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TECHNICAL POSITION

ON

WASTE FORM

(Revision 1)

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Technical Position on Waste Form

A. INTRODUCTION

The regulation, "Licensing Requirements for Land Disposal of Radioactive Waste," 10 CFR Part 61, establishes a waste classification system based on the radionuclide concentrations in the wastes. Class B and C waste are required to be stabilized. Class A wastes have lower concentrations and may be segregated without stabilization. Class A wastes may also be stabilized and disposed of with stabilized Class B and C wastes. All Class A liquid wastes, however, require solidification or absorption to meet the free liquid requirements. Structural stability is intended to ensure that the waste does not degrade and (a) promote slumping, collapse, or other failure of the cap or cover over a near-surface disposal trench and thereby lead to water infiltration, or (b) impart a substantial increase in surface area of the waste form that could lead to an increase in leach rate. Stability is also a factor in limiting exposure to an inadvertent intruder since it provides greater assurance that the waste form will remain in a recognizable and nondispersable state. Structural stability of a waste form can be provided by the waste form itself (as with activated stainless steel components), by processing the waste to a stable form (e.g., solidification), or by emplacing the waste in a container or structure that provides stability (e.g., high integrity container or engineered structure).

This technical position on waste form was initially developed in 1983 to provide guidance to both fuel-cycle and non-fuel-cycle waste generators on waste form test methods and results acceptable to the NRC staff for implementing the 10 CFR Part 61 waste form requirements. It has been used as an acceptable approach for demonstrating compliance with the 10 CFR Part 61 waste stability criteria. This position includes guidance on (1) the processing of wastes into an acceptable, stable waste form, (2) the design of acceptable high integrity containers, (3) the packaging of filter cartridges, and (4) minimization of radiation effects on organic ion-exchange resins. The regulation, 10 CFR 20.311, requires waste generators and processors to certify that their waste forms meet the requirements of Part 61 (including the requirements for structural stability). The recommendations and guidance provided in this technical position are an acceptable method upon which to base such certification by waste generators. One way of demonstrating conformance with the general recommendations contained in this technical position is to reference an approved Topical Report, because such reports are reviewed and approved in accordance with the acceptance criteria contained in this technical position. Additional actions (e.g., plant-specific process control procedures) by waste generators, however, to demonstrate that a stabilized plant-specific waste stream satisfies Part 61 waste form requirements, will be needed.

Since the initial issuance of the Technical Position, it has been the intent of the NRC staff to provide additional guidance on waste form as it became necessary to address other pertinent waste form issues. One such issue involves the use of cement to stabilize low-level wastes. Field experience and laboratory testing of cement-solidified low-level radioactive waste has indicated that some unique chemical and physical interactions can occur between the cement constituents and the chemicals and compounds that can exist in the

waste materials. Therefore, an appendix (Appendix "A") dealing with the qualification testing, performance confirmation and reporting of mishaps involving cement-stabilized waste forms has been included in this revision to the Technical Position. (Reporting of mishaps is addressed for other types of waste forms in Section C.6 of the main body of this Technical Position).

To provide more comprehensive guidance on cement stabilization of low-level radioactive waste, Appendix A addresses several areas of concern that were not considered in the May 1983, Revision 0, version of this Technical Position. Thus, information and guidance on cement waste form specimen preparation, statistical sampling and analysis, waste characterization, process control program (PCP) specimen preparation and examination, surveillance specimens and reporting of mishaps are provided in Appendix A. The guidance provided in Appendix A is the culmination of an extended period of study and information gathering and exchange between the NRC staff and representatives of various sectors of the nuclear industry, including government laboratories, cement processing vendors, other waste form vendors, nuclear utilities, state regulatory agencies, and industry representative organizations such as the Nuclear Management Resources Council (NUMARC) and the Electric Power Research Institute (EPRI). Especially useful in the development of the guidance in Appendix A was the information exchanged in a Workshop on Cement Stabilization of Low-Level Radioactive Waste (Ref. 1).

B. BACKGROUND

Historically, waste form and container properties were considered of secondary importance to good site selection; a properly operated site having good geologic and hydrologic characteristics was considered the only barrier necessary to isolate low-level radioactive wastes from the environment. As experience in operating low-level waste disposal sites was acquired, however, it became apparent that the waste form should play a significant role in the overall plan for managing these wastes.

The regulation for near-surface disposal of radioactive wastes, 10 CFR Part 61, includes requirements which must be met by a waste form to be acceptable for near-surface disposal. The regulation includes a waste classification system which divides waste into three general classes: A, B, and C.

The classification system is based on the overall disposal hazards of the wastes. Certain minimum requirements must be met by all wastes. These minimum requirements are presented in Section 61.56(a) and involve basic packaging criteria, prohibitions against the disposal of pyrophoric, explosive, toxic and infectious materials, and requirements to solidify or absorb liquids.

In addition to the minimum requirements, Class B and C wastes are required to have structural stability. As stated in Section 61.56(b) of the rule, stability requires that the waste form maintain its structural integrity under the expected disposal conditions. Structural stability is necessary to inhibit (a) slumping, collapse, or other failure of the disposal unit (if an engineered structure is not used) resulting from degraded wastes which could lead to water infiltration, radionuclide migration, and costly remedial care programs and (b) radionuclide release from the waste form that might ensue due to increases in

leaching that could be caused by premature disintegration of the waste form. Stability is also considered in the intruder pathways where it is assumed that wastes are recognizable after the active control period, and that, therefore, continued inadvertent intrusion would be unlikely. To the extent practical, Class B and C waste forms should maintain gross physical properties and identity over a 300 year period.

To ensure that Class B and C wastes will maintain stability, the following conditions should be met:

- a. The waste should be a solid form or in a container or structure that provides stability after disposal.
- b. The waste should not contain free standing and corrosive liquids. That is, the wastes should contain only trace amounts of drainable liquid, and, as required by 10 CFR 61.56(b)(2), in no case may the volume of free liquid exceed one percent of the waste volume when wastes are disposed of in containers designed to provide stability, or 0.5 percent of the waste volume for solidified wastes.
- c. The waste or container should be resistant to degradation caused by radiation effects.
- d. The waste or container should be resistant to biodegradation.
- e. The waste or container should remain stable under the compressive loads inherent in the disposal environment.
- f. The waste or container should remain stable if exposed to moisture or water after disposal.
- g. The as-generated waste should be compatible with the solidification medium or container.

A large portion of the waste produced in the nuclear industry, including waste from nuclear power plants, is in a form which is either liquid or in a wet solid form (e.g., resins, filter sludge, etc.) and requires processing to achieve an acceptable form for burial. The wet wastes, regardless of their classification, are required to be either absorbed or solidified. To assure that this processing will consistently produce a product which is acceptable for disposal and will meet disposal site license conditions, nuclear power plant licensees are required to process their wastes in accordance with a plant-specific process control program (PCP). Guidance for such PCPs was provided in NRC Standard Review Plan Section 11.4, "Solid Waste Management Systems," NUREG-0800 (Ref. 2) and its accompanying Branch Technical Position ETSB 11-3, "Design Guidance for Solid Waste Management Systems Installed in Light-Water-Cooled Nuclear Power Reactor Plants," (revised in July 1981). However, 10 CFR Part 61 became effective in January 1983, providing requirements regarding waste form, and superseding certain of the guidance previously provided in NUREG-0800. Licensee's PCPs provide assurance that the processing of wet radioactive wastes will result in waste forms that meet the requirements of 10 CFR Part 61 and low-level waste disposal sites licenses.

Plant-specific PCPs developed and approved without consideration of Part 61 should be revised to provide assurance that applicable Part 61 requirements will be satisfied. In many cases, licensee PCPs are based on generally applicable (generic) PCPs contained in vendor-submitted topical reports that are reviewed by the NRC for referencing in licensing actions.

The guidance in this technical position may also serve as the basis for qualifying generic PCPs for Class B and C wastes. Applicable generic test data (e.g., topical reports) may be used for generic PCP qualification, and may be used in part as the basis for a plant-specific PCP. PCPs for solidified Class A waste products that are to be segregated from Class B and C wastes need only demonstrate that the product is a free-standing monolith with no more than 0.5 percent of the waste volume as free liquid.

An alternative to processing some Class B and C waste streams, particularly ion exchange resins and filter sludges, is the use of a high integrity container (HIC). The high integrity container would be used to provide the long-term stability required to meet the structural stability requirements in 10 CFR Part 61. The design of the high integrity container should be based on its specific intended use in order to ensure that the waste contents, as well as interim storage and ultimate disposal environments, will not compromise its integrity over the long-term. As with waste solidification, a PCP for dewatering wet solids in HICs or liners should be developed and utilized to ensure that the free liquid requirements in 10 CFR Part 61 are being met.

