Table C-46 SWE mass balance and percent recovery for Column B.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1st	840	810	30	96.4
2nd	840	796	44	94.8
3rd	840	802	38	95.5
4th	840	788	52	93.8
5th	840	800	40	95.2
6th	840	795	45	94.6
7th	840	775	65	92.3
Σ	5880	5566	314	

Table C-47 Input and output volume of SWE from Column C.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				756
4	840	142		
5				
6				658
7	840		98	
8				
9				689
10	840	123		
11				
12				679
13	840		85	
14				
15				713
16	840	117		
17				
18				697
19	840		80	
20				
21 (End)				708

Table C-48 SWE mass balance and percent recovery for Column C.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1 st	840	756	84	90.0
2 nd	840	800	40	95.2
3 rd	840	787	53	93.7
4 th	840	802	38	95.5
5 th	840	798	42	95.0
6 th	840	814	26	96.9
7 th	840	788	52	93.8
Σ	5880	5545	335	

Table C-49 Input and output volume of SWE from Column D.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				806
4	840	113		
5				
6				670
7	840		78	
8				
9				708
10	840	102		
11				
12				684
13	840		74	
14				
15				708
16	840	107		
17				
18				667
19	840		71	
20				
21 (End)				723

Table C-50 SWE mass balance and percent recovery for Column D.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1 st	840	806	34	96.0
2 nd	840	783	57	93.2
3 rd	840	786	54	93.6
4 th	840	786	54	93.6
5 th	840	782	58	93.1
6 th	840	774	66	92.1
7 th	840	794	46	94.5
Σ	5880	5511	369	

Table C-51 Input and output volume of SWE from Column E.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				791
4	840	124		
5				
6				662
7	840		86	
8				
9				714
10	840	97		
11				
12				669
13	840		80	
14				
15				709
16	840	115		
17				
18				651
19	840		68	
20				
21 (End)				726

Table C-52 SWE mass balance and percent recovery for Column E.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1 st	840	791	49	94.2
2 nd	840	786	54	93.6
3 rd	840	800	40	95.2
4 th	840	766	74	91.2
5 th	840	789	51	93.9
6 th	840	766	74	91.2
7 th	840	794	46	94.5
Σ	5880	5492	388	

Table C-53 Input and output volume of SWE from Column F.

Time (Of day)	SWE Application Input (mL)	Sampling from Output (mL)		
		Port 1	Port 2	Port 3
1 (Start)	840			
2				
3				749
4	840	118		
5				
6				664
7	840		82	
8				
9				716
10	840	107		
11				
12				672
13	840		75	
14				
15				727
16	840	95		
17				
18				667
19	840		68	
20				
21 (End)				722

Table C-54 SWE mass balance and percent recovery for Column F.

Number of Application	SWE Input (mL)	Σ Output (mL)		% Recovery
		Output	Difference	
1 st	840	749	91	89.1
2 nd	840	782	58	93.1
3 rd	840	798	42	95.0
4 th	840	779	61	92.7
5 th	840	802	38	95.5
6 th	840	762	78	90.7
7 th	840	790	50	94.0
Σ	5880	5462	418	

Table C-56 Col. A- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.000	7	0.011	3	0.061
10	0.000	13	0.000	6	0.047
16	0.000	19	0.000	9	0.007
				12	0.003
				15	0.000
				18	0.000
				21	0.000

Table C-57 Col. B- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.282	7	0.387	3	0.068
10	0.094	13	0.097	6	0.064
16	0.030	19	0.020	9	0.395
				12	0.140
				15	0.076
				18	0.067
				21	0.028

Table C-58 Col. C- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	6.125	7	7.362	3	0.061
10	2.188	13	1.478	6	0.492
16	0.678	19	0.654	9	8.233
				12	2.449
				15	1.231
				18	1.152
				21	0.484

Table C-59 Column D- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.007	7	0.012	3	0.309
10	0	13	0.001	6	0.141
16	0	19	0	9	0.013
				12	0.005
				15	0.001
				18	0
				21	0

Table C-60 Col. E- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.252	7	0.382	3	0.308
10	0.094	13	0.085	6	0.150
16	0.031	19	0.021	9	0.353
				12	0.138
				15	0.079
				18	0.072
				21	0.030

Table C-61 Col. F- Cl avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	6.948	7	7.641	3	0.310
10	2.577	13	1.803	6	0.326
16	0.559	19	0.855	9	8.604
				12	2.334
				15	0.987
				18	1.123
				21	0.472

Table C-62 Col. B- Fluoride avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.034	7	0.039	3	0.000
10	0.012	13	0.010	6	0.000
16	0.003	19	0.002	9	0.036
				12	0.017
				15	0.008
				18	0.005
				21	0.002

Table C-63 Col. C- Fluoride avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.595	7	0.639	3	0.000
10	0.201	13	0.171	6	0.000
16	0.050	19	0.036	9	0.832
				12	0.302
				15	0.156
				18	0.112
				21	0.047

Table C-64 Col. E- Fluoride avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.035	7	0.033	3	0.000
10	0.014	13	0.011	6	0.000
16	0.004	19	0.003	9	0.032
				12	0.014
				15	0.007
				18	0.006
				21	0.003

