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Review of Literature Pertinent to the Aqueous Conversion of Radionuclides to Insoluble Silicates with Selected References and Bibliography (Revised)

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December 1973

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**Atlantic Richfield Hanford Company
Richland, Washington 99352**

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REVIEW OF LITERATURE PERTINENT TO THE AQUEOUS
CONVERSION OF RADIONUCLIDES TO INSOLUBLE SILICATES
WITH SELECTED REFERENCES AND BIBLIOGRAPHY (REVISED)

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REVIEW OF LITERATURE PERTINENT TO THE AQUEOUS
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INTRODUCTION

Radioactive waste fission products and residual actinides in chemical processing plant wastes need to be isolated from biological processes until the radionuclides decay to innocuous levels. A promising method for effecting isolation is to convert the wastes to a highly insoluble material for final storage. The product should be inert for maximum immobility regardless of storage mode during long-term and any interim storage. Naturally-occurring systems that may yield suitable product forms are the aluminosilicate mineral groups which form much of the earth's crust.

Methods being investigated convert nuclear waste into crystalline aluminosilicate minerals wherein each of the nuclide cations are contained within individual molecular cages that constitute the crystal lattices. In the course of the investigation, the literature pertinent to immobilization of cations by silicates, with emphasis on aqueous systems, was reviewed. This report summarizes the literature and includes a bibliography to assist other investigators.

SUMMARY

The literature available on mineral synthesis was found to be extensive, however, applications with respect to converting radionuclides to synthetic crystalline minerals appears to be limited. The concept of incorporating mixed fission products and actinides from nuclear waste solutions inside crystalline aluminosilicate mineral lattices via a bulk process appears to be novel.

The Atomic Energy Commission national laboratories have studied waste immobilization in silicates. Their contributions include studies on high-temperature clay processes and ion exchange "fixation" processes. Previous workers have concentrated on "fixing" fission products on clay minerals by ion exchange processes. In such processes the fission products are leachable. Some investigators fixed the fission products more securely by heating the clay minerals to temperatures in the range of 1,000° C. However, the reaction of cesium with bentonite that produces relatively insoluble crystalline products of low leachability apparently was overlooked.

Two types of aqueous cation "fixation" in clay minerals are possible in which the degree of fixation and the mechanism differ. "Fixation" of cesium in a pollucite crystal structure is a mechanism that involves a three-dimensional cage and complete transformation of the aluminosilicate

lattice during the synthesis. Cesium fixed in a three-dimensional aluminosilicate lattice is immobilized to a greater degree than cesium in a "relatively fixed" condition between one-dimensionally collapsed lamellae of a clay mineral.

Aqueous synthesis of cation aluminosilicates is termed "hydrothermal" at elevated pressures. At atmospheric pressure the synthesis is called the "Gel Process" or the "Clay Reaction Process" if a reactant is a clay mineral. The Clay Reaction Process is a means of converting soluble radioactive cations to insoluble microcrystalline aluminosilicates. The type of aluminosilicate depends on the composition of the liquid waste and the reactants used.

Another application of the Clay Reaction Process is the conversion of relatively pure radioactive cesium salts to the mineral pollucite. Pollucite can be easily formed by reacting bentonite, pyrophyllite or kaolinite plus silica gel with alkaline solutions of cesium salts (CsCl , CsOH , CsNO_3 , etc.). With these materials, the reaction goes through a phase where the clay is converted to an amorphous material (gel) and much of the aluminum and silicon in the clay are dissolved. At temperatures of 80° to 100° C the reaction of these materials produces spherical crystallites of pollucite as a product. The type of product obtained from these reactions is partly controlled by the type of anions in the alkaline solution. The anions, in some circumstances, are considered to behave as structural templates in zeolite crystallizations, causing specific aluminosilicate structures to form. For example, halides, phosphate, perchlorate and some other anions cause sodalite to form.

The reaction of the clays with alkaline waste occurs at a relatively low temperature (70° to 100° C). At 100° C the reaction is reasonably fast, reaching completion after less than one day. The product is a hard mass of small insoluble cancrinite crystals. All clay minerals do not behave the same in the Clay Reaction Process. There is an influence of composition and structure of the mineral as yet only partially understood. Other cations do not behave as cesium but all in Groups II through VIII of the periodic table form insoluble silicates or aluminosilicates, giving promise to immobilization of all radionuclide cations that are present in chemical processing waste solutions.

REVIEW

Recognition of the advantages of converting fission products and residual actinides in chemical processing wastes to highly immobilized product forms led to studies of aluminosilicate mineral synthesis. Preliminary studies resulted in the STOPPER process which encapsulates individual ions such as cesium in molecular aluminosilicate lattices. STOPPER is the coined term for the Stone Process for Permanent Encapsulation of Radionuclides as described by Brownell, Chiaramonte and Isaacson.⁽¹⁾

Extension of the concept led to the discovery of the Clay Reaction Process reported by Barney.^(2,3) The processes of interest in this review can be grouped into three categories: hydrothermal syntheses, gel processes and clay reaction processes.

Synthesis of cation aluminosilicates in an aqueous system at all temperatures and pressures can be termed "hydrothermal," but in this review this term will be used for reactions conducted at pressures greater than atmospheric. Reactions in aqueous systems at temperatures of 100° C or less, in general, form gels prior to crystallization and are termed the "Gel Process." If one of the reactants is a clay mineral, the term "Clay Reaction Process" is used. The Clay Reaction Process involves a complete transformation of the aluminosilicate lattice and in this regard differs from "fixation" of cations in clay minerals by ion exchange processes or lattice collapse. The extent of the relationships between the processes has not been evaluated as yet.

The term "Clay Reaction Process" is the name given to the aqueous, atmospheric-pressure process of silicification of cations in radioactive waste liquors with a clay material such as bentonite as one of the reactants.^(2,3) The process is similar to the Gel Process, also conducted at atmospheric pressure, in which reactants such as sodium silicate and sodium aluminate are used.^(4,5) Also argillaceous minerals found in deep sea sediments, including montmorillonite, chlorite and illite, have been used as reactants in the Clay Reaction Process.⁽⁶⁾

Reaction at atmospheric or low pressure between argillaceous materials such as the clay minerals is a process that occurs naturally in ocean and lake sediments. This "reverse weathering," as it is called, is the subject of considerable recent study.⁽⁷⁻¹⁰⁾ The opposite process, "weathering," in which clay minerals and cations are released as a result of the decomposition of complex silicates is an extensively studied subject which is basic to the understanding of geology, mineralogy, and soil science and is the subject of extensive literature not included in this review.

Hydrothermal Synthesis

Barrer and his associates, over a period of 20 years, have conducted a classic series of studies on the elevated-temperature (>100° C) hydrothermal chemistry of silicates⁽¹¹⁻²⁵⁾ with Part IV⁽¹⁴⁾ describing the synthesis of cesium aluminosilicate (pollucite) and Part XII⁽²²⁾ the synthesis of strontium aluminosilicates. In general, the cation aluminosilicates were synthesized from silica gel and alumina by crystallization from aluminosilicate gels at moderate temperatures, usually 150° C or greater, in a closed autoclave suitable for high pressure.

However in Part VIII⁽¹⁸⁾ the lower-temperature growth of aluminosilicate crystals was studied with temperatures as low as 60° C. Gels of composition $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot n\text{SiO}_2$ ($1 \leq n \leq 12$) were prepared from sodium aluminate

and a stable dispersion of silica gel. At temperatures in the range of 65° C to 110° C crystallizations were conducted in Pyrex glass. Crystallizations often were rapid (two to three hours) and gave good yields of a zeolite "species Q" ($\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 4\text{H}_2\text{O}$) at temperatures of 85° C to 110° C from alumina-rich gels. The reaction slowed as the silica content of the gel increased. Crystallization was better and more rapid from gels with a large excess (300 percent) of sodium hydroxide. The influence of high NaOH concentration is especially marked at the low-temperature range of 60° to 85° C. With n in the range of four to six, the NaOH concentration at 300 percent and 85° C temperature, another zeolite species ($\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 5\text{SiO}_2 \cdot 5.4\text{H}_2\text{O}$) crystallized. Other crystalline species identified included analcite, mordenite, basic nosean, basic sodalite, harmotome-phillipsite, and nepheline hydrate.

In another series Barrer and associates have reported on the synthesis of cation aluminosilicates at low temperatures (<100° C) in studies on the Chemistry of Soil Minerals.⁽²⁶⁻³⁷⁾ Part V⁽³⁰⁾ of this second series contains the first known report of the synthesis of pollucite (along with cancrinite and a cesium zeolite) from a clay mineral (kaolinite) at temperatures of 80° C.

The conventional method of forming synthetic pollucite in an aqueous medium has involved the hydrothermal process at elevated temperatures and pressures.⁽¹⁴⁾ In experiments at Hanford, synthetic pollucite has been made at atmospheric pressure and 90° C.^(1,3,4) One procedure involves holding a mixture for two weeks at 90° C containing a 4 weight percent solution of cesium hydroxide (CsOH), sodium metasilicate ($\text{Na}_2\text{SiO}_3 \cdot 9\text{H}_2\text{O}$) and aluminum hydroxide [$\text{Al}(\text{OH})_3 \cdot \text{H}_2\text{O}$] in a molar ratio of 1:2:1, to produce a microcrystalline pollucite. If freshly precipitated ferric hydroxide is substituted for the aluminum hydroxide in the same molar ratio, a microcrystalline iron analog of pollucite is obtained. X-ray diffraction analyses confirmed the presence of pollucite in these products.⁽⁵⁾

Evidence of the stability of the pollucite cage is found in the absence of sodium aluminosilicates in the microcrystalline product even though sodium was present in solution at twice the molar concentration of cesium. Formulations similar to that given for pollucite but without cesium hydroxide yielded faujasite, a sodium zeolite. But, as no trace of faujasite was found with cesium hydroxide in the system, cesium is believed to control the structure of the product. The formation of cesium ferrosilicate at atmospheric pressure from a highly alkaline solution in which ferric hydroxide has low solubility also indicates the strong influence of cesium in producing the ferrosilicate lattice.

