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ORNL
FOREIGN TRIP REPORT

ORNL/FTR-2749

DATE: November 12, 1987

SUBJECT: Report of Foreign Travel by A. J. Mattus, Project Engineer, Engineered Waste Disposal Technology Group, Chemical Technology Division

TO: Herman Postma

FROM: A. J. Mattus

PURPOSE: To participate in a collaborative study of mixed waste immobilization in various types of U.S. and European cements at the Centre d'Études Nucléaires de Saclay in France.

SITES VISITED:

9/28/87	Centre d' Études Nucléaires de Saclay (CEN-S)	Saclay, France	A. Bernard
9/29/87	Centre d'Études Nucléaires de Fontenay-aux-Roses	Fontenay-aux-Roses, France	M. Jorda
9/30-10/27/87	Centre d'Études Nucléaires de Saclay (CEN-S)	Saclay, France	A. Bernard
10/28/87	Centre d'Études Nucléaires de Fontenay-aux-Roses	Fontenay-aux-Roses, France	J. Oliver
10/29-10/30/87	Centre d'Études Nucléaires de Saclay, (CEN-S)	Saclay, France	A. Bernard

ABSTRACT: The traveler participated in a joint collaborative study of waste immobilization together with French scientists at the Centre d'Études Nucléaires de Saclay (CEN-S) in France. This collaborative effort came about as a result of a July 26, 1983, agreement between the U.S. Department of Energy and the Commissariat à l'Énergie Atomique due to our common interest in

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the use of cement-based waste forms and radioactive wastes. Two types of liquid wastes that are both common and problematic for both the United States and France were of primary interest in this assignment. Two synthetic surrogate wastes, one based upon phosphate and the other nitrate, were utilized. Both wastes are classed as low-level radioactive wastes here in the United States; the phospate waste represents a decontamination waste from our Hanford, Washington, facilities, and the nitrate waste represents waste in our Melton Valley storage facility at Oak Ridge National Laboratory (ORNL). In the study, four types of French cement were used with our phosphate-based surrogate while three types were used with the nitrate-based waste; one of the cements was nearly identical to one of our U.S. Portland cements. The exact formulations used with each type of waste were decided upon jointly between my fellow U.S. colleague and our French coworkers at the Saclay research facility before my arrival. Waste forms were cast into various geometries to facilitate their use in both physical and chemical (leaching) tests following 28 days of curing.

Although the traveler spent most of his time working at the Saclay facility, a few days were spent at the French Centre d' Études Nucléaires de Fontenay-aux-Roses discussing the procedures to be followed for the leaching of radioactive samples. In addition, technical discussions were held with a number of their staff on matters of waste form testing, radionuclide transport and diffusion, carbonate complexation and precipitation equilibria, and general laboratory methods used by the French.

Report of Foreign Travel to Saclay, France

September 26-October 31, 1987

A. J. Mattus

The purpose of this foreign assignment has been to test various cement-based waste forms that have been prepared in cooperation with other French coworkers utilizing European cements and U.S. surrogate wastes. The basis for the cooperative exchange is the implementation of the July 26, 1983, agreement between the Commissariat à l'Énergie Atomique (CEA) and the U.S. Department of Energy in the field of radioactive waste treatment and immobilization. The assignment called for the formulation of liquid surrogate wastes that were of mutual interest to France and the United States. The nitrate-based waste is common to both countries in that ion-exchange processes in both countries produce alkaline, sodium nitrate-based, low-level radioactive wastes. This waste is presently stored in the Melton Valley storage tanks at ORNL. The phosphate-based waste is present at the Westinghouse Hanford Company in Hanford, Washington, and like the French nuclear industry, this liquid waste originates from decontamination processes. Due to the common interest in these two types of waste, the joint research agreement and subsequent assignment of the traveler were mutually beneficial to both countries. The traveler not only performed the testing of the resulting waste forms but also was able to learn of the differences and similarities that exist between the two waste immobilization programs, including the emphasis and interpretation of different types of tests. The traveler has learned much about the superior French waste immobilization program and will now describe many of the technical and programmatic differences in addition to the activities performed while on assignment at CEA.

