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Characteristics of Low-Level Radioactive Waste

Decontamination Waste Program Annual Report for Fiscal Year 1991

Prepared by N. Morcus, J. W. McConnell, Jr., D. W. Akers

Idaho National Engineering Laboratory EG&G Idaho, Inc.

Prepared for U.S. Nuclear Regulatory Commission

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Decontamination Waste Program Annual Report for Fiscal Year 1991

Manuscript Completed: April 1992 Date Published: June 1992

Prepared by N. Morcos, J. W. McConnell, Jr., D. W. Akers

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Prepared for Division of Regulatory Applications Office of Nuclear Regulatory Research U.S. Nuclear Regulatory Commission Washington, DC 20555 NRC FIN A6359 Under DOE Contract No. DE-AC07-76ID01570

ABSTRACT

The objective of the Low-Level Radioactive Waste-Decontamination Waste Program (FIN A6359), funded by the United States Nuclear Regulatory Commission (NRC), is to provide base-line data on the physical stability and leachability of solidified waste streams generated in the decontamination process of primary coolant systems in operating nuclear power stations. The data include information on the chemical composition and characterization of these waste streams. In addition, this work is intended to evaluate waste form characterization tests identified in the "Technical Position on Waste Form" (Revision 1), prepared by the Low-Level Waste Management Branch of the NRC. This Branch Technical Position clarifies the methods to meet the requirements of 10 CFR Part 61. Samples of LOMI decontamination waste stream resins solidified in Portland cement, and unsolidified waste stream resins were obtained from the Peach Bottom commercial Luclear power station. The radioisotopic composition of the waste stream was determined, and the solidified samples were leached in demineralized and simulated sea water. The compressive strengths of samples immersed for 90 days in the two leachants was determined. In addition to the samples obtained from Peach Bottom, filter sludge waste stream and solidified waste forms were obtained from Nine Mile Point nuclear power station. The radioisotopic composition of the Nine Mile Point waste stream was determined.

> FIN A6359—Chare eristics of Low-Level Radioactive Waste: Decontamination Waste

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EXECUTIVE SUMMARY

The United States Code of Federal Regulations (CFR), Section 10, Part 61 (10 CFR 61), "Licensing Requirements for Land Disposal of Radioactive Wastes,"1 establishes a waste classification system based on radionuclide concentrations. Furthermore, the Low-Level Waste Management Branch of the NRC has clarified interpretation of the relevant regulatory guides with respect to when te solidification and waste performance in the "Technical Position on Waste Form," Revision 1.2 This Branch Technical Position (BTP) presents recommendations and guidance on waste performance test methods that satisfy the requirements of the NRC with respect to waste solidification and disposal. In addition, it provides guidance to establish waste solidification process formulation parameters that will produce free-standing solidified waste forms that meet the requirements of 10 CFR 61.

The research program at the Idaho National Engineeling Laboratory (INEL) is intended to provide the NRC with performance data on solidified waste that was obtained from nuclear power station waste streams and solidified by the generators. Solidified waste performance characteristics that were evaluated included assessment of the waste form stability, and its ability to meet the requirements of 10 CFR 61. In addition, this research effort addresses the impact of the waste stream chemical composition on the properties of the solidified waste. The waste streams evaluated under this program consist of solidified ionexchange resins that were used in processing waste generated in the decoatamination process of primary coolant plant systems. These resins are routinely solidified in cement, usually at the reactor site. The cement types used for solidification include Portland or mixtures of Portland cement and pozzolana. Program activities include determining the leachability of solidified radionuclides, stable metals, and organic chelating agents. In addition, the compressive strengths of leached waste-form specimens were measured before initiation of the leaching studies, and at specified immersion times in the leachant. The program at INEL also includes provision of technical assistance to the NRC on an as-needed basis. Work performed during the 1991 fiscal year was a continuation of previous work and is summarized below.

We obtained decontamination radwaste stream samples and solidified radwaste samples from two nuclear power stations. The chemical and radiological composition of the sampled waste streams were characterized. The most prominent radioisotopes observed were ⁶⁰Co, ⁵⁵Fe, and ⁶⁵Zn.

We initiated a waste form performance assessment in accordance with the recommended tests in the BTP. Leaching characteristics were determined for de-ionized and simulated sea water leachants. The resulting leachates were analyzed for inorganic, organic, and radiological constituents. Furthermore, a long-term waste form leachability test was initiated. This test was performed in accordance with a Long-Term Leaching Program Plan.

Compressive strength measurements were performed on solidified samples from two resin waste streams obtained from Peach Bottom in October, 1989. The samples were tested after seven and 90 days of immersion in deionized and simulated sea water. The results were compared with those of samples which were not immersed in the two leachants. No significant differences were observed among all samples.

Preparations to characterize the contents of the bulged Millstone solidified waste liner were completed, and the task was postponed to FY-93.

NUREG/CR-5601 was completed and is titled "Effect of pH on the Release of Radionuclides and Chelating Agents from Cement-Solidified Decontamination Ion-Exchange Resins Collected from Operating Nuclear Power Stations."³ In . addition, two journal articles were submitted to Waste Management journal for publication after review by the NRC Program Manager. The titles of the two journal articles are "Effect of Waste Form Size on the Leachability of Radionuclides Contained in Cement-Solidified Evaporator Concentrates Generated at Nuclear Power Stations." by C. V. McIsaac and S. T. Croney,⁴ and "Leachability of Chelated Decontamination Ion-Exchange Resins Solidified in Cement or Cement

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and Fly Ash at Nuclear Power Stations," by C. V. McIsaac (to be published).

Technical expertise was provided as requested and specified by the NRC Research Program Manager.

Characteristics of Low-Level Radioactive Waste-Decontamination Waste Program Annual Report for Fiscal Year 1991

1. INTRODUCTION

The objective of the Low-Level Radioactive Waste-Decontamination Waste Program (FIN A6359), funded by the United States Nuclear Regulatory Commission (NRC), is to provide baseline data on the physical stability and leachability of solidified waste streams generated in the decontamination process of primary coolant systems in operating nuclear power stations. In addition, program work includes characterizing the chamical composition of these waste streams.

The NRC has directed this work to evaluate waste form characterization tests identified in the "Technical Position on Waste Form," Revision 1.² prepared by the Low-Level Waste Nanagement Branch of the NRC. This Branch Technical Position (BTP) clarifies the methods to meet the requirements of 10 CFR 61¹ with respect to waste form stabil' y and leachability.

Table 1 lists the tasks associated with the program, and summarizes the work accomplished and the current status of each task.

Sections 2, 3, and 4 present findings from the analysis of waste samples taken from Peach Bottom, Nine Mile Point, and Oyster Creek, respectively. Section 5 presents the status of the bulged Millstone liner.

Sections 6, 7, 8, and 9 describe the documents included in the program and the plans they support. Sections 10 and 11 explain the meetings that participants attended and the status of the annual program review.

their decision to solidify their warte.

Task	Status
Obtain decontamination radwaste from a nuclear power station such as Oyster Creek, which will use the LOMI decontamination process, and low-level waste evaporator concentrates from Nine Mile Point.	 it is a dge samples and thirty solidified waste for as were obtained from Nine Mile Point nuclear power static. Plans to collect waste and samples fr. Oyster Creek nuclear power station could be not be completed due to uncertainty about

Table 1. Status of the low-level radioactive waste program.

Table 1, (continued).

Task

2. Develop and begin implementing a chemical composition characterization program plan for the solidified waste streams obtained. During waste stream characterization, include analyses (at the parts per million level) for elements such as metals, organics such as chelating agents, and radiological constituents such as gamma-ray emitters. ⁹⁰Sr, ⁵⁵Fe, ⁶³Ni, and ²⁴¹Pu. Consider waste streams from Nine Mile Point and another plant such as Ovster Creek.

> Report the final results of this task under Tasks 3 and 6. Report the progress in the FY-91 annual report.

3. Initiate waste form performance assessment in accordance with the recommended tests in the BTP, including compressive strength, radiation stability, biodegradation, leachability, immersion, thermal cycling, and free liquid amount. Use characterized and solidified decontamination waste streams obtained from nuclear power stations under Task 1 for this task.

Analyze leachates for metals such as calcium, boron, organic compounds such as chelating agents, and radiological constituents such as gamma-ray emitters, ⁹⁰Sr. ⁵⁵Fe, ⁶³Ni, and ²⁴¹Pu

4. Analyze relevant data for waste forms that fail to meet BTP criteria. Determine, for these forms, if their failure may be correlated to the presence of trace chemicals in the waste streams from which they were derived. Present the final results in a letter report. Status

The program plan for chemical waste stream characterization was completed. line Mile Point filter sludge and Peach Bottom ion exchange resin waste streams were characterized for radioisotopic content. Complete chemical analysis of these waste streams was initiated in September, 1991.

Samples from Oyster Creek nuclear power station were not obtained because this plant has not decided to solidify its waste during the course of this fiscal year. Examination of these samples will continue in FY-92 with additional measurements.

 Nine Mile Point waste stream characterization indicates that it is Class A waste Based on the BTP, this waste classification does not require solidification. We therefore deterred this task into FY-92. We expect at that time to obtain waste that is Class B.

 Because no waste forms were assessed this fiscal year, none failed to meet the BTP.

Table 1. (continued).

Task	Status

- 5. Determine generic-scaling factors and dose-to-curie factors for long-lived and hard-to-measure radionuclides found in waste streams and activated metals. Radionuclides of concern are listed in Tables I and 2 of 10 CFR 61.55, and may include ⁹³Nb, ¹²⁶Sn, ^{108m}Ag, ³⁶Cl, ¹²⁹I, ¹⁴C, and ⁹⁹Tc. Base the scaling factors on measurements performed on actual waste from nuclear power stations. Document the results in annual and NUREG/CR reports.
- Begin leaching characterization of waste forms obtained under Task 1 in accordance with American Nuclear Society (ANS) 16.1.⁵ Use deionized and simulated sea water for leachants. Determine leachability indexes for ⁹⁰Sr, ⁵⁵Fe, ⁶³Ni, ²⁴¹Pu, metals, radionuclides, and major chemical components such as chelating agents, boron, and calcium.

Determine the compressive strength of these waste forms at the end of the leaching study. Document results in annual and NUREG/CR reports.

7. Evaluate the stability of solidified waste forms under long-term leaching conditions. Leach actual solidified waste from nuclear power stations in deionized and simulated sea water for a period of at least one year in accordance with a Long-Term Leaching Program Plan (LTLPP), Develop and write the LTLPP by March 21, 1991. Change leachates every 43 days after the standard 90 days sampling protocol. Analyze leachates for calcium and other metals, boron, organic compounds such as chelating agents, and radiological constituents such as gammaray emitters, 90Sr, 55Fe, 63Ni, and 241Pu.

 No work has been performed on this task as it requires specific initiating cues from the NRC Program Manager.

6. The waste steam from Nine Mile Point was obtained under Task 1. Analysis of this waste stream indicated that it was Class A waste. This classification precluded further assessment of the solidified waste in accordance with the BTP.

7. A Long-Term Leaching Plan was developed and circulated for review. Comments were received from the NRC Program Manager and were addressed. No samples were available from commercial nuclear power stations to proceed with this task. The task was deferred to FY-93.

Table 1. (continued).

Task

- 8. Perform compressive strength measurements on samples obtained from two resin waste streams at Peach Bottom in October, 1989. Test the samples after 7 and 90 days of immersion in deionized and simulated sea water. Compare the compressive strengths of these samples with those of samples that were not immersed in the two leachants. Report the results of these measurements in a NUREG/CR draft, which must be completed by July 30, 1991. Include in a letter report an evaluation of the source and effects of ammonia released during solidification of these samples.
- Obtain core samples from three different 0 parts of the bulged Millstone liper to determine the homogeneity of the waste distribution in the cement solidification matrix. Examine the samples by gammaray spectroscopy. Also, perform SEM analysis to determine the microstructure of the resin-cement matrix. In addition, measure the immersion and compressive strength of these samples to determine the cause of liner bulging. Using the SEM method, also examine granular samples obtained from the unsuccessful solidification of Three Mile Island and Quad Cities wastes. Document the results of these measurements in a NUREG/CR report. which will include case histories of unsuccessful and expanded solidifications from other nuclear power stations.
 - Save a portion of all leachate samples for use in a separate study. This study will determine the chemical forms of radionuclide-chelating agent complexes leached from the solidified waste forms. Retain samples for FY-92.

Status

8

The compressive strengths of solidified LOMI resin waste forms from Peach Bottom were measured for immersed (90 days) and non-immersed samples. Immersed samples were placed in deionized and simulated sea water. Results indicate, within measurement errors, that all samples have similar compressive strengths.

9 The necessary safety analysis report and work procedure were completed for this task. However, the scope of this task was modified to include sampling and analysis of the liner gas head space. This change in scope necessitated rewitting the necessary safety analysis and work procedure. The actual work for this task has been deferred to FY-93.

 Samples of all leachates have been saved, and will be forwarded to Pacific Northwest Laboratory for further analysis.

Table 1. (continued).

Task	Status
 Inclusion of the second se	and when we want to a set of the

- U. ^a long-term leaching data to evaluate the applicability of the Clifton-Pommerstein Model in predicting the stability and leaching behavior of solidified waste forms. Leach data to be used include releases of calcium and other metals, boron, major gamma-ray emitters, ⁹⁰Sr, ⁵⁵Fe, ⁶³Ni, and ²⁴¹Pu. Summarize results in a letter report to be issued in FY-92.
- Initiate development of a program plan to evaluate effectiveness of non-destructive mechanical and chemical methods. This will be used to determine the stability of actual solidified radioactive wastes. The program will include identification of minor constituents in actual waste streams. obtained from nuclear power stations, short- and long-term leach data from actual plant-solidified waste and process control program samples, other test results as outlined in BTP tests, and results obtained from the Millstone liner examination. These mechanical and chemical waste form assessment methods. can provide the information required to determine waste form stability.

Relate wasts form assessment data to waste disposal site acceptance parameters, and, where applicable, identify and recommend new waste form acceptance tests for waste disposal sites.

Summarize results for this task in a letter report. Work for this task was deferred to FY-95.

 Work on this task can begin after completion and assessment of results from Yasiks 3, 6, 7, 8, and 9.

Table 1. (continued).

Task

13. Initiate assessment of full-scale solidified radioactive waste forms on the basis of tests performed on small-scale waste forms. Data from Brookhaven National Laboratory and the INEL indicate that leachability indexes do not follow the trends suggested in ANS 16.1. Review these data and investigate the practicality of leaching large waste forms. Test full-size waste forms to compare them to core samples removed from full-scale solidified liners. Cores from the Millstone liner may be used for this assessment.

Summarize results for this task in a letter report.

- 14 Complete NUREG/CR-5601 and journal articles on the effects of waste form size on leachability of radionuclides and chemicals from solidified decontamination waste by March 31, 1991.
- 15. Provide necessary technical expertise as requested and specified by the NRC Research Program Manager within budgetary constraints. Subjects may include decontamination processes; solidification of ion-exchange resins; leaching of radionuclides, metals and chelating agents; and analysis of waste streams for radionuclides, metals, and chemicals. In addition, assistance may be provided on an as-needed basis with respect to research topics on waste form stability, waste testing, characterization, and classification.

Status-

 Data collection for this task was predicated on using the solidified Millstone Liner. However, access to the solidified waste form in the liner is postponed to FY-93.

- NUREG/CR-5601³ was issued in June, 1991. In addition, two journal articles were submitted to Waste Management journal for publication after review by the NRC Program Manager.
- Technical expertise was provided as requested and specified by the NRC Program Manager within budgetary constraints.

Table 1. (continued).

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	Task					Status			
lssue mon	tily and	quarterly	progress	16.	Monthly	and quarterly	reports	Warre	

covering research activities performed in FY-91 by September 30, 1991 as a NUREG/CR report. Complete and publish the FY-90 annual report as a NUREG/CR report.

reports. Complete a draft annual report

17. Ensure proper disposal of all radioactive materials generated during the course of this research effort in approved disposal facilities. These may be at INEL or at commercial disposal sites. This disposal activity will include reseating and possibly overpacking the Millstone liner prior to its disposal. Monthly and quarterly reports were issued in accordance with the requirements.

17. Radioactive waste materials generated to date from this work were disposed of according to proper procedures. However, disposal of the bulged Millstone liner will incur expected additional costs.

2. PEACH BOTTOM LOMI DECONTAMINATION WASTE STREAM

From the Peach Bottom nuclear power station, LOMI decontamination waste stream and small scale waste forms were collected on March '1, 1991. The waste was analyzed for radionuclidic and chemical compositions at both Peach Bottom and INEL. Radiochemical analysis of the unsolidified waste stream was performed at INEL on two samples to determine the waste classification. The measured concentrations of ⁵⁵Fe, ⁶⁰Co, and ⁶³Ni in the waste stream indicate that it is Class A waste TRU content was determined to be <10 nCi/g. Table 2 summarizes the results. Analyses for stable metals in this waste stream were initiated in September, 1991.

Peach Bottom LOMI resins solidified in Portland cement were immersed in two different leachants for a period of 90 days, and the compressive strengths of these specimens were measured. The leachants were deionized and simulated sea water. After 30 days of immersion, small amounts of resin and cement debris (<1g) were observed in the leaching containers bottoms. All samples maintained their physical integrity and remained without cracks during the leaching experiment. Table 3 summarizes the compressive strength measurement results. The compressive strength of additional samples from this solidified waste stream will be assessed periodically throughout FY-92.

In addition, waste forms obtained from the process control program were cast into paper drinking cups and consequently were not true cylinders. Compressive strength measurements of this type of solid will be calculated by using the smaller of its two diameters. The rationale for selecting this calculational method is presented, by R. Neilson in Appendix A.

	Peach Bottom LOMI resin waste samples		
Radioisotope	Sample A waste activity (Ci/m ³)	Sample B waste activit (Ci/m ³)	
54Mn	2.86E - 02	3.03E - 02	
55Fe	9.38E-01	8.90E-01	
60Co	3.12E + 00	$3.17E \pm 00$	
63Ni	6.44E-02	3.90E - 02	
65Zn	3.97E - 01	4.06E - 01	
125Sb	1.22E - 02	1.13E-02	
137Cs	2.61E - 03	2.18E-03	
238pu	5.69E - 05	3.28E - 05	
239Pu	1.62E - 05	1.33E-05	
24) Pu	1.24E - 02	1.14E - 02	
241 Am	4.33E - 05	2.70E-05	
242Cm	7.81E - 06	5.03E - 06	
244Cm	1.02E - 04	7.39E-05	

Table 2. Radiochemical	analyses of Peach	Bottom LOMI resin waste.
------------------------	-------------------	--------------------------

Table 3. Compressive strength test measurements of solidified Peach Bottom LOMI waste stream resins.

Sample	Area (in. ²)	Yield load (lb)	Yield strength (psi)
1. Unleached	2.713	3370	1242
2. Unleached	2.695	2620	972
3. Unleached	2.690	3250	1208
Average $\pm \sigma$		3000 ± 402	1140 ± 147
Average 2.0			
1. DI water	2.722	3240	1190
2. DI water	2.541	3390	1334
Average $\pm \sigma$		3315 ± 106	1262 ± 101
Areidge a v			
1. Sea water	2.739	2720	992
	2.727	3810	1396
			1194 ± 285
 Sea water Sea water Average ± σ 	2.739	3810 3265 ± 770	1396

3. NINE MILE POINT FILTER SLUDGE WASTE STREAM

Samples of filter sludge waste stream, 30 solidified waste forms, and process control parameter samples were obtained from Nine Mace Point nuclear power station during an in-plant waste solidification evolution. Sample collection and full liner solidification were observed by INEL personnel. The radioisotopic composition of the

unsolidified waste stream was determined and is shown in Table 4. Stable metal analysis and full analysis of this waste stream were initiated in September, 1991. Appendix B describes the collection process and the analysis results obtained from the waste generator.

4. OYSTER CREEK EVAPORATOR CONCENTRATE WASTE

Oyster Creek Nuclear Power Station was contacted to determine the date of the evaporator concentrate solidification. Changes in Revision 1 of the RTP resulted in Chem Nuclear Systems.

Inc., delaying the solidification schedule at Oyster Creek. Confirmation that the waste is Class B must be received by INEL prior to obtaining this waste stream.

5. BULGED MILLSTONE LINER

Preparations for sampling the bulged Millstone liner were made. The safety review and necessary working procedures were prepared and approved. The hot cell space was also scheduled for July, 1991, together with the necessary veradorsupplied labor for core drilling.

The condition of this liner was rather abnormal, and there was concern about potential hydrogen gas generation and buildup in the liner. Consequently, the NRC requested the INEL to

Toble 4

evaluate the solidification and liner conditions with respect to headspace gas analysis prior to venting or coring the liner. Any headspace gas analyses, if deemed necessary, must be performed prior to venting or coring the liner, and necessitated rewriting the safety analysis and work procedures, and rescheduling hot cell space. The liner manufacturer information indicated that the upper liner manhole consisted of a standard 55 gallon 17H cover. These covers will sustain a

Table 4. Nine Mile Point filters	Different Particula	esterni i contratione	A Look Turk months	
			PALLINE LINE DOTTED.	
The second		the second s		

Isotope	INEL (A)	INEL (B)	Nine Mile Point
⁵⁴ Mn	$7.59\mathrm{E} = 3 \pm 6.5\mathrm{E} - 5$	6.74E-3±6.8E-5	$1.05E - 2 \pm 8.7E - 4$
58Co	$2.74\mathrm{E} = 3 \pm 2.8\mathrm{E} = 4$	$2.55E - 3 \pm 1.6E - 4$	3.53E-3±8.1E-4
⁶⁰ Co	$4.64\mathrm{E}-1\pm1.1\mathrm{E}-3$	4.55E-1±1.5E-3	$6.01E - 1 \pm 1.7E - 2$
125Sb	— N/D —	$3.66E - 3 \pm 3.8E - 5$	N/D
134Cs	$3.7\mathrm{E} - 3 \pm 3.0\mathrm{E} - 5$	3.20E - 3 ± 3.0E - 5	$5.56E - 3 \pm 6.8E - 4$
137Cs	$4.46E\!-\!1\pm\!1.1E\!-\!3$	3.94E-1±2.7E-3	$8.03E - 1 \pm 2.5E = 2$
154E0	N/D	2.15E-4±2.1E-5	N/D

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leak above an internal container pressure of 2 to 7 psi.⁶ In addition, the foam or rubber gaskets used in these covers allow hydrogen to diffuse. Moreover, private discussions with Dr. B. Siskind of Prookhaven National Laboratory regarding the radiation doses required to cause hydrogen gageneration from irradiation of organic ion exchanger material and water hydrolysis indicate that a radiation dose of greater than 10⁸ to 10⁹ RAD is required. The Millstone liner internal dose was ten orders of magnitude lower than this necessary dose (0.17 RAD over four years and ignoring decay corrections). Hydrogen gas buildup within the liner was ruled out. However, due to changes in priority, this task was posiponed to FY-93 by the NRC Program Manager.

6. CHEMICAL WASTE STREAM CHARACTERIZATION

A Program Plan, "Characterization of Chemical Wastes Present in Radioactive Waste Forms," was completed and will be implemented (see Appendix C).

7. LEACHING PLANS

The "NRC Leaching Program Plan" was completed. It includes sampling methods and dire,tions for short-term leaching in accordance with ANS 16.1. Comments from the NRC Project Manager were solicited and incorporated. A Long Term Leaching Plan was completed. Comments from the NRC Program Manager were solicited and incorporated. The plan will be published in FY-92.

8. NUREG/CR PUBLICATION

MUREG/CR-5601³ was published and distributed in July, 1991. It entailed learning and compressive strength data and a discussion on Brunswick and FitzPatrick solidified waste-form specimens.

9. JOURNAL ARTICLES PUBLICATION

Two journal articles were submitted to Waste Management journal for publication after review by the NRC Program Manager Section 9.1 summarizes the journal article by C. V. McIsaac and S. T. Croney,⁴ and Section 9.2 summarizes the article by C. V. McIsaac (to be published).

9.1 Effect of Waste Form Size on the Leachability of Radionuclides Contained in Cement-Solidified Evaporator Concentrates Generated at Nuclear Power Stations

Bone acid evaporator concentrate waste specimens solidified in type m mortar cement were obtained from pressurized water reactor, and specimens of sulfate evaporator concentrate waste solidified in Portland type I cement were obtained from a boiling water reactor station. The specimens varied in size. The physical dimensions, surface areas, and volumes of the collected waste-form specimens are summarized in Tables 5 and 6 for the boric acid and sulfate waste forms, respectively.

The study was undertaken to correlate radionuclide leachability from small- and full-scale waste forms. The samples were leached in deionized water at 25°C using a modified International Atomic Energy Agency (IAEA) leaching procedure. Release rates, effective diffusivities, and leachability indexes of ⁶⁰Co, ¹³⁷Cs, and combined ⁸⁰Sr and ⁹⁰Sr were determined using

Sample number	Waste loading (vol%)	Diameter × height (cm)	Surface area (cm ²)	Volume (cm ³)	Volume-to-surface crea ratio (crt;)
İ	76.5	5×10	1.78×10^2	1.70×10^2	0.95
2	76.5	5×10	1.78×10^{2}	$1.70 imes 10^2$	0.95
3	77.0	15×15	1.27×10^3	3.44×10^3	2.70
4	76.3	56×76	1.95×10^{d}	1.94×10^5	9.99
5	77.1	56×76	1.85×10^{4}	1.92×10^{8}	10.40

Table 5. Physical parameters of boric acid v aste forms.

Table 6. Physical parameters of sulfate waste forms.

Sample number	Waste loading (vol%)	Diameter × height (cm)	Surface area (cm ²)	Volume (cm ³)	Volume-to-surface area ratio (cm)
1	88.3	6×8	1.94×10^2	2.04×10^2	1.05
2	81.5	15×15	1×10^3	2.70×10^{3}	2.51
3	81.3	34×54	$7.49 imes 10^3$	4.80×10^4	6.42

diffusion release models given in ANS 16.1. The results indicate that predicted long-term releases of ⁶⁰Co from large-sca'e waste forms on the basis data from mail-scale waste forms are overestimated by as much as a factor of five after one year of leaching.

The solidified specimens were leached with a modified IAEA leaching procedure using deionized water at 25°C. The release rates and leachability indexes for ⁶⁰Co, combined ⁸⁹Sr and ⁹⁰Sr, and ¹³⁷Cs from both types of solidified wastes were calculated in accordance with the diffusion models of ANS 16.1. The calculated values are summarized in Tables 7 and 8 for boric acid and sulfate waste forms, respectively.

Leach tests were conducted at two commercial nuclear power stations on different sizes of specimens of boric acid and sulfate evaporator concentrate waste solidified in cement to correlate radionuclide leaching from small- and full-scale waste forms. Tests were performed using a modified IAEA leaching procedure using deionized water at 25°C. Release rates, effective diffusivities, and leach_bility indexes of ⁶⁰Co. ¹³⁷Cs, and combined ⁸⁰Sr and ⁶⁰Sr were determined for the different sizes of waste-form specimens using the diffusion release models of ANS 16.1

When the effective diffusivities determined for two 5 × 10-cm boric acid waste-form specimens were used to predict long-term releases of 60 Co from two 56 × 76-cm boric acid waste forms, predicted cumulative fractional releases (CFRs) of 60 Co from the larger specimens were factors of between 3.6 and 5.2 higher than actual CFRs after a leaching time of one year. Predicted CFRs of 137 Cs from the two 56 × 76-cm boric acid waste form specimens after one year of leaching were between 25% and 42% lower than actual CFRs of

		Average re		
Nuclide	Diameter », height (cm)	$(\mu \mathcal{C}i/cm^2\cdot s)$	(F/cm ² · s) ⁵	Leachability index
⁵⁰ Co	5 × 10	2.62×10^{-9}	1.96×10^{-10}	9.51 ± 0.07
wit a	5×10	$4.(8) \times +0^{30}$	2.99×10^{-10}	9.4 ± 0.1
	15 × 15	1.30×10^{-8}	5.11×10^{-11}	9.4 ± 0.1
	56 × 76	6.01×10^{-9}	3.94×10^{-13}	9.45 ± 0.07
	56 × 76	5.07×10^{-9}	3.33×10^{-13}	9.37 ± 0.07
89Sr. 90Sr	5×10		State State	b
31, 31	5 × 10	3.28×10^{-12}	2.89×10^{-9}	6.79 ± 0.06
	15 × 15	6.81×10^{-12}	3.10×10^{-10}	6.9 ± 0.2
	56×76	3.45×10^{-12}	2.81×10^{-12}	7.09 ± 0.09
	56 × 76	2.24×10^{-12}	1.87×10^{-17}	7.3 ± 0.1
I ^M Cs	5×10	2.56×10^{-9}	2.70×10^{-9}	631 ± 0.03
and a	5 × 10	2.65×10^{-0}	2.79×10^{-9}	6.26 ± 0.03
	15×15	4.00×10^{19}	2.07×10^{-10}	6.27 ± 0.02
	15 × 15 56 × 76	4.72×10^{10}	4.37×10^{-12}	6.14 ± 0.03
	56 × 76	5.33×10^{-9}	4.93×10^{-12}	$6.07~\pm~0.03$

Table 7. Release rates and leachability indexes for boric acid waste forms.

a. Fraction of initial inventory released per cm2 per second.

b. Leachates were not analyzed for radiostrontium.

and the second second	1	A	no for culture w	oucto friemic.
Table 8. Releas	e rates and leas	chability index	es foi suitaic v	cupic rolinis.

		Average re		
Nuclide	Diameter × height (cm)	(µCi/cm ^{2+s})	(F/cm ² · s) ^a	1 *achability index
⁶⁰ Co	6×8	7.32×10^{-10}	1.56×10^{-11}	11.3 ± 0.1
	15×15	1.23×10^{-9}	2.02×10^{-12}	10.4 ± 0.1
	34×54	-9.39×10^{-10}	8.72×10^{-14}	10.6 ± 0.1
⁸⁰ Sr, ⁹⁰ Sr	6×8	1.08×10^{-11}	2.54×10^{-10}	9.1 ± 0.3
1211 571	15 x 15	1.13×10^{-11}	$2.0^{\pm} \times 10^{-11}$	8.6 ± 0.2
	34×54	1.09×10^{-11}	$= 1.12 \times 16^{-12}$	8.6 ± 0.2
13 ⁷ Cs	6×8	2.41×10^{-9}	3.42×10.9	5.90 ± 0.04
	15 × 15	3.24×10^{-8}	-3.66×10^{-10}	5.75 ± 0.04
	34×54	3.52×10^{-8}	2.38×10^{-11}	$5.89 \div 0.06$

a. Fraction of mitial inventory released per cm² per second.

 137 Cs. Predicted CFRs of 89 Sr and 90 Sr from the two 56 × 76-cm boric acid waste forms ranged between 15% lower to 30% higher than actual CFRs of strontium.

In the case of the sulfate waste forms, whose surfaces cracked during leaching, the predicted CFR of ⁶⁰Co from the 34 × 54-cm waste-form specimen after one year of leaching, calculated using the effective diffusivity determined for the 6 × 8-cm specimen, was essentially identical to the CFR estimated using the data for the 34 × 54-cm waste form. The predicted CFR of ¹³⁷Cs from the 34 × 54-cm waste form after 20 days of leaching was equal to the CFR estimated using the leaching data for the 34 × 54-cm waste form.

The leaching results for the boric acid waste forms, which did not experience surface cracking during leaching, showed that when the effective diffusivity of ⁶⁰Co determined for a 90-day leach test was used to predict lor,g-term releases of ⁶⁰Co, the CFR of ⁶⁰Co was overpredicted by as much as a factor of two after a leaching time of one year.

