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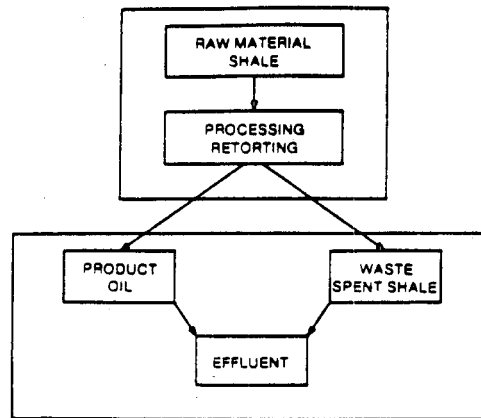


Figure 1. Research Philosophy.

There is general agreement that retorting of oil shale presents problems regarding solid waste disposal and water contamination. Leachates from spent shales, as well as contaminated process waters, present the potential for environmental degradation whose magnitude will be closely linked to the commercial shale oil process utilized. Studies concerning the characterization of potential contaminants in various shales, shale wastes, or waste waters have been conducted; however, much of this information is not applicable to the development of predictive capabilities. A better understanding of the fundamental chemical principles operative during generation of effluents and leachates should provide a realistic framework for assessment of water management and solid waste management problems. Further research into the water quality and solid waste problems associated with recovery of shale oil is required so that control technology requirements can be specified.

### 3. OBJECTIVES:

The objectives of this research are to determine the nature, magnitude, and time dependence of the major and trace element releases as functions of the raw shale mineralogy, retorting conditions, and spent shale mineral assemblages. These experimental studies will focus on retorting variable regimes characteristic of most retorting processes. As an adjunct objective, the relation of laboratory results to those obtained from both bench-scale and pilot-scale retorts, when both have been operated under similar retorting conditions, will be defined. The goal is to develop a predictive capability for spent shale chemistry as a function of the raw material feedstock and process parameters.

### 4. PROJECT DESCRIPTION:

Our experimental program is being conducted to acquire quantitative information about the nature, magnitude, and time dependence of the organic compound, major, and trace element releases as functions of the raw shale mineralogy, retorting conditions, and spent shale mineral assemblages. These studies will continue for western shales from the Green River formation, but the same rationale and scientific approach are applicable to

eastern shales from regions being considered for development. In addition, the influence of the chemical and mineralogical properties of solid spent shales on the behavior and control of other effluent streams codisposed with the solid wastes is crucial for defining environmental behavior. These experimental studies focus on retorting variable regimes that include parameters characteristic of most retorting processes.

To accomplish these goals, several parallel experimental activities will be performed: (1) characterization of the raw and spent shales to gain a more detailed understanding of the effects of retorting on the mineral, trace element, and organic contents of the shales; (2) laboratory heating experiments using controlled conditions of time, temperature, and atmosphere to establish correlations of retorting parameters, mineralogy, and trace element content of these laboratory samples; and (3) laboratory leaching experiments designed to ascertain the composition of leachates and kinetic and mechanistic aspects of leachate generation. Determining the relationships among spent shales generated from laboratory-scale, pilot-scale, and field retorts will be approached by using a small pilot-scale retort capable of simulating various retorting processes under conditions of physical and chemical control that are more rigorous than now possible.

The importance of solids characterization in the assessment of health and environmental consequences of oil shale processing has been recognized. Such information is essential for understanding water quality impacts from energy development. Aided by solids characterization information, various issues can be explored: (1) the role of process parameters in the formation of wastes and effluents; (2) mineral/water interaction during leaching and/or weathering; (3) the influence of the microstructure of the solids on the leaching process; (4) the availability of major ions for solubility control determined by spent mineral assemblages; and (5) trace element residences and their role in contaminant mobility.

The interaction between shale and water to produce leachates is a complicated process that depends on both the nature of the shale and the quality of the leaching medium. In addition, depending on the type of contact that occurs, both thermodynamic and kinetic considerations must be examined to understand the chemistry and composition of these aqueous solutions. Our approach to this research area is to understand shale/-water reactions in the context of the interactions of the raw material and the retorting process. These are the independent variables that determine the mineral assemblages in the spent shale; these mineral assemblages ultimately define the composition of leachates generated from contact with the shale materials.