September 28, 1987 Centre d'Études Nucléaires de Saclay (CEN-S)

The traveler spent this first day at the CEN-S facility participating in a discussion with his American coworker, T. L. Sams, who was already on assignment at CEN-S, and French engineers P. Bouniol, E. Revertegat, and A. Bernard, the latter also serving as the group leader of the Concrete and Coating Laboratory. The group discussed the results to date for tests conducted with the initial formulations, which were chosen before the traveler's arrival, and the status of testing protocols, whether already performed or pending. All applicable data obtained to date and the schedule of physical testing were reviewed. In addition to reviewing those test protocols of interest to us, we discussed those tests that were of most interest to the French and their reasons for conducting them.

On this first day, the traveler was given a tour of the laboratory facilities that were routinely used in their daily testing, and it quickly became apparent to the traveler that the materials testing and characterization of cement-based waste forms were of primary importance to the French. The laboratory is primarily involved in the physical testing of cement-based waste forms and the application of X-ray diffraction

techniques to performance and the chemical mechanisms involved in waste form alteration in differing chemical environments.

Additional time was spent on this first day discussing the costs associated with our proposed test program with R. Atabek, who is our equivalent of a section head at the ORNL and who is in control of the Concrete and Coating Laboratory. The work performed to date, as well as the remaining tests and required chemical analyses, was briefly discussed. During this meeting, the traveler and T. L. Sams were invited to the Fontenay-aux-Roses laboratory, which is located just south of Paris, for an all-day tour the following day. The purpose of the trip was to introduce us to research staff members who were involved in various aspects of waste immobilization testing and modeling as well as to give a general overview of the waste management support program at the Fontenay facility.

September 29, 1987 Fontenay-aux-Roses

Immediately upon arrival at the Fontenay facility, the traveler met with M. Jorda, the head of the Service d' Étude des Stockages de Dechets to discuss the line and matrix management structure at this site. At this time, M. Jorda briefly discussed the job function of each member of his staff. In addition to meeting the head of this department, we were also briefly introduced to the service assistant, F. Dubois. Following this discussion, a tour of the laboratory facilities was made, during which we met with four researchers who were working in different areas of waste immobilization or special waste storage-related problems.

The traveler's first meeting was with A. Lajudie, who is involved with the storage of vitrified high-activity waste, both short-term storage above ground and long-term storage 500 m below the surface. Steel vessels containing the vitrified waste are cooled in open air above ground for 30 years and then are placed into clay-lined auger holes. The clay lining is composed primarily of bentonite but also contains graphite and sand. Various mixtures are being studied for their thermal properties and their ability to expand or contract in the presence of water. The production of the clay wedges under high pressure was observed. The highest temperature at the surface of the glass is expected to be 170°C, while the maximum temperature of the outer clay surface should be 120°C; this outer surface will be in contact with granite.

Next, the traveler met with M. Petit and toured his laboratory. This researcher was primarily studying the interaction of water with rock and the clay material described above. His work was material science, related and involved, using TEM and SEM instrumentation to observe changes to both chemical and physical properties of natural and man-made materials under conditions encountered at four different waste sites in France. The primary goal of his work was to predict long-term changes in such materials following use in disposal environments.

The traveler met next with J. Oliver, who was involved primarily in the leaching and transport behavior of waste species in various types of waste matrix materials, especially cement-based materials. He showed us

special diffusion cells in which they study the rates of diffusion of waste species from lime-saturated water from one side of the cell to the other through a thin slab of the cement matrix material. The traveler was shown the curves of fractional release as a function of time and observed the deviation from the expected slope with short-term tests versus the long-term tests which are most often used by the French. Additionally, we discussed the hot cell leaching experiments that were under the control of J. Oliver. Discussions also included the reasons that the French thought their pozzolanic cements performed so well with difficult waste types. A detailed design of their diffusion cell was given to the traveler at this meeting so that we might construct the same type in the United States. The traveler also discussed a new phenomenon observed by the French in which radiocesium entered the vapor phase at room temperature and passed through a diffusion cell in which grout separated the two sides.

