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PERFORMING A CHEMICAL DURABILITY TEST ON HIGH-LEVEL NUCLEAR WASTE GLASS (U)

by

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PERFORMING A CHEMICAL DURABILITY TEST ON RADIOACTIVE HIGH-LEVEL NUCLEAR WASTE GLASS by D. C. Beam, J. A. Napier, J. D. McCurry, and N. E. Bibler, Westinghouse Savannah River Company, Savannah River Site, Aiken, South Carolina 29802.

ABSTRACT

Savannah River Site (SRS), as a result of 25 years of producing nuclear defense materials for the USA, currently is storing ~30 million gallons of highly radioactive nuclear wastes. These wastes are stored as caustic slurries in large underground double-walled steel tanks. The Defense Waste Processing Facility (DWPF) nearing completion at SRS will incorporate the radionuclides in these wastes into solid borosilicate glass for final disposal in a geologic repository. Because of the variability of the wastes in the tanks, borosilicate glasses of different compositions will be produced by the DWPF during the 20-25 years required to solidify all the wastes at SRS. A chemical durability test, the Product Consistency Test (PCT), has been developed at SRS to measure the consistency of the durability of these glasses. Because of fission products and transuranic elements in the wastes, the glasses will be highly radioactive (surface radiation dose rate of ~2000 rad/hr). This paper describes the remote and hands-on procedures for performing the PCT on these radioactive glasses. Results will be presented that indicate the good precision of the PCT and indicate some of the chemistry involved in leaching radioactive elements from the glass.

The PCT is a crushed glass leach test where glass particles ranging from 100 to 200 mesh are placed in deionized water for seven days at 90°C. The durability of the glass is determined by measuring the amounts of soluble elements such as B, Li, and Na, that leach from the glass into the water during this time. Other elements and radionuclides such as Cs-137, Sr-90, and Pu-238, in the glass are also measured. Normally the PCT is performed in triplicate for each glass with duplicate blanks. All procedures that involve the glass have to be performed remotely in the SCF. These procedures are crushing, grinding, sieving, washing, and drying the glass and performing the test itself in a 90°C oven. After the tests, the leach vessels are cooled and the leachates decanted from the glass samples. The radioactivity of the leachates themselves is then low enough so they can be safely transferred to a radiochemical hood where they can be handled directly. Here they are filtered, acidified, and analyzed by nonradioactive and radioactive techniques. Comparison of the results in triplicate tests for the major soluble elements B, Li, and Na in the glass indicates an excellent precision for the PCT. The relative standard deviations were in the range 2-5%. Normalized concentrations for the elements (the leachate concentration of an element or radionuclide normalized to its concentration in the glass) are a measure of how fast that element leaches from the glass. Results indicate that leach rates are in the order Li≈B≥Na>K>Si>Al>Cs-137>Pu-238>Fe>Sr-90. These rates are affected by the chemistry of the leaching process.

ACKNOWLEDGMENT

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TITLES OF SLIDES

1. Title Slide: "Performing a Chemical Durability Test on Radioactive High-Level Nuclear Waste Glass," by D. C. Beam, J. A. Napier, J. D. McCurry, and N. E. Bibler

2. Second Title Slide: "Performing the Product Consistency Test (PCT) on Radioactive High-Level Nuclear Waste Glass," by D. C. Beam, J. A. Napier, J. D. McCurry, and N. E. Bibler.

3. Outline of Presentation.

4. High-Level Radioactive Wastes Stored at Savannah River Site.

5. High-Level Radioactive Waste Glass to be Produced at Savannah River Site.

6. Major Components of Two SRS Radioactive Glasses.

7. Purposes of the Product Consistency Test.

8. Development Goals of the Product Consistency Test.

9. General Procedures for the Product Consistency Test.

10. Shielded Cell Procedures for the Product Consistency Test.

11. Picture of the PCT Apparatus in a Shielded Cell.

12. Radiochemical Hood Procedures for the Product Consistency Test.

13. Analyses Performed on the Product Consistency Test Leachates.

14. PCT Results - Leachate Concentrations for the PCT Performed on Two Radioactive Glasses.

15. Equation for the Calculation of Normalized Concentration.

16. PCT Results - Normalized Concentrations (Grams Glass/Liter) for the PCT Performed on Two Radioactive Glasses.

17. PCT Results - Leaching B and Li from Gamma Irradiated SRS Glass.

18. Summary.

SUMMARY OF SLIDES

Slide 1:

At the Savannah River Site (SRS) in South Carolina, we have developed a chemical durability test or leach test for measuring the durabilities of nuclear waste glasses. My talk today will be about performing this test on radioactive nuclear waste glasses typical of those that will be produced to immobilize the wastes stored at SRS.