## 5. RESULTS:

Characterization studies of both raw and spent shales are being carried out by a number of techniques, including x-ray diffraction, scanning electron microscopy and energy dispersive x-ray spectrometry, major and trace element compositions by neutron activation analysis, and the leaching behavior of the solids by a standard batch leaching procedure (this is used as a solids characterization tool). Several raw shale cores from development areas of the Piceance Basin have been investigated.

These studies indicated the importance of the minor accessory minerals of the assemblage in determining the environmental behavior of the raw material, as well as the behavior of the spent shales produced from retorting. A variety of spent shales from laboratory-scale, pilot-scale, and field retorts have been characterized. These studies suggested the relative importance of process variables in determining the mineral reactions that occur during processing and the subsequent environmental behavior of the material after disposal. These classical characterization techniques will continue to be used to explore the major and minor minerals present in raw shales, to study the interaction of the raw material and the process in the generation of the spent shale solids, and to investigate the identity and behavior of the spent shale minerals present as a result of specific process variables.

A new capability being developed at Los Alamos is the proton microprobe. This instrumentation is used for proton-induced x-ray emission spectrometry (PIXE), which can characterize trace element residences in mineral grains. This technique, which is more sensitive than the electron microprobe, can detect trace constituents in minerals at bulk concentration levels of 10 ppm or more. Survey work to determine the applicability of this technique to the oil shale matrix has been accomplished, and the preliminary results are promising. Further work with this technique is planned. The main focus of this work is to determine the trace element chemistry occurring during shale oil extraction and processing as a function of fundamental process variables and the environment of the material in the original matrix. This information is essential to understanding the environmental behavior of products, wastes, and effluents from oil shale processing.

The high-temperature behavior of mineral species present in the oil shale matrix currently is being investigated. Laboratory experiments designed to investigate the interaction of the raw material and important process parameters in the formation and behavior of the solid waste have been accomplished. Mineralogic changes during processing are influenced by the environment of the matrix constituents in the raw material and by the process variables. An understanding of this reaction chemistry is requisite to understanding environmental behavior.

An experiment was conducted to explore the influences of shale type, temperature, and gas atmosphere on the mineralogy and leachability of spent shale. The study involved five shales from the Green River formation in Colorado, four atmospheres (air, nitrogen, CO<sub>2</sub>, and CO), and three temperatures (600, 800, and 1000°C) spanning the range expected for oil shale retorting. The temperatures were selected because different mineral reactions were expected at each. In this design, temperature is a qualitative variable as are atmosphere and shale type. Additionally it was expected that mineral reactions at a given temperature or for a given shale type might differ depending on atmosphere. Thus, a full factorial design was used. Retorted shales were analyzed by x-ray diffraction and extracted by a standard aqueous leaching procedure. Aqueous leachates were analyzed for inorganic chemical species.

A full factorial design for three qualitative variables (five shales, four atmospheres, and three temperatures) requires  $5 \times 4 \times 3 = 60$  experiments.

Some of these were replicated to determine the experimental error. The data were analyzed by a multiway analysis of variance, assuming the possibility of two-way interactions by using the general linear model procedure contained in the Statistical Analysis System (SAS).

The raw mineralogy data consist of peak heights of the major reflection of each mineral type, as discussed in the experimental section. These peak heights are approximately linearly related to the concentration of the mineral in the matrix; however, standards for calibration are not available. For our purposes, absolute concentrations are not necessary as we are mainly interested in the relative changes that occur from experiment to experiment. In similar matrices, relative peak heights provided the comparisons we desire. Summary results for the general linear model analysis of variance of the mineralogy data are listed in Table 1.

Table 1. General Linear Model Summary: SAS Analysis of Variance Shale Mineralogy.