The traveler then met with A. Vitorge to discuss the geochemical work being performed in the Fontenay hot cell laboratory. This French researcher is involved in establishing the limits of solubility of various transuranic elements, especially in carbonate/bicarbonate environments. This work is related to modeling and speciation of various waste species; some seemingly very basic studies are being performed in hot cells, since not all literature dealing with thermodynamic data is considered accurate enough for their purposes. The results of their work are immediately used to update their release models following disposal and possible incursions of groundwaters.

September 30 - October 27, 1987 Centre d'Études Nucléaires de Saclay (CEN-S)

The traveler spent much of the first week aiding in the preparation of additional blank waste form specimens for our pending leaching tests. A variety of French cements were used to mix the formulations using different ratios of various additives. Waste forms were cast for leaching experiments as well as measurements of expansion and contraction, compressive strength, bleed water, X-ray diffraction, and mercury porosimetry. The waste forms that were prepared this week contained the phosphate-based waste, since the nitrate-based waste had been prepared before my arrival.

The French cure their waste forms for 28 d just as we do; as a result, this amount of time had to elapse before some tests, such as leaching, could be started. The nitrate-based samples were prepared early and were to be removed from their molds approximately 3 weeks after my arrival on-site. Phosphate-containing waste forms prepared using a slag cement, referred to as CLK cement, which contains mostly slag, were observed to be very corrosive to the iron molds that are used to prepare compressive strength samples. In addition, phosphate-containing waste forms made with a cement from France, which is similar to our Portland Cement and referred to as CPA, experienced extensive cracking during curing. The reasons for the cracking are unknown; thus, X-ray diffraction studies of this material will be obtained to investigate the cause or causes of this phenomenon.

The most impressive investigative tool that the French have perfected for understanding the physical and chemical changes occurring in cement-based materials is X-ray diffraction. They have been able to relate the physical properties of waste forms, such as compressive strength, porosity, shrinkage and expansion, and to some degree, leaching resistance, to the presence of various crystal phases that appear as the cement cures. This technology is relatively new for this purpose and was introduced in England approximately 3 years ago. The traveler held many meetings with E. Revertegat, the engineer in charge of these studies. There were opportunities to discuss the interaction of species such as phosphate, calcium, and nitrate upon the formation of certain crystal phases which are only stable under given conditions. The traveler's background in solution chemistry as well as some experience in X-ray diffraction provided much insight into the usefulness of this tool and the folly associated with the use of empirical techniques like ours in the United States.

The French have gained a lot of technical insight into the formation of expansive phases of cement that occur when certain anions are present, phases we refer to as ettringite. These exact, unwanted phases of cement are now causing thousands of drums of cemented waste to expand and break open at our Y-12 facility in Oak Ridge. The traveler believes that there is further need to study this technique more closely, jointly with the French, and would like to participate in a near-future exchange to study all of their X-ray techniques for use and implementation here in Oak Ridge. This approach is needed in our U.S. waste immobilization program to aid in reducing the costs of a nearly empirical approach to laboratory testing.

The traveler also was given the opportunity to learn about their long-term tests, in which the interaction of salt brine with discs of cement was being studied. Discussions with E. Revertegat revealed that they are studying the phase changes occurring in cement discs in the presence of salt brine over a 3-year period in order to investigate the action of chloride and sodium on these materials. These data will be important for burial of some wastes at a depth at which brine is present and would also be important to our hydrofracture closure plan here in Oak Ridge. The traveler observed that some of the discs were cracking due to the formation of chloride-based ettringite.

Additionally, discussions with P. Bouniol also revealed that they are studying the crystal-phase changes occurring in cement discs in saturated lime solutions in which various anions are present. Phase diagrams showing regions of stability are being generated from the resulting data for many types of formulations. Again, in these studies, X-ray diffraction techniques are being used to explain observed changes in the solid cement-based discs.