Part IX⁽³⁴⁾ of Barrer's series on soil minerals covers ion exchange and ion fixation in synthetic fluorhectorites and is especially pertinent to the Clay Reaction Process. In this article Barrer and Jones discuss three theories on the fixation of Cs^+ in clay minerals. The first theory is that of Wear and White⁽³⁸⁾ which proposes that fixation of large

univalent ions, such as K^+ in the layer silicates of clay minerals, is a result of the location of negative charge from isomorphous substitution. Cation substitution in the tetrahedral layer brings the source of negative charge as near as possible to the K^+ ions. This is the explanation given for potassium ion fixation.

The second theory, which was that proposed by Page and Baver,⁽³⁹⁾ is specific for the K^+ ion. It ascribes fixation to the ionic fit in the six-membered oxygen rings that form the surface of the tetrahedral layer.

The third theory relates ion fixation to swelling.⁽⁴⁰⁾ If swelling occurs, expansion of the lattice and ion hydration must reduce the free energy of the system more than the increased separation of opposite charges increases the free energy. Barrer supports this theory and some of his analysis is included later in this review (after consideration of other references on cation fixation as related to structure). Some of the other studies on the synthesis of aluminosilicates in aqueous systems are described in references 41 to 51.

The Gel Process

Patrick at Johns Hopkins University⁽⁴¹⁾ reported on tests with alumina silica hydrogels termed "artificial clays" for the fixation of radionuclides as feldspathic solids. The hydrogels were produced from aluminum hydroxide and silicic acid and reacted with alkaline silicates or alkaline earth silicates at temperatures not exceeding 100°C . The hydrogels were considered superior to kaolin and other clays in their "ability to react with silicates at temperatures not exceeding 100°C ."⁽⁴¹⁾ Commercial hydrogels of the type used for cracking catalysts in the petroleum industry were suggested for immobilizing radionuclides. A hydrogel containing one formula weight of Al_2O_3 and six formula weights of SiO_2 was proposed for immobilizing $\text{Cs}(\text{OH})$ and $\text{Sr}(\text{OH})_2$. The use of alumina silica hydrogels parallels the preliminary studies on the "Gel Process" conducted at Atlantic Richfield Hanford Company (ARHCO).^(4,5)

Gels can be prepared by use of sodium aluminate and sodium silicate solutions in combination with solutions prepared from the radionuclide salt cakes or soluble cesium and strontium salts. The gel structure results from polymerization of aluminate and silicate anions. The composition and structure of the hydrous polymer gel is controlled by the size and structure of the polymerizing species.⁽⁴²⁾ Differences of the species in the silicate solutions lead to differences in structure of the gels and in the lattices of crystals that may be produced by subsequent crystallization.

Aluminosilicate gels are produced by mixing alkaline solutions of silicates and aluminates in a limited region of mixture compositions.⁽⁴³⁾ The time required for gel formation at room temperature depends strongly

on the composition of the mixture. It is minimal for mixtures in which R, the molar ratio of Si/Al, is 1.00 and increases with changes in R from unity. In fact, gels are not formed if R deviates widely from unity or if the pH is high.

Aluminosilicate gels are typical colloidal structures, and gel skeletons are formed by joining contacting particles of colloidal size. The alkali cations such as Na^+ , Cs^+ , or Sr^{++} act as orientation centers for the aluminosilicate polymers. Layers of water molecules exist around the ions, and each ion with its associated primary hydration sphere undergoes Brownian motion as a whole.⁽⁴⁴⁾ The number of water molecules involved in primary hydration is dependent on the ionic size. For ions with radii of 1.3 to $2.0\text{\AA}$, the most stable grouping occurs with a coordination number of six and for smaller ions with a coordination number of four.⁽⁴⁵⁾

In a system of $\text{Na}_2\text{O}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2\cdot\text{H}_2\text{O}$ the silicate gel will be a hydrated complex with a coordination number of four. The stability of this complex increases with sodium concentration. As a result, the aluminosilicate compounds also will be formed with a coordination number of four.

The large number of water molecules involved in the hydration of aluminosilicate in alkaline solution is a major factor in the formation of crystalline zeolites. These hydrated ions have high stability. As a result, alumina and silica in solution will tend to crystallize out around the molecules of water. This produces microcrystalline aluminosilicate nuclei around the water molecules. As these grow, they form lattices with vacancies at the sites occupied by the water molecules. Synthetic zeolites with lattices of zeolite A and zeolite X are formed in this manner. They result from the growth at neutral pH around a group of 20 water molecules. According to Litvin,⁽⁴³⁾ "The large-scale hydrated molecules which exist under these conditions serve as crystallization centers and determine the lengths of the 'links' in forming zeolites. As the temperature is increased there is a progressive breakdown of the large-scale hydrated molecules, and the aluminum-silicon-oxygen gel adapts to the lighter hydrated crystallization centers." Zeolites with smaller link lengths are formed; for example, chabazite from phillipsite. A further increase of temperature leads to a complete breakdown of the hydration of the gel molecules, and sodium crystallization centers (still hydrated) begin to play an important part. Zeolite-like phases are formed in this way, such as analcite. Finally, at temperatures above 300° to 350°C there begins the formation first of nepheline-hydrate I with an open structure, and then of nepheline-hydrate II and nepheline itself, and more rarely albite.

Thus, gels produced at low temperature and pressure are most likely to produce crystallization centers that are precursors of zeolites. Products with minimum hydration such as nepheline are preferred to the zeolites on the basis of minimizing solubility. The temperature is limited to that of a boiling solution if atmospheric pressure is used. However, the pH can be increased at atmospheric pressure by increasing

the sodium ion concentration which has much the same effect as increasing the temperature. According to Litvin,⁽⁴³⁾ "At 150° C chabazite crystallizes, then phillipsite, sodalite, and finally analcime as the sodium content is increased. If the temperature and the concentration are increased (particularly when both are increased simultaneously), the role of sodium crystallization centers increases. At high temperature (>250° C) and high sodium concentrations (excess of caustic soda), the aluminum-silicon-oxygen gel adapts itself to the sodium centers forming a complete gamma phase of the cancrinite type. Active crystallization centers can also be formed by other large ions...such as chloride."

The discussion given above, based on Litvin's work,⁽⁴³⁾ relates to the $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ system and natural minerals. However, a number of other systems behave in the same manner, including the $\text{Na}_2\text{O} \cdot \text{ZnO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ system, for example. Litvin points out that, "One of the crystallochemical properties of zinc is its preference for tetrahedral coordination. In the system $\text{Na}_2\text{O} \cdot \text{ZnO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$ the zinc, in forming the zinc-containing phase, will surround itself with four oxygen atoms and in turn will predetermine several of the properties of the phase."

Sodium centers and other Group I cation (cesium) centers become the important factor in this system as the sodium concentration is increased. Now not only the silicon-oxygen tetrahedra, but also the zinc-oxygen tetrahedra are forced to adapt to the nucleus. In solutions containing low concentrations of NaOH, new phases (as well as willemite) can be formed whose centers are hydrated sodium or cesium ions. Further increase in the sodium concentration leads to the appearance of other new zinc-silicate phases. Litvin reports the formation of six new phases in the system $\text{Na}_2\text{O} \cdot \text{ZnO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$.⁽⁴³⁾

Litvin concludes that a similar approach to crystallization in the systems $\text{Na}_2\text{O} \cdot \text{CdO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$, $\text{Na}_2\text{O} \cdot \text{MnO} \cdot \text{SiO}_2 \cdot \text{H}_2\text{O}$, etc., makes it possible to produce a whole series of new compounds of desired properties by taking into account the crystallochemical properties of cadmium and manganese.⁽⁴³⁾ This can be extended to a great variety of complex silicate systems such as may occur in the Clay Reaction Process with various waste compositions.

Clay Reaction Process (G. S. Barney)

The Clay Reaction Process in which cesium is immobilized in good yield at 100° C in an aluminosilicate lattice using a clay mineral was described by Barney.⁽³⁾ Bentonite was considered to be superior to other clay materials tested because of its greater reactivity with various waste solutions and also because of its high silica content. The progress of the reaction was measured by X-ray diffraction tests of the solid products.

The Clay Reaction Process is a means for converting water-soluble, radioactive cations to solid, insoluble aluminosilicates. The type of aluminosilicate obtained from this process depends on the composition of the liquid waste and the type of clay used in the reaction.

Barrer and co-workers have studied the synthesis and properties of salt-filled cancrinites and sodalites.^(25,30,31,32) They reacted kaolinite with alkaline solutions of many different kinds of salts and found that in almost all cases the salts were entrained in the aluminosilicate lattice. The entrained salts could not be released except by decomposition of the aluminosilicate by strong mineral acids.

The type of product obtained from these reactions is partly controlled by anions in the alkaline solution. The presence of nitrate ion in solution causes the formation of cancrinite; whereas halides, phosphate, perchlorate and other anions cause sodalite to form. According to Barrer, the anions, in some circumstances, behave as structural templates in zeolite crystallizations, causing specific aluminosilicate structures to form.

One application of the Clay Reaction Process is the conversion of relatively pure radioactive cesium salts to the mineral pollucite. Pollucite can be easily formed by reacting bentonite, pyrophyllite, or kaolinite plus silica gel with alkaline solutions of cesium salts (CsCl , CsOH , CsNO_3 , etc.). The reaction occurs at 80° - 100° C and produces spherical crystallites of pollucite. With clays, the reaction goes through a phase where the clay is converted to an amorphous material and much of the aluminum and silicon in the clay are dissolved.⁽³⁾ As pollucite begins to form, the aluminum and silicon are removed from solution, indicating that the origin of the pollucite is the aluminum, silicon and cesium in solution.