Table C-65 Col. F- Fluoride avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.638	7	0.707	3	0.000
10	0.230	13	0.173	6	0.000
16	0.066	19	0.021	9	0.902
				12	0.340
				15	0.150
				18	0.131
				21	0.055

Table C-66 Col. B- Nitrite-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.009	7	0.010	3	0.000
10	0.003	13	0.003	6	0.000
16	0.001	19	0.001	9	0.008
				12	0.006
				15	0.003
				18	0.002
				21	0.001

Table C-67 Col. C- Nitrite-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.141	7	0.157	3	0.000
10	0.059	13	0.053	6	0.000
16	0.010	19	0.017	9	0.035
				12	0.168
				15	0.050
				18	0.043
				21	0.018

Table C-68 Col. E- Nitrite-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.008	7	0.009	3	0.000
10	0.004	13	0.003	6	0.000
16	0.001	19	0.001	9	0.006
				12	0.004
				15	0.003
				18	0.002
				21	0.001

Table C-69 Col. F- Nitrite-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.168	7	0.150	3	0.000
10	0.085	13	0.064	6	0.000
16	0.022	19	0.030	9	0.025
				12	0.184
				15	0.065
				18	0.038
				21	0.016

Table C-70 Col. B- Bromide avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.022	7	0.025	3	0.000
10	0.008	13	0.007	6	0.000
16	0.001	19	0.002	9	0.037
				12	0.010
				15	0.005
				18	0.005
				21	0.002

Table C-71 Col. C -Bromide avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.390	7	0.464	3	0.000
10	0.192	13	0.142	6	0.000
16	0.057	19	0.067	9	0.676
				12	0.227
				15	0.111
				18	0.127
				21	0.053

Table C-72 Col. E- Bromide avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.021	7	0.023	3	0.000
10	0.009	13	0.007	6	0.000
16	0.003	19	0.002	9	0.034
				12	0.010
				15	0.005
				18	0.005
				21	0.002

Table C-73 Col. F- Bromide avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.397	7	0.441	3	0.000
10	0.239	13	0.136	6	0.000
16	0.061	19	0.083	9	0.644
				12	0.193
				15	0.095
				18	0.108
				21	0.045

Table C-74 Col. B- Nitrate-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.025	7	0.030	3	0.000
10	0.010	13	0.008	6	0.000
16	0.009	19	0.006	9	0.031
				12	0.013
				15	0.007
				18	0.006
				21	0.003

Table C-75 Column C- Nitrate-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.313	7	0.347	3	0.000
10	0.225	13	0.194	6	0.000
16	0.061	19	0.071	9	0.460
				12	0.300
				15	0.090
				18	0.033
				21	0.014

Table C-76 Col. E- Nitrate-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.019	7	0.025	3	0.000
10	0.010	13	0.008	6	0.000
16	0.008	19	0.006	9	0.018
				12	0.012
				15	0.006
				18	0.005
				21	0.002

Table C-77 Col. F- Nitrate-N avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.510	7	0.349	3	0.000
10	0.344	13	0.223	6	0.000
16	0.153	19	0.087	9	0.332
				12	0.254
				15	0.101
				18	0.051
				21	0.021

Table C-78 Col. B- Phosphate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.120	7	0.146	3	0.000
10	0.113	13	0.086	6	0.000
16	0.081	19	0.067	9	0.032
				12	0.067
				15	0.046
				18	0.046
				21	0.019

Table C-79 Col. C- Phosphate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	2.682	7	2.328	3	0.000
10	2.172	13	1.720	6	0.000
16	1.178	19	1.443	9	3.545
				12	3.205
				15	1.656
				18	1.733
				21	0.728

Table C-80 Col. D- Phosphate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.000	7	0.067	3	3.125
10	0.000	13	0.000	6	1.982
16	0.000	19	0.000	9	0.417
				12	0.007
				15	0.000
				18	0.000
				21	0.000

Table C-81 Col. E- Phosphate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.147	7	0.183	3	3.095
10	0.109	13	0.076	6	2.924
16	0.084	19	0.054	9	0.026
				12	0.047
				15	0.037
				18	0.034
				21	0.014

Table C-82 Col. F- Phosphate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	3.406	7	3.674	3	3.210
10	2.889	13	2.014	6	2.951
16	1.289	19	1.539	9	2.119
				12	2.833
				15	1.811
				18	1.750
				21	0.735

Table C-83 Col. B- Sulfate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.154	7	0.120	3	0.000
10	0.043	13	0.033	6	0.000
16	0.022	19	0.033	9	0.143
				12	0.110
				15	0.046
				18	0.035
				21	0.015

Table C-84 Col.C - Sulfate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	2.080	7	1.500	3	0.000
10	1.174	13	0.803	6	0.000
16	0.724	19	0.438	9	2.813
				12	2.100
				15	0.729
				18	0.804
				21	0.338

Table C-85 Col. D- Sulfate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.079	7	0.194	3	1.351
10	0.000	13	0.000	6	0.526
16	0.000	19	0.000	9	0.185
				12	0.049
				15	0.000
				18	0.000
				21	0.000

Table C-86 Col. E- Sulfate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	0.253	7	0.351	3	1.119
10	0.060	13	0.055	6	0.982
16	0.033	19	0.029	9	0.179
				12	0.101
				15	0.057
				18	0.061
				21	0.026

