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US field testing programs and results

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Abstract

The US has been active in four major international in situ or field testing programs over the past two decades, involving the burial of simulated high-level waste forms and package components. These joint international efforts include: (1) burial of over 100 simulated Savannah River Site (SRS) high-level waste glass samples in the granite site at Stripa in Sweden, (2) burial of SRS glasses in clay at Mol, Belgium, (3) in situ testing of SRS waste forms and natural glass analogs in limestone at Ballidon in the UK, and (4) field testing of almost 2000 SRS and international waste form samples and package components in the salt site at the Waste Isolation Pilot Plant (WIPP) in the US. These programs are designed to supplement laboratory testing studies in order to obtain the most complete and realistic picture possible of waste glass behavior under realistic repository-relevant conditions. Waste glass performance thus far has been seen to be very good, and even better in the field than would be predicted by many so-called standardized laboratory leaching tests. Published by Elsevier Science B.V.

1. Background

Located in the US today are approximately 100 million gallons of high-level radioactive waste (HLW) containing in excess of one billion curies of radioactivity. Most of this inventory is a result of defense programs and is handled and managed on three main sites; the Hanford reservation in Richland, Washington, the Savannah River Site (SRS) in Aiken, South Carolina and the Idaho Chemical Processing Plant at Idaho Falls. While most of this inventory by volume is at Hanford, most of the radioactivity is contained at Savannah River.

A long-term objective of the high-level waste management program at the SRS, in Aiken, South Carolina, is to safely and effectively immobilize, approximately 34 million gallons of HLW, into high integrity, waste glass forms. This waste has been stored for many years in a liquid or semi-liquid form in large underground tanks on site. The waste is now being removed from these storage vessels and sent to a vitrification building, the Defense Waste Processing Facility (DWPF), where it is being processed into borosilicate waste glass logs. The waste

glass products are currently being stored on site in an interim storage building, before they will be shipped to a geologic federal repository for permanent disposal. An important part of the immobilization strategy is to produce high-quality waste glass products and ultimately, to understand and assess waste glass behavior, especially in the final resting place of the forms, the geologic repository. In order to assess, understand and to be able to predict the long-term behavior of these waste glass products, a multi-phase experimental effort has been in progress for several decades [1].

An important part of the SRS waste glass program involves assessing glass behavior under geologic, repository-relevant conditions, which includes: (a) laboratory-scale waste glass leaching studies, (b) repository simulation tests (ex., rock-cup experiments), and (c) in situ or field experiments. The most realistic tests that can be performed are also the most complex, and are representative of field experiments. By combining all of the elements of the repository-related program, involving standardized tests conducted under controlled conditions as well as experiments containing the variability of the repository, one can obtain the most complete picture possible of performance of waste glass products in an actual disposal scenario. This paper will provide an overview of US field testing efforts and discuss in more detail, results for in situ testing of SRS waste glasses in

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the materials interface interactions tests or MIIT program [2].

2. International in situ testing programs

2.1. Stripa tests (Sweden – granite)

The first major international field tests, involving burial of simulated US high-level waste glass forms and potential package components, were begun in 1982. These tests were conducted in the granite site of the Stripa mine located in Sweden. This joint effort was conducted by the Swedish Nuclear Fuels Safety Division of the Nuclear Fuel Supply (SKBF/KBS) in Sweden, and involved the participation of a variety of countries and facilities, including the University of Florida and the SRS, from the US [3]. Over 100 simulated SRS waste glasses were buried and over the next several years, samples were extracted from burial, analyzed, and results documented by the team. The samples were fabricated in the shape of ‘pineapple slices’ and ‘mini-cans’ with holes down the centers so they could be stacked on heater rods and heated to 90°C temperatures after em-

placement. Various stacking sequences were used which involved waste form samples as well as potential package components, including canister and overpack metals as well as possible backfill materials. The interfaces and subsequent interactions were examined and characterized by a variety of analytical techniques.

Among the objectives of this joint study was to evaluate the behavior of the SRS waste glass forms as part of a multi-barrier waste isolation system and in a realistic geologic repository setting (Fig. 1). This effort also helped to establish a characterization methodology for comparing and assessing field data with laboratory test results and using these data to better define leaching mechanisms and long-term performance of waste glass systems.

It was found from this study that the SRS waste glass compositions performed very well in the field, in fact even better in the field than would be expected from many so-called ‘standardized laboratory leaching tests’, which often use more aggressive leachants and leaching conditions [4].

