Quality Assurance Plan

for

A Project Entitled:

"Earthen Liners: a Study of Transit Time"

USEPA Cooperative Agreement Mo. CR-812650-01

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CONTENTS

1.0 INTRODUCTION

Earthen liners are commonly used to prevent groundwater contamination from waste-disposal facilities. Although performance requirements typically exist for the hydraulic conductivity of a liner, these specifications are generally based on laboratory tests of liner samples. Few studies have been done, however, to relate these laboratory measurements to the actual field performance of earthen liners. The purposes of this study are: (1) to determine transit times for water and tracer movement through a full-scale, compacted earthen liner, and (2) to test the accuracy and practicality of available methods for predicting the transit time of water and solutes through compacted earthen liners.

This document presents the quality assurance (QA) plan for a cooperative study to be conducted by the Illinois State Geological Survey (ISGS) and the U.S. Environmental Protection Agency (USEPA) in studying transit time in a full-scale earthen liner. This plan has been prepared to fulfill the requirements of the award conditions and has been prepared according to guidelines and specifications given in QAMS-005/80 (December 29, 1980).

2.0 PROJECT DESCRIPTION

2.1 Objectives and Information Utilization

The primary objectives of this project are to:

(1) Determine transit times for water and solute (tracer) movement through a partially saturated, compacted, earthen liner. A field-scale liner will be constructed using full-scale equipment.

(2) Test the accuracy and practicality of available methods for predicting transit time of water and nonreactive solute (tracer) movement through partially-saturated, compacted, earthen liners.

The data collected during this study will be used to determine the hydraulic and engineering characteristics of an experimental, field-scale, compacted earthen liner. The data will primarily be used to examine the rates of water and solute (tracer) movement through the liner. Data from laboratory studies will be examined to determine the degree of correlation with field data. Conclusions will be made on the basis of these data regarding the performance of actual field-scale, compacted earthen liners and the adequacy of field and laboratory tests for predicting liner performance.

2.2 Experimental Design

This project consists of three phases, each with multidisciplinary parts: 1) laboratory characterization of materials; 2) construction and testing of a pilot-scale liner and; 3) construction and monitoring of a field-scale liner. The project involves the disciplines of hydrogeology and soil

physics, engineering geology and geochemistry. Each discipline has clearly defined roles within the project, although coordination and interaction among the disciplines will be necessary for the successful completion of the study.

The project is divided into the three phases outlined above. The first phase is selection and characterization of the earthen materials that will be used in the field scale liner. This phase will involve laboratory studies of physical, engineering and hydrologic characteristics of possible liner materials. Phase one will be finished by early summer, 1986.

The second phase is the construction and monitoring of the prototype liner. The purpose of this phase is to test construction methods and monitoring techniques. A limited number of measurements will be taken of soil properties of the test liner.

Phase three is the construction and monitoring of the field-scale clay liner. This liner will be enclosed in a building to help protect it from freezing and precipitation so that the experiments can be better controlled. Extensive monitoring will be performed using tracers and various indirect methods for determining soil moisture movement. Tracers will be incorporated in building the liner for later studies on effective porosity. The liner will be ponded and the outflow will be measured to determine the water balance and the saturated hydraulic conductivity of the liner.

3.0 PROJECT ORGANIZATION AND RESPONSIBILITIES

3.1 Project Organization

The project organization structure is illustrated in Figure 3 1. The project will be conducted within the Groundwater, Engineering Geology and Geochemistry Sections of the Illinois State Geological Survey's General and Environmental Geology and Chemical Groups. K. Cartwright is the Principal Investigator, R. A. Griffin is the Project Manager and B. L. Herzog is the Quality Control Officer for this project.

3.2 Key Staff and Responsibilities

As principal investigator, K. Cartwright will be ultimately responsible for the management of the overall project; this includes cost control, planning and reporting. As project manager, Dr. Griffin will be responsible for operation and coordination of the project. Quality control officer, Ms. Herzog, will be responsible for surveillance of the accuracy, precision and completeness of all data collected for this project. B. L. Herzog, K. A. Albrecht, T. M. Larson, W. J. Su and W. J. Morse will be conducting the investigations of hydrogeologic properties and moisture movement. K. A. Albrecht will design the sampling strategy. Engineering properties will be determined by W. J. Su and C. J. Stohr, while particle size analyses will be performed by R. J. Roeper. Laboratory column studies of moisture movement will be performed by T. H. Larson. Tracer analyses will be performed by Dr J. B. Risatti and I. G. Krapac. Dr. Risatti will also conduct studies of microbiological degradation in the liner.

3.3 Cooperation with EPA Officer

K. Cartwright will maintain periodic contact with the EPA's project officer, Mr. K. Dotson, to facilitate the cooperative planning implicit to this agreement. Mr. Dotson will be kept informed of the project's progress with Quarterly Reports and consulted at critical junctions during the project.

Figure 3.1: Project Organization

University of Illinois
Board of Trustees

Illinois State Geological Survey
M. W. Leighton, Chief

Principal Investigator
K. Cartwright, Principal Geologist
General and Environmental Geology Group

Project Manager
R. A. Griffin, Geochemist and Acting Section Head
Groundwater Section

Quality Assurance Officer
B. L. Herzog, Associate Geologist
Groundwater Section

Sampling Strategy	**Engineering Geology**	**Particle size**	**Laboratory Hydraulic Conductivity**	**Field hydraulic Properties**	**Tracer Analysis Studies**	**Microbial Degradation**
K. A. Albrecht	W. J. Su C. J. Stohr	R. J. Roeper	B. L. Herzog K. A. Albrecht W. J. Morse	K. A. Albrecht B. L. Herzog	J. B. Risatti I. G. Krapac	J. B. Risatti

Column Studies
T. M. Larson
J. B. Risatti

4.0 QUALITY ASSURANCE OBJECTIVES

The ultimate validity of the final procedures and results of this study is dependent upon the accuracy and precision of the experimental data generated during this project. Therefore, the main objective of this quality assurance project plan is to ensure the reproducibility and validity of the data collected in all investigations. Besides ensuring that data of known quality are generated, the quality assurance plan is also intended to document and verify the conditions and techniques under which the data are obtained.

The quality assurance plan for this project falls into three categories:

- Preventive
- Assessment
- Corrective

Instrument maintenance, calibration checks, and replicate analyses are activities designed to prevent incorrect data acquisition. The sampling strategy will be designed to assure that the data obtained will be representative of the system. Data handling errors are being minimized through the use of computerized systems for data storage and statistical analysis. Assessment activities include performance and systems audits and performance monitoring. These are intended to indicate when the validity of any experimental procedure or data is questionable. This will then trigger the corrective activites of evaluating the problem, identifying a solution and acting to eliminate the problem.

Estimates of the quality assurance objectives for each of the tests anticipated for use in this project are given in Table 4.1. No precision or accuracy values are given for several tests listed in Table 4.1 because no

such values are available and a guess at the values would be inappropriate. Most of the missing values are for tests of hydraulic properties. The actual values cannot be determined before the tests are performed; then precision and accuracy can be calculated as discussed in Section 10. Descriptions of the individual tests follow in Section 5.

.1: Summary of Estimated Precision, Accuracy and Completeness Objectives for Each Test Described in Section 5.

er	Method	Precision (CV(%))	Accuracy (% Bias)	Completeness (%)
e Size	Sieve	5%	5%	100%
utions	Hydrometer	5%	5%	100%
	X-ray Sedigraph	1%	5%	100%
c Gravity	Pycnometer	5%	5%	100%
re	Conventional Oven	.1%	5%	100%
	Microwave Oven	1%	5%	100%
	Seaman Nuclear	5%	10%	95%
	Troxler Nuclear	5%	10%	95%
	Speedy Moisture Tester	10%	10%	95%
	Seaman Nuclear	5%	10%	95%
	Troxler Nuclear	5%	10%	95%
	Drive Cylinder	5%	10%	95%
	Rubber Balloon	5%	10%	95%
	Sand Cone	5%	10%	95%
rg	Liquid Limit	NA	NA	100%
	Plastic Limit	NA	NA	100%
	Shrinkage Factor	NA	NA	100%
tion	Standard Proctor	10%	10%	90%
	Modified Proctor	10%	10%	90%
	Havard Miniature	NA	NA	90%
	Double Hydrometer	5%	5%	95%
sion	Pinhole Test	5%	5%	95%
ted	Triaxial Test	NA	NA	95%
lic	Harvard-core miniature	NA	NA	95%
tivity	permeameter	NA	NA	95%
ater Velocity	Laboratory Miscible displacement	NA	NA	95%
sion Coefficient	model fitted from breakthrough			
	data	NA	NA	95%
re Retention teristic $\theta(\Psi)$	Stepwis Equilibrium	0.1	NA	95%
uated Hydraulic	Steady-state	NA	NA	90%
tivity $K(\Psi),K(\theta)$	transient	NA	NA	90%
	calculation	NA	NA	90%
ivity, $K(\theta),\theta(\Psi)$	onestep	NA	NA	90%

able 4.1: (cont.)

arameter	Method	Precision	Accuracy	Completeness
nfiltration Rate (in-situ)	Double-ring ponded	NA	NA	100%
esident Solute oncentrations (in-situ)	Soil-water Suction Samplers	NA	NA	95%
lux at Liner Bottom and lux-averaged Solute oncentrations (in-situ)	Drains	NA	NA	95%
il Exchange Capacity	$MgCl_2$percolation	5%	10%	100%
tal Organic Carbon	TOC Ionanalzyer	3%	3%	100%
l	Electrode & Digital Ionanalyzer	3%	3%	100%
ter Extractables	ICAP&IC	5%	5%	100%
l EXTRACTABLES	ICAP&IC	5%	5%	100%
tch Absorption	Roy, et al	10%	NA[a]	90%
organic analysis	ICAP	5%	5%	100%
	IC	10%	5%	100%
	Liquid Scintillation	5%	5%	90%
ganic analysis	GC	5%	3%	100%
	HPLC	5%	3%	100%

n appropriate standard is unknown to the investigators, thus an estimate of accuracy is not ssible.

5.0 ANALYTICAL AND EXPERIMENTAL QUALITY ASSURANCE PROCEDURES

5.1 Sampling Strategy - Statistical Aspects

5.1.1 Actual Liner

Design of the sampling strategy for characterization of the field-scale clay liner will be based on decisions regarding tolerable errors, treatment of spatial dependence, and time and labor constraints as they pertain to project goals. Conventional and geostatistical methods will be used to evaluate conservative sample numbers to ensure parameter means to within 'reasonable' errors with a high level of confidence.

A two-stage sampling plan will be employed. A large number of samples will be taken - more than is indicated by conventional and geostatistical estimates. Since sampling is a relatively inexpensive procedure compared to the laboratory testing and because we will only have one opportunity to obtain some of the samples before ponding the clay liner, this is a safeguard to guarantee adequate sample number, n. Initially, only about half of the samples will be analyzed. If results are sufficiently accurate, then no further analyses will be necessary. It is anticipated that the area will be quite uniform. Additional samples will be analyzed as necessary until sufficiently accurate results are obtained.