During the first few hours of reaction the concentrations of both silicon and aluminum rise as some of the bentonite goes into solution. Formation of aluminosilicate gel causes the rise to reach a maximum of 5 to 9 percent for aluminum (weight percent of Al in clay) after about 10 hours. Thereafter, the concentration of aluminum in solution falls rapidly and is close to zero after about 50 hours of reaction. At the time that the aluminum concentration is falling, the silicon concentration is continuing to rise but more slowly. Barney notes that, "Pollucite formation also marks the beginning of the loss of silica from solutions. These observations are consistent with a mechanism in which the intermediate cesium zeolite reacts with dissolved silica to form pollucite."⁽³⁾

Barney conducted other experiments using cesium chloride and bentonite to determine the feasibility of conversion of CsCl to pollucite by the Clay Reaction Process and obtained results as listed in Table I.

TABLE I

Results of Reaction of Bentonite* with CsCl (After Barney)⁽³⁾

<u>Mixture Number</u>	<u>Solution</u>	<u>Yield (grams)</u>	<u>Percent Cs₂O</u>	<u>Product</u>
98	20 ml H ₂ O	1.04	12.0	Bentonite with adsorbed Cs ⁺
99	20 ml 0.75M NaOH	1.29	29.9	Poor pollucite
100	20 ml 1.50M NaOH	1.27	34.2	Good pollucite
101	20 ml 3.00M NaOH	1.16	27.2	Pollucite + other

*1.0 gram bentonite and 10.0 grams CsCl were used in each experiment.

The results in Table I show the importance of high alkalinity such as associated with 1.5M or higher NaOH.

Cation Fixation, Structure and Interlayer Charge of Clay Minerals

The Clay Reaction Process "fixes" radionuclide cations such as cesium and strontium in an aluminosilicate lattice. Also soil scientists have long known that some cations can be adsorbed by clay minerals in a relatively nonexchangeable or "fixed" state by a process termed cation fixation. "Fixation" thus is a relative term used in the literature in at least two senses: (1) the "relatively fixed" position, for example, of potassium in muscovite mica; and, (2) the "fixed" position, for example, of cesium within the pollucite crystal structure. The energy required to remove the cesium from pollucite is greater than that required to remove potassium from muscovite. Potassium, cesium, and barium are included in the cations that undergo cation fixation. Of these, potassium has been the most widely studied because of its importance in fertilization of soil. Fixation of K⁺ has been studied in a variety of clay minerals including illite,⁽⁵²⁾ montmorillonite,⁽⁵³⁾ vermiculite,⁽⁵⁴⁾ pyrophyllite,⁽⁵⁵⁾ and mica.⁽⁵⁶⁾ Early studies of fixation of Cs⁺ in clay minerals were reported by Martin, et al.,⁽⁵⁸⁾ and Barshad.⁽⁵⁴⁾ The kaolinite group was found to have a much lower capability for fixation of cations than the other groups studied.^(53,56,57,59) Barshad⁽⁵⁴⁾ concluded that this capability of cation fixation is dependent on the magnitude of the interlayer charge of the clay mineral. Because the cation must penetrate the interlayer to be fixed, the reaction is time and temperature dependent.⁽⁵⁵⁾

Dorothy Carroll made an early review of "Ion Exchange in Clays and Other Minerals"⁽⁵⁹⁾ with a selected bibliography of 150 references. Her discussion of the structural features of the clay minerals is helpful in visualizing a mechanism of structural rearrangement in the Clay Reaction Process. The clay minerals are made up of layers in octahedral coordination. One of the simplest clay minerals is kaolinite, termed a 1:1 layer silicate with one silica tetrahedral layer and one alumina octahedral layer. Montmorillonite, however, is a 2:1 layer silicate and consists of two silica tetrahedral layers separated by one alumina layer. Montmorillonite (bentonite) has been found to be more reactive in the Clay Reaction tests than kaolinite, and the 2:1 structure rather than the 1:1 structure offers one explanation for this observation. Halloysite type clay minerals have the same structure as kaolinite and, at first glance, would seem to be less satisfactory than montmorillonite type clay minerals. However, the halloysite or endellite variety with $4\text{H}_2\text{O}$ has a higher exchange capacity than the metahalloysite form with $2\text{H}_2\text{O}$.

The mica group minerals are 2:1 layer silicates and include the micas (muscovite, phlogopite, biotite and paragonite), hydrous micas (illites), vermiculites, glauconites, and celadonites. The vermiculite structure is electrically unbalanced, chiefly because of Al^{+3} substitution for Si^{+4} . Other substitutions within the mica lattice may partly compensate for the unbalance. The exchange capacity is about equal or higher than that of montmorillonite. For example, Carroll gives an exchange capacity of 70 to 100 for montmorillonite and 100 to 150 for vermiculite in milliequivalents per 100 grams at pH 7.⁽⁵⁹⁾

The following arrangements of structural units are given for a vermiculite according to Carroll, except that Cs^+ has been substituted for Mg^{++} at the exchange surface.

TABLE II

Balance of Charges for One Layer of Vermiculite
with Cesium at Exchange Surface

	Y H_2O Double Layers of Water	Charge	Charge (X=1)	One Layer of Vermiculite
	Cs^+	$+1$	$+1$	
Si-tetrahedron	6 0^{-2}	-12	-12	
	(4-X) $\text{Si}^{+4} \cdot \text{Al}^{+3}$	$+(16-X)$	+15	
Al-octahedron	4 0^{-2} 2(OH) $^{-1}$	-10	-10	
	6 (Mg Fe) $^{+2}$	+12	+12	
	4 0^{-2} 2(OH) $^{-1}$	-10	-10	
Si-tetrahedron	(4-X) $\text{Si}^{+4} \cdot \text{Al}^{+3}$	(16-X)	+15	
	6 0^{-2}	-12	-12	
	Cs^+		$+1$	
			0	
	Y H_2O Double Layers of Water			

According to Barshad^(54,60-69) the total interlayer charge of the crystal lattice in clay minerals affects the accessibility of the interlayer cation in three ways: it affects the interlayer expansion, the number of empty hexagonal cavities in the interlayer position, and the interlayer attractive force that holds the lattice layers together. The explanation advanced for "fixation" of cations in clay minerals is that it is due to an interlayer charge which originates from the isomorphous substitution of Al^{+3} for Si^{+4} in the tetrahedral layers of the lattice. This is the case for the vermiculites,⁽⁵⁴⁾ but not for montmorillonite.^(64,65) Since one Al^{+3} must be substituted for one Si^{+4} for each Cs^{+} ion in a pollucite aluminosilicate cage, the substituted vermiculite tetrahedral lattice is more similar to a pollucite lattice than the montmorillonite lattice.

The nature of the exchangeable cation (shown as Cs^{+} above) influences the thickness of the interlayer of water⁽⁵⁹⁾ because the cation rests between the layers of vermiculite. Mg^{+2} is the usual exchangeable cation in vermiculite, and the bound water forms a hydration envelope of six water molecules for each Mg^{+2} . Unbound water fills the space between the octahedral groups of bound water. In the case of Mg^{+2} cations, the d(002) spacing is $14.39\text{\AA}$, giving a water layer thickness of $5.11\text{\AA}$. In the case of Na^{+} cations, the d(002) spacing is $14.8\text{\AA}$, and the water thickness is $5.55\text{\AA}$. However in the case of K^{+} the lattice is collapsed to $10.6\text{\AA}$, giving an approximate water layer thickness of only $1.34\text{\AA}$. Jacobs⁽⁶⁶⁾ states that a collapsed lattice of about $10\text{\AA}$ spacing is requisite for the fixation of cesium by vermiculite. Also according to Jacobs the fixation mechanism and the fixation capacity differ depending on whether the lattice is collapsed before or during the fixation process.⁽⁶⁶⁾ In another article,⁽⁶⁷⁾ Jacobs and Tamura comment that, "In the case of previously collapsed lattices, the 'fixed' cations are held with high affinity at exchange sites on the edge of the interlayer spacing while lattices that collapse during the fixation process physically entrap the interlayer cations in a true fixation process."

Jacobs and Tamura reported on ion fixation in clay minerals^(67,68) and later on improving cesium selectivity of bentonites by heat treatment.⁽⁶⁹⁾ They compared Arizona bentonite, Wyoming bentonite, kaolinite, illite, and chemically-treated vermiculite with regard to cesium sorption and desorption. It was concluded that, "There are three types of sorbed cesium in the collapsed lattice systems: (1) easily exchangeable cesium (for example, cesium retained by illite at high levels of cesium saturation); (2) cesium 'fixed' at lattice edges (for example, cesium retained by illite, micas, potassium-treated vermiculites, and heat-treated bentonites at extremely low levels of cesium saturation); and, (3) cesium 'fixed' by entrapment during lattice collapse (for example, cesium trapped by vermiculite at high levels of cesium saturation). The easily exchangeable cesium would be readily removable by any leaching cation, while cesium 'fixed' at lattice edges would be expected to be displaced only by another ion having steric favorability for such an exchange site. On the other hand, entrapped ions should be liberated by those cations which would permit

lattice expansion. Desorption data show this to be true. Stable cesium is slightly more effective than hydrogen but is markedly more effective for removing the cesium 'fixed' at the lattice edges of illite. Hydrogen ions appear to be more efficient in removing cesium from vermiculite, though very little of the entrapped cesium can be removed unless the lattice is destroyed. The inability of cesium to undergo isotopic exchange attests to the tenacity with which these interlayer 'fixed' cations are held."⁽⁶⁷⁾ However, Ames points out (see Discussion) that cesium is not held this tightly by clay minerals. "Fixed" cations can be removed by maintaining very low levels of the "fixed" cation in the contacting sodium solutions.

Experiments at ARHCO with clay minerals, such as montmorillonite (bentonite),⁽²⁾ sea sediment,⁽⁶⁾ and aluminosilicates⁽⁵⁾ have indicated the formation of crystalline pollucite at 90° C. The strong tendency to the formation of pollucite from Cs⁺ in the presence of clays and other aluminosilicates is another effect of this immobilization of Cs⁺. However, the aluminosilicate must undergo a structural transformation to become pollucite, and this transformation is favored by pH>10 and/or the input of thermal energy.