Slide 2

I use this second title slide to introduce you to the name of the test - the Product Consistency Test or PCT [1]. It is called this because the primary purpose of the test is to measure the consistencies of the durabilities of the glasses produced at SRS [2]. We have already used this test at SRS to measure the durabilities of both radioactive and nonradioactive glasses [1-3]. In my talk, I will concentrate on using the test on radioactive glasses.

Slide 3:

This is a brief outline of my talk. First, I'll give you some background information on the high-level nuclear wastes stored at SRS and the glasses that will be produced to immobilize these wastes. Second, I'll describe in some detail what's involved in performing the PCT on radioactive glass. Last, I'll present some typical results that we have obtained thus far for radioactive glasses containing actual waste and nonradioactive glasses containing simulated wastes.

Slide 4:

Currently at SRS, we have approximately 30 million gallons of high level radioactive waste [4]. This waste is stored as caustic sludges and supernates in one-million-gallon, double-walled tanks. These tanks are closely monitored by both SRS and state health and environmental control personnel. The material consists primarily of iron, aluminum and manganese hydrous oxides. These result from the chemical separation processes used to obtain and purify uranium, plutonium, and tritium for national defense purposes. The major radionuclides are fission products such as strontium-90 and cesium-137, and neutron activation products such as plutonium-238 and curium-244.

Slide 5:

Currently at SRS, construction of a facility called the Defense Waste Processing Facility (DWPF) is nearing completion. In this facility, the waste slurries will be mixed with a glass forming frit and melted at 1150°C to form a solid glass[5]. Over a 20-25 year period, approximately 700,000 gallons of glass in about 6,000 stainless steel canisters will be produced. The primary components in this glass will be silica, boron, and sodium from the frit and iron, aluminum and manganese from the waste. All the radionuclides including those that were mentioned earlier will comprise only approximately one weight percent of the glass. The solid glass product will have a dose rate of around 5,500 rad per hour at the surface of the canister and thus cannot be handled without appropriate radioactive shielding. Currently DOE plans to ship these canisters of glass to a geologic repository where they will be disposed of several hundred meters below the surface of the earth.

Slide 6

Several radioactive glasses have already been produced and tested in the research facilities at SRS [3,6,7]. The major compositions of two of the glasses are shown in this slide. These compositions are different because they each contained different waste and were made with different frits. Note especially the difference in the Al and Fe contents and the amount of radioactivity in each.

Slide 7:

The purpose of the PCT will be to measure the durabilities of the SRS nuclear waste glasses that will be produced by the DWPF. It will also be used to compare the durability of all glasses that will be produced during the 20 to 25 years of operation of the DWPF.

Slide 8:

In the developmental stages of this test, there were certain goals that we felt had to be achieved. First, we wanted a test that would be sensitive to glass composition and homogeneity. Also, the test had to be both precise and reliable. Last, but not least, it had to have a procedure that could be performed remotely with manipulators in a hot cell environment because of the intense radioactivity of the glass.

Slide 9:

After much experimental work, a procedure was developed. The general procedure is shown on this slide. Crushed glass is used so that the surface area of the glass exposed to the water is large. It is sieved to 100-200 mesh to get a reproducible surface area from test to test. The glass is then washed ultrasonically with distilled water and absolute alcohol to remove the fines. We then leach the glass in ASTM Type I water in stainless steel or teflon vessels at 90°C for seven days. The leachates are then filtered, acidified, and submitted for analyses.

Slide 10:

This slide gives you the details of the procedures that have to be performed remotely. Essentially all the procedures that involve the glass are performed with manipulators. Because of the radioactivity of the glass, stainless steel leach vessels have to be used rather than teflon™. The proper amount of water and glass (10 mL water per gram of glass) are put into the steel vessel. It is sealed and the final assembly is weighed and put into an oven where the temperature is continuously monitored at 90°C. After seven days, the assembly is removed from the oven and weighed again to determine if any water was lost during the heating in the oven. The leachate is then carefully decanted to a clean bottle. The glass is so durable that the radioactivity of the leachate is small enough so it can be removed from the cell and transferred to a radiochemical hood where it can be handled directly. The glass is then removed from the steel leach vessels and they are rinsed to determine how much radioactivity sorbed on the steel.