2.2. MOL field tests (Belgium – Boom clay)

The High Activity Disposal Experimental Site (HADES) Underground Research Facility is part of the SCK/CEN Nuclear Research Center at Mol and is located in Boom clay in the northeast part of Belgium. This important and unique underground research facility (URL) is supported by the European Communities and its primary mission is to conduct a wide range of experiments related to disposal of high-level and long-lived radioactive wastes. Major tests conducted include geomechanics, hydrology, corrosion, waste form studies, and retention and migration of radionuclides. This URL became operational in 1983 and was expanded in 1987 with an additional experimental gallery, which further increased in situ testing possibilities. The URL is shown in Figs. 2(a) and (b), a test assembly prior to emplacement [5].



Fig. 1. Installation of in situ tests underground.

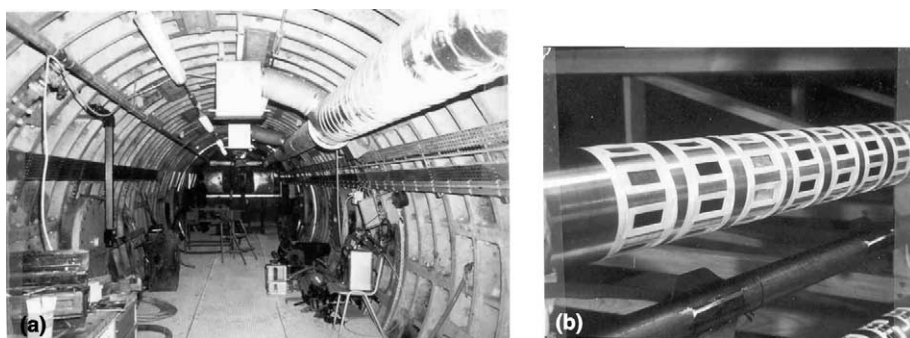


Fig. 2. (a) HADES URL in Mol, Belgium. (b) Samples loaded in test assemblies prior to emplacement.

The HADES underground research laboratory is considered by many as the best research facility of its type in the international waste management community. Over the years, many excellent studies and important results have been obtained, relevant to waste management efforts around the world, which, have been well documented [6]. In 1986, a series of SRS simulated HLW glass samples, similar in composition to those used in the Stripa study, became part of the HADES program. These samples were in the configuration of rectangles and were placed in the stainless steel assemblies shown in Fig. 2(b), before being buried in Boom clay approximately 224 m below the surface. They were buried for 2 years at temperatures of 90°C and also ambient conditions (16°C). Analyses of these glasses showed leaching depths of the glasses larger than those observed in the Stripa tests, with both selective and congruent dissolutions occurring [7]. These data and information were very useful not only with respect to geologic burial sites, but also for potential clay-like backfills that are also being considered as part of a multi-barrier isolation system for disposal of waste forms.

2.3. Ballidon field tests (England – limestone)

An interesting study was initiated in 1970 by the British Glass Industry Research Association and involved the burial of archaeological and modern glass specimens in carboniferous limestone at Ballidon in Derbyshire, in the UK. The original objective was to bury commercially relevant glass formulations and to dig them out and assess them after time periods of 1, 2, 4, 8, 16, 32, 64, 128, 256, and 512 years. The descriptions and compositions of the original glasses, along with shapes and measurements, are described elsewhere [8].

In 1986, following extraction of samples at five sites within the mound, a joint effort was started between the SRS and Dr Roy Newton, of the University of Sheffield, involving emplacement of an additional 46 samples in the recently vacated positions (Fig. 3). This included a series of SRS simulated HLW glasses, along with a standard glass and a glass analog. Samples were to be retrieved at time periods of 1, 2, 5, and 16 years. The 1-, 2-, and 5-year samples have been excavated, characterized and evaluated and are reported elsewhere [9]. The 16-year samples are scheduled to be retrieved in the year 2002.

These Ballidon tests represent the simplest of the field tests involving burial of US glasses. All the tests were conducted at ambient temperature and simply involved digging holes into the limestone mound and burying the glasses in shallow formations, which were then marked by pole markers. Post-burial analyses of these glasses showed that they exhibited very good durability in the limestone environment and produced two major leach-



Fig. 3. SRS glass samples being buried at Ballidon, UK.

ing regions; an outermost precipitated layer several microns thick and below that, a glass interaction zone, less than 1 μm thick, which also consisted of sub-layers.