Mineral	R <sup>2</sup> <sup>a</sup>	F <sup>b</sup>	p <sup>c</sup>	Shale <sup>d</sup>	Atm	Temp
Quartz	0.99	56.2	0.0001	0.0001	0.100	0.0001
Calcite	0.97	19.7	0.0001	0.0001	0.0016	0.0001
Dolomite	0.91	7.12	0.0001	0.0033	0.147	0.0001
Albite	0.98	29.8	0.0001	0.0001	0.458	0.0001
Orthoclase	0.84	3.57	0.0009	0.0001	0.812	0.0001
Analcime	0.96	17.4	0.0001	0.0001	0.105	0.0001
Illite	0.91	6.83	0.0001	0.0001	0.398	0.0001
Periclase	0.88	4.97	0.0001	0.0001	0.074	0.0001
Wollastonite	0.86	4.29	0.0002	0.0001	0.323	0.0001
Akermanite	0.97	19.9	0.0001	0.0001	0.341	0.0001
Diopside	0.97	22.6	0.0001	0.012	0.0072	0.0001
Merwinite	0.85	3.76	0.0006	0.0001	0.23	0.0043
Leucite	0.82	3.14	0.0023	0.0002	0.848	0.0008
Monticellite	0.89	5.40	0.0001	0.0001	0.737	0.0001
Magnetite	0.89	5.36	0.0001	0.0178	0.0001	0.0001
Hematite	0.86	4.20	0.0002	0.749	0.0001	0.0033
Anhydrite	0.95	13.7	0.0001	0.0001	0.0001	0.0081
Hauyne	0.85	3.92	0.0004	0.427	0.0002	0.0006
Troilite	NS					
Feldspars	0.91	7.32	0.0001	0.0001	0.791	0.0001

<sup>a</sup>R<sup>2</sup> indicates fit to the model. <sup>b</sup>F-test statistic. <sup>c</sup>Probability that the F-test is significant. <sup>d</sup>Probability that each variable type is significant in the model.

Each shale type consists of a mineral assemblage that, although not without some variation within each shale, is characterized by its own particular mineralogical composition. Thus, the analysis of variance indicates the importance of the mineralogy of the spent shale minerals that survived the retorting, as well as the minerals that are products of the solid-state reactions that occur during retorting. Table 1 indicates the importance of shale type for describing the variance in each raw shale



mineral response. Spent shale minerals resulting from conversion reactions during retorting also can be influenced by raw shale composition because the solid-state reactions in the mineral matrix are governed by stoichiometry. Excesses or deficiencies of reactants, such as quartz and carbonates, will determine the range of possible reaction products.

Results from the general linear model analysis of variance indicate that atmosphere is much less important in determining spent shale mineralogy than is shale type or temperature. The ANOVA indicates that atmosphere is significant only for the minerals calcite, diopside, magnetite, hematite, anhydrite, and hauyne. No significance is indicated for any other minerals as suggested by data in Table 1. The air atmosphere enhances formation of diopside, magnetite, hematite, anhydrite, and hauyne. The CO<sub>2</sub> atmosphere promotes the formation of magnetite, probably by favoring partial oxidation of Fe(II). The CO<sub>2</sub> also inhibits the decomposition of calcite by discouraging CO<sub>2</sub> evolution.

Temperature is the most important independent variable controlling the minerals that survive, disappear, or form as a result of retorting. It is instructive to discuss the trends with temperature in the context of similar mineral behaviors. The raw shale minerals quartz, albite, and orthoclase exhibit similar behaviors. This behavior is consistent with metamorphic silication reactions and feldspar decomposition reactions that occur at high temperature. Calcite, dolomite, analcime, and illite also decrease with increasing temperature but are completely decomposed or reacted by 800°C, whereas the quartz and feldspars still exist at that temperature. Thus, we observe little change in calcite and other minerals between 800 and 1000°C.

The spent shale minerals also are significantly influenced by temperature. The intermediate product periclase begins to appear at 600°C, has a maximum at 800°C, and has essentially disappeared by 1000°C. Other intermediate product silicates, wollastonite and merwinite, also have a maximum at 800°C, but they still have significant concentrations at 1000°C. The main product metamorphic minerals akermanite, diopside, leucite, and monticellite are most evident at 1000°C. Akermanite also is present in 800°C experiments, suggesting that this silicate is forming at lower temperatures than are diopside, leucite, and monticellite. The iron oxides, hematite and magnetite, also have maxima at 800°C, indicating that these phases are being incorporated into the metamorphic silicates at the higher temperatures by reactions in the solid-state matrix.