Slide 11:

This is a picture of the equipment in a shielded cell at SRS. In it you can see the steel mortar and pestle for crushing the glass, the sieves, a metal leach vessel, and some crushed glass and water in the beakers in the foreground. The water is put into a sealed container, put into the cell, and poured into the leach vessel after the glass is in it. The 90°C oven is in the background. A wrench assembly for sealing the vessels is in the center of the picture. A storage vessel for the glass is in the left of the picture. A funnel for transferring the crushed glass is in the top of the vessel. Notice that the glass of the vessel has turned brown because of the radiation in the cell. The cell operators are so skillful that they can perform the test without contaminating the leachant with any radioactive contaminants in the cell. This was confirmed with blank tests with the glass absent.

Slide 12

The leachate is taken to a radiochemical hood where it is transferred to a clean vessel. An aliquot is submitted for anion analysis. Another aliquot is taken to measure the final pH of the leachate. We then filter the leachate with a clean 0.45 micron filter to ensure that there are no particles of glass in the leachate. This filtrate is acidified and submitted for cation and radiochemical analyses. Acidification ensures that no cations precipitate prior to analyses. If small particles of glass had remained in the leachate, they would be dissolved during acidification; thus, we filter the leachate.

Slide 13:

This slide lists in detail the analyses that are performed on the PCT leachates when radioactive glass is tested. In some cases, only 15 mL of water are used in the test; thus the final leachate has to be diluted to obtain sufficient sample for all the analyses that have to be performed. The pH is measured with a glass electrode that remains in the radiochemical hood. Anions are measured primarily to check the purity of the water and the cleanliness of the leach vessel. Anions measured are F^-, Cl^-, NO_2^-, NO_3^-, and $SO_4^=$. All but one of the cations leached from the glass are measured by inductively coupled plasma-atomic emission spectroscopy. Boron is also measured by this technique. Atomic absorption spectroscopy is used for K. The detection system of each of the above instruments is located in a radiochemical hood to prevent personnel contamination. The concentrations of the radionuclides are measured by calibrated counting techniques. Gamma and beta counting are used for the fission products and alpha counting is used for the actinides.

SLIDE 14:

This slide shows the leachate concentrations and final pH values for the PCT performed on two radioactive glasses. The errors given are the standard deviations of the results of triplicate tests. Based on the standard deviations of the major elements in the glass, the relative precision of the test is 2-3%. The initial pH for the tests with both glasses was nominally 7. As a result of leaching during the PCT, the pH increases due to alkali ions in the glass exchanging with hydrogen ions in the leachate. The less durable the glass, the higher the pH becomes. Thus, it appears that the 200R glass is slightly less durable than the 165/42 glass. For each glass, the concentrations of the various nonradioactive and radioactive elements are different because of the different concentrations in the glass and their different chemistries in the glass and the leachate. Consequently, the concentrations themselves are not a good indication of the durability of the glass.

SLIDE 15:

To get an indication of the durability of the glass, it is necessary to normalize the leachate concentrations of the various elements to their respective concentrations in the glass. The equation for doing this is given in this slide. Normalized concentrations can be considered the concentration of glass in the PCT leachate based on that particular element in the glass. Of course this concentration is only the true concentration if all the elements in the glass leach at the same rate, are soluble in the leachate, and do not sorb on the leach vessel or the glass itself. Because of the many elements in nuclear waste glasses, this is never the case. However, the normalized concentrations of the soluble elements are good measures of the maximum rates that elements can be dissolved or released from the glass.

SLIDE 16:

This slide shows the normalized concentrations for two radioactive glasses. The best elements for indicating the durability of the glass are B, Li, and Na. These elements are soluble in the leachate. For the 200R glass, these elements have nearly equal normalized concentrations indicating that they are leaching essentially at the same rate. For the 165/42 glass, Li is indicating the fastest rate. From these results, it is clear that the 165/42 glass is more durable than the 200R glass. The 165/42 glass is more durable primarily because of its higher SiO_2 content (57.2% compared to 45.1% for 200R glass). The maximum rate of dissolution of the glass is given by Li, and usually B, and Na. This rate represents the fastest rate that radionuclides can be released from the glass. Three factors affect the release of an element from the glass. First, the element may be released slower from the glass because to its larger size or higher valence. This is probably the reason that K, Si, Al, and Sr-90 have lower normalized concentrations. Second, the element may sorb on the glass or on the leach vessel itself. This is the case for Cs-137 and Pu-238 [7]. Lastly, the element may be insoluble in the leachate. This is certainly the case for Pu-238.

During operation of the DWPF, the PCT will be applied to the glasses that will be produced. The maximum leachability of the glass will be equated to the normalized concentration of Li or B. Currently, this maximum leachability is being defined based on tests at SRS in conjunction with the US geologic repository program [8]. Once the maximum leachability has been defined, no glass that has a leachability higher than this will be shipped to the geologic repository.

SLIDE 17:

This slide shows you the results for B and Li for several PCT tests performed on samples of a nonradioactive simulated nuclear waste glass. These results were part of a test to determine if gamma radiation affected the durability of the glass [9]. Samples of the glass were irradiated with Co-60 gamma rays to three different doses. The PCT in triplicate was then performed on these samples as well as the unirradiated glass. The results for B and Li are clearly indicate that the radiation did not affect the durability of glass. These results also indicate the excellent precision of the PCT. As a result, the PCT is a good test for determining if and how some external factor affects the durability of a glass.

SLIDE 18:

With this slide I want to briefly summarize my talk. At SRS we have developed a durability test for radioactive nuclear waste glasses. This test can be performed reliably using manipulators in a hot cell environment. The results are very precise and an excellent measure of the durability of the glass. Because the PCT is such a good test, we are now in the process of proposing it to the American Society for Testing Materials as a national standard test for determining the durability of glasses, both radioactive and nonradioactive.

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SLIDE 1

PERFORMING A CHEMICAL DURABILITY TEST ON RADIOACTIVE HIGH-LEVEL NUCLEAR WASTE GLASS

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SLIDE 2

PERFORMING THE PRODUCT CONSISTENCY TEST (PCT) ON RADIOACTIVE HIGH-LEVEL NUCLEAR WASTE GLASS

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SLIDE 3

OUTLINE OF PRESENTATION

- BACKGROUND INFORMATION
- PROCEDURES FOR THE PCT
- TYPICAL RESULTS FOR RADIOACTIVE AND NONRADIOACTIVE GLASSES

SLIDE 4

HIGH-LEVEL RADIOACTIVE WASTES AT SRS

- ~ 30 MILLION GALLONS STORED IN DOUBLE WALLED TANKS
- STORED AS CAUSTIC SLUDGE AND SUPERNATE
- PRIMARILY Fe, Al, AND Mn HYDROUS OXIDES
- Sr-90 AND Pu-238 in SLUDGE, Cs-137 IN SUPERNATE

SLIDE 5

HIGH-LEVEL RADIOACTIVE GLASS TO BE PRODUCED AT SRS

- ~ 0.7 MILLION GALLONS OF GLASS

  IN ~ 6000 STAINLESS STEEL CANS
- MIXTURE OF WASTE AND FRIT MELTED AT 1150°C
- PRIMARILY Si, B, Na, Fe, Al, and Mn OXIDES
- ~ 1 WT % RADIONUCLIDES, Cs-137, ETC.
- DOSE RATE, ~ 5500 RAD/HR AT SURFACE

SLIDE 6

MAJOR COMPONENTS OF 200R AND 165/42 SRS RADIOACTIVE GLASSES

Nonradioactive Oxides (wt%)

Oxide	200R	165/42
B_2O_3	9.46	8.24
Li_2O	3.01	5.16
Na_2O	13.29	12.0
K_2O	3.49	0.0
SiO_2	45.1	56.6
U_3O_8	0.4	0.17

Oxide	200R	165/42
Al_2O_3	4.62	10.9
Fe_2O_3	12.3	6.49
CaO	1.47	0.37
MgO	1.25	1.07
MnO_2	2.61	2.05
NiO	0.53	0.64

Radionuclides (mCi/100 g glass)

Isotope	200R	165/42
Cs-137	1.76	9.1
Sr-90	50.7	1090.
Sb-125	0.09	0.34
Eu-154	0.23	3.8
Eu-155	0.18	8.6
Pu-238	1.0	4.2

PRODUCT CONSISTENCY TEST (PCT)

PURPOSES:

- MEASURE THE DURABILITY OF SRS NUCLEAR WASTE GLASSES

- COMPARE DURABILITIES OF ALL SRS GLASSES THROUGHOUT DWPF OPERATION (~ 20 YEARS)

PRODUCT CONSISTENCY TEST (PCT)

DEVELOPMENT GOALS

- WANT TEST SENSITIVE TO GLASS COMPOSITION AND HOMOGENEITY
- WANT TEST TO BE PRECISE AND RELIABLE
- WANT PROCEDURE THAT CAN BE PERFORMED REMOTELY

SLIDE 9

GENERAL PROCEDURES FOR THE PCT

- CRUSHED, SIEVED 100-200 MESH GLASS
- WASH GLASS TO REMOVE FINES
- LEACH IN DI WATER IN TEFLON OR SS VESSELS
- LEACH 7 DAYS AT 90°C
- ANALYZE FILTERED, ACIDIFIED LEACHATES

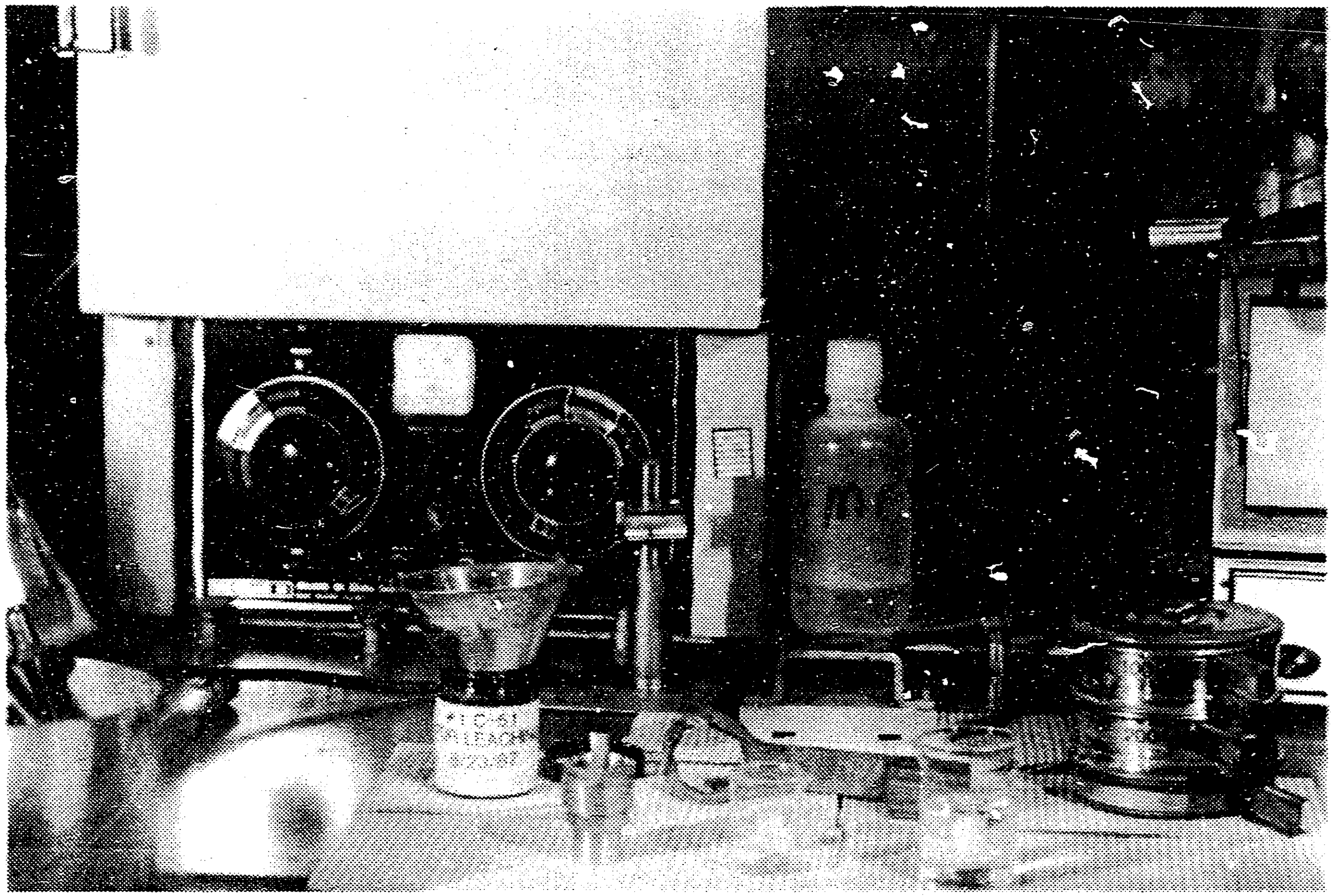
SLIDE 10

SHIELDED CELL PROCEDURES FOR PCT

- CRUSH, GRIND, SIEVE, WASH, AND DRY GLASS
- WEIGH VESSEL, GLASS, WATER, AND FINAL ASSEMBLY
- PUT IN 90°C OVEN, MONITOR TEMPERATURE
- AFTER 7 DAYS, REMOVE FROM OVEN AND WEIGH
- DECANT LEACHATE, TRANSFER TO HOOD
- REMOVE GLASS AND RINSE VESSELS