2.4. WIPP MIIT tests (US – salt)

The mission of the Waste Isolation Pilot Plant or WIPP was defined by the US Congress as ‘providing a research and development facility to demonstrate the safe disposal of radioactive waste resulting from the defense activities and programs of the United States’. Construction of the WIPP began in 1981 in the salt beds in Carlsbad, New Mexico. The WIPP excavation consists of more than four miles of tunnels at a depth of approximately 655 m in the Salado Formation. This facility will be used: (a) to permanently isolate transuranic (TRU) waste generated from US defense programs, and (b) to serve as an ‘underground laboratory’ to evaluate and demonstrate safe and effective disposal of TRU wastes (simulated and actual), as well as defense high-level waste (simulated DHLW only) [10].

The materials interface interactions test (MIIT) program, started in 1986, represents the first in situ or field tests to be conducted in the US, involving burial of simulated (non-radioactive) high-level waste glasses and potential package components [11,12]. Included in the study were over 900 waste form samples comprising 15 different systems supplied by seven countries. Also included were almost 300 potential canister or overpack metal samples of 11 different metals along with more than 500 geologic and backfill specimens. There were almost 2000 relevant interactions that characterized this five-year field testing effort, which was conducted in the bedded salt site at the WIPP, near Carlsbad, New Mexico [12]. The MIIT program involved the participation of a variety of national and international organizations, laboratories and universities in France,

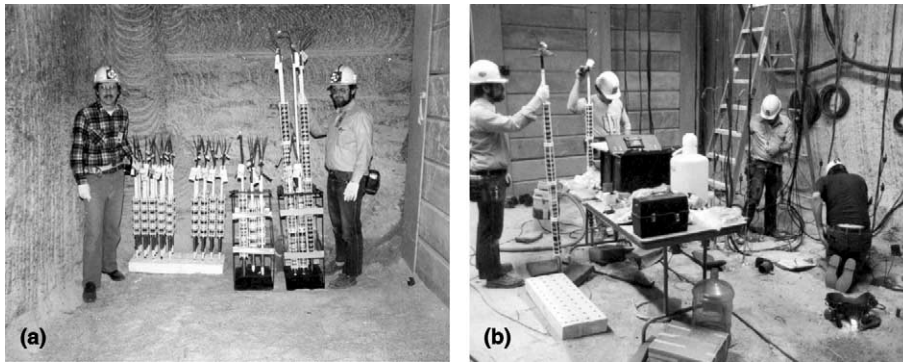


Fig. 4. (a) 50 MIIT assemblies prior to installation. (b) Installation of MIIT test assemblies in WIPP.

Germany, Belgium, Canada, Japan, the UK, Sweden, as well as the US. This program represents one of the largest, most cooperative ventures of its type in the international waste management community. The 50 MIIT assemblies and their installation at WIPP are shown in Figs. 4(a) and (b).

MIIT was operated and managed jointly by the SRS (Aiken, South Carolina) and Sandia National Laboratories (Albuquerque, New Mexico), and sponsored by the US Department of Energy. There were many participants involved, some who supplied samples, others who conducted post-test analyses and some who participated in a Peer Review process of the effort. US participants included the Hanford Waste Vitrification Project, Battelle Pacific Northwest Laboratory, the University of Florida, Catholic University of America, University of New Mexico, the Pennsylvania State University, Clemson University, and the Materials Characterization Center. International representatives include Hahn–Meitner Institute and Kernforschungszentrum Karlsruhe (Germany), Centre D’Etudes Nucleaires (France), the Atomic Energy of Canada (Canada), British Nuclear Fuels (UK), Studiecetrum Voor Kernenergie/Centre D’Etude de L’Energie Nucleaire (Belgium), Japan Atomic Energy Research Institute (Japan), and the Chalmers Institute of Technology (Sweden) [13,14].

There were many unique features associated with the design of the MIIT experiments. Among the most important features was that meaningful solution data could be obtained for the first time in a field experiment involving burial of the US waste glasses. This represented an important contribution to assessing and understanding waste glass leaching behavior. Glass, metal, and geologic samples, along with brine leachate solutions, were obtained from WIPP at pre-determined time intervals over the five-year testing program. A summary of some of the more interesting and relevant findings, involving SRS waste glass evaluated in the MIIT program, is given below [15].