The multiway ANOVA also analyzes two-way interactions among the independent variables and indicates where significant interactions are occurring. For the raw shale minerals quartz, calcite, dolomite, albite, orthoclase, analcime, and illite, the interaction between shale type and temperature is significant. It is most significant for albite, analcime, and calcite, probably because of the complete lack of these minerals at 1000°C and the variable amount in the original raw shale. The interaction between atmosphere and temperature is important for calcite because of the influence of CO<sub>2</sub> on the decomposition of this mineral. The interaction between shale and temperature also is significant for the spent shale minerals wollastonite, akermanite, diopside, merwinite, leucite, monticellite, and the

mixed feldspars. This indicates the importance of temperature and stoichiometry on the solid-state metamorphic silicate formation reactions that occur during heating. The interaction of shale and atmosphere is important for hematite and anhydrite, indicating the influence of the air atmosphere and stoichiometry on the formation of these minerals. The interaction of atmosphere and temperature is important in the formation of diopside and magnetite. In the case of diopside, the combination of the air atmosphere and 1000°C favors diopside formation.

The spent shale leaching data for the 60 experiments will be published in a journal article that has been submitted. Table 2 shows results of calculations of the SAS general linear model (analysis of variance) for the various soluble species analysed in the study.

Table 2. General Linear Model Summary: SAS Analysis of Variance Elemental Mobilities.

Element	R <sup>2</sup> <sup>a</sup>	F	P	Shale	Atm	Temp
Mg	0.83	3.37	0.0014	0.05	0.0002	0.002
Si	NS			0.057	0.0073	
B	0.77	2.28	0.019	0.013	0.013	0.085
Cu	NS					
Cd	0.77	2.33	0.0166	0.0001	0.0884	0.363
Ca	0.80	2.66	0.0071	0.0762	0.0001	0.0068
Tl	0.79	2.53	0.0100	0.0046	0.136	0.0586
Fe	0.77	2.23	0.0215	0.298	0.0019	0.0048
Mo	0.93	9.76	0.0001	0.0001	0.0001	0.0050
Mn	0.75	2.10	0.0301	0.0007	0.187	0.0143
V	0.91	7.20	0.0001	0.0005	0.0001	0.0001
Pb	0.86	4.17	0.0003	0.0004	0.0001	0.0002
Co	0.82	3.06	0.0028	0.0036	0.0004	0.0005
Al	0.82	3.09	0.0026	0.0173	0.0001	0.1628
Ba	0.77	2.32	0.0170	0.0195	0.0058	0.0035
Sr	0.81	2.71	0.0152	0.0270	0.0074	0.0017
K	0.90	6.29	0.0001	0.0001	0.0001	0.0746
Ni	NS					
Li	0.81	2.88	0.0042	0.0011	0.0006	0.0600
Na	0.96	14.5	0.0001	0.0001	0.0001	0.0001
pH	0.93	8.82	0.0001	0.0001	0.0001	0.0001
Cond.	0.78	2.40	0.014	0.4241	0.0001	0.0002
F <sup>-</sup>	NS			0.0595		
Cl <sup>-</sup>	0.92	6.62	0.0001	0.0001	0.0123	0.0005
SO <sub>4</sub> <sup>+2</sup>	0.95	11.73	0.0001	0.0156	0.0001	0.0009
CO <sub>3</sub> <sup>+3</sup>	NS					

<sup>a</sup>NS= model not significant for this species.

Several minor and trace elements leachable from spent shales are dependent on the original raw shale that was used in the experiment. Boron and potassium solubilities are significantly higher from spent shales produced

from the LW-156 core material. Boron also was soluble from the raw material. Cadmium and titanium were significantly more leachable from spent shales produced from the Colony run-of-mine shale. Molybdenum, aluminum, and sodium were statistically higher in spent shale leachates from the tract C-a-A raw shale composite. Conversely, lithium concentrations were significantly lower from spent shale leachates produced from the tract C-a-B raw shale composite. The association of the similar behavior of these elements is probably due to the residence of these elements in a mineral phase in the raw shale. Fluoride and sulfate were significantly higher for the tract C-a composites, while fluoride also was higher for the Logan Wash core. Chloride was higher from spent shales generated from the tract C-a-B raw shale composite. There were no significant differences in leachability from spent shale because of shale type for the following elements: magnesium, silicon, calcium, iron, manganese, vanadium, cobalt, barium, nickel, and strontium.