SLIDE 11

APPARATUS FOR PERFORMING THE PCT IN A SHIELDED CELL ENVIRONMENT



RADIOCHEMICAL HOOD PCT PROCEDURES

- TRANSFER LEACHATE TO CLEAN VESSEL
- SUBMIT FOR ANION ANALYSES
- MEASURE pH
- FILTER LEACHATE (0.45 MICRON)
- ACIDIFY AND SUBMIT FOR CATION ANALYSES
- SUBMIT FOR RADIOCHEMICAL ANALYSES

SLIDE 13

ANALYSES PERFORMED ON PCT LEACHATES

- pH
- ANIONS BY ION CHROMATOGRAPHY
- CATIONS BY PLASMA SPECTROSCOPY AND ATOMIC ABSORPTION
- RADIONUCLIDES BY GAMMA , ALPHA, AND BETA COUNTING

LEACHATE CONCENTRATIONS FOR THE PCT PERFORMED ON TWO RADIOACTIVE GLASSES

(Results of Triplicate Tests)

Nonradioactive Elements (ppm)	200R Glass	165/42 Glass
B	33.8 ± 1.8	10.4 ± 0.3
Li	13.6 ± 0.2	13.1 ± 0.4
Na	97.3 ± 3.5	32.1 ± 0.9
K	15.5 ± 0.4	-
Si	98.1 ± 1.1	87.1 ± 2.8
Al	7.4 ± 1.0	19.6 ± 0.6
Radionuclides (nCi/mL)		
Cs-137	3.5 ± 0.034	11.0 ± 0.4
Sr-90	1.89 ± 0.08	210. ± 17
Pu-238	0.98 ± 0.04	5.5 ± 1.9
Sb-125	0.53 ± 0.01	1.5 ± 0.2
Final pH	10.5	10.3

SLIDE 15

CALCULATION OF NORMALIZED CONCENTRATIONS

(INDICATES CONCENTRATION OF GLASS BASED ON A SPECIFIC ELEMENT)

$$N_i = C_{il}/C_{ig}$$

N_i = Normalized Concentration Based on Element i (grams glass/liter)

C_{il} = Concentration of Element i in Leachate (grams i/liter)

C_{ig} = Concentration of Element i in Glass (grams i/gram glass)

NORMALIZED LEACHATE CONCENTRATIONS

(GRAMS GLASS/LITER)

FOR THE PCT PERFORMED ON TWO RADIOACTIVE GLASSES

(Results of Triplicate Tests)

Nonradioactive Elements	200R Glass	165/42 Glass
B	1.1 ± 0.06	0.41 ± 0.01
Li	0.97 ±0.01	0.53 ± 0.02
Na	0.99 ± 0.04	0.34 ± 0 01
K	0.57 ± 0.01	-
Si	0.45 ± 0.005	0.35 ± 0.01
Al	0.30 ± 0.004	0.32 ± 0.01
Radionuclides		
Cs-137	0.20 ± 0.002	0.12 ± 0.004
Sr-90	0.004 ± 0.0002	0.019 ± 0.001
Pu-238	0.098 ± 0.004	0.13 ± 0.01
Sb-125	0.59 ± 0.004	0.44 ± 0.06

SLIDE 17

LEACHING B AND LI FROM GAMMA IRRADIATED SRP GLASS USING THE PCT CRUSHED GLASS TEST

Dose (gray) and Test No	Concentrations (ppm) in Leach Solutions	
	B	Li
Unirradiated		
Test 1	15.2	17.7
Test 2	15.2	17.7
Test 3	15.4	18.1
4.0×10^6		
Test 1	14.6	17.3
Test 2	15.0	17.8
Test 3	14.3	17.0
4.7×10^7		
Test 1	15.1	18.3
Test 2	15.2	18.4
Test 3	15.1	18.4
3.1×10^8		
Test 1	15.6	18.5
Test 2	15.0	18.1
Test 3	15.6	18.5

SLIDE 18

SUMMARY

THE PRODUCT CONSISTENCY TEST (PCT) IS:

- A CRUSHED GLASS LEACH TEST IN ASTM WATER
- PRECISE AND CAN BE PERFORMED REMOTELY
- SUITABLE FOR AN ASTM STANDARD TEST

END

DATE FILMED

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