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TECHNICAL JUSTIFICATIONS FOR THE TESTS AND CRITERIA IN THE WASTE FORM TECHNICAL POSITION APPENDIX ON CEMENT STABILIZATION*

B. Siskind and M. G. Cowgill
Nuclear Waste and Materials Technology Division
Department of Nuclear Energy
Brookhaven National Laboratory
Upton, New York 11973

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ABSTRACT

As part of its technical assistance to the Nuclear Regulatory Commission (NRC), Brookhaven National Laboratory (BNL) developed a background document for the cement stabilization appendix, Appendix A, to Rev. 1 of the Technical Position on Waste Form (TP). Here we present an overview of this background document, which provides technical justification for the stability tests to be performed on cement-stabilized waste forms and for the criteria posed in each test, especially for those tests which have been changed from their counterparts in the May 1983 Rev. 0 TP. We address guidelines for procedures from Appendix A which are considered in less detail or not at all in the Rev. 0 of the TP, namely, qualification specimen preparation (mixing, curing, storage), statistical sampling and analysis, process control program specimen preparation and examination, and surveillance specimens. For each waste form qualification test, criterion or procedural guideline, we consider the reason for its inclusion in Appendix A, the changes from Rev. 0 of the TP (if applicable), and a discussion of the justification or rationale for these changes.

INTRODUCTION

Over the past few years there has been increasing interest in matters dealing with waste form stability and topical report reviews. Several groups have expressed concern about these matters, including the Advisory Committee for Reactor Safeguards, which has identified a need to define better the scientific bases for some of the criteria and tests in the NRC TP (1), and the Nuclear Utilities Management and Resources Council, which commissioned a study on the technical bases for meeting the waste form stability requirements of 10 CFR Part 61 (2). Generators of low-level radioactive waste (LLW), vendors of solidification processes, state regulatory bodies, and LLW disposal site operators have also voiced concern.

The overall level of concern has been increasing since the issuance of the TP in 1983 as a result of a growing body of evidence -- from test data as well as from field experience -- that some waste forms may not have the long-term stability characteristics required by 10 CFR Part 61 even though they meet the criteria of Rev. 0 of the TP. The Workshop on Cement Stabilization of LLW held May 31 through June 2, 1989, which we will refer to here simply as the Cement Workshop, considered this evidence in greater detail (3). Further NRC concerns

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were expressed in a report by NRC staff to the Director of the Office of Nuclear Material Safety and Safeguards (4).

As part of the BNL Technical Assistance Program to NRC, we developed a document (5) to provide technical justification for the tests and criteria described in Appendix A, Cement Stabilization, to Revision 1 of the TP, dated January 1991. In the present paper, we present an overview of that report.

BACKGROUND

The May 1983 TP provided guidance on waste form test methods and results which would be considered acceptable in demonstrating compliance with the requirements of 10 CFR Part 61 for structural stability. For solidified waste forms, the tests essentially involved subjecting the waste forms to standardized compression testing before and after irradiation, biodegradation, immersion, and thermal cycling. The TP also specified a leaching test. The tests were selected for their relative simplicity and reproducibility and most of them were based on tests which had been developed for specific applications to non-radioactive materials. These test methods were intended to provide confidence that the waste forms would possess the desired long-term (300-year) stability, although the duration of the tests themselves were relatively short term, i.e., minutes to weeks. These test conditions did not match nor were they intended to duplicate exactly the conditions that might exist in a disposal facility. In some ways the TP tests were accelerated tests, but in a more fundamental sense they were screening tests to eliminate material formulations and designs that did not exhibit sufficient assurance of long-term stability. The introduction to the Cement Workshop discusses this in more detail.