C. REGULATORY POSITION

1. Solidified Class A Waste Products

- a. Solidified Class A waste products which are segregated from Class B and C wastes should be free standing monoliths and have no more than 0.5 percent of the waste volume as free liquids as measured using the method described in ANS 55.1 (Ref. 4).
- b. Class A waste products which are not segregated from Class B and C wastes should meet the stability guidance for Class B and C wastes provided below.

2. Stability Guidance for Processed (i.e., Solidified) Class B and C Wastes

The stability guidance in this technical position for processed wastes should be implemented through the qualification of the individual licensee's PCP. Generic test data may be used for qualifying generic PCPs, and incorporated as part of the individual licensee's (i.e., plant-specific) PCP. Tests to demonstrate waste form stability through a generic testing program include the following:

- a. Solidified waste specimens should have compressive strengths of at least 60 psi when tested in accordance with ASTM C39 (Ref. 5). Compressive strength tests for bituminous products should be performed in accordance with ASTM D1074 (Ref. 6).

Many solidification agents (such as cement) will be easily capable of meeting the 60 psi limit for properly solidified wastes. For such cases, process control parameters should be developed to achieve maximum practical compressive strengths, not simply to achieve the minimum acceptable compressive strength; (see Section II.B of Appendix A for further guidance on cement-stabilized wastes).

- b. Waste specimens should be resistant to thermal degradation. The heating and cooling chambers used for the thermal degradation testing should conform to the description given in ASTM B553, Section 3 (Ref. 7). Samples suitable for performing compressive strength tests in accordance with ASTM C39 or ASTM D1074 should be used. Samples should be placed in the test chamber and a series of 30 thermal cycles carried out in accordance with Section 5.4.1 through 5.4.4 of ASTM B553. The high temperature limit should be 60°C and the low temperature limit -40°C. Following testing the waste specimens should have the maximum practical compressive strengths; (a minimum compressive strength of 60 psi as tested using ASTM D1074 is acceptable for bituminized waste forms--for cement-stabilized wastes see Section II.C of Appendix A).
- c. The specimens for each proposed waste stream formulation should remain stable after being exposed in a radiation field equivalent to the maximum level of exposure expected from the proposed wastes to be solidified. Specimens for each proposed waste stream formulation should be exposed to a minimum of 10E+8 Rads in a gamma irradiator or equivalent. If the maximum level of exposure is expected to exceed 10E+8 Rads, testing should be performed at the expected maximum accumulated dose. Following irradiation the irradiated specimens should have the maximum practical compressive strengths (a minimum compressive strength of 60 psi as tested using ASTM D1074 is acceptable for bituminized waste forms--for cement-stabilized wastes see Appendix A).
- d. Specimens for each proposed waste stream formulation should be tested for resistance to biodegradation in accordance with both ASTM G21 and ASTM G22 (Refs. 8 & 9, respectively). No indication of culture growth should be visible. Specimens should be suitable for compression testing in accordance with ASTM C39 or ASTM D1074, as applicable. Following the biodegradation testing, specimens should have the maximum practical compressive strengths (a minimum compressive strength of 60 psi as tested using ASTM D1074 is acceptable for bituminized waste forms--see Section II.E of Appendix A for guidance on biodegradation testing of cement-stabilized wastes).

For polymeric or bitumen products, some visible culture growth from contamination, additives, or biodegradable components on the specimen surface that does not relate to overall substrate integrity

may be present. For these cases, additional testing should be performed. If culture growth is observed upon completion of the biodegradation test for polymeric or bitumen products, the test specimens should be removed from the culture and washed free of all culture and growth with water, with only light scrubbing. An organic solvent compatible with the substrate may be used to extract surface contaminants. The specimen should be air dried at room temperature and the test repeated. Specimens should have observed culture growths rated no greater than 1 in the repeated ASTM G21 test. The specimens should have no observed growth in the repeated ASTM G22 test. Compression testing should be performed in accordance with ASTM C39 or ASTM D1074, as applicable, following the repeated G21 and G22 tests. The minimum acceptable compressive strength for bituminized waste forms is 60 psi. Maximum practical compressive strengths should be established for other media.

If growth is observed following the extraction procedure, longer term testing of at least six months should be performed to determine biodegradation rates. The Bartha-Pramer Method (Ref. 10) is acceptable for this testing. Soils used should be representative of those at disposal facilities. Biodegradation extrapolated for full-size waste forms to 300 years should produce less than a 10 percent loss of the total carbon in the waste form.

- e. Leach testing should be performed for a minimum of 90 days (5 days for cement-stabilized waste forms--see Section II.F of Appendix A for cement-stabilized wastes) in accordance with the procedure in ANS 16.1 (Ref. 11). Specimen sizes should be consistent with the samples prepared for the ASTM C39 or ASTM D1074 compressive strength tests. In addition to the demineralized water test specified in ANS 16.1, additional testing using other leachants specified in the Standard should also be performed to confirm the solidification agents leach resistance in other leachant media. It is preferred that the synthesized sea water leachant also be tested. In addition, it is preferable that radioactive tracers be utilized in performing the leach tests. For proposed nuclear power station waste streams, cobalt, cesium, and strontium should be used as tracers. The leachability index, as calculated in accordance with ANS 16.1, should be greater than 6.0.
- f. Waste specimens should maintain maximum practical compressive strengths as tested using ASTM C39 or ASTM D1074, following immersion for a minimum period of 90 days. Immersion testing may be performed in conjunction with the leach testing; (see Section II.G of Appendix A for guidance on cement-stabilized wastes).
- g. Waste specimens should have less than 0.5 percent by volume of the waste specimen as free liquids as measured using the method described in ANS 55.1. Free liquids should have a pH between 4 and 11; (for cement-solidified water, free liquids should have a minimum pH of 9--see Section II.H of Appendix A).

- h. If small, simulated laboratory size specimens are used for the above testing, test data from sections or cores of the anticipated full-scale products should be obtained to correlate the characteristics of actual size products with those of simulated laboratory size specimens. This testing may be performed on non-radioactive specimens. Correlation testing should be performed using 90-day immersion (including post-immersion compression) tests on the most conservative waste stream(s) intended for use for the particular solidification medium; i.e., the waste stream that presents the most difficulty in consistently producing a stable product(s). For cement-solidified waste forms, the mixed bead resin waste stream is expected to be the most conservative. For bituminized wastes, the sodium sulfate waste stream should be used. The full-scale specimens should be fabricated using solidification equipment the same as or comparable to that used for processing actual low-level radioactive wastes in the field.
- i. Waste samples from full-scale specimens should be destructively analyzed to ensure that the product produced is homogeneous to the extent that all regions in the product can expect to have compressive strengths representative of the compressive strength as determined by testing lab-scale specimens (i.e., that meet the criteria called out in Section C2.a. above). Full-scale specimens may be fabricated using simulated non-radioactive products; however, the specimens should be fabricated using solidification equipment that is the same as or comparable to that used in the field for actual low-level radioactive wastes.

3. Radiation Stability of Organic Ion-Exchange Resins

To ensure that organic ion exchange resins will not undergo adverse degradation effects from radiation, resins should not be generated having loadings that will produce greater than $10E+8$ Rads total accumulated dose. For Cs-137 and Sr-90 a total accumulated dose of $10E+8$ Rads is approximately equivalent to a 10 Ci/ft concentration in resins in the unsolidified, as-generated form. In the event that the waste generator considers it necessary to load resins higher than $10E+8$ Rads, it should be demonstrated that the specific resin will not undergo radiation degradation at the proposed higher loading. The test method should adequately simulate the chemical and radiologic conditions expected. A gamma irradiator or equivalent should be utilized for these tests. There should be no adverse swelling, acid formation or gas generation that will be detrimental to the proposed final waste product.

4. High Integrity Containers

- a. The maximum allowable free liquid in a high integrity container should be less than one percent of the waste volume as measured using the method described in ANS 55.1. A process control program

should be developed and qualified to ensure that the free liquid requirements in 10 CFR Part 61 will be met upon delivery of the wet solid material to the disposal facility. This process control program qualification should consider the effects of transportation on the amount of drainable liquid which might be present.