The conventional method of estimating the number of samples can be found in any standard statistics book:

$$n = \left[\frac{t_{C_2} \bullet CV}{F} \right]^2 \qquad [1]$$

where n is the number of samples required to estimate the true mean to within F percent error from the sample mean at the C confidence level; t is the two-tailed Student's t-value for (n-1) degrees of freedom and CV is the coefficient of variation (sample standard deviation divided by the sample mean) in percent (Snedecor and Cochran, 1980). This equation is solved iteratively since the value of t is a function of n. The method is based on the central limit theorem and assumes a normal population distribution (although it is commonly used for data which are not normal). Finite variance and random independent samples are also assumed. No accounting of spatial dependence can be made using this approach.

Many of the physical and chemical soil properties of concern in this study, such as bulk density, texture, Atterburg limits and cation exchange capacity, tend to be normally distributed and generally exhibit little spatial dependence. Therefore, conventional sample number estimation is appropriate. Means for these properties will be estimated to within a reasonable error i.e., $\leq$ 10 percent. Initial variance estimates for each parameter will either be obtained from pertinent studies already documented; or if none are available, best estimates will be used. Sample numbers estimated using the conventional approach will be conservative values since they do not account for spatial dependence. Most of the analyses for these properties are also relatively inexpensive and simple so there are generally few time or labor constraints to limit sample numbers.

Flow and transport parameters like hydraulic conductivities, pore water velocities and dispersion coefficients however tend to be lognormally distributed and exhibit significant spatial dependence. Conventional statistics may be used on data which have been logtransformed; however, often the variation may still be very high and the required sample numbers

prohibitive. Schemes have been devised which take advantage of the knowledge of spatial dependence to optimize sample numbers. That is, because samples spaced close together are similar, fewer samples are needed to characterize the area. Geostatistics and kriging provide a framework for expressing this variability in a quantitative manner.

In geostatistics, soil properties are regionalized variables (i.e., variables that are distributed in space). Statistical behavior of regionalized variables is described by their first-order and second-order moments repectively as:

$$E\{x_i\} = m_i \qquad [2]$$

$$\gamma(x_i, x_{i+h}) = \tfrac{1}{2}\,\mathrm{var}\ \{x_i - x_{i+h}\} \qquad [3]$$

where x is the variable of interest observed at locations i (i=1,...n) and E is the expectation or mean value (Journel and Huijbregts, 1978). In practice it is assumed that the expected value is constant over the region and estimated by the sample mean. Semivariance is denoted γ and h is the vector that separates observations x_i and x_{i+h} and is referred to as the lag. Plots of $\gamma(h)$ versus h are semivariograms which in geostatistics may be used to determine precision or sampling effort (as opposed to the variance controlling sampling effort in classical statistics). Property values at locations between sampling points may be obtained by the kriging interpolation method.

Webster and Burgess (1984) showed that for small land areas true variances can be much less than those apparent using classical theory and that observations which are autocorrelated in space may have their sampling effort overestimated 2-3 times by classical predictions. In a series of papers,

McBratney, Webster and Burgess (McBratney, Webster and Burgess (1981); McBratney and Webster (1981, 1983); Burgess, Webster and McBratney (1981); Burgess and Webster (1980 a,b,)) presented theory, methods (including a fortran program), and examples of a geostatistical method to maximize precision and minimize sampling effort for small isolated parcels of land. The method assumes that the maximum standard error of a kriged estimate is a reasonable measure of the goodness of a sampling scheme. The maximum tolerable estimation variance or kriging variance ($\sigma^2_{K_{max}}$) is defined for point and block values respectively as:

$$\sigma^2_K = \sum_{i=1}^{n} \lambda_i \ \gamma(x_i, x_0) + \psi \qquad [4]$$

and $$\sigma^2_{KB} = \sum_{i=1}^{n} \lambda_i \ \gamma(x_i, V) + \psi_B - \gamma(V,V) \qquad [5]$$

where λ_i are weights chosen so that they sum to 1 to ensure unbiased estimates while minimizing the estimation variance; $\gamma(x_i, x_0)$ is the semivariance between locations x_i and x_0, and $\gamma(x_i, V)$ and $\gamma(V,V,)$ are average semivariances between the observation points and block V and between points within the block respectively. The quantities ψ and ψ_B are Lagrange parameters associated with the minimization process. Equations 4 or 5 (depending on whether estimates are required for points or blocks) are solved for a range of spacings. Graphs of $\sigma^2_{K_{max}}$ against sampling density are plotted and the density required to achieve the desired precision is read from the graph.

Methods described by McBratney, Webster and Burgess will be evaluated along with others (e.g., Russo (1984)) which are based on similar principles. The most suitable method will be selected to determine the optimal number of samples for spatially dependent flow and transport parameters for the ISGS field-scale clay liner. Optimizing the number of samples is desirable because of the difficulty, expense and time required to perform hydraulic conductivity and miscible displacement tests on compacted clays.

A major obstacle to using geostatistics to optimize the sampling scheme is the need to know the semivariogram in advance. Reconnaissance studies are usually necessary to obtain this information since it is important to have the variogram for the properties of interest on the same soil type and area to be studied. Ironically, a very large number of samples is required to obtain results necessary to calculate how few samples could have been taken in the first place to yield the same information.

Rogowski et al. (1985 and personal communication) have collected a large amount of data on flow characteristics of a compacted clay liner of similar size to the one that will be built for the ISGS study. Their estimates of variance and semivariance will be used to help design the most efficient and informative sampling plan for the ISGS clay liner.

Rogowski found coefficients of variation of about 200 percent for hydraulic conductivities, strongly suggesting lognormal distributions. Verification of the population distribution functions will be performed; if they indicate lognormality, equation [1] will be used to calculate n using the logtransformed data. Medians will be estimated by constraining the upper confidence limit to some tolerable error, for example the percent error will be selected to correspond to approximately 0.5 orders of magnitude for the

upper limit. This will yield a conservative estimate of n to use as a guideline for sampling flow parameters. Since we know that the conventional approach will overestimate n, the sample number estimate will be tempered using the semivariograms already available from Rogowski and the optimization methods previously described.

Samples will be spaced on a uniform square grid. This is the best design for geostatistics and semivariance analyses since it provides equally spaced observation points and minimizes the Kriging variance between points. Random sampling schemes are unbiased and commonly preferred among statisticians, but may cluster data spatially unless a large number are taken. Random schemes may also compound systematic and random error.

Scale effects will also be considered in the sampling scheme. Core volume influences means and variances of flow and transport parameters (Albrecht (1985); Sisson and Wierenga (1981); Daniel (1984)). Generally, increasing sample volume decreases variance and, if volume is too small, means may be inaccurate. Rogowski used large infiltration rings (30 cm diameter), so we may expect differences in variance for differences in ring sizes that we use. Sample numbers will be adjusted proportionally so that fewer large rings will be needed than small rings to characterize the area.

Rogowski has not cited evidence of anisotropy, however an effort will be made to examine the possibility of its presence in the ISGS clay liner by employing a two-dimensional semivariogram program.

5.1.2 Selecting Materials for Liner

Soil materials near the site location will be screened to select the most likely candidates for use in the clay liner. After this preliminary assessment to narrow down the number of possible materials, laboratory tests

will be run to determine the most suitable material (especially $K_s \leq 10^{-7}$ cms^{-1} or lowest K_s). Because there is no previous information on these soils and no well-defined area to characterize, the sampling scheme is quite arbitrary. The best that we can do is make some assumptions regardiing representativeness, variance, and homogeneity and guess the number of samples. At least 3-4 bulk samples evenly distributed over the exposed area will be taken per material. At least two to three replicates will be analyzed from each bulk sample. Bulking is a convenient method to reduce costs or increase precision for soil properties that are determined on disturbed soil (Webster, 1977). Bulked samples are also expected to provide a reasonable representation of the material used for the liner since the liner will be constructed from bulked material.

5.1.3 Prototype

Fewer samples will be taken in the prototype than in the actual liner. Only enough samples will be taken to ensure that specifications for the clay liner can be met before it is actually built. Numbers of samples taken in the prototype will primarily depend on time and labor restrictions for sampling and analyses. Results will provide insight to the variability of our own system (as compared to Rogowski's) and will be considered when designing the sampling scheme for the actual liner.

5.2 Sampling and Storage

5.2.1 Scope

Attention to sampling, handling and sample storage is important for interpreting test results. Test results may differ because of the behavior of

clay minerals and the hydration history. Some researchers (i.e., Sangrey et al., 1976, and Sherard et al., 1976) have noted significant changes in test results, due to the method of sample storage. It is necessary to maintain natural soil moisture because some irreversible changes in the engineering properties of the soil may be caused by dehydration. Therefore, a procedure must be implemented to limit the loss of natural water from samples and prevent change of material properties because of dehydration.

5.2.2 Sampling

Soil samples for the study will be collected both at outcrops and from borings.

• 5.2.2.1 Outcrops

Outcrop samples should be taken by exposing a fresh face where soils are moist. Samples may be collected using a hand pick, and shovel or by driving a sampling tube or auger. Possible samplers include the 'Tube density sampler' (Soiltest Model No. CN-1020 or CN-1025), the 'Corps of Engineers surface soil sampler' (Soiltest Model No. CN-1030 or CN-1031) the 'Soil sampling tube' (Soiltest Model No. A-2), the 'Compact sampler' (Soiltest Model No. A-43), and the 'Soil Conservation Service bucket auger' (Soiltest Model No. DR-30 or DR-31).

• 5.2.2.2 Borings

Recommended ASTM procedures for sampling from borings will be drilling rig, as outlined below:

(1) ASTM D1452, Soil investigation and sampling by auger borings. This method covers the use of augers in soil investigations and sampling where disturbed samples are acceptable.

(2) ASTM D1586, Penetration test and split-barrel sampling of soils This method describes a procedure to obtain representative relatively undisturbed soil samples suitable for identification and classification laboratory tests. These sampling procedures are also used to measure the resistance of the soil by counting the number of blows required to achieve a specified depth of penetration of a standard sampler.

(3) ASTM D1587, Thin-walled tube sampling of soils. This method describes a procedure to recover relatively undisturbed soil samples suitable for laboratory tests.

5.2.3 Storage

Samples should be sealed and stored (preferably in 100% humidity) immediately after collection and preparation of a field description. Depending upon the amount of the sample needed, three different storage methods will be used as follows:

- 5.2.3.1 Small Samples

Moisture-tight sample containers made of glass with 4- and 8-oz capacity will be used for moist samples for determination of moisture content and mineral analysis.

- 5.2.3.2 Medium Sample

Each sample will be put into a ziploc bag or double sealed plastic bags first, and then will be stored in an adequate moisture-tight container (e.g., wax-paper sealed can).

- 5.2.3.3 Large Samples

Double-sealed, large-capacity, plastic bags will be used. Bagged samples are then stored in a large insulated chest with water or ice in the bottom. Samples prepared for long-term storage will be kept in a plastic container with water in the bottom to maintain near 100 percent humidity.