The May 1983 TP, while dealing with solidified LLW generically, provided no guidance on how a topical report submitted by the vendor of a cement solidification process should treat the details of waste form preparation (e.g., mixing, cure time, cure temperature) appropriate for specific binder materials, nor did it provide guidance on testing waste forms which have been fabricated from binder materials which require times comparable to the duration of some of the more time-consuming tests (i.e., several weeks), if not much longer, to attain their ultimate compressive strength. Furthermore, the TP tests would not necessarily screen out waste forms which exhibit what we will term "atypical cement strength behavior" -- namely, a decrease in compressive strength with time. Also, except to state that waste specimen compositions should be "based on the range of waste stream chemistries expected," no guidance was given as to how closely the chemical composition of the surrogate waste used in a vendor's qualification program had to correspond to that of the actual waste encountered in the field. During the NRC's review of the cement solidification vendors' topical reports, these areas -- mixing, curing, atypical cement strength behavior, and composition of surrogate waste used in qualification testing -- would prove to be major potential problem areas revealed as a result of laboratory testing. Based on the extensive experience of the concrete industry as summarized in textbooks such as Mindess and Young (6) as well as in the Concrete Manual, 8th Ed. (7), it is almost a trivial statement to say that the preparation, especially the mixing and curing, of a waste form using a cementitious binder material, e.g., portland cement, are crucial to the subsequent performance of the waste form.

During the past several years, a number of solidifications which were performed at reactor sites did not go as planned. These unsuccessful solidifications fell into the following categories: premature solidification (e.g., Quad

Cities - Unit 2), incomplete solidification (e.g., J.A. FitzPatrick), postponed solidification (e.g., Peach Bottom), and bulging of liners (e.g., TMI-2, Millstone-1). These problems which occurred in the field with full-scale "real-world" waste forms indicated a need for modifications and additions to the May 1983 TP in the area of cement solidification of LLW. For more details on the unsuccessful solidifications, we refer the reader to the Working Group 1 discussions in the Cement Workshop proceedings (3). The Working Group 1 participants, however, believed that the vast majority of "solidifications" (over 95%) have been successful.

WASTE FORM QUALIFICATION TESTING

Compression

According to 10 CFR 61.56(b)(1), "a structurally stable waste form will generally maintain its physical dimensions and its form under the expected disposal conditions such as weight of overburden and compaction equipment...." [emphasis added]. Taking into consideration the potential additional loads such as those from trench compaction equipment and waste contents, the May 1983 TP set the compressive strength criterion at 50 psi (345 kPa). This criterion was raised later to 60 psi (414 kPa) to reflect an increase in burial depth at Hanford to 55 feet (16.8 m).

To provide greater assurance that there will be sufficient cementitious material present in the waste form not only to withstand the burial loads but also to maintain general "dimension and form" (i.e., not fall apart or disintegrate), Appendix A to the TP recommends a compressive strength criterion "representative of the values that are reasonably achievable with current cement solidification processes." Appendix A also notes that portland cement mortars, consisting of mixtures of cement, silica sand and water, are readily capable of achieving compressive strengths of 5000 to 6000 psi (35 to 41 MPa), values which are approximately two orders of magnitude greater than the minimum values required to resist deformation under load in a burial trench. Appendix A then notes that LLW constituents "are not in most cases capable of providing the physical and chemical functions of silica sand in a cement mortar." As a result, Appendix A recommends a mean compressive strength of at least 500 psi for waste form specimens cured for a minimum of 28 days. Since data are lacking on the actual stability of cement-solidified LLW for long time durations compared to the laboratory qualification tests, subsequent information may require revisions to the 500 psi compressive strength criterion. This "provisional" compressive strength criterion of 500 psi in a sense quantifies the May 1983 TP requirement of "maximum practical compressive strength" cited in the previous section.

Unlike the May 1983 TP, Appendix A to the TP addresses scatter in the compressive strength data. Such scatter occurs because cement is a brittle ceramic which fails in tension or shear rather than compression at regions of localized stress concentration or microstructural flaws. Appendix A recommends that sufficient specimens -- a minimum of ten -- should be tested to determine the mean compressive strength and standard deviation. Since data on the variability in compressive strength values of such waste forms are lacking, a precision criterion in the form of an acceptable variance or standard deviation must be deferred.