3. MIIT results

3.1. Compositional correlations

The compositions of the different waste forms provided by each of the international participants in MIIT have been correlated by Ramsey and Wicks [16] using compositional ternaries. These interesting correlations were made based on structural considerations, bonding energies, and surface layer characteristics. These efforts suggest that for the variety of waste glass systems studied world-wide, the behavior of many of these systems, such as the borosilicate waste forms, should be very similar, including leaching mechanisms. In Fig. 5, a compositional ternary relating MIIT systems based on structural role of components as network formers, intermediates and modifiers within the random network of glass, is shown.

3.2. Post-test analyses of SRS waste glass (SRL 165/ TDS)

An integrated study approach was used to characterize and assess waste form behavior and the interaction

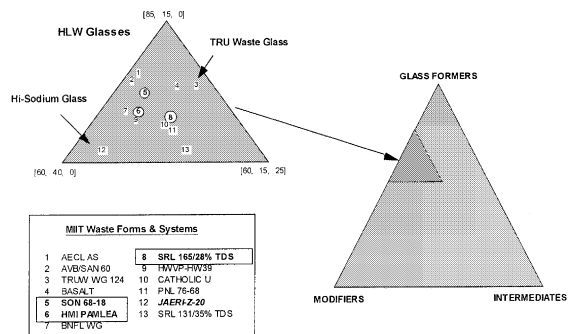


Fig. 5. Compositional correlations of MIIT waste glass compositions.

layers and zones produced during burial. The performance of SRS waste glass, based on both (a) surface studies and (b) solution analyses, was seen to be very good, after burial in the salt environment at WIPP for time periods of 1, 2, and 5 years [2,17–20].

3.2.1. Integrated study approach

The integrated study approach combines solution analyses with detailed bulk and surface studies of reacted waste forms. A variety of analytical techniques were used to characterize the composition, chemistry and structure of glass and waste components on, in and through leached surface layers. Depth profiling was also used. The MIIT study emphasized optical microscopy (OM), scanning electron microscopy (SEM), energy dispersion X-rays (EDX), electron microprobe analysis (EMP), secondary ion-mass spectrometry (SIMS), Auger electron spectroscopy (AES), transmission electron microscopy (TEM), Fourier transform infrared reflection spectroscopy (FTIRRS) and wide angle X-ray diffraction (WAXD). In addition to the various bulk and surface studies performed, solution analyses of brine leachates were conducted via inductively coupled plasma mass spectroscopy (ICP-MS). The integrated study approach and analytical techniques are summarized in Fig. 6.

3.2.2. Precipitated and glass reaction zones

Precipitated and glass reaction zones were analyzed and measured as a function of time and other relevant conditions by Tacca and Wicks using SEM/EDX [21]. Noted were two distinct regions; an outermost precipitated layer and an inner glass reaction zone. The outermost layer consists mainly of precipitated salt phases and includes crystalline as well as amorphous regions as determined by SEM, EDX and WAXD. These phases were attributed to the brine and salt precipitates that form on the glass surface during the MIIT leaching tests. There are two important points to note concerning the glass reaction region underneath the precipitated layers; (1) the amount of interaction of the glass with sur-

rounding brine environment, as measured by the intrusion of Mg and Cl from the brine into the glass, is very low and (2) the rate of interaction decreases with increasing time. These observations indicated that chemical durability of the glass is very good and actually becomes better with increasing time.

The reacted glass surfaces were investigated in more detail by other analytical techniques, including FTIRRS, by Clark et al. [22] at the University of Florida, and by SIMS, by Lodding et al. [23] of Chalmers University in Sweden. FTIRRS showed that the disruption to the glass structure was qualitatively small, while SIMS provided one of the most detailed tools for mapping individual elements throughout leached layers of the burial glasses. SIMS showed that the precipitated layer actually consists of two individual layers and the reaction zone of the glass consists of at least three individual leached layers (Fig. 7) [23].

By combining studies of SEM, EDX, FTIRRS, WAXD, TEM, EMP, and most importantly, the SIMS studies of Lodding, a composite picture of leaching of SRS waste glass in WIPP was developed and is summarized in Fig. 8 [24].