Table C-87 Col. F- Sulfate avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	3.521	7	3.617	3	1.273
10	1.525	13	1.193	6	1.079
16	0.833	19	0.916	9	3.314
				12	2.195
				15	1.052
				18	1.323
				21	0.556

Table C-88 Col. B- Ammonia avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	48	7	36	3	0
10	5	13	6	6	0
16	3	19	4	9	18
				12	2
				15	3
				18	4
				21	1

Table C-89 Col. C- Ammonia avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	126	7	120	3	1
10	102	13	76	6	13
16	33	19	17	9	105
				12	64
				15	17
				18	37
				21	16

Table C-90 Col. E- Ammonia avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	51	7	29	3	0
10	17	13	8	6	0
16	6	19	3	9	12
				12	4
				15	2
				18	2
				21	1

Table C-91 Col. F- Ammonia avg. conc. (mg/L)

Day	Port 1	Day	Port 2	Day	Port3
4	185	7	177	3	0
10	173	13	111	6	0
16	132	19	25	9	95
				12	58
				15	45
				18	49
				21	21

Table C-92 Col. A- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	29	7	36	3	21
10	35	13	45	6	26
16	36	19	39	9	27
				12	21
				15	33
				18	29
				21	39

Table C-93 Col. B- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	63	7	29	3	27
10	39	13	36	6	36
16	48	19	27	9	36
				12	36
				15	32
				18	35
				21	41

Table C-94 Col. C- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	680	7	416	3	50
10	386	13	321	6	108
16	206	19	122	9	456
				12	351
				15	235
				18	257
				21	108

Table C-95 Col. D- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	72	7	188	3	326
10	21	13	168	6	69
16	27	19	36	9	80
				12	191
				15	359
				18	69
				21	27

Table C-96 Col. E- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	282	7	293	3	215
10	69	13	201	6	115
16	63	19	87	9	128
				12	285
				15	420
				18	120
				21	72

Table C-97 Col. F- avg. COD

Day	Port 1	Day	Port 2	Day	Port3
4	876	7	562	3	64
10	446	13	372	6	130
16	240	19	140	9	529
				12	410
				15	273
				18	306
				21	129

Mass Balance Calculation

Table C-98 Cumulative concentration for Chloride - Column A.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.803	0.061	0.049	0.049
2 nd	Port 1	0.133	0.000	0.000	
	Port 3	0.675	0.047	0.032	0.081
3 rd	Port 2	0.093	0.011	0.001	
	Port 3	0.700	0.007	0.005	0.087
4 th	Port 1	0.124	0.000	0.000	
	Port 3	0.688	0.003	0.002	0.089
5 th	Port 2	0.089	0.000	0.000	
	Port 3	0.693	0.000	0.000	0.089
6 th	Port 1	0.128	0.000	0.000	
	Port 3	0.683	0.000	0.000	0.089
7 th	Port 2	0.087	0.000	0.000	
	Port 3	0.706	0.000	0.000	0.089

Table C-99 Cumulative concentration for Chloride - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.068	0.055	0.055
2 nd	Port 1	0.121	0.282	0.034	
	Port 3	0.675	0.064	0.043	0.132
3 rd	Port 2	0.084	0.387	0.033	
	Port 3	0.718	0.395	0.283	0.448
4 th	Port 1	0.119	0.095	0.011	
	Port 3	0.669	0.140	0.094	0.553
5 th	Port 2	0.082	0.097	0.008	
	Port 3	0.718	0.076	0.055	0.616
6 th	Port 1	0.108	0.030	0.003	
	Port 3	0.687	0.067	0.046	0.665
7 th	Port 2	0.075	0.020	0.002	
	Port 3	0.700	0.028	0.020	0.686

Table C-100 Cumulative concentration for Chloride - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.061	0.046	0.046
2 nd	Port 1	0.142	6.125	0.870	
	Port 3	0.658	0.492	0.324	1.240
3 rd	Port 2	0.098	7.362	0.721	
	Port 3	0.689	8.233	5.673	7.634
4 th	Port 1	0.123	2.188	0.269	
	Port 3	0.679	2.449	1.663	9.566
5 th	Port 2	0.085	1.478	0.126	
	Port 3	0.713	1.231	0.878	10.569
6 th	Port 1	0.117	0.678	0.079	
	Port 3	0.697	1.152	0.803	11.451
7 th	Port 2	0.080	0.654	0.052	
	Port 3	0.708	0.484	0.343	11.846

Table C-101 Cumulative concentration for Chloride - Column D.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.806	0.309	0.249	0.249
2 nd	Port 1	0.113	0.007	0.001	
	Port 3	0.670	0.141	0.094	0.344
3 rd	Port 2	0.078	0.012	0.001	
	Port 3	0.708	0.013	0.009	0.354
4 th	Port 1	0.102	0.000	0.000	
	Port 3	0.684	0.005	0.003	0.358
5 th	Port 2	0.074	0.001	0.000	
	Port 3	0.708	0.001	0.001	0.359
6 th	Port 1	0.107	0.000	0.000	
	Port 3	0.667	0.000	0.000	0.359
7 th	Port 2	0.071	0.000	0.000	
	Port 3	0.723	0.000	0.000	0.359