The May 1983 TP, while indicating that "maximum practical" values should be developed for the compressive strength, provided no guidance as to what these values might be, either for individual waste form recipes or as a general goal. Without such guidance, a tendency evolved among the vendors of cement (and other) LLW solidification processes to concentrate primarily on meeting the minimum compressive strength value of 50 psi (later revised upward to 60 psi). In addition, this was sometimes interpreted as a "mean" minimum compressive strength, which meant that it was possible to meet the criterion although individual test samples might actually fall below the minimum value. These interpretations were considered inappropriate not only because there was apparently little attempt to optimize the properties of the solidifications but also they impinged on the basic engineering principle of including safety factors in designs. This was particularly relevant as the TP specifically stated that the mechanical design strength of a high integrity container (HIC) "should be justified by conservative design analyses" (Section C.4.d in the TP).

To alleviate this potential problem, Appendix A provides guidance as to what would be considered a mean practical value for cement-solidified LLW, namely, 500 psi for specimens cured for at least 28 days. Appendix A, recognizing that the technical basis for selecting this value is limited at the moment, states that this criterion may be subject to revision. The 500 psi value is considered to be indicative of the presence of sufficient cementitious material to ensure typical cement-like behavior. Furthermore, it is a strength level reasonably achievable in cement-solidified wastes, as has been demonstrated in several laboratory-scale studies. For example, Piciulo et al. (8) found that for cement-solidified mixed-bed bead resins, a waste stream which can be difficult to solidify in cement, mean compressive strengths in excess of 500 psi are achievable for certain vendor formulations, even after being subjected to the 90-day water immersion test. In addition, Boris (9) found that 500 psi was achievable for a variety of waste streams solidified in cement. Only one cement-solidified waste stream, oil, did not attain 500 psi while several others (boric acid, bead resins at higher loadings) attained 500 psi only after cure periods of greater than 28 days (e.g., 56 to 84 days).

Thermal Cycling

Section 61.56(b)(1) of 10 CFR Part 61 addresses "internal factors," which include temperature and temperature effects. Also, an actual waste form will experience temperature excursions, particularly during storage and transport prior to disposal. Although the specific minimum and maximum temperatures (-40°C and 60°C, respectively) may not actually be attained during storage, transport, or disposal of the waste forms, this test can function as a screening test to eliminate waste formulations with poor stability.

The thermal cycling test in TP Appendix A contains several modifications to the May 1983 TP thermal degradation test. First, the specimens are to be allowed to come to thermal equilibrium at the high and low temperature limits as confirmed by the center temperature measurement of at least one specimen. In addition, Appendix A requires the specimens to be examined visually for evidence of significant cracking, spalling, or bulk disintegration after 30 thermal cycles. (Photographic evidence is required of defects judged to be insignificant.) Finally, Appendix A specifies compression testing for waste forms free of significant defects after 30 thermal cycles; the compressive strength should be greater than 500 psi.

It is generally recognized that thermal cycling can cause progressive deterioration in the mechanical properties of many materials, including cements and concretes. The potential for damage to structural concrete from repeated temperature variations has been a source of concern in the nuclear power industry for many years -- see, for example, Lankard et al. (10). In such cases, one of the contributing factors to the deterioration is the loss of moisture when the cycling involves elevated temperatures. At the other end of the temperature spectrum, a different concern manifests itself - that of the freezing of the pore water and the attendant volume increase. The "frost" damage sustained by highway surfaces results from cycling which involves alternate freezing and thawing, and the problems and cures are now well documented in the many textbooks available, e.g., Mindess and Young (6) and Neville (11). It is the problem of freeze-thaw damage that is particularly relevant to the thermal cycling of cement-based low-level radioactive waste forms.

In order to obtain a degree of consistency in test procedures, it is desirable to assure that specimens become equilibrated thermally, at least at the high and low points of the thermal cycle. The simplest method of ascertaining that this equilibrium has been achieved is by monitoring and comparing the centerline and the surface temperatures of the specimen.

As with the basic compressive strength requirements for pre-test material, the criterion of 500 psi minimum is based on values that are considered reasonably achievable and gives some assurance of adequate structural strength.