5.3 Sample Preparation

5.3.1 Scope

Described here are the methods for preparation of soil samples as received from the field for particle size analysis, determination of Atterberg limits and some compaction tests. For most of engineering property tests, the separation method will be described separately with each procedure.

A laboratory test of an inappropriate sample is often worse than no test at all. Certainly the use of a preparation method which would significantly change the measured soil property should not be allowed. Two prepartion methods will be described in this section.

Some soils never totally dry out in nature; these soils may change characteristics greatly when dried. This is especially true for organic-rich soils and some clay minerals, such as halloysites, which undergo irreversible changes when dried. Therefore, for most of the tests, the wet preparation

method is often recommended when the true particle size, liquid limit, plastic limit, and shrinkage limit are required. For this reason, the mineral composition of the soil will be determined if the dry preparation method is considered for use.

5.3.2 Dry Preparation

The standard procedure suggested by ASTM (American Society for Testing Materials) D-421 will be used.

5.3.3 Wet Preparation

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2217 will be used with one modification. Although the oven drying at low temperature (140°F or 60°C) is allowed for the removal for excess water from the samples, it will not be used for this study. Only air drying will be used. Excess water after preparation will be removed by air drying or vacuuming through a Pasture-Chamberlain porcelain candle filter (Type-F, nominal size of 0.1 micrometer). The vacuum filter system has been set up and used successfully at the Geotechnical Laboratory of Illinois State Geological Survey. With aid of the vacuum system, drying time will be reduced significantly.

5.4 Description and Classification

5.4.1 Scope

Both after sampling and before testing, the sample should be examined and described. There are at least three reasons why this description procedure is extremely desirable.

(1) The procedure gives the described an indication of the expected soil behavior.

(2) It may aid in the interpretation of the results obtained.

(3) A correlation check of laboratory description against that made in the field and recorded in the boring log may detect a possible error in sample numbering.

Soil classification systems commonly used in engineering enable one to use the experience of others. Such systems also facilitate communication between widely separated groups of engineers using the same system. It gives the experienced engineer some idea of the probable soil behavior before test results are obtained. It also offers a guideline for the engineer to set up a reasonable and practical testing program.

There are many detailed soil classification systems currently employed. Most of these systems were designed for use in a particular branch of soils work and are, therefore, limited in their application. The Unified Soil Classification System (as stated in ASTM D-2487) will be employed in this study because it is widely used by the engineering agencies of the U.S. government, including the Bureau of Reclamation and the Army Corps of Engineers, in city building codes, and by many commerical soil testing laboratories and geotechnical consulting firms.

5.4.2 Description

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2488 will be used.

5.4.3 Classification

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2487 will be used.

5.5 Engineering Index Properties

5.5.1 General

Economics often imposes a practical limit on the quantity, and sometimes quality, of testing that is performed to identify and classify a soil mass. On the other hand, enough testing is also necessary to enable the engineer to obtain the behavior and characteristics of the soil mass. Therefore, a set of basic index properties which can be determined quickly and easily is often needed.

There are five basic index tests will be carried out for the project. They are (1) particle size distribution, (2) specific gravity, (3) moisture content, (4) in-situ density, (5) Atterberg Limits. Each basic test may have several different methods. The methods, which will be carried out in this project, depend upon the following factors:

(1) suitability of the method to be used in this project
(2) the field situation;
(3) the precision, accuracy, and completeness of the method;
(4) the required turnaround time of the result.

5.5.2 Particle Size Distribution

- 5.5.2.1 Scope

Most geotechnical engineering laboratories routinely run particle size analysis on virtually every soil sample. Particle size distribution curves have been widely used in the identification and classification of soils.

In this chapter, three procedures of analysis are presented: sieve, hydrometer, and X-Ray Sedigraph methods. The sieve method consists of shaking of the soil through a stack of wire screens with openings of known sizes; the definition of particle diameter for the sieve method is, therefore, the side dimension of a square hole. The other two methods are based upon Stoke's equation for the velocity of a freely falling sphere; the definition of particle diameter for these methods is, therefore, the diameter of a sphere of the same density which falls at the same velocity as the particle in question. A combination of procedures will be used for soils with a large particle size range. The large particle size portion is analyzed by the sieve method and the finer portion is analyzed by one of the other three methods.

- 5.5.2.2 Sieve Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-422 will be used.

- 5.5.2.3 Hydrometer Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-422 will be used.

- 5.5.2.4 X-Ray Sedigraph Method

The Sedigraph procedure given by Micromeritics, Inc., Chicago, Illinois will be used. The equipment used for this test is the model of No. 5000D, by Micromeritics, Inc., Chicago, Illinois, which is capable of analyzing particle sizes ranging from 100 to 0.1 micrometers. The method is a non-destructive approach and a duplicate test for the same sample is possible.

5.5.3 Specific Gravity

- 5.5.3.1 Scope

The specific gravity of a soil is often used in relating the weight of the soil to its volume. Thus, knowing any three parameters among specific gravity, unit weight, moisture content, void ratio or porosity, and degree of saturation, the fourth one can be computed. These parameters are needed in nearly all problems in soil engineering. Several methods can be used to determine the specific gravity of a soil, but only the pycnometer method will be used. This method is basically simple, the result is reasonably accurate, and the test can be duplicated. Duplicate samples will be tested to secure reasonably accurate results. If the difference between two results are too great, a third test must be carried out.

- 5.5.3.2 Pycnometer Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-854 will be used.

A pycnometer having a capacity of at least 100 ml is suggested by ASTM. In order to increase accuracy and representative of the test, a pynometer of either 500 or 1000 ml capacity will be used.

5.5.4 Moisture Content Measurement

- 5.5.4.1 Scope

Among the most frequently determined soil characateristics is water content. The water content of a soil mass is defined as the ratio (usually as a percentage) of the weight of water to the weight of dry soil grains in the mass. Although the water content is one of the easiest properties of a soil to determine, it is also one of the most useful. For example, much research has shown that water content is a good indication of the shear strength of a saturated soil, and that the water content of a compacted soil is strongly related to the hydraulic conductivity of the soil.

A specimen for determination of water content should be weighed as soon as possible after it has been obtained to minimize the effect of drying. For some in-situ tests, such as the nuclear method, the test should be carried out immediately after the damp soil surface is exposed.

Both laboratory and field tests will be carried out. Duplicate tests will be conducted to secure reasonably accurate results. If the difference between the duplicates is too large, the third test will be carried out.

- 5.5.4.2 Laboratory Tests

Both the conventional oven method and the microwave method will be used. The advantages to using the microwave oven over the conventional oven is a substantial time savings and low purchase price. The microwave method is often used when fast turn-around is necessary, such as for the inspection of field compaction.

- 5.5.4.2.1 Conventional Oven Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2216 will be used.

- 5.5.4.2.2 Microwave Oven Method

The method suggested by Lade and Nejadi-Badadai (1976) will be the basic guideline used to develop the procedure for this project. Due to the complex heating process by microwaves, the nature of the action of microwaves, and the sensitivity of clay minerals to drying temperature, the amount of water driven off in the microwave oven may not correspond to the amount removed by evaporation in a conventional oven at a given temperture. Water contents determined using microwave oven are often higher than those determined using a conventional oven, especially for cohesive soils. It was found that the differences between the two methods decrease with decreasing plasticity of the soil. Therefore, a systematic correlation between the two methods for testing of the liner materials must be determined.

The type of microwave oven to be used for the test has not been decided. Calibration and correlation with different power ranges of microwaves will be an integral step before a final procedure is established. A large number of tests will be carried out for both methods. The results will then be statistically analyzed in order to develop empirical equations to be used for field tests.

- 5.5.4.3 Field Tests

Field tests are generally required to determine if the specification, especially for compaction, has been met. Both destructive and nondestructive methods will be used. The destructive method will be either by Speedy

moisture tester (by chemical reaction) or by the microwave method as described above. The nondestructive method to be used is nuclear radiation. The amount of neutron loss is related to the amount of hydrogen atoms present, which in turn is related to the water content. Both Seaman and Troxler instruments have been obtained and will be used for the project.

- 5.5.4.3.1 Nuclear Method
- 5.5.4.3.1.1 Seaman Method:

The standard procedure suggested by ASTM (American Society for Testing Materials) D-3017 will be used. Similar procedures are given by Seaman Nuclear Corporation, Oak Creek, Wisconsin.

- 5.5.4.3.1.2 Troxler Method:

The testing procedure suggested by Troxler Laboratories, Raleigh, North Carolina, will be used. At present, only a downhole model is available for the project.

- 5.5.4.3.2 Speedy Moisture Tester

The standard procedure suggested by AASHTO (American Association of State Highway and Transportation Officials) T-217 will be used. Similar procedures are also given by Soiltest, Inc. Evanston, Illinois.

5.5.5 In-Situ Density Measurement

- 5.5.5.1 Scope

Field tests are generally required to determine if the specification, especially for compaction, has been met. Both destructive and nondestructive methods will be used. The destructive method will be one or more of the drive

cylinder method, rubber balloon method, and sand cone method. The nondestructive method is using nuclear radiation. The amount of gamma ray detected is related to the unit weight of the soil mass. Both Seaman and Troxler instruments have been equiped and will be used for the project.

• 5.5.5.2 Nuclear Method

- 5.5.5.2.1 Seaman Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2922 will be used. Similar procedures are given by Seaman Nuclear Corporation, Oak Creek, Wisconsin.

- 5.5.5.2.2 Troxler Method

The testing procedure suggested by Troxler Laboratories will be used. At present, only a downhole model is available for the project.

• 5.5.5.3 Drive Cylinder Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2937 will be used.

• 5.5.5.4 Rubber Balloon Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-2167 will be used.

• 5.5.5.5 Sand Cone Method

The standard procedure suggested by ASTM (American Society for Testing Materials) D-1556 will be used.

5.5.6 Atterberg Limits Test

- 5.5.6.1 Scope

Atterberg limits results are normally used for qualitative purposes. They provide a basis for the classification and identification of fine-grained soils. It may be also implied for limited quantitative intentions, especially when there is a need for the engineering properties in a very short time. This assumption may be valid in most cases because

> "the chemical and mineral composition, size, and shape of the soil particles influence the adsorbed water films on the particles. Because such soil properties as compressibility, permeability, and strength, as well as the limits, are dependent on the water films, approximate relationships exist between these properties and the limits. .....
>
> For a given soil, we can often set up definite semi-empirical relationships between a property and the limits or indices. From such expressions, we can then make predictions of the properties of another sample soil by knowing the limits. Such a procedure is often very helpful because the limits are usually more easily determined than the compressibility, permeability, or strength. .....

> They are also often used directly in specifications for controlling soil for use in fill and in semi-empirical methods of design. .....
>
> .....even though the limits do not furnish numbers which can be substituted directly into scientifically derived formula, they are extremely useful to the soil engineers." (Lambe, 1962)

- 5.5.6.2 Liquid Limit

The standard procedure suggested by ASTM (American Society for Testing Materials) D-423 will be used.