Each of the various precipitated and reacted glass layers are discussed below:

α_0 – *Outermost precipitated salt layer*: The outermost precipitated layer was studied by Vernez of the CEA in France [25], Harker of Rockwell International [26], Ewing while at the University of New Mexico [27] and SRS. Both amorphous as well as highly crystalline phases were observed, including $MgCl_2$, KCl, $CaSO_4$, NaCl, and a variety of mixed silicates, along with additional minor phases. The layer was generally very heterogeneous and varied in thickness. The chemistry of the layer was dominated by large quantities of Mg and Cl derived from the surrounding brine, along with other components such as Ca, Na, S, and Si. The salt layer, which was formed primarily as a result of the geologic environment and not from leaching the glass, is expected to effect subsequent glass leaching.

α_1 – *Precipitated glass layer*: Under the outermost precipitated salt layer, immediately adjacent to the glass surface is a thin, precipitated glass layer. This layer formed when elements from the glass leached and precipitated in this region. The layer is more uniform than the salt precipitated layer and characterized by large amounts of elements from the brine, including Mg and Cl, along with Si. This layer is also relatively depleted in elements such as Al, Zr, and Fe, which are generally the least leachable species within the glass. This observation, along with ratios of other components present, morphology and subsequent brine analyses show that this is a precipitated region and not a selectively leached part of the original glass.

β_0 – *Major depletion zone*: Directly under the precipitated layers is where glass begins and represents a

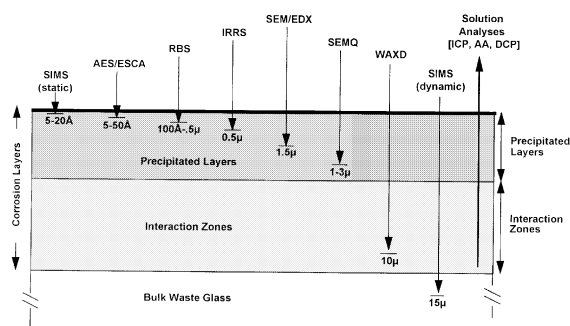


Fig. 6. Analytical tools used to study MIIT glasses.

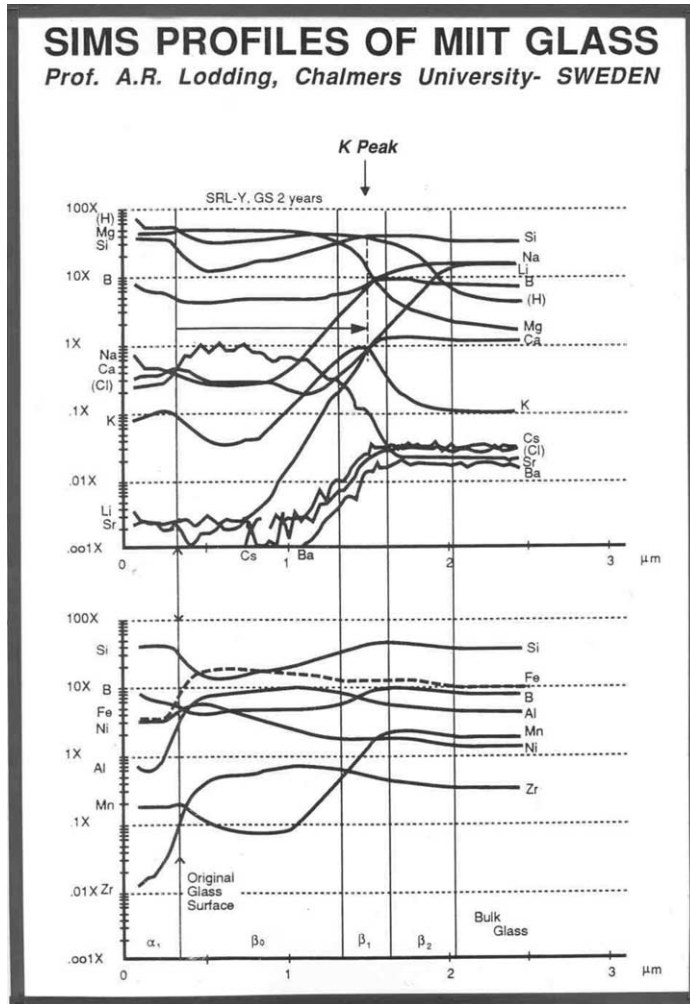


Fig. 7. SIMS Profiles of SRS waste glass after two-years burial.