Irradiation

There is a large body of evidence that exposure to radiation can influence the stability of various waste-stream/binder combinations. [For a discussion of some of this evidence in relation to the May 1983 TP irradiation test, we refer the reader to Bowerman et al. (12).]

The irradiation test in Appendix A for cement-solidified LLW incorporates several modifications to the corresponding test in the May 1983 TP. One major modification is that irradiation qualification testing need not be conducted unless the cement waste forms contain ion-exchange resins or other organic LLW material. As in the May 1983 TP, if the maximum level of exposure is expected to exceed 10^8 rad, testing should be performed on specimens exposed to the expected maximum dose. In addition, Appendix A requires the specimens to be examined visually after irradiation for evidence of significant cracking, spalling, or bulk disintegration and, as for the thermal cycling test, specifies compression testing for waste forms free of significant defects after irradiation; the compressive strength should be greater than 500 psi.

These changes are justified since cement and concrete exhibit excellent radiation resistance, even at doses of 10^9 or 10^{10} rad. Organic materials, in contrast, are generally susceptible to a variety of radiation effects. Because of the lack of information on the effects of radiation on many organic wastes and the resulting cement waste forms, the irradiation testing of cement waste forms incorporating organic wastes should continue to be performed until an extensive data base has been compiled.

Biodegradation

Appendix A exempts from biodegradation testing cement waste forms not containing carbonaceous materials since such waste forms generally do not support fungal or bacterial growth (3, 12, 13). The absence of such growth results largely from the lack of a carbonaceous food source for the microorganisms used in the test protocols.

Leach Testing

Appendix A makes two significant changes to the leach test specified in Section C.2.e. of the May 1983 TP. First, the leach test period is reduced from 90 days to 5 days. Second, the bulk of the leach testing may be conducted with only one leachant if sufficient preliminary testing is performed to identify which of the leachants, deionized water or synthetic sea water, is the more aggressive one. An acceptable method for identifying the more aggressive leachant is to perform 24-hour (or longer) leaching measurements using both leachants (separately) and to use the leachant with the higher leach rate for the remaining days of testing.

The leach testing identified in the TP is, in effect, a quality control measure. The major concern is that the leachability index should be greater than 6.0. It can be demonstrated by a simple calculation that this requirement can be checked in an abbreviated (5-day) test; for details, see the supporting background document (5). Using the appropriate curve from ANSI/ANS-16.1 (14), it may be shown that for a cylinder with a length and diameter of 2.5 cm (about 1 in) approximately 92% of the species of interest have become leached from the waste form at 5 days. Similar calculations show that for smaller specimens a very high proportion of species with leachability index of 6.0 will be released in a 5-day leach test and little additional meaningful information will probably be gained from extending the test to 90 days. Specimens used in 90-day leach testing often fall into this category.

Originally, the requirement for a 90-day test rather than a 5-day test derived from the possibility that the leaching process might not be time-independent, as is assumed in the ANSI/ANS procedure, and that there might be a change from a diffusion-controlled mechanism between 5 and 90 days. This is a valid concern when using the information to implement a model for predicting long-term leach rates. However, as stated previously, this was not the intent of the 90-day test requirement in TP Rev. 0. Rather, it sought only to establish if such a change existed. In the event that the leach-rate-controlling mechanism became something other than diffusion (e.g., erosion, corrosion, dissolution), the effects would probably be observed visually or by mechanical testing. This was recognized in the TP by the inclusion of a 90-day immersion test which could be performed in conjunction with the leach testing. Thus, the presence of the immersion test requirement assures that an assessment will be available of the degradation caused by mechanisms other than diffusion-controlled leaching.

Immersion Testing

As in the case of several of the tests discussed above, Appendix A requires specimens of those waste forms subjected to the immersion testing to be examined visually for evidence of significant cracking, spalling, or bulk disintegration and specifies compression testing for waste forms free of significant defects after immersion testing; the compressive strength should be either greater than 500 psi or not less than 75 percent of the pre-immersion compressive strength (as measured on specimens that have been cured for a minimum of 28 days). If the post-immersion compressive strength is greater than 500 psi but less than 75 percent of the as-cured, pre-immersion compressive strength, the immersion testing interval should be extended to a minimum of 180 days. For these cases, sufficient compressive strength testing should be conducted (e.g., at 120, 150, and 180 days) to establish that the compressive strengths level off and do not continue to decrease with time.