- 5.5.6.3 Plastic Limit and Plasticity Index

The standard procedure suggested by ASTM (American Society for Testing Materials) D-424 will be used.

- 5.5.6.4 Shrinkage Factor

The standard procedure suggested by ASTM (American Society for Testing Materials) D-427 will be used.

5.6 Compaction Characteristics

5.6.1 Scope

The purpose of a laboratory compaction test is to determine the proper amount of mixing water to use when compacting the soil in the field and the resulting degree of density which can be expected from compaction at this optimum water content. To accomplish this, a laboratory test which will give

a degree of compaction comparable to that obtained by field procedures is necessary. Because a variety of compaction equipment and procedures which produce different degrees of compaction have been developed, it is necessary to employ different amounts of compacting energy in the laboratory test. At least three compaction methods with different compacting energies will be used in this project. They are the (1) Standard Proctor compaction method, (2) Modified Proctor compaction method, and (3) Harvard Miniature compaction method.

5.6.2 Standard Proctor Compaction

The standard procedure suggested by ASTM (American Society for Testing Materials) D-698 will be used.

5.6.3 Modified Proctor Compaction

The standard procedure suggested by ASTM (American Soceity for testing Materials) D-1557 will be used.

5.6.4 Harvard Miniature Compaction

This is a small scale compaction test which employ a mold of 1 5/16-in inside diameter and 2.816-in long for a total volume of 1/454 cubic foot. This method is believed to more closely approximate the action of sheepsfoot roller. Different compaction effort for the method can be achieved by selecting the most appropriate number of layers, number of tamps per layer and the tamping force. There is no ASTM standard procedure for this method. The procedure given by SOILTEST, INC. (1970) and Wilson (1950) will be used as a the guideline to develop this testing procedure.

5.7 Soil Dispersion/Erodability

5.7.1 Scope

It is often desirable to have less dispersive or erodible soil as a liner material. Certain fine-grained soils in nature are highly erodible and dispersive. These soils erode by a process in which the individual collidal clay particles readily go into suspension, whereas the erosion process for ordinary clays is quite different, requiring considerable velocity in the eroding water. Two laboratory tests for identifying dispersive or erodible soils will be used as described in the following sections.

5.7.2 Double Hydrometer Method

This method is also called SCS (Soil Conservation Service) Laboratory Dispersion Test, which has been widely used since 1940. The particle size distribution of a soil is first measured using the standard hydrometer test (ASTM D-422), in which the sample is dispersed in the hydrometer bath with strong mechanical agitation and a chemical dispersant. A second hydrometer test is made without strong mechanical agitation and without a chemical dispersant. By definition, 'percent dispersion' is the ratio of the amount of clay sized particles of 5 micrometers measured in the two tests. The test procedure which will be used is described in detail by Sherard et al. (1972).

5.7.3 Pin Hole Test

This test is quite simple. Distilled water flows through a 1.0 mm diameter hole made in a compacted soil (using the Harvard miniature compaction method). For dispersive soils, the water rapidly becomes cloudy and the hole

quickly erodes. For nondispersive soils, the water is clear and there is no erosion. The detailed test procedure is given by Sherard et al. (1976).

5.8 Laboratory Hydraulic Conductivity

5.8.1 Triaxial Cell Test

Hydraulic conductivity tests will be performed in the laboratory using the Triaxial test. The testing system (figure 5.1) manufactured by Wykeham Farrance Inc. will be used for permeability tests. The principal features of the system, the sample preparation method and the testng procedures are discussed below.

Three principal features are necessary for the hydraulic conductivity test. They are (1) Triaxial cell apparatus, (2) Bishop type self compensating mercury control apparatus, and (3) Twin burette cell volume change apparatus.

- 5.8.1.1 Triaxial cell apparatus

The Triaxial cell is a standard low pressure cell. As shown in the Figure 2, the major parts are the basal plate with sample pedestal, the removable cylinder and top plate, the cap, and the rubber membrane. The cell can accommodate 1.5-4.0-inch diameter samples. However, only 2.0-inch diameter samples will be used for this project. The basal plate is equipped with five draining ducts each of which leads to a non-volume change valve. Two of these drains lead vertically into the base of the sample through sample pedestal; one is used for applying back-pressure while the other is used for measuring the amount of outflow. A third drain is the duct for the confining fluid and pressure. The remaining two drains, which are connectd to the cap, are used to apply back-pressure and to impose the head of water during actual hydraulic conductivity testing.

A 6.0-inch diameter transparent plexiglass cylinder, which facilitates the testing of different sized samples (from 1.5 to 4.0 inch diameter), will be used. The top plate, as shown in Figure 2, is equipped with a draining valve and duct for confining liquid or for air leasing.

The cap is made of plexiglass and completed with two drain holes to be connected to the basal plate for applying back-pressure and for imposing a water head during testing.

A rubber membrane 0.010 - 0.015 inch thick and approximate 6.0 inches long will be used. The membrane will be sealed against the cap and pedestal by rubber O-rings. The O-rings will have an internal diameter of 1.75 inches.

5.8.2. Samples Preparation for Triaxial Test

• 5.8.2.1 General Description

The samples will be prepared in a humid room, with every reasonable precaution taken to minimize drying and disturbance of the soil. The sample size used for this project will be 4.5 inch high by 2 inch in diameter.

• 5.8.2.2 Sample Trimming Procedures

A sample approximately 5.5 inch in length and 2.5 inch in width or diameter will be cut from undisturbed block sample, shelby tube sample, or Proctor compacted sample. A miter box and wire saw (or knife) will be used to trim enough soil and to make the ends parallel. The sample will then be placed in the lathe or the trimmer, (e.g., WF32560 model by Wykeha Farrance Inc.) and trimmed to a circular cross section. The sample will again be placed in the miter box and cut it to a length of approximately 4.5 inch by trimming both ends. From the trimmings, three representative sub-samples for

water content measurement will be obtained. These subsamples will be taken from the center of the core and from near each end.

Sample volume and density will be calculated from measurements of sample length, circumference and mass.

5.8.3 Testing Procedures for Triaxial Cell

There are three major steps in the testing procedure. They are: (1) sample set-up in the triaxial cell, (2) back pressure saturation, and (3) permeability testing. Since no ASTM standards exist for these procedures, they are outlined in detail below.

• 5.8.3.1. Sample Set-up in the Triaxial Cell

(1) In the humid room, the sample is enclosed within the rubber membrane by using a membrane stretcher.

(2) The basal plate, sample pedestal and line connecting the basal place to the burettes are de-aired by fluship with de-aired water. A porous stone, which has been boiled in water to remove the air, is then placed at the top of the sample pedestal.

(3) Water is allowed to run out of the sample pedestal slowly to prevent entrapping air. Filter paper separates the sample from the bottom porous stone. Next the membrane is moistened, overlapped, and bound to the sample pedestal with three or four O-rings.

(4) The cap with the upper porous stone and filter paper on the top of the sample. During this step, water should be running slowly through the cap and porous stone to prevent entrapping air. As with the basal plate, the cap and line connecting the cap to the burette will be de-aired.

(5) The upper end of the membrane moistened, rolled and bound (by O-rings) to the sides of the cap.

(6) Vertical rods are screwed into the basal plate.

(7) Next the bottom rubber gasket is wet and the plexiglass cyliber centered on it. The upper rubber gasket is also wet and placed on the top of the cylinder.

(8) The upper plate is placed tightened with the top nuts of the bertical rods.

(9) Water is added to the cell chamber by opening valve E_2 and air releasing valve I (see figure 5.1). The water level is brought up to the top plate, then both valves are closed. Care must be taken to that no aire is allowed into the chamber.

(10) The desired confining chamber pressure is set by adjusting the self Compensating Mercury Control and the regulator. The confining chamber pressure slowly built up by cracking valve screw control cylinder.

• 5.8.3.2 Procedure of Back Pressure Saturation

(1) After cell chamber has been filled with water, and the desired confining chamber pressure has been built up, both the chamber pressure line and the back pressure line are connected to a same self compensative mercury control (see figure 5.1). In such an arrangement, both the chamber pressure and the back pressure can be raised simultaneously.

(2) The pressure will be raised stepwise. An increment of 10 psi with five to ten minutes settling time between pressure increases will be used.

(3) The required back pressure depends on the initial degree of saturation of the sample. The theoretical required back pressure calculated by Bishop and Henkel (1962), as given in Table 5.1, will be followed.

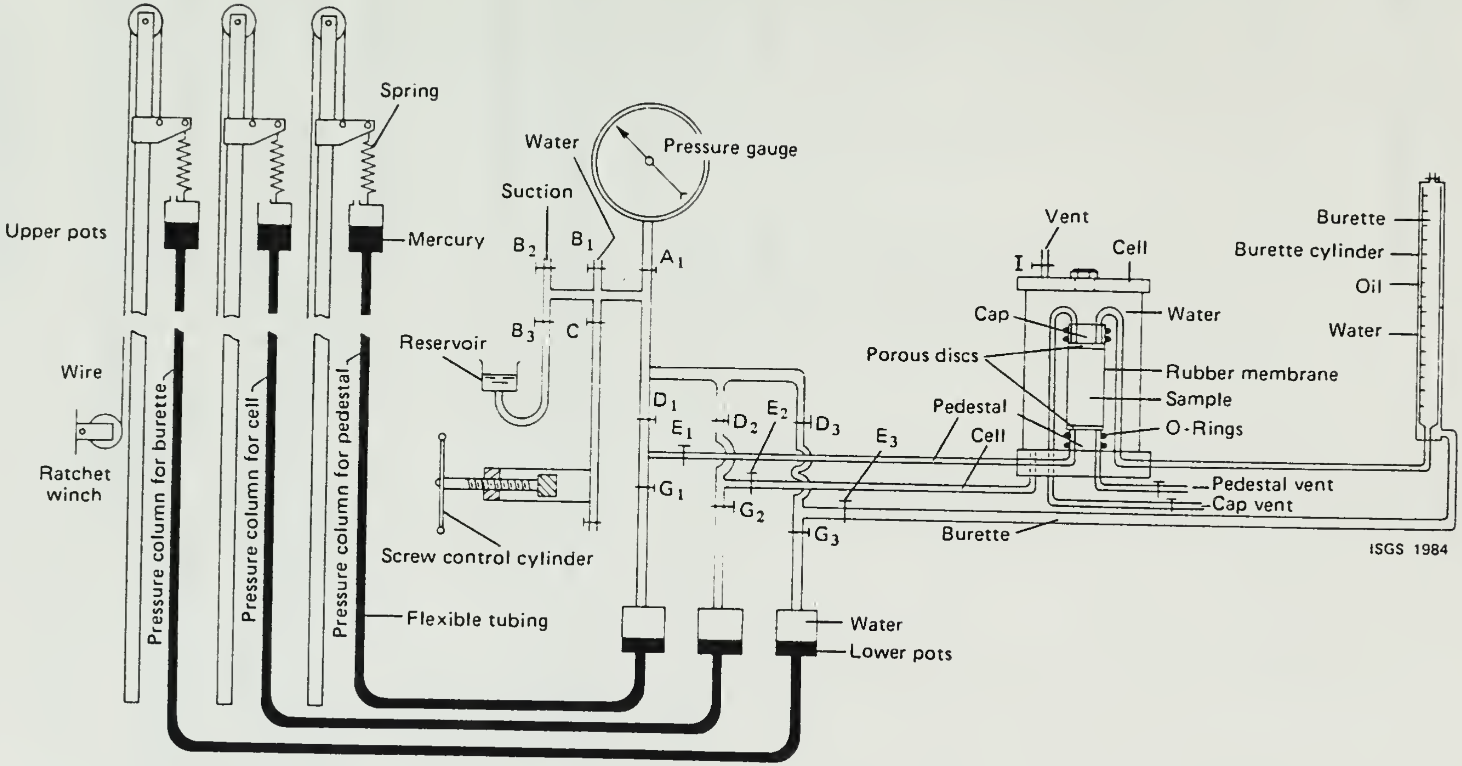

Figure 5.1. Schematic diagram of triaxial cell apparatus.