Jacobs conducted time studies of cesium reaction with bentonite and collapsed-lattice clay minerals illite and biotite.⁽⁷⁰⁾ He found that equilibrium between bentonite and salt solutions was attained within a few minutes after contact and concluded that interlayer fixation of cesium occurred when cesium was sorbed on basal exchange sites followed by lattice collapse of the mineral. Jacobs reported that with expanding minerals such as montmorillonite, "No fixation of Cs⁺ was observed."⁽⁶⁹⁾ However, in Ames' test of Cs⁺ and sea sediments, montmorillonite converted to pollucite at 90° C in a high pH environment.⁽⁶⁾

Jacobs⁽⁶⁶⁾ considers treated vermiculite to behave similarly to illite. Tests at Hanford⁽⁴⁾ with sea mud at 90° C showed the disappearance of illite and the appearance of pollucite. Thus pollucite may be a product of cesium reaction with treated vermiculite under the proper pH and temperature conditions. Jacobs comments that potassium-treated vermiculite develops an illitic structure and exhibits a cesium sorption curve nearly identical to that of natural illite. In the case of sodium-treated vermiculite, the sorption curve is more complex. As the cesium concentration is increased, cesium ions are first fixed along the edge of vermiculite platelets and, as these positions are filled, the cesium ions move to the interlayer exchange sites and cause "a collapse of the vermiculite lattice." The interlayer cesium is thus physically entrapped by the closing lattice and is very tenaciously held. In fact, very little of the entrapped cesium is available for isotopic exchange with stable cesium.⁽⁶⁶⁾

Jacobs also reported on cesium fixation by hydrobiotite-vermiculite⁽⁷¹⁾ (zonolite, a commercial form of vermiculite), noting that collapse of vermiculite layers after cesium sorption causes entrapment of the cesium

sorbed at the basal exchange sites in a process of interlayer fixation. Shainberg and Kemper reported on electrostatic forces between bentonite clays and Na^+ , Cs^+ and Ca^{+2} cations.⁽⁷²⁾ Na^+ -saturated bentonite and Cs^{+2} -saturated bentonite and ionic mixtures of the two clays were used. They observed that the affinity of bentonite for Cs^+ is 10 to 20 times the affinity for Na^+ and commented that, "The usual explanation for the high affinity of clay for Cs^+ is that a large cation like cesium is not hydrated and can come very close to the negative charge on the particle."

Barrer and Jones reviewed three theories for the fixation of Cs^+ in their study of fluorhectorite.⁽³⁴⁾ The clay mineral hectorite belongs to the smectite group, trioctahedral division, and has no substitution in the tetrahedral layer. The negative charge in the fluorhectorite arises only from substitution in the octahedral layer. Since fixation of K^+ , Rb^+ and Cs^+ occurs in fluorhectorite, Barrer and Jones conclude that substitution of Al^{+3} for Si^{+4} in the tetrahedral layer (as in the case of vermiculite) is not essential for cation fixation. This is consistent with fixation in montmorillonite reported in other studies but does not deny the advantages of such substitution. The diameter of the hole in the hexagonal oxygen ring has been reported to be about $2.5\text{\AA}$ based on studies of zeolites with similar ring structures.⁽⁴⁴⁾ Barrer and Jones consider the differences in cation diameters [K^+ ($2.66\text{\AA}$), Rb^+ ($2.96\text{\AA}$) and Cs^+ ($3.38\text{\AA}$)] to rule out the theory of the fixation of K^+ ion because of the snug fit of K^+ ion into the oxygen hexagon. However, if K^+ , Rb^+ and Cs^+ are all larger than the ring opening and Li^+ and Na^+ , which are not fixed, are smaller, then the theory of the importance of ionic dimensions is supported.

Barrer and Jones⁽³⁴⁾ in studies on fluorhectorite observed the clearest correlation between ion fixation and the inability of the fixed ions to hydrate in their intracrystalline environment. The smaller Group I cations, Li^+ and Na^+ , were observed to remain hydrated in preference to becoming more closely bound to the lattice oxygens. The larger Group I cations, K^+ , Rb^+ and Cs^+ , shed their waters of hydration and locate between two clay lamellae. They are partially recessed into each of two rings of oxygen hexagons, one from a lower and one from an upper tetrahedral layer on the lamellae surfaces. This provides a coordination number of 12 oxygens for Cs^+ .

The alkaline earth metal cations, Ba^{+2} , Ca^{+2} , and Sr^{+2} , all cause fluorhectorite to swell due to hydration, but the larger alkali cations, K^+ , Rb^+ and Cs^+ , do not. The cation diameter of Ba^{+2} is $2.70\text{\AA}$ as compared to $2.66\text{\AA}$ for K^+ which demonstrates that fixation involves more than a critical diameter for the cation. The magnitude of the valence charge would be expected to contribute to hydration.

The result in a fluorhectorite system with mixed cations in solution is that the large univalent cations K^+ , Rb^+ and Cs^+ will replace smaller univalent cations Na^+ and Li^+ , as well as the divalent cations such as Ca^{+2} , Sr^{+2} and Ba^{+2} , losing their waters of hydration in this immobilizing process. Barrer and Jones consider this loss of hydration to be

"the prime cause of ion fixation." Ions such as Na^+ that have a high affinity for hydration do so and push the lamellae apart. Norrish⁽⁴⁰⁾ reported a lattice expansion for sodium-montmorillonite in sodium chloride solution of $40\text{\AA}$ in the (001) plane whereas cesium-montmorillonite did not exceed $15\text{\AA}$ (in water).

According to Barrer and Jones, "Fluorhectorites have a greater power to remove Cs^+ than any crystalline zeolite so far examined." This is a strong statement considering Barrer's extensive 20-year experience with zeolites.

The selective sorption and fixation of cations by clay minerals was reviewed in 1972 by Sawhney.⁽⁷³⁾ He discussed the role of interlayer collapse in vermiculite on K^+ or Cs^+ sorption and compared the fixation of K^+ and Cs^+ by different clay minerals. He states that, "Cesium ions with a smaller hydration energy than K^+ ions hold the layers together more strongly than K^+ ions so that hydrated Ca^{+2} and Mg^{+2} ions which can expand layers held by K^+ ions are ineffective in producing layer expansion in Cs^+ -saturated minerals. Consequently, Cs^+ ions are not as readily replaced by these ions as are K^+ ions."

Fixation by Heating

In most earlier studies of the use of clay minerals the objective was to remove limited quantities of radionuclides from aqueous waste, usually by adsorption and/or ion exchange. However, some of these earlier studies, such as those at Brookhaven National Laboratory,⁽⁷⁴⁻⁷⁸⁾ involved "fixation" of radionuclides in selected clay minerals, including montmorillonite and glauconite, with further immobilization resulting from heating to about 1000°C .

The earliest work on fixing radioactivity in stable, solid media probably was that of Hatch and co-workers with montmorillonite clay.⁽⁷⁴⁻⁸²⁾ They found that one gram of clay would fix about 1.2 milliequivalents of cations. Subsequent heating of the clay to about 1000°C completed the fixation. A pilot plant with 15-foot high columns packed with extruded clay was tested at Brookhaven and gave high decontamination factors for strontium-90 from leach solutions. One disadvantage of this "clay" process was the inability of the clay to fix ruthenium. Heating the clay to 565°C after cation fixation from fission product solutions permanently fixed about 90 percent of the activity in fission product waste, but heating to 915°C permanently fixed over 99.8 percent.⁽⁷⁷⁾ Removal of zirconium and aluminum from fission product solutions prior to treatment with clay was considered to be advantageous.⁽⁸⁴⁾

A similar clay process was studied by Amphlett and others^(85,86) of AERE at Harwell, England, except that the clay was mixed to a paste with partly-hydrolyzed ethyl orthosilicate dried and fixed at 350°C , crushed and sieved to produce a granular product. Amphlett⁽⁸⁵⁾ considered the granular product to have greater physical stability than the extruded product, but had the disadvantages of greater cost and a reduction in fixation

capacity as a result of heating to 350° C. In the Harwell studies,⁽⁸⁶⁾ the clay was pretreated with ammonium salt solution to produce the cationic form of the clay preferred by Grover because it gave about 25 percent greater cation fixation. The clay used in the Harwell study was of a silica-bonded montmorillonite type identified as Surry Fuller's Earth. Amphlett⁽⁸⁵⁾ reported that the fired clays had satisfactory resistance to leaching in tests using distilled water and sea water but increased leaching with dilute acids. Initial leach rates after a few days for clay granules (Fuller's Earth Unicon, Ltd.) fired at 1250° C for one hour were about 0.001 percent per day using distilled water. This converts to a leach rate of approximately $4 \times 10^{-7} \text{ g.cm}^{-2}.\text{day}^{-1}$ based on an estimated specific surface of 25 cm²/g for the clay granules. The leach rate in 0.1N nitric acid was two orders of magnitude greater initially but decreased to above $4.10^{-6} \text{ gm.cm}^{-2}.\text{day}^{-1}$ after 50 days of leaching.

Mixtures of clays, ceramic fluxes and solutions of radioactive wastes which were dried and sintered to ceramic masses were studied at Oak Ridge National Laboratory.⁽⁸⁷⁻⁸⁹⁾ Clay samples including metabentonites, montmorillonites, halloysite, nontronite, kaolinite, vermiculite, and a Conasauga shale (predominantly illite) were studied with regard to their abilities to remove radionuclide cations.⁽⁹⁰⁾ Lacy⁽⁹¹⁾ found that Oak Ridge clay was composed chiefly of montmorillonite and kaolinite and, when slurried with contaminated water, was quite effective in removing the cations ¹⁴¹Ce, ¹⁴⁴Ce-¹⁴⁴Pr, ⁹⁵Zr-⁹⁵Nb, ¹⁴⁰Ba-¹⁴⁰La, ⁹⁰Sr-⁹⁰Y and some mixtures of fission products. For ¹⁰⁶Ru-¹⁰⁶Rh and for other mixtures of fission products, removal was not as satisfactory. The possibility of trapping radioactive wastes in ceramic materials was reported by Robinson.⁽⁹²⁾ In 1962 Robinson prepared an extensive review⁽⁹³⁾ of the early studies on the use of clay minerals in "Ion-Exchange Minerals and Disposal of Radioactive Wastes — A Survey of Literature."