- b. High integrity containers should have as a design goal a minimum lifetime of 300 years. The high integrity container should be designed to maintain its structural integrity over this period.
- c. The high integrity container design should consider the corrosive and chemical effects of both the waste contents and the disposal environment. Corrosion and chemical tests should be performed to confirm the suitability of the proposed container materials to meet the design lifetime goal.
- d. The high integrity container should be designed to have sufficient mechanical strength to withstand horizontal and vertical loads on the container equivalent to the depth of proposed burial assuming a cover material density of 120 lbs/ft³. The high integrity container should also be designed to withstand the routine loads and effects from the waste contents, waste preparation, transportation, handling, and disposal site operations, such as trench compaction procedures. This mechanical design strength should be justified by conservative design analyses.
- e. For polymeric material, design mechanical strengths should be conservatively extrapolated from creep test data. It should be demonstrated for high integrity containers fabricated from polymeric materials that the containers will not undergo tertiary creep, creep buckling, or ductile-to-brittle failure over the design life of the containers.
- f. The design should consider the thermal loads from processing, storage, transportation and burial. Proposed container materials should be tested in accordance with ASTM B553 in the manner described in Section C2(b) of this technical position. No significant changes in material design properties should result from this thermal cycling.
- g. The high integrity container design should consider the radiation stability of the proposed container materials as well as the radiation degradation effects of the wastes. Radiation degradation testing should be performed on proposed container materials using a gamma irradiator or equivalent. No significant changes in material design properties should result following exposure to a total accumulated dose of 10 E+8 Rads. If it is proposed to design the

high integrity container to greater accumulated doses, testing should be performed to confirm the adequacy of the proposed materials. Test specimens should be prepared using the proposed fabrication techniques.

High integrity container designs using polymeric materials should also consider the effects of ultra-violet radiation. Testing should be performed on proposed materials to show that no significant changes in material design properties occur following expected ultra-violet radiation exposure.

- h. The high integrity container design should consider the biodegradation properties of the proposed materials and any biodegradation of wastes and disposal media. Biodegradation testing should be performed on proposed container materials in accordance with ASTM G21 and ASTM G22. No indication of culture growth should be visible. The extraction procedure described in Section C2(d) of this technical position may be performed where indications of visible culture growth can be attributable to contamination, additives, or biodegradable components on the specimen surface that do not affect the overall integrity of the substrate. It is also acceptable to determine biodegradation rates using the Bartha-Pramer Method described in Section C2(d). The rate of biodegradation should produce less than a 10 percent loss of the total carbon in the container material after 300 years. Test specimens should be prepared using the proposed material fabrication techniques.
- i. The high integrity container should be capable of meeting the requirements for a Type A package as specified in 49 CFR 173.411 and 173.412. Conditions that may be encountered during transport or movement are to be addressed by meeting the requirements of 10 CFR 71.71. j. The high integrity container and the associated lifting devices should be designed to withstand the forces applied during lifting operations. As a minimum the container should be designed to withstand a 3g vertical lifting load.
- k. The high integrity container should be designed to avoid the collection or retention of water on its top surfaces in order to minimize accumulation of trench liquids which could result in corrosive or degrading chemical effects.
- l. High integrity container closures should be designed to provide a positive seal for the design lifetime of the container. The closure should also be designed to allow inspections of the contents to be conducted without damaging the integrity of the container. Passive vent designs may be utilized if needed to relieve internal pressure. Passive vent systems should be designed to minimize the entry of moisture and the passage of waste materials from the container.

- m. Prototype testing should be performed on high integrity container designs to demonstrate the container's ability to withstand the proposed conditions of waste preparation, handling, transportation and disposal.
- n. High integrity containers should be designed, fabricated, and used in accordance with a quality assurance program. The quality assurance program should address the following topics concerning the high integrity container: fabrication, testing, inspection, preparation for use, filling, storage, handling, transportation, and disposal. The quality assurance program should also address how wastes which are detrimental to high integrity container materials will be precluded from being placed into the container. Special emphasis should be placed on fabrication process control for those high integrity containers which utilize fabrication techniques such as polymer molding processes.

5. Filter Cartridge Wastes

For Class B and C wastes in the form of filter cartridges, the waste generator should demonstrate that the selected approach for providing stability will meet the requirements in 10 CFR Part 61. Encapsulation of the filter cartridge in a solidification binder or the use of a high integrity container are acceptable options for providing stability. When high integrity containers are used, waste generators should demonstrate that protective means are provided to preclude container damage during packaging handling and transportation.

6. Reporting of Mishaps

In all future reviews and approvals of stabilization media and high integrity containers, waste generators, vendors and processors will, as a condition of approval, be asked to commit to reporting any knowledge they may have of misuse or failure of their waste forms and containers. Such mishaps include, but are not necessarily limited to, the following:

- a. The failure of high integrity containers used to ensure structural stability. Such failure may be evidenced by changed container dimensions, cracking, or injury from mishandling (e.g., dropping or impacting against another object).
- b. The misuse of high integrity containers, as evidenced by a quantity of free liquid greater than one percent of container volume, or an excessive void space within the container; (such use is in violation of 10 CFR 61.56(a)).
- c. The production of a solidified Class B or C waste form that has any of the following characteristics:
 - 1. greater than 0.5 percent volume of free liquid.

2. concentrations of radionuclides greater than the concentrations demonstrated to be stable in the waste form in qualification testing accepted by the regulatory agency.
3. greater or lesser amounts of solidification media than were used in qualification testing accepted by the regulatory agency.
4. contains chemical ingredients not present or accounted in qualification testing accepted by the regulatory agency.
5. shows instability evidenced by crumbling, cracking, spalling, voids, softening, disintegration, nonhomogeneity, or change in dimensions.
6. evidences processing phenomena that exceed the limiting processing conditions identified in applicable topical reports or process control programs, such as foaming, excessive temperature, premature or slow hardening, production of volatile material, etc.

Waste form mishaps should be reported to the NRC's Director of the Division of Low-Level Waste Management and Decommissioning and the designated State disposal site regulatory authority within 30 days of knowledge of the incident. For any such waste form mishap occurrence, the affected waste form should not be shipped off-site until approval is obtained from the disposal site regulatory authority. The reason for this is that the low-level waste generators and processors are required by 10 CFR 20.311 to certify that their waste forms meet all applicable requirements of 10 CFR Part 61, and waste forms that are subject to the types of mishaps mentioned above may not possess the required long-term structural stability. When mishaps of the nature described above occur, it is expected that, before the waste form is shipped to a disposal facility, either adequate mitigation of the potential effects on the waste form or an acceptable justification concerning the lack of any potential significant effects of the affected waste form on the overall performance of the disposal facility would be provided.

D. IMPLEMENTATION

This technical position reflects the current NRC staff position on acceptable means for meeting the 10 CFR Part 61 waste stability requirements. Therefore, except in those cases in which the waste generator, vendor, and/or processor proposes an acceptable alternative method for complying with the stability requirements of 10 CFR Part 61, the guidance described herein will be used in the evaluation of the acceptability of waste forms for disposal at near-surface disposal facilities.

E. REFERENCES

1. "Proceedings of the Workshop on Cement Stabilization of Low-Level Radioactive Waste," NUREG/CP-0103, October 1989.
2. "Standard Review Plan for the Review of Safety Analysis Reports for Nuclear Power Plants," (LWR Edition), NUREG-0800, July, 1981.
3. "Update on Waste Form and High Integrity Container Topical Report Review Status, Identification of Problems with Cement Solidification, and Reporting of Waste Mishaps," NRC Information Notice No. 90-xx, (in preparation).
4. ANS 55.1, "American National Standard for Solid Radioactive Waste Processing System for Light Water Cooled Reactor Plants," American Nuclear Society, 1979.
5. ASTM C39, "Compressive Strength of Cylindrical Concrete Specimens," American Society for Testing and Materials, 1979.
6. ASTM D1074, "Compression Strength of Bituminous Mixtures," American Society for Testing and Materials, 1980. 7. ASTM B553, "Thermal Cycling of Electroplated Plastics," American Society for Testing and Materials, 1979.
8. ASTM G21, "Determining Resistance of Synthetic Polymeric Materials to Fungi," American Society for Testing and Materials, 1970.
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10. R. Bartha, D. Pramer, "Features of a Flask and Method for Measuring the Persistence and Biological Effects of Pesticides in Soils," Soil Science 100 (1), pp. 68-70, 1965.
11. ANS 16.1, "Measurement of the Leachability of Solidified Low-Level Radioactive Wastes," American Nuclear Society Draft Standard, April 1981.

Appendix A

Cement Stabilization

I. INTRODUCTION

This Appendix to the Technical Position on Waste Form provides guidance to waste generators and processors who intend to use cementitious materials such as Portland and pozzolonic-type cements to solidify and stabilize low-level radioactive wastes in accordance with the requirements of 10 CFR Part 61 (Ref. A1(a)). This guidance is applicable for cementitious waste forms destined for disposal in shallow-land disposal sites and engineered structures where the regulatory authorities require stable waste forms. It is expected that the guidance described herein would be used by NRC staff in any Topical Report evaluation of the acceptability of cement waste forms for disposal at near-surface disposal facilities. Waste generators using cement solidification systems and media not approved generically through the Topical Report review process may use this guidance to conduct testing to demonstrate that waste forms satisfy the requirements of Part 61. NRC regulation 10 CFR 20.311 (Ref. A1(b)) requires waste generators to certify that their waste forms meet the requirements of Part 61 (including the requirements for structural stability). Waste generators whose cement waste formulations meet the provisions of this Technical Position will be able to certify that the formulations meet the requirements of Part 61. The disposal site regulatory authorities, however, have the ultimate responsibility for accepting or rejecting the waste.