TABLE 1. Theoretical Required Back Pressure

Initial degree of saturation of sample, %	Back Pressure, Psi
95	36
90	72
85	108
80	144
75	180
70	216

(4) After the desired back pressure has been reached the sample is allowed to sit overnight.

• 5.8.3.3 Permeability Test

The flow rate through the sample in a triaxial cell depends on the combination of both consolidation and seepage. Therefore, a consolidation test will be carried out before the hydraulic conductivity test in order to ensure the sample has been fully consolidated.

- 5.8.3.3.1 Consolidation Test

(1) Close both back pressure line and cap drainage line.

(2) Raise the cell pressure to desired value.

(3) Open cap drainage line and strat timing simultaneously.

(4) Take readings of volume change from burette at 0, 1/4, 1/2, 1, 2 1/2, 4, 6 1/4 --- hours until volume stabilizes.

(5) Prepare Δv vs. 1/2t (volume change vs. square root of elaspe time) Curve.

(6) Use Δv vs t curve to determine t_{100}.

(7) Determine Cv from equation

$$Cv = \frac{2}{t_{100}}$$

where 2H is the height of sample.

- 5.8.3.3.2 Permeability Test

(1) Close cap drainage line.

(2) Raise backpressure line to desired pressure until a gradient of 5-20 is superimposed.

(3) Open cap drainage line and start timing.

(4) Prepare Q vs t (Quantity of flow vs time) curve. If no major error exists this should be a straight line.

(5) Determine hydraulic conductivity K by equation

$$K = \frac{QL}{thA}$$

where Q: quantity of flow
L: length of sample
t: time
h: pressure gradient
A: cross section area of sample

- 5.8.3.3.3 Multiple Consolidation and Permeability Test

(1) After step 5.8.3.3.2.(5) has been finished, close both backpressure line and cap drainage line.

(2) Increase cell pressure to desired value.

(3) Repeat step 5.8.3.3.1. (3)-(7) of the consolidation test.

(4) Repeat step 5.8.3.3.2. (1)-(5) of the hydraulic conductivity test.

(5) Repeat step (1)-(4) until the greatest desired cell pressure is reached.

5.8.4 Harvard-Core Minature Permeameter Tests

Laboratory tests of hydraulic conductivity for this study will also be conducted using a falling head permeameter that accommodates small-diameter Harvard-type cores (modified from Soiltest, Inc., Model K-620). This test is described in detail here because no standard method exists. This device is shown schematically in Figure 5.2.

- 5.8.4.1 Description of Apparatus

The hydraulic gradient is increased by applying air pressure to a pressure tank containing water. A flexible membrane is used to separate air and water in the tank. The pressure can be manipulated by adjusting the air pressure, or by adding or deleting water. The Harvard-cell sample mold is a stainless steel cyliner, open at each end, with a height of 7.16 cm and a diameter of 3.33 cm. The sample molds, base, and cap of the permeameter units, porous stones, and clamps were obtained from a Soiltest Inc., K-620 Permeameter. The base contains the inlet port and the porous stone through which water is introduced to the bottom of the sample. The cap contains the drain port and holds the manometer. The cylindrical sample mold is held

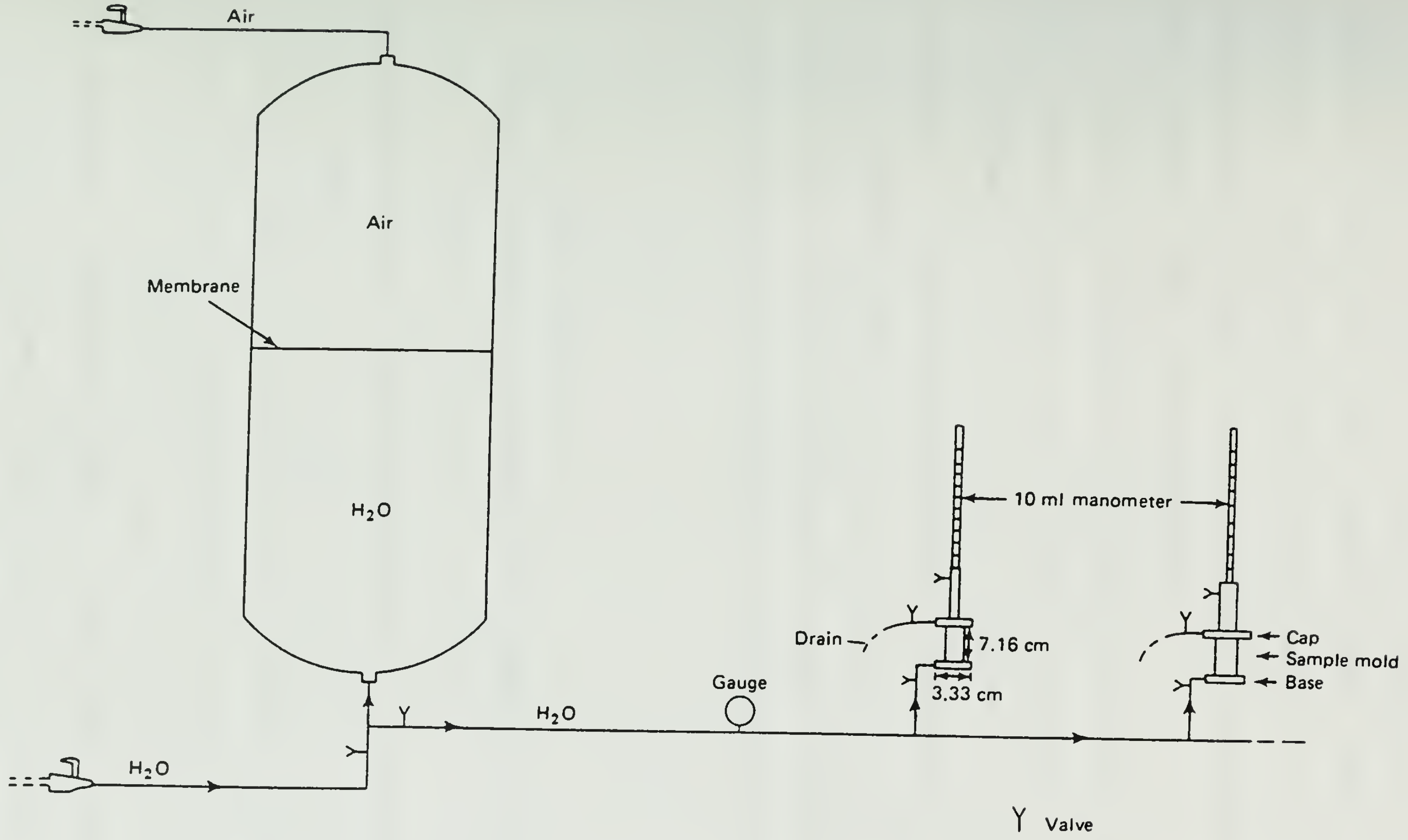

Figure 5.2. Schematic diagram of miniature permeameter for falling head tests.

firmly between the base and the cap by two bolts. A seal is achieved by using a rubber gasket at each end of the mold. A filter paper and a screen are placed at the top of the sample and filter paper overlies the porous stone at the bottom. During saturation and actual testing of the sample, water flows upward through the sample into the vertical 10 ml manometer. The upper end of the manometer is open to the atmosphere.

The pressure supplied to the sample from the tank is measured with a gauge on the inlet water line at the base of the sample. The tailwater head is the distance, in cm, from the top of the sample to the water level in the manometer. The actual gradient applied to the sample at a given time is the pressure head corresponding to the pressure on the inlet gauge minus the tailwater head. Since the inlet pressure remains constant, the change in tailwater head as the test progresses is the decrease in head or the falling head.

• 5.8.4.2 Sample Compaction

Prior to recompaction, samples are mechanically crumbled so that the silt and clay aggregates are reduced to less than 2 mm in diameter. The smples are then passed through a 2-mm sieve to remove all gravel-sized particles. The interior surface of the permeameter is coated with silicon high-vacuum grease to eliminate channeling along the walls of the mold. The sample is placed in the mold in five lifts and excess material is trimmed from the top. The top of each lift is scarified prior to placement of each subsequent lift. Samples are compacted in the mold by hand, by using a solid steel cylinder that was slightly smaller in diameter than the mold. The dry density of each sample is calculated after the completion of each test to determine compaction.

5.8.4.3 Test Procedure

The mold and sample are placed in the permeameter apparatus; a filter paper are placed on each end of the sample and a screen is placed on the top. Tap water is added to the sample under pressure until water flowed through the discharge line. For the more fine-grained samples, it may take several days for discharge to appear. A vacuum line is then attached to the discharge line in an attempt to remove entrapped and dissolved air from the sample. Vacuum is applied to each sample until saturation, when no more air is observed to be drawn from the sample. The test will begin no sooner than an hour after the vacuum is disconnected.

At least two samples from each bulk sample collected will be tested in the Harvard-type minature permeameter apparatus. Approximately fifteen bulk samples from five outcrops representing at least three material types will be tested before choosing the material to be used for the liner.

5.8.4.4 Hydraulic Conductivity Calculation

Hydraulic conductivity was determined using the equation:

$$K = \frac{aL}{AT} \ln(H_i/H_f)$$

where:

K = hydraulic conductivity (cm/s)

a = cross-sectional area of the manometer (cm^2)

L = length of the sample (cm)

A = cross-sectional area of the sample (cm^2)

T = time (s)

H_1 = initial pressure head = $P-H_1$ (cm)

H_f = Final pressure head = $P-H_2$ (cm)

P = pressure head from the pressure source (cm)

H_1 = initial tailwater head (cm)

H_2 = Final tailwater head (cm)

5.9. Unsaturated Flow Parameters

5.9.1 Moisture Retention Characteristics

Standard equilibrium stepwise pressure desorption methods (Klute, 1965; ASTM D2325-68; ASTM D3152-72) will probably be used to measure the moisture retention characteristic ($\theta(\psi)$) of the clay liner material. Tempe cells and pressure chamber equipment are already available. Precision should be within 0.1%.