For certain waste streams, e.g., bead resins, chelating agents, that have been found to exhibit complex relationships between cure time and immersion resistance, immersion testing should be performed on specimens that have been cured 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°C. After the specimens have been dried, they should meet the post-immersion test criteria specified above for compressive strength and for visible defects.

The extended testing regime specified in the event that the post-immersion compressive strength is greater than 500 psi but less than 75 percent of the pre-immersion value is intended to provide reasonable assurance that the compressive strength does not continue to decrease at a similar rate during subsequent periods of immersion, possible even reaching zero. The immersion followed by drying which Appendix A specifies for certain "problem" waste streams solidified in cement simulates to an extent wetting followed by drying which could occur under certain storage and disposal conditions.

Free-Standing Liquids

As in Section C.2.h. of the May 1983 TP, the free liquid should not exceed 0.5 percent of the waste specimen volume as measured using the method described in Appendix 2 of ANSI/ANS-55.1 (15). The minimum pH of the free liquid, however, is increased to 9 from the May 1983 TP value of 4. Since cement is an alkaline material, evidence of acidic free liquids is indicative of properties (and thus performance) not typical of cement and probably results from improper waste form preparation or curing.

Full-Scale Testing

The requirements of Section 61.56 of 10 CFR Part 61 are for full-size LLW waste forms. It is, therefore, necessary to establish the applicability of the laboratory-scale testing discussed in the previous sections to the full-scale product in the field by correlating the characteristics of full-scale products with those of laboratory-scale specimens.

The May 1983 TP addressed this need for establishing scale-up from laboratory to field by specifying that test data should be obtained for sections or cores of anticipated full-scale products and by setting a homogeneity guideline for the compressive strength of samples from the full-scale specimen, namely, > 50 psi. Appendix A specifies that these cores or sections should meet the compressive strength criterion called out in the section on compression testing in Appendix A, namely, 500 psi (but subject to change in light of subsequent information).

QUALIFICATION TEST SPECIMEN PREPARATION

Mixing

As noted in Appendix A, experience in preparation of lab-scale and full-scale cement-solidified waste forms has shown that the method employed in mixing the ingredients can have a dramatic influence on the properties and characteristics of the waste form. Therefore, Appendix A requires 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 qualification specimens as the full-scale mixing equipment will impart to the full-scale waste forms and that the degree of mixing is sufficient to ensure production of homogeneous waste forms.

Curing

As discussed earlier, experience in preparation of lab-scale specimens of cement-solidified waste forms has shown that curing time and curing conditions affect the stability of the waste form product. Therefore, the curing conditions for laboratory-scale qualification test specimens should, to the extent practical, duplicate the conditions obtained with full-scale products. The qualification specimen temperature, in particular, should duplicate the waste form centerline temperature profile as a function of time for the largest full-scale waste form to be qualified. Normally, the compressive strength at 28 days approaches seventy-five percent or more of the "peak" value, but 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 peak) strength prior to subjecting the remaining specimens to qualification testing. It is necessary to determine that the specimens are essentially cured before subjecting them to qualification testing in order to assure "prototypic cement strength behavior;" that is, that the strength increases with time.

Storage

Specimens to be used for the qualification testing of Section II of Appendix A should be stored in sealed containers following the peak heat-of-hydration period of curing in order to simulate the environment that would be obtained in a typical full-scale waste form liner. Storage in a sealed container will also prevent loss of water that might affect the performance of the waste form specimens during subsequent testing. Storage of the specimens in sealed containers is in accord with established practice for test specimens of structural concrete (16).