The data indicate that leach-testing small-scale waste-form specimens will yield conservative predictions of long-term releases of radionuclides from full-scale waste forms. Because release rates and effective diffusivities of radionuclides generally decrease as cumulative leaching time increases, the use of a relatively short-term leach test such as that recommended by the NRC will ensure that predictions of long-term releases from full-scale waste forms are conservative.

9.2 Leachability of Chelated Decontamination Ion-Exchange Resins Solidified in Cement or Cement and Fly Ash at Nuclear Power Stations

Leach tests were conducted on six small-scale specimens of cement-solidified ion-exchange resin wastes. The ion-exchange resins had been used to process reagent solutions following chemical decontaminations of primary coolant systems at five commercial light water reactors. The decontaminations were performed using AP/ Citrox, Can-Decon, Dow NS-1, and LOMI processes. The ion-exchange resin wastes were loaded with radionuclides, transition metals, and organic chelating gents and were solidified in either Portland type I neat cement or in a mixture of Portland type I cement and fly ash. The Dow NS-1 and Can-Decon #1 resin wastes were solidified in Portland type I neat cement. The remaining waste resins were solidified in a mixture of Portland type I cement and fly ash.

Waste-form specimens were leached in deionized water at 23°C using the ANS 16.1 procedure. Release rates, effective diffusivities, and leachability indexes of radionuclides, chelating agents, and stable metals were determined using ANS 16.1 diffusion release models. Releases of radionuclides, chelating agents, and metals from waste forms that degraded during leaching were similar to releases from waste forms that maintained their physical integrity during leaching. The presence of chelating agents in the waste forms did not adversely affect the leachability of the waste forms. The actual dimensions of the six waste-form specimens that were leached, their volumes, surface areas, waste loadings, and their curing times are summarized in Table 9.

Release rates from the Can-Decon #1 waste form were generally lower than those from the Dow NS-1 waste form but were often higher than those from the Can-Decon #2 and LOMI waste forms. Release rates of ⁵⁵Fe, ⁶⁰Co, ⁶³Ni, ⁹⁰Sr, ^{13°}Cs, ²⁴¹Fu, citrir acid, oxalic acid, EDTA, DTPA, picolinic acid, and stable nickel are summarized in Tables 10 and 11. The release rates shown are simple arithmetic averages of release rates for individual leaching intervals. They are expressed in Table 10 as quantity released per second per cm² of sample external geometric surface area (µCi/cm²⁺s or µg/cm²⁺s). Release rates presented in Table 11 are expressed as fraction of initial inventory released per second per cm² of sample externa¹ geometric surface area (F/cm²⁺s).

Waste-form identification	Aging time (days)	Binder material	Waste loading (wt %)	Diameter × height (cm)	Surface area (cm ²)	Mass (g)	Volome (cm ³)	Volume-to- sufface atea ratio (cm)
AP/C(trox (mixed-bed)	710	Type I-P	60	4.4×9.5	164	205.2	147.8	0.90
AP/Citrox (cation)	802	Type 1-P	60	-4.4×10.2	173	215.6	137.7	
Can-Decon #1	684	Type I	61	4.8×6.2	115	125.9	93.5	0,81
Can-Decon #2		Type I-P		4.8×8.9	(69	207.7	158.4	0.94
Dow NS-1	654	Type 1	62	4.8×0.2	173	215.2	164.1	0.95
LOME		Type I-P	63	4.6×9.5	- 171	202.5	158.6	(1.93

Table 9. Physical parameters of ion-exchange resin waste forms.

The uncertainties shown in Tables 10 and 11 are standard deviations of the mean calculated at the one-sigma confidence level. The AP/Citrox waste forms crumbled during the first (2-hour) and second (5-hour) leaching intervals. Consequently, the average release rates did not include the first and second leaching intervals. These results are shown in the rows with headings of >2 h and >7 h, respectively.

The release rate of ⁶⁰Co from the AP/Citrox cation resin waste form increased sharply during the second leaching interval. However, release rates of the remaining radionuclides generally decreased significantly during the second leaching interval.

The average fractional release rates of ⁶⁰Co, ⁶³Ni, ⁹⁰Sr, citric acid, oxalic acid, and EDTA from the Dow NS-1 waste form are summarized in Table 10. They were significantly higher than corresponding release rates from the other waste forms. However, the fractional release rate of ¹³⁷Cs from the Dow NS-1 waste form was one to two orders of magnitude lower than those of ¹³⁷Cs from the other waste forms.

In all cases, maximum release rates occurred during the first two leaching intervals. Minimum release rates occurred during the last three leaching intervals. These last intervals were normally 14, 28, and 43 days long. In the case of the AP/ Citrox mixed-bed resin waste form, which disintegrated completely within 1 hour following immersion in leachant, release rates of radionuclides, chelating agents, and stable nickel decreased significantly after 7 hours of leaching.

CFRs of radionuclides, chelating agents, and stable nickel at the end of 90 days of leaching are summarized in Table 11. They were calculated from data obtained from unfiltered leachates and duey ranged from 0.002% from the Dow NS-4 waste form to 6.4% from the AP/Citrox cation cusin waste form for ⁵⁵Fe. The CFR results for the latter waste form were different from those of the other specimens. In particular, 92% of the total quantity of ⁵⁵Fe released from the AP/Citrox cation resin waste form occurred during the first 2 hours of leaching.

CFRs of ⁶⁰Co from the AP/Citrox cation resin. Can-Decon #1 and #2, and LOMI waste forms were similar to those of ⁵⁵Fe and ranged from about 0.1% to 0.2%. Cumulative releases of ⁶⁰Co from the Dow NS-1 and AP/Citrox mixed-bed resin waste forms were about 6% and 2%, respectively.

Nearly the entire inventory of 63Ni was released from the Dow NS-1 waste form. CFRs of 63Ni from the remaining waste forms ranged from clear one of the second of the

b. Concentration of radiomicfide or uteral in result waste was not determined

Quantity relears d per second per crin² of specimen external geometric surface area.

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Table 10. Average absolute release rates a

Journal Articles

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AP/Citros 16 \pm 0.8 \times 10 ⁻¹⁰ 3.6 \pm 1.9 \times 10 ⁻¹⁰ 3.5 \pm 1.9 \times 10 ⁻¹² 2.1 \pm 1.6 \times 10 ⁻¹⁶ 2 b 1.2 \pm 0.9 \times 10 ⁻¹⁰ 2.1 \pm 1.3 \times 10 ⁻¹⁰ 2.5 \pm 2.1 \times 10 ⁻¹² 2.4 \pm 2.0 \times 10 ⁻¹⁰ 57 h 3.2 \pm 0.2 \times 10 ⁻¹¹ 7.9 \pm 2.4 \times 10 ⁻¹¹ 1.8 \pm 1.8 \times 10 ⁻¹² 2.4 \pm 2.0 \times 10 ⁻¹⁰ 62 h 2.2 \pm 0.2 \times 10 ⁻¹¹ 7.9 \pm 2.4 \times 10 ⁻¹¹ 1.8 \pm 1.8 \times 10 ⁻¹² 2.4 \pm 1.4 \times 10 ⁻⁷ 7 h 2.0 \pm 1.0 \times 10 ⁻¹¹ 1.7 \pm 1.2 \times 10 ⁻¹¹ 3.1 \pm 1.3 \times 10 ⁻¹³ 3.3 \pm 1.3 \times 10 ⁻¹⁶ 7 h 2.0 \pm 1.0 \times 10 ⁻¹¹ 5.9 \pm 2.6 \times 10 ⁻¹² 2.0 \pm 1.0 \times 10 ⁻¹³ 2.4 \pm 1.4 \times 10 ⁻¹⁷ Can-Decon #1 3.3 \pm 2.2 \times 10 ⁻¹² 5.9 \pm 2.3 \times 10 ⁻¹⁶ 6.9 \pm 3.8 \times 10 ⁻⁹ 5.2 \pm 4.0 \times 10 ⁻⁹ LOMI 8.2 \pm 7.7 \times 10 ⁻¹² 5.1 \pm 1.4 \times 10 ⁻¹¹ 6.5 \pm 5.2 \times 10 ⁻¹² 7.7 \pm 2.8 \times 10 ⁻¹⁶ Dow NS -1 6.3 \pm 5.6 \times 10 ⁻¹² 5.1 \pm 1.4 \times 10 ⁻¹¹ 6.5 \pm 5.2 \times 10 ⁻¹² 7.7 \pm 2.8 \times 10 ⁻¹⁰ LOMI 8.2 \pm 7.7 \times 10 ⁻¹² 5.1 \pm 1.4 \times 10 ⁻¹¹ 2.9 \pm 2.0 \times 10 ⁻¹⁰ 7.7 \pm 2.8 \times 10 ⁻¹⁰ 7.7 \pm 2.8 \times 10 ⁻¹⁰ Can-Decon #1 </th <th></th> <th>STE</th> <th>60Co</th> <th>63N)</th> <th>90Sr</th>		STE	60Co	63N)	90Sr	
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$\begin{array}{c ccccccccccccccccccccccccccccccccccc$		$1.6 \pm 0.8 \times 10^{-10}$	$3.6 \pm 1.9 \times 10^{10}$	$3.5 \pm 1.9 \times 10^{-12}$		
a^{7} h $3.2 \pm 0.2 \times 10^{-11}$ $7.9 \pm 2.4 \times 10^{-11}$ $3.8 \pm 1.8 \times 10^{-10}$ $4.4 \pm 3.5 \times 10^{-11}$ cation $9.2 \pm 9.2 \times 10^{-9}$ $1.6 \pm 1.0 \times 10^{-11}$ $3.8 \pm 1.8 \times 10^{-10}$ $1.4 \pm 1.4 \times 10^{-11}$ s^{7} h $2.2 \pm 0.7 \times 10^{-11}$ $1.7 \pm 1.2 \times 10^{-11}$ $3.1 \pm 1.3 \times 10^{-10}$ $3.4 \pm 1.3 \times 10^{-11}$ can -Decon #1 $3.3 \pm 2.2 \times 10^{-12}$ $5.9 \pm 2.3 \times 10^{-12}$ $8.1 \pm 3.4 \times 10^{-11}$ $2.6 \pm 1.2 \times 10^{-9}$ can -Decon #2 -5 5.6 ± 10^{-13} $5.6 \pm 2.3 \times 10^{-12}$ $8.1 \pm 3.4 \times 10^{-11}$ $2.6 \pm 1.2 \times 10^{-9}$ can -Decon #2 -5 5.6 ± 10^{-13} $5.6 \pm 2.3 \times 10^{-12}$ $8.1 \pm 3.4 \times 10^{-11}$ $2.6 \pm 1.2 \times 10^{-9}$ can -Decon #2 -5 $5.6 \pm 2.3 \times 10^{-10}$ $6.9 \pm 3.8 \times 10^{-9}$ $5.2 \pm 4.0 \times 10^{-9}$ $c5.0 \times 10^{-9}$ can -Decon #2 5.6 ± 10^{-13} $5.6 \pm 2.3 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-19}$ $2.9 \pm 1.9 \times 10^{-9}$ $c5.0 \times 10^{-9}$ can -Decon #1 $5.6 \pm 0.9 \times 10^{-8}$ $ca^{-9} \times 10^{-11}$ $2.9 \pm 1.0 \times 10^{-10}$ $ca^{-1} \times 1.5 \times 10^{-11}$ 1.4×10^{-11} $2.9 \pm 1.9 \times 10^{-10}$ $1.5 \pm 1.0 \times 10^{-9}$ $ca^{-1} \times 1.5 \times 10^{-11}$ $1.5 \pm 1.0 \times 10^{-9}$			2.1 ± 1.3 × 10 ⁻¹⁰	2.5 ± 2.1 × 10 ⁻¹²		
cation $9.2 \pm 9.2 \times 10^{-9}$ $1.6 \pm 1.0 \times 10^{-11}$ $1.8 \pm 1.8 \times 10^{-10}$ $1.4 \pm 1.4 \times 10^{-7}$ >2 h $2.2 \pm 0.7 \times 10^{-11}$ $1.7 \pm 1.2 \times 10^{-11}$ $3.1 \pm 1.3 \times 10^{-16}$ $3.3 \pm 1.3 \times 10^{-17}$ S7 h $2.0 \pm 1.0 \times 10^{-11}$ $5.9 \pm 2.6 \times 10^{+12}$ $2.0 \pm 1.0 \times 10^{+13}$ $2.4 \pm 1.4 \times 10^{-17}$ Can-Decon #1 $3.3 \pm 2.2 \times 10^{+12}$ $5.9 \pm 2.3 \times 10^{+12}$ $8.1 \pm 3.4 \times 10^{+17}$ $2.6 \pm 1.2 \times 10^{-9}$ Can-Decon #2 6 $3.8 \pm 1.3 \times 10^{+12}$ 6^{-5} $5.2 \pm 4.0 \times 10^{-10}$ Dow NS-1 $6.3 \pm 5.6 \times 10^{-13}$ $5.6 \pm 2.3 \times 10^{+10}$ $6.9 \pm 3.8 \times 10^{-9}$ $5.2 \pm 4.0 \times 10^{-10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-11}$ $6.5 \pm 5.2 \times 10^{-12}$ $7.7 \pm 2.8 \times 10^{-10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-19}$ $7.7 \pm 2.8 \times 10^{-10}$ MP/Citrox mixed-bed $1.8 \pm 0.9 \times 10^{-8}$ $e^{9.2} \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $1.0 \pm 0.7 \times 10^{+10}$ $5.1 \pm 1.0 \times 10^{-8}$ $e^{-3.7} \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $1.0 \pm 0.7 \times 10^{+10}$ $1.0 \pm 0.7 \times 10^{+10}$ $5.2 \pm 1.0 \times 10^{-8}$ $e^{-3.7} \times 10^{-11}$ <t< td=""><td></td><td></td><td>$7.9 \pm 2.4 \times 10^{11}$</td><td>$3.8 \pm 1.8 \times 10^{-13}$</td><td></td></t<>			$7.9 \pm 2.4 \times 10^{11}$	$3.8 \pm 1.8 \times 10^{-13}$		
2 h $2.2 \pm 0.7 \times 10^{-11}$ $1.7 \pm 1.2 \times 10^{-11}$ $3.1 \pm 1.3 \times 10^{-13}$ $3.3 \pm 1.3 \times 10^{-16}$ 57 h $2.0 \pm 1.0 \times 10^{-11}$ $5.9 \pm 2.6 \times 10^{-12}$ $2.0 \pm 1.0 \times 10^{-11}$ $2.4 \pm 1.4 \times 10^{-11}$ Can-Decom #1 $2.5 \pm 2.2 \times 10^{-12}$ $5.9 \pm 2.3 \times 10^{+12}$ $8.1 \pm 3.4 \times 10^{+11}$ $2.6 \pm 1.2 \times 10^{-9}$ Can-Decom #2 -5 $3.8 \pm 1.3 \times 10^{+16}$ -5^{-16} $-2.5 \oplus .5^{-2} \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{+11}$ $6.5 \pm 5.2 \times 10^{+12}$ $7.7 \pm 2.8 \times 10^{+10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{+11}$ $6.5 \pm 5.2 \times 10^{+12}$ $7.7 \pm 2.8 \times 10^{+10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{+11}$ $6.5 \pm 5.2 \times 10^{+10}$ $7.7 \pm 2.8 \times 10^{+10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{+11}$ $6.5 \pm 5.2 \times 10^{+10}$ $7.7 \pm 2.8 \times 10^{+10}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{+10}$ $2.9 \pm 2.0 \times 10^{+10}$ $7.7 \pm 2.8 \times 10^{+10}$ LOMI $8.2 \pm 7.7 \times 10^{-13}$ $5.6 \pm 2.0 \times 10^{-9}$ -5.5×10^{-12} $1.0 \pm 0.7 \times 10^{+10}$ S7 h $5.0 \pm 2.0 \times 10^{-9}$ $<5.6 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{+10}$ $-5. \times 10^{+10}$ Can-Decon #1			1.6 ± 1.0 × 10 ⁻¹¹	$1.8 \pm 1.8 \times 10^{-10}$		
57 h 2.0 ± 1.0 × 10 ⁻¹¹ 5.9 ± 2.6 × 10 ⁻¹² 2.0 ± 1.0 × 10 ⁻¹³ 2.4 ± 1.4 × 10 ⁻¹¹ Can-Decon #1 3.3 ± 2.2 × 10 ⁻¹² 5.9 ± 2.3 × 10 ⁻¹² 8.1 ± 3.4 × 10 ⁻¹¹ 2.6 ± 1.2 × 10 ⁻⁹ Can-Decon #2 -8 3.8 ± 1.3 × 10 ⁻¹² -9^{5} -2^{5			$1.7 \pm 1.2 \times 10^{-11}$	$3.1 \pm 1.3 \times 10^{-13}$		
Can-Decon #1 $3.3 \pm 2.2 \times 10^{-12}$ $5.9 \pm 2.3 \times 10^{-12}$ $8.1 \pm 3.4 \times 10^{-11}$ $2.6 \pm 1.2 \times 10^{-9}$ Can-Decon #2 8 $3.8 \pm 1.3 \times 10^{-16}$ 9^{-9} $c5.0 \times 10^{-9}$ Dow NS-1 $6.3 \pm 5.6 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-16}$ $6.9 \pm 3.8 \times 10^{-9}$ $5.2 \pm 4.0 \times 10^{-9}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-16}$ $6.5 \pm 5.2 \times 10^{-12}$ $7.7 \pm 2.8 \times 10^{-10}$ MP/Citrox mixed-bed $1.8 \pm 0.9 \times 10^{-8}$ $s^{-9.2 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $27 h$ $5.0 \pm 2.0 \times 10^{-9}$ $s^{-1.6 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $27 h$ $5.0 \pm 2.0 \times 10^{-9}$ $s^{-1.6 \times 10^{-11}$ $2.9 \pm 1.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $52 h$ $1.5 \pm 1.0 \times 10^{-8}$ $s^{-3.7 \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-10}$ $52 h$ $1.5 \pm 0.8 \times 10^{-9}$ $s^{-3.7 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-10}$ $52 h$ $5.0 \pm 2.0 \times 10^{-10}$ $s^{-7.7 \times 10^{-10}$ $s^{-7.7 \times 10^{-10}}$ $s^{-7.7 \times 10^{-10}}$ $s^{-7.7 \times 10^{-10}}$ $s^{-7.7 \times 10^{-10}}$ $s^{-7.7 \times 10^{-$				$2.0 \pm 1.0 \times 10^{-13}$		
Can-Decon #2 $-b^6$ $3.8 \pm 1.3 \times 10^{-14}$ $-b^6$ $c5.0 \times 10^{-10}$ Dow NS-1 $6.3 \pm 5.6 \times 10^{-15}$ $5.6 \pm 2.3 \times 10^{-16}$ $6.9 \pm 3.8 \times 10^{-9}$ $5.2 \pm 4.0 \times 10^{-9}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-11}$ $6.5 \pm 5.2 \times 10^{-12}$ $7.7 \pm 2.8 \times 10^{-10}$ MP/Citros 13^{-16} $2^{-241}P_0$ Crine acid Oxalic acid AP/Citros $c3.7 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $23 h$ $1.5 \pm 1.0 \times 10^{-8}$ $c9.2 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $24 h$ $1.5 \pm 1.0 \times 10^{-8}$ $c3.7 \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-40}$ $cation 2.3 \pm 1.0 \times 10^{-9} c4.5 \times 10^{-11} 2.5 \pm 1.0 \times 10^{-11} 3.9 \pm 1.5 \times 10^{-11} cation 2.3 \pm 1.0 \times 10^{-9} c4.5 \times 10^{-11} c.5 + 10^{-11} 3.9 \pm 1.5 \times 10^{-11} cation 2.5 \pm 1.0 \times 10^{-9} c4.5 \times 10^{-11} c.5 + 10^{-10} c.5 + 10^{-10} cation 2.6 \pm 4.2 \times 10^{-10} c4.5 \times 10^{-11} c.5 + 10^{-10} c4.5 \times 10^{-10} cation 2.5 \pm 0.6 \times 10^{-9} c4.5$			$3.9 \pm 2.3 \times 10^{-12}$	8.1 ± 3.4 × 10 ⁻¹¹	$2.6 \pm 1.2 \times 10^{-9}$	
Dow NS-1 $6.3 \pm 5.6 \times 10^{-13}$ $5.6 \pm 2.3 \times 10^{-10}$ $6.9 \pm 3.8 \times 10^{-9}$ $5.2 \pm 4.0 \times 10^{-9}$ LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-11}$ $6.5 \pm 5.2 \times 10^{-12}$ $7.7 \pm 2.8 \times 10^{-10}$ MPCitrox Image: the transmitted transmitter					$<5.0 \times 10^{-10}$	
LOMI $8.2 \pm 7.7 \times 10^{-12}$ $5.1 \pm 1.4 \times 10^{-17}$ $6.5 \pm 5.2 \times 10^{-12}$ $7.7 \pm 2.8 \times 10^{-10}$ $1^{33}Cs$ $2^{241}Pu$ Citric acid Oxalic acid. AP/Citros mixed-bed $1.8 \pm 0.9 \times 10^{-8}$ $eg_{2} \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-40}$ $57 h$ $5.0 \pm 2.0 \times 10^{-9}$ $et_{1.6} \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-40}$ $57 h$ $5.0 \pm 2.0 \times 10^{-9}$ $et_{1.6} \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-40}$ $cation 2.3 \pm 1.0 \times 10^{-9}$ $et_{3.0} \times 0^{-9}$ et_{-5} et_{-5} $cation 2.3 \pm 1.0 \times 10^{-9}$ $et_{3.0} \times 0^{-9}$ et_{-5} et_{-5} $cation 2.3 \pm 1.0 \times 10^{-9}$ $et_{3.0} \times 10^{-11}$ et_{-5} et_{-5} $cation 2.3 \pm 0.6 \times 10^{-9}$ $et_{3.0} \times 10^{-11}$ et_{-5} et_{-5} $cation 2.15 \pm 0.8 \times 10^{-9}$ $et_{3.0} \times 10^{-11}$ et_{-5} et_{-5} $cation 2.15 \pm 0.6 \times 10^{-9}$ $et_{4.6} \times 10^{-11}$ et_{-5} et_{-5} $cation 4.2 \pm 1.5 \pm 0.6 \times 10^{-9}$ $et_{4.6} \times 10^{-11}$ et_{-5} et_{-5} $cation 4.2 \pm 1.0 \times 10^{-8}$ $et_{6.5} \times 10^{-12}$ $1.3 \pm 0.5 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ Can-Decon #2 $1.5 \pm 0.6 \times 10^{-9}$ $et_{4.6} \times 10^{-11}$ et_{-5}				$6.9 \pm 3.8 \times 10^{-9}$	$5.2 \pm 4.0 \times 10^{-9}$	
AP/Citrox $x = 0.9 \times 10^{-8}$ $x = 9.2 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-10}$ >2 h $1.5 \pm 1.0 \times 10^{-8}$ $< 3.7 \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-10}$ >7 h $5.0 \pm 2.0 \times 10^{-9}$ $< 1.6 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $3.9 \pm 1.5 \times 10^{-11}$ cation $2.3 \pm 1.0 \times 10^{-9}$ $<3.0 \times 10^{-9}$ 6×10^{-11} 6×10^{-11} >2 h $1.5 \pm 0.8 \times 10^{-9}$ $<3.0 \times 10^{-9}$ 6×10^{-11} 6×10^{-11} >2 h $1.5 \pm 0.8 \times 10^{-9}$ $<3.7 \times 10^{-11}$ 6×10^{-11} 6×10^{-11} >2 h $1.5 \pm 0.6 \times 10^{-9}$ $<3.7 \times 10^{-11}$ 6×10^{-10} 6×10^{-10} Can-Decon #1 $2.2 \pm 1.0 \times 10^{-8}$ $<6.5 \times 10^{-12}$ $1.3 \pm 0.5 \times 10^{-10}$ $5.6 \pm 3.1 \times 10^{-10}$ Can-Decon #2 $1.5 \pm 0.6 \times 10^{-9}$ $<1.4 \times 10^{-10}$ $3.3 \pm 2.4 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ LOMI $5.6 \pm 1.9 \times 10^{-9}$ $<4.6 \times 10^{-11}$ 1.4×10^{-10} 6×10^{-10} LOMI $5.6 \pm 1.9 \times 10^{-9}$ 6×10^{-11} 6×10^{-9} 6×10^{-9} $2.1 \times -0.5 \times 10^{-9}$	LOMI			$-6.5~\pm~5.2~\times~10^{-12}$	$7.7 \pm 2.8 \times 10^{-10}$	
mixed bed $1.8 \pm 0.9 \times 10^8$ $\approx 9.2 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-30}$ >2 h $1.5 \pm 1.0 \times 10^8$ $<3.7 \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-10}$ >7 h $5.0 \pm 2.0 \times 10^9$ $<1.6 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $3.9 \pm 1.5 \times 10^{-10}$ cation $2.3 \pm 1.0 \times 10^9$ $<3.0 \times 10^9$ $<3.0 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $3.9 \pm 1.5 \times 10^{-10}$ >2 h $1.5 \pm 0.8 \times 10^9$ $<3.0 \times 10^{-10}$ $ \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ >2 h $1.5 \pm 0.8 \times 10^9$ $<3.7 \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ Can-Decon #1 $2.2 \pm 1.0 \times 10^{-8}$ $<1.0 \times 10^{-11}$ $ \times 10^{-10}$ $ \times 10^{-10}$ Can-Decon #2 $1.5 \pm 0.6 \times 10^9$ $<1.6 \times 10^{-10}$ $3.3 \pm 2.4 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ Dow NS-1 $<2.5 \times 10^{-10}$ $<3.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^8$ $4.3 \pm 2.3 \times 10^{-10}$ $ \times 10^{-10}$ Cation $ \times 10^{-10}$ Cation $ \times 10^{-10}$ $ \times$		137Cs	240pg	Citric ació	Oxalic acid	
mixed bed $1.8 \pm 0.9 \times 10^8$ $\approx 9.2 \times 10^{-11}$ $2.9 \pm 2.0 \times 10^{-10}$ $2.9 \pm 1.9 \times 10^{-30}$ >2 h $1.5 \pm 1.0 \times 10^8$ $<3.7 \times 10^{-11}$ $9.6 \pm 7.1 \times 10^{-11}$ $1.0 \pm 0.7 \times 10^{-10}$ >7 h $5.0 \pm 2.0 \times 10^9$ $<1.6 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $3.9 \pm 1.5 \times 10^{-10}$ cation $2.3 \pm 1.0 \times 10^9$ $<3.0 \times 10^9$ $<3.0 \times 10^{-11}$ $2.5 \pm 1.0 \times 10^{-11}$ $3.9 \pm 1.5 \times 10^{-10}$ >2 h $1.5 \pm 0.8 \times 10^9$ $<3.0 \times 10^{-10}$ $ \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ >2 h $1.5 \pm 0.8 \times 10^9$ $<3.7 \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ $ \times 10^{-11}$ Can-Decon #1 $2.2 \pm 1.0 \times 10^{-8}$ $<1.0 \times 10^{-11}$ $ \times 10^{-10}$ $ \times 10^{-10}$ Can-Decon #2 $1.5 \pm 0.6 \times 10^9$ $<1.6 \times 10^{-10}$ $3.3 \pm 2.4 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ Dow NS-1 $<2.5 \times 10^{-10}$ $<3.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^8$ $4.3 \pm 2.3 \times 10^{-10}$ $ \times 10^{-10}$ Cation $ \times 10^{-10}$ Cation $ \times 10^{-10}$ $ \times$	And you and the second second second second second					
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$\begin{array}{c ccccccccccccccccccccccccccccccccccc$						
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$\begin{array}{c ccccccccccccccccccccccccccccccccccc$						
$\times 2$ H $1.3 \pm 0.5 \pm 10^{-10}$ $< 10^{-10}$ $< 10^{-11}$ -5^{-1} -3^{-3} S7 h $8.6 \pm 4.2 \times 10^{-10}$ $< 10^{-10}$ $< 6.5 \times 10^{-12}$ $1.3 \pm 0.5 \times 10^{-10}$ $5.6 \pm 3.1 \times 10^{-10}$ Can-Decon #1 $2.2 \pm 1.0 \times 10^{-8}$ $< 1.4 \times 10^{-10}$ $3.3 \pm 2.4 \times 10^{-10}$ $5.6 \pm 3.1 \times 10^{-10}$ Dow NS-1 $< 2.0 \times 10^{-10}$ $< 3.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^{-8}$ $4.3 \pm 2.3 \times 10^{-8}$ LOMI $5.6 \pm 1.9 \times 10^{-9}$ $< 4.6 \times 10^{-11}$ -2^{-1} -2^{-1} -2^{-1} AP/Citros mixed-bed -5 -5^{-1} -5^{-1} -4^{-10} -5^{-1} -5^{-1} AP/Citros -5^{-1}	cation					
$\times 7$ h 5.0 $\pm 4.4 \times 10^{-8}$ $< 6.5 \times 10^{-12}$ $1.3 \pm 0.5 \times 10^{-10}$ $5.6 \pm 3.1 \times 10^{-10}$ Can-Decon #2 $1.5 \pm 0.6 \times 10^{-9}$ $< 1.4 \times 10^{-10}$ $3.3 \pm 2.4 \times 10^{-10}$ $5.6 \pm 3.1 \times 10^{-10}$ Dow NS-1 $< 2.0 \times 10^{-10}$ $< 3.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^{-8}$ $4.3 \pm 2.3 \times 10^{-9}$ LOMJ $5.6 \pm 1.9 \times 10^{-9}$ $< 4.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^{-8}$ $4.3 \pm 2.3 \times 10^{-9}$ EDTA DTPA Picolinic acid Nickel AP/Citrox $= 5$ $= 5$ $= 5$ $= 4.7 \times 10^{-10}$ ~ 2 h $= 3$ $= 5$ $= 5$ $< 2.2 \times 10^{-9}$ ~ 7 h $= 3$ $= 5$ $= 5$ $< 2.2 \times 10^{-9}$ ~ 7 h $= 3$ $= 5$ $= 5$ $< 4.7 \times 10^{-10}$ ~ 2 h $= 3$ $= 5$ $= 5$ $< 4.7 \times 10^{-10}$ ~ 7 h $= 3$ $= 5$ $= 5$ $< 4.7 \times 10^{-10}$ $< 4.7 \times 10^{-10}$ ~ 7 h $= 3$ $= 5$ $= 5$ $= 5$ $< 1.0 \times 10^{-9}$ $< 1.0 \times 10^{-9}$ ~ 1 h $= 6$ $= 5$ $= 5$ $= $						
Car-Decon #2 1.5 \pm 0.6 \times 10 $3.3 \pm 2.4 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ Car-Decon #2 0.6×10^{-9} $<3.6 \times 10^{-11}$ $3.3 \pm 2.4 \times 10^{-10}$ $1.8 \pm 1.0 \times 10^{-10}$ Dow NS-1 2.0×10^{-10} $<3.6 \times 10^{-11}$ $1.1 \pm 0.6 \times 10^{-8}$ $4.3 \pm 2.3 \times 10^{-8}$ LOMJ $5.6 \pm 1.9 \times 10^{-9}$ $<4.6 \times 10^{-11}$ $= 6$ -6 -6 AP/Citrox $= 6$ -5 $= 6$ -6 1.0×10^{-9} -6 AP/Citrox $= 5$ -5 -5 -5 -6 -6 -6 AP/Citrox $= 5$ -5 -5 -6 -6 -6 -6 AP/Citrox $= 5$ -5 -6 -5 -6 -6 -6 -1.0×10^{-9} -6 $>2 h$ -5 -5 -5 -6 -1.0×10^{-9} -10^{-10} -5 -1.0×10^{-9} -1.0×10^{-9} $>7 h$ -5 -5 -5 -5 -6 $-1.7 \pm 0.9 \times 10^{-10}$ -6 <th< td=""><td>>7 h</td><td></td><td></td><td></td><td></td></th<>	>7 h					
Carlo Decon #2 1.8 \pm 0.6 \pm 10 ⁻¹⁰ <3.6 \pm 10 ⁻¹¹ 1.1 \pm 0.6 \pm 10 ⁻⁸ 4.3 \pm 2.3 \pm 10 ⁻⁹ LOMJ 5.6 \pm 1.9 \times 10 ⁻⁹ <3.6 \pm 10 ⁻¹¹ 1.1 \pm 0.6 \times 10 ⁻⁸ 4.3 \pm 2.3 \times 10 ⁻⁹ EDTA DTPA Picolinic acid Nickel AP/Citrox <2.2 \times 10 ⁻⁹ x2 h <2.2 \times 10 ⁻⁹ >2 h <2.2 \times 10 ⁻⁹ ×7 h <4.7 \times 10 ⁻¹⁰ cation 9.9 \pm 5.7 \times 10 ⁻¹⁰ ×7 h 9.9 \pm 5.7 \times 10 ⁻¹⁰ ×7 h 9.9 \pm 5.7 \times 10 ⁻¹⁰ ×7 h 9.9 \pm 5.7 \times 10 ⁻¹⁰ ×7 h 9.9 \pm 5.7 \times 10 ⁻¹⁰ ×7 h Can-Decon #1 2.0 \pm 0.6 \times 10 ⁻⁹ Dow NS-1 1.7 \pm 0.8 \times 10 ⁻⁸ 3.1 \pm 1.2 \times 10 ⁻⁹ <	Can-Decon #1					
LOMJ $5.6 \pm 1.9 \times 10^{-9}$ $<4.6 \times 10^{-11}$ $=^{-5}$ $=^{-5}$ EDTA DTPA Picolinic acid Nickel AP/Citrox $=^{-5}$ $=^{-5}$ $<2.2 \times 10^{-6}$ $>2 h$ $=^{-5}$ $=^{-5}$ $<2.2 \times 10^{-6}$ $>2 h$ $=^{-5}$ $=^{-5}$ $<2.2 \times 10^{-6}$ $>2 h$ $=^{-5}$ $=^{-5}$ $<2.2 \times 10^{-6}$ $>7 h$ $=^{-5}$ $=^{-5}$ $<2.2 \times 10^{-6}$ cation $=^{-5}$ $=^{-5}$ $=2^{-5}$ $>2 l$ $=^{-5}$ $=^{-5}$ $1.2 \pm 1.2 \times 10^{-9}$ $>2 l$ $=^{-5}$ $=^{-5}$ $1.7 \pm 0.9 \times 10^{-10}$ $>7 h$ $=^{-5}$ $=^{-5}$ $1.7 \pm 0.9 \times 10^{-10}$ $>7 h$ $=^{-5}$ $=^{-5}$ $1.7 \pm 0.9 \times 10^{-10}$ $>7 h$ $=^{-5}$ $=^{-5}$ $=^{-5}$ $=^{-5}$ $>2 l$ $= 0.6 \times 10^{-6}$ $=^{-5}$ $=^{-5}$ $=^{-6}$ $>2 l$ $=0.6 \times 10^{-6}$ $=^{-5}$ $=^{-5}$ $=^{-5}$ $>2 l$ $=0.6 \times 10^{-6}$ $=^{-5}$ $=^{-5}$ <td>Can-Decon #2</td> <td></td> <td></td> <td></td> <td></td>	Can-Decon #2					
EDTA DTPA Picolinic acid Nickel AP/Citrox -3 -4 -5 -2 -4 -2 -10^{-9} -2.2×10^{-9} >2 h -3 -4 -5 -5 -10^{-1} -2.2×10^{-9} >7 h -5 -5 -5 -10^{-1} -2.2×10^{-9} cation -5 -5 -5 -10^{-1} -2.2×10^{-9} >2 l -5 -5 -5^{-1} -2.2×10^{-9} $-4.4 \pm 2.2 \times 10^{-9}$ >3 h -5^{-1} -5^{-1} -5^{-1} -5^{-1} -5^{-1} Can-Decoh #1 $2.0 \pm 0.6 \times 10^{-9}$ -5^{-1} -5^{-1} -5^{-1} Dow NS-1 $1.7 \pm 0.8 \times 10^{-8}$ $3.1 \pm 1.2 \times 10^{-9}$ -5^{-1} $4.0 \pm 2.1 \times 10^{-9}$	Dow NS-1				$4.3 \pm 2.3 \times 10^{10}$	
AP/Citrox $= 2^{0}$ $= 1^{0}$ $= 2^{0}$ $= 1^{0}$ $= 2^{0}$	LOMI	$5.6 \pm 1.9 \times 10^{-9}$	<4.6 × 10 ⁻¹⁴			
mixed-bed -3 -4°		EDTA	DTPA	Picolinic acid	Nickel	
mixed-bed -3 -4°						
>2 h -3 -4 -3 -4 -3 -4					10 1 1 10 10	
$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} $1.2 \pm 1.2 \times 10^{-8}$ $9.9 \pm 5.7 \times 16^{-11}$ >7 h a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} $4.4 \pm 2.2 \times 10^{-11}$ Can-Decon #1 $2.0 \pm 0.6 \times 10^{-9}$ a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} Can-Decon #2 $5.4 \pm 1.8 \times 10^{-10}$ a^{-5} a^{-5} a^{-5} a^{-5} a^{-5} Dow NS-1 $1.7 \pm 0.8 \times 10^{-8}$ $3.1 \pm 1.2 \times 10^{-9}$ a^{-5} $4.0 \pm 2.1 \times 10^{-9}$						
>2 1. $\{c}$ $\{c}$ $\{c}$ $\{c}$ $\{c}$ $9.9 \pm 5.7 \times 10^{-11}$ >7 h $\{c}$ $\{c}$ $\{c}$ $\{c}$ $\{c}$ $4.4 \pm 2.2 \times 10^{-11}$ Can-Decon #1 $2.0 \pm 0.6 \times 10^{-9}$ $\{c}$ $\{c}$ $\{c}$ $1.7 \pm 0.9 \times 10^{-19}$ Can-Decon #2 $5.4 \pm 1.8 \times 10^{-10}$ $\{c}$ $\{c}$ $\{c}$ $\{c}$ Dow NS-1 $1.7 \pm 0.8 \times 10^{-8}$ $3.1 \pm 1.2 \times 10^{-9}$ $\{c}$ $4.0 \pm 2.1 \times 10^{-9}$						
$\begin{array}{cccccccccccccccccccccccccccccccccccc$						
Can-Decon #1 2.0 \pm 0.6 \times 10.9 $-s$ $-s$ $-s$ 1.7 \pm 0.9 \times 10.9 10^{-10} Can-Decon #2 5.4 \pm 1.8 10^{-10} $-s$ $-s$ $-s$ $-b$ Dow NS-1 1.7 \pm 0.8< <td>10^{-8} 3.1 \pm 1.2 40^{-9} $-s$ 4.0 \pm 2.1 10^{-9}</td> <td></td> <td></td> <td></td> <td></td> <td></td>	10^{-8} 3.1 \pm 1.2 40^{-9} $-s$ 4.0 \pm 2.1 10^{-9}					
Can-Decon #2 5.4 \pm 1.8 \times 10 ⁻¹⁰ - ^c - ^b Dow NS-4 1.7 \pm 0.8 \times 10 ⁻⁸ 3.1 \pm 1.2 \times 10 ⁻⁹ - ^c 4.0 \pm 2.1 \times 10 ⁻⁹						
Dow NS-1 $1.7 \pm 0.8 \times 10^{-8}$ $3.1 \pm 1.2 \times 10^{-9}$ $-^{-2}$ $4.0 \pm 2.1 \times 10^{-9}$	Can-Decon #1					
	Can-Decoh #2 -					
LOMI -2° $4.8 \pm 1.5 \times 10^{-0}$ $2.6 \pm 1.8 \times 10^{-0}$	Dow NS-1					
	LOMI			$4.8 \pm 1.5 \times 10^{-9}$	$2.6 \pm 1.8 \times 10^{-10}$	

Table 11. Average fraction release rates (F/cm²·s).^a

a Fraction of initial inventory released per s per cm2 of specimen external geometric surface area

b. Concentration of radionuclide or metal in resin waste was not determined.

c. Chelating agent was not present or was present at only a trace level in resin waste.

about 0.02% in the case of the AP/Citrox mixedbed resin waste form to about 1% in the case of the Can-Decon #1 waste form.

Approximately 91% of the inventory of ⁹⁰Sr in the AP/Citrox cation resin waste form was released during the first 2 hours of leaching. CFRs of ⁹⁰Sr from the remaining specimens ranged from about 2% for the AP/Citrox mixedbed resin waste form to about 38% in the case of the Can-Decon #1 waste form.

Plutorium-241 was detected in only one leachate sample—the first leachate for the AP/Citrox cation resin waste form. The ²⁴¹Pu detected in this leachate sample may have been attached to particulate matter suspended in the leachate solution. The CFR of ²⁴¹Pu from the AP/Citrox cation resin waste form was <2.2% after 90 days of leaching. CFRs of ²⁴¹Pu from the remaining specimens tanged from <0.024% in the case of the Can-Decon #1 waste form to <2.4% in the case of the Can-Decon #2 waste form. The latter CFP for ²⁴¹Pu is artificially high because the concentration of ²⁴¹Pu in the Can-Decon #2 waste resin was

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much lower than in the other resin wastes, but the detection limit concentration for ²⁴¹Pu was about the same for all leachate samples.

The data presented in Table 12 show that with the exception of the result for ⁶³Ni for the Dow NS+1 waste form, only small fractions of the inventories of the solidified ⁶⁰Co and ⁶³Ni were leached from the waste forms. CFRs of ⁶⁰Co and ⁶³Ni from the AP/Citrox cation resin waste form, which completely decomposed during early phases of leaching, were comparable to CFRs of ⁶⁰Co and ⁶³N. from waste forms that remained intact during leaching. These data indicate that large fractions of the quantities of these two radiomiclides in the waste forms were unavailable for leaching. A relevant finding of the study was that almost all of the Ca⁺² in tobomorites and xonotlite was irreversibly replaced by Co⁺² or Ni^{*2}.

Table 13 st immarizes the leachability indexes for the waste-form samples evaluated. They all exhibit a leachability index above the limit value of 6 delineated in the BTP, except the result for EDTA from the Dow NS-1 waste form, which was 5.7.

Waste-form identification	⁵⁵ Fe ^a	60Co	63N10	90Spd
AP/Citrox				
mixed-bed	1.3×10^{-2}	1.7×10^{-2}	1.5×10^{-4}	2.3×10^{-2}
cation	6.4×10^{-2}	1.7×10^{-3}	1.2×10^{-3}	9.9×10^{-1}
Can-Decon #1	1.2×10^{-4}	1.5×10^{-3}	9.6×10^{-3}	3.8×10^{-1}
Can-Decon #2	b	1.3×10^{-3}		$< 8.8 \times 10^{-2}$
Dow NS-1	2.2×10^{-5}	6.3×10^{-2}	9.4×10^{-1}	2.4×10^{-1}
LOMI	2.7×10^{-4}	1.3×10^{-3}	5.7×10^{-4}	2.3×10^{-1}
	137Cs	241 pua	Citric acid	Oxalic acid
-xP/Citrox				
mixed-bed	9.9×10^{-1}	$<3.4 \times 10^{-3}$	8.4×10^{-3}	-1.1×10^{-2}
cation	6.8×10^{-1}	$<2.2 \times 10^{-2}$	Section 2.	
Can-Decon #1	9.8×10^{-1}	$<2.4 \times 10^{-4}$	1.4×10^{-2}	$= 2.4 \times 10^{-2}$
Can-Decon #2	3.6×10^{-1}	$<2.4 \times 10^{-2}$	2.2×10^{-2}	1.8×10^{-2}
Dow NS-1	$< 8.6 \times 10^{-3}$	$<2.0 \times 10^{-3}$	4.3×10^{-1}	5.1×10^{-1}
LOMI	$1.0 \times 10^{+0}$	$<2.8 \times 10^{-3}$		
			Picolinic	
	EDTA	DTPA	neid	Nickel ^a
AP/Citrox				
mixed-bed	- A.		¢	$<1.5 \times 10^{-1}$
cation	and the second second		C	8.7×10^{-2}
Can-Decon #1	2.3×10^{-1}			1.4×10^{-2}
Can-Decon #2	7.1×10^{-2}			b
Dow NS-1	1.0 × 10*0	4.2×10^{-1}		$1.0 \times 10^{+0}$
LOMI	1.0 ~ 10	mand the star	8.1×10^{-1}	3.0×10^{-2}

Table 12. Cumulative fraction releases.

a. Not all leachases were analyzed for this radionuclide or metal. IFRs for leachates that were not analyzed for this radionuclide or metal were estimated by averaging release rates measured for adjacent leaching intervals. Estimated IFRs were added to measured IFRs to obtain the CFR.

b. Concentration of radionuclide or metal in ion-exchange resin waste was not determined.

c. Chelating agent was not present or was present at only a trace level in resin waste

AP/Citrox Image of the set of		63N	⁶⁰ Co	ss _{Fe}	Waste-form identification
mixed-bed 10.9 ± 0.4 10.5 ± 0.5 14.5 ± 0.5 >2 h 11.0 ± 0.5 10.6 ± 0.6 14.6 ± 0.5 >7 h 11.3 ± 0.4 10.9 ± 0.6 15.2 ± 0.6 cation 10.9 ± 1.2 12.7 ± 0.4 14.6 ± 1.5 >2 h 12.0 ± 0.6 12.5 ± 0.4 15.9 ± 0.6 >7 h 11.9 ± 0.9 12.7 ± 0.4 15.9 ± 0.6 Can-Decon #1 14.9 ± 0.6 13.2 ± 0.1 11.1 ± 0.6 Can-Decon #2 $-h$ 12.8 ± 0.1 $-h$ Dow NS-1 16.1 ± 0.5 92.2 ± 0.1 7.4 ± 0.6 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 $2 h$ $-a^{h}$ >12.3 $-a^{h}$ $2 h$ $-a^{h}$ >12.3 $-a^{h}$ $2 h$ $-a^{h}$ >12.3 $-a^{h}$ $2 h$ $-a^{h}$ >12.3 $-a^$	⁵⁰ Sr			Charles and the second s	identification
>2 h 11.0 ± 0.5 10.6 ± 0.6 14.6 ± 0.5 >7 h 11.3 ± 0.4 10.9 ± 0.6 15.2 ± 0.5 cation 10.9 ± 1.2 12.7 ± 0.4 14.6 ± 1.5 >2 h 12.0 ± 0.6 12.5 ± 0.4 15.9 ± 0.5 Can.Decon #1 14.9 ± 0.6 13.2 ± 0.1 11.1 ± 0.5 Can.Decon #2 h 12.8 ± 0.1 h Dow NS-1 16.1 ± 0.5 9.2 ± 0.1 7.4 ± 0.1 LOMI 14.6 ± 0.6 12.8 ± 0.1 h LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.5 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.5 LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.5 1001 13.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.5 2 h h >11.5 13.5 ± 0.5 2 h h >11.5 13.5 ± 0.5 137Cs 241Pu Citric acid AP/Citrox h >11.5 h mixed-bed h >11.5 h 2 h h h >10.6 h					
>7 h 11.3 ± 0.4 10.9 ± 0.6 15.2 ± 0. cation 10.9 ± 1.2 12.7 ± 0.4 14.6 ± 1. >2 h 12.0 ± 0.6 12.5 ± 0.4 15.7 ± 0. >7 h 11.9 ± 0.9 12.7 ± 0.4 15.9 ± 0. Can-Decon #1 14.9 ± 0.6 13.2 ± 0.1 11.1 ± 0. Can-Decon #2 $-h$ 12.8 ± 0.1 $-h$ LOMI 14.6 ± 0.6 12.8 ± 0.1 $-h$ $-2 h$ $-h$ -13.2 -13.2 $-2 h$ $-h$ -13.2 -13.2 $-7 h$ $-h$ -12.3 -15.2 $-7 h$ $-h$ -11.9 -5.2 $-5.2 h$ $-h^2$ -12.3 -5.2 Cation $-h^3$ -14.3 -16.2 <td< td=""><td>5 11.1 ± 0.7</td><td>14.5 ± 0.5</td><td>4 10.5 ± 0.5</td><td>10.9 ± 0.4</td><td></td></td<>	5 11.1 ± 0.7	14.5 ± 0.5	4 10.5 ± 0.5	10.9 ± 0.4	
cation 10.9 ± 1.2 12.7 ± 0.4 $14.6 \pm 1.57 \pm 0.4$ >2 h 12.0 ± 0.6 12.5 ± 0.4 15.7 ± 0.4 >7 h 11.9 ± 0.9 12.7 ± 0.4 15.9 ± 0.6 Can-Decon #1 14.9 ± 0.6 13.2 ± 0.1 11.1 ± 0.6 Can-Decon #2 $-b$ 12.8 ± 0.1 $-b$ Dow NS-1 16.1 ± 0.5 9.2 ± 0.1 7.4 ± 0.6 LOM1 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOM1 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOM1 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 LOM1 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0.6 $137Cs$ $241P_0$ Citric acid AP/Citrus $-a^a$ >11.7 11.5 ± 0.6 $22 h$ $-a^a$ >12.3 $-a^c$ Can-Decon #1 6.1 ± 0.3 >14.3 10.8 ± 0.6 Can-De	5 10.9 ± 0.8	14.6 ± 0.6	$5 = 10.6 \pm 0.6$	11.0 ± 0.5	
>2 h 12.0 ± 0.6 12.5 ± 0.4 15.7 ± 0. >7 h 11.9 ± 0.9 12.7 ± 0.4 15.9 ± 0. Can-Decon #1 14.9 ± 0.6 13.2 ± 0.1 11.1 ± 0. Can-Decon #2 $-h$ 12.8 ± 0.1 $-h$ Dow NS-1 16.1 ± 0.5 9.2 ± 0.1 7.4 ± 0. LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0. 1000 13.7 ± 0.5 9.2 ± 0.1 7.4 ± 0. LOMI 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0. 1001 14.6 ± 0.6 12.8 ± 0.1 13.5 ± 0. 13.7 Cs 241 p0 Citric acid 2 h $-a^{4}$ >11.5 1.5 ± 0. 2 h $-a^{4}$ >12.3 $-a^{5}$ Car-Decon #1 6.7 ± (1<	i 11.4 ± 0.7	15.2 ± 0.4	4 10.9 ± 0.6	11.3 ± 0.4	>7 h
>7 h 11.9 \pm 0.9 12.7 \pm 0.4 15.9 \pm 0. Can-Decon #1 14.9 \pm 0.6 13.2 \pm 0.1 11.1 \pm 0.5 Can-Decon #2 $-b^h$ 12.8 \pm 0.1 $-b^h$ Dow NS-1 16.1 \pm 0.5 9.2 \pm 0.1 7.1 \pm 0. LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0. LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0. LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0. LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0. LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0. AP/Citrox		14.8 ± 1.3	2 12.7 ± 0.4	10.9 ± 1.2	cation
Can-Decon #1 14.9 \pm 0.6 13.2 \pm 0.1 11.1 \pm 0. Can-Decon #2 -3^{h} 12.8 \pm 0.1 -5^{h} Dow NS-1 16.1 \pm 0.5 9.2 \pm 0.1 7.4 \pm 0.1 LOMI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0.1 100MI 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0.1 110 14.6 \pm 0.6 12.8 \pm 0.1 13.5 \pm 0.1 128 2.1.7 11.1 \pm 0.5 2.2 \pm 0.1 7.4 \pm 0.1 100 13.5 \pm 0.1 13.5 \pm 0.1 13.5 \pm 0.1 13.5 \pm 0.1 2 h $-a^{a}$ >12.3 11.5 \pm 0.1 15.5 \pm 0.1 >2 h $-a^{a}$ >12.3 15.5 \pm 0.1 15.5 \pm 0.1 137Cs 244 Pu Citric acid 15.5 \pm 0.1 10.1 10.0 \pm 0.1 solon $-a^{b}$ >12.3 $-a^{c}$ a^{c} a^{c} a^{c} 2 h $-a^{b}$ >10.1 10.0 \pm 0.1 10.0 \pm 0		15.7 ± 0.6	6 12.5 ± 0.4	12.0 ± 0.6	
Can-Decon #2 $-b$ 128 ± 0.1 $-b$ Dow NS-1 16.1 ± 0.5 9.2 ± 0.1 7.4 ± 0.1 LOMI 14.6 ± 0.6 42.8 ± 0.1 13.5 ± 0.1 Image: the end of the en		15.9 ± 0.8	9 12.7 ± 0.4	11.9 ± 0.9	>7 h
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Table 13. Leachability indexes.

a. CFR was greater than 20%: therefore, the approximate semi-infinite tredium diffusion model could not be used to calculate the effective diffusivity

b. Concentration of radionaclide or metal in resin waste was not determined.

c. Chelating agent was not present or was present at only a trace level in resin waste-

10. MEETINGS ATTENDED

C. V. McIsaac attended "Waste Management '91" in Tucson, Arizona, and presented the experimental results obtained during FY-90 for the Low-Level Radioactive Waste—Decontamination Waste Program (FIN A6359). The results were presented in a poster paper titled "Effects of Leachant Chemical Composition and pH on Leachability of Cement-Solidified Decontamination Ion-Exchange Resin Waste."

11. ANNUAL PROGRAM REVIEW

The Annual Program Review by the NRC was completed in July, 1991.

12. REFERENCES

- Code of Federal Regulations, 10 CFR 61, "Licensing Requirements for Land Disposal of Radioactive Wastes," Office of the Federal Register, December 1982.
- U.S. Nuclear Regulatory Commission, "Technical Position on Waste Form," Revision 1, Low-Level Waste Management Branch, Washington, D.C., January 1991.
- C. V. McIsaac, D. W. Akers, and J. W. McConnell, Effect of pH on the Release of Radionuclides and Chelating Agents from Cement-Solidified Decontamination Ion-Exchange Resins Collected from Operating Nuclear Power Stations, NUREG/CR-5601, EGG-2605, June 1991.
- C. V. McIsaac and S. T. Croney, "Effect of Waste Form Size on the Leachability of Radionuclides Contained in Cement-Solidified Evaporator Concentrates Generated at Isuclear Power Stations," Waste Management, Vol. 11, pp. 271–282, 1991.
- American Nuclear Society, 16.1, "Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure," February 1986.
- 6. Morcos et al., Properties of Radioactive Wastes and Waste Containers NUREG/CR-2617, April 1982.

Appendix A

Information Regarding the Compression Testing of Tapered Cylindrical Waste Forms

Appendix A

Information Regarding the Compression Testing of Tapered Cylindrical Waste Forms

Mr. Phillip R. Reed Waste Management Branch Office of Nuclear Regulatory Research U.S. Nuclear Regulatory Commission Washington, D.C. 20555

INFORMATION REGARDING THE COMPRESSION TESTING OF TAPERED CYLINDRICAL WASTE FORMS - RMN-34-91

Dear Mr. Reed:

Mr. John McConnell asked me to provide information regarding the compression testing of tapered cylindrical waste forms in support of the Branch Technical Fosition (BTP) stability guidance for processed (solidified) Class B and C wastes. Specifically, 1 will address whether the waste forms can be tested in accordance with ASTM C-39 "Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens" and the significance of test results.

Waste forms obtained from the Peach Bottom Atomic Power Station were collected and solidified in paper cups. The resultant waste forms are cylindrical with a length of approximately two inches, but are tapered along the cylindrical axis. The minor diameter of the waste form is approximately two inches and the major diameter is approximately three inches.

ASTM C-39 does not have a restriction regarding taper of the specimen along the cylindrical axis. However, since the test method is normally applied to concrete cores or molded cylindrical specimens, it is understood that the normal specimen is a right cylinder (no taper). The test procedure can be applied to a tapered specimen: the taper presents no fundamental problem in application of the compressive test. However, a significant question develops regarding the calculation of the test specimen compressive strength.

The Standard Test Method includes a procedure by which the compressive strength of the test specimen is calculated. In this test method, the diameter of the test specimen is determined to the nearest 0.01 inch (0.25 mm) by averaging two diameters about the midpoint of the specimen. This average diameter is used to calculate the cross-sectional area of the specimen. The compressive strength is determined by dividing the maximum load supported by the specimen during the test and dividing this value by the cross-sectional area. This calculation procedure is probably not appropriate for specimens with significant tapers along the cylindrical axis.

Mr. Phillip R. Reed March 1, 1991 RMN-34-91 Page 2

A right circular cylinder geometry under load has a constant stress level along its length (except near the specimen ends as is discussed later) since the specimen cross section at right angles to the cylindrical axis is constant. In a tappred specimen, the stress distribution changes along the length of the test specimen since the cross section supporting the load also changes along the length. Neglecting end effects and assuming a specimen of uniform strength throughout its volume, the tapered specimen should fail at its smallest diameter since it is in this cross section that the stress level is the highest.

The situation is complicated by end effects. Near the ends of the specimen, frictional constraints with the test machine platens affects the stress distribution by restricting lateral expansion at the ends of the specimen. In many uniaxial compression tests, cylinders with a length to diameter ratio of two or more are often specified (although not in ASTM C-39) so that the stresses in the central portion of the specimen are only slightly affected by contact with the platens. However it is often noted in uniaxial compression tests that failure starts where the circumference of the cylinder is in contact with the platen. Filon (1902) showed for cylinders with a length to diameter racio of one that the stress concentration at the circumferential contact is 1.69. As a result, the effect of lateral constraint is often to initiate failure at the contact between the specimen and the platen at a load value less than that corresponding to the true uniaxial strength of the specimen. This gives rise to the conical fragments which are often observed in these tests.

Sulfur cement or metal end caps are often applied to specimens for uniaxial compression testing. The primary reason for this is to eliminate bending moments during testing which result from departure of the specimen ends from perpendicularity to the cylinder axis (ASTM C-39 specifies a maximum departure from perpendicularity of 0.5 degrees at either end). The use of end caps also minimizes lateral constraint between the test machine platens and the specimen.

This suggests that an reasonable approach to the uniaxial compression testing of a tapered cylindrical specimen would be to use its minimum diameter, and not its midheight diameter, in the calculation of compressive strength. The effect of using this minimum diameter to calculate the specimen cross section rather than the diameter at the midheight of the specimen is illustrated below for the geometry of the tapered Peach Bottom specimens and assuming a load at failure of 5000 lbf: Mr. Phillip R. Reed March 1, 1991 RMN-34-91 Page 3

Specimen Diameter, inches	Cross Sectional Area, inch ²	Compressive Strength,
2.0 (minimum diameter)	3.14	1,590
2.5 (midheight diameter)	4.91	1,020

For this geometry, the difference in the calculated compressive strengths is approximately 55%. Note however, that the strength calculated using the minimum diameter is more likely to approximate the actual material compressive strength, assuming that the test specimen is uniform in strength throughout its volume. If the strength is not uniform throughout its volume, the specimen will fail at the cross section where the stress first exceeds the material strength. If the location of failure is not near the minimum di _ter (highest applied stress level), calculating the compressive strength using the minimum diameter will over predict the strength (by the ratio of the cross sectional area at the failure location to that at the minimum diameter).

It would be desirable to use the cross sectional area at the location of failure initiation for the calculation of compressive strength. However, since specimen failure during compression testing tends to be violent and produce conical fragments, it is often difficult to determine where failure initiated. For this reason, it is not recommended that compressive strength be calculated by using the cross sectional area at the point of failure initiation.

Note that compression testing of tapered specimens is not unique. Earlier NRC sponsored work at both Idaho National Engineering Laboratory (INEL) and BNL has used tapered specimens. This occurred because tapered molds were used to facilitate specimen release after curing. The taper in these molds was small. Measurements made on an EPICOR test specimen indicates a taper of approximately 0.28° along the cylinder axis. Such a small taper has a negligible effect on calculated compressive strength. (For the particular specimen measured, which had a length of 2.551 inches, a minimum diameter of 1.737 inches, and a maximum diameter of 1.762 inches, the assumption of a 5,000 lbf load at failure results in a calculated compressive strength of 2,110 psi using the minimum diameter and 2,080 psi using the midheight diameter.)

The use of the minimum diameter is recommended for calculation of compressive strength in the tapered Peach Bottom specimens since it is believed to provide the best reasonably obtained estimation of strength. The question could arise as to whether this approach will drive other people towards the use of tapered specimens since it could provide an overestimate of compressive strength for Mr. Phillip R. Reed March 1, 1991 RMN-34-91 Page 4

marginal and/or nonuniform waste forms. If this is a concern, it could be resolved by requiring the use of the midheight diameter for the compressive strength calculation. This would provide a more conservative (lower) estimate of compressive strength although at a probable loss of accuracy relative to use of the minimum diameter in the calculation.

Please call me at (208) 526-8274 if you have any questions regarding the information provided.

Yours truly. sect the healson, for

Robert M. Neilson, Jr. Manager, Metals and Ceramics Materials Technology Group

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Appendix B

Trip Report of the Collection of Solidification Samples at Nine Mile Point Nuclear Plant Unit 1

Appendix B

Trip Report of the Collection of Solidification Samples at Nine Mile Point Nuclear Plant Unit 1

Date: April 2, 1991

To: J. W. McConnell, MS 1406

From: M. R. Winberg, MS 7113 MAlluber

Subject: TRIP REPORT OF THE COLLECTION OF SOLIDIFICATION SAMPLES AT NINE MILE POINT NUCLEAR PLANT UNIT 1- MRW-26-91

On March 11, 1991, John McConnell and I arrived at Nine Mile Nuclear Plant Unit 1 (NMP-1) to collect samples from their solidification of filter sludge. The source of this filter sludge consisted of lowconductivity wastes from the reactor water cleanup system and floor drain wastes primarily from the Elevation .55 decontamination. These wastes were transferred through the plant radwaste system and collected on polyproplylene wound septum (Precoat) filters. The waste was then cleaned off the filters and collected in a line for solidification. The volume of waste transferred to the liner was 120 ft³. The plant history shows that these wastes were expected to be Class B wastes. Unfortunately, when the chemistry reports were collected and analyzed, the waste was determined to be Class A. That analysis is attached.

The preparation of the PCP sample was performed by the Chem-Nuclear (CNSI) technician on February 21, 1991. Consequently, we were unable to witness mixing of the PCP sample used to calculate the mixing pa. meters of the liner. We were later able to inspect the PCP sample and it appeared to have cured properly. Sampling of the filter sludge prior to solidification was performed on March 13, 1991. The stir paddles were turned on for approximately 10 minutes to allow the contents of the liner to mix before sampling. Sampling of the liner was accomplished using a plastic beaker attached to a long handle. The beaker was dipped into the waste mixture in the liner through the camera access door on the fill head using the handle to collect 500 ml of sample. The beaker was then removed from the liner and poured into a bucket. Photographs were taken of this operation from the top of the liner. Approximately 1 liter of sludge was sampled and divided into 2 aliquots; 1) 500 ml for mixing of additional PCP samples in cylindrical containers for shipment to INEL for compressive strength tests (these samples were mixed on March 19, 1991, after we left, using the same recipe as the original PCP sample), and 2) 500 ml for shipment to INEL for waste characterization. The radiation reading on the 1 liter container of sludge was 320 mrem/hr.