Early studies on the use of clays for containing radionuclides also were conducted at Los Alamos Scientific Laboratory.^(94,95) Clay-waste mixtures were extruded to produce bricks for leaching tests, using a wide range of natural and commercially-available clays. The bricks possessed good physical properties but had relatively poor leaching resistance unless fired at high temperature. Bricks fired at 1200° C were glassy and had lower leach rates than those fired at 1000° C which were not glassy and were more porous.

Cation Exchange Characteristics

There is some opinion that ion exchange characteristics of the clay minerals may have minor influence on the Clay Reaction Process. Inclusion of literature on this subject may be considered to have limited relevancy but, as yet, this has not been clearly established. Therefore, the literature specific to modes of cation fixation and the relationship of structure of the minerals to fixation has been reviewed. The characteristics of the clay minerals in the soils, river beds and sea sediments as well as specific aluminosilicate systems were considered in relation to cation fixation. Some of the more pertinent papers have been included in the references.

The sorption of strontium and cesium in Savannah River Plant (SRP) soil was studied by Fenimore and Horton.⁽⁹⁶⁾ They reported that cesium was retained in SRP soil because of fixation in mica minerals, and that fixation was greatly increased by sodium ions present in waste solutions. Strontium was readily leached from the waste-soil mixture by dilute solutions of acid or salts, but cesium was not readily leached by any solution tested. Earlier studies⁽⁹⁷⁾ on the fixation of mixed fission-product activity by soils of the Savannah River Project were conducted at Mound Laboratory, and the activity removal was correlated with the clay content of the soils.

The U. S. Geological Survey (USGS) has reported on the role of stream sediment components in radionuclide sorption,⁽⁹⁸⁾ the distribution of radionuclides in bottom sediment,⁽⁹⁹⁾ and the uptake and transport of radionuclides by stream sediments.⁽¹⁰⁰⁾ Sorption of cesium-137 in contrast to strontium-90 and cobalt-60 was controlled by clay minerals.⁽⁹⁸⁾ Sorption of radiocontaminants by stream sediments is the rule rather than the exception, but the extent of the sorption is dependent upon a large number of variables: the nature of the sediment; the substance being sorbed; the available surface area; the concentration and types of solute in solution; time; and, to a lesser extent, temperature.⁽¹⁰⁰⁾ The absorption of cesium on clay minerals, montmorillonite, kaolinite, illite and halloysite was studied by Wahlberg and Fishman of the USGS.⁽¹⁰¹⁾ They found that halloysite was very effective in absorbing cesium from extremely dilute cesium solutions. A number of related studies on the interaction of river sediments and radionuclides were reported by personnel of USGS⁽¹⁰²⁻¹⁰⁶⁾ and ORNL.⁽¹⁰⁷⁻¹²¹⁾

Early studies on the interaction of radionuclide and soil minerals also were conducted at Hanford.⁽¹²²⁻¹³⁷⁾ During the period 1960 to 1964 extensive studies⁽¹³⁸⁻¹⁶⁷⁾ were conducted at the Hanford site by Ames and co-workers on aluminosilicates, clinoptilolite in particular, to remove cesium and strontium from stored waste in preparation for the In-Tank-Solidification program. Aluminosilicates were used at the Hanford reservation to remove cesium from waste liquor until 1970.

The National Academy of Sciences (NAS) in 1971 reviewed the influence of "Radioactivity in the Marine Environment" with Chapter 6 devoted to Marine Sediments and Radioactivity.⁽¹⁶⁸⁾ The references for this chapter contained a number of pertinent additional references⁽¹⁶⁹⁻¹⁹³⁾ of which only a few were available for review.

Duursma and Gross⁽¹⁶⁸⁾ noted that sea sediments, like river sediments, have a substantial capacity to remove radionuclides from sea water, and that certain radionuclides are more strongly sorbed on sediment particles than others. Mineral-related reactions, each with a degree of specificity, appear to be controlled by chemical species of the radionuclides and the physico-chemical conditions of the sediments and sedimentary particles. In some cases the sorption of radionuclides by sediments is dependent upon the "history of the sediment-water system prior to the introduction of the radionuclide." Among the processes

by which radionuclides move in sea sediments are: (1) diffusion through the interstitial water; (2) movement with the interstitial water expelled from the sediment by compaction; and, (3) groundwater discharge through the sea bottom. "Where sediment accumulation is sufficiently rapid, the radioactive particles may be covered by later sedimentary deposits before these relatively slow movements can move the nuclides through the sediment-water interface."

Distribution coefficients defined as the dimensionless ratio of the amount of bound radionuclide per unit volume of dry sediment to the amount of radionuclide per unit volume of sea water were reported for different radionuclides. The values of the distribution coefficients reported ranged from about 1×10^2 for strontium-90 to 10^5 for promethium-147 with cesium-137 and a number of other radionuclides in the 10^3 - 10^5 range. The larger the distribution coefficient, the stronger the binding (sorption) of the radionuclide to the sediment.

International Atomic Energy Agency (IAEA) Technical Report No. 78⁽¹⁹⁴⁾ briefly treats the use of clay minerals attapulgite, halloysite, kaolinite, illite, montmorillonite and vermiculite for ion exchange with radioactive wastes. Natural zeolites analcime, chabazite, clinoptilolite, harmotome, heulandite, natrolite, sodalite and several synthetic zeolites also are discussed briefly. The report contains an extensive bibliography of which references 195-205 are pertinent to the Clay Reaction Process. Studies on three earth materials, a lignitic material, an alluvium clay, and a silicate mineral, clinoptilolite, showed that the clinoptilolite was superior. Levi and Micheley⁽²⁰⁶⁾ of the Federal Republic of Germany reported on "Ion Diffusion in Vermiculite" and commented that, "It is well known that several clay minerals occurring in soils are able to fix certain radionuclide ions particularly cesium...in a cesium-saturated vermiculite, only the fraction of Cs ions adsorbed at crystal imperfections is exchangeable. For the bulk of the cesium ions no diffusion could be observed even at 100° C over a period of three months." In the case of vermiculite loaded with two ions, one of which was cesium and the other an alkaline earth, the latter remains mobile. According to Levi and Micheley⁽²⁰⁶⁾ this shows that, "The presence of the non-hydrated (cesium) and, consequently, fixed ion does not inhibit the migration of other ions in the vermiculite lattice. The conclusion is that it is not lattice-contraction associated with cesium uptake, but rather the state of the particular ion in the lattice that is responsible for the fixation phenomenon." Thus these authors disagree with the theory of Barshad⁽⁵⁴⁾ that lattice collapse is the chief explanation for the fixation of cesium ion in vermiculite. The authors claim that the chemical bond of the individual cesium ion should be considered responsible for fixation rather than the state of the lattice as a whole.

International Atomic Energy Agency Technical Report No. 136 on "Uses of Minerals in the Treatment of Radioactive Waste"⁽²⁰⁷⁾ published in 1972 is the most recent IAEA publication relating to the Clay Reaction Process.

It contains a section on aluminosilicates and summarizes the causes for cation exchange in clay minerals as follows: "(1) unsatisfied valences produced by broken bonds at surfaces and edges of particles; (2) unbalanced charges caused by isomorphous substitutions of cations; for example, Al^{+3} substituted for Si^{+4} giving one net negative charge; (3) disassociation of structural OH^- radicals, the H^+ of which may be replaced by metallic cations; and, (4) accessibility of structural cations other than H^+ which becomes exchangeable under certain conditions; for example, at low pH values Al^{+3} ions move from the octahedral units to the exchange positions."

International Atomic Energy Agency Technical Report No. 136 gives the principal cause of cation exchange in montmorillonites, illites, and vermiculites as isomorphous replacement. Exchange takes place in montmorillonite "at three sites: on the flat surfaces, on the edges, and between the silica and alumina layers where ions are loosely held to neutralize deficiencies in these layers caused by isomorphic replacements."

Mineral replacement reactions are defined as "those between a mineral and a constituent of the solution, resulting in the formation of a new crystal lattice and the inclusion of radioisotopes in the final product."⁽²⁰⁷⁾ Nontronite is a dioctahedral clay mineral of the smectite group having a crystal structure of montmorillonite in which most of the octahedral Al^{+3} ions are replaced by Fe^{+3} . The latter connect two tetrahedral layers, forming a non-swelling octahedral double layer silicate. As pollucite can be synthesized from montmorillonite and from ferric hydroxide, as demonstrated for the Gel Process, nontronite also should produce a pollucite with some ferric group substitutes for alumino groups. A list of 179 references is given in reference 168, of which 11⁽²⁰⁸⁻²¹⁸⁾ were selected as new and pertinent.

The most recent review of synthetic aluminosilicates, "Synthetic Inorganic Ion Exchangers - II," was published in 1972 in Talanta Review.⁽²¹⁹⁾ This review covered published papers in the field for the period 1965-1970 for both western and eastern countries, with emphasis on those not easily available. A total of 403 references are listed, but only a few that were new to this study were reviewed. References 220-223 are from this review and are considered particularly pertinent.