STATISTICAL SAMPLING AND ANALYSIS

Materials with more or less homogeneous microstructures (such as metals) will often show minimal variability within a specific batch of material and the performance of two or three tests may be considered sufficient to obtain reliable information. However, materials with heterogeneous structures, such as concrete and cement-based waste forms, will provide data which have less reproducibility. This problem of variability is recognized in the concrete industry and is treated in detail in such texts as Neville (11) and the Bureau of Reclamation's <u>Concrete Manual</u> (7). It follows that an adequate description of the properties of these materials can only be obtained by performing more than two or three tests and establishing a reasonable statistical base.

Appendix A does not provide at this time a precision criterion for an acceptable variance about the mean because insufficient data exist on cement-solidified LLW to permit such a formulation. We agree with this assessment. When sufficient data have been assembled, the precision criterion could possibly take a format similar to that recommended by the ACI Committee 214 (17) relative to standards of control on concrete. Most relevant to cement-solidified LLW would appear to be ratings based on the coefficient of variation, a parameter which normalizes the standard deviation with respect to the mean compressive strength.

In accord with ANSI/ANS-16.1, Appendix A recommends that the confidence range and correlation coefficient be reported with the leach index. Since no precision criterion has yet been established for the ANSI/ANS-16.1 leach test, acceptance criteria for the confidence range and correlation coefficient will be deferred pending the collection and analysis of sufficient data to establish such criteria.

WASTE CHARACTERIZATION

As noted in Appendix A, although some reactor waste streams are relatively well characterized and free of secondary ingredients, some, such as ion exchange resins, filter sludges and floor drain liquids, may contain chemicals that can significantly retard or accelerate the hydration of cement or otherwise adversely affect cement waste form performance. The proceedings of the Cement Workshop (3) document such incidents. Especially noteworthy is the effect of a parts-per-million concentration of an organic chelating agent on the setting of the cement-solidified decontaminated supernatant in the reprocessing waste tank at West Valley. Also noteworthy are the several incidents involving problems with cement solidification in liners containing ion-exchange resin waste from power-plant cooling system decontaminations; these problems were attributed to various reactions involving picolinic acid, an organic chelate decontamination reagent.

Radwaste system managers and processors should be cognizant of the types of chemicals that may produce problems for solidification and stabilization of LLW in cement. The introduction of such chemicals into cement solidification systems should be avoided or specifically compensated for in the cement solidification formulation for that waste stream. Mixing of different wastes in holding tanks and transfer of liquid wastes without adequate flushing of lines should be avoided. This section of Appendix A provides a list of chemicals which may adversely affect the setting and stability of cement.

PROCESS CONTROL PROGRAM SPECIMEN PREPARATION AND EXAMINATION

General

As Appendix A indicates, the purpose of the recipe portion of the process control program (PCP) is to provide assurance that the formulations used in the qualification testing program correspond to those actually used in the field. Appendix A specifies that the following process variables which influence the solidification of LLW in cement be identified and constrained within acceptable limits:

a. Type of waste (e.g., boric acid, powdered resin, bead resin -- including type where applicable, e.g., mixed bed, cation strong acid);

b. Other waste characteristics influencing the final waste form (e.g., pH, oil content, chelating agents, water content);

c. Ingredients and additives (e.g., cement, water, silica fume, fly ash) and their order of addition;

d. Physical process parameters (e.g., temperature ranges, mixing equipment, curing times).

Appendix A also requests information in the PCP on representative sampling of the feed waste for verification specimens, typical and maximum batch sizes, number of PCP verification specimens, and where adjustments could be made to the feed material in the event of out-of-range process parameters.

Preparation of PCP Specimens

The purpose of the PCP specimens is to verify correspondence between the actual full-scale waste forms being prepared and the laboratory-scale qualification test specimens. Therefore, the preparation procedures and conditions of the PCP specimens should correspond as closely as possible to those of the qualification test specimens while at the same time simulating to the extent practical those of the actual full-scale field waste forms.