A one-step outflow method (Kool et al., 1985) will be evaluated as an alternative to the stepwise desorption. The method is discussed in the following section on hydraulic conductivity.

The feasibility of determining the adsorption relation as input into a hysteresis model will also be evaluated. Equipment modifications will be necessary if adsorption is desired.

5.9.2 Hydraulic Conductivity and Diffusivity

Methods for assessing the hydraulic conductivity ($K(\psi)$ and $K(\theta)$) and hydraulic diffusivity ($D(\theta)$) of the clay liner will be reviewed. Three basic approaches to determine hydraulic conductivity are (1) steady-state methods, (2) unsteady or transient methods and (3) calculation methods. Roberts (1984) reviewed these three types of methods in detail. Diffusivity is conventionally determined by the pressure outflow method.

Daniel (1983) and Hamilton et al. (1981) described an unsteady method (instantaneous profile) for determining unsaturated hydraulic conductivity. It was modified specifically for compacted clay liner materials by adding psychrometers to increase the range if tensions which could be measured. Results suggest that this test may be a promising one.

Another method which will be considered is described by Kool et al. (1985 a,b) and Parker et al. (1985). Water retention and hydraulic conductivity functions (and hydraulic diffusivity) are determined simultaneously from one-step pressure outflow experiments on soil cores. The $K(\psi)$ function is obtained by a parameter estimation procedure. An attractive feature of this method is the much shorter testing time than conventional step-wise methods. This is especially important for compacted clays which take a long time to equilibrate at each pressure step. Extension of this one-step parameter estimation method to adsorption experiments to characterize hysteretic behavior is currently being studied by its authors. Hysteresis has never been evaluated for compacted clays, however its role in governing flow and transport through a liner is probably significant. An attempt will be made in the ISGS study to evaluate hysteresis. Experimental and numerical problems are anticipated due to the unique characteristics of compacted clays.

5.10 Performance Monitoring and Transit Times

After construction, the field-scale liner will be flooded with water and a series of different tracers will be applied at staggered time intervals. Transit times of both water and solutes through the liner will be measured directly. This will be accomplished by instrumenting the liner to collect data on moisture and tracer movement. Moisture movement through the liner will be assessed by measuring infiltration-rates, soil-water matric

potentials, soil moisture contents and drainage. Solute movement through the liner will be assessed by measuring solute concentrations in soil-water samples drawn from suction-lysimeters installed in the liner profile and from the drainage effluent. Water and solute monitoring methods are described in the next sections.

5.10.1 Indirect Field Methods for Determination of Soil Moisture

• 5.10.1.1 Soil Cores

Statistically representative soil samples will be periodically taken using shelby tube samplers as discussed in Section 5.1. Soil moisture contents will be determined gravimetrically from these cores by subjecting them to oven drying at 105°C for 24 hours. Moisture contents will be compared to results obtained from tensiometers, tensiometer/piezometers, and soil moisture blocks.

• 5.10.1.2 Soil Moisture Resistance Blocks

Soil moisture resistance blocks are used to measure soil matric suction as a function of resistance between two electrodes in a porous block.

Soil salinity, texture and temperature are assumed constant. Delmhorst Model 227 cylindrical soil moisture blocks will be used. These gypsum blocks have an effective measurement range of 100-15,300 cm H_2O and are buffered against salts. Calibration curves will be determined for representative blocks by measuring the extraction of soil pore water in the laboratory by pressure plate apparatus.

Use Agronomy Monograph 9 part 1

• 5.10.1.3 Tensiometers

Tensoimeters will be used to measure in situ matric suction in the trench liner. They will be individually constructed of porous ceramic cups, nylon tubing and brass pressure fittings. Each tensiometer will be connected to a Micro Switch piezometric differential pressure transducer rated at either 0-5 psi (0-352 cm H_2O) or 0-15 psi (0-1,055 cm H_2O), and referenced to atmospheric pressure. The 0-5 psi transducers will allow better resolution of soil matric suction in the higher saturation range of less than 300 cm H_2O, while the 0-15 psi transducers will allow measurement of early pre-wetting front conditions with higher soil tensions.

• 5.10.1.4 Operation

As equilibrium between fluid in the tensiometer and soil is achieved, a subatmospheric pressure (matric suction) is measured by a pressure transducer. As water content increases, this vacuum is reduced by the influx of water into the tensiometer through the porous ceramic cup. When saturation is reached, a zero value (equivalent to atmospheric pressure) is recorded.

• 5.10.1.5 Tensiometer/Piezometers

These instruments will be constructed similarly to the tensiometers, except that they will be connected to Micro Switch pressure transducers rated from -3 to +3 psi, allowing measurement of soil matric suction values to -211 cm H_2O and positive pressure head up to +211 cm H_2O. They will be able to measure the range from low values of matric suction through saturation and into the positive pressure head range as a wetting front advances through the soil material.

- 5.10.1.6 Calibration

Calibration of a representative number of transducers used with tensiometers and tensiometer/piesometers will by conducted by measuring the extraction of soil pore water in the laboratory using a pressure plate apparatus.

- 5.10.1.7 Measurements

Installation of multiple tensiometers, soil moisture resistance blocks and tensiometer/piezometers will allow the calculation of the vertical hydraulic gradiant (i) as:

$$i = [(S_{n+1} + d_{n+1})-(S_n + D_n)]/(d_{n+1} - d_n)$$

where $S_1, S_2, S_3,...S_n$ = matric suction values in cm H_2O

$d_1, d_2, d_3,...d_n$ = instrument depth in cm

In-situ matric suction values will be converted to soil moisture content using soil moisture characteristic curves developed from laboratory analyses.

- 5.10.1.8 Data Collection

Near-continuous data collection will be accomplished using Campbell Scientific CR21X dataloggers in conjunction with AM32 input multiplexers. The CR21X is capable of providing power to the entire electronic measurement system at predetermined intervals and recording the resulting output data. All output data are recorded digitally on magnetic tape. The monitoring instruments will be powered for no less than one full minute prior to taking a measurement so that signal stabilization occurs.

• 5.10.1.9 Data Recovery

Data recorded on magnetic tape will be periodically retrieved by field personnel and returned to the office. Special software and hardware obtained from Campbell Scientific will allow direct transfer of data to an IBM PC computer for processing. Spot checks will be performed on 1-5 percent of the stored data to insure that field parameters are reliable.

• 5.10.1.10 Maintenence

Periodic changing of magnetic tapes will be required weekly. Tensiometers will be visually inspected for leaks and will be flushed and refilled with deaired water as required. This becomes necessary because cavitation can occur at soil matric suction values of greater than approximately 816 cm H_2O.

Field personnel will periodically check electronic systems for failures of transducers or soil moisture blocks as well as for battery replacement.

• 5.10.1.11 Replication

Each instrument will have at least 2 replicates (at the same depth) to insure accurate results. The CR21X datalogger system will allow monitoring of numerous instruments with a miniumum of maintenence.

5.10.2 In-Situ Infiltration Measurements

The conventional double-ring ponded infiltration method will be used to measure the infiltration rate in-situ (Boersma, 1965). Infiltration rates will be used in unsaturated flow models to predict transit time of water movement through the lines.

5.10.3 Resident Solute Concentrations

Nests of small porous-cup soil-water suction samplers (lysimeters) will be installed at various depths in the liner. Soil-solution samples will be collected at regular time intervals using conventional methods (Everett, Wilson and Hoylman, 1984). Solute concentrations measured at known locations and times will be used in unsaturated transport models to predict transit time of solutes through the liner.

5.10.4 Flux at Liner Bottom and Flux Averaged Solute Concentrations

The field-scale liner will be installed on a base which will allow the collection of drainage (a gravel layer, geocomposite, or combination of the two). Drainage from this layer will be quantified and analyzed for tracer concentration. Direct measurement of transt times of water and solutes through the liner will be made.

5.11 Dual-Energy Gamma-Ray Column Experiments

A dual-energy, gamma-ray system including 300 mCi of 60 KeV Am^{241} and 300 mCi of 660 KeV Cs^{137} will be used as a nondestructive method to simultaneously measure moisture content and bulk density of a porous medium during column experiments of moisture transport.

5.11.1 Recalibration

The dual-energy gamma-ray system owned by the ISGS has been used in the past for moisture transport studies (Johnson, et al., 1983). Prior to beginning a new experiment, the system will be recalibrated. One-minute single energy counts will be made using each isotope independently. Standard target materials (water and aluminum) will be varied in thickness from zero to

ten centimeters in one centimeter increments. This test will provide baseline values for Am energy transmission in the low energy detection window. Results will also be used to optimize column thickness for peak Am energy transmission. The same test will be repeated in the dual-energy mode after replacing both isotopes in the apparatus. The difference in the low energy spectra between the single-source Am test and the dual-source test will provide an indication of energy downscatter from the high energy Cs isotope. Results from this comparison will be used to compute a downscatter correction to Am counts.

5.11.2 Attenuation Constants

Calibration columns will be packed at known moisture and density with separate soil components to be evaluated in this study. Replicate runs on each soil material will be made to determine an attenuation constant for that material. The attenuation factor should be a constant value, independant of moisture and density.

5.11.3 Column Experiments

Each soil column will include a reference segment containing an aluminum standard block. Each pass of the gamma-ray beam through the column will include the reference segment and a blank segment. These counts will be used to compensate for system drift, decay of the isotopes and variations in laboratory conditions.

Initial bulk density and moisture content values will be determined gravimetrically during construction of each column. These initial values will be used for off-set corrections to be applied to subsequent moisture and

density determinations made radiometrically to obtain absolute values from initially relative measurements.

Miniature porous cup tensiometers will be inserted into the columns at various points to obtain independent measurements of moisture content. The tensiometers will be monitored at irregular intervals throughout the experiment.

Column tests will be run in replicate.

5.12 Geochemistry Contributions

5.12.1 Determination of Chemical Properties of Liner Materials

Upon selection on the basis of engineering and hydrologic properties, of an appropriate soil(s) for construction of the in-situ liner, chemical testing of the material will be performed. Statistically representative subsamples shall be collected and tested (see section for details on sampling strategies). The initial tests to determine the soils "chemical" suitability as a potential liner material will be; 1) cation exchange capacity (CEC), 2) anion exchange capacity (AEC), 3) total organic carbon (TOC), 4) 1:1 soil to water pH and electrical conductivity (EC), 5) water extractable and KCl extractable constituents, 6) batch type adsorption experiments using tracers such as Bromide (Br^-) and pentaflurobenzoic acid (PFBA). A brief outline of the test procedures, expected errors, and references where additional information can be obtained are given below.