Diagenesis of Clay Minerals

Diagenesis of clay minerals refers to changes that occur in these materials during geologic time. It is pertinent to the Clay Reaction Process because it describes some of the typical reaction paths toward structural and chemical equilibrium that can be anticipated for clay minerals placed in different environments. A review of transformation of clay minerals during diagenesis and early metamorphism was given by Dunoyer de Segonzac.⁽²²⁴⁾ The crystallochemical processes that occur during diagenetic evolution are summarized as follows: (1) substitution of aluminum in the tetrahedral layer; (2) filling of octahedral sites

either by interlayer cations or by cations from outside the lattice; (3) interlayer exchange between crystal lattice and interstitial solution; and, (4) crystal growth. In the review, six different groups were considered: (1) kandite group minerals; (2) smectite group minerals; (3) mixed-layer minerals; (4) micaceous clay minerals or illites; (5) micas; and, (6) chlorites.

The kandite clay mineral group (or kaolinite group) includes six species: kaolinite, anauxite, metahalloysite, halloysite, dickite and nacrite. These minerals are sensitive to the environmental conditions in zones of the earth's crust where they are formed. The kandite minerals are unstable in an alkaline system but are stable in an acidic condition. At the pH of sea water, kaolinite is stable at 95° C or less but undergoes illitization at 100° C and above.⁽²²⁵⁾ At around 300° C kaolinite transforms into pyrophyllite and andalusite almost independent of pressure.⁽²²⁶⁾

The smectite minerals (or montmorillonoids) are hydrated silicates and, like kaolinite, are characteristic of certain environmental conditions. According to Millot,⁽²²⁷⁾ the smectites occur in four types of environments: (1) in soil profiles; (2) in basic chemical sedimentation; (3) in bentonites; and, (4) in hydrothermal veins. In well-drained soils, the smectites are ferriferrous.⁽²²⁸⁾ In basic sedimentation the environment is alkaline and can be either lacustrine or marine. These smectites are magnesian and may be associated with attapulgite and sepiolite. Smectites in hydrothermal veins are principally magnesian and of the saponite species. The term bentonite is generic, implying a smectite clay produced by the transformation of volcanic ash.⁽²²⁹⁾ The term has been enlarged to include other types of deposits of smectite regardless of origin.⁽²²⁴⁾

Montmorillonite-bearing sediments undergo progressive transformation with depth including dehydration, absorption of alkali ions, rearrangement of the lattice and conversion to mixed-layer minerals of the illite-montmorillonite type. The temperature for transformation of montmorillonites into illite montmorillonite mixed-layer minerals is 70° to 95° C as determined from borings. This temperature is lower than determined experimentally for natural clays.⁽²³⁰⁾ In borehole tests with Gulf Coast (Texas, Louisiana) clays, Burst observed⁽²³¹⁾ that montmorillonites were converted to mixed-layer illite-montmorillonites at about 95° C and finally to illite by loss of swelling interlayers.

In Whitehouse's and McCarter's experiments, transformations at normal temperatures and pressures converted montmorillonite to chlorite, illite and mixed-layer minerals by interaction with artificial sea water.⁽²³²⁾ Slaughter and Milne⁽²³³⁾ confirmed that magnesium was fixed between the layers of montmorillonite to form chlorite. At higher temperatures up to 350° C chlorite continues to be formed; but at even higher temperatures, corrensite, talc or chlorite may be produced, depending on the quantity of magnesium present.⁽²³⁴⁾ Additions of correct

proportions of calcium and magnesium carbonates resulted in formation of pyroxene and plagioclase rather than of corrensite (a regular mixed-layer chlorite-montmorillonite) and chlorite from degraded 2:1 clay minerals, ^(235,236) as well as talc in saline formations. ⁽²³⁷⁾ Laboratory temperatures necessary for promotion of the reaction are higher than the natural temperatures required, which seldom exceed 150° C. Dunoyer ⁽²³⁸⁾ observed conversion of montmorillonite to a well-crystallized mineral similar to cancrinite with concomitant formation of a heat-labile chlorite and either illite or illite-montmorillonite. Several workers ⁽²³⁹⁻²⁴²⁾ have considered the thermodynamic equilibrium of montmorillonite, and all agree that montmorillonites are stable at temperatures and pressures much higher than those involved in diagenesis. Experiments on dehydration of montmorillonite showed a threshold of dehydration between 130° to 150° C at atmospheric pressure, increasing to 260° C at 15,000 atmospheres of pressure. ⁽²³⁰⁾

Loss of interstitial water reduces the lattice spacing of montmorillonite to 9.57Å. In the presence of 10 percent KCl, illitization occurs at the threshold of dehydration but not at lower temperatures. At a reaction pressure of 5,000 atmospheres the spacing of the illite product is 10.88Å. ⁽²³⁰⁾ However, in montmorillonite from well borings of the Logbaba (Cretaceous) area, from the Dovala Basin, Cameroon, Africa, Dunoyer found dehydration to be complete at 75° C (rather than 130° C) and illitization to be initiated at 150° C, again indicating that critical temperatures for transformations in geological deposits are lower than those determined in laboratory measurements. ⁽²²⁴⁾ Dehydration causes collapse of the montmorillonite layers in a reversible process as long as bound cations of the alkali metals (K^+ , Rb^+ and Cs^+) have not sealed the structure. The chemical composition of the interstitial solutions is important in the transformation of montmorillonites as the adsorption of Mg^{+2} , Na^+ and K^+ between the layers creates random mixed-layering of illite-montmorillonite or chlorite-montmorillonite. Conversion of montmorillonite to these mixed-layer products is a natural process in diagenesis.

Potassium bentonites or metabentonites are examples of montmorillonitic clays formed by the devitrification of volcanic products which have been partially illitized by an environment with K^+ present, possibly a marine environment. In ordinary bentonites the level of potassium is very low, and the dominant mineral is montmorillonite. This is transformed into illite by contact with K^+ containing solutions. The dominant mineral in the metabentonite is an irregular, mixed-layer illite-montmorillonite. The illitization of metabentonites is incomplete because, according to Dunoyer, ⁽²²⁴⁾ the layers have a weak charge.

In general, the early diagenesis of montmorillonite results in conversion to irregular mixed-layer minerals, such as the conversion of bentonite to metabentonite, an illitic-montmorillonite, in the presence of the potassium cation. As diagenesis and adsorption of K^+ continues in a K^+ environment, a regular mixed-layer mineral, alleverdite (rectorite), appears. The end product of diagenesis in a K^+ environment is the simple

and ordered mineral illite. Diagenesis in an environment rich in the divalent magnesium cation with the resultant adsorption of Mg^{+2} leads successively to chlorite-montmorillonite or chlorite-vermiculite which are irregular mixed-layer minerals, then to regular mixed-layer corrensite, and finally to the simple and ordered mineral chlorite. These diagenetic changes are not simply the incorporation of elements into the lattice but also involve a reordering of the crystal structure.

The degradation of 2:1 layers in vermiculite produces a mixed-layer chlorite-vermiculite. Shover⁽²⁴³⁾ demonstrated conversion of vermiculite to chlorite by fixation of magnesium in a vermiculite-bearing shale and measured the degree of diagenetic recrystallization. The chlorite-vermiculites undergo a partial collapse on heating but become more stable to heat as diagenesis to chlorite progresses.

Illites are intermediate between montmorillonites and micas but should not be considered fragmented micas. Mixed-layer montmorillonites undergo a transformation involving a decrease in the percent of expandable layers, a decrease in cation exchange capacity, and an increase in both the tetrahedral charge and the alkali metal concentration while the octahedral charge remains constant.⁽²⁴⁴⁾

In general the diagenetic and metamorphic changes in clay minerals follow the following steps: (1) dehydration of interlayer spaces; (2) adsorption of potassium and/or magnesium from interstitial solutions; (3) redistribution of ions within the lattices, particularly the migration of aluminum from the octahedral layer to the tetrahedral layers; and, (4) ordering of the lattices, growth of the crystals, and massive authigenesis (neof ormation) of stable illite and chlorite.

Dunoyer⁽²²⁴⁾ has reviewed the isomorphic replacements in the crystal lattices during diagenesis of clay minerals. In general one ion can replace another only if the ionic radii are sufficiently close and only if the charge equilibrium is preserved. Substitutions may occur either in the tetrahedral or octahedral layers, or in both. Igneous and metamorphic rocks exposed to weathering produce minerals with more than one Al^{+3} for four tetrahedra. Micas have one Al^{+3} for four tetrahedra as given by the general formula of Si_3AlO_{10} for mica tetrahedral layers. The tetrahedral charge is the average number of Al^{+3} substituted for Si^{+4} per tetrahedron. This value for montmorillonites is in the range of 0.1 to 0.4 but rises to 0.5 for illites and in later diagenesis increases to 0.9 as the illites are gradually transformed to white micas called sericites. These differ from muscovite in that they are partial or complete sodium minerals such as paragonitic muscovites or paragonites.⁽²⁴⁵⁾

Other Pertinent Literature

The literature reviewed in the previous sections was divided into groups of related articles for convenience. The literature reviewed in this section simply is not related to the previous groups. The study of the synthesis of hectorite falls into this category and has provided a key to a better understanding of the chemistry of clay minerals.

Hectorite, an unusual clay mineral from Hector, California, was first described by Foshag and Woodford⁽²⁴⁶⁾ as a magnesian bentonite in which practically all the aluminum has been replaced by magnesium. It is the magnesian end member of the montmorillonite group. The almost total absence of Al_2O_3 , the presence of essential Li_2O , and the dominance of F over OH establish the Hector clay as a distinct mineral species.⁽²⁴⁷⁾ The name hectorite was proposed by Strese and Hofmann⁽²⁴⁸⁾ who conducted extensive synthesis of the mineral at atmospheric pressure. The clay-like product, a "two-dimensional gel," was prepared by the slow addition, with stirring, of a MgCl_2 solution to a boiling potassium silicate solution. Barrer and Dicks⁽²⁹⁾ synthesized alkylammonium hectorites at low temperature of about 100°C with crystallization complete in five to seven days. The stability and decomposition products of hectorite were reported by Sand and Ames,⁽²⁴⁹⁾ with discussion of the unique deposit at Hector, California, by Ames, et al.⁽²⁵⁰⁾ Granquist and Pollack⁽²⁵¹⁾ conducted synthesis at 300°C and 1200 psig pressure. The starting mixture was based upon the formula for the mineral of $[(\text{Li}_x\text{Mg}_{6-x})\text{Si}_8\text{O}_{20}(\text{OH})_4]_x \cdot x\text{Na}^+$. The pressure synthesis involved reaction of an aqueous slurry containing freshly precipitated $\text{Mg}(\text{OH})_2$ with additions of NaOH and/or LiOH or LiF . Kinetics and mechanism of the reaction were reviewed.

