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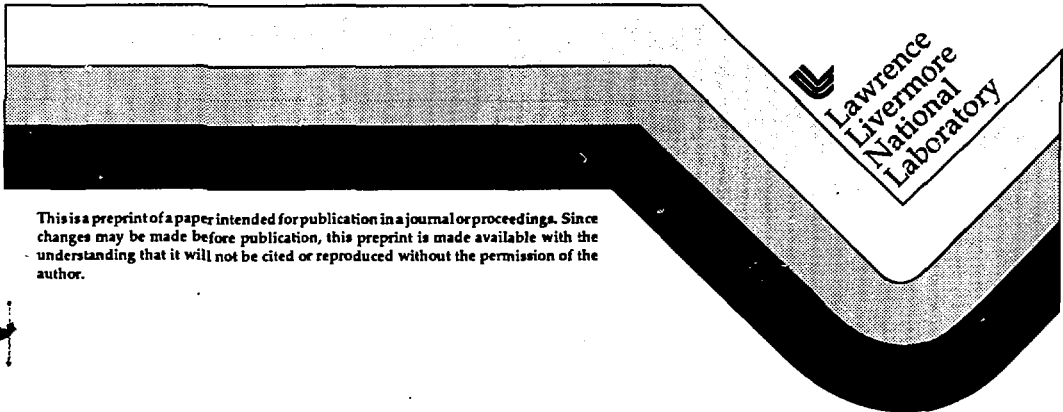
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# QUALIFICATIONS OF AND ACCEPTANCE CRITERIA FOR TRANSPORTING SPECIAL FORM RADIOACTIVE MATERIAL

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**ABSTRACT**

A special form radioactive material is a radioactive material that is in an inert, insoluble, indispersible form such that even in the event of an accident, it will not be dispersed into the environment in a way that could have an adverse impact on public health and safety. Methods of qualifying a special form radioactive material are discussed. Interpretation of acceptance criteria are proposed for the transportation of Type B quantities of a special form radioactive material.

**INTRODUCTION**

A special form radioactive material is defined in 10 CFR 71 (Code of Federal Regulations, Title 10, Section 71, Office of Federal Register - Proposed) as a radioactive material which satisfies the following conditions.

- (1) It is either a single piece or is contained in a sealed capsule that can be opened only by destroying the capsule;
- (2) The piece or capsule has at least one dimension not less than 5mm; and
- (3) It satisfies the requirements of §71.75 ....

A similar definition of special form radioactive material is given in 49 CFR 173 (Office of Federal Register-Proposed) and IAEA Safety Series No. 6 (International Atomic Energy Agency 1985).

A special form radioactive material is inert, insoluble, and indispersible so that even in an accident, radioactive material will not escape into the environment to adversely impