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WASTE MANAGEMENT

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by

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1.0 GOALS AND OBJECTIVES

Waste Management Project personnel are conducting research to characterize waste materials from advanced coal utilization processes and to develop innovative management practices for coal utilization waste disposal. The purpose of the characterization work is to predict the environmental impacts of wastes from several processes being developed at the University of North Dakota Energy and Mineral Research Center (UNDEMRC). The project is currently evaluating the chemical, physical, and leachate production properties of wastes from an atmospheric fluidized bed combustion (AFBC) process, a low-temperature coal gasification process, and a hot-water-drying coal slurry preparation process.

Project personnel are also developing methods for constructing fly ash liners at waste disposal sites, evaluating the use of new leaching tests for coal utilization wastes, and developing statistical procedures for analyzing soils data collected at candidate waste disposal sites. The purpose of these studies is to assist utility companies in the implementation of new environmental regulations in a cost-effective manner.

2.0 ACCOMPLISHMENTS

2.1 Waste Characterization

Eleven different waste materials were characterized during this reporting period. Six wastes were characterized from AFBC tests which used a coal slurry fuel. Four wastes were characterized from low-temperature coal gasification runs performed for the Hydrogen Production Project, and one waste was characterized from a coal preparation operation associated with the hot-water-drying coal slurry process. The eleven waste materials were all generated from coal utilization processes being developed at UNDEMRC.

The waste materials were tested for leachate trace metals and trace organics, elemental composition, mineral composition, and selected physical properties.

The results of the waste characterization activities are summarized as follows:

- EPA-EP leachates produced from all eleven of the coal utilization wastes contained trace metal concentrations well below the maximum allowable contaminant levels specified by the Resource Conservation and Recovery Act (RCRA) for hazardous waste classification.

- No significant levels of trace organic compounds were detected in the leachates produced from the eleven coal utilization wastes.

- The characterization studies indicated that no significant or unusual regulatory problems should be encountered for the disposal of the eleven coal utilization process wastes which were evaluated.

2.2 Fly Ash Liner Study

The purpose of the fly ash liner study is to develop cost-effective liner materials for utility waste disposal sites using mixtures of fly ash, water, hydrated lime, and/or Portland cement.

The fly ash liner study was started in the first year of the Waste Management project with a laboratory testing program, and was continued in the second year with additional laboratory tests and two fly ash liner field tests.

In the first year of the project, a series of laboratory tests were performed to develop liner formulas for six different types of fly ash wastes. The results of the formulation tests showed that minimum lime and/or cement additions ranging from 3 percent to 9.5 percent (of dry weight) were required to produce liner materials with permeability coefficients less than 1×10^{-7} cm/sec and unconfined compressive strengths greater than 400 psi.

In the second year of the project, four-square-foot by six-inch-thick fly ash liner slabs were constructed in the laboratory for leachate compatibility tests. Six liner slabs were constructed using the formulas developed in the preceding laboratory work. The slabs were submerged in fly ash leachates for five months to evaluate their stability under simulated field conditions. At the end of the five-month leachate exposure period, core samples from the liner slabs were tested for permeability coefficient and compressive strength. The results of the tests conducted on the liner cores indicated that the permeability and strength characteristics of the liner slabs were still acceptable after the five-month exposure period. The permeability coefficients of the liner cores typically ranged between 1×10^{-8} cm/sec and 1×10^{-9} cm/sec which means that the six liner materials had performed significantly better than the original design criteria of 1×10^{-7} cm/sec. The unconfined compressive strengths of the liner cores typically ranged between 1000 psi and 2000 psi, which exceeded the original design criteria for the liner materials of 400 psi.

For the fly ash liner field tests, two liner test sections were installed at sites located in Indiana and Texas. The formulas used for the field test sections were based on the preceding laboratory work. The dimensions of the liners installed for the field tests were 40 feet by 40 feet by 2 feet thick. The first liner slab was constructed at the H. W. Pirkey Plant located in Hallsville, Texas. The Pirkey Plant is owned by the Southwestern Electric Power Company. The second liner slab was constructed at the R. M. Schahfer Plant located near Wheatfield, Indiana. The Schahfer Plant is owned by the Northern Indiana Public Service Company. Each of these companies provided approximately 25 percent of the funds for the field tests.

The principal operations required for constructing the liner sections included placing the fly ash waste, mixing in appropriate amounts of lime and cement, mixing in water to obtain the correct moisture level, and then compacting the liner mixture. The liner sections were constructed in four lifts of six inches thick. The construction process for each liner section took approximately three days using a three-man crew. When the test sections were completed, a double-ring infiltrometer apparatus was placed on the surface to estimate the permeability of the liner in the field.

The results of the liner construction activities have been very encouraging. Construction activities at the field test sites were completed on schedule and no significant problems were encountered. The initial reports from the field sites indicate that both test sections are curing properly, and that no apparent expansion or cracking of the liners has occurred during the curing process.

Three sets of core samples were obtained from the Texas liner section for laboratory analysis, and one set of core samples was obtained from the Indiana liner section. These cores were tested for relevant structural properties such as compressive strength, tensile strength, modulus of elasticity, coefficient of thermal expansion, Poisson's Ratio, and permeability coefficient. The results of the tests performed on the core samples collected at the field sites generally indicate that these materials have acceptable physical properties for use as liner materials.

2.3 Numerical Modeling of Disposal-Related Soil Properties

The process of evaluating soil properties at candidate waste disposal sites could be improved by including a procedure for identifying inconsistent permeability data obtained from laboratory tests. Such data can result from improper sample collection, sample storage, or laboratory testing. Having the capability to check a set of test results for consistency is important because a single permeability measurement that does not meet the specified regulatory criteria for soil liners may exclude a candidate disposal site from being permitted.

The purpose of this research task is to develop a statistical procedure for checking the consistency of permeability data from candidate waste disposal sites in the Texas lignite region. This procedure can then be used to screen newly aquired soils data to identify test results that appear to be inconsistent with other data collected in this region.

A soils data-screening procedure was developed by compiling a relatively large data set containing information on the permeability coefficient, liquid limit, plasticity index, and percent passing a #200 sieve of soil samples collected at five power stations in east-central Texas. The screening procedure was based on a statistical model which predicted the permeability of a soil sample from its plasticity index and percent passing a #200 sieve. To screen the data set, each of the measured permeability coefficients was compared to its predicted permeability coefficient, and the difference between the two values was used as a measure of consistency for the data. If it was found that there was less than a five percent chance that a measured permeability coefficient would have been predicted by the model, it was concluded that the permeability measurement was significantly different from the rest of the data set. This finding could then be used as a basis for removing the inconsistent measurement from the data set.

2.4 Leaching Test Evaluation

The Environmental Protection Agency (EPA) is proposing to amend its hazardous waste identification regulations under Subtitle C of RCRA by

expanding the Toxicity Characteristic to include additional chemicals and introducing a new extraction procedure to evaluate the Toxicity Characteristic. These changes to the solid waste regulations are being developed to meet a specific mandate of the Hazardous and Solid Waste Amendments of 1984.

The proposed changes to the Toxicity Characteristic evaluation procedure will: 1) expand the characteristic to include 38 additional compounds, 2) revise the maximum allowable contaminant levels by applying compound-specific dilution/attenuation factors based on a groundwater transport model, and 3) introduce a second-generation leaching procedure, the Toxicity Characteristic Leaching Procedure (TCLP), which has been developed to address the mobility of both organic and inorganic compounds and to resolve the operational problems of the existing EP leaching method.

In response to the proposed changes to the solid waste regulations, a study was conducted at UNDEMRC to evaluate the use of the trace organic leaching procedure developed at the Morgantown Energy Technology Center (METC) to facilitate the use of follow-on TCLP leaching tests. The study was done by performing replicate leaching tests on two coal gasification tar samples using both the METC and TCLP procedures. The METC leaching tests were used to identify the various classes of nonvolatile trace organics in the waste leachates, and the TCLP tests were used to quantitatively identify the organics which had specific regulatory criteria. The results of the study indicated that the METC procedure was an effective means of screening the gasifier tar leachates for nonvolatile organic compounds.

3.0 WASTE CHARACTERIZATION

This section summarizes the results of the waste characterization studies conducted during the second year of the DOE-UNDEMRC Cooperative Agreement.

3.1 Waste Materials Studied

Eleven waste materials from advanced coal combustion processes being developed at UNDEMRC were studied in the second year of the Cooperative Agreement. These wastes included the following:

- Four spent bed materials from coal gasification tests performed by the Hydrogen Production Project.
- A spent bed material, a primary cyclone ash, a secondary cyclone ash, a baghouse fly ash, and two composite ashes from atmospheric fluidized bed combustion (AFBC) tests which used a coal slurry fuel.
- A heavy fraction from float-sink tests performed for the coal-cleaning operation of the hot-water-drying coal slurry process.

The gasification waste samples included two gasifier bed materials from hydrogen production runs which used Martin Lake, Texas, lignite. Both runs were performed at a temperature of 800^{o}C and a 3:1 steam-to-carbon molar

ratio. One of the runs used a limestone bed material and the other run used a silica sand bed material with a trona catalyst. Ten weight percent of trona was added to the coal for the catalyzed run. The third gasifier bed material characterized was produced from a hydrogen production run with a Velva, North Dakota, lignite. This run used a limestone bed, an 800°C gasification temperature, and a 2:1 steam-to-carbon ratio. The fourth gasifier bed material characterized was produced from a hydrogen production run with a Wyodak, Wyoming, subbituminous coal. This run used a limestone bed, an 800°C gasification temperature, and a 2:1 steam-to-carbon ratio.

The AFBC waste samples were produced in tests performed at UNDEMRC in a 1,000,000 Btu/hr, bubbling bed combustion unit. The fuel used was a Sarpy Creek, Montana, subbituminous coal which was burned in the form of an aqueous slurry. The waste samples studied included a spent bed material (silica sand), a primary cyclone ash, a secondary cyclone ash, a baghouse fly ash and a composite ash collected from the same combustion run. The sixth AFBC waste characterized was a composite ash produced from a slurry combustion run that used limestone addition directly to the coal slurry to increase SO_2 capture.

The AFBC composite ash samples were prepared by blending the various process waste streams in direct proportion to the amounts of material produced during the combustion test. The proportions used were 90 wt% primary cyclone ash, 8 wt% secondary cyclone ash, and 2 wt% baghouse fly ash.

The coal slurry used for the AFBC tests was produced at UNDEMRC with the hot-water-drying process. Wastes collected from slurry combustion runs may have different characteristics than wastes produced from pulverized, dry coal combustion runs because the slurry preparation process removes some water-soluble material from the coal prior to combustion. To evaluate the differences in waste composition resulting from combustion of the coal in the form of a slurry, the waste characterization data presented in this report was compared to characterization data collected in previous studies from waste samples produced with the same coal burned in a dry form (1).

The waste sample from the hot-water-drying coal slurry process was produced from a float-sink run performed on a Beulah-Zap, North Dakota, lignite. The float-sink operation was part of the coal cleaning procedure used for the slurry process.

3.2 Waste Characterization Methods

The testing program developed to evaluate disposal requirements for wastes from the advanced coal utilization processes consisted of analyses of the trace elements and trace organics contained in waste leachates, analyses of the chemical and mineral compositions of the wastes, and testing of the relevant physical properties of the wastes. The waste characterization protocol is summarized in Table 1. The principal objective of this characterization protocol is to identify any potential regulatory problems which may develop from the disposal of these wastes when the advanced processes are implemented on a commercial scale.

TABLE 1

THE WASTE CHARACTERIZATION PROTOCOL

A. Waste Leachate Testing

1. EPA-EP batch extraction and inorganic trace element analyses
2. ASTM D 3987 batch extraction and inorganic trace element analyses
3. Trace organic analyses of waste leachates
4. Column leaching tests and inorganic trace element analyses

B. Waste Chemical and Mineral Analyses

1. Major and trace inorganic elemental analyses
2. Mineral analysis
3. Trace organic analysis

C. Waste Physical Property Testing

1. Hydraulic Conductivity (also referred to as permeability coefficient)
2. Bulk density
3. Specific surface area
4. Loss-on-ignition

Leaching tests are often used to evaluate the regulatory status of solid wastes. The purpose of the EPA-EP batch leaching test is to determine whether a solid waste should be classified as a hazardous waste under the regulations established by the Resource Conservation and Recovery Act (RCRA). The regulations state that a solid waste may be classified as a hazardous waste based on leachate trace metal content if the levels exceed the maximum contaminant levels listed in Table 2. A detailed description of the procedure used for the EPA-EP leaching test is contained in Reference 2.

The RCRA regulations, in addition to defining the characteristics of hazardous wastes, also specify the allowable in situ contaminant levels for usable groundwater deposits adjacent to waste disposal facilities. In this regard, the regulations state that the disposal facility shall not cause trace metal concentrations in an underground drinking water source to exceed their primary drinking water standards. The primary drinking water standards for regulated trace metals are equal to 1/100 of the concentrations listed in Table 2.

The ASTM D-3987 batch leaching test is also useful for evaluating the potential impacts of coal utilization wastes on groundwater. The results of

TABLE 2

MAXIMUM ALLOWABLE LEVELS OF LEACHATE CONTAMINANTS FOR RCRA HAZARDOUS WASTE CLASSIFICATION

Element	Maximum Leachate Concentration (mg/l)
Arsenic	5.0
Barium	100.0
Cadmium	1.0
Chromium	5.0
Lead	5.0
Mercury	0.2
Selenium	1.0
Silver	5.0

this test are particularly important when the waste will be subjected to alkaline leaching conditions after disposal. Since leachates from western low-rank coal ashes typically have pH values between 10 and 13, this test is included in the characterization protocol.

Column leaching tests provide information about the rates at which trace elements will be extracted from wastes after they have been placed at a disposal site. This type of information is an important supplement to batch leaching data because it indicates the time frame during which the waste will have the greatest impact on the surrounding groundwater.

The experimental procedure used for the column leaching tests involved compacting the waste material into 1.5-inch by 3.5-inch cylinders and then passing distilled water through the cylinders under a constant hydraulic head. The cylinders were confined in a triaxial cell using a rubber membrane during the leaching tests. When the wastes were sufficiently permeable, leachate samples were collected after 2, 4, 6, 8, and 10 pore volumes had passed through the cylinders. The leachates were analyzed for sodium, calcium, aluminum, barium, chromium, and magnesium. Only the composite ash samples were studied with the column leaching procedure because of the relatively large number of analyses required for each test.

Trace organic compounds which leach from solid wastes are becoming more of a regulatory concern due to the proposed addition of phenolics, benzene, and toluene to the list of regulated leachate contaminants (3). For this reason, trace organic analyses of the coal utilization wastes and their leachates were included in the characterization protocol. The trace organics were analyzed using a quantitative gas chromatography/mass spectrometry (GC/MS) scanning procedure developed at the Morgantown Energy Technology Center (4).

Understanding the elemental composition of a waste is useful for evaluating a number of relevant disposal properties such as exothermal hydration potential, abrasiveness, and self-hardening potential. Elemental analyses were performed on the waste samples using energy-dispersive x-ray fluoresence.

The mineral composition of a waste can also influence its handling and disposal properties. For example, a fly ash with a high quartz content will tend to be abrasive, while a fly ash with a high lime content will tend to be cementitious. The mineral compositions of the waste samples were determined using a powder x-ray diffraction technique.

The hydraulic conductivity of a waste is a measure of the rate at which water will pass through the material under a given hydraulic gradient, and it indicates the potential for leachate production after disposal. Hydraulic conductivity was measured using a falling-head permeameter; the test method is contained in Reference 5. The hydraulic conductivity of a material is often referred to as the permeability coefficient.

The bulk density of a waste is useful for predicting the volume requirements for transportation equipment and land disposal facilities. Bulk density was measured for this study by a conventional volume displacement technique.

The specific surface area of a waste may affect its leaching behavior. In general, the greater the specific surface area, the higher will be the rate at which soluble inorganic constituents leach from the waste. Specific surface area was measured in this study with a Quantachrome, single-point, monosorb instrument, using the BET liquid nitrogen adsorption principal.

The loss-on-ignition test measures the unburned carbon content of a waste. A high unburned carbon content may inhibit cementitious reactions which otherwise would tend to reduce the permeability coefficient of the waste. Loss-on-ignition was measured for this study using ASTM Method C 311-68.

3.3 Waste Characterization Test Results

This section discusses the results of the leaching tests, chemical and mineral analyses, and physical property tests performed on the UNDEMRC advanced process wastes. The results of the waste characterization tests are tabulated in Appendix 1.

3.3.1 Leaching Test Results

The results of the EPA-EP leaching tests performed on the UNDEMRC process wastes indicate that none of these materials would be classified as hazardous wastes based on their leachate trace metal contents. A comparison between the EPA-EP leachate trace metal concentrations listed for the various wastes in Appendix 1 and the maximum allowable levels listed in Table 2 shows that the leachate trace metal concentrations were well below their RCRA limits.

Some states require special handling of wastes that produce ASTM leachate trace metal concentrations which exceed their primary drinking water standards by a factor of 25 because the groundwater has a limited capacity to dilute the leachate. Selenium was the only measured trace metal that exceeded its primary drinking water standard by more than a factor of 25 in any of the ASTM leachates. This occurred in the baghouse fly ash from the AFBC tests performed with Sarpy Creek subbituminous coal. The selenium concentration of the ASTM leachate was 0.3 mg/l as compared to the commonly used regulatory criteria of 0.25 mg/l (i.e., 25 times the primary drinking water standard).

The ASTM leachate data suggests that mixing the various waste streams from the AFBC process prior to disposal would be a good management practice because mixing tends to reduce the amount of leachable selenium per unit volume of waste. For example, when the baghouse fly ash from the AFBC process was mixed with the other waste streams to produce the composite ash, the ASTM leachate selenium concentration was reduced from 0.3 mg/l to less than 0.02 mg/l.

The leachate characteristics of the wastes from coal-slurry-fired AFBC runs were quite similar to the characteristics of analogous wastes from dry-coal-fired combustion runs. Characterizations of AFBC wastes produced from dry combustion of Sarpy Creek subbituminous coal were reported in the 1986-1987 Waste Management Project report (1). A comparison between the leachate data from the dry-combusted and slurry-combusted coals showed that the leachate trace metal concentrations from the two types of wastes were approximately equal, and therefore it was concluded that the slurry process had very little effect on the leaching behavior of the process wastes.

Trace organic analyses were performed on ASTM leachates from the UNDEMRC advanced process wastes. The leachate samples were prepared for analysis by performing acid and base solvent extractions. The organic extracts were then analyzed using GC/MS. The results of the analyses showed that no significant amounts of trace organics were present in any of the eleven UNDEMRC waste leachates studied. The minimum detection limit for the trace organics was approximately 20 mg/l in the leachate.

Column leaching tests performed on the two composite ash samples from the AFBC slurry combustion runs indicated that sodium and calcium were the elements extracted in the highest concentrations in the first six to ten pore volumes which passed through the samples. The elemental concentrations, however, did not appear to be high enough to pose a significant threat of contamination to the groundwater.

An interesting aspect of the column leaching tests was that the permeabilities of both of the composite ashes decreased as the tests progressed. The permeability coefficient of the composite ash produced without limestone addition decreased from 2.7×10^{-5} to 1.0×10^{-6} cm/sec during the test, and the permeability coefficient of the composite ash produced with limestone addition decreased from 6.3×10^{-5} to 6.4×10^{-8} cm/sec. The permeability of the ash from the limestone addition combustion run was reduced by three orders of magnitude after only six pore volumes had passed through the sample. In fact, the sample became so impermeable that the test had to be stopped after six pore volumes because it was not possible to collect enough leachate to do the necessary analyses.

The large decrease in the permeability coefficient of the limestone addition composite ash was probably caused by the occurrence of pozzolanic reactions between the silicates and aluminates in the ash and the calcium contributed by the limestone. This type of behavior is commonly observed in fly ashes which undergo extensive pozzolanic reactions.

3.3.2 Elemental and Mineral Analyses

The elemental and mineral analyses of the UNDEMRC advanced process wastes indicate that the materials are typically made up of silicon, aluminum, and calcium compounds, with lesser amounts of sodium, sulfur, and iron also being present. The major silicon-containing mineral phase detected in the wastes was quartz (SiO_2). The prominent calcium-containing mineral phases detected in the wastes were anhydrite ($CaSO_4$), melilite ($Ca_2Al_2SiO_7$), calcite ($CaCO_3$), and lime (CaO). (See Appendix 1 for a detailed listing of the elemental and mineral compositions of the wastes.)

The use of silica sand as a bed material for the AFBC and hydrogen production processes may produce highly abrasive waste materials due to the high silica content. This fact should be considered when designing handling equipment for waste transport and disposal operations.

The use of limestone additives as a means of trapping sulfur in both the AFBC and hydrogen production processes tends to increase the calcium content of the wastes. Increasing the calcium content may stimulate pozzolanic reactions in the wastes when they come into contact with water. Pozzolanic reactions are important because they can lead to significant reductions in the permeability of a waste after it has been placed at a disposal site. The dramatic reduction in the permeability coefficient of the AFBC composite ash produced with limestone addition during the column leaching test is a good example of how pozzolanic reactions can reduce the potential environmental impact of the waste.

3.3.3 Waste Physical Properties

The physical properties measured for the UNDEMRC waste characterization study included the permeability coefficient, bulk density, specific surface area, and loss-on-ignition. The results of the physical property tests are listed in Appendix 1.

The ranges of the physical properties measured for the UNDEMRC advanced process wastes are shown in Table 3. The highest permeability coefficient was measured for an AFBC, silica sand bed material and the lowest permeability coefficient was measured for a coal gasification, limestone bed material. For comparison purposes, it can be noted that the general criterion for selecting "impermeable" soils for waste disposal site liners is a permeability coefficient less than 1×10^{-7} cm/sec. Based on this criterion the UNDEMRC advanced process wastes could be classified as highly to moderately permeable. The bulk densities and surface areas measured for the advanced processes wastes were similar to other measurements obtained for other coal utilization wastes studied at UNDEMRC and elsewhere (6).

TABLE 3

RANGES OF PHYSICAL PROPERTIES OF THE UNDEMRC ADVANCED PROCESS WASTES

Physical Property	Range	
	High	Low
Permeability Coefficient (cm/sec)	1.3×10^{-1}	4.8×10^{-6}
Bulk Density (gm/ml)	3.3	1.38
Specific Surface Area (m^2/gm)	7.23	0.07
Loss on Ignition (wt%)	13.8	0.0

3.4 Waste Characterization Conclusions

The characterization data generated for the eleven UNDEMRC advanced process wastes does not indicate that any major regulatory problems should be encountered for the disposal of these materials on a commercial scale.

The EPA-EP leaching test results clearly show that the AFBC, hydrogen production, and coal slurry preparation wastes would not be classified as hazardous wastes based on their leachate trace metal contents under the existing RCRA regulations.

There were no significant amounts of trace organic compounds found in any of the UNDEMRC waste leachates.

The column leaching tests performed on the composite ashes showed that significant reductions in the permeability coefficients of these materials occurred during the course of the tests, particularly for the AFBC ash produced with limestone addition. The permeability reductions were probably caused by pozzolanic reactions between the ash and the limestone. The observed behavior indicates that the permeabilities of the composite ashes may decrease by several orders of magnitude after the materials have been placed in a permanent disposal site.

The use of silica sand bed materials in the advanced coal utilization processes may produce abrasive waste materials which require special procedures to protect transportation and handling equipment.

4.0 FLY ASH LINER STUDY

This section summarizes the results of the fly ash liner development study for the second year of the DOE-UNDEMRC Cooperative Agreement. The purpose of this study is to develop methods for constructing cost-effective liner materials for utility waste disposal sites using fly ash.

In the first year of the study, six fly ashes were tested to evaluate their suitability as liner construction materials. Each of the six fly ashes was initially tested for selected chemical and physical properties related to their use as liner materials. Screening tests were then performed to determine the approximate amounts of hydrated lime and/or portland cement that must be added to the fly ashes to produce acceptable liner materials. Based on the results of the screening tests, designed laboratory experiments were performed using systematically varied lime, cement, and water addition rates to develop individual liner formulas for each of the six different fly ashes.

The results of the characterization tests indicated that the fly ashes had acceptable physical properties for use as cementitious-type liner materials and that none of the ash materials would be classified as a hazardous wastes based on their leachate trace metal contents. The results of the formulation experiments showed that minimum cement and/or lime additions ranging from 3% to 9.5% (of dry weight) were required to produce liner materials with permeabilities significantly lower than 1×10^{-7} cm/sec and unconfined compressive strengths which generally exceeded 400 psi. The formulas determined for the six fly ashes are shown in Table 4. A detailed summary of the results of the first year of the fly ash liner study is contained in Reference 1.

In the second year of the fly ash liner study, laboratory tests were performed on liner slabs with dimensions of four square feet by six inches thick. The slabs were constructed to test the working properties of the liner materials and to verify the permeability and strength characteristics predicted by the formulation experiments. After completion of the slab tests, two of the fly ash liner formulas were tested in the field by constructing liner sections with dimensions of 40 feet by 40 feet by 2 feet thick. The two field tests were conducted at the power stations where the fly ashes were produced. Approximately 25 percent of the funding for the field tests was provided by the companies that owned the power stations. Liner core samples were collected from the field sites and a series of relevant physical properties were tested to evaluate the performance of the liners. The results of the laboratory-scale and field-scale liner tests from the second year of the study are summarized in Section 4.2 of this report.

4.1 Materials and Methods

4.1.1 Fly Ash Sources

The fly ashes used in the liner study were obtained from six utility companies located in five different states. The Northern States Power Company (NSP) supplied a fly ash - scrubber powder mix. This material was produced at NSP's River Side Station (Minnesota), firing a mixture of Sarpy Creek subbituminous coal and 10-15% coke. The Basin Electric Power Cooperative

TABLE 4

FLY ASH LINER FORMULAS

Liner Ash Source	Liner Composition			
	Lime (wt%)	Portland Cement (wt%)	Fly Ash (wt%)	Water (% of dry wt)
Northern States Power Co.	4	0	96	38
Basin Electric Power Co.	3	0	97	21
Texas Utilities Generating Co.	3	3	94	18
Southwestern Electric Power Co.	1.5	5	93.5	18
Northern Indiana Public Service Co.	3	5	92	19
Central Illinois Public Service Co.	3.5	6	90.5	20

supplied a fly ash produced from a Beulah lignite at the Antelope Valley Station in Beulah, North Dakota. The Texas Utilities Generating Company supplied a fly ash produced from a Texas lignite at the Big Brown Station in Fairfield, Texas. The Southwestern Electric Power Company (SWEP) supplied a fly ash produced from a Texas lignite at the H. W. Pirkey Station near Hallsville, Texas. The Northern Indiana Public Service Company (NIPS) supplied a 50%-50% mix of fly ash and scrubber sludge produced at the R. M. Schahfer Station in Wheatfield, Indiana. The NIPS fly ash was produced from an Illinois #6 coal and the scrubber sludge was obtained from a lime-based wet scrubber. The Central Illinois Public Service Company (CIPS) supplied a fly ash obtained from an ash pond located at the Meridosia Station located in Meridosia, Illinois. The CIPS fly ash was produced from an Illinois #6 bituminous coal.

4.1.2 Laboratory Test Methods for the Liner Slabs

The six liner slabs were constructed in the laboratory using the formulas listed in Table 4. For each slab, approximately 2.5 cubic feet of liner material was prepared in a paddle-type mixer and compacted in a 27-inch-diameter by six-inch-long PVC pipe section. The slab was compacted with approximately 100 psi of uniformly applied static pressure using three inch lifts. Figures 1 through 4 illustrate how the the liner slabs were mixed and compacted. After the liner slabs had cured for 28 days at 70^{o}F, they were placed in a leachate compatibility test device and loaded with six inches of

unconsolidated fly ash and 1.5 feet of water. Figure 5 shows the leachate compatibility test setup. The test devices were constructed so that any leachate that passed through the liner slabs would be collected below the slab.

The liner slabs were left in contact with the fly ash leachate for four months to test the stability of the liner material. During the four month tests, no measureable amounts of leachate passed through any of the liner slabs, and so the test devices were modified to exert 5 psi of additional head on the slabs. The slabs were then tested with 5.65 psi of head for another month, but it was still not possible to force any leachate through the liners. Finally, the liner slabs were taken out of the test devices and five cores were cut from each slab for bench scale permeability and strength tests. Figure 6 shows some of the core specimens taken from the six liner slabs.

The permeability coefficients of the liner cores were measured with a triaxial apparatus using a flexible membrane for sample confinement. It was possible to put 40 psi of head on the liner specimens and obtain permeability measurements in a reasonable time period using this apparatus. The liner cores were also tested for unconfined compressive strength using ASTM Method D 1633-84, "Compressive Strength of Molded Soil-Cement Cylinders." The durability of the liner materials to withstand freeze-thaw and wet-dry weathering cycles was tested by first inducing stresses in the core specimens with a vacuum saturation treatment and then testing their residual compressive strengths.

4.1.3 Field Test Construction Methods

Liner test sections were constructed for the fly ash liner study at two field sites in August and September of 1987. The first liner was constructed at the H. W. Pirkey Power Plant located in Hallsville, Texas. The Pirkey Plant is owned by the Southwestern Electric Power Company. The second liner was constructed at the R. M. Schahfer Power Plant located in Wheatfield, Indiana. The Schahfer Plant is owned by the Northern Indiana Public Service Company.

The liner section at the Pirkey Plant was made by mixing 5 wt% portland cement, 1.5 wt% hydrated lime, and approximately 18% water (% of dry wt.) with Texas lignite fly ash. The liner section at the Schahfer Plant was made by mixing 5 wt% portland cement, 3 wt% hydrated lime, and approximately 30% water (% of dry wt.) with a 50:50 mix of Illinois #6 fly ash and lime-based scrubber sludge.

The dimensions of the liner sections constructed for the field tests were 40 feet by 40 feet by 2 feet thick. This liner size was sufficiently large to permit the use of representative field-scale construction techniques. The lateral dimensions of the liners should also allow for development of maximum thermal warping stresses. The thickness of the liner was selected because two feet is a typical design specification required by many states for clay liners.

Figure 1. Batch Mixing of Fly Ash for Liner Material

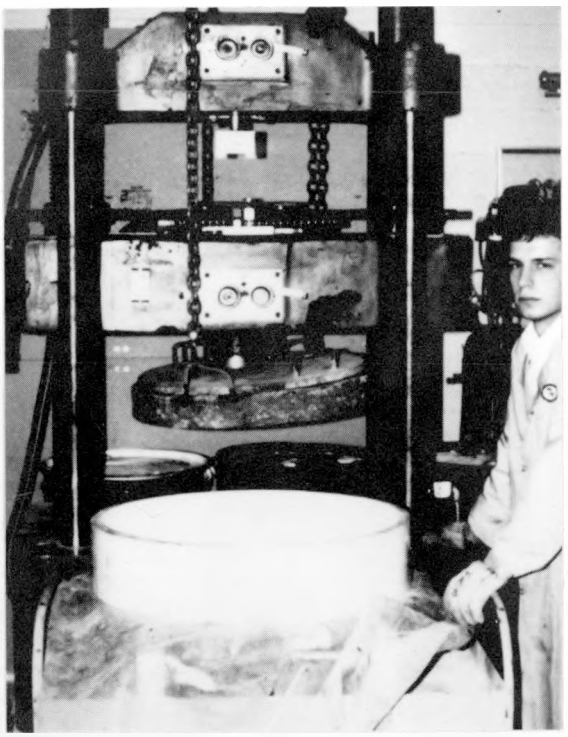

Figure 2. Compacting Apparatus Fly Ash Liner Slabs

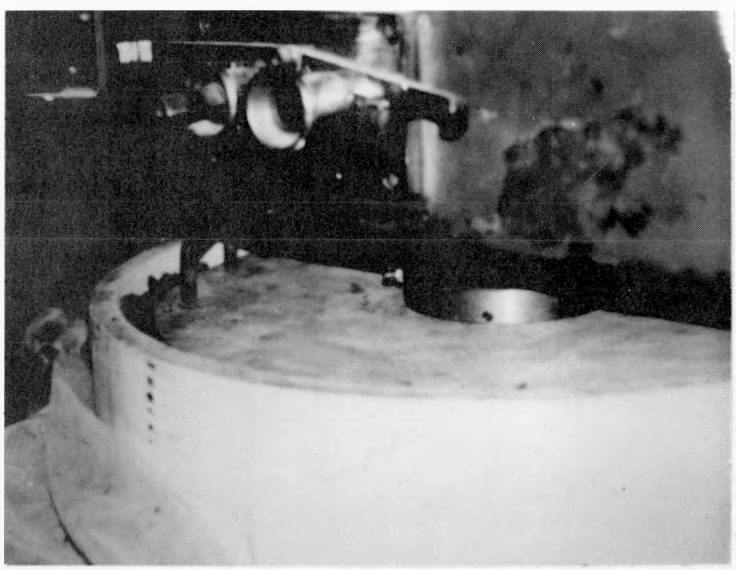

Figure 3. Fly Ash Liner Slab Liner Being Compacted.

Figure 4. Completed Fly Ash Slab.

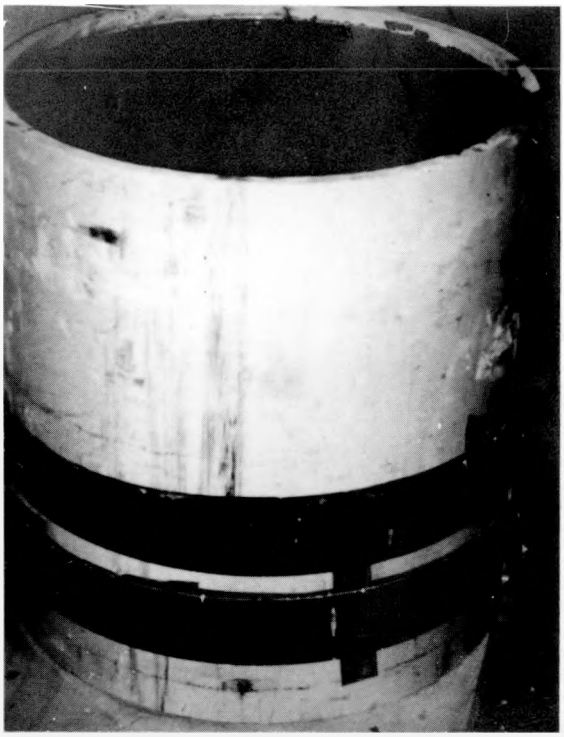

Figure 5. Leachate Compatibility Test Device for Fly Ash Liner Slabs

The liner sections were constructed so that the top surface was flush with the surrounding soil surface. This type of placement exposes the liner to ambient weather conditions throughout the test period and represents a "worst case" scenario in terms of the thermal stresses encountered.

The sections were installed in four consecutive six inch lifts. Each lift was compacted using a vibratory steel drum roller. Three passes were made with the roller to obtain liner densities similar to those produced in the laboratory. To ensure a good cure, the liner was compacted within two hours after the water was added.

Each liner lift was constructed by spreading a six-inch layer of fly ash and then tilling in the additives. The lime and cement were added by laying out the proper number of bags of material in an evenly spaced pattern, spreading the materials by hand, and then tilling it into the fly ash. Next, water was added and the mixture was tilled again. A five-foot, tractor-mounted tiller was used to mix in the additives. The proper amounts of hydrated lime, Type-1 portland cement and water used for the liner slabs were based on the formulas determined in the preceding laboratory study.

In-place mixing was used to construct the fly ash liner sections because discussions with contractors, who have experience with fly ash pavement construction, indicated that in-place mixing would provide adequate control over the composition of the liner material and would cost about 50 percent less than batch mixing for a full-size project.

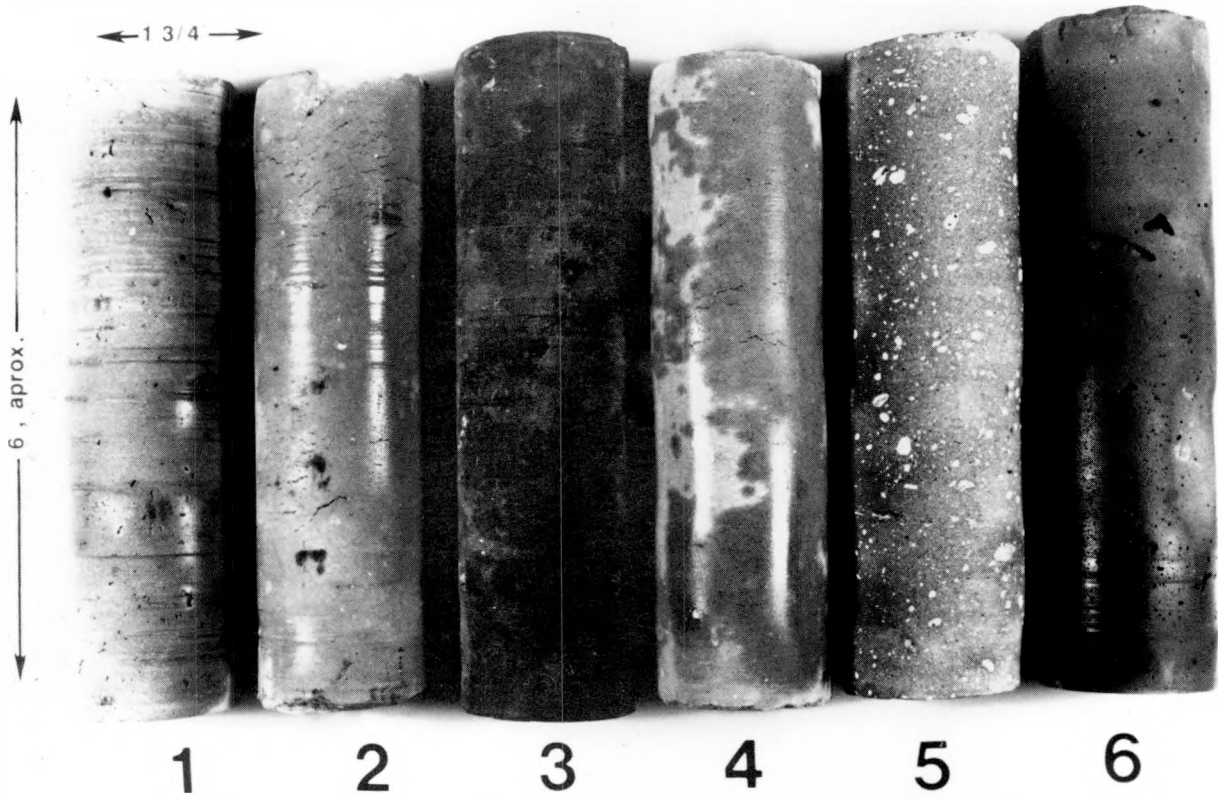

Figure 6. Core Samples Taken from the Fly Ash Liner Slabs Prepared in the Laboratory.

Core Number Identification:

1. Northern States Power Scrubber Waste.
2. Basin Electric Fly ash.
3. Central Illinois Fly Ash.
4. Texas Utilities Fly Ash.
5. Northern Indiana Fly Ash-Scrubber Sludge Mix.
6. Southwestern Electric Power Fly Ash.

After each of the slabs was completed, a double-ring infiltrometer was installed to estimate the permeability using ASTM method D 3385-75. The infiltrometer was inserted into the top lift of the liner slab immediately after the material was compacted. The infiltrometer at the Texas site was filled with 18 inches of water and the test was started after the liner had cured for about six weeks. The infiltration rate was determined by periodically measuring the rate at which the water level dropped in the infiltrometer barrels. The infiltrometer tests at the Indiana site were delayed until spring of 1988 because of freezing weather.

4.1.4 Test Methods for Liner Cores Collected in the Field

The physical properties of the fly ash liner sections are being evaluated by periodically taking core samples at the field sites and testing the cores in the laboratory. Four sets of cores will eventually be collected at each field site over a one-year period. The properties for which the cores are being tested include the permeability coefficient, unconfined compressive strength, Poisson's Ratio, modulus of elasticity, coefficient of thermal expansion, and tensile strength. The methods used for these tests are listed in Table 5.

During the second year of the cooperative agreement, three sets of core samples from the Texas field site were tested, and one set of core samples from the Indiana field site was tested. The first set of core samples was taken from the Texas site seven weeks after the liner was installed, the second set was taken fourteen weeks after the liner was installed, and the third set was taken seven months after the liner was installed. The first set of core samples was taken from the Indiana site ten weeks after the liner was installed. Each set was to consist of six, 1.5-inch diameter cores and six, 3-inch diameter cores; however it was not possible to collect all of the 1.5-inch core samples from the Indiana site because they tended to crumble when they were extracted from the liner. Figure 7 shows the locations of where the core samples were taken from the fly ash liner sections.

The core samples from the Texas site were composed of a brown, well-cemented ash material, similar to the fly ash liner materials produced in the preceding laboratory study with Texas lignite fly ash. The most obvious difference between the cores from the field liners and the materials produced in the laboratory was the degree of mixing. The in-place mixing technique used to construct the field liners was clearly not as thorough as the batch mixing technique used for the laboratory study because there were visible clumps of unmixed fly ash in the field cores.

The cores obtained from the Texas site were typically about ten inches long and broken into two or three pieces. The longest core fragments were about six inches long. It appeared that the cores had a tendency to crack between the top two lifts when they were being extracted from the liner. The separate core fragments appeared to be well-cemented and showed very little tendency to crumble.

TABLE 5

PHYSICAL PROPERTY TEST METHODS FOR THE LINER CORES COLLECTED AT THE FIELD SITES

Physical Property	Test Method
Unconfined Compressive Strength	ASTM D 1633 - 84
Modulus of Elasticity	ASTM C 469 - 83
Poisson's Ratio	ASTM C 469 - 83
Tensile Strength	Test done with direct loading of specimen to failure
Coefficient of Thermal Expansion	ASTM E 228 - 71

The core samples from the Indiana site were composed of a gray, mortar-like material which contained particles of white scrubber sludge. The three-inch cores from the Indiana slab were typically about ten inches long and each was broken into three or four pieces. The Indiana cores definitely showed a greater tendency to crumble than the Texas cores. The Indiana cores, however, were collected in freezing weather and the samples were frozen when they arrived at UNDEMRC for testing. Only one set of cores was collected from the Indiana site during this reporting period due to the cold weather.

4.2 Fly Ash Liner Test Results

This section summarizes the results of the fly ash liner tests conducted in the laboratory and in the field. The four-square-foot liner slabs were prepared in the laboratory in January of 1987, and the leachate compatibility tests performed on the slabs were completed in June of 1988. The 1600-square-foot liner test sections were constructed in the field in August and September of 1987, and the field tests are scheduled to be completed in the spring of 1989.

4.2.1 Results of the Laboratory Slab Tests

Leachate compatibility tests were performed to evaluate the durability of the six fly ash liner slabs prepared in the laboratory. For these tests, the

liner slabs were exposed to aqueous leachates for four months under a head of 0.65 psi, and then exposed to the leachates for one additional month under a head of 5.65 psi. No measurable amounts of leachate passed through the liner slabs in the five-month test period. These results indicated that the slabs did not significantly deteriorate during the prolonged contact with the leaching solutions and that the permeabilities of the slabs were all significantly lower than 1×10^{-7} cm/sec.

After the leachate compatibility tests were concluded, the liner slabs were removed from the test devices and five core samples were cut from each slab. The permeability coefficient and unconfined compressive strength of each liner material was determined by performing bench-scale tests on the core samples. The results of the permeability and strength tests are listed in Table 6. The liners were originally designed to meet a permeability criterion of 1×10^{-7} cm/sec and an unconfined compressive strength criterion of 400 psi both before and after vacuum saturation treatment. The results presented in Table 6 show that the liner slabs surpassed both of these criteria even after five months of contact with a leaching solution. The permeability coefficients for the liner cores were typically one to two orders of magnitude lower (less permeable) than the design criteria, and the compressive strengths were typically three to four times higher than the design criteria.

Table 6 also lists the dry densities and porosities of the liner cores. The dry densities ranged from 78 lbs/cu ft to 117 lbs/cu ft and the porosities ranged from 26% to 45%. Porosity is a measure of the void volume within the liner material expressed as a percent of the total volume of the specimen.

4.2.2 Liner Slab Construction Report

The construction of the fly ash liner test sections at the Texas and Indiana field sites was accomplished on schedule. No significant problems were encountered during construction.

Three days were required to construct each of the liner sections. The material used for the Texas liner was a Texas lignite fly ash. The material used for the Indiana liner was a 50%-50% mixture of Illinois #6 bituminous coal fly ash and lime-based wet scrubber sludge. Figures 8 through 15 illustrate the various operations involved in the fly ash liner construction process. See Section 4.1.3 for a description of the field construction methods.

Reports from the two field test sites indicate that both liners are curing properly since no significant cracking has been observed and the liners appear to be quite hard. A heavy rain occurred at the Texas site a few hours after the liner was installed. The rain caused some superficial marking of the liner surface, but it did not cause any significant structural damage to the liner.

4.2.3 Liner Core Test Results

Three sets of liner cores from the Texas site and one set of liner cores from the Indiana site were tested during this reporting period. The fly ash

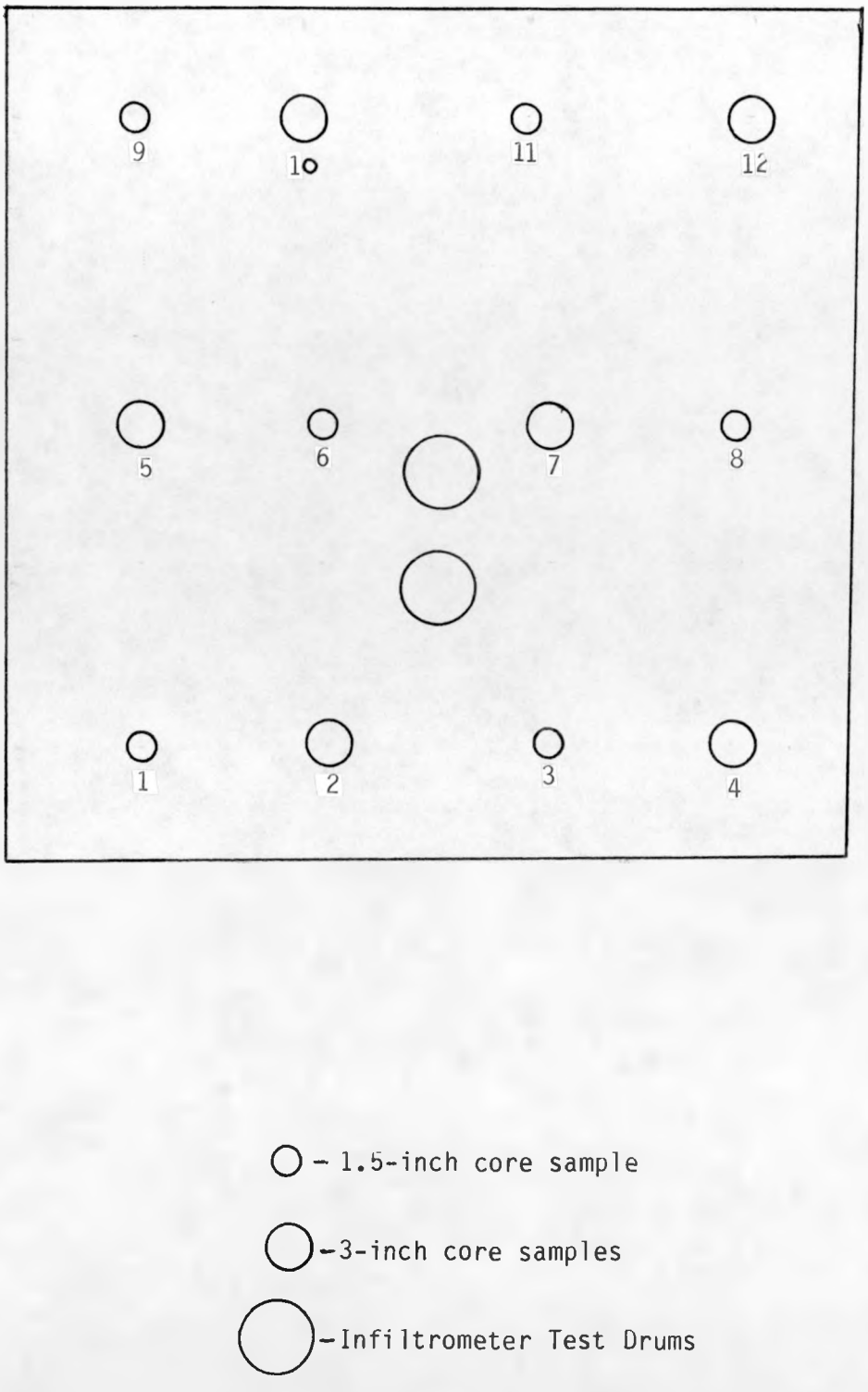

Figure 7. Core Sample Locations on the Fly Ash Liner Slabs

liner core samples were tested at UNDEMRC for permeability coefficient, unconfined compressive strength, modulus of elasticity, Poisson's Ratio, tensile strength, coefficient of thermal expansion, dry density, and porosity.

The results of the permeability tests are shown in Table 7. Four samples were tested from each set of cores collected in the field. The log mean coefficient of permeability for each of the core sets was below 1×10^{-7} cm/sec, although one core from the first and third sets collected at the Texas site and two cores collected at the Indiana site exhibited permeability coefficients greater than 1×10^{-7} cm/sec.

The results of infiltrometer tests performed at the Texas site are shown in Table 8. The infiltration rate of the liner was determined by periodically measuring the drop in the water level in the infiltrometer barrels. There was a relatively small amount of infiltration into the liner in the first five weeks after the test was started. After five weeks however, there was no measurable infiltration into the liner.

An infiltrometer test does not, strictly speaking, measure the permeability of the liner. However, the data from the infiltrometer test can be used to estimate liner permeability based on the head in the infiltrometer and the depth to which the apparatus is inserted into the liner. The permeability coefficient of the liner appears to be less than 1×10^{-7} cm/sec based on the fact that there was no measurable drop in the water level in the apparatus after the first five weeks of the test. These results are important because they corroborate the permeability measurements obtained in the laboratory with the liner core samples.

The physical properties measured for the three sets of liner cores collected at the Texas site are shown in Table 9, and the physical properties measured for the cores collected at the Indiana site are shown in Table 10. The physical properties measured for the fly ash liner cores are similar to properties reported in the literature for other fly ash-type paving materials (7).

The unconfined compressive strengths of the liner cores were generally lower than the strengths obtained in previous laboratory tests with these fly ashes. The lower strength of the liner materials placed in the field may have resulted from the in-place type of mixing which was used, but it also may be due to the fact that the average curing temperatures which the liners have been exposed to in the field over the winter were lower than the curing temperature used in the laboratory. The moduli of elasticity of the liner materials are intermediate between the moduli of elasticity of clay and concrete (approximately 5×10^3 psi and 5×10^6 psi, respectively), which indicates that the liner material is somewhat more flexible than concrete. The coefficients of thermal expansion measured for the core samples were quite low and similar to the coeficient of thermal expansion typically measured for concrete.

4.3 Fly Ash Liner Test Conclusions

The results of the tests conducted on the laboratory scale fly ash liner slabs indicated that all six liner materials displayed excellent stability after extended exposure to leaching solutions. The permeabilities of the

TABLE 6

PHYSICAL PROPERTIES OF FLY ASH LINERS AFTER FIVE MONTHS OF EXPOSURE TO LEACHATES IN THE LABORATORY

Liner Ash Source	Permeability Coefficient (cm/sec)	Compressive Strength (psi)	Compressive Strength After Vac. Sat. (psi)
Northern States Power Co.	4.0×10^{-9}	1454	1100
Basin Electric Power Co.	2.2×10^{-9}	1057	1153
Texas Utilities Generating Co.	5.7×10^{-9}	2334	1845
Southwestern Electric Power Co.	9.0×10^{-9}	1268	2782
Northern Indiana Public Service Co.	3.0×10^{-9}	863	1067
Central Illinois Public Service Co.	1.6×10^{-8}	1014	1154

Liner Ash Source	Dry Density (lbs/cu ft)	Porosity (%)
Northern States Power Co.	78	41
Basin Electric Power Co.	106	38
Texas Utilities Generating Co.	112	26
Southwestern Electric Power Co.	117	31
Northern Indiana Public Service Co.	84	45
Central Illinois Public Service Co.	106	37

Figure 8. Spreading Fly Ash (Texas)

Figure 9. Adding Cement and Lime (Texas)

Figure 10. Adding Water to Liner Mix (Texas)

Figure 11. Completed Liner Lift (Texas)

Figure 12. Spreading Cement and Lime (Indiana)

Figure 13. Tilling In Additives (Indiana)

Figure 14. Compacting a Liner Lift (Indiana)

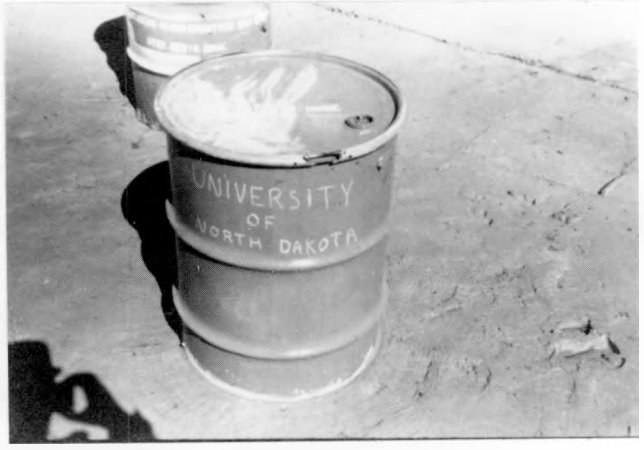

Figure 15. Infiltrometer Apparatus (Indiana)

liner slabs after the five month exposure period were all significantly lower (less permeable) than the 1 x 10^{-7} cm/sec general design criteria, and the compressive strengths of the slabs were all significantly higher than the 400 psi general design criteria. A visual examination of the slabs at the end of the exposure period confirmed that no cracking or softening of the liner materials had occurred.

The results obtained thus far from the two fly ash liner field tests are also very encouraging. Tests performed on core samples collected at the field sites show that the permeability coefficients of the liner materials were generally below 1 x 10^{-7} cm/sec, although the compressive strengths of the field liners were substantially lower than the compressive strengths of the materials prepared in the laboratory. The permeability characteristics observed in the laboratory tests were corroborated by the results of infiltrometer tests performed in the field at the Texas liner site. The values for modulus of elasticity and coefficient of thermal expansion measured for the liner core samples indicate that the liner materials are more flexible than concrete and that they should not undergo excessive thermal expansion.

5.0 NUMERICAL MODELING OF DISPOSAL-RELATED SOIL PROPERTIES

The process of evaluating soil properties at candidate waste disposal sites could be improved by including a procedure for identifying inconsistent permeability data obtained from laboratory tests. Such data can result from improper sample collection, sample storage, or laboratory testing. Having the capability to check a set of test results for consistency is important because a single permeability measurement that does not meet the specified regulatory criteria for soil liners may stop a candidate disposal site from being permitted.

The usual method for evaluating soil properties is to perform borings and collect in situ soil samples at various depths. Samples collected in this manner are logged and sent to the laboratory for testing. The test results, which are included in the application for a waste disposal permit, are typically reported directly from the laboratory data with little or no attempt to statistically screen the results. A screening procedure for permeability data could benefit a utility company in the process of developing a new waste disposal site by providing a statistically valid basis for removing questionable observations from the data set. Such a procedure would also benefit state regulatory agencies by supplying higher quality data for the decision process involved in granting permits.

In this research task, a statistical procedure was developed for checking the consistency of permeability data from candidate waste disposal sites in the Texas lignite region. This procedure can be used to screen newly aquired soils data to identify test results that appear to be inconsistent with other soils data collected in this region.

TABLE 7

PERMEABILITIES OF CORE SAMPLES FROM THE FLY ASH LINER FIELD TEST SITES

Southwestern Electric Power Co. - Core Set #1

	Core 1	Core 2	Core 3	Core 4	Log Mean
Permeability Coefficient (cm/sec)	7.6×10^{-8}	7.3×10^{-8}	1.1×10^{-7}	1.1×10^{-8}	5.1×10^{-8}

Southwestern Electric Power Co. - Core Set #2

	Core 1	Core 2	Core 3	Core 4	Log Mean
Permeability Coefficient (cm/sec)	2.0×10^{-8}	1.1×10^{-8}	3.8×10^{-8}	6.7×10^{-8}	2.7×10^{-8}

Southwestern Electric Power Co. - Core Set #3

	Core 1	Core 2	Core 3	Core 4	Log Mean
Permeability Coefficient (cm/sec)	4.9×10^{-8}	1.2×10^{-7}	4.5×10^{-8}	1.8×10^{-8}	4.7×10^{-8}

Northern Indiana Public Service Co. - Core Set #1

	Core 1	Core 2	Core 3	Core 4	Log Mean
Permeability Coefficient (cm/sec)	1.8×10^{-7}	2.6×10^{-8}	1.7×10^{-7}	7.5×10^{-8}	8.8×10^{-8}

TABLE 8

INFILTROMETER TEST RESULTS - SOUTHWESTERN ELECTRIC POWER FIELD SITE

Date of Measurement	Water Level* (inches)	Time Interval (days)	Change in Water Level (inches)	Water Temperature (^{o}F)	Infiltration Rate (cm/sec)	Estimated Permeability Coefficient (cm/sec)
10/29/87	16	22	2.0	62	2.7×10^{-6}	5×10^{-7}
11/11/87	17.75	14	0.25	61	5.3×10^{-7}	9×10^{-8}
11/25/87	18	14	0	59	0	$< 1 \times 10^{-8}$
12/9/87	18	14	0	58	0	$< 1 \times 10^{-8}$
01/05/88	18	25	0	48	0	$< 1 \times 10^{-8}$

* The water level in the infiltrometer barrel was 18 inches at the start of the test, and the water level was readjusted to 18 inches after each reading was taken.

TABLE 9

CORE SAMPLE PHYSICAL PROPERTY TEST RESULTS:
SOUTHWESTERN ELECTRIC POWER FIELD TEST

Physical Property	Core Set #1	Core Set #2	Core Set #3
Unconfined Compressive Strength (psi)	346	360	430
Modulus of Elasticity (psi)	2.3×10^5	7.9×10^5	1.0×10^6
Poisson's Ratio	NR*	0.35	0.19
Tensile Strength	61	60	114
Coefficient of Thermal Expansion (in/^{o}F)	3.5×10^{-6}	5.2×10^{-6}	1.4×10^{-6}
Dry Density (lbs/cu ft)	112	113	109
Porosity (%)	33.4	33.3	32.0

* Reproducible measurements of Poisson's Ratio could not be obtained for this sample.

5.1 Model Development Methods

5.1.1 Data Set Description

The data screening procedure was developed by statistically analyzing test results from 105 different soil samples collected in east-central Texas. The data was obtained from the permit files of the Texas Department of Water Resources (TDWR). The TDWR's files contain a large amount of uniform soils data because Texas has longstanding guidelines for the permeability coefficient, percent passing a #200 sieve, plasticity index, and liquid limit of soils being used for liners at Class I and Class II waste disposal sites. The TDWR's recommended soil specifications are listed in Table 11 (8).

TABLE 10

CORE SAMPLE PHYSICAL PROPERTY TEST RESULTS:
NORTHERN INDIANA PUBLIC SERVICE FIELD TEST

Physical Property	Core Set #1
Unconfined Compressive Strength (psi)	200
Modulus of Elasticity (psi)	9.6×10^4
Poisson's Ratio	0.28
Tensile Strength (psi)	26
Coefficient of Thermal Expansion (in/^{o}F)	1.3×10^{-5}
Dry Density (lbs/cu ft)	80
Porosity (%)	48.6

The soils data was obtained from the permit files of five fossil fuel power plants located in east-central Texas. The five plants where the data was collected included the Limestone Power Plant located in Jewett, Texas; the Martin Lake Power Plant located in Rusk County, Texas; the Gibbons Creek Power Plant located in Carlos, Texas; the San Miguel Power Plant located in Atascosa County, Texas; and the Pirkey Power Plant located in Hallsville, Texas. The depths at which the soil samples were collected ranged from the surface to approximately 150 feet below the surface. In addition to having data on permeability coefficient, percent passing a #200 sieve, plasticity index, and liquid limit, the dry density and moisture content were also reported for many of the samples. The data set included both recompacted and undisturbed soil samples. The full data set is contained in Appendix 2.

TABLE 11

TEXAS DEPARTMENT OF WATER RESOURCES'
RECOMMENDED SOIL SPECIFICATIONS FOR WASTE DISPOSAL SITE LINERS

Parameter	Specification
Permeability Coefficient	$< 1 \times 10^{-7}$ cm/sec
Liquid Limit	>30%
Plasticity Index	>15%
Percent Passing a #200 Sieve	>30%

5.1.2 Statistical Analysis Procedures

The statistical analysis which was performed to develop the soils data screening procedure was done using the SAS computer software package (9). The UNIVARIATE procedure was used to calculate simple statistics such as the mean and standard deviation for the data set. The FREQ procedure was used for plotting frequency histograms to study the distribution of the data set. The CORR procedure was used to calculate correlation coefficients between the various soil properties, and the REG procedure was used to perform multiple-variable regression analyses on the data set.

The full data set was initially analyzed in this manner. It was then separated into five groups based on the different sites where the soil samples had been collected, and each group was analyzed independently. Since all five data groups were distributed in basically the same manner, the decision was made to develop the screening procedure using the full data set instead of studying each group separately.

5.2 Model Development - Results and Discussion

The basic statistics calculated for the soils data set are listed in Table 12. Since most of the soil samples were clays, the mean values for log permeability coefficient, liquid limit, plasticity index, and percent passing a #200 sieve were well within the acceptable ranges of the TDWR's liner criteria. All of the soil properties displayed relatively wide ranges of values, and all of the properties followed skewed distributions.

After the initial statistical analyses, correlation coefficients between the various soil properties were determined. It was hoped that at least one of the other measured soil properties would exhibit a strong correlation with permeability, and thus provide a simple means of checking the consistency of

the permeability data. Unfortunately, there were no strong correlations found between permeability and the other soil properties. The best correlations were found between the liquid limit and the plasticity index (0.97) and between the dry density and the moisture content (-0.79). Values for correlation coefficients can range between -1.0 and 1.0. (A value of -1.0 indicates the highest possible negative correlation, a value of 1.0 indicates the highest possible positive correlation, and a value of 0.0 indicates that no correlation exists between the variables.)

The correlation coefficient matrix for all of the properties in the soils data set is listed in Table 13. The high positive correlation between liquid limit and plasticity index is explained by the fact that most of the soils were classified as either Type CH or Type CL clays, and these types of materials follow the relationship represented by the A-line on Casagrande's plasticity chart (10). The high negative correlation between dry density and moisture content is explained by the fact that a lower dry density generally indicates a larger pore volume, which permits more moisture to be stored in the soil.

The soils data set was next analyzed using a multiple-variable, nonlinear regression procedure to develop a permeability model based on the combined effects of liquid limit, plasticity index, and percent passing a #200 sieve. It was decided not to include either dry density or moisture content in the regression model because both of these variables were missing too many values. The regression procedure was used to fit the permeability data to an empirical quadratic model containing squared terms and cross-product terms. The goal of this model was to be able to accurately predict the permeability of an individual soil sample based on its measured values for liquid limit, plasticity index, and percent passing a #200 sieve. Since it was considered advantageous to keep the model as simple as possible, some preliminary work was done to determine whether it was necessary to include all three soil properties in the model.

The degree of fit between a data set and a regression model is generally measured by the squared multiple correlation coefficient (r^2). The value of the r^2 parameter can range from 0.0 to 1.0 with 1.0 representing the best possible fit between the measured data and the values predicted by the model. Values of r^2 were calculated for a series of permeability models using the RSQUARE option of the REG procedure to determine which combinations of terms yielded the best model. The results of this analysis indicated that plasticity index was the variable which had the largest effect on the permeability model, percent passing a #200 sieve had the next largest effect, and liquid limit had the smallest effect on the model.

To determine the combined effects of the variables, a permeability model was first developed using terms containing only the plasticity index variable, then terms containing the percent passing a #200 sieve variable were added to the model, and finally terms containing the liquid limit variable were added to the model. The r^2 values obtained for each of the three types of models are listed in Table 14. Scatter diagrams for the models which illustrate their predictive capabilities are shown in Figures 16, 17, and 18. These regression models were generated using the BACKWARD option of the REG procedure. The BACKWARD option uses backward elimination to sequentially remove non-significant terms from the model. Based on a comparison of the r^2

TABLE 12

STATISTICAL SUMMARY OF THE SOILS DATA

	Liquid Limit (%)	Plasticity Index (%)	Percent Passing a #200 Sieve (%)	Log Permeability Coefficient (cm/sec)	Dry Density (lbs/cu.ft.)	Moisture Content (% dry wt.)
Number of Observations	101	101	82	105	84	98
Minimum Value	27	3	7	-9.24	67	9
Maximum Value	145	141	99	-1.67	123	40
Mean	58	34.5	73.7	-7.33	95	24.8
Standard Deviation	24.2	22.2	19.1	1.38	10.4	5.7
Type of Distribution	Skewed Low	Skewed Low	Skewed Low	Skewed High	Skewed Low	Skewed Low

values obtained for the various models and the goodness of fit between the measured and predicted permeabilities as indicated by the scatter diagrams, it was concluded that the model containing two independent variables produces almost as good of a fit to the data as the model containing three independent variables. Therefore, the equation selected to model the permeability data had the following form:

$$\log \text{Perm} = 0.73 - 0.067\ PI - 0.17\ P200 + 0.00038\ PI^2 + 0.0011\ P200^2$$

where PI is the plasticity index,
and P200 is the percent passing a #200 sieve.

The next step in developing the data-screening procedure was to use the two-variable permeability model to identify those soil samples that were significantly different from the other samples in the data set based on the inrelationship between the permeability coefficient, the plasticity index, and the percent passing a #200 sieve. The most useful technique for doing this was to examine the differences between the measured permeability coefficients and the permeability coefficients predicted by the model.

These differences are called residuals. The residuals determined for a specific model can be standardized by transforming them into "studentized residuals" or t-values. The R option for the REG procedure automatically calculates t-values for all of the samples in the data set. The decision as to whether a permeability measurement for a specific soil sample does or does not fit the model can be made by comparing its t-value to some critical value. If the t-value for a soil sample exceeds the critical value, it can be concluded that the permeability measurement does not fit into the model and that the sample is significantly different from the rest of the data set.

The CLI option for the REG procedure automatically compares the t-values generated by the model to a reasonable critical value by calculating the 95 percent confidence interval for each of the predicted permeability values. If a measured permeability value falls outside the confidence interval, there is less than a five percent chance that this value would have been predicted by the model and that the large observed difference between the measured and predicted values occured by random experimental error. When a measured permeability value falls outside the 95 percent confidence interval, that fact may then be used as a basis for deleting the measurement from the data set. The 95 percent confidence intervals for the three permeability models are illustrated in figures 16, 17, and 18.

When the Texas soils data was analyzed in the manner described above, four permeability measurements were outside the 95 percent confidence interval. These permeabilities were for Sample Numbers 6, 8, 13, and 82 listed in Appendix 2. It is worth noting that three of the four outlying permeability measurements were obtained from the same site, which may indicate that a different type of permeability test was used for the samples collected at this site.

TABLE 13

CORRELATION COEFFICIENT MATRIX FOR THE SOILS DATA SET

	Permeability Coefficient	Dry Density	Liquid Limit	Plasticity Index	% Passing a #200 Sieve	Moisture Content
Dry Density	-0.015	---	-0.35	-0.23	-0.078	-0.79
Liquid Limit	-0.44	-0.35	---	0.97	0.38	0.44
Plasticity Index	-0.48	-0.23	0.97	---	0.35	0.36
% Passing Index #200 Sieve	-0.59	-0.078	0.38	0.35	---	0.47
Moisture Content	-0.28	-0.79	0.44	0.36	0.47	---

TABLE 14

ONE-, TWO-, AND THREE-VARIABLE REGRESSION MODELS FOR PREDICTING SOIL PERMEABILITY

One-Variable Model*, r^2=0.47

$$\log Perm = -0.32 - 0.18\ P200 + 0.001\ P200^2$$

Two-Variable Model*, r^2=0.505

$$\log Perm = 0.73 - 0.067\ PI - 0.17\ P200 + 0.00038\ PI^2 + 0.0011\ P200^2$$

Three-Variable Model*, r^2=0.56

$$\log Perm = -2.65 - 0.33\ PI + 0.25\ LL - 0.16\ P200 - 0.0034\ PI^2 - 0.0038\ LL^2 + 0.001\ P200^2 + 0.0077\ PI\ LL$$

* where PI is the plasticity index, LL is the liquid limit, and P200 is the Percent Passing a #200 Sieve.

It must be remembered that the procedure developed to identify inconsistent permeability data is based on statistical principals and assumptions, and therefore is not an absolute means of rejecting a specific measurement. It is advisable to combine some additional information about the soil sample with the statistical analysis to optimize the data screening procedure. Specifically, it would be a good idea to record a description of the soil sample before and after the permeability test for later reference. If there is a question about the validity of a measurement later on, then the sample description might provide a simple physical explanation for the apparent inconsistency. For example, the presence of visible cracks or sand lenses in a sample might explain an unusually low permeability. If there is no obvious physical explanation for the unusual test result, then it may be concluded that the result is inaccurate.

5.3 Model Development - Summary and Conclusions

The data screening procedure developed in this section is summarized as follows:

Step 1 Assemble a relatively large data set (i.e., a reference data set) from the same general area where the data being tested was collected. If possible, the data set should contain measurements of permeability coefficient, liquid limit, plasticity index, and percent passing a #200 sieve. Make the reference data set as large as possible.

Step 2 Develop a quadratic regression model using the SAS computer analysis system to generate a predictive equation for sample permeability. The independent variables for the model can include the plasticity index, the percent passing a #200 sieve, and the liquid limit of the sample. Use the BACKWARD option of the REG procedure to produce the regression equation.

Step 3 Determine whether the permeability measurements fall within the 95 percent confidence interval for the predicted value. If the measured value is not within the 95 percent confidence interval, remove that sample from the data set.

The data screening procedure described above is designed to identify permeability measurements that are inconsistent with the rest of the reference data set based on the interrelationships exhibited between the various soil properties. This inconsistency does not necessarily mean that a permeability measurement is erroneous; however, it does indicate a high probability that the measurement is in some way different from the other measurements. The screening procedure should be used along with any other available information about the sample to make the final decision whether or not to remove the sample from the data set.

6.0 LEACHING TEST EVALUATION

The Environmental Protection Agency (EPA) is proposing to amend its hazardous waste identification regulations under Subtitle C of the Resource Conservation and Recovery Act (RCRA) by expanding the Toxicity Characteristic

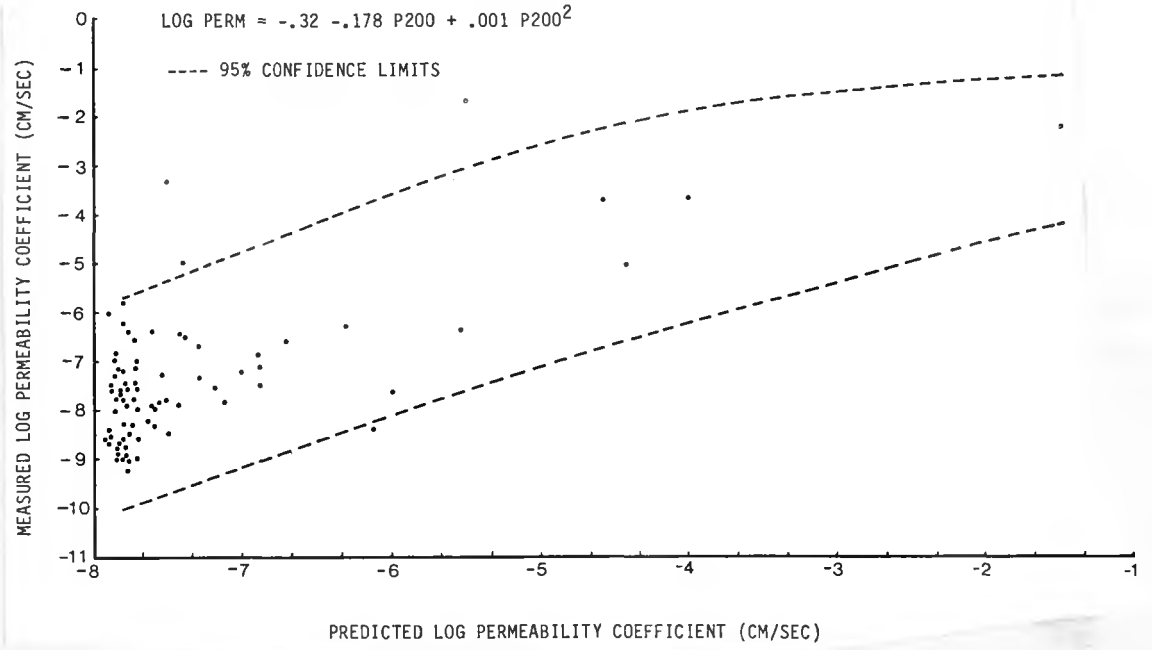

Figure 16. Scatter Diagram for the One Variable Permeability Model

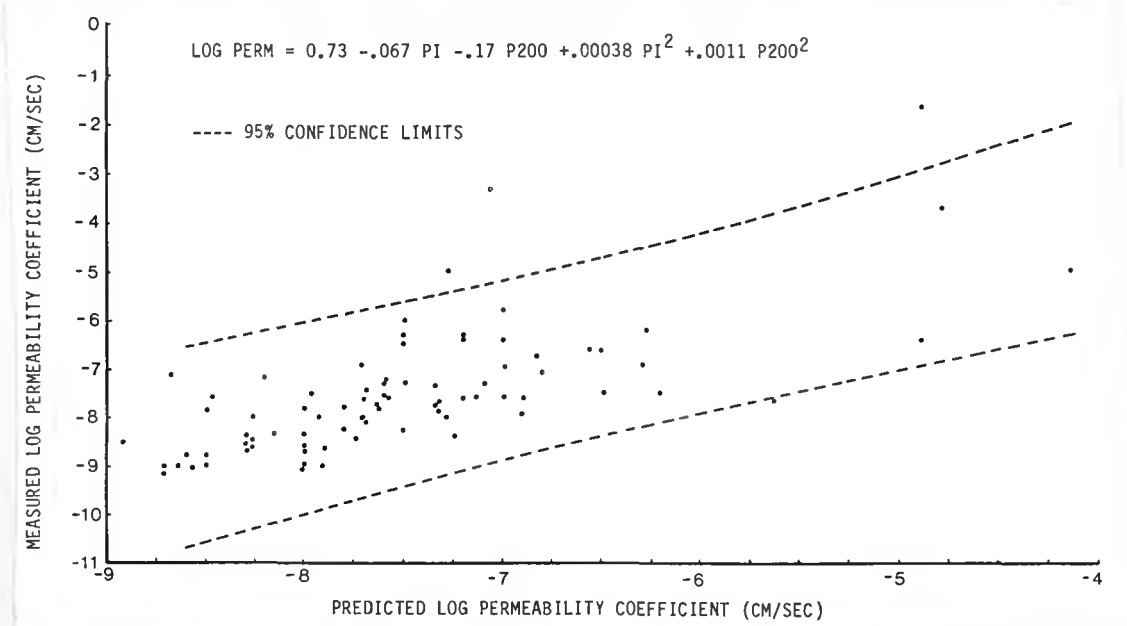

Figure 17. Scatter Diagram for the Two Variable Permeability Model

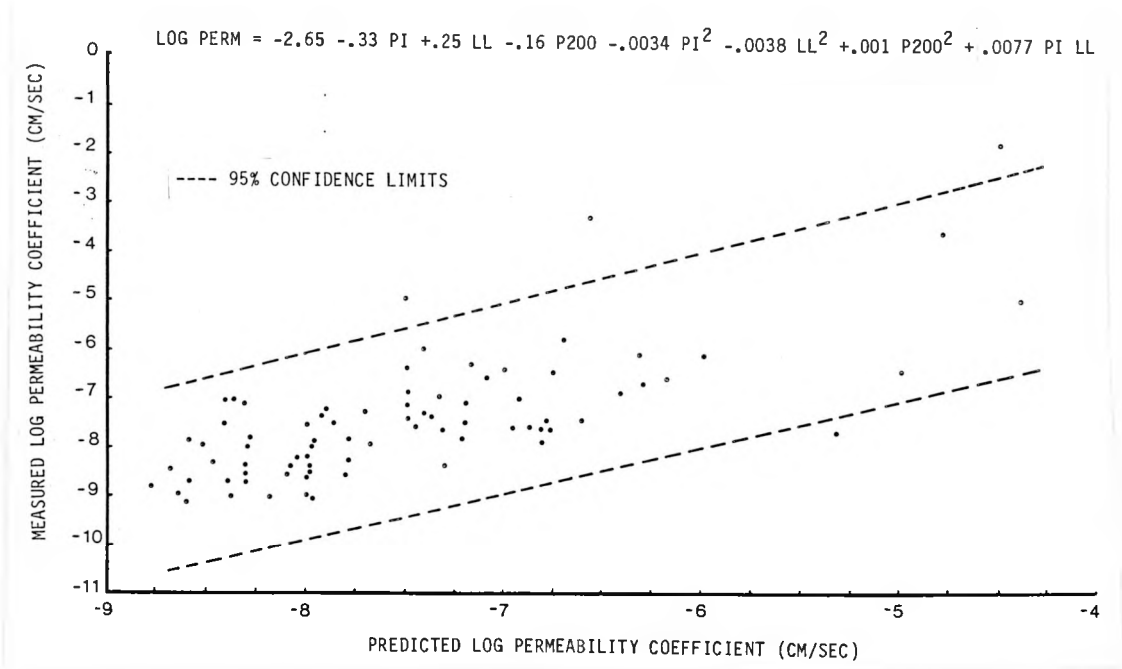

Figure 18. Scatter Diagram for the Three-Variable Permeability Model

to include additional chemicals and introducing a new extraction procedure to evaluate the Toxicity Characteristic. These changes to the solid waste regulations are being developed to meet a specific mandate of the Hazardous and Solid Waste Amendments of 1984.

The proposed changes to the Toxicity Characteristic evaluation procedure will 1) expand the characteristic to include 38 additional compounds, 2) revise the maximum allowable contaminant levels by applying compound-specific dilution/attenuation factors based on a groundwater transport model, and 3) introduce a second-generation leaching procedure, the Toxicity Characteristic Leaching Procedure (TCLP), which has been developed to address the mobility of both organic and inorganic compounds and to resolve the operational problems of the existing EP leaching method (3).

In response to the proposed changes to the soild waste regulations, a study was conducted to evaluate the use of the trace organic leaching procedure developed at the Morgantown Energy Technology Center (METC) to facilitate the use of follow-on TCLP leaching tests. The study was done by performing replicate leaching tests on two coal gasification tar samples using both the METC and TCLP procedures. The METC leaching tests were used to identify the various classes of nonvolatile trace organics in the waste leachates, and the TCLP tests were used to quantitatively identify the organics which had specific regulatory criteria. The results of the study indicated that the METC procedure was an effective means of screening the gasifier tar leachates for nonvolatile organic compounds.

6.1 Materials and Leaching Methods

6.1.1 Tar Samples Used for Leaching Tests

The two tar samples used for the METC and TCLP leaching tests were produced at UNDEMRC in a pilot-scale fixed bed coal gasifier using Indian Head lignite. The first sample tested was a "dry" tar which had been distilled to remove the light oil fraction. The second sample tested was an "oily" tar which had not been distilled. The light oil fraction contained significant amounts of volatile aromatic compounds such as benzene and toluene. Since benzene and toluene are both on the EPA's proposed list of regulated organic compounds, the "oily" tar sample was used to test the ability of the two leaching tests to detect this type of volatile organics.

6.1.2 The METC Leaching Procedure

For the METC procedure, an aqueous leachate is generated from the waste using distilled and deionized water without pH adjustment. The leaching procedure basically follows the ASTM D - 3987 method which uses a 1:4 solids-to-water ratio. The leachates thus produced are spiked with 20 mg/l each of 2-fluorophenol and azulene to check the efficiency of subsequent solvent extraction and analysis steps.

The organics are removed from the leachate by solvent extraction with methylene chloride. Both base and acid fractions are produced using appropriate leachate pH adjustment. The two solvent fractions are each reduced to ten milliliters using a Kuderna-Danish concentrator, and 20 ml/g of tetradecane is added to both of the concentrated fractions as an internal standard.

At UNDEMRC, the concentrated acid and base fractions were analyzed separately using a GC/MS equipped with a 60 meter x 0.32 mm ID, DB-5 capillary column. Hydrogen carrier gas was used at a flow rate of 21 cm/sec, and the samples were injected on-column at 350^{o}C.

The METC procedure recognizes the inherent diversity of the organic content of waste samples and notes that, in addition to the procedures described above, specialized procedures such as head space and liquid chromatography may be used to more completely evaluate a specific waste. A detailed description of the METC leaching procedure is given in Reference 4.

6.1.3 The TCLP Leaching Procedure

The TCLP procedure uses a leaching solution containing a sodium acetate buffer which has a pH of 5.0. The procedure also uses a 1:20 solids-to-water ratio and an 18-hour shake period. A detailed description of the TCLP procedure is contained in Reference 3.

Since the preceding METC leaching tests had already indicated that phenolics were the only types of regulated nonvolatile organics in the tar samples, each of the TCLP leachates was analyzed for phenolics by direct aqueous injection into a gas chromatograph equipped with a 30 meter x 0.32 mm,

OV-351 capillary column. Hydrogen was used as the carrier gas at a flow rate of 42 cm/sec. The aqueous leachate sample was injected on-column at 240°C.

In addition to the direct injection analyses, an alternate form of the TCLP called the zero-head-space extraction procedure was also performed on each of the gasifier tars because it was known that these samples contained volatile organics. The purpose of the zero-head-space procedure was to produce and analyze a leachate without allowing any of the volatile compounds to escape. The procedure used a specially designed, stainless steel extraction cylinder equipped with an internal piston. This type of cylinder permitted the waste to contact the leaching solution but did not permit the formation of a vapor phase. After the procedure was completed, the leachate sample was drawn from the extraction vessel into a gas-tight syringe and placed directly in a purge-and-trap apparatus. The purge-and-trap apparatus then injected the volatile organics into a GC/MS for analysis.

6.1.4 Leaching Test Evaluations

The METC and TCLP leaching procedures were evaluated by performing ten replicate tests with each of the procedures on the two different gasifier tar samples. Each tar sample was completely mixed before aliquots were taken for the leaching tests. In all, a total of 40 leaching tests were performed.

Quantitative estimates of the concentrations of the various organics in the tar leachates produced with the METC procedure were determined based on the relationship between the areas of the peaks measured for the identified compounds and the areas of the peaks measured for the fluorophenol and azulene spikes. For the METC procedure, only those organics which appeared to be present in the leachates at concentrations equal to or greater than the spikes (i.e., 20 mg/l) were included in lists of identified compounds.

For each of the tar samples tested with the TCLP, five replicates were done with the zero-head-space procedure and five replicates were done with the standard procedure. Each of the zero-head-space extracts was analyzed by purge-and-trap injection into a GC/MS equipped with the same column and carrier gas as were used to analyze the METC leachates. Deuterated standards were used to quantify the amounts of benzene and toluene in the leachates. The leachates produced with the standard TCLP procedure were analyzed for phenols and cresols using gas chromatography.

The analytical results from the replicate leaching tests were examined to evaluate the reproducibility of the METC and TCLP methods and the use of the METC procedure as a screening method for follow-on TCLP tests.

6.2 Leaching Test Results

The existing RCRA regulations specify maximum contaminant levels for six organic compounds in waste leachates. Four of the six are insecticides and the other two are herbicides. Therefore, none of the currently regulated organics would normally be present in coal utilization by-products. The proposed changes to the RCRA regulations specify maximum contaminant levels for 43 organic compounds. Most of the added organics are chlorinated solvents which would not normally be contained in gasifier tars; however, seven of the

new organics are nonchlorinated aromatics which could be present in significant amounts in gasifier tars. These seven compounds, along with their proposed regulatory maximum contaminant levels, are listed in Table 15.

The results of the METC leaching tests conducted on the "dry" and "oily" tar samples are listed in Tables 16 and 17, respectively. Only those compounds with leachate concentrations greater than 20 mg/l were included in the tables. All of the compounds detected in the leachates were aromatics, and the phenolics were present in the highest concentrations. The test results appear to be reasonable since these types of organics are normally found in fixed-bed coal gasification tars. The analyses obtained with the METC leaching procedure for the two tars were similar except that the dimethylphenols and naphthalene were detected only in the "dry" tar leachate.

The relative standard deviations listed for the various organics were based on the replicate leaching tests. Some of the compounds identified with the METC leaching test and listed in the Tables 16 and 17 were not detected in all of the replicates, but the two classes of regulated nonvolatile organics, the phenol and cresols, were detected in all of the replicate leaching tests. This means that the test had good reproducibility in terms of identifying the general classes of organics that should be examined with follow-on tests. In addition to identifying the types of regulated nonvolatile organics in the tars, the METC leaching tests identified several other types of toxic organics such as aniline and naphthalene, which are of general interest in evaluating the environmental impact of the waste.

TABLE 15

ORGANIC COMPOUNDS PROPOSED FOR REGULATION
WHICH MAY BE PRESENT IN GASIFIER TAR

Compound	Proposed Maximum Allowable Contaminant Level
Phenol	14.4
o-Cresol	10.0
m-Cresol	10.0
p-Cresol	10.0
Benzene	0.07
Toluene	14.4
Pyridine	5.0

TABLE 16

TRACE ORGANIC COMPOUNDS DETECTED IN THE GASIFIER "DRY" TAR METC LEACHATES

Compound	Number of Leachate Replicates In Which Compound Was Detected	Mean Estimated Leachate Concentration (mg/l) And Percent Standard Deviation
Phenol	10	300 ± 60%
o-Cresol	6	120 ± 27%
p-Cresol	10	342 ± 73%
2,3-Dimethyl Phenol	3	54 ± 81%
2,4-Dimethyl Phenol	2	60 ± 4%
2-Ethyl Phenol	8	124 ± 72%
3,5-Xylenol	8	60 ± 73%
Benzyl Alcohol	2	40 ± 28%
Naphthalene	2	64 ± 95%
Aniline	5	48 ± 64%

The methods used to analyze the METC leachates were intended specifically to detect nonvolatile organics, and therefore no volatile organics were detected in any of the METC leachates. The METC procedure notes that other analytical methods, such as head space analysis, may be necessary to evaluate some types of samples. It is probable that either the head space method or the purge-and-trap method of sample injection would produce good analytical results of volatile organics in gasifier tars when used in conjunction with the METC leaching procedure.

The results of the TCLP leaching tests performed on the "dry" and "oily" gasifier tar samples are listed in Tables 18 and 19, respectively. Six regulated organics were detected in each tar sample. The TCLP leaching method and the associated analyses appeared to do an excellent job of quantitatively identifying the phenolics which the METC method had indicated were present in the tar leachates. The results of the TCLP tests indicated that the "oily" tar leached higher levels of both nonvolatile and volatile organics. This finding was not unusual because the distillation operation used to produce the dry tar would remove some of the phenolics and most of the benzene and toluene.

TABLE 17

TRACE ORGANIC COMPOUNDS DETECTED IN THE GASIFIER "OILY" TAR METC LEACHATES

Compound	Number of Leachate Replicates In Which Compound Was Detected	Mean Estimated Leachate Concentration (mg/l) And Percent Standard Deviation
Phenol	10	392 ± 21%
p-Cresol	10	180 ± 42%
o-Cresol	9	219 ± 33%
m-Cresol	2	173 ± 13%
2 Ethyl Phenol	9	105 ± 41%
3,5 Xylenol	10	133 ± 37%
Benzyl Alcohol	6	77 ± 13%
Aniline	4	54 ± 26%

The levels of phenolics measured with the TCLP were generally higher and the relative standard deviations were lower than those measured with the METC leaching test, particularly for the oily tar sample. The results of either the METC or the TCLP leaching tests, however, would cause the gasifier tars to be classified as hazardous wastes based on the proposed maximum contaminant levels for phenol and cresols.

The zero-head-space form of the TCLP was found to be an effective means of measuring the volatile organics in the tar leachates.

6.3 Leaching Test Evaluation - Summary and Conclusions

The results of the replicate leaching tests performed on the two coal gasification tars indicated that the METC leaching procedure was an effective and reproducible means of screening the wastes to determine their potential environmental impacts due to leaching of nonvolatile trace organics. The acid and base methylene chloride extracts prepared from the tar leachates did not detect volatile organics, and it is suggested that either head space or purge- and trap-sample injection should be used with the METC precedure to identify volatile organics.

TABLE 18

TRACE ORGANIC COMPOUNDS DETECTED IN THE GASIFIER "DRY" TAR TCLP LEACHATES

Nonvolatile Compounds	Number of Leachate Replicates In Which Compound Was Detected	Mean Estimated Leachate Concentration (mg/l) And Percent Standard Deviation
Phenol	5	470 ± 47%
p-Cresol	5	368 ± 41%
o-Cresol	5	167 ± 51%
m-Cresol	5	319 ± 44%
Volatile Compounds		
Benzene	5	0.1 ± 53%
Tolulene	2	0.016 ± 56%

TABLE 19

TRACE ORGANIC COMPOUNDS DETECTED IN THE GASIFIER "OILY" TAR TCLP LEACHATES

Nonvolatile Compounds	Number of Leachate Replicates In Which Compound Was Detected	Mean Estimated Leachate Concentration (mg/l) And Percent Standard Deviation
Phenol	5	742 ± 16%
p-Cresol	5	503 ± 19%
o-Cresol	5	233 ± 21%
m-Cresol	5	442 ± 18%
Volatile Compounds		
Benzene	5	1.46 ± 12%
Toluene	5	0.68 ± 15%

The TCLP was found to be a highly reproducible means of quantitatively identifying trace organics in the tar leachates. The organics identified included phenol, cresols, benzene, and toluene. A comparative summary of the conclusions drawn concerning the two tests is listed in Table 20.

7.0 REFERENCES

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TABLE 20

SUMMARY OF THE FINDINGS FROM THE LEACHING TEST STUDY

METC Leaching Procedure	TCLP Leaching Procedure
Procedure is good for screening the leachate for the presence of various classes of trace organics. The test results were reproducible.	Procedure is good for quantifying specific trace organic compounds in the leachate, however the leachate must first be screened to determine the types of compounds to look for.
Procedure identifies a wide range of nonvolatile trace organics, including materials that are not specifically regulated but still are known to be toxic.	Levels of regulated organics measured with TCLP were generally substantially higher and had lower standard deviations than measurements obtained with the METC procedure. This is important when measurements are used to determine the regulatory status of a waste.
Purge-and-trap or equivalent sample injection method would have to be used with the METC procedure to effectively detect volatile organics.	Purge and trap sample injection used in conjunction with GC/MS analysis was an effective means of measuring volatile organics in gasifier tar leachates.

APPENDIX 1

EPA-EP LEACHING TEST RESULTS FOR THE AFBC SLURRY COMBUSTION WASTES

AFBC Waste Materials:

Element*	Bed Material	Primary Cyclone Ash	Secondary Cyclone Ash
Arsenic	<0.01	0.012	<0.01
Barium	0.67	0.38	0.3
Cadmium	<0.04	<0.04	<0.04
Chromium	<0.05	0.06	0.12
Lead	<0.6	<0.6	<0.6
Mercury	<0.002	<0.002	<0.002
Selenium	<0.02	<0.02	<0.02
Silver	<0.5	<0.5	<0.5

AFBC Waste Materials:

Element*	Baghouse Fly Ash	Composite Ash (No Limestone Added)	Composite Ash (Limestone Added)
Arsenic	<0.01	<0.01	<0.01
Barium	0.24	0.4	0.14
Cadmium	<0.04	<0.04	<0.04
Chromium	0.18	0.04	<0.05
Lead	<0.6	<0.6	<0.6
Mercury	<0.002	<0.002	<0.002
Selenium	<0.02	<0.02	<0.02
Silver	<0.5	<0.5	<0.5

* All elemental concentrations measured for the EP leachates have units of mg/l.

EPA-EP LEACHING TEST RESULTS FOR THE HYDROGEN PRODUCTION BED MATERIALS

Coal Gasification Waste Materials:

Element*	Texas Lignite Silica Sand Bed	Texas Lignite Limestone Bed
Arsenic	0.003	0.006
Barium	0.44	0.93
Cadmium	<0.05	<0.05
Chromium	<0.05	<0.05
Lead	<0.6	<0.6
Mercury	<0.0003	<0.0003
Selenium	0.003	0.008
Silver	<0.5	<0.5

Coal Gasification Waste Materials:

Element*	North Dakota Lignite Limestone Bed	Wyoming Subbituminous Coal Limestone Bed
Arsenic	<0.002	<0.002
Barium	14	5.5
Cadmium	<0.05	<0.05
Chromium	<0.05	<0.05
Lead	<0.6	<0.6
Mercury	<0.0003	<0.0003
Selenium	0.003	0.002
Silver	<0.5	<0.5

EPA-EP LEACHING TEST RESULTS FOR THE COAL SLURRY PREPARATION WASTE

Element*	Waste Leachate Concentration
Arsenic	0.011
Barium	2.5
Cadmium	<0.05
Chromium	<0.05
Lead	<0.6
Mercury	<0.001
Selenium	<0.002
Silver	<0.5

* All elemental concentrations measured for the EPA-EP leachates have units of mg/l.

ASTM D-3987 LEACHING TEST RESULTS FOR THE AFBC WASTES

AFBC Waste Materials:

Element*	Bed Material	Primary Cyclone Ash	Secondary Cyclone Ash
Aluminum	13	<0.6	55
Arsenic	<0.01	<0.01	<0.01
Barium	0.8	1.0	0.5
Boron	0.5	0.9	14
Cadmium	<0.04	<0.04	<0.04
Calcium	130	720	560
Chromium	<0.05	<0.05	0.3
Iron	<0.1	<0.1	<0.1
Magnesium	<2	<2	<2
Manganese	<0.05	<0.05	<0.05
Lead	<0.6	<0.6	<0.6
Potassium	<5	35	50
Selenium	<0.02	<0.02	<0.02
Silicon	4.6	0.9	0.9
Sodium	<5	15	28
Strontium	2.8	34	48
Sulfate	300	130	2500
pH	11.3	12.5	10.2

AFBC Waste Materials:

Element*	Baghouse Fly Ash	Composite Ash (No Limestone Added)	Composite Ash (Limestone Added)
Aluminum	42	<0.6	<0.6
Arsenic	<0.01	<0.01	<0.01
Barium	0.45	0.4	0.6
Boron	5.4	0.47	0.2
Cadmium	<0.04	<0.04	<0.04
Calcium	600	450	980
Chromium	0.42	0.07	0.13
Iron	<0.1	<0.1	<0.1
Magnesium	<2	<2	<2
Manganese	<0.05	<0.05	<0.05
Lead	<0.6	<0.6	<0.6
Potassium	100	49	76
Selenium	0.3	<0.02	<0.02
Silicon	1.0	4.3	<0.5
Sodium	80	28	32
Strontium	61	46	40
Sulfate	3000	1300	1200
pH	10.2	11.9	12.5

* All elemental concentrations for the ASTM D-3987 leachates have units of mg/l except pH.

ASTM LEACHING TEST RESULTS FOR THE HYDROGEN PRODUCTION BED MATERIALS

Coal Gasification Waste Materials:

Element *	Texas Lignite Silica Sand Bed	Texas Lignite Limestone Bed
Aluminum	8.7	<0.5
Arsenic	<1.0	<1.0
Barium	<0.03	1.3
Cadmium	<0.05	<0.05
Calcium	6.3	800
Chromium	<0.05	<0.05
Lead	<0.6	<0.6
Selenium	0.016	0.01
Silicon	47	<5.0
Sodium	280	10
Sulfate	30	60
pH	11.9	12.55

Coal Gasification Waste Materials:

Element*	North Dakota Lignite Limestone Bed	Wyoming Subbituminous Coal Limestone Bed
Aluminum	<0.5	<0.5
Arsenic	<1.0	<1.0
Barium	11	19
Cadmium	<0.05	<0.05
Calcium	800	760
Chromium	<0.05	<0.05
Lead	<0.6	<0.6
Selenium	0.005	<0.002
Silicon	<0.5	<0.5
Sodium	30	19
Sulfate	<10	<10
pH	12.7	12.7

* All elemental concentrations measured for the ASTM leachates have units of mg/l.

ASTM LEACHING TEST RESULTS FOR THE COAL SLURRY PREPARATION WASTE

Element*	Waste Leachate Concentration
Aluminum	<0.5
Arsenic	<1.0
Barium	0.06
Cadmium	<0.05
Calcium	1.3
Chromium	<0.05
Lead	<0.6
Selenium	0.003
Silicon	1.7
Sodium	180
Sulfate	300
pH	7.3

* All elemental concentrations measured for the ASTM leachates have units of mg/l.

ELEMENTAL COMPOSITIONS OF THE AFBC WASTES

AFBC Waste Materials:

Element*	Bed Material	Primary Cyclone Ash	Secondary Cyclone Ash
Silicon	43.0%	23.8%	19.6%
Aluminum	2.1%	7.4%	10.3%
Iron	0.2%	3.1%	2.7%
Calcium	0.9%	11.1%	15.1%
Magnesium	0.2%	1.4%	2.3%
Sodium	0.0%	0.0%	0.3%
Potassium	0.0%	0.8%	1.4%
Sulfur	0.1%	1.4%	2.3%
Phosphorous	0.0%	0.1%	0.2%
Titanium	0.1%	0.5%	0.7%
Chlorine	64	<20	<20
Chromium	21	70	81
Manganese	20	211	280
Nickel	<10	<10	<10
Copper	23	20	23
Zinc	19	10	22
Arsenic	<20	<20	36
Barium	157	1230	839
Strontium	181	1710	3140

AFBC Waste Materials:

Element*	Baghouse Fly Ash	Composite Ash (No Limestone Added)	Composite Ash (Limestone Added)
Silicon	15.7%	18.2%	17.8%
Aluminum	9.8%	10.7%	10.1%
Iron	2.8%	2.8%	2.5%
Calcium	17.8%	15.8%	18.4%
Magnesium	2.6%	2.3%	2.0%
Sodium	0.7%	0.3%	0.0%
Potassium	1.3%	1.3%	1.3%
Sulfur	4.3%	2.8%	3.0%
Phosphorous	0.3%	0.2%	0.2%
Titanium	0.8%	0.7%	0.6%
Chlorine	<20	<20	<20
Chromium	95	79	76
Manganese	264	244	247
Nickel	<10	<10	<10
Copper	24	23	23
Zinc	21	21	16
Arsenic	44	37	27
Barium	652	736	669
Strontium	3490	3230	2590

* Elemental concentrations for the waste materials are in units of either % of dry weight or ≈g/gram waste.

ELEMENTAL COMPOSITIONS OF THE HYDROGEN PRODUCTION BED MATERIALS

Coal Gasification Waste Materials:

Element*	Texas Lignite Silica Sand Bed	Texas Lignite Limestone Bed
Silicon	34.5%	2.4%
Aluminum	1.2%	0.5%
Iron	0.6%	0.7%
Calcium	6.9%	54.5%
Magnesium	0.2%	0.3%
Sodium	2.4%	<0.1%
Potassium	<0.1%	<0.1%
Sulfur	0.7%	0.6%
Phosphorous	<0.01%	0.02%
Titanium	0.07%	0.06%
Arsenic	<20	<20
Barium	250	140
Copper	<10	<10
Chromium	<20	<20
Manganese	40	110
Nickel	<10	<10
Strontium	300	210
Zinc	<10	<10

Coal Gasifiacation Waste Materials:

Element*	North Dakota Lignite Limestone Bed	Wyoming Subbituminous Coal Limestone Bed
Silicon	2.8%	2.6%
Aluminum	1.0%	1.2%
Iron	1.3%	1.6%
Calcium	58.2%	58.6%
Magnesium	0.8%	0.9%
Sodium	<0.1%	<0.1%
Potassium	<0.1%	<0.1%
Sulfur	0.1%	0.07%
Phosphorous	0.02%	0.02%
Titanium	0.1%	0.1%
Arsenic	<20	<20
Barium	710	460
Copper	30	<10
Chromium	<20	<20
Manganese	240	240
Nickel	<10	<10
Strontium	310	360
Zinc	<10	<10

* Elemental concentrations for the waste materials are in units of either % of dry weight or micrograms/gram of waste. All analyses were done by X-ray fluorescence.

ELEMENTAL COMPOSITION OF THE COAL SLURRY PREPARATION WASTE

Element*	Float-Sink Waste Composition
Silicon	2.8%
Aluminum	1.1%
Iron	2.0%
Calcium	5.7%
Magnesium	0.4%
Sodium	<0.1%
Potassium	<0.1%
Sulfur	1.5%
Phosphorous	0.1%
Titanium	0.44%
Arsenic	<5
Barium	560
Copper	6
Chromium	<10
Manganese	18
Nickel	<5
Strontium	440
Zinc	<5

* Elemental concentrations for the waste materials are in units of either % of dry weight or micrograms/gram of waste. All analyses were done by X-ray fluorescence.

COLUMN LEACHING TEST RESULTS FROM THE AFBC COMPOSITE ASHES

Ash Produced Without Limestone Addition

Pore Volume Number	Leachate Concentration (mg/l)						
	Ca	Na	Mg	Al	Ba	Cr	SO_4
1	390	170	<1.0	<0.3	<0.03	<0.05	2305
4	180	150	<1.0	<0.3	1.3	<0.05	2000
6	140	100	<1.0	1.0	2.2	<0.05	<10
8	230	22	<1.0	0.89	2.9	<0.05	<10
10	240	13	<1.0	0.89	2.4	<0.05	<10

Ash Produced With Limestone Addition

Pore Volume Number	Leachate Concentration (mg/l)						
	Ca	Na	Mg	Al	Ba	Cr	SO_4
1	1400	220	<1.0	<0.3	<0.03	<0.05	2300
4	1300	110	<1.0	<0.3	<0.03	<0.05	1875
6*	260	280	<1.0	<0.3	1.6	<0.05	<10

* It was not possible to collect more than six pore volumes due to the low permeability of the sample.

MINERAL COMPOSITIONS OF THE AFBC WASTES

Sarpy Creek Bed Material:

Major phases - Quartz (SiO_2)

Sarpy Creek Primary Cyclone Ash:

Major phases - Quartz (SiO_2)

Trace phases - Anhydrite ($CaSO_4$), Melilite ($Ca_2Al_2SiO_7$)

Sarpy Creek Secondary Cyclone Ash:

Major phases - Quartz (SiO_2), Melilite ($Ca_2Al_2SiO_7$)

Sarpy Creek Baghouse Fly Ash:

Major phases - Quartz (SiO_2), Anhydrite ($CaSO_4$), Melilite ($Ca_2Al_2SiO_7$)

Sarpy Creek Composite Ash (No Limestone Addition):

Major phases - Quartz (SiO_2), Anhydrite ($CaSO_4$), Melilite ($Ca_2Al_2SiO_7$)

Sarpy Creek Composite Ash (Limestone Addition):

Major phases - Quartz (SiO_2), Anhydrite ($CaSO_4$), Melilite ($Ca_2Al_2SiO_7$)

MINERAL COMPOSITIONS OF THE HYDROGEN PRODUCTION BED MATERIALS

Texas Lignite - Silica Sand Bed:

Major Phases: Quartz (SiO_2)

Texas Lignite - Limestone Bed:

Major Phases: Calcite ($CaCO_3$), Lime (CaO)

Minor Phases: $Ca(OH)_2$

Trace Phases: Quartz (SiO_2)

North Dakota Lignite - Limestone Bed

Major Phases: Calcite ($CaCO_3$), Lime (CaO)

Minor Phases: $Ca(OH)_2$

Trace Phases: Quartz (SiO_2)

Wyoming Subbituminous Coal - Limestone Bed

Major Phases: Calcite ($CaCO_3$), Lime (CaO)

Minor Phases: $Ca(OH)_2$

Trace Phases: Quartz (SiO_2)

PHYSICAL PROPERTIES OF THE AFBC WASTES

AFBC Waste Materials:

	Bed Material	Primary Cyclone Ash	Secondary Cyclone Ash
Permeability Coefficient (cm/sec)	1.3×10^{-1}	5.3×10^{-5}	1.3×10^{-3}
Bulk Density (gm/ml)	2.6	2.0	2.2
Specific Surface Area (m^2/gm)	0.07	5.24	3.27
Loss On Ignition (Wt%)	0.0	1.5	0.25

AFBC Waste Materials:

	Baghouse Fly Ash	Composite Ash (No Limestone Added)	Composite Ash (Limestone Added)
Permeability Coefficient (cm/sec)	2.1×10^{-3}	2.7×10^{-5}	6.3×10^{-5}
Bulk Density (gm/ml)	2.1	2.0	2.1
Specific Surface Area (m^2/gm)	4.84	3.77	2.8
Loss On Ignition (Wt%)	0.84	0.5	0.5

PHYSICAL PROPERTIES OF THE HYDROGEN PRODUCTION BED MATERIALS

Coal Gasification Waste Materials:

Physical* Property	Texas Lignite Silica Sand Bed	Texas Lignite Limestone Bed
Permeability Coefficient (cm/sec)	7.0×10^{-3}	6.2×10^{-3}
Bulk Density (gm/ml)	3.3	2.1
Specific Surface Area (m^2/gm)	2.37	2.33
Loss On Ignition (Wt%)	2.49	30.1

Coal Gasification Waste Materials:

Physical* Property	North Dakota Lignite Limestone Bed	Wyoming Subbituminous Coal Limestone Bed
Permeability Coefficient (cm/sec)	4.8 x 10-6	6.0 x 10-6
Bulk Density (gm/ml)	2.32	2.30
Specific Surface Area (m^2/gm)	4.8	7.23
Loss On Ignition (Wt%)	13.8	12.1

PHYSICAL PROPERTIES OF THE COAL SLURRY PREPARATION WASTE

Physical* Property	Beulah-Zap North Dakota Lignite
Permeability Coefficient (cm/sec)	7.0×10^{-3}
Bulk Density (gm/ml)	1.38
Specific Surface Area (m^2/gm)	ND*
Loss On Ignition (Wt%)	87.6

* Reproducible test results could not be obtained for this sample.

APPENDIX 2

SOILS DATA SET

OBS.#	Site	Re-Comp* or Not Re	Soil Class	Dry Density (#/ft^3)	Liquid Limit (%)	Plasticity Index (%)	Moisture Content (%)	% Passing #200	log Perm (cm/sec)
1	A	NR	CL	.	41	24	18	64	-6.42
2	A	NR	CH	.	53	29	21	.	-7.67
3	A	NR	CH	.	67	24	25	46	-6.14
4	A	NR	CL-ML	.	45	19	24	61	-6.70
5	A	NR	CL	.	43	21	24	69	-6.40
6	A	NR	CL	.	33	11	24	37	-1.67
7	A	NR	CH	.	54	29	27	88	-6.00
8	A	NR	CL	.	44	26	16	63	-4.96
9	A	NR	CL	.	47	32	15	61	-8.23
10	A	NR	CL	.	44	24	25	29	-3.72
11	A	NR	CL	.	49	33	20	70	-7.80
12	A	NR	CL-ML	.	48	21	22	93	-5.77
13	A	NR	CL	.	48	21	22	67	-3.31
14	A	NR	CH	.	106	86	23	44	-8.39
15	B	NR	CH	90	54	31	30	.	-7.08
16	B	NR	CL	80	48	28	40	.	-6.62
17	B	NR	SC	79	36	13	26	.	-4.77
18	B	NR	SC	103	42	24	21	.	-8.10
19	B	NR	CH	92	53	30	25	.	-7.47
20	B	NR	CH	88	74	52	29	.	-7.32
21	B	NR	CH	84	55	33	27	.	-6.16
22	B	NR	SM	89	34	8	20	.	-4.96
23	B	NR	CL	97	39	23	18	55	-7.09
24	B	NR	CH	86	55	30	26	64	-6.58
25	B	NR	CH	92	62	39	31	75	-9.05
26	B	NR	CH	92	65	40	29	71	-8.60
27	B	NR	SC	80	36	10	30	.	-7.54
28	B	NR	CL	99	48	28	26	74	-7.27
29	B	NR	CH	71	59	32	40	.	-7.01
30	B	NR	CH	82	57	29	35	86	-7.49
31	B	NR	CH	87	68	44	32	93	-8.60
32	B	NR	CH	91	71	47	28	85	-8.70
33	B	NR	CL	92	40	18	22	.	-7.44
34	B	NR	CH	91	53	27	26	.	-6.92
35	B	NR	CH	92	69	46	29	77	-8.38
36	B	NR	CH	85	61	38	34	88	-8.64
37	B	NR	CH	92	54	34	28	70	-8.25
38	B	NR	CH	82	64	40	33	.	-7.77
39	B	NR	CH	94	67	45	26	73	-8.01
40	B	NR	CH	92	54	34	28	77	-9.00
41	B	NR	CH	86	70	46	34	82	-8.60

* Re-Comp - Recompacted soil samples used for permeability tests.
Not Re - Nonrecompacted soil samples used for permeability tests.

SOILS DATA SET (CONT.)

OBS.#	Site	Re-Comp* or Not Re	Soil Class	Dry Density (#/ft^3)	Liquid Limit (%)	Plasticity Index (%)	Moisture Content (%)	% Passing #200	log Perm (cm/sec)
42	B	NR	CH	93	60	39	26	75	-8.96
43	B	NR	CH	80	59	36	32	77	-7.52
44	C	NR	CH	97	52	28	27	80	-7.29
45	C	NR	CH	94	52	26	30	78	-6.33
46	C	NR	CH	105	56	35	19	90	-8.05
47	C	NR	SC	114	40	18	18	91	-7.57
48	C	NR	CL-CH	102	45	20	24	90	-7.57
49	C	NR	CL	102	46	25	23	87	-7.64
50	C	NR	CL	96	45	16	25	96	-6.60
51	C	NR	CL	98	45	20	25	77	-7.57
52	C	NR	CL	100	42	19	24	90	-7.00
53	C	NR	CL	97	47	20	26	89	-6.40
54	C	NR	CL	102	45	21	25	96	-7.89
55	C	NR	CL	98	52	29	26	85	-7.57
56	C	NR	CL	108	41	21	21	75	-8.39
57	C	NR	CL	107	58	39	21	84	-8.42
58	C	NR	CL-CH	101	61	39	20	96	-7.42
59	C	NR	CH	99	56	32	27	99	-7.00
60	C	NR	CL	99	47	24	26	89	-7.82
61	D	NR	.	.	30	13	.	55	-6.92
62	D	NR	.	.	114	79	.	98	-7.60
63	D	NR	.	.	140	114	.	99	-7.22
64	D	NR	.	.	101	71	.	88	-9.24
65	D	NR	.	.	103	70	.	91	-9.00
66	D	NR	CH	85	65	32	28	54	-7.52
67	D	NR	CH	84	97	62	31	75	-7.12
68	D	NR	CH	94	108	80	25	71	-8.51
69	D	NR	CH	94	120	90	31	96	-8.85
70	D	NR	CH	93	145	113	29	75	-9.07
71	D	NR	CH	81	93	67	36	95	-7.92
72	D	NR	CH	88	78	51	29	66	-8.47
73	D	NR	CL-CH	101	73	50	21	89	-8.72
74	D	NR	CL	96	111	85	27	98	-9.03
75	D	NR	CH	102	73	53	20	74	-8.77
76	D	NR	OH	67	58	3	28	74	-7.47
77	D	NR	CH	90	130	106	29	92	-8.96
78	D	NR	CL	113	30	14	12	58	-7.47
79	D	NR	SM	106	.	.	9	24	-3.37
80	D	NR	SM	99	.	.	12	7	-2.21
81	E	RE	CH	100	52	33	20	68	-7.96
82	E	RE	SC	100	39	17	22	42	-7.66
83	E	RE	CL	97	44	22	24	51	-6.62
84	E	RE	CH	96	56	30	24	60	-7.74
85	E	RE	CH	92	66	43	26	71	-8.40
86	E	RE	CH	93	53	32	26	69	-8.00

SOILS DATA SET (CONT.)

OBS.#	Site	Re-Comp* or Not Re	Soil Class	Dry Density (#/ft^3)	Liquid Limit (%)	Plasticity Index (%)	Moisture Content (%)	% Passing #200	log Perm (cm/sec)
87	E	RE	.	94	53	29	28	81	-7.20
88	E	RE	.	90	56	31	26	80	-6.89
89	E	RE	.	94	55	35	26	90	-7.58
90	E	RE	.	98	42	28	22	57	-7.25
91	E	RE	.	96	43	23	23	81	-7.35
92	E	RE	.	88	55	29	28	82	-7.70
93	E	RE	.	91	54	37	28	84	-8.00
94	E	RE	CL	93	64	39	24	72	-7.80
95	E	RE	CL	96	.	.	23	.	-7.89
96	E	RE	CH	84	.	.	30	.	-8.00
97	F	RE	CL	117	37	18	16	.	-8.16
98	F	RE	CL	123	29	12	17	.	-7.09
99	F	RE	CL	105	38	19	17	.	-7.66
100	F	RE	CL	121	34	15	17	.	-7.39
101	F	RE	CL	118	29	11	18	.	-7.55
102	F	RE	CL	100	35	17	17	.	-7.82
103	F	RE	CL	121	35	17	16	.	-7.70
104	F	RE	CL	.	27	10	.	38	-6.42
105	F	RE	CL	.	29	12	.	29	-5.05