Table C-102 Cumulative concentration for Chloride - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0.309	0.244	0.244
2 nd	Port 1	0.124	0.252	0.031	
	Port 3	0.662	0.150	0.099	0.375
3 rd	Port 2	0.086	0.382	0.033	
	Port 3	0.714	0.353	0.252	0.660
4 th	Port 1	0.097	0.095	0.009	
	Port 3	0.669	0.138	0.092	0.761
5 th	Port 2	0.080	0.086	0.007	
	Port 3	0.709	0.079	0.056	0.824
6 th	Port 1	0.115	0.031	0.004	
	Port 3	0.651	0.072	0.047	0.875
7 th	Port 2	0.068	0.021	0.001	
	Port 3	0.726	0.030	0.022	0.898

Table C-103 Cumulative concentration for Chloride - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0.309	0.231	0.231
2 nd	Port 1	0.118	6.948	0.820	
	Port 3	0.664	0.326	0.216	1.268
3 rd	Port 2	0.082	7.641	0.627	
	Port 3	0.716	8.604	6.160	8.055
4 th	Port 1	0.107	2.577	0.276	
	Port 3	0.672	2.334	1.568	9.899
5 th	Port 2	0.075	1.803	0.135	
	Port 3	0.727	0.987	0.717	10.752
6 th	Port 1	0.095	0.559	0.053	
	Port 3	0.667	1.123	0.749	11.554
7 th	Port 2	0.068	0.855	0.058	
	Port 3	0.722	0.472	0.341	11.952

Table C-104 Cumulative concentration for Fluoride - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.034	0.004	
	Port 3	0.675	0.000	0.000	0.004
3 rd	Port 2	0.084	0.039	0.003	
	Port 3	0.718	0.036	0.026	0.033
4 th	Port 1	0.119	0.012	0.001	
	Port 3	0.669	0.017	0.011	0.046
5 th	Port 2	0.082	0.010	0.001	
	Port 3	0.718	0.008	0.006	0.052
6 th	Port 1	0.108	0.003	0.000	
	Port 3	0.687	0.005	0.003	0.056
7 th	Port 2	0.075	0.002	0.000	
	Port 3	0.700	0.002	0.001	0.058

Table C-105 Cumulative concentration for Fluoride - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	0.595	0.085	
	Port 3	0.658	0.000	0.000	0.085
3 rd	Port 2	0.098	0.639	0.063	
	Port 3	0.689	0.832	0.573	0.720
4 th	Port 1	0.123	0.201	0.025	
	Port 3	0.679	0.302	0.205	0.950
5 th	Port 2	0.085	0.171	0.015	
	Port 3	0.713	0.156	0.111	1.076
6 th	Port 1	0.117	0.050	0.006	
	Port 3	0.697	0.112	0.078	1.160
7 th	Port 2	0.080	0.036	0.003	
	Port 3	0.708	0.047	0.033	1.196

Table C-106 Cumulative concentration for Fluoride - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0.000	0.000	0.000
2 nd	Port 1	0.124	0.035	0.004	
	Port 3	0.662	0.000	0.000	0.004
3 rd	Port 2	0.086	0.033	0.003	
	Port 3	0.714	0.032	0.023	0.030
4 th	Port 1	0.097	0.014	0.001	
	Port 3	0.669	0.014	0.009	0.041
5 th	Port 2	0.080	0.011	0.001	
	Port 3	0.709	0.007	0.005	0.047
6 th	Port 1	0.115	0.004	0.000	
	Port 3	0.651	0.006	0.004	0.051
7 th	Port 2	0.068	0.003	0.000	
	Port 3	0.726	0.003	0.002	0.053

Table C-107 Cumulative concentration for Fluoride - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0.000	0.000	0.000
2 nd	Port 1	0.118	0.638	0.075	
	Port 3	0.664	0.000	0.000	0.075
3 rd	Port 2	0.082	0.707	0.058	
	Port 3	0.716	0.902	0.646	0.779
4 th	Port 1	0.107	0.230	0.025	
	Port 3	0.672	0.340	0.228	1.033
5 th	Port 2	0.075	0.173	0.013	
	Port 3	0.727	0.150	0.109	1.154
6 th	Port 1	0.095	0.066	0.006	
	Port 3	0.667	0.131	0.087	1.248
7 th	Port 2	0.068	0.021	0.001	
	Port 3	0.722	0.055	0.040	1.289

Table C-108 Cumulative concentration for Nitrite-N - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.009	0.001	
	Port 3	0.675	0.000	0.000	0.001
3 rd	Port 2	0.084	0.010	0.001	
	Port 3	0.718	0.008	0.006	0.008
4 th	Port 1	0.119	0.003	0.000	
	Port 3	0.669	0.006	0.004	0.012
5 th	Port 2	0.082	0.003	0.000	
	Port 3	0.718	0.003	0.002	0.014
6 th	Port 1	0.108	0.001	0.000	
	Port 3	0.687	0.002	0.001	0.016
7 th	Port 2	0.075	0.001	0.000	
	Port 3	0.700	0.001	0.001	0.017

Table C-109 Cumulative concentration for Nitrite-N - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	0.141	0.020	
	Port 3	0.658	0.000	0.000	0.020
3 rd	Port 2	0.098	0.157	0.015	
	Port 3	0.689	0.035	0.024	0.059
4 th	Port 1	0.123	0.059	0.007	
	Port 3	0.679	0.168	0.114	0.181
5 th	Port 2	0.085	0.053	0.004	
	Port 3	0.713	0.050	0.036	0.221
6 th	Port 1	0.117	0.010	0.001	
	Port 3	0.697	0.043	0.030	0.252
7 th	Port 2	0.080	0.017	0.001	
	Port 3	0.708	0.018	0.013	0.266