The traveler also observed waste forms that contained embedded Zircaloy-cladding material. P. Bouniol described this new technique, in which the cuttings are placed in a wire basket before embedding, producing a metal-free zone around the waste. The technique utilizes plasticizers and vibrating tables to obtain complete filling of the Zircaloy hulls,

without any observed voids. The traveler had just finished a paper study on this very topic before going to France, and new data and reports returned will supplement our data in this area of solid encapsulation. This type of waste is a problem for both countries and is an area where possible future exchange should be considered. P. Bouniol gave the traveler a photograph of the waste forms containing the cladding hulls under different conditions and was told that this photograph was for personal use and not to be duplicated; the photograph has been marked and treated as "business confidential." Reports of their work could not be given to the traveler; however, British reports on a process that is similar to theirs were presented to the traveler.

During the traveler's assignment at CEN-S, he also observed that the French have been able to fix as much as 55 vol % anion-exchange resin in cement-based materials without experiencing the swelling and cracking that usually destroy our U.S. waste forms upon immersion in water. Discussions held with P. Bouniol and A. Bernard revealed that the technology is classified, which prevents disclosing the means by which this is accomplished. It is the traveler's opinion that they are coating the resins with highly cross-linked polystyrene before fixing them in concrete. The traveler was told that the waste forms observed had been soaking in water for 5 months and showed no sign of cracking. It is unlikely that we will be able to work jointly in this area with the French because they appear to be ready to market the technology for financial gain. The traveler also observed a few polystyrene-based waste forms containing ion-exchange resins; these waste forms were split open and deformed, probably as a result of swelling after immersion in water. Based upon the traveler's experience, these waste forms were probably not cross-linked very much, if at all, because of the absence of contracted edges.

While there, the traveler also learned how to operate their mercury porosimeter and discovered the limitation encountered when it is used with cement-based wastes forms containing a lot of water. Interestingly, most of the French cement-based waste forms contain little water and therefore are much harder and less porous than ours; the French have been studying super plasticizers for many years and use them in most of their waste forms. The traveler observed many containers of different plasticizers and learned that they are used with their waste forms containing cladding hulls as well as resins. Discussions with P. Conche regarding their use of plasticizers revealed that when used with vibrating tables, dense waste forms containing few entrained air bubbles were obtained and the resulting volume of the waste form was reduced by as much as 5% when entrained air is removed from processing.

The traveler also met with J. C. Nominé to discuss his experiments with full-scale radioactive waste forms (some as heavy as 3 metric tons), some of which have been under way for as long as 8 years. The static leaching was performed inside steel vessels which were open to the air. Most of the waste forms viewed by the traveler were cement-based; however, two epoxy-based waste forms were also seen. The traveler was told by Nominé that work with epoxy and resin as well as nitrate was under way at Grenoble. The traveler was also given the opportunity to suit up and

enter their hot cell area where a 200-L epoxy waste form, which had been immersed in water for 3 years, had just been removed and sawed in half. The waste form contained a lot of miscellaneous material, such as plastic and alpha-contaminated waste. The top of the waste form appeared to contain more waste than the bottom, apparently due to flotation of some of the waste as a result of density differences prior to hardening of the form. The traveler also discussed polymer impregnation of waste forms with Nominé and was told that work had been done in the past by A. Bernard with the concept of impregnating cement-based waste forms, but it was no longer being done. The traveler was told that Electricity de France (EDF) was using polymers to encapsulate ion-exchange resins, based upon the original German process; however, the process has been modified by the French. The modified process employs mobile units and is referred to as the STEAJE Process.