Portland and pozzolonic cements have been observed to exhibit unique chemical and physical interactive behavior when used with certain materials and chemicals encountered in some low-level radioactive waste streams. Therefore, this Appendix specifically addresses cement waste form qualification only and is not intended to be applied generically to all stabilization agents (although many of the provisions discussed are, in principle, applicable to other media). This Appendix thus complements, and does not replace, the main body of the Technical Position on Waste Form.

Included in this Appendix are descriptions of methods that may be used in cement waste form qualification testing. Associated acceptance criteria that may be used by NRC staff or others to evaluate the acceptability of the test results are also provided. Included in this waste form testing guidance are descriptions of acceptable procedures for sample preparation and statistical treatment of data. In addition, this Appendix provides guidance on waste stream characterization, process control program (PCP) recipe qualification and specimen examination, surveillance specimen preparation and testing, and procedures for reporting of cement waste form preparation mishaps. This guidance on cement waste forms is intended to provide the best available information on an acceptable approach for demonstrating that a cement-solidified low-level radioactive waste form will possess the long-term (300-year) structural stability that is required by Part 61 for Class B and Class C wastes.

Linkage between the waste form qualification test recommendations in this Technical Position and the requirements of Part 61 is provided in 10 CFR 61.56(b)(1), where it is stated that "a structurally stable waste form will generally maintain its physical dimensions and form, under the expected disposal conditions such as weight of overburden and compaction equipment, the presence of moisture and microbial activity, and internal factors such as radiation effects and chemical changes." The discussion provided in Section II of this Appendix addresses the details of the test procedures and acceptance criteria recommended for cement-stabilized wastes. Further information on test specimen preparation and analysis of data is provided in Section III and Section IV, respectively.

II. WASTE FORM QUALIFICATION TESTING

A. General

As indicated in Section C.2 of the main body of this Technical Position, generic test data may be used "for qualifying process control programs." That is, a low-level radioactive waste generator/processor may perform qualification testing, as described in the following subsections of this Appendix, to qualify recipes for a range of waste compositions (concentrations and loadings) for a given type of waste stream. It is incumbent upon the party providing 10 CFR 20.311 certification, however, to show that the composition(s) of the waste form specimens used in the qualification testing adequately covers the range of waste compositions that will be encountered in the field. An acceptable approach to qualification testing is to perform the tests not only at the maximum waste loading but also at lower loadings (at least one), with appropriate variations in water/cement ratios and proportions of additives. It should not be necessary to perform all the qualification tests for all of the waste loadings, but adequate justifications should be provided for any omissions.

Each individual waste stream should be qualified with test data obtained for that specific waste stream. In cases where two or more waste streams are combined, it should be demonstrated that the specimen compositions used in the qualification testing adequately cover the range of compositions that are intended to be stabilized in the field. This may be accomplished by performing the full series of qualification tests on the "worst-case" composition only, along with one or more tests on alternate compositions, sufficient to show that the selected "worst-case" was chosen correctly.

B. Compression

It is stated in 10 CFR 61.56(b)(1) that "a structurally stable waste form will generally maintain its physical dimensions and form under expected disposal conditions such as weight of overburden and compaction equipment...." Assuming a cover material density of 120 lbs./cu.ft., a minimum compressive strength criterion of 50 psi was established in section C.2.b. of the 1983 Revision 0 portion of this Technical Position. To reflect the increase in burial depth (from 45 to 55 feet) at Hanford, Washington, the minimum compressive strength criterion for generic waste forms was later increased from 50 to 60 psi.

However, as further noted in the above-cited section C.2.a., for solidification agents that are easily capable of meeting the 50 (now 60) psi minimum compressive strength, the waste forms should achieve "maximum practical compressive strengths," not just the "minimum acceptable compressive strength." This provision was included in the Rev. 0, 1983 Technical Position in recognition of the fact that mere resistance to deformation under burial loads is, in itself, inadequate evidence that the waste form microconstituents are bonded together sufficiently well to ensure that the waste form will not over time fall apart due to internal stresses that are chemically, physically, or irradiation induced.

Portland cement mortars, which are comprised of mixtures of cement, lime, silica sand and water, are readily capable of achieving compressive strengths of 5000 to 6000 psi; that is approximately two orders of magnitude greater than the minimum compressive strength required to resist deformation under load in current low-level waste burial trenches. Therefore, to provide greater assurance that there will be sufficient cementitious material present in the waste form to not only withstand the burial loads, but also to maintain general "dimensions and form" (i.e., to not disintegrate) over time, it is recommended that cement-stabilized waste forms possess compressive strengths that are representative of the values that are reasonably achievable with current cement solidification processes. Taking into consideration the fact that low-level radioactive waste material constituents are not in most cases capable of providing the physical and chemical functions of silica sand in a cement mortar, a mean compressive strength equal to or greater than 500 psi is recommended for waste form specimens cured for a minimum of 28 days (see Section III.B of Appendix A). This value of compressive strength is recommended as a practical strength value that is representative of the quality of cementitious material that should be used in the waste form to provide assurance that it will maintain integrity and thus possess the long term structural capability required by Part 61.

Compressive strengths of cement-stabilized waste forms should be determined in accordance with procedures described in ASTM Standard C39: Compressive Strength of Cylindrical Concrete Specimens (Ref. A2). It is recommended that the compressive strength test specimens be right circular cylinders, 2 to 3 inches in diameter, with a length-to-diameter (L/D) ratio of approximately two. Because hydrated cement solids are brittle ceramic materials that fail in tension or shear rather than compression, and at regions of localized stress concentration or microstructural flaw, there tends to be considerable scatter in the strength test data even if all processing variables are kept relatively constant. Therefore, sufficient specimens should be tested to determine the mean compressive strength and standard deviation. Because of the many variables involved, a decision regarding the specific number of specimens to be tested is left to the judgement of the waste processor/qualifier; in no case, however, should the number of as-cured (pre-environmental test) compressive strength test specimens be less than ten. This approach should continue until there are sufficient data available to permit judgements to be made regarding what is reasonably achievable, from a statistical standpoint, in compressive strength testing of low-level waste test specimens. No precision criterion, in the form of an acceptable variance or standard deviation, is recommended at this time.

[For the purposes of verification of Process Control Program (PCP) parameters (see discussion in Section VI of Appendix A), compressive strength tests and/or penetrometer hardness tests should be performed after the qualification test specimens have been allowed to cure for approximately 24 hours. The results of these tests should be retained and made available for comparison with the results of similar tests that should be performed on PCP specimens fabricated from actual radioactive wastes in the field; (see Appendix A, Section VI.C for details).]

C. Thermal Cycling

Though thermal effects are not called out specifically as an item of concern in 10 CFR 61.56(b)(1), as other factors are, cement-stabilized low-level radioactive waste forms should be demonstrated to be resistant to thermal degradation. There are three basic reasons for this: (1) Section 61.56(b)(1) of Part 61 lists "internal factors" as a condition that must be considered in assuring that a waste form will retain structural stability, and temperature and thermal effects are internal factors; (2) thermal cycling of the waste form will occur, particularly during the storage and transport phase of the waste form's performance "life;" and (3), experience has shown that the thermal cycling test has served well in distinguishing between "strong" and "weak" solidified waste forms. The thermal cycling test imposes a stress (due to differential thermal expansion) between the various microconstituents of the waste form and between different regions of the waste form. By cycling between the maximum and minimum temperatures called for in the test, any cracks initiated in the test specimen may propagate and eventually measurably weaken the waste form. The extent of any degradation that might occur will be a function of various factors such as the amount of cementitious material in the waste form, the bond strength between the materials present, and the morphology of the microconstituents in the waste form microstructure. Thus, the thermal cycling test, by subjecting the waste form specimens to a short-term cyclic thermal stress, challenges the structural capability of the specimens and thus serves as a very useful vehicle for screening out unfavorable "weak" formulations.

The heating and cooling chambers used in determining the thermal cycling resistance of cement-stabilized waste forms should, as stated in Section C.2.b. of the main body of this Technical Position, conform to the description given in ASTM Standard Test Method B553 (Ref. A3). However, because that test method addresses thermal cycling of electroplated plastics, not cement-solidified waste materials, some modifications to the test procedure are necessary. Test specimens suitable for performing compressive strength tests in accordance with ASTM C39 should be used. The specimens should be tested "bare;" i.e., not in a container. Specimens should be placed in the test chamber, and a series of 30 thermal cycles should be carried out in accordance with Section 5.4.1 through 5.4.4 of ASTM B553, with the additional proviso that the specimens should be allowed to come to thermal equilibrium at the high (50 degrees C) and low (-40 degrees C) temperature limits. Thermal equilibrium should be confirmed by measurements of the center temperature of at least one specimen (per test group). A minimum of three specimens for each waste formulation should be subjected to the thermal cycling tests.