Representative samples of each batch of the actual feed waste required to be stabilized, not simulated waste, should be used to prepare the PCP specimens. The composition of the simulated waste cannot match that of the actual waste, especially with regard to the constituents present at small concentrations, say, in the parts-per-million range. The feed waste material should be solidified using the laboratory qualification test program recipe for the given waste stream, mixed with the cement and additives in a manner duplicating to the extent practical full-scale mixing conditions, and cured under conditions similar to those used in the laboratory qualification test program.

PCP Specimen Examination and Testing

At least 24 hours before solidifying actual full-scale waste forms, PCP verification specimens should be prepared. These should be examined for (and found free of) visible defects and at 24 hours exhibit a compressive strength within two standard deviations of the mean compressive strength exhibited by the laboratory-scale qualification test specimens at 24 hours. Penetrometer tests may be used if correlations between these tests and the ASTM C39 compressive strength tests have been obtained for the waste stream formulation in question.

In addition, sufficient PCP specimens should be prepared to permit the retention, examination, and testing of surveillance specimens as per Section VII of Appendix A. The PCP surveillance specimens should be stored in sealed containers at room temperature.

The 24-hour PCP verification specimens provide a "signature" of the solidification of the full-scale waste form as well as an indication as whether the waste feed is sufficiently similar in composition to the surrogate waste used in the laboratory-scale qualification test specimens so that the qualification testing program is applicable. Significant deviations from the 24-hour qualification specimen compressive strength values could be indicative of problem constituents or a waste form formulation not bracketed by the composition ranges of the qualification testing program.

SURVEILLANCE SPECIMENS

As stated in Appendix A, the purpose of the surveillance specimens is to provide confirmation that the waste forms prepared for certain waste streams (such as bead resins, chelates, and floor drain wastes) are performing as expected. The surveillance specimens should have chemical compositions representative of that of the full-scale waste form in question. Because of this similarity as well as the similarity between the preparation conditions and procedures of the surveillance specimens and those of the actual full-scale waste forms, any deterioration of the surveillance specimens, whether it be a significant decrease in compressive strength or significant visible degradation, is likely to be indicative of a similar deterioration of the full-scale waste form.

At 6 and 12 months after preparation, the surveillance specimens should be examined and be free of significant visible degradation. The compressive strength (or penetrometer value) should be determined for at least one specimen at these times and should be no more than two standard deviations below the mean of the compressive strength of equivalently-cured qualification test specimens. At 12 months one or more surveillance specimens should be subjected to immersion for at least 14 days in the more aggressive leachant and then dried in ambient air for at least 48 hours. The specimens should meet the compressive strength and visible degradation criteria for the 6- and 12-month unimmersed test specimens. In the event the PCP specimens fail any of these tests, the NRC and disposal site regulatory authorities should be notified.

REPORTING OF MISHAPS

10 CFR 20.311 requires waste generators and processors to certify that their waste forms meet the requirements of Part 61, including those for structural stability. Waste form processing mishaps may be indicative of lack of such long-term stability. Information on the conditions and observations surrounding the mishaps are not only essential to ensure that proper corrective action can be taken and that the possibility of future mishaps can be minimized. It will also increase the database on the linkage between solidification and stabilization of qualification specimens and actual waste forms.

Therefore, cement waste form processing mishaps, including but not limited to incomplete solidifications, swollen or disintegrated waste forms, waste forms not prepared in accord with an approved PCP, and waste form preparations accompanied by unusual exothermic reactions, should be reported to the NRC's director of the Division of Waste Management and Decommissioning and the designated disposal site authority within 30 days of the incident. These waste forms should not be shipped off site until approval is obtained from these authorities.

CONCLUSIONS

We conclude that there is a sound technical basis for the tests and criteria described in Cement Stabilization, which constitutes Appendix A to Revision 1 of the NRC Technical Position on Waste Form, dated January 1991. We also conclude that these tests and criteria provide reasonable assurance that LLW stabilized in cement in accordance with these tests and criteria will meet the stability requirements for LLW in 10 CFR Part 61. Furthermore, although disposal standards for LLW can be made completely generic, based on the experience with cement stabilization of LLW, disposal acceptance criteria and the associated testing will have to be based on the properties of the solidification medium. For example, we would expect that the testing and criteria for bitumen as a LLW solidification medium will be different from those for cement.