Table C-110 Cumulative concentration for Nitrite-N - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0.000	0.000	0.000
2 nd	Port 1	0.124	0.008	0.001	
	Port 3	0.662	0.000	0.000	0.001
3 rd	Port 2	0.086	0.009	0.001	
	Port 3	0.714	0.006	0.004	0.006
4 th	Port 1	0.097	0.004	0.000	
	Port 3	0.669	0.004	0.003	0.009
5 th	Port 2	0.080	0.003	0.000	
	Port 3	0.709	0.003	0.002	0.011
6 th	Port 1	0.115	0.001	0.000	
	Port 3	0.651	0.002	0.001	0.013
7 th	Port 2	0.068	0.001	0.000	
	Port 3	0.726	0.001	0.001	0.013

Table C-111 Cumulative concentration for Nitrite-N - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0.000	0.000	0.000
2 nd	Port 1	0.118	0.168	0.020	
	Port 3	0.664	0.000	0.000	0.020
3 rd	Port 2	0.082	0.150	0.012	
	Port 3	0.716	0.025	0.018	0.050
4 th	Port 1	0.107	0.085	0.009	
	Port 3	0.672	0.184	0.124	0.183
5 th	Port 2	0.075	0.064	0.005	
	Port 3	0.727	0.065	0.047	0.235
6 th	Port 1	0.095	0.022	0.002	
	Port 3	0.667	0.038	0.025	0.262
7 th	Port 2	0.068	0.030	0.002	
	Port 3	0.722	0.016	0.012	0.276

Table C-112 Cumulative concentration for Bromide - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.022	0.003	
	Port 3	0.675	0.000	0.000	0.003
3 rd	Port 2	0.084	0.025	0.002	
	Port 3	0.718	0.037	0.027	0.031
4 th	Port 1	0.119	0.008	0.001	
	Port 3	0.669	0.010	0.007	0.039
5 th	Port 2	0.082	0.007	0.001	
	Port 3	0.718	0.005	0.004	0.043
6 th	Port 1	0.108	0.001	0.000	
	Port 3	0.687	0.005	0.003	0.047
7 th	Port 2	0.075	0.002	0.000	
	Port 3	0.700	0.002	0.001	0.049

Table C-113 Cumulative concentration for Bromide - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	0.390	0.055	
	Port 3	0.658	0.000	0.000	0.055
3 rd	Port 2	0.098	0.464	0.045	
	Port 3	0.689	0.676	0.465	0.566
4 th	Port 1	0.123	0.192	0.024	
	Port 3	0.679	0.227	0.154	0.744
5 th	Port 2	0.085	0.142	0.012	
	Port 3	0.713	0.111	0.079	0.835
6 th	Port 1	0.117	0.057	0.007	
	Port 3	0.697	0.127	0.089	0.930
7 th	Port 2	0.080	0.067	0.005	
	Port 3	0.708	0.053	0.038	0.973

Table C-114 Cumulative concentration for Bromide - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0.000	0.000	0.000
2 nd	Port 1	0.124	0.021	0.003	
	Port 3	0.662	0.000	0.000	0.003
3 rd	Port 2	0.086	0.023	0.002	
	Port 3	0.714	0.034	0.024	0.029
4 th	Port 1	0.097	0.009	0.001	
	Port 3	0.669	0.010	0.007	0.036
5 th	Port 2	0.080	0.007	0.001	
	Port 3	0.709	0.005	0.003	0.040
6 th	Port 1	0.115	0.003	0.000	
	Port 3	0.651	0.005	0.003	0.043
7 th	Port 2	0.068	0.002	0.000	
	Port 3	0.726	0.002	0.002	0.045

Table C-115 Cumulative concentration for Bromide - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0.000	0.000	0.000
2 nd	Port 1	0.118	0.397	0.047	
	Port 3	0.664	0.000	0.000	0.047
3 rd	Port 2	0.082	0.441	0.036	
	Port 3	0.716	0.644	0.461	0.544
4 th	Port 1	0.107	0.239	0.026	
	Port 3	0.672	0.193	0.130	0.700
5 th	Port 2	0.075	0.136	0.010	
	Port 3	0.727	0.095	0.069	0.779
6 th	Port 1	0.095	0.061	0.006	
	Port 3	0.667	0.108	0.072	0.857
7 th	Port 2	0.068	0.083	0.006	
	Port 3	0.722	0.045	0.033	0.895

Table C-116 Cumulative concentration for Nitrate-N - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.025	0.003	
	Port 3	0.675	0.000	0.000	0.003
3 rd	Port 2	0.084	0.030	0.003	
	Port 3	0.718	0.031	0.022	0.028
4 th	Port 1	0.119	0.010	0.001	
	Port 3	0.669	0.013	0.009	0.038
5 th	Port 2	0.082	0.008	0.001	
	Port 3	0.718	0.007	0.005	0.043
6 th	Port 1	0.108	0.009	0.001	
	Port 3	0.687	0.006	0.004	0.048
7 th	Port 2	0.075	0.006	0.000	
	Port 3	0.700	0.003	0.002	0.050