J. W. McConnel. Ap:il 2, 1991 MRW-26-91 Page 2

The CNSI solidification system was composed of four parts: 1) a large bulk trailer which contained the proprietary cement: 2) a blower system to move the cement through a 4 inch line to the fill head: 3) a fill head which attaches to the top of the liner and connects to the stirring paddles, and 4) a control console used to monitor the solidification process and control cement feeding operations. The fill head also houses the camera system used to view the inside of the liner during solidification an a cyclone air cleaning system to minimized dust generation du ng cement feeding. In addition, a thermocouple is inserted into liner down the shaft of the stirring paddles to monitor the exotherm created from the chemical reaction of the curing c ment.

An attempt was made to solidify the liner on March 14, 1991. The solidification was not performed due to equipment problems (a dust leak in the fill head and a faulty valve actuator on the community feed system). Repairs were completed on March 14 and the actual solidification was accomplished the following day.

Solidification was performed on March 15, 1991. The process began at 11:00 a.m. with addition of cement to the liner. The mixing of the PCP showed that the liner pH did not require the addition of lime or sodium hydroxide to raise the pH to the required level, Therefore, the only material added to the waste in the liner was the cement. Cement was added to the liner in 100 15, increments until approximately 2:00 p.m. when a total of 7140 lbs. of cement had been added to the liner. The density of the cement is 173 lb/ft³. The addition of cement increased the volume of the liner by 41.3 ft³ for a total of 161.3 ft³ of waste. The waste loaded percent was 74.4% and the visite to cement ratio was 2.91. The total liner volume was 169.5 ft³. The paddles continued to spin for an additional 30 min. as required by the CNSI procedure. At this time sampling of the waste/cement rixture was accomplished.

Sampling was performed at approximately 3:00 p.m. on March 15, 1991. Sampling of the liner was accomplished using a plastic beaker attached to a long handle. The CNSI technician inserted the beaker into the liner via the camera access door on the fill head. The beaker was dipped using the handle into the cement/waste mixture and collected 500 ml of sample. The beaker was then removed from the liner and poured into a bucket. This process was repeated until 6 gts. of sample were collected. Photographs of this process were taken from ground level. The bucket was then brought down from the liner to a sample staging area where the sample was transferred from the bucket into the 2 in. diameter by 4 in. long sample containers

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J. W. McConnell April 2, 1391 MRW-26-91 Page 3

for final curing. The sample was poured from the bucket to the sample containers using a plastic beaker. Thirty (30) samples were collected in this manner and place in the CNSI oven and baked at 140 degrees F for three days. Photographs were taken of the collection process and loading the samples into the oven. The radiation reading on the bucket of sample collected from the liner was 400 mrem/hr at contact while the radiation readings of the individual sample containers were about 60 mrem/hr at contact. Photographs that were taken will be available upon printing by EG&G Photographic Services.

Information collected at the plant and included here consist of chemistry reports detailing radionuclide content, waste classific ion, and gamma spectroscopy report on the sludge before solidification.

The solidification appears to have gone well. The sludge itself was a slightly viscous, brown-black, liquid. The waste/cement mixture was smooth and consistent and was thin enough to be able to be poured into the sample containers. With the exception of the mixing of the PCP samples, EG&G was able to observe all phases of the solidification and sampling of the liner. Shipping of the solidified liner to Barnwell occurred March 21, 1991.

/*tached are copies of all information collected at the plant. If fere are any questions please contact me at 6-4042.

Attachments: As stated.

cc: D. W. Akers, MS 2114
J. W. Mandler, MS 2219
C. V. McIsaac, MS 7113
W. Serrano, MS 7113
M. R. Winberg ltr file

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BATCH # :		1 (3)	(4)	1 (5) 1	(6)	(7)	(8)	(9)	(10)	(11)	(12)	(13)	(14)
(1) 61.55 Table 1	(2) Nuclide uCi/ml	Class	Class A Fector	Class	Cless C	61.55 Table 2	Nuclide uCi/ml	Cless A	Class A Factor	Class	Class B Factor	Class	Class C Factor
C-1, C-14(m) Ni-59(m) Nb-94(m) Tc-99 Tc-99 T-129 Pu-241(b) Cm-242(b) TRU(b)	3.357E-06 3.825E-05 1.480E-05 8.729E-01	0.8 8 22 0.02 0.3 0.008 350 2000 10(c)	4.196E-06 1.275E-04 1.850E-03 2.494E-03 1.355E-07 3.008E-02	8 80 220 0.2 3 5.08 3500 20000 100(c)	4.196E-07 1.275E-05 1.850E-04 2.494E-04 1.355E-08 3.088E-03	H-3 Co-60 Ni-63 Ni-63(0) Sr-90 Cs-137 T1/2 <syr< td=""><td>9.364E-04 4.455E-01 2.828E-02 6.698E-03 5.921E-01 1.790E+00</td><td>40 700 3.5 35 0.04 1 700</td><td>2.341E-05 6.362E-04 8.080E-03 1.525E 01 5.921E-01 2.557E-03 7.558E-03</td><td>XXX XXX 70 7.00 150 4.4 XXX</td><td>N.L. N.L. 4.040E-04 4.066E-05 1.346E-02 N.L. 1.390E-02</td><td>*** *** 700 7000 7000 4600 ***</td><td>N.L. N.L. 4.0406-0 8.712E-0 1.287E-0 N.L. 1.700E-0</td></syr<>	9.364E-04 4.455E-01 2.828E-02 6.698E-03 5.921E-01 1.790E+00	40 700 3.5 35 0.04 1 700	2.341E-05 6.362E-04 8.080E-03 1.525E 01 5.921E-01 2.557E-03 7.558E-03	XXX XXX 70 7.00 150 4.4 XXX	N.L. N.L. 4.040E-04 4.066E-05 1.346E-02 N.L. 1.390E-02	*** *** 700 7000 7000 4600 ***	N.L. N.L. 4.0406-0 8.712E-0 1.287E-0 N.L. 1.700E-0
			3.535E-02 aste must De		3.535E-03 d es STABLE								
	NOTES:	(a) ect	ivated metal	s only		1	echnician:				DATE		
			ressed as no			Ve	rified by:				DATE		
		(c) tot N.L. no	al of transi	ganics.	listed	Re	viewed by:				DATE		

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Appendix B

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Contra a

NINE MILE POINT NUCLEAR STATION UNIT-1 Rede-1 14-MAR-91 13:17:00

ANALYSIS PARAMETERS

MCA UNIT NUMBER: 1 / ADC UNIT NUMBER: 1.0 DETECTOR NUMBER: 1 / SECMETRY NUMBER: 12 SPECTRUM SIZE: 4096 CHANNELS FROM MCA REGION H1 ORDER OF SMOOTHING FUNCTION: 5 NUMBER OF BACKGROUND CHANNELS: 4 ON EACH SIDE OF PEAK PEAK CONFIDENCE FACTOR: 80.02 IDENTIFICATION ENERGY WINDOW: +- 1.00 KEV ERROR GUOTATION: 1.00 SIGMA UNCERTAINTY

MULTIPLET ANALYSIS PERFORMED

REGULAR OUTPUT SPECTRAL DATA READ DIRECTLY FROM MULTICHANNEL ANALYZER ANO ANALYZED BY: J WOODS

SAMPLE DESCRIPTION: FS THICKENER TANKS GEOMETRY DESCRIPTION: FOLY BOTTLE 3 CM SAMPLE SIZE: 2.0000E+00 ml / CONVERSION FACTOR: 1.0000E+00 STANDARD SIZE: 1.0000E+03 ML AN LYSIS LIBRARY FILE: ANLOOI

COLLECT STARTED ON 14-MAR-91 AT 12:42:12

COLLECT	LIVE TIMF:	2000. SECONDS
	REAL TIME:	2080 SECONDS
	DEAD TIME:	(3.85 3

DECAYED TO O. DAYS, 0.0000 HOURS BEFORE THE START OF COLLECT

ENERGY CALIBRATION PERFORMED 14-MAR-91 EFFICIENCY CALIBRATION PERFORMED 24-001-90

NINE MILE FOINT NUCLEAR STATION UNIT-1 ReGe-1 14-MAR-91 13:17:00

PEAK ANALYSIS

PK	CENTRCID CHANNEL	ENER"Y Kev	e MHH E MHH	BACKGND COUNTS	NET AREA COUNTS	ERROR	NUCLIDES
2242	1208.45 1322.31 1599.86 1603.46 1600.39 1603.51	661.65 795.87 802.17 910.74	1.3 1.4 1.4	3032. 3461. 4491.	110352. 787. 128. 473.	2.3 19.9 30.7 22.4	CS-134 CS-127 CS1348 CS134C I=1325 KA-TSE, MA-5-

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Appendix B

ERROR QUOTATION AT 1.00 SIGHA PEAK CONVIDENCE LEVEL AT 80.0% C ~ MULTIPLET ANALYSIS CONVERGED NORMALLY ? ~ MULTIPLET ANALYSIS CONVERGED BUT GFIT > 4

NINE MILE POINT NUCLEAR STATION UNIT-1 ReGa-1 14-MAR-91 13:17:00

SAMPLE: ES THICKENER TANKS DATA COLLECTED ON 14-MAR-91 AT 12:42:12 DECAYED TO O. DAYS, 0.0000 HOURS BEFORE THE START OF COLLECT.

RADIONUCLIDE ANALYSIS REPORT

NUCLIDE ACTIVITY CONCENTRATION IN UCI/#1

	MEASURED	ERROR	CORRECTED	ERROR
C0-58 C0-60 C0-608 CS-104 CS-1348	3.53E-02 + 6.01E-01 + 6.04E+01 + 5.56E+03 + 6.73E-03 +	- 1.70E-02 - 1.69E+02 - 6.80E=04	3.53E-03 *- 6.01E-01 *- 6.04E-01 *- 5.56E-03 *- 6.73E-03 *-	1.70E-02 1.69E-02 6.80E-04
CS-134C CS-137 MN-54	1.50E-02 + 8.03E-01 + 1.05E-02 +	- 4.63E-03 - 2.60E-03	1.50E-02 + 8.03E-01 +- 1.05E-02 +	2.60E-02
TOTAL	2.05E+00 +	- 3.57E-02	2.05E+00 +-	3.572-02

ERROR QUOTATION AT 1.00 SIGMA

NINE MILE POINT NUCLEAR STATION UNIT-1 ReGe+1 14-MAR-91 13:17:00

NO DOSE EQUIVALENT 1-131 VALUES LISTED FOR IDENTIFIED NUCLIDES.

ALL DETECTED PEAKS WERE USED IN THE ANALYSIS

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B-8

Characterization of Chemical Wastes Present in Radioactive Waste Forms

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Characterization of Chemical Wastes Present in Radioactive Waste Forms

INTRODUCTION

The U.S. Nuclear Regulatory Commission (NRC) has defined a strategy for conducting research on issues of concern in its efforts to assure safe disposal of low-level radioactive waste. This strategy, defined in the Low-Level Radioactive Waste Research Program Plan,¹ provides requirements for research on the stability and leachability of radioactive waste forms. Research being performed at the Idaho National Engineering Laboratory (INEL) as part of FIN A6359 includes requirements to assess the quantities of chemical wastes present in waste streams collected from operating commercial light water reactors (LWRs). This research is being performed to determine those trace chemicals, both organic and inorganic, at the parts-per-million level that could affect the stability of cement solidified low-level radioactive waste.

This program plan addresses the methods used for sampling radioactive waste streams, identifying chemical components of those samples, and analyzing the samples for chemical components that could affect the stability of the waste forms. The four sections of this plan are: sampling and field measurements, chemical waste identification, laboratory analysis for chemical constituents, and conclusions. The sampling section defines methods for sampling waste streams at commercial facilities and field screening techniques. The chemical waste identification section identifies waste form components that could impact solidification including non-aqueous organics, aqueous organics, and inorganics. Finally, the laboratory analysis section provides brief descriptions of relevant analytical methods that would be used to qualitatively and quantitatively assess these constituents.

SAMPLING AND FIELD MEASUREMENTS

This section addresses the proposed methods for sampling waste streams prior to solidification and possible screening techniques that could be used at the facility. Perhaps the most critical aspect of this analytical plan is proper sampling. This is particularly difficult at commercial reactors due to the radiation fields from the sample and the location in the reactor

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plant where the sample is to be obtained. Also, the active production aspect of most plants makes waste stream sampling or setting up field screening techniques difficult. Further, access to appropriate sampling locations may require extensive, time-consuming planning to overcome plant health and safety restrictions.

Sampling and field techniques have been addressed in the form of protocols by a number of federal agencies and research groups. Field screening procedures have been prepared by the Environmental Protection Agency (EPA) for hazardous but not radioactive environments², and sampling of waste tanks containing medium to high levels of mixed fission products for subsequent organic compound analysis has been described in detail in a current Oak Ridge Report by Audrey et al.³ EPA, ASTM, or Standard Methods of sampling usually have to be modified because of the difficulty and hazards of handling radioactive waste forms and waste streams.^{4,5} These techniques are included in the sampling procedure described below.

Three types of samples from commercial reactor waste streams are desirable. They are the liquid waste stream before solidification, the cement-resin or evaporator waste taken during solidification, and a core from the solidified monolith (if possible). Waste from uquids can be sampled from: the plant chemical and volume control sampling system (CVCS), the pre-solidification holding tank, or the liner before the addition of any solidification agent. Representative sampling of these systems requires that the waste must be fully mixed prior to sampling and that the sample line must be completely flushed out before a sample is acquired. Consequently, either the sample line must be flushed for 5-10 minutes before a sample is obtained or the sample must be obtained directly from a holding tank or liner that is being stirred. In some cases, where these approaches are impractical, an automatic proportional composite sampler may be required to provide a homogenous sample. This sampler uses a pumping system to periodically obtain a sample from the waste stream and put it in a composite sample.

Radioactive liquids in a holding tank or the liner can be sampled with a vacuum pump. For most inorganic analysis sampling protocols, sampling without exposure to air is not a requirement; however, for samples where volatile organic analysis (VOA) or certain inorganic species analyses are required, limited exposure to air or other precautions must be taken.

Where volatile species are present, the sample is drawn from a waste stream or the waste holding tank using a vacuum pump into a precleaned 250-ml organic free jar. Approximately

15-25 ml of headspace is left in the jar. The samples are then screened and tagged. Refrigeration is usually required in the EPA VOA sampling protocol, however, this is not considered necessary for waste stream samples which have already been exposed to higher temperatures.

Sampling the waste and concrete slurry mixture in the liner, after the soldification agents have been added, will be done with the large plastic syringe that has been used to obtain slurry samples as part of this program. The length of this device is 4-5 feet to allow samples to be obtained from various depths in the liner. Samples are typically obtained several feet below the surface.

Obtaining a solid core sample from the solidified monolith for examinatin will require preplanning and extensive practice with cold samples. A lead shield on the top of the liner with a small hole large enough to allow a standard concrete core bore entry is required. Also, plans to patch the liner after the sample is obtained must be completed. Subsequent standard VOA analysis for a solid material as implemented in the INEL laboratories is followed. The sample will be maintained in a closed container prior to sampling and the head space gas will be sampled and analyzed. This approach requires special procedures to assure that the head space gas is retained for analysis. It is important that the samples where volatile organics are present be transported to the analysis laboratory as soon as possible, because the EPA protocol recommends that the maximum holding period for volatile samples is about 2 weeks.

Initial testing of the sampling and handling procedures as defined in this program plan is required because only a laboratory containing the proper glove box and/or hot-cells and appropriate training can ensure the safety of the operation. Testing the receiving, sample splitting, and analysis procedures, especially the glove box sparge and trap, is essential. In all cases, the sample analyses will be performed in a laboratory where accurate measurements can be performed using standard protocols that have been modified for the radioactivity of the samples.

In addition to the waste stream and waste form samples themselves, nonradioactive resin samples and decontamination chemical solution samples should be obtained where possible. These samples will be used for calibration of the analysis instrumentation. In addition, all possible plant information, operational procedures, and vendor information should be obtained to determine which chemical constituents are present that may adversely effect solidification.