In a subsequent study Granquist and Pollack⁽²⁵¹⁾ described the hydrothermal synthesis of a randomly-interstratified dioctahedral 2:1 layer silicate which contained only Al^{+3} in the octahedral sites. The imbalance in charge caused by substitution of Al^{+3} for Si^{+4} in the tetrahedral layer was compensated by Na^+ ions present during synthesis, which was conducted at 300°C and a pressure of about 1200 psig and which required reaction times of one to two days.

Granquist, et al.,⁽²⁵²⁾ described the large-scale (100 gallon), rapid (several hours), batch-synthesis of a crystalline aluminian smectite at 300°C and 1240 psig pressure. The rapid crystallization was considered to be related to the use of NH_4^+ as the charge-balancing cation and to partial F/OH substitution. Two continuous methods of crystallization also were studied. These processes may be of interest in use of clay minerals at pressures greater than atmospheric.

Ames⁽²⁵⁵⁾ reported on the products obtained by aqueous reaction at 300°C of different clay minerals and cesium compounds. Results from several experiments showing the effect of pH on the final product are given in Table III. All of the bentonite runs with an initial pH greater than 10.0 resulted in the formation of well-crystallized pollucite. No reaction occurred in these mixtures at an initial pH of less than 10.0. These results indicate that at least part of the bentonite must be solubilized in order to form a pollucite with cesium. If the silica and alumina are already in soluble form, as they are in the metasilicate and aluminate mixtures, pollucite is free to crystallize directly.

Ames⁽²⁵⁵⁾ also used another clay mineral, vermiculite from the Transvaal, South Africa. This mineral is predominately vermiculite with a small amount of hydrobiotite and biotite. The hydrothermal products formed

with the vermiculite also are listed in Table III. The collapsed vermiculite product had an X-ray diffraction peak height somewhat larger for the product from run M-47 than for run M-46, indicating that a part of the vermiculite may have been solubilized in run M-46 with an initial pH of 12.5. However, most of the vermiculite survived, and lattice collapse to $10\text{\AA}$ indicates that some of the cesium was removed. Survival of the vermiculite after such drastic treatment without destruction of the lattice structure was unexpected.

A synthetic in-tank-solidification liquid was prepared by Ames⁽²⁵⁵⁾ using analytical data for liquid from tank 112-BY. Vermiculite was added to the simulated tank liquid, and the mixture was shaken for 23 hours. The vermiculite survived quite well, and X-ray diffraction tests showed that it also had partially collapsed to $10\text{\AA}$ and that some cesium was removed from the solution.

The ability of the vermiculite to persist in high caustic solutions is believed to be due to its characteristic of collapsing in the presence of cesium. Unlike bentonite, this makes the vast surfaces represented by the interlayers unavailable for caustic attack.⁽²⁵⁵⁾

TABLE III

Hydrothermal Reaction Products for Various Clay Minerals
and Cesium Compounds (at 300°C)⁽²⁵⁵⁾

<u>Run Number</u>	<u>Materials</u>	<u>Conditions</u>	<u>Initial pH</u>	<u>Final pH</u>	<u>Products</u>
M-41	Bentonite + $\text{Cs}_2\text{CO}_3 + \text{H}_2\text{O}$	300°C Two days	10.2	5.6	Good pollucite
M-42	Bentonite + $\text{CsCl} + \text{H}_2\text{O}$	300°C Two days	6.9	3.7	Bentonite
M-43	Sodium metasilicate + sodium aluminate + $\text{CsCl} + \text{H}_2\text{O}$	300°C Two days	12.9	13.0	Good pollucite
M-44*	Bentonite + $\text{CsCl} + \text{H}_2\text{O}$	90°C Two weeks	8.3	8.4	Bentonite
M-45	Bentonite + $\text{CsOH} + \text{H}_2\text{O}$	300°C Two days	12.7	6.2	Good pollucite
M-46	Vermiculite + $\text{CsOH} + \text{H}_2\text{O}$	300°C Two days	12.5	12.5	Collapsed vermiculite ($10\text{\AA}$)
M-47	Vermiculite + $\text{CsCl} + \text{H}_2\text{O}$	300°C Two days	6.5	5.2	Collapsed vermiculite ($10\text{\AA}$)

*Compare with Barney's run 98 (see Table I).⁽³⁾

In another study, Barrer and Falconer⁽²⁵⁷⁾ reported on the influence of structure on the feldspathoids cancrinite and sodalite in relation to exchangeable ions using a basic cancrinite $M_2O \cdot Al_2O_3 \cdot 2.4SiO_2 \cdot 0.6MOH \cdot xH_2O$ and a basic sodalite $M_2O \cdot Al_2O_3 \cdot 2.5SiO_2 \cdot 0.34MOH \cdot xH_2O$ where M is a monovalent cation. In these studies the silver basic feldspathoids were prepared and treated with chloride solutions of other monovalent cations.

Barrer and Falconer⁽²⁵⁷⁾ stated that, "Many experiments were made under controlled conditions using salts of the ions Li, Na, K, Rb, Cs, Ag, and Tl. With basic cancrinite 100 percent exchange could be effected with Li, Na, and Ag while partial exchange occurred with K, Tl, and Rb. Of the alkali metals, only Cs was not exchanged. In basic sodalite complete exchange was again achieved with Li, Na, and Ag while exchanges which were at least partial were found also with K, Tl, Rb and Cs. Earlier study has already established that ions which exchange with analcite include H, Li, Na, K, Rb, NH_4 , Ag, Tl and $PbOH^+$ but not Cs [Barrer and Hinds⁽²⁵⁸⁾ and Barrer⁽²⁵⁹⁾]. Basic cancrinite and analcite are thus similar in excluding Cs while chabazite [Barrer and Sammon⁽²⁶⁰⁾], which has a structure essentially akin to that of sodalite, resembles sodalite in accepting Cs."

Silver basic sodalite and silver basic cancrinite were treated with chlorite solutions of various Group I cations with results as given in Table IV.

TABLE IV

Cation and Hydrogen Ion Exchange⁽²⁵⁷⁾

<u>Exchange Form</u>	<u>Exchange</u>	<u>Conditions</u>	<u>Cations Entering Crystals as Percent of Initial Ag Content</u>
Ag Basic Sodalite	Ag $\rightarrow$ Rb	RbCl _{aq.} at 85° C for 5 days	38.2 Rb 8.1 H
	Ag $\rightarrow$ Cs	CsCl _{aq.} at 85° C for 5 days	15.2 Cs 17.9 H
Ag Basic Cancrinite	Ag $\rightarrow$ H	Distilled water at 85° C for 1 day	No change
	Ag $\rightarrow$ Na	NaCl _{aq.} at 85° C for 1 day	80.2 Na 11.5 H
	Ag $\rightarrow$ K	KCl _{aq.} at 85° C for 2 days	56.5 K 17.7 H
	Ag $\rightarrow$ Rb	RbCl _{aq.} at 85° C for 2 days	8.0 Rb 17.7 H
	Ag $\rightarrow$ Cs	CsCl _{aq.} at 85° C for 2 days	0 Cs 21.4 H

Barrer and Meier⁽²⁶¹⁾ reported on the structural and ion sieve properties of the synthetic zeolite known as Linde A. Structurally, the zeolite is composed of large α -cages of the type found in sodalite and very small cubic cages. These cages are formed of AlO_4 and SiO_4 tetrahedra, and in the case of this zeolite the formula is: $Na_{12} [12AlO_2 \cdot 12SiO_2] NaAlO_2 \cdot 29H_2O$ with the $NaAlO_2$ included in the β -cages. X-ray diffraction tests combined with ion exchange provided better understanding of the molecular sieve phenomena. The sodium zeolite, NaA was found to provide "a double sieve with respect to cations." The two critical values of the cationic diameters which govern the ion exchange properties of NaA were found to be about $4.5\text{\AA}$ for α -cages and $2.5\text{\AA}$ for β -sites.

The β -cages are of the sodalite structure formed by six-membered rings with a free diameter of $2.5 \pm 0.2\text{\AA}$ as determined by X-rays. The Tl^+ ion with a diameter of $2.9\text{\AA}$ cannot pass through the $2.5\text{\AA}$ free diameter in the six-membered ring as demonstrated by ion exchange measurements.

Keller⁽⁵¹⁾ investigated silicates of tetravalent elements from thorium to americium. Hydrothermal synthesis was used to prepare $ThSiO_4$, $USiO_4$, $PuSiO_4$, $NpSiO_4$, and $AmSiO_4$ with a zircon ($ZrSiO_4$) structure. These silicates were crystalline analogs of zircon with essentially the same lattice constants. Zircon and thorite are very stable and are found in beach and river sands.⁽²⁶²⁾ Silicate mineral reactivity is considered by Garrels and MacKenzie.⁽²⁶³⁾

DISCUSSION

There is some opinion that ion exchange characteristics of the clay minerals may have minor influence on the Clay Reaction Process. Inclusion of literature on this subject may be considered to have limited relevancy but, as yet, this has not been clearly established. Knowledge of the mechanisms of the different methods of cation fixation may be helpful in identifying the mineral and fixation method best suited to immobilizing a specific ion.