Table C-117 Cumulative concentration for Nitrate-N - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	0.313	0.044	
	Port 3	0.658	0.000	0.000	0.044
3 rd	Port 2	0.098	0.347	0.034	
	Port 3	0.689	0.460	0.317	0.395
4 th	Port 1	0.123	0.225	0.028	
	Port 3	0.679	0.300	0.204	0.626
5 th	Port 2	0.085	0.194	0.016	
	Port 3	0.713	0.090	0.064	0.707
6 th	Port 1	0.117	0.061	0.007	
	Port 3	0.697	0.033	0.023	0.737
7 th	Port 2	0.080	0.071	0.006	
	Port 3	0.708	0.014	0.010	0.752

Table C-118 Cumulative concentration for Nitrate-N - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0.000	0.000	0.000
2 nd	Port 1	0.124	0.019	0.002	
	Port 3	0.662	0.000	0.000	0.002
3 rd	Port 2	0.086	0.025	0.002	
	Port 3	0.714	0.018	0.013	0.017
4 th	Port 1	0.097	0.010	0.001	
	Port 3	0.669	0.012	0.008	0.026
5 th	Port 2	0.080	0.008	0.001	
	Port 3	0.709	0.006	0.004	0.031
6 th	Port 1	0.115	0.008	0.001	
	Port 3	0.651	0.005	0.003	0.035
7 th	Port 2	0.068	0.006	0.000	
	Port 3	0.726	0.002	0.002	0.037

Table C-119 Cumulative concentration for Nitrate-N - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0.000	0.000	0.000
2 nd	Port 1	0.118	0.510	0.060	
	Port 3	0.664	0.000	0.000	0.060
3 rd	Port 2	0.082	0.349	0.029	
	Port 3	0.716	0.332	0.238	0.327
4 th	Port 1	0.107	0.344	0.037	
	Port 3	0.672	0.254	0.171	0.534
5 th	Port 2	0.075	0.223	0.017	
	Port 3	0.727	0.101	0.073	0.624
6 th	Port 1	0.095	0.153	0.015	
	Port 3	0.667	0.051	0.034	0.673
7 th	Port 2	0.068	0.087	0.006	
	Port 3	0.722	0.021	0.015	0.694

Table C-120 Cumulative concentration for O-Phosphate - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.120	0.015	
	Port 3	0.675	0.000	0.000	0.015
3 rd	Port 2	0.084	0.146	0.012	
	Port 3	0.718	0.032	0.023	0.050
4 th	Port 1	0.119	0.113	0.013	
	Port 3	0.669	0.067	0.045	0.108
5 th	Port 2	0.082	0.086	0.007	
	Port 3	0.718	0.046	0.033	0.148
6 th	Port 1	0.108	0.081	0.009	
	Port 3	0.687	0.046	0.032	0.189
7 th	Port 2	0.075	0.067	0.005	
	Port 3	0.700	0.019	0.014	0.207

Table C-121 Cumulative concentration O-Phosphate - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	2.682	0.381	
	Port 3	0.658	0.000	0.000	0.381
3 rd	Port 2	0.098	2.328	0.228	
	Port 3	0.689	3.545	2.442	3.051
4 th	Port 1	0.123	2.172	0.267	
	Port 3	0.679	3.205	2.176	5.495
5 th	Port 2	0.085	1.720	0.146	
	Port 3	0.713	1.656	1.180	6.821
6 th	Port 1	0.117	1.178	0.138	
	Port 3	0.697	1.733	1.208	8.167
7 th	Port 2	0.080	1.443	0.115	
	Port 3	0.708	0.728	0.515	8.798

Table C-121 Cumulative concentration for O-Phosphate - Column D.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.806	3.125	2.519	2.519
2 nd	Port 1	0.113	0.000	0.000	
	Port 3	0.670	1.982	1.328	3.847
3 rd	Port 2	0.078	0.067	0.005	
	Port 3	0.708	0.417	0.295	4.147
4 th	Port 1	0.102	0.000	0.000	
	Port 3	0.684	0.007	0.005	4.152
5 th	Port 2	0.074	0.000	0.000	
	Port 3	0.708	0.000	0.000	4.152
6 th	Port 1	0.107	0.000	0.000	
	Port 3	0.667	0.000	0.000	4.152
7 th	Port 2	0.071	0.000	0.000	
	Port 3	0.723	0.000	0.000	4.152

Table C-123 Cumulative concentration for O-Phosphate - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	3.095	2.448	2.448
2 nd	Port 1	0.124	0.147	0.018	
	Port 3	0.662	2.924	1.936	4.402
3 rd	Port 2	0.086	0.183	0.016	
	Port 3	0.714	0.026	0.019	4.437
4 th	Port 1	0.097	0.109	0.011	
	Port 3	0.669	0.047	0.031	4.479
5 th	Port 2	0.080	0.076	0.006	
	Port 3	0.709	0.037	0.026	4.511
6 th	Port 1	0.115	0.084	0.010	
	Port 3	0.651	0.034	0.022	4.543
7 th	Port 2	0.068	0.054	0.004	
	Port 3	0.726	0.014	0.010	4.557