Several techniques have applications for on-line analyses and field screening of gaseous, liquid and solid waste streams.² Most are considered qualitative and few if any have been tried or verified for radioactive streams. These analysis techniques do not appear to meet the programmatic requirements for analysis at the parts-per-million level. However, the difficulty in obtaining or transporting samples to the lab may preclude analysis of certain components due to holding time or sample preservation considerations. In this case, a mobile lab or field program would then be essential. However, current indications are that holding time and sample preservation are not of particular concern for the chemical components of primary interest.

CHEMICAL WASTE IDENTIFICATION

The Hanford and Barnwell waste disposal state licenses require that wastes with activities greater than 1 μ Ci/cm³ be stabilized. Stability of these wastes may be achieved by dewatering and disposal in a high integrity liner, or solidifying the waste. For a typical LWR, the estimated fractional breakdown of solidified wastes is 45% letdown system and decon resins, 22% boric acid evaporator waste, 15% sludges, 11% concentrator wastes, and 4.5% oil.⁶ Solidification problems from inorganics such as sulfates and borates have generally been resolved, however, problems have occurred from the presence of unknown organics in oils, evaporator wastes and sludges or from organic byproducts in decontamination or other ion-exchange resins.

Tables 1 and 2 are compiled from an NRC list of waste constituents that may cause problems with cement solidification.⁷ These constituents have been classified in three broad categories: inorganic, organic-aqueous and organic-nonaqueous components. This classification system lends itself to the unique handling, sampling and analysis technique requirements associated with each type of sample. Instrumental analytical procedures have been identified for the analysis of samples of these types:

- Inorganic cations and anions inductively coupled plasma spectroscopy (ICP) and ion chromatography (IC)
- Organic-aqueous compounds liquid chromatography (LC)
- Organic-nonaqueous volatile substances gas chromatography/mass spectroscopy (GC/MS).

	Туре	Chemical	Analytical Method
Inorganic	Non-metallic anions	borates phosphates sodium hypochlorite ammonia sulfates nitrates pH total solids total dissolved solids	IC ^a , LC ^b , ICP ^c IC IC IC IC IC WM ^d WM
	Metals	lead zinc iron	WM ICP ICP, AA ^c ICP
	Metals-oxidizing agents	manganese chromium	ICP, IC ICP, AA, IC
Organic-Aqueous	Carboxylic acid chelates	formic acid-formates oxalic acid oxalates citric acid-citrates picolinic acid-picolinates	IC IC IC LC
	Chelating agents	NTA EDTA "Decon Solutions"	LC, IC LC, IC LC, IC
	Surfactants	soap detergent	LC, IC, WM LC, IC, WM

Table C-1. Aqueous soluble waste constituents that may affect cement solidification.

a. IC = ion chromatography

b. LC = liquid chromatography

c. ICP = inductively coupled plasma

d. WM = standard wet methods

c. AA = atomic absorption

The list was generated from NRC Notice No. 90-31.

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	Туре	Chemical	Analytical Method
Organic-Nonaqueous	Aromatics	"oils"	GC/MS ^a
corganie-reonadaeosas	Theomatics	benzene	GC/MS
		toluene	GC/MS
		xylene	GC/MS
	Petroleum distillates	vegetable oil additives	GC/MS, WM ^b
	a water owned another the	hexane	GC/MS
		oil and grease	WM
		paint thinners	GC/MS
		industrial cleaners	GC/MS
	Chlorinated solvents	trichlorethane	GC/MS
		trichloro-trifluorethane	GC/MS
		dicholorbenzene	GC/MS
		tri-chloroethene	GC/MS
		tetra chloroethene	GC/MS
		methylene chloride	GC/MS
		dry-cleaning solvents	GC/MS
	Ketones	acetone	GC/MS
		methyl ethyl ketone	GC/MS
		methyl isobuty! ketone	GC/MS

Sec.

Table C-2. Organic waste constituents that may affect cement solidification.

a GC/MS = gas chromatography/mass spectroscopy

b. WM = standard wet methods

The list was generated from NRC Notice No. 90-31.7

Standard wet methods (WM) may also be used for general water quality parameters. Tables 3 and 4 list specific chemical detection limits for these analytical techniques on non-radioactive samples. Detection limits have not been identified for radioactive samples, although the limits should be similar depending on whether the samples needed to be diluted to reduce their radioactivity for analysis.

The chemical components listed in Tables 3 and 4 are further categorized to give an indication of the probable contents of nonspecific constituents such as decontamination solutions or vegetable oil additives. A best guess is made on what method might be most practical in the analysis of generic components, although some research may be required to define the best analytical method with the fewest interferences for specific components.

LABORATORY ANALYSIS FOR CHEMICAL CONSTITUENTS

This section addresses the specific chemical analysis procedures that have been identified for the chemical components identified in the previous section. Standard Environmental Protection Agency (EPA), ASTM or Standard Method procedures⁸ can be modified to assess the concentrations of the four broad chemical groups that may affect solidification. These groups of chemical compounds are: volatile organics, dissolved organics (organic anions), dissolved metals (cations), and inorganic anions. The procedure for the general analysis is given along with a brief outline of the procedure itself. In addition, some of the procedural modifications necessary for handling radioactive are included where they have been identified. Further, quality assurance requirements for the specific procedures are identified.

Volatile Organics

Volatile organic compounds may be identified and quantified in radioactive aqueous samples containing mixed fission products using a modified version of the EPA purge-and-trap procedure for nonradioactive samples.⁹ A gas chromatography/mass spectrometer and special 'sorption trap are required. Optimum detection limits for liquid and solid samples using this technique are listed in Table 4. The use of this technique for radioactive solid samples requires modification of EPA SW-846 methods (see Reference 4).

Element	Method Detection Limits ^a (µg/L, ppb)
Aluminum	200
Antimony	150
Arsenic	250
Barium	200
Beryllium	5
Cadmium	20
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	200
Magnesium	5000
Manganese	15
Mercury	1 ^b
Nickel	40
Potassium	5000
Selenium	400
Silver	30
Sodium	5000
Thallium	500
Vanadium	50
Zinc	20
Anions ^c	
Fluoride	5
Chloride	15
Nitrite-N	5
O-Phosphate-P	60
Nitrate-N	15
Sulfate	200
Citric	500
Oxalic	200
Formic	200
Picolinic	100

Table C-3. Detection limits for ICP and IC analyses.

a. These MDLs are the ICP (inductively coupled plasma), IC (ion chromatography), and cold vapor AA (atomic absorption) detection limits obtained in pure water that have been met using the procedure. The detection limits for radioactive samples may be considerably higher depending on the sample matrix.⁸

b. Furnace AA is required to attain this MDL. Mercury is not determined in the ICP method.

c. Ion chromatography of aqueous solutions.

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Table C-4. Detection limits for GC/MS.	Table C-4.	Detection	limits for	GC/MS.
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			Detection Limits ^{a,b}		
	Analyte	CAS Number	Water So (µg/L)	il/Sediment (<u>µg/kg)^c</u>	
	Chloromethane	74-87-3	10	10	
	Bromomethane	74-83-9	10	10	
	Vinyl chloride	75-01-4	10	10	
	Chloroethane	75-00-3	10	10	
5,	Methylene chloride	75-09-2	5	5	
б.	Acetone	67-64-1	10	10	
7.	Carbon disulfide	75-15-0	5	5	
8.	1,1-Dichloroethene	75-35-4	5	5	
9.	1,1-Dichloroethane	75-35-3	5	5	
10.	1,2-Dichloroethene(total)	540-59-0	5	5	
	Chloroform	67-66-3	5	5	
12.	1,2-Dichloroethane	108-06-2	5	5	
13.	2-Butanone	78-93-3	10	10	
14.	1,1,1-Trichloroethane	71-55-6	5	5	
15.	Carbon tetrachloride	56-23-5	5	5	
16.	Vinyl acetate	108-05-4	10	10	
17.	Bromodichloromethane	75-27-4	5	5	
	1,2-Dichloropropane	78-87-5	5	5	
	cis-1,3-Dichloropropene	10061-01-5	5	5	
	Trichloroethene	79-01-6	5	5	
21.	Dibromochloromethane	124-48-1	5	5	
22	1,1,2-Trichloroethane	79-00-5	5	5	
23.	Benzene	71-42-2	5	5	
24.	trans-1,3-Dichloropropene	10061-02-6	5	5	
	Bromoform	75-25-2	5	5	
	4-Methyl-2-pentanone	108-10-1	10	10	
27	2-Hexanone	591-78-6	10	10	
	Tetrachloroethene	127-18-4	5	5	
29		108-88-3	5	5	
	1,1,2,2-Tetrachloroethane	79-34-5	5	5	
	Chlorobenzene	108-90-7	5	5	
	Ethyl benzene	100-41-4	5	5	
	Styrene	100-42-5	5	2 5	
	Xylenes (total)	133-02-7	5	2 5	

a. Specific detection limits are highly matrix dependent. The detection limits listed herein are provided for guidance and may not always be achievable.⁸

b. Detection limits listed for soil/sediment are based on wet weight. The detection limits calculated by the laboratory for soil/sediment, calculated on dry weight basis as required by the contract, will be higher.

c. Contract-required detection limits (CRDL) for volatiles at medium levels in soil/sediment are 125 times the listed CRDL for volatiles at low levels in soil/sediment.

The first step in the procedure is sparging the sample. A special teflon sampling head is screwed onto a standard volatile organic (VOA) vial. A teflon tube from this vial goes to a bulkhead fitting on the wall of the glove box wall. The trap is attached on the cold side, outside the glove box and is designed for EPA Method 624.^{4,8} The column contains 3% SP-2100, 15 cm of Tenax GC column, and 8 cm of grade 15 silica gel with the exit end attached to a Rotometer.⁹

Surrogates and spikes are necessary to determine the efficiency for each organic of concern. A Purgeable Surrogate Standards Mix-CLP will be purchased containing 25 ppm each of two selected common aromatic and two chlorinated compounds. Test mixtures containing 25 ppm of 10-15 regulatory compounds from the hazardous substances list called the Purgeable Mix A, B and C standards will be purchased, mixed, and run according to EPA procedures.⁸ The surrogates are run alone and added to samples for the measurements. Blanks are also analyzed to test the residual material deposited on the trap column and within the glovebox.

The trap is desorbed by heating into a GC column that separates the specified chemical components. Detection is by mass spectrometry. Each organic is quantitated by the total signal output or peak area. Identification is by comparative retention time (when it comes out of column), and mass spectrum. The goal of the purge and trap design is to minimize radioactive contamination and perform the analysis on a cold GS/MS. The handling of the small purge vial and all manipulations in the glove box are difficult and require practice. Previous experience shows recoveries of 18-27%. Most compounds of concern for this study show a 30-40% recovery at the 50 ppb level. From recent conferences talks and notices, the problems caused by inorganic components in organic samples have been largely solved.^{1,6,7,9,10,11} Quality assurance for this analysis is performed using standard EPA procedures that require regular calibration and periodic duplicate sample analyses.

Metals

Metal analyses are performed using inductively coupled plasma spectroscopy (ICP), atomic absorption (AA), and direct elemental x-ray fluorescence (XRF). The procedures for these analyses are well defined EPA procedures. Non-destructive elemental analyses can be performed by XRF in the field on both liquid and solid samples and most metals of interest such as Zn, Cr, Fe and Mn are detectable at the 10-100 ppm range. This technique when used in the field is considered a qualitative analysis method that is not usable for quantitative analysis.

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However, both ICP azid AA (quantitative techniques) require dissolution of the sample with mineral acids before introduction of the sample into the instrument. Isolation of the instrument from radioactive contamination is not possible as was done with GC/MS. Consequently a dedicated radioactive instrument in a hood is needed. Analyses of this type are routinely performed at the INEL.

In the AA and ICP procedures, a dissolved sample is sparged into the hot excitation. in the instrument. ICP generates a 6000 deg zone with electrically energized plasma. AA use of flame torch or a furnace to heat the sample. The spectrum from atomic excitation is characteristic of each element and is thus used for identification and the relative excitation is compared with a standard for quantitative analysis. These techniques are total elemental analysis techniques and do not provide information on speciation, oxidation, or the chemical form of the metal. Quality assurance is assured through calibration of the instruments with standards that are traceable to the National Institute of Standards and Technology (NIST).

Organic and Inorganic Anion Analysis

Organic and inorganic anions may be identified and quantified in radioactive aqueous samples containing mixed fission products with Liquid Chromatography (LC). LC is useful for certain organic acids, chelates, surfactants and oxidizing metallic anions. All types of liquid chromatography require a high pressure pump, injection valves, a separation column and detector. For many organics a refractive index, or a u'tra violet/visible (UV/VIS) detector is used.

In this procedure, the dissolved sample is injected onto a column with some solvent or solution carrying it through the column to the detector. The amount of the substance is determined by the peak area measured relative to a standard concentration. The use of this technique for radioactive solid samples requires modification in the sample preparation method to prevent losses of the organic components. In this case the sample would be dissolved in a closed environment and the cover gas trapped for analysis.

The principal technique used for organic ions is ion chromatography, a variant of liquid chromatography. IC requires a high pressure pump, injection valves, ion exchange separation column, a supressor column and conductivity detector. Aqueous samples are injected into the column and separated by ion exchange affinity. Peaks are identified by retention time and

quantified by peak area. The principal ions and detection limits for liquid samples are listed in Table 4. Most of these dissolved anions are found in the power plant waste streams. Those identified as affecting solidification are listed in Table 2. Quality assurance for this technique is performed using standard EPA procedures that require duplicate sample analyses and regular system calibration.

In addition to these specialized instrumental analyses, many general water characteristics are routinely determined by wet chemical methods. Gravimetric, electrode, titrations and visible spectrophotometric techniques are used for critical solidification parameters such as total solids, pH, hardness, oil and grease, and total organic carbon. Standard methods for these are well defined though modifications for medium to high level radioactive samples to minimize individual exposure are not standardized.

CONCLUSIONS

This program plan provides the general methodology to be used to sample waste streams at commercial light water reactors and analyze waste stream samples for organic and inorganic constituents that may affect the solidification of waste forms. Sampling methods at the reactor will be limited by power plant operational requirements and the need to minimize radiation exposure to individuals. It is expected that most waste stream samples will be obtained through the plant sampling system, and that after the solidification agent has been added samples of the slurried waste form will be obtained with the large syringe system now being used to obtain waste form samples.

Prohable components of the waste stream and waste form specimens have been identified along with the analytical methods needed to perform the qualitative and quantitative assessment of these components. Specific sampling plans will be prepared for each commercial reactor to be sampled that identify analyses specific to that plant based plant and vendor information on the waste stream to be characterized. Quality assurance requirements for the identified analysis techniques have been well documented in EPA, ASTM and Standard Method Procedures. These requirements are strictly observed in the laboratories used for these analyses.

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United States Nu leachability of so operating nuclear these waste strear "Technical Positio	the Low-Level Radioactive Waste—Decontamination clear Regulatory Commission (NRC), is to provide blidified waste streams generated in the decontamina power stations. The data include information on the c ns. In addition, this work is intended to evaluate wasto on on Waste Form" (Revision 1), prepared by the Low-1 nical Position clarifies the methods to meet the require	base-line data on the physical stability and ition process of primary coolant systems in hemical composition and characterization of e form characterization tests identified in the level Waste Management Branch of the NRC

this Branch rectained Position clarifies the methods to meet the requirements of 10 CFR Part 61. Samples of LOMI decontamination waste stream resins solidified in Portland cement, and unsolidified waste stream resins were obtained from the Peach Bottom commercial nuclear power station. The radioisotopic composition of the waste stream was determined, and the solidified samples were leached in demineralized and simulated sea water. The compressive strengths of samples immersed for 90 days in the two leachants was determined. In addition to the samples obtained from Peach Bottom, filter sludge waste stream and solidified waste forms were obtained from Nine Mile Point nuclear point waste stream. The radioisotopic composition of the Nine Mile Point waste stream was determined.

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