Many of the major, minor, and trace elements studied in these experiments are influenced by the atmosphere present during retorting. Magnesium, silicon, potassium, and sodium are more leachable from shales exposed to the air atmosphere; shales treated with nitrogen, carbon dioxide, and carbon monoxide are much less leachable. Vanadium is mobilized from shales treated with either air or carbon dioxide atmospheres, while molybdenum and aluminum are significantly more mobile from the carbon-dioxide-treated shales. Molybdenum and aluminum also are mobile from the air atmosphere treatments. Calcium, iron, lead, cobalt, lithium, and strontium are significantly less soluble in shales treated with the carbon dioxide atmosphere, presumably because of the influence the carbon dioxide atmosphere has on the carbonate decomposition. The leachate concentrations of chloride and sulfate were significantly higher from the air-treated shales. This is a consequence of the air atmosphere providing conditions for oxidation of sulfur. There were no significant differences in leachability from spent shales because of atmosphere for the following elements: boron, cadmium, titanium, manganese, barium, and nickel.

The leachate concentrations of calcium, lead, cobalt, barium, and strontium peak at 800°C with the peaks at 1000°C being significantly smaller than those at 600°C. This is a consequence of alkaline earth oxide formation from the decomposition of carbonates. At 1000°C these oxides have been incorporated into the metamorphic silicate phases. Iron and nickel also peak at 800°C, but there is no significant difference between 600°C and 1000°C. Magnesium and vanadium are significantly more mobile from 1000°C shales, while molybdenum is more mobile from both 800°C and 1000°C shales. Sulfate concentrations also are significantly higher in leachates from 1000°C-treated shales. Sodium mobility decreases with increasing temperature. There are no significant temperature effects for the following elements: silicon, boron, cadmium, titanium, manganese, aluminum, potassium, and lithium.

The multiway ANOVA also analyzes two-way interactions among the independent variables and indicates where significant interactions are occurring. The interaction of atmosphere and temperature is the most important interaction for the greatest number of elements. The leachability of magnesium, boron, lead, vanadium, barium, potassium, lithium, and sodium, as

well as pH, is influenced by the interaction of temperature and atmosphere. Molybdenum and aluminum mobility and pH are influenced by the interaction of shale and atmosphere. In addition, the solubility of titanium and sodium is influenced by the interaction of shale type and temperature.

Any discussion of the leachability of spent shales has two distinct aspects, the concerns associated with increased salinity of surface and groundwaters and the concerns related to increased concentrations of toxic trace elements. The present study and previous results indicate that the solubility of most major cations is reduced to low levels and conductivity values are reduced with the formation of high-temperature silicate products during processing. For the alkaline earths and the alkali metals (except potassium), the concentration levels vary in a manner that suggests that these elements form oxides at intermediate temperatures that are reasonably soluble. At the more extreme conditions that may be encountered from an in situ retort, these oxides have reacted to form silicates that decrease their solubility.

The minor and trace elements leachable from these spent shales are not mitigated by formation of high-temperature products. This study indicates a positive correlation between increased mobilities of molybdenum, vanadium, and sulfate and high-temperature silicate products. This is verified by examination of the grand means for the temperatures. Although these elements, as well as others, are variable in the raw material, the effects with temperature are evident. It is not apparent whether the high-temperature products exclude these elements from their structures or whether the decomposition of the original hosts prevents further reaction with the silicate matrix. It does indicate that extreme retorting conditions (high-temperature oxidizing conditions) do not render all minor and trace elements insoluble.

These results indicate the importance of a knowledge of both the raw shale chemical and mineralogical compositions, and details of the processing conditions are necessary for definition of potential environmental problems associated with spent shale disposal. This work also suggests that a predictive model based upon shale type and processing conditions is a attainable and useful goal.

#### 6. KEY ACCOMPLISHMENTS:

- Completed an overview of health, environmental effects, and potential "show stoppers" in oil shale development.
- Elucidated the importance of both raw material and process in the identity and behavior of spent shale wastes (Occidental raw and spent shales from the Logan Wash site).
- Completed a balanced factorial design experiment to investigate the influence of shale type, temperature, and atmosphere on spent shale behavior.
- Compared the behavior of spent shales from laboratory experiments with shales generated from MIS retorting by OOSI at Logan Wash, Colorado.
- Completed a study of the partitioning of minerals, inorganics, and organics as a function of particle size in a raw shale from

Anvil Points, Colorado.