Table C-124 Cumulative concentration for O-Phosphate - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	3.210	2.404	2.404
2 nd	Port 1	0.118	3.406	0.402	
	Port 3	0.664	2.951	1.959	4.766
3 rd	Port 2	0.082	3.674	0.301	
	Port 3	0.716	2.119	1.517	6.584
4 th	Port 1	0.107	2.889	0.309	
	Port 3	0.672	2.833	1.904	8.797
5 th	Port 2	0.075	2.014	0.151	
	Port 3	0.727	1.811	1.317	10.265
6 th	Port 1	0.095	1.289	0.122	
	Port 3	0.667	1.750	1.167	11.554
7 th	Port 2	0.068	1.539	0.105	
	Port 3	0.722	0.735	0.531	12.190

Table C-125 Cumulative concentration for Sulfate - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0.000	0.000	0.000
2 nd	Port 1	0.121	0.154	0.019	
	Port 3	0.675	0.000	0.000	0.019
3 rd	Port 2	0.084	0.120	0.010	
	Port 3	0.718	0.143	0.103	0.131
4 th	Port 1	0.119	0.043	0.005	
	Port 3	0.669	0.110	0.074	0.210
5 th	Port 2	0.082	0.033	0.003	
	Port 3	0.718	0.046	0.033	0.246
6 th	Port 1	0.108	0.022	0.002	
	Port 3	0.687	0.035	0.024	0.272
7 th	Port 2	0.075	0.033	0.002	
	Port 3	0.700	0.015	0.010	0.285

Table C-126 Cumulative concentration for Sulfate - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	0.000	0.000	0.000
2 nd	Port 1	0.142	2.080	0.295	
	Port 3	0.658	0.000	0.000	0.295
3 rd	Port 2	0.098	1.500	0.147	
	Port 3	0.689	2.813	1.938	2.380
4 th	Port 1	0.123	1.174	0.144	
	Port 3	0.679	2.100	1.426	3.951
5 th	Port 2	0.085	0.803	0.068	
	Port 3	0.713	0.729	0.520	4.539
6 th	Port 1	0.117	0.724	0.085	
	Port 3	0.697	0.804	0.560	5.184
7 th	Port 2	0.080	0.438	0.035	
	Port 3	0.708	0.338	0.239	5.458

Table C-127 Cumulative concentration for Sulfate - Column D.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.806	1.351	1.089	1.089
2 nd	Port 1	0.113	0.079	0.009	
	Port 3	0.670	0.526	0.352	1.450
3 rd	Port 2	0.078	0.194	0.015	
	Port 3	0.708	0.185	0.131	1.596
4 th	Port 1	0.102	0.000	0.000	
	Port 3	0.684	0.049	0.034	1.630
5 th	Port 2	0.074	0.000	0.000	
	Port 3	0.708	0.000	0.000	1.630
6 th	Port 1	0.107	0.000	0.000	
	Port 3	0.667	0.000	0.000	1.630
7 th	Port 2	0.071	0.000	0.000	
	Port 3	0.723	0.000	0.000	1.630

Table C-128 Cumulative concentration for Sulfate - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	1.119	0.885	0.885
2 nd	Port 1	0.124	0.253	0.031	
	Port 3	0.662	0.982	0.650	1.567
3 rd	Port 2	0.086	0.351	0.030	
	Port 3	0.714	0.179	0.128	1.724
4 th	Port 1	0.097	0.060	0.006	
	Port 3	0.669	0.101	0.068	1.798
5 th	Port 2	0.080	0.055	0.004	
	Port 3	0.709	0.057	0.040	1.842
6 th	Port 1	0.115	0.033	0.004	
	Port 3	0.651	0.061	0.040	1.886
7 th	Port 2	0.068	0.029	0.002	
	Port 3	0.726	0.026	0.019	1.906

Table C-129 Cumulative concentration for Sulfate - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	1.273	0.953	0.953
2 nd	Port 1	0.118	3.521	0.415	
	Port 3	0.664	1.079	0.716	2.085
3 rd	Port 2	0.082	3.617	0.297	
	Port 3	0.716	3.314	2.373	4.755
4 th	Port 1	0.107	1.525	0.163	
	Port 3	0.672	2.195	1.475	6.393
5 th	Port 2	0.075	1.193	0.089	
	Port 3	0.727	1.052	0.765	7.248
6 th	Port 1	0.095	0.833	0.079	
	Port 3	0.667	1.323	0.882	8.209
7 th	Port 2	0.068	0.916	0.062	
	Port 3	0.722	0.556	0.401	8.673

Table C-130 Cumulative concentration for COD- Column A.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L.)	Mass (mg)	Cumulative mass
1 st	Port 3	0.803	21	17	17
2 nd	Port 1	0.133	29	4	
	Port 3	0.675	26	18	38
3 rd	Port 2	0.093	36	3	
	Port 3	0.700	27	19	61
4 th	Port 1	0.124	35	4	
	Port 3	0.688	21	14	79
5 th	Port 2	0.089	45	4	
	Port 3	0.693	33	23	106
6 th	Port 1	0.128	36	5	
	Port 3	0.683	29	20	131
7 th	Port 2	0.087	39	3	
	Port 3	0.706	39	28	162