• 5.12.1.1 Sample Preparation

All analyses or procedures carried out on the soil samples will be done using air-dried samples. To correct for the mass of water, the moisture content will be determined in triplicate separate subsamples. This will be accomplished gravimetrically by determining the mass difference between air-dried samples and samples subjected to oven-drying for 24 hrs at 105°C (Standard Method D2216-71, ASTM, 1972).

$$\frac{A-0}{0} \times 100 = \%\ \text{moisture}$$

where A = air-dried mass

0 = oven-dried mass

• 5.12.1.2 Soil Exchange Capacity

Cation exchange/anion exchange capacity of the soil(s) will be ascertained using the magnesium chloride percolation method described in Breimer and Leffelaar (1976). The procedure involves the percolation of a known concentration $MgCl_2$ solution through the soil. Ammonium nitrate is then percolated through the soil to remove adsorbed magnesium and chloride ions. The percolates from each of the steps are quantitatively collected and analyzed. Magnesium and Cl concentrations in the percolates will be determined by inductively coupled argon plasma (ICAP) emission spectroscopy and ion chromatography (IC), respectively. See section 5.12.4.1 for details concerning inorganic analysis. The CEC and AEC of the soil can then be calculated using the data generated by this procedure.

- 5.12.1.3 Total Organic Carbon

Total organic carbon (TOC) of the soil will be ascertained by a Dohrman DC-80 total organic carbon analyzer system with the sludge and sediment samples accessory. Detection of organic carbon is accomplished by combustion of the sample at 850°C in an oxygen atmosphere. The resulting CO_2 is quantified with a non-dispersive infra-red detector (NDIR).

- 5.12.1.4 Soil pH

The pH of the soil(s) will be ascertained by making a 1:1 (weight/volume) soil to deionized water mixture and determining the mixtures pH with a combination pH electrode and an Orion Model 801A digital ionalyzer. The electrical conductivity will be determined by collecting an aliquot of the water soil mixture before determination of the pH and filtering the aliquot through a 0.45 μm filter. The conductivity of the filtrate will be ascertained by a Yellow Springs Instrument Model 31 conductivity bridge.

- 5.12.1.5 Soil Extractables

The water extractable constituents will be ascertained using a 1:1 (weight/volume) soil to deionized water mixture. This mixture will be allowed to equilibrate and then filtered through a 0.45 μm filter. The most prevalent chemical constituents in the filtrate will be determined by ICAP and IC. The procedure for the KCl extractable constituents is identical to the above procedure except a 1M KCl solution instead of deionized water is used. Errors associated with these procedures would be predominantly related to the analysis of individual constituents in the filtrates, weighing of the material and measurement of the extractant. Cummulative errors are not expected to effect the data by more than ± 5%.

• 5.12.1.6 Adsorption Studies

Batch type adsorption experiments will be performed as outlined in Roy et al. (1985). In brief, the batch adsorption approach consists of mixing a solution containing aqueous solute(s) of known composition and concentrations with a given mass of soil, for a period of time. The solution is then separated from the soil and analyzed to determine changes in the solute concentration. Experimental variables such as temperature, equilibration time, soil to solution ratio and mixing method are addressed in the adsorption procedure.

5.12.2 Miscible Displacement and Microbial Degradation

Laboratory clay-column experiments will be conducted to determine if and how transit times of chemical tracers are affected by microbial activity. Aerobic and strict anaerobic methods will be employed on enriched material obtained from the Wilsonville, Illinois hazardous waste site. Growth and maintainance of strict anaerobes for seeding columns will be in a Coy Freter-type anaerobic chamber, using strict anaerobic techniques (Balch and Wolfe, 1976). Techniques will be used that represent actual ground water conditions beneath hazardous waste sites (temperature, pH, solution ionic strength and nutrient level). Columns will be designed with water jackets that will provide a constant temperature environment. The columns and water jackets will be constructed of polycarbonate cylinders with tops and bottoms of Delrin plastic.

A preliminary evaluation of microbial effects on liner porosity will be obtained by comparison of flow rates through innoculated laboratory columns versus the flow rate through sterile columns used as controls. All columns will first be sterilized by radiation (Cobalt 60), selected columns will then

be innoculated with microbial cultures while uninnoculated columns will be used as controls.

Chemical tracers will be introduced and water samples will be collected from various depths of the columns. Tracers to be used include: iodide (I^-), bromide (Br^-), m-trifluoromethyl benzoic acid (m-TFMBA), pentafluorobenzoic acid (PFBA), o-trifluoromethylbenzoic acid (o-TFMBA), 2,6-difluorobenzoic acid (2,6-DFBA), and tritiated water (HTO). The inorganic and organic tracers will be monitored by ion, gas and high pressure liquid chromatography. Tritiated water will be measured by radio assay using a Packard Tri-Carb 2000CA liquid scintillation analyze. Refer to section 4.0 for quality assurance procedures for these analytical methods.

It is anticipated that flow rates through the columns will be extremely slow (LeFairre, M. H., and G. Peyton, personal communication, Illinois State Water Survey). The regulated supply of nitrogen to the water supply tank into the column will maintain a high fixed pressure source on the column. The flow measurement device will be a pipet graduated in 0.01 ml increments. Flow rate will be determined by measuring the time required for the water/nitrogen interface to move from an upper to lower mark in an accumulator. A tracer injection port will be used to avoid disturbance of the solvent in the column.

Rate of flow will be determined by recording the length of time needed for a known volume of water to enter the column. Hydraulic gradient, I, will be calculated as follows:

$$I = (70 - 28)(P_1 - P_m)/L$$

where P_i is the pressure (lbs/in^2) for flow into the column, P_m is outflow pressure (0 psi) and L is the column's length. The constant (70 - 28) is used to convert from pounds per square inch to centimeters of water. Hydraulic

conductivity will be determined by the flow rate into and out of the columns and from the hydraulic gradient by solving Darcy's equation:

$$K = Q/(I*A)$$

where A is the cross-sectional area of the column.

Rate of flow will also be determined by collecting fractions of a specified number of drops coming out of the column. To avoid difficulties arising from differing sizes of drops, we will determine the volume of the collected drops from their weights in the collection vials. The difference in weights of vials before and after sample collection and knowing the collection time will allow the calculation of flow rate out of the column.

Collected samples, obtained from a number of columns simultaneously, will be used to assess the precision of our system. Parameters of data aquisition will be determined as laboratory columns are set up. Preliminarily, we will determine four values for each column; flow rates into and out of the columns, and hydraulic head before entering and after exiting the column. Effective porosity of the columns will be determined by analysis of break-through curves derived from effluent collected in scintillation vials.

5.12.3 Routine Quality Assurance Practices

Sample containers, preservation techniques and holding times will be selected to minimize contamination and losses of the analyte(s) of interest. Test equipment shall be selected based on their compatability with the procedures used throughout this study.

Recommendations regarding sample containers, preservation techniques and holding times given in Methods for Chemical Analysis of Water and Wastes (U.S. EPA, 1975) will generally be followed.

Blanks will be carried out for all procedures used in this study. The use of blanks will minimize the generation of spurious data due to contamination, unknown, and known losses of test analytes and incompatabilities between laboratory materials and the test analytes.

Reagent grade chemicals and type IV deionized water (U.S. EPA, 1979) will be used in all test procedures.

5.12.4 Analytical Procedures

• 5.12.4.1 Inorganic Analysis

Test solutions generated from the determination of soil property, procedures, and the miscible displacement studies will be analyzed via an inductively coupled argon plasma (ICAP) emission spectrometer and/or ion chromatography. The ICAP is capable of simultaneously analyzing for 24 elements (Table 1) in aqueous solutions. Ion chromatography will be utilized for those anions not analyzable via ICAP; i.e. Br, Cl, F, NO_3, SO_4.

- 5.12.4.1.1. ICAP Emission Spectrometry

A Jarrell-Ash Model 975 Plasma AtomComp will be employed for analysis of inorganic constituents (Table 5.2). This system incorporates a DEC PDP-8A central processing unit which performs automatic background correction of individual spectral lines and interelement correction as required.

To insure that the data generated by the Environmental Geology Unit of the Geochemistry Section of the ISGS are reliable and reproducible, several quality control guidelines have been established. These are the quality of water, reagents, standard stock solutions and calibration standard solutions used.

Table 5.2. ICAP analyzable elements.

Constituent	Detection Limit	Constituent	Detection Limit
Al	.05	Mg	.003
As	.05	Mn	.01
B	.002	Mo	.01
Ba	.001	Na	1.42
Be	.002	Ni	.01
Ca	.01	Pb	.03
Cd	.01	Sb	.03
Co	.01	Se	.05
Cr	.03	Si	.03
Cu	.01	Sn	.11
Fe	.03	V	.04
K	1.03	Zn	.01

For the preparation of standards ultra high purity grade reagents will be used. Standard stock solutions are purchased from Spex Industries, Inc. The stock solutions will be prepared in a 5% ultra pure HNO_3 matrix. Typically the stock solutions are multicomponent having concentrations of 100 mg/L. These standards will be periodically checked against EPA quality control samples to determine their stability. Appropriate dilutions of the stock solutions, using the same matrix, will be made daily and used to calibrate the ICAP.

Reference trace metal and ICAP quality control samples obtained from the U.S. EPA Quality Assurance Branch and Environmental Resources Associates will be analyzed before, during, and after sample analysis to check calibration standards and instrument drift. If agreement between ICAP results and the expected values or established control limits are not within ± 5%, analysis is terminated and corrective action and instrument recalibration are initiated (U.S. EPA, 1980). Instrument calibration will be checked every 5 to 10 samples. If the ICAP results are not within ± 3% of the expected calibration

standard values, the instrument will be recalibrated. If recalibration does not result in values within the ± 3% range other corrective actions are implemented. To check instrument and background correction factors, reference samples spiked with known interfering elements will be analyzed monthly. Each sample is analyzed 2 to 3 times and a mean value is reported. However, for each analysis the ICAP measures 2 burns. From these six results the data processing unit calculates a coefficient of variation (CV). If the CV is greater than 5% the sample is reanalyzed.

- 5.12.4.1.2 Ion Chromatography

A Dionex Model 2110i ion chromatograph (IC) and a Hewlett-Packard 3390A Integrator will be used to analyze Br, Cl, F, NO_3, and SO_4. The reagents, standards, and quality control assessments for the ion chromatograph are similar to those of the ICAP.

Type IV water will be used to make all standards, dilutions, and test solutions. Reagent grade compounds of the potassium salts will be used to make up all standards. These standards will be periodcally checked against EPA reference samples to determine their stability. Mixed calibration standard solutions will be prepared from stock standard solutions. These will be prepared weekly to ensure stability. Reference mineral and nutrient quality control samples obtained from the U.S. EPA Quality Assurance Branch and Environmental Resources Associates will be analyzed before, during, and after sample analysis to check calibration standards and instrument drift. If agreement between IC results and expected values are not within ± 5%, analysis is terminated and corrective action is instituted. Generally, detection limits for all constitutents are 0.005 mg/L. However, using a concentrator column and larger injection loops detection limits may be improved to 0.001

mg/L. A coefficient of variation (CV) is determined by mulitple consecutive analysis (3) of the same sample. If the CV is greater than 10% corrective action is taken.