The traveler was aware that Nominé had done full-scale leaching tests with bitumen-based waste forms from reading his publications in the past, and since the traveler had just finished an extensive test program using bitumen, this topic was discussed. It seems that the French are continuing to use bitumen to immobilize alpha- and beta-contaminated sodium nitrate waste which is similar to that which exits our evaporators at ORNL and which is stored at Melton Valley. The traveler was told that the LUWA thin-film evaporator is currently used and that drums weighing 273 kg and containing 40% sodium nitrate salt are being produced.

The traveler reviewed photographs of our bitumen test samples for Nominé and discussed the swelling and degradation problems that we encountered here in the United States during our 1986 study. The traveler was told of the same observations in the French studies, and we continued to discuss the osmotic forces involved in the swelling and water vapor pressure depression. If plans of using bitumen here in Oak Ridge are ever realized, for any type of waste, a joint effort in this area to share our experiences would be of immeasurable value in offsetting potential long-term problems with this type of superior waste form.

Other interesting work discussed with J. C. Nominé was the current testing program with waste forms of different sizes. In his laboratory, he is leaching waste forms that are right circular cylinders of varying sizes: 0.2, 2, 20, and 200 L. The fractional release of waste species is expected to be directly related to the volume-to-surface ratios of the different-sized forms, but this has not been the case. It appears that the fractional release is much worse for the larger samples and would indicate that small-scale laboratory waste forms may be underestimating the effect of scale, an important piece of data for modelers. The traveler was told that no answer has been found for this phenomenon. The traveler suggested that the B.E.T. surface area differences be investigated instead of considering only the geometric surface area. Nominé was receptive to the ideas and will investigate. The traveler was given the supporting data for these scale studies, and the traveler intends to stay in touch with J. C. Nominé regarding future revelations from this work.

The traveler was given a tour of a hot cell building under construction in which an archive is being built for the storage of various radiologically hot samples from all parts of France. The samples will be stored in steel tubes for easy removal and will be monitored by camera and gas sampling tubes for possible long-term changes during storage. The researcher, J. C. Nominé, is in charge of this facility. Such an archive for representative samples of waste might be considered here in the United States but not simply for monitoring; availability of test specimens should be another underlying reason for archiving.

During this time period, the traveler was busy performing leach tests using the U.S. ANS 16.1 leach procedure. The waste forms prepared using the French molds were larger than ours, so larger leaching vessels had to be improvised from 5-L beakers. The leach tests are continuing even now, since they are each scheduled to run for 90 d. The waste forms containing nitrate were all started by the traveler, and those for the phosphate-based waste are scheduled to start November 2, in the traveler's absence. Communication by Telex and telephone will be maintained to monitor progress. The leachates generated from these tests will be analyzed for the cold elements, cesium, nitrate, uranium, and cobalt; cold strontium analyses are not possible for a number of reasons. The lesser degree of analytical sensitivity associated with cold elemental analysis caused the traveler to discuss possible hot leaching using radioelements with R. Atabek. It was decided that a limited number of hot leaches could be performed for us at the Fontenay-aux-Roses laboratory, where hot cells are available for such work. The French would not permit the traveler to aid in the preparation of the hot samples. The traveler was told that he must choose formulas for the two wastes, which were to be based upon interest and physical data to date gathered in their laboratory. The traveler, in conjunction with his American coworker, T. L. Sams, chose a slag cement called CLC and a French cement referred to as CPA for these tests.

October 28, 1987 Centre d'Études Nucléaires de Fontenay-aux-Roses (CEN-F)

The traveler went to the CEN-F laboratory to meet with Dr. J. Oliver with the primary purpose of speaking about the leaching procedure that they would have to follow after preparing the radiologically hot waste forms. The traveler discussed the details of our ANS 16.1 leach test and left copies of the test along with detailed written instruction regarding sample preparation and the test. The traveler also provided isotope activity values which approximately matched those in our Oak Ridge Melton Valley tanks as well as those of the Hanford facilities; Oliver stated that the level of activity was not too high for their use. The radioelements used will be ^{137}Cs, ^{60}Co, and ^{90}Sr; uranium cannot be followed because they do not have a hot cell fluorimeter for this purpose.