Table C-131 Cumulative concentration for COD - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	27	22	22
2 nd	Port 1	0.121	63	8	
	Port 3	0.675	36	24	54
3 rd	Port 2	0.084	29	2	
	Port 3	0.718	36	26	82
4 th	Port 1	0.119	39	5	
	Port 3	0.669	36	24	111
5 th	Port 2	0.082	36	3	
	Port 3	0.718	32	23	137
6 th	Port 1	0.108	48	5	
	Port 3	0.687	35	24	166
7 th	Port 2	0.075	27	2	
	Port 3	0.700	41	29	197

Table C-132 Cumulative concentration for COD - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	50	38	38
2 nd	Port 1	0.142	680	97	
	Port 3	0.658	108	71	205
3 rd	Port 2	0.098	416	41	
	Port 3	0.689	456	314	560
4 th	Port 1	0.123	386	47	
	Port 3	0.679	351	238	846
5 th	Port 2	0.085	321	27	
	Port 3	0.713	235	168	1041
6 th	Port 1	0.117	206	24	
	Port 3	0.697	257	179	1244
7 th	Port 2	0.080	122	10	
	Port 3	0.708	108	76	1331

Table C-133 Cumulative concentration for COD - Column D.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.806	326	263	263
2 nd	Port 1	0.113	72	8	
	Port 3	0.670	69	46	317
3 rd	Port 2	0.078	188	15	
	Port 3	0.708	80	57	388
4 th	Port 1	0.102	21	2	
	Port 3	0.684	191	131	521
5 th	Port 2	0.074	168	12	
	Port 3	0.708	359	254	788
6 th	Port 1	0.107	27	3	
	Port 3	0.667	69	46	837
7 th	Port 2	0.071	36	3	
	Port 3	0.723	27	20	859

Table C-134 Cumulative concentration for COD - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	215	170	170
2 nd	Port 1	0.124	282	35	
	Port 3	0.662	115	76	281
3 rd	Port 2	0.086	293	25	
	Port 3	0.714	128	91	398
4 th	Port 1	0.097	69	7	
	Port 3	0.669	285	191	595
5 th	Port 2	0.080	201	16	
	Port 3	0.709	420	298	909
6 th	Port 1	0.115	63	7	
	Port 3	0.651	120	78	994
7 th	Port 2	0.068	87	6	
	Port 3	0.726	72	52	1053

Table C-135 Cumulative concentration for COD - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	251	188	188
2 nd	Port 1	0.118	876	103	
	Port 3	0.664	130	86	377
3 rd	Port 2	0.082	562	46	
	Port 3	0.716	529	379	802
4 th	Port 1	0.107	446	48	
	Port 3	0.672	410	276	1126
5 th	Port 2	0.075	372	28	
	Port 3	0.727	273	198	1352
6 th	Port 1	0.095	240	23	
	Port 3	0.667	306	204	1579
7 th	Port 2	0.068	140	10	
	Port 3	0.722	129	93	1682

Table C-136 Cumulative concentration for Ammonia - Column B.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.810	0	0	0
2 nd	Port 1	0.121	48	6	
	Port 3	0.675	0	0	6
3 rd	Port 2	0.084	36	3	
	Port 3	0.718	18	13	22
4 th	Port 1	0.119	5	1	
	Port 3	0.669	2	1	24
5 th	Port 2	0.082	6	0	
	Port 3	0.718	3	2	27
6 th	Port 1	0.108	3	0	
	Port 3	0.687	4	2	30
7 th	Port 2	0.075	4	0	
	Port 3	0.700	1	1	31

Table C-137 Cumulative concentration for Ammonia - Column C.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.756	1	1	1
2 nd	Port 1	0.142	126	18	
	Port 3	0.658	13	9	27
3 rd	Port 2	0.098	120	12	
	Port 3	0.689	105	72	112
4 th	Port 1	0.123	102	13	
	Port 3	0.679	64	43	168
5 th	Port 2	0.085	76	6	
	Port 3	0.713	17	12	187
6 th	Port 1	0.117	33	4	
	Port 3	0.697	37	26	216
7 th	Port 2	0.080	17	1	
	Port 3	0.708	16	11	229

Table C-138 Cumulative concentration for Ammonia - Column E.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.791	0	0	0
2 nd	Port 1	0.124	51	6	
	Port 3	0.662	0	0	7
3 rd	Port 2	0.086	29	2	
	Port 3	0.714	12	8	18
4 th	Port 1	0.097	17	2	
	Port 3	0.669	4	3	22
5 th	Port 2	0.080	8	1	
	Port 3	0.709	2	2	24
6 th	Port 1	0.115	6	1	
	Port 3	0.651	2	1	26
7 th	Port 2	0.068	3	0	
	Port 3	0.726	1	1	27

Table C-139 Cumulative concentration for Ammonia - Column F.

Order of Application	Extraction port	Volume (Litre)	Avg. Conc. (mg/L)	Mass (mg)	Cumulative mass
1 st	Port 3	0.749	0	0	0
2 nd	Port 1	0.118	185	22	
	Port 3	0.664	0	0	22
3 rd	Port 2	0.082	177	15	
	Port 3	0.716	95	68	104
4 th	Port 1	0.107	173	19	
	Port 3	0.672	58	39	162
5 th	Port 2	0.075	111	8	
	Port 3	0.727	45	33	203
6 th	Port 1	0.095	132	13	
	Port 3	0.667	49	33	249
7 th	Port 2	0.068	25	2	
	Port 3	0.722	21	15	265