REFERENCES

1. WILLIAM KERR, Chairman, Advisory Committee for Reactor Safeguards, Letter to U.S. NRC Chairman, Lando W. Zech, Jr., November 10, 1987.

2. 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.

3. M. TOKAR, P. REED, B. JAGANNATH, and J. CLIFTON, Workshop on Cement Stabilization of Low-Level Radioactive Waste, Gaithersburg, MD, May 31 - June 2, 1989, U.S. Nuclear Regulatory Commission and National Institute of Standards and Technology, NUREG/CP-0103, NISTIR 89-4178, October 1989.

4. U.S. Nuclear Regulatory Commission, "Current Status and Proposed Action for Regulation of LLW Stability," Report to the Director, Office of Nuclear Material Safety and Safeguards, prepared by the Division of Low-Level Waste Management and Decommissioning Staff, June 21, 1988, enclosed with Memorandum from Richard L. Bangart, Acting Director, LLWMD, to Hugh L. Thompson, Director, NMSS, dated June 24, 1988.

5. B. SISKIND and M. G. COWGILL, Brookhaven National Laboratory, "Technical Justifications for Proposed Tests and Criteria in the Draft Cement Stabilization Appendix to the Technical position on Waste Form," WM-3175-12, February 1991.

6. S. MINDESS and J.F. YOUNG, Concrete, Prentice-Hall, Inc., Englewood Cliffs, NJ (1971).

7. U.S. Department of Interior, Water and Power resources Service (formerly U.S. Bureau of Reclamation), Concrete Manual, 8th Ed., U.S. Government Printing Office, Washington, DC (1975).

8. 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," Technical Report, Nuclear Waste and Materials Technology Division, Brookhaven National Laboratory, September 1987.

9. G. F. BORIS, "Testing of Variables which Affect Stability of Cement Solidified Low-Level Waste," in Waste Management '89, Tucson, AZ, February 26-March 2, 1989, R. G. Post, Ed., Volume II, Low-Level Waste, pp. 401-405.

10. D.R. LANKARD, D.L. BIRKIMER, F.F. FONDRIEST and M.J. SNYDER, "Effects of Moisture Content on the Structural Properties of Portland Cement Concrete Exposed to Temperatures up to 500 F", in "Temperature and Concrete" ACI Publication SP 25, American Concrete Institute, Detroit, MI, pp. 59-102 (1971).

11. A.M. NEVILLE, Properties of Concrete, Pitman Publishing Inc., Marshfield, MA 02050 (1983)

12. B. S. BOWERMAN, K. J. SWYLER, D. R. DOUGHERTY, R. E. DAVIS, B. SISKIND, and R. E. BARLETTA, "An evaluation of the Stability Tests Recommended in the Branch Technical Position on Waste Form and Container Materials," NUREG/CR-3829, BNL-NUREG-51784, March 1985.

13. P. L. PICIULO, J. W. ADAMS, M. S. DAVIS, L. W. MILIAN, and C. I. ANDERSON, "Release of Organic Chelating Agents from Solidified Decontamination Wastes," Topical Report, NUREG/CR-4709, BNL-NUREG-52014, July 1986.

14. ANSI/ANS -16.1-1986, "Measurement of the Leachability of Solidified Low-Level Radioactive Wastes by a Short-Term Test Procedure," American Nuclear Society, La Grange Park, IL (1986)

15. ANSI/ANS -55.1-1979, "American National Standard for Solid Radioactive Waste Processing System for Light Water Cooled Reactor Plants," American Nuclear Society, La Grange Park, IL (1979)

16. ASTM C 192-86, Standard Practice for Making and Curing Concrete Specimens in the Laboratory, American Society for Testing and Materials, Philadelphia, PA (1988).

17. American Concrete Institute Committee 214, "Proposed Revision of ACI 214-65: Recommended Practice for Evaluation of Strength Test Results of Concrete," ACI Journal, May 1976, 265-278.

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