Following exposure to 30 thermal cycles the specimens should be examined visually and should be free of any evidence of significant cracking, spalling, or bulk disintegration; i.e., visible evidence of significant degradation would be indicative of failure of the test. Because it is not possible to provide an a priori assessment of the significance of visible defects, taking into consideration the wide range of possible defect configurations, no definition of "significant degradation" is provided here. The organization performing the tests should (1) assess whether visible defects are significant, and (2) obtain and retain photographic evidence of any defects that are judged to be insignificant for future reference. If there are no significant visible defects, the test specimens should be subjected to compression strength testing in accordance with ASTM C39 and should have mean compressive strengths that are equal to or greater than 500 psi.

D. Irradiation

In accordance with the requirements of 10 CFR 61.56(b)(1), and as indicated in Section C.2.c. of the main body of this Technical Position, irradiation testing of solidified waste forms should be conducted on specimens exposed to a minimum dose of $10E+8$ rads. The $10E+8$ rads radiation dose is approximately equivalent to the dose that would be acquired by a waste form over a 300-year period, if the waste form were loaded to a Cesium-137 or Strontium-90 concentration of 10 Ci/cu.ft. This is the recommended (Ref. A3) maximum activity level for organic resins based on evidence that while a measurable amount of damage to the resin will occur at $10E+8$ rads, the amount of damage will have negligible effect on power plant or disposal site safety. However, cementitious materials are not affected by gamma radiation to relatively high cumulative doses (e.g., greater than $10E+9$ rads--Ref. A4) considerably in excess of $10E+8$ rads. Therefore, for cement-stabilized waste forms, irradiation qualification testing need not be conducted unless (1) the waste forms contain ion exchange resins or other organic media or (2) the expected cumulative dose on waste forms containing other materials is greater than $10E+9$ rads. Testing should be performed on specimens exposed to (1) $10E+8$ rads or the expected maximum dose greater than $10E+8$ rads for waste forms that contain ion exchange resins or other organic media or (2) the expected maximum dose greater than $10E+9$ rads for other waste forms. In cases where irradiation testing is warranted, a minimum of three specimens should be tested for each waste formulation being qualified.

Following the irradiation exposure the specimens should be examined visually and should be free of any evidence of significant cracking, spalling, or bulk disintegration; i.e., visible evidence of significant degradation would be indicative of failure of the irradiation test. If there are no significant visible defects (see Section II.C for discussion of "significant degradation"), the test specimens should be subjected to compressive strength testing in accordance with ASTM C39 and should have mean compressive strengths that are equal to or greater than 500 psi.

E. Biodegradation

As indicated in 10 CFR 61.56(b)(1), a structurally stable waste form is one that will be relatively unaffected by "microbial activity." Generic (not specific to type of waste form) recommendations for biodegradation testing provided in Section C.2.e. of the main body of this Technical Position indicate that ASTM Standard Practice G21 (Ref. A5) and G22 (Ref. A6) are suitable methods of test for determining susceptibility to fungi and bacteria, respectively. Experience in biodegradation testing of cement-stabilized waste forms has shown (Refs. A7-A9), however, that they generally do not support fungal or bacterial growth. The principal reason for this appears to be that the fungi and microbes used in the G21 and G22 tests require a source of carbon for growth, and in the absence of any carbonaceous materials in the waste stream, there is no internal food source available for culture growth. Consequently, for cement-stabilized waste forms, biodegradation qualification testing need not be conducted unless the waste forms contain carbonaceous materials (e.g., ion exchange resins or oils).

For cement-stabilized waste forms containing carbonaceous materials, there should be no evidence of culture growth during the G21 and G22 tests. The test specimens (at least three for each organic waste stream formulation being qualified) should also be free of any evidence of significant cracking, spalling or bulk disintegration; i.e., visible evidence of significant degradation would be indicative of failure of the test. If there are no significant visible defects following the test exposures (see Section II.C of this Appendix for discussion of "significant degradation"), the test specimens should be subjected to compression strength testing in accordance with ASTM C39 and should be shown to have mean compressive strengths equal to or greater than 500 psi.

F. Leach Testing

Resistance to leaching of radionuclides is not specifically mentioned in Part 61, nor is radionuclide containment called out as a specific requirement for low-level waste packages. Minimization of contact of waste by water is a fundamental concern of Part 61, however, as evidenced by the statement in Section 61.7 that "...a cornerstone of the program is stability...so that... access of water to the waste can be minimized (emphasis added). Migration of radionuclides is thus minimized..." In addition, there are several statements in Section 61.51 that address minimization of contact of water with waste. These statements are in recognition of the fact that contact of waste with water is the first step in a potentially major pathway for radionuclide release and migration off-site. Thus, "leaching," or release of radionuclides from a waste form through contact with water is a first step in subsequent migration of the radionuclides from the waste through the groundwater and off the site. Therefore, leaching is a phenomenon that is of fundamental interest in waste disposal.

The leach testing procedure specified in Section C.2.e. of the main body of this Technical Position is ANSI/ANS 16.1: Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure (Ref. A10). In the ANSI/ANS 16.1 test, a test specimen is completely immersed in a measured volume of water, which is changed on a prescribed schedule. Upon removal, the leachant is analyzed for the radionuclides (or elements) of interest. The data obtained by this procedure are expressed as a material parameter of the leachability of each leached species. This parameter is called the "Leachability Index" (L), which is the arithmetic mean of the L values obtained for each leaching interval (where the L value is the logarithm of the inverse of the effective diffusivity). The leachability index, as calculated in accordance with ANSI/ANS 16.1, should be greater than 6.0.

The period of time specified for the leach test in the above-cited Section C.2.e. of this Technical Position is a minimum of 90 days, and the test period called out in the Standard corresponds to 90 days. This time period was selected as a means of determining whether there might be a change in leach mechanism with time; (as explained in the Standard, early leach rates observed with solidified waste forms are most often explained by diffusion--other mechanisms, such as erosion, dissolution, or corrosion, would generally be discernible only after longer leaching times). However, any leaching that involves other mechanisms such as erosion, dissolution, corrosion or other chemical or physical phenomena would most likely be readily observed visually and through mechanical testing. Such observations would be made as part of the immersion test, which is a 90-day test. These facts, coupled with comparisons of 5-day and 90-day data (Ref. A11) on cement waste forms that showed that the percentage differences between 5-day and 90-day leach indices were relatively small for most specimens, indicate that a 5-day leach testing period is sufficient for cement-solidified wastes.

The leachant specified in ANSI/ANS 16.1 is deionized water. It is stated in the above-cited Section C.2.e. of this Technical Position that additional testing using other leachants should also be performed to confirm the solidification agents leach resistance in other leachant media. Synthesized sea water leachant is listed as a preferred alternate leachant. The basis for this is, that while leachability indices are generally lower (i.e., leach rates are higher) for tests conducted in demineralized water than in sea water (Ref. A11), this is not true in all cases for all waste streams. For reasons of economy, however, it is desirable to limit the bulk of the testing to one leachant. If it can be shown that the chosen leachant is the most aggressive one, testing with one leachant is appropriate. Since it is not possible to initially predict (Ref. A9) which leachant (deionized water or synthesized sea water) would be most aggressive, sufficient preliminary testing should be conducted to identify the most aggressive leachant for each waste form formulation being qualified, and that leachant should be used for the balance of the testing (if only one is used). An acceptable method of identifying the most aggressive leachant is to perform 24 hour (or longer) leaching measurements on both leachants and to use the leachant that resulted in the lowest leach indices (i.e., highest leach rate) for the remaining days of testing.

G. Immersion Testing

No "Standard Method of Test" for immersion testing has been adopted for low-level radioactive waste, but as indicated in Section C.2.f. of the main body of this Technical Position, immersion testing may be performed in conjunction with the leach testing (which is to be performed in accordance with ANSI/ANS 16.1). However, in contrast with the period of time (5 days) necessary for leach testing of cement-stabilized wastes, immersion testing should be performed for a minimum period of 90 days. The immersion testing should be performed in either deionized water or synthesized sea water. The immersion liquid should be selected on the basis of short-term (24-hour or longer) leach tests that identify the most aggressive immersion medium (see discussion of leach testing).

The test specimens (at least three for each waste stream formulation being qualified) should be cured for a minimum cure time of 28 days (see Section III, "Specimen Preparation," of Appendix A for details) prior to being immersed. Following immersion, the specimens should be examined visually and should be free of any evidence of significant cracking, spalling, or bulk disintegration. If there are no significant visible defects (see Section II.C of this Appendix for discussion of "significant degradation"), the specimens should be subjected to compressive strength testing in accordance with ASTM C39 and should have post-immersion mean compressive strengths that are equal to or greater than 500 psi and not less than 75 percent of the pre-immersion test (i.e., as-cured) mean compressive strength. If the post-immersion mean compressive strength is less than 75 percent of the as-cured specimens' pre-immersion mean compressive strength, (but not less than 500 psi) the immersion testing interval should be extended (using additional specimens) to a minimum of 180 days. For these cases, sufficient compressive strength testing should be conducted (for example, after 120, 150, and 180 days of immersion) to establish that the compressive strengths level off and do not continue to decline with time.