- Evaluated the application of the Los Alamos nuclear microprobe to the characterization of trace element residences in shale materials.
- Established the use of chemometrics as a major tool for evaluating large data bases in oil shale research and for relating field and laboratory results.
- Conceptualized and evaluated experimentally a multistaged leaching control for abandonment of underground retorts.
- Coordinated activities with other DOE laboratories, industry laboratories, and universities.

## 7. FUTURE WORK:

New approaches for the characterization of trace constituents in the oil shale matrix should provide more detailed information on the environments of these minor constituents in the matrix of both the raw and spent materials. This information, which could yield insights into the behavior of trace constituents during processing, is necessary for developing generalized predictive models for the magnitude and extent of elemental and organic partitioning into product and effluent phases, as well as for providing insights into the potential of environmental degradation caused by fugitive emissions or waste disposal. These activities may identify both matrix and process influences on the behavior of trace constituents that until now were not amenable to study by direct observation.

Two unique methods for solids characterization are being investigated for studying associations among trace elements and mineral components from laboratory or natural environments: the Los Alamos nuclear microprobe and the electron microprobe/automated imaging analysis (EMX/AIA). Exploratory work using these methods demonstrates their usefulness in such studies. Preliminary experiments to characterize spent shale samples were conducted. In addition to the major matrix elements magnesium, aluminum, potassium, calcium, and iron, this broadbeam (20- to 50-micron spot) reconnaissance study detected the minor and trace elements titanium, chromium, manganese, nickel, copper, zirconium, gallium, arsenic, rubidium, and strontium. Quantitative data reduction is in progress; initial results are consistent with 10-ppm detection limits. Further work in this area promises to yield heretofore unattainable data on the microscale processes controlling mobilization and transport of contaminants in the environment.

The EMX/AIA technique has been used on raw shale samples for particle analysis. The technique has been easily adapted to the oil shale matrix, and particle analysis of this type is providing data on the nature and size distribution of mineral grains that occur in this matrix. The automated imaging analysis and the energy dispersive spectroscopy results are being analyzed by sophisticated pattern recognition techniques that may ultimately lead to a quantitative, as well as qualitative, determination of the minerals present in the matrix. This particle analysis technique is being researched further.

Our laboratory experiments with well-characterized shales and carefully controlled process variables will be refined to include the influences of residence time and particle size on the reactions that occur in the oil shale matrix. These results will be supplemented by extensive characterization of materials from other bench-scale reactors and reference retorts to determine those properties of the shales that can be predicted from this research. In addition, these studies will be coupled with our previous studies of temperature and atmosphere to define the extent that each variable influences the formation and behavior of products, effluents, and wastes. These activities will be extended to include lower temperatures than expected in surface retorts and much longer time scales to begin to understand the effects of elevated temperatures in spent shale piles on spent shale chemistry.

Research to determine relationships among raw shale materials, process parameters important in surface retorting, and mineralogy and leaching behavior of the resultant spent shales will be continued. The goals are to determine the range of trace element mobilities expected from surface retorted spent shales, to understand the water/solid interactions responsible for solubilization of the trace elements away from the solids, and to identify solution chemical controls that may alter leachate composition after leachate movement away from the solids. Efforts will focus on determining those elements whose mobility is controlled by kinetic processes that have not been identified. The possibility of surface reactions for either mobilization of elements away from the solids or retardation of contaminants by the solids will be examined in the context of the bulk solution chemistry. Experimental activities also will elucidate the influence of leaching medium on leachate chemistry and the subsequent potential for transport after bulk solution properties are determined. These results, which will extend the knowledge gathered concerning the interplay of raw material and the process variables in the generation and behavior of the wastes, are essential for understanding the water management and solid waste management problems that may be encountered during surface retorting operations.

While the thermodynamic and kinetic influences on the generation and behavior of solid wastes and effluents are being defined, the relationships among spent shales from laboratory-scale, pilot-scale, and field retorts that were operated with similar raw material and process conditions will be investigated. These characterization efforts will try to determine the extent of scaling problems and will identify those shale characteristics that are not affected by moving from the laboratory or pilot-scale to a commercial operation.

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