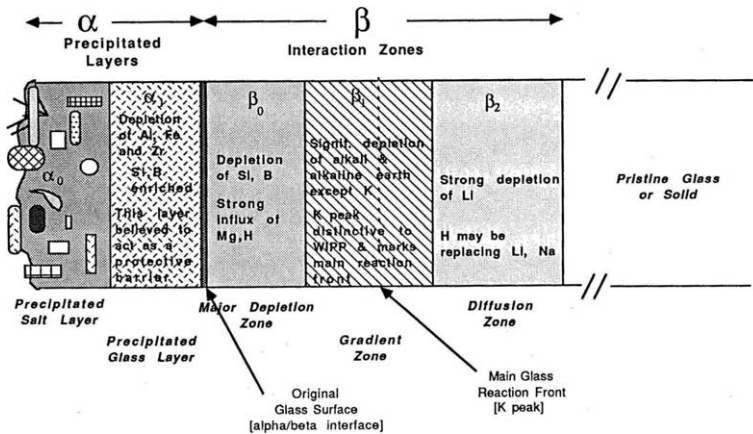


Fig. 8. Schematic representation of precipitated and leached layers in MIT waste glasses.

major depletion zone in the outermost glass surface. Here major components of the glass are depleted in this area. The region is further characterized by the intrusion of major brine components such as Mg. The original glass surface, the α/β interface, is located in this region.

β_1 – *Gradient zone*: Below the major depletion zone is the gradient zone, which is characterized by depletion of alkali and alkali earth components of the glass and enrichment in the main brine component, Mg. One of the most interesting features of this zone is the presence of a potassium peak. The distance from the α/β interface or glass surface to the potassium peak in the β_1 zone, represents the main reaction front of the glass after interacting with the surrounding environment.

β_2 – *Diffusion zone*: This innermost glass leached layer is believed to be similar to the gel layer which initially forms on simple glasses during leaching. The zone is characterized by depletion in Li from the glass and enrichment of H from the solution. The thickness is consistent with diffusion calculations of these components in the bulk glass.

3.3. Brine analyses

The MIIT program is the only in situ testing effort of this type which allows solution analyses to be obtained and subsequently, correlated with surface studies. Brine analyses were performed on leachates from selected boreholes containing SRL 165/TDS waste glass undoped and glass doped with Eu and Yb as chemical tracers. After measuring concentrations in solution, leach rates were calculated based on sample characteristics and testing conditions. In addition to assessing glass performance in the field, the solution analyses were used to define the original position of the glass surface in more detail. Analyses were performed by Macedo and co-workers at the Catholic University of America and are summarized in Table 1 [28,29].

Table 1
Leach rates and leaching depths of MIIT glasses based on brine analyses

Waste Glasses	Leach rates	Leach depths
A. SRL 165/TDS		
Zr	1.6×10^4 g/m ² day	0.02 $\mu\text{m}/\text{yr}$
Li		0.36
La	3.5	0.042
Eu	3.0	0.036
B. FR JSSA		
Y	7.4	0.090
Ce	1.2	0.014
Pr	3.1	0.027
Nd	1.2	0.015

There are several important observations that can be made from these data. First, the leaching behavior of SRS waste glass is very similar to other important international waste glass compositions used in this study. This includes the Japan–Switzerland–Sweden (JSS) composition. This observation was predicted by the compositional ternaries discussed earlier. Next, the actual leach rates are very low, generally less than 1 g/m² day. This is noted based on both brine analyses and by calculations of SIMS profiles, even for very mobile and non-radioactive species such as Li. Finally, if one takes into account the geometry of the stored waste, leaching depths can also be calculated and related to previously defined Nuclear Regulatory Commission release rate criteria [10 CFR Part 60]. For the MIIT tests, these data showed a release rate of species of interest of less than one part in 100,000 for all elements investigated, thus far [24,28,29].

4. Summary

In situ or field tests provide an important contribution to the overall goals of assessing, understanding and predicting long term behavior of waste forms in geologic burial sites. Field experiments are often complex and difficult to perform, but can also be the most realistic tests that can be conducted. Hence, they can be especially useful in helping to support and validate laboratory test findings and proposed models, and contribute to providing additional assurance in the safe and effective disposal of radioactive wastes. Based on all data currently available, the performance of nuclear waste glass systems can be excellent, when tested under realistic conditions, as determined by many studies performed by many investigators world-wide.

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