For certain waste streams (viz., bead resins, chelates, filter sludges, and floor drain wastes) that have been found to exhibit complex relationships of cure time and immersion resistance (Ref. A12), additional immersion testing should be performed on specimens that have been cured (in sealed containers) for a minimum of 180 days. The immersion period should be for a minimum of 7 days, followed by a drying period of 7 days in ambient air at a minimum temperature of 20 degrees Celsius. After the specimens are dried, they should meet the post-immersion test visual and compressive strength criteria specified above.

Free Standing Liquids

It is stated in 10 CFR 61.56(b)(2) that "...liquid wastes, or wastes containing liquid, must be converted into a form that contains as little free standing or noncorrosive liquid as is reasonably achievable, but in no case shall the liquid exceed...0.5% of the volume of the waste for waste processed to a stable form." Correspondingly, waste test specimens should have less than 0.5 percent by volume of the waste specimen volume as free liquids as measured using the method described in Appendix 2 of ANSI/ANS 55.1 (Ref. A13). Inasmuch as cement

is an alkaline material, evidence of acidic free liquids is indicative of improper waste form preparation or curing. Therefore, any free liquid from cement-stabilized waste forms should have a minimum pH of 9.

I. Full-scale Testing

It is expected that the testing performed in accordance with the guidance provided in Sections A through H above will be carried out on small, laboratory scale specimens. As indicated in Section C.2.h. of the main body of this Technical Position, therefore, it is necessary to correlate the characteristics of full-size products with those of laboratory size specimens. The full-scale specimens should be fabricated using solidification equipment that is the same as or comparable to that used in processing real low-level waste forms in the field. The correlation of full-scale product characteristics should be accomplished by performing (1) compressive strength tests on as-cured material (cured for a minimum of 28 days), and (2) 90-day immersion tests that include post-immersion compressive strength tests (See Section II.G above) for the most conservative waste stream(s) being qualified.

Test specimens obtained from the full-scale waste forms by coring or sectioning should be destructively analyzed to ensure that the product produced is homogeneous to the extent that all regions in the product can be expected to have compressive strengths that meet the criteria called out in Section II.B above.

III. QUALIFICATION TEST SPECIMEN PREPARATION

A. Mixing

Experience in preparation of lab-scale and full-scale cement-solidified waste forms (Ref. A9) has shown that the method employed in mixing the ingredients can have a dramatic influence on the reactivity of the materials, the structure of the solidified waste form, and the resultant properties and characteristics of the waste form. Important parameters include type of equipment and mixing time because they will determine the amount of energy imparted to the ingredients used in the solidification recipe. This is especially important in cases where properties and characteristics of small, lab-scale specimens are used to predict the behavior of large, full-scale products. In preparing laboratory-sized qualification test specimens, it should be shown by analysis and/or testing that the type of equipment used, the mixing time, the speed of the mixer, etc. will, in combination, impart the same degree of mixing to the laboratory specimens as the full-scale mixing equipment and procedure will impart to full-scale waste forms and that the degree of mixing is sufficient to ensure production of homogeneous waste forms.

B. Curing

The curing conditions for small, laboratory-scale qualification test specimens, should, to the extent practical, be the same as the conditions obtained with full-scale products. Inasmuch as cement constituents exhibit a significant exothermic heat of hydration, while possessing low thermal conductivity, the interior temperature of large, full-scale cement waste forms may be elevated

significantly (approaching even the boiling point of water). To ensure that the laboratory specimens endure curing conditions that are reasonably similar to those of full-size products, the waste form centerline temperature profile as a function of time should be obtained for the largest full-sized waste form to be qualified for each waste stream. That profile should be duplicated, to the extent practical, in the laboratory specimens. An acceptable method is to cure the specimens in a suitable oven for a period of time equivalent to the peak heat of hydration period. For the purposes of this Technical Position that period of time is taken to be that required for the centerline temperature of a full-scale waste form to decrease to a near-ambient (30 degrees Celsius or lower) temperature level.

Care should be taken to ensure that the waste loadings and cement concentrations in the full-scale waste forms provide sufficient margin to preclude reaching the boiling point of the pre-solidification mix. This is necessary to ensure that the waste form formulations will not be subject to uncontrolled variations due to water losses caused by evaporation during set. Uncontrolled porosities due to vapor bubble formation and rapid set due to elevated temperatures will also be avoided by limiting the maximum temperatures in the cement-solidified waste forms.

The compressive strength of hydrated cement and concrete solids increases asymptotically as the mixtures cure. Normally, the strength at 28 days approaches seventy-five percent or more of the "peak" value, though when pozzolonic cements are used the time required to reach peak strength may be extended. Sufficient test specimens should be prepared to determine the compressive strength increase with time to ensure that the specimens have attained sufficient (i.e., greater than 75% of the projected peak) strength prior to subjecting the remaining specimens to the qualification testing called out in Sections II.C through II.G. of this Appendix.

C. Storage

Test specimens that will be subjected to the qualification testing described in Section II of this Appendix should be kept in sealed containers during curing and storage. This is intended to simulate the environment that would be obtained in a typical full-scale waste form liner and will prevent loss of water that might affect the performance of the waste form specimens during subsequent testing.

IV. STATISTICAL SAMPLING AND ANALYSIS

As noted in the discussion of compressive strength testing (see Section II.B above), there tends to be considerable scatter in the compressive strength data obtained on brittle ceramic materials such as cement. Therefore, sufficient specimens should be tested in the as-cured condition to provide enough data to establish a mean and standard deviation, though for reasons discussed in Appendix A Section II.B, the number of as-cured specimens to be tested is left to the judgement of the waste formulation qualifier. For statistical purposes, however, the number of as-cured (pre-environmental test) compressive strength specimens should be ten or greater for a given formulation. Further discussion

of the rationale for this provision is provided in Section II.B of this Appendix. For the minimum quantities of test specimens recommended in the respective subsections of this Appendix, the specimens tested should have a post-test mean compressive strength that is equal to or greater than 500 psi. Note that for the immersion tests, a slightly different acceptance criterion is identified, in subsection II.G of this Appendix. Variations in individual specimen compression strength need not be considered.

Other than the determinations of compressive strength, the only other parameter of interest in qualification testing of low-level waste forms that lends itself to statistical treatment is the leachability index. ANSI/ANS 16.1 (Ref. A10) uses the confidence range and correlation coefficient as measures of discrepancies in the measurements of leachability. The Standard requires that the confidence range and correlation coefficient be reported with the Leachability Index. As is the case of the ASTM C39 Compressive Strength standard, however, no precision criterion has been established yet for the ANSI/ANS 16.1 leach test.

V. WASTE CHARACTERIZATION

The importance of waste characterization was extensively discussed at the May/June Workshop on Cement Stabilization of Low-Level Radioactive Waste that was held in Gaithersburg, MD. The Proceedings (Ref. A9) of the Workshop, particularly the efforts of Working Group 4, record the discussions and provide useful information on the routine characterization of typical waste streams. Waste characterization would typically be expected to include as a minimum the identification of major constituents in the waste (including primary ions and salts or other solids), density, pH, temperature, radioactive isotopes, and a check for the presence of secondary ingredients that could significantly affect the hydration of the cement.

Some waste streams, such as pressurized water reactor (PWR) primary coolant system borated water, are relatively well-characterized and free of secondary ingredients. There are other waste streams, however, such as ion exchange resins, filter sludges and floor drain liquids, that may contain chemicals that can significantly retard or accelerate the hydration of cement or in other ways adversely affect cement waste form performance (Ref. A9). It is impractical for a waste processor to perform qualification testing on every possible combination and concentration of secondary constituents in a given type of waste stream. Nor is it considered practical or necessary for a waste generator to perform a complete quantitative chemical analysis on every batch of waste that is produced. It is, however, incumbent on radwaste system managers and processors to be cognizant of the types of chemicals that may produce problems in using cement in the solidification and stabilization of low-level radioactive waste. The introduction of such chemicals into waste treatment systems that utilize cement stabilization media should be avoided or specifically compensated for in the formula used for stabilizing that waste stream. If the waste processor is a vendor or is otherwise not the generator of the waste, it is incumbent on all parties to be in adequate communication with each other with regard to the types and quantities of chemical ingredients in the waste and the capability of the waste formulation to provide long-term

structural stability to the waste form. As a part of process control, mixing of different wastes in holding tanks and transfer of liquid wastes without adequate flushing of lines should be generally avoided, because such mixing might introduce ingredients into the waste that were not present in the qualification test program that was conducted for the waste stream in question.