Two types of aqueous cation "fixation" are possible using clay minerals in which the degree of fixation and the mechanism differ. Clay minerals such as vermiculite hold large univalent cations K^+ , Rb^+ and Cs^+ in a "relatively fixed" position as a result of one-dimensional collapse of the lattice. "Fixation" of cesium in a pollucite crystal structure is a different mechanism and involves a three-dimensional cage and complete transformation of the aluminosilicate lattice during the synthesis. The energy required to remove cesium fixed in a three-dimensional aluminosilicate lattice is greater than that required to remove it from a relatively fixed condition between one-dimensionally collapsed lamellae of a clay mineral.

The ion fixation hypothesis supported by Barrer and Jones⁽³⁴⁾ proposes that the ions with a low hydration energy are absorbed on the lamellar interface. The ion becomes non-hydrated in the process and lowers the energy of the system. The hydration of an ion manifests itself in

diffusing the charge and increasing the distance between the cation and the negatively-charged lamella. Both of these effects weaken the electrostatic bond and increase the mobility of the ion, making it less fixed. The presence of hydrated ions swells the mineral. This thesis is supported by experiments of Barrer who notes that the clearest correlation for ion fixation is the inability of the fixed ions to hydrate.

The variation of cation fixation with different cations also may be explained by the "snug fit" hypothesis. The univalent cations K^+ , Rb^+ and Cs^+ , are large enough to approach all of the oxygens in the hexagons of the tetrahedral layer, and they partially penetrate these vacancies to form relatively strong electrostatic bonds. The smaller cations, Li^+ and Na^+ , are unable to do this and are more loosely bound. The smaller cations are also the ones that are expected to have the greatest extent of hydration. In this way the "snug fit" also explains the swelling of the clay mineral. The collapsed lattice may be looked upon as having each cation joining the lamellae by bonding into two hexagons, one on each side.

The insertion of Cs^+ between layers of oxygen hexagons by itself does not create pollucite; for one thing, the spacial arrangement of the oxygens is incorrect. Two similar hexagonal rings of oxygen are associated with Cs^+ in the pollucite aluminosilicate cage, but half of these, or six oxygens, are associated with the adjacent aluminosilicate cages for other Cs^+ ions in the unit cell of pollucite. Thus six additional oxygens and three additional Si^{+4} are required in the conversion of a clay mineral lattice to a pollucite lattice.

A third hypothesis is that Al^{+3} substitution for Si^{+4} in the tetrahedral layer and the subsequent concentration of the negative charge in those layers is responsible for cation fixation. This theory is unable to explain the fixation in fluorhectorites where there is substitution only in the octahedral layer. However, the thesis might be contributory since vermiculite with greater Al^{+3} substitution in the tetrahedral layers has a higher capacity for cation uptake than other clay minerals.

The high cation capacity in vermiculite does not necessarily imply an absolute attachment. Ames⁽²⁵³⁾ discussed the degree of fixation of K^+ and Cs^+ in clay minerals and noted that Scott and Smith⁽²⁵⁴⁾ found that 100 percent of the supposedly fixed K^+ in vermiculite was exchangeable with Na^+ if the K^+ concentration in the equilibrating Na^+ solution was kept very low. At less than 7 parts per million K^+ in the Na^+ equilibrating solution, 100 percent of the K^+ in biotite was exchangeable. Only 0.1 ppm K^+ in the equilibrating solution restricted muscovite K^+ exchange to 17 percent. (most reagent grade NaCl contains up to 3 ppm K^+ in a 1N NaCl solution.) Isotopic exchange simply would not work because the lattice will not be expanded by K^+ to enable the solution K^+ to get at the "fixed" K^+ in the mineral for exchange. Just the opposite conditions are necessary for lattice expansion. The K^+ in the equilibrating Na^+ solution must be kept very low in concentration (20 ppm K^+ in the equilibrating Na^+ solution is low enough for vermiculite to exchange

100 percent of its "fixed" K^+). If the K^+ in the equilibrating Na^+ solution is precipitated as potassium tetraphenylboron as fast as it is removed from the vermiculite, 100 percent of the K^+ "fixed" on the vermiculite is removed in 10 hours.⁽²⁵³⁾

Under the right conditions (very low Cs^+ in a Na^+ equilibrating solution), the vermiculite may release a sizeable portion of its "fixed" cesium. The above situation might occur if the vermiculite were placed in a dynamic natural flow system. There is a question as to whether or not the Cs^+ really can be "fixed" without being enclosed within a crystal lattice cage, such as pollucite.⁽²⁵³⁾

A natural solid solution series exists between analcime ($NaAlSi_3O_8 \cdot H_2O$) and pollucite ($CsAlSi_3O_8$), with all of the intermediate member compositions possible. In a residual liquor containing more than 10 moles per liter of sodium, the question arises as to how much cesium the analcime-pollucite will contain. The work of Regis, Sand, Calmon and Gilwood⁽⁴⁸⁾ in a $Na_2O \cdot Al_2O_3 \cdot SiO_2 \cdot H_2O$ system suggests that hydroxysodalite, $Na_8[Al_6Si_6O_{24}](OH)_2$, is the stable phase in a residual liquor system, if pure.

Ames⁽²⁵³⁾ notes that there are three possible positions for the same cation in open aluminosilicate structures with large cavities such as sodalite, cancrinite or a zeolite. The three positions can be illustrated in a Type A zeolite analyzed by Barrer.⁽²⁵⁴⁾ The composition of the Type A zeolite was given by Barrer as $Ag_{12}[12AlO_2 12SiO_2] Ag_{10} 9AgNO_3$. If the silver positions from left to right are described as (a), (b), and (c) positions, the position (a) silver cations are in the large cavity formed by the junction of four sodalite cages and function to neutralize the negative charges in the framework caused by substitution of Al^{+3} for Si^{+4} ions. In the (b) position the silver aluminate is structurally trapped within a sodalite cage and normally cannot be removed without destroying the cage. Inclusion of $AgAlO_2$ in the cage is a chance event, dependent on the concentration of $AgAlO_2$ in the growth solution. Zero to one moles of $AgAlO_2$ could have been included in this position depending upon the chemical growth environment. The $AgNO_3$ in (c) position is an occluded salt. It is relatively loosely bound and takes the place of water molecules normally found filling the large central cavity.

The studies by Barrer and associates have aided in identification of six-membered rings of SiO_4 and AlO_4 tetrahedra in pollucite, analcime, sodalite and the sodalite β -cages of Linde A zeolite. These six-membered rings have a free diameter of about $2.5\text{\AA}$ too small to permit passage of the $3.38\text{\AA}$ diameter Cs^+ ion. As a result, if Cs^+ ion is trapped within any of these cages by the formation of the cages around the Cs^+ ion, the cesium becomes immobilized with respect to ion exchange or leaching. Each cesium atom trapped in this manner is individually encapsulated within a stone-like aluminosilicate mineral. This process was described by Brownell, Chiaramonte and Isaacson⁽¹⁾ and by Isaacson and Brownell⁽⁶⁾ who termed it "STOPPER," the Stone Process for Permanent Encapsulation of Radionuclides.

The high-temperature (approximately 1000° C) clay process for immobilization of radionuclides studied at Brookhaven⁽⁷⁴⁻⁸³⁾ and Harwell^(85,86) may be regarded as a hybrid between the ion exchange process at low temperature and a high-temperature, silicate-melt process such as Thermal^(6,265-267) or Basalt Melt.^(2,266) Amphlett⁽⁸⁵⁾ comments on the high-temperature clay process as follows: "On heating to several hundred degrees both the water content and the exchange capacity are reduced, and above 900° C the structure changes to that of an aluminosilicate lattice within which the cations are firmly fixed; the exchange capacity is then negligible and cations are only leached out in minute amounts except by reagents which attack the lattice chemically." Thus three temperature ranges exist for immobilization of radionuclide cations in aluminosilicate lattices: approximately 100° C for clay reaction and gel processes; approximately 1000° C for high-temperature clay process; and, approximately 1200° C for most silicate melt processes.

A majority of the studies reviewed may be termed "cesium oriented." This is considered to be the result of at least three factors: cesium is the most difficult of the cations to immobilize and has been given much attention; cesium undergoes some unusual "fixation" reactions which have been given considerable study; and, the caging of cesium in aluminosilicate lattices illustrates the "stone" encapsulation process. Consideration must be given to the other cations in the fission products and actinides. In this regard all cations in the Group II through Group VIII of the periodic table form both silicate and complex silicate products that are stable, highly immobile and of very low solubility.⁽²⁶⁴⁾

Keller's studies on the hydrothermal formation of thorium, uranium, plutonium, neptunium and americium silicates (zircon analogs) show promise for immobilizing these cations. At the lower temperatures of the Clay Reaction Process complex silicates may result rather than zircon analogs, but the product solubility can be expected to remain low. Zircon and thorite are found in beach and river sands⁽²⁶²⁾ as residues after leaching for geologic time periods and show very low cation mobility.

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The bibliography of articles considered pertinent to the Clay Reaction Process is included at the end of the list of references. A number of articles not mentioned in the references have been added to this bibliography, therefore, it has been broken down into groups of related subjects for convenience. These groups are:

- A. Clay Materials which describes the different clay minerals and discusses the nomenclature associated with each.
- B. Structure is the group of articles directly associated with the structures of clays.
- C. Technology and Chemistry gives the background of the chemistry of the clay reactions and associated theory and technology.
- D. Adsorption and Ion Exchange, subdivided as General (D-1) and Radionuclide Application (D-2), is the largest group and covers the exchange and sorption of ions. Only selected articles considered to have value in the explanation of fixation and clay reactions are included in this group.
- E. Cation Exchange is an extension of Adsorption and Ion Exchange which covers general cation exchange theory and application.
- F. "Fixation," also subdivided into General (F-1) and Radionuclide Application (F-2), is a relative term used in the literature in at least two senses: (1) the "relatively fixed" position, for example, of potassium in muscovite mica; and, (2) the "fixed" position, for example, of cesium within the pollucite crystal structure. The energy required to remove the cesium from pollucite is greater than that required to remove potassium from muscovite.
- G. Diagenesis includes articles on a postdepositional process by which clays are transformed to sedimentary rocks.

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