The samples will be prepared November 9, cured for 28 d, and leached into 1988 for a total of at least 90 d. Due to the traveler's interest in the reported phenomenon regarding cesium volatility at room temperature in their special cell, as noted on the first visit, a more detailed followup discussion was held on the topic. The traveler was told that this was

of interest to the French simply because, on a few occasions, ^{137}Cs was observed to move from liquid tanks of waste to other areas when the migration was not attributed to misting of waste movement. The traveler was given vapor pressure data from a report but was refused a copy of the complete report without clearance from management. The traveler gave Oliver a possible explanation for this migration. This phenomenon is very interesting and may have some bearing upon the safety of storage of this isotope in certain environments. The traveler will stay in communication with this French researcher in order to keep abreast of any new explanations or data surrounding this anomaly.

October 29-October 30, 1987 Centre d'Études Nucléaires de Saclay (CEN-S)

The traveler spent most of the remaining time scheduling the leaching test changeout times with the French technicians and gathering all data obtained to date. Time was spent going over the analytical methods that should be used for the analysis of the leachates. It was agreed that some of the analyses will be performed by a private laboratory in Northern France, which will be cost-effective, while uranium analyses for the cold leaches will be performed in the CEN-F facility.

On the last day in the CEN-S facility, the traveler introduced American coworker, W. W. Pitt, to the Saclay staff and to R. Atabek of CEN-F. All data and results obtained to date were discussed, and written documentation of tests and some conclusions to date were exchanged.

SUMMARY AND CONCLUSIONS

It became immediately apparent to the traveler that the French have more to teach us in the field of waste form development and testing than we have to offer them. It is the traveler's opinion that the emphasis by the French is more scientific than our approach to waste form development in that they strive to understand the reasons why waste form materials may exhibit the properties they do. They do not take a purely empirical approach to laboratory testing of waste forms and, as a result, probably spend much less money and time achieving results. When working with impure natural materials that comprise cement-based waste forms, an analytical tool such as X-ray diffraction, together with some knowledge of solution chemistry and thermodynamics, can make a great difference. They have come close to correlating crystal growth and stability data with observed physical and chemical properties of waste form materials. This approach is needed in the United States to aid in explaining deviations in the expected performance of waste forms and other field-related materials, such as packing or barrier materials. The traveler has even observed the application of their technique to troubleshooting the reason for cracking construction materials in buildings in downtown Paris. With experience in X-ray technology and a background in material science, the traveler would like to study the French techniques in the future, especially as related to leaching performance and physical and chemical properties. The degree of personal and technical rapport established between the traveler and the French researchers should be built upon in the future through continued collaboration, especially in the area of X-ray diffraction.

APPENDIX A

Itinerary

September 26-27, 1987	Travel to Saclay, France
September 28, 1987	Saclay, France
September 29, 1987	Fontenay-aux-Roses, France
September 30- October 27, 1987	Saclay, France
October 28, 1987	Fontenay-aux-Roses, France
October 29-30, 1987	Saclay, France
October 31, 1987	Return to Oak Ridge

APPENDIX B

Persons Contacted

Rosemarie Atabek	CEA, Fontenay, France
Andre' Bernard	CEA, Saclay, France
Elaine Revertegat	CEA, Saclay, France
Pascal Bouniol	CEA, Saclay, France
Pascal Conche	CEA, Saclay, France
Jean-Claude Nominé	CEA, Saclay, France
Guy Baudin	CEA, Fontenay, France
Michel Jorda	CEA, Fontenay, France
A. Lajudie	CEA, Fontenay, France
Jean-Claude Petit	CEA, Fontenay, France
Michel Pigeon	Laval University, Civil Engineering Dept., Sainte-Foy, Quebec, Canada
A. Vitorge	CEA, Fontenay, France
J. Oliver	CEA, Fontenay, France
P. Cote	Burlington, Ontario, Canada
Wolfgang Attwenger	Seibersdorf, W. Germany

APPENDIX C

Literature Acquired

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