To assist waste generators and processors in developing a sense of greater awareness of low-level radioactive waste stream ingredients that may adversely affect the setting and stability of cement-solidified waste forms, a list of such chemicals is provided in Table I. This list is not intended to be all-inclusive. Moreover, some of the constituents listed may be considered hazardous materials, as defined by Environmental Protection Agency (EPA) criteria, and which thus, if mixed with radioactive material, could be classified as a "mixed waste." Any questions about low-level radioactive wastes that might be classified as mixed wastes should be directed to the EPA.

Low-level radioactive waste generators and processors who intend to stabilize Class B and Class C waste with cement should either (a) prevent the contamination of, (b) limit to the extent practical, or (c) pre-treat as appropriate, waste streams that may contain the chemicals and constituents in Table I. It is the responsibility of the waste generator and processor to ensure that the cement formulation used for a given waste stream is qualified for the waste stream chemical constituents and concentrations in question.

VI. PCP SPECIMEN PREPARATION AND EXAMINATION

A. General

The purpose of a Process Control Program (PCP) is to describe the envelope within which processing and packaging of low-level radioactive wastes will be accomplished to provide reasonable assurance of compliance with low-level waste requirements. All commercial nuclear power plants have plant specific PCPs. The guidance provided in this section of this Appendix is not, however, intended to address facility-specific PCPs, which, in addition to containing a general description of the methods for controlling the processing and packaging of radioactive waste, may also contain a description of the system and operating procedures, instructions on manifest preparation, and a discussion of administrative controls. Rather, this guidance addresses only the recipe portion of cement stabilization of low-level waste; that is, the guidance addresses the nature of the information that should be provided in a generic PCP concerning the type and quantity of ingredients used in the cement waste form formulation, the order of addition, and the method, process, and time required for mixing the ingredients in the preparation of verification and surveillance specimens as well as the full-scale waste forms. Also provided is guidance on the preparation of PCP "verification" and surveillance specimens and the type of examinations and testing that should be performed on those specimens.

This information on verification specimens is intended to provide assurance that the formulations used in the qualification testing program correspond to those actually used in the field. The surveillance specimen program, described in Section VII of this Appendix, is intended to provide verification that the waste forms are remaining stable with time.

For each low-level radioactive waste formulation, the generic PCP should address the boundary conditions (i.e., bounding process parameters) for processing the waste to provide reasonable assurance that the final waste form will meet 10 CFR Part 61 stability requirements. The process parameters will be influenced by (a) the characteristics of the waste prior to processing, (b) the qualities of the solidification medium, as influenced by additives, and (c) the physical/chemical process of preparing the waste into a final waste form. Variables that influence the process and have an effect on the product, and that should be, therefore, be identified and restricted within acceptable bounds for each waste form include the following:

1. Type of waste (e.g., bead resin, including type--anion/cation/mixed/manufacturer/weak acid/strong acid, percent depleted, powdered resins, boric acid, sludges);
2. Waste characteristics having influence on the final waste form (e.g., pH, oil content, chelating agents, water content, maximum concentration of secondary ingredients);
3. Additives (e.g., type of cement, water, lime, silica fume, fly ash, furnace slag,) and the order of addition;
4. Physical process parameters (e.g., maximum temperature, mixing equipment required, mixing and curing times).

The generic PCP should indicate how representative samples of the feed waste are to be obtained for preparing PCP verification and surveillance specimens. The PCP should identify typical and maximum batch sizes and the number of PCP specimens to be taken for each batch. The PCP should describe where adjustments could be made to the feed waste material, in the event that certain feed material parameters that may be encountered in the field fall outside of the acceptable range for processing. These adjustments should not be undertaken if the resultant waste stream feed material and stabilized waste form were to be chemically or physically different from that qualified in laboratory testing.

If, during the course of full-scale waste form preparation at a nuclear power plant, it should become necessary to effect an ad hoc, impromptu change in the approved recipe or procedure to avoid an incomplete or otherwise unsatisfactory solidification condition, the change should be reviewed and approved by the facility licensee pursuant to the provisions of 10 CFR 50.59. This process should be followed in all such cases where ad hoc changes are necessary whether or not a generic PCP has received approval as part of a Topical Report review process. Inasmuch as the affected waste form would lack assurance of long-term

structural stability (because it was produced under conditions that were outside of the envelope of the conditions used in the qualification tests), it is anticipated that the resultant waste form would not be accepted for disposal at a disposal site without the expressed approval of the disposal site regulatory authorities. It is also anticipated that, prior to accepting the waste, the regulatory authority would require either (1) adequate mitigation of any potential adverse effects on the long-term structural stability of the waste form or (2) an acceptable justification concerning the lack of any potential significant effect of the affected waste form on the overall performance of the facility. Alternatively, the disposal site regulatory authority could accept the affected waste for disposal with the provision that the required structural stability would be provided at the disposal facility by means of an engineered structure.

After the generic PCP has been reviewed and approved by the NRC, the PCP parameters and procedures should be followed as described in the Topical Report (or other documentation) so that the 10 CFR 20.311 certification can be made without the need for additional justification that the cement-solidified waste meets the requirements of 10 CFR Part 61. Once a generic PCP has been approved by the NRC any subsequent changes to the generic PCP should be reviewed and approved by the NRC. Any incomplete or otherwise unsatisfactory solidification condition known to waste generators and processors is requested to be reported to the NRC (Director, Division of Low-Level Waste Management and Decommissioning) within 30 days after such an occurrence is known (see Section VIII). The actions taken to produce an acceptable waste form after the initial unsatisfactory solidification condition was identified should be described.

B. Preparation of PCP Specimens

Prior to plant-specific solidification of full-scale waste forms, representative samples of the feed waste should be obtained in sufficient quantity to prepare the desired number of PCP specimens. The feed waste material should be solidified using the recipe that has been qualified in laboratory testing for the given waste stream. Mixing of the waste materials with the cement and additives should be accomplished in a manner that duplicates, to the extent practical, the mixing conditions that are obtained with full-scale mixing. The specimens should be cured under conditions similar to those used in the laboratory qualification test program. PCP specimens should be prepared for each batch of waste that is required to meet the 10 CFR Part 61 structural stability criteria. For the purposes of the guidance provided in this Technical Position, a "batch" is herein defined as any quantity of waste stream feed material that is from a single source (e.g., a holding tank), that is processed as a single batch (even though it maybe subdivided in more than one unit waste form; e.g., liner), and that, therefore, possesses unvaried, single operation, batch characteristics.

C. PCP Specimen Examinations and Testing

1. Short-term (24-hour PCP Verification) Specimens -

Prior to solidifying full-scale waste forms, plant-specific PCP verification specimens should be prepared, in accordance with procedures described above,

for examination and compressive strength testing. The specimens should be free of significant visible defects, such as cracking, spalling or disintegration and should exhibit less than 0.5% by volume of the specimen as free liquid. As a measure of process control, the specimens should, within a 24-hour period after preparation, be subjected to an ASTM C39 compressive strength test; (penetrometer measurements may be substituted, as described below). The compressive strength values should be within two standard deviations of the mean compressive strength values obtained at 24 hours for test specimens prepared and tested as part of the associated laboratory generic qualification test program for the waste formulation. Alternatively, penetrometer tests can be used in lieu of C39 compressive strength measurements if acceptable correlation data demonstrating the relationship between the compressive strength values and penetrometer values have been obtained for the waste stream formulation in question. If penetrometer tests are used, the mean penetrometer hardness values obtained on the verification specimens should be within two standard deviations of the mean obtained on the qualification test specimens for that formulation. If the compressive strength or penetrometer measurements do not meet the above criteria, a second set of PCP specimens should be prepared and retested. The second set of PCP specimens should be fabricated using either the same formula or an adjusted one that falls within the compositional envelope of the qualification tests conducted for that waste stream.

2. Long-term Surveillance Specimens -

The guidance herein addressing long-term surveillance specimens is directly applicable to waste generators and to vendors processing wastes at licensed facilities who intend to certify, in accordance with the provisions of 10 CFR 20.311, that the cement-solidified waste meets the structural stability requirements of 10 CFR Part 61. Sufficient PCP specimens should be prepared to permit the retention, examination and testing of surveillance specimens. The surveillance specimens should be stored in sealed containers at normal room temperatures. The examination and testing of surveillance specimens is described in Section VII of this Appendix.

VII. SURVEILLANCE SPECIMENS

The purpose of the surveillance specimens is to provide confirmation that the waste forms prepared for certain waste streams, (in particular bead resins, chelates, filter sludges, and floor drain wastes) are performing as expected. At periods of time equal to 6 months and 12 months after preparation, the surveillance specimens should be examined visually and should be free of evidence of significant cracking, spalling or bulk disintegration (see Section II.C of Appendix A for discussion of "significant degradation"). At least one specimen should be subjected to an ASTM C39 compressive strength (or penetrometer) test at the 6 and 12 month periods. The mean compression strength (or penetrometer) value(s) obtained should be not more than two standard deviations below the mean of the as-cured strength or penetrometer values obtained with the qualification test specimens cured for an equivalent period of time.

At 12 months after preparation, one or more PCP surveillance specimens should be subjected to an immersion test. The duration of the immersion test should be a minimum of 14 days. Upon removal from the immersion liquid, which should be either deionized water or synthesized sea water (see Section II.F of this Appendix) the specimens should be allowed to dry in ambient air for a minimum of 48 hours. The specimens should then be examined visually and should be free of significant surface or bulk defects such as cracking, spalling, or bulk disintegration. Following the immersion test, the specimen(s) should be subjected to an ASTM C39 compressive strength (or penetrometer) test. The test results should meet the criteria discussed above.

If the PCP surveillance specimens tested either by the vendor of an NRC-approved Topical Report or by a utility or other licensee, should fail any of the above tests, the wastes previously solidified may not meet the stability requirements of 10 CFR Part 61. Therefore, the NRC (Director, Division of Waste Management and Decommissioning) and licensee (if other than the waste processor that shipped the suspect waste to the disposal facility) should be notified in writing within 30 days. In turn, the licensee should notify the disposal facility operator and regulatory authority if the 10 CFR 20.311 certification as to waste stability was invalidated by this finding. The licensee's report should satisfy the information needs of the regulatory authority and should describe the waste stream solidified, the waste formulation used, the number of full-scale waste forms that had been produced, date of shipment, manifest numbers, and the results of the tests. The report should also contain a discussion of the significance of the test results and proposed changes, if any, that might have to be made to the waste formulation to ensure that, for the waste stream in question, future waste forms would be stable.

For all waste processors (including utility licensees and vendors of NRC-approved Topical Reports), it is recommended that a summary report that addresses the results of PCP surveillance specimen preparations and examinations should be prepared annually by the waste processor and submitted to the NRC (Director, Division of Waste Management and Decommissioning). The report should document the results of all visual examinations and immersion, compression, and/or penetrometer tests performed on the cement-stabilized waste form surveillance specimens during the calendar year. The annual report should be submitted within 90 days of the end of each calendar year. A commitment to provide this information will be made a condition of approval for all future license applications, topical report submittals or other regulatory actions that deal with cement waste forms, where the waste generators and/or processors desire NRC endorsement of their 10 CFR 20.311 certifications.

VIII. REPORTING OF MISHAPS

Known cement waste form processing mishaps, including but not restricted to, cement waste forms that have not solidified completely, waste forms that have swelled and/or disintegrated, waste forms that were not prepared in accordance with an approved PCP, and waste form preparations that resulted in unusual exothermic reactions, should be reported by the cognizant waste processor to the NRC (Director of the Division of Waste Management and Decommissioning)

within 30 days of the time that the vendor becomes aware of the incident. Licensees should also report such mishaps to the disposal site regulatory authority since such an event may indicate the waste form will or does not satisfy the stability requirements of 10 CFR Part 61. If the mishap becomes known to the waste generator and/or processor before the waste forms are shipped off-site, the affected waste form(s) should not be shipped until approval is obtained from the disposal site regulatory authority. A commitment to report and deal with waste form mishaps as discussed above will be made a condition of approval for all future license applications, topical report submittals, or other regulatory actions that deal with cement waste forms, where the waste generators and/or processors desire NRC endorsement of their 10 CFR 20.311 certifications.

IX. IMPLEMENTATION

This Appendix to the Technical Position on Waste Form reflects the current NRC staff position on an acceptable means for meeting the 10 CFR Part 61 structural stability requirements for cement waste forms. Therefore, except in those cases in which the waste generator, vendor, and/or processor proposes an acceptable alternative method for complying with the stability requirements of 10 CFR Part 61, the guidance described herein will be used by the NRC staff in all future evaluations of the acceptability of cement waste forms for disposal at near-surface disposal facilities.

X. REFERENCES

- A1(a). Part 61 - Licensing Requirements for Land Disposal of Radioactive Waste, Code of Federal Regulations, Title 10: Energy.
- A1(b). "Method for Obtaining Approval of Proposed Disposal Procedures," Subsection 311 of Part 20 (20.302), Code of Federal Regulations, Title 10: Energy.
- A2. American Society for Testing and Materials Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens, ASTM C39, October 1984.
- A3. D.R. MacKenzie, M. Lin, and R.E. Barletta, "Permissible Radionuclide Loading for Organic Ion Exchange Resins for Nuclear Power Plants," Brookhaven National Laboratory Draft Report, BNL-NUREG-30668, January 1982.
- A4. P. Soo and L. W. Milian, "Sulfate-Attack Resistance and Gamma-Irradiation Resistance of Some Portland Cement Based Mortars," Brookhaven National Laboratory Report, NUREG/CR-5279, March 1989.
- A5. American Society for Testing and Materials Standard Practice for Determining Resistance of Synthetic Polymeric Materials to Fungi, ASTM G21, 1985.
- A6. American Society for Testing and Materials Standard Practice for Determining Resistance of Plastics to Bacteria, ASTM G22, 1985.
- A7. P.L. Piciulo, C.E. Shea, and R.E. Barletta, "Biodegradation Testing of Solidified Low-Level Waste Streams," Brookhaven National Laboratory Report, NUREG/CR-4200 (BNL-NUREG-51868), May 1985.
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- A9. Proceedings of the Workshop on Cement Stabilization of Low-Level Radioactive Waste, U.S. Regulatory Commission Report, NUREG/CP-0130, October 1989.
- A10. American National Standards Institute/American Nuclear Society American National Standard for Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure, ANSI/ANS 16.1-1986, April 14, 1986.
- A11. W. Chang, L. Skoski, R. Eng, and P.T. Tuite, "A Technical Basis for Meeting the Waste Form Stability Requirements of 10 CFR 61," Nuclear Management and Resources Council, Inc. Report, NUMARC/NESP-002, April 1988.

A12. P. L. Piciulo, J. W. Adams, J. H. Clinton, and B. Siskind, "The Effect of Cure Conditions on the Stability of Cement waste Forms after Immersion in Water," Brookhaven National Laboratory Report, WM-3171-4, August 1987.

A13. American National Standards Institute/American Nuclear Society American National Standard for Solid Radioactive Waste Processing System for Light Water Cooled Reactor Plants, Appendix 2, March 1979.

Table I

LIST OF WASTE CONSTITUENTS THAT MAY CAUSE PROBLEMS WITH CEMENT SOLIDIFICATION

POTENTIAL PROBLEM CONSTITUENTS WHICH MAY BE EXPECTED IN THE WASTE STREAM

Inorganic Constituents

Borates [1]
Phosphates [1]
Lead salts [2]
Zinc salts
Ammonia and ammonium salts
Ferric salts
"Oxidizing agents" [1]
 (often proprietary)
 Permanganates [1]
 Chromates [2]
Nitrates [1]
Sulfates [1]

Organic Constituents - Aqueous Solutions

Organic acids [1]
 Formic acid (and formates)
"Chelates" [1],[3]
 Oxalic acid (and oxalates)
 Citric acid (and citrates)
 Picolinic acid (and picolines)
 EDTA (and its salts)
 NTA (and its salts)
"Decon solutions" [1]
 Soaps and detergents [1]

Organic Constituents - Oily Wastes

Benzene [1],[2]
Toluene [1],[2]
Hexane [1]
Miscellaneous hydrocarbons
Vegetable oil additives

POTENTIAL PROBLEM CONSTITUENTS THAT MAY BE AVOIDED BY HOUSEKEEPING OR PRETREATMENT [4]

Generic Problem Constituents

[5]

Oil [1] and grease
"Aromatic oils" [1]
"Organic solvents" [1],[2]
Dry-cleaning solvents [1],[2]
"Industrial cleaners" [1],[2]
Paint thinners [1],[2]
"Decon solutions" [1]
Soaps and detergents [1]

Specific Problem Constituents - Organic

Acetone [1],[2]
Methyl ethyl ketone [2]
Trichloroethane [2]
Trichlorotrifluoroethane [2]
Xylene [2]
Dichlorobenzene [2]

Specific Problem Constituents - Inorganic

Sodium hypochlorite [1]

NOTES:

- [1] These constituents have been specifically identified by vendors as having the potential to cause problems with cement solidification of low-level wastes.
- [2] The presence of these constituents may result in the generation of mixed wastes. The Environmental Protection Agency should be contacted for more information.
- [3] All of these chelating agents could also be identified as "organic acids."
- [4] Good housekeeping and pretreatment could also be effective in preventing problems with cement solidification for many of the constituents listed in the top list.
- [5] These specific constituents also fall into several of the "generic" problem constituents "categories" listed at the left.

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