- 5.12.4.1.3 Radio Assay - Liquid Scintillation Analysis

A Packard Tri-Carb model 2000CA liquid scintillation analyzer will be used to analyze tritated water. The instrument incorporates computerized spectrum analysis, an efficiency tracing technique to eliminate quenching and luminescence correction. Tritium standards will be analyzed during and after samples analyses to check calibration standards and instrument drift. If agreement between tritium standards and analyzer results are not within ± 5% analyses is terminated and correction action and instrument recalibration are initiated.

• 5.12.4.2 Organic Analysis

Test solutions generated by the miscible displacement studies will be analyzed for organic constituents using gas chromatography and high pressure liquid chromatography.

- 5.12.4.2.1 Gas Chromatography

A varion 3500 capillary gas chromatograph equipped with ion trap and electron capture detectors will be used in this study. Analysis procedures, when possible, will follow U.S. EPA prescribed procedures (U.S. EPA, 1979). Gas chromatography will be used to ascertain possible nonpolar degradation products of the anionic organic tracers listed in section 5.12.2.

Type IV water or ultra pure solvents will be used to make all standards, dilutions, and test solutions. Reagent grade or ultra high purity compounds will be used to make all standards.

Calibration checks of the instrumentation will be an integral step in sample analysis to ensure the accuracy and reliability of the data. Calibration standards will be prepared from commercially available analyte solutions (the concentrations being certified). The GC systems will be checked with the test compound at the beginning of each day and at intervals of every five samples. The response factors obtained for the calibration replicates will be averaged and used to calculate the amounts of analyte in the samples. If any of the response factors, retention times, or the internal standard peak areas are outside of the limits established at the beginning of the analyses, the GC system will be checked for problems and the instrument recalibrated.

A quality control (QC) program will be established. The minimum requirements of this program consist of an initial demonstration of laboratory capability and the analysis of spiked samples as a continuing check on performance. Appropriate QC samples, if possible, will be obtained from the Quality Assurance Branch, U.S. Environmental Protection Agency. The samples, obtained from the U.S. EPA, will be used as independent checks of technique. methodology, and standards, not as replacement for the standards. Replicates or spike samples will be routinely analyzed as part of the laboratory's quality control program. The laboratory will maintain performance records to define the quality of data that is generated. Ongoing performance checks will be compared with established performance criteria to determine if the results of analyses are within the accuracy and precision limits expected of the method.

To establish the ability to generate acceptable accuracy and precision, the analyst will perform the following operations using a QC sample as a check sample.

(1) Analyze three aliquots of the unspiked check sample.

(2) For each compound to be measured, select a spike concentration representative of twice the level found in the unspiked check sample or a level equal to 10 times the expected detection limit, whichever is greater. Prepare a spiking solution by diluting the compounds (QC samples) in methanol, acetone, or other acceptable solvent at the appropriate levels.

(3) Spike a minimum of three aliquots of the check sample with the spiking solution to achieve the selected spike concentrations. Spike the samples after they have been transferred to separatory funnels for extraction. Analyze the spike aliquots according to the method previously described.

(4) Calculate the average percent recovery, (R), and the standard deviation of the percent recovery, (S), for all compounds. The average recovery must be greater than 90% for all compounds to be measured. The percent relative standard deviation of the percent recovery, (S/R x 100), must be less than 10% for all compounds measured.

- 5.12.4.2.2 High Pressure Liquid Chromatography

A Perkin-Elmer Series 3 Liquid Chromatograph, LC-6ST detector/oven with a variable UV-IR detector interfaced by a Perkin Elmer Sigma 10 data acquisiton system will be used for determination of anionic organic tracer species (Section 5.12.2). Analysis procedures will follow those described in Bowman

(1984). Quality control, calibration determinations, and test solution preparation will be similar to those listed for gas chromatograph (Section 5.4.2.).

5.12.5 Data Validation

Data validation is an ongoing, continual process. The actual validation of data is accomplished during the analysis of various chemical components as discussed in Sections 5.12.4.1 and 5.12.4.2.

5.12.6 Data Reporting

All chemical analysis data will be tabulated and entered into a computerized data handling program. Reporting of data will be accomplished through quarterly and annual project reports. To limit the possible error due to data transcription the data base will be checked against the original data at least once. Also, spot check calculations will be done by hand on approximately 10% of the data to check for transcribing errors not caught during the first check.

5.12.7 Preventive Maintenance

Preventive maintenance of instrumentation and equipment used during the project will be performed according to the following procedures and schedules.

- 5.12.7.1 Plasma Emission Spectrometer

Operation	Schedule
Computer system	Every month
Clean air filters	Every three months
Change peristaltic pump tubing	Every three months

Clean torch	Every three months
Clean mirrors	Yearly
Change or clean drain tubing	Yearly
Change or clean inlet lines	Yearly
Check connections for leaks	Every two months
Change water filter	Yearly

Spare parts include: Torch, nebulizer, photo multiplier tubes, tubing, filters

5.12.7.2 Ion Chromatograph

Operation	Schedule
Regenerant flow rate	Weekly
Check connections for leaks	Daily
Lubricate pumps	Monthly
Clean or change eluent tubing	Yearly
Replace analytical column	When loss of anion resolution occurs
Replace suppressor column	When background conductivity is below acceptable limits

Spare chromtagraphic parts on hand include: Syringes, tubing, gaskets, fittings, supressor column.

5.12.7.3 Gas Chromatograph

Operation	Frequency
Replace carrier gas O_2 scrubber	Every six months
Recharge carrier gas H_2O scrubber	Every two months
Clean chassis air filter	Every six months

Clean detectors	When poor response is obtained
Replace injection septum	Every 80-100 runs or as required
Replace column	When poor resolution is obtained

Spare chromatographic parts on hand include: columns, packing materials. Swagelok^R fittings, carrier gas H_2O scrubber, septa, syringes.

- 5.12.7.4 High Pressure Liquid Chromatograph

Operation	Frequency
Check eluant flow rate	Daily
Check connections for leaks	Daily
Clean or change tubing	Yearly
Replace analytical column	When loss of resolution occurs
Replace injection septum	As required

5.12.8 Procedures for Assessing Data Accuracy, and Precision

The quality assurance activities implemented in the study will provide a basis to assess the accuracy and precision of the measurement systems. The generalized form of the equations which will be used to calculate accuracy and precision are given in section 10.0. Quality assurance objectives for these parameters were presented in Table 4.1.

6.0 SAMPLE CUSTODY

Strict chain of custody procedures involving locked storage are not required for this program. Since each soil sample will remain at the Survey, an exhaustive sample tracking system is not necessary. However, each sample will be assigned a unique number, denoting the date collected and sampling location. These sample numbers, collection data and sample location at the Survey will be entered on sample data sheets and distributed to members of the research team. This method will protect the samples from erroneous identification

7.0 DATA VALIDATION AND REPORTING

7.1 Data Validation

Data validation is an ongoing, continual process. The actual validation of data is accomplished during the analysis of various project components as discussed in various parts of Section 5.

7.2 Treatment of Outliers

Safeguards which will be taken against outliers (extreme observations) will include circumspection in performing experimental procedures, measurements and data recording. Data will also be carefully inspected before statistical analysis. If suspicious or anomalous observations occur, then inquiries will be made to determine the cause. If no explanation or correct value can be found, the test will be rerun. If it is not possible to rerun the test, then data analysis will be done both with and without the outlier to determine its effect on the conclusions. Skewness and kurtosis test may be done to help detect the presence of outliers.

7.3 Data Reporting

All data will be tabulated and entered into a computerized data handling program. Reporting of data will be accomplished through quarterly and annual project reports as described in Section 2.3. To limit the possible error due to data transcription the data based will be initially checked against the original data at least once. Also, spot check calculations will be done by hand on approximately 10 percent of the data to check for transcribing errors not caught during the first check.

8.0 PERFORMANCE AND SYSTEM AUDITS

A formal performance audit is inappropriate for this program because of the performance measures already incorporated into the program design and analytical procedures. However, external audits by EPA may be instituted at the discretion of the QA or EPA project officer at any time.

Due to the organization of this project an internal audit is inherent in that our procedures. If any problems are identified immediate corrective action will be instituted.

9.0 PREVENTIVE MAINTENANCE

Preventive maintenance of instrumentation and equipment used during the project will be performed according to the procedures and schedules described with the various analytical procedures described in section 5.

10.0 Procedures for Assessing Data Precision, Accuracy, and Completeness

The quality assurance activities implemented in the study will provide a basis to assess the accuracy and precision of the measurement systems. These calculations will be made to determine if the quality assurance objectives outlined in Table 4.1 are met. The generalized form of the equations which will be used to calculate accuracy and precision are given below:

10.1 Precision

$$\text{Standard deviation (s)} = \frac{\sum_{i=1}^{n} (x_i - \bar{x})^2}{n-1}$$

Where x_1 is the experimentally determined value for the i^{th} measurement, n is the number of measurements performed, and $\bar{x}$ is the mean of the experimentally determined values. The standard deviation is frequently expressed as the coefficient of variation which is the variability about the mean experimentally determined value, x, expressed as a percentage.

$$\text{Coefficient of variation, \%} = \frac{s100}{\bar{x}}$$

Where s is the standard deviation calculated according to the above equation.

10.2 Relative difference between duplicates

$$\text{R.D. (\%)} = \frac{\Sigma|x_1 - x_2|}{1/2\ (x_1 + x_2)} = 100$$

10.3 Accuracy

$$\text{Percent accuracy} = \frac{(X-T)\ 100}{T}$$

Where X is the average value of a set of measurements of the analyte determined experimentally and T is the true or reference value of the analyte being measured. X and T are in the same units.

10.4

Completeness describes the percentage of useable data points obtained. Percent completeness is calculated by the following equation:

$$\text{\% completeness} = \frac{\text{\#of valid data points}}{\text{\#of planned data points}} \times 100$$

11.0 CORRECTIVE ACTION

The need for corrective action occurs when a circumstance arises which threatens the quality of the data output. In order for corrective action to be initiated, awareness of a problem must exist. In most instances, the personnel conducting the laboratory and field analyses are in the best position to recognize problems which will affect data quality. Keen awareness on their part can frequently detect minor instrument changes, drifts or malfunctions which can be corrected, thus preventing a major breakdown of the system. The acceptance criteria for the QA/QC data have been described, as have corrective actions, in previous sections. The project personnel will have the prime responsibility for recognizing the need for corrective action.

If major problems do appear, B. L. Herzog (QA officer) will have the responsibility of determining the appropriate corrective action. This will be done in consultation with senior project staff.

12.0 QUALITY ASSURANCE REPORTS TO MANAGEMENT

The results of activities to assess the quality of the data generated during the project will be reported and discussed with the QA Officer, B. L. Herzog, by project staff at periodic meetings.

All quality assurance procedures employed and the results obtained will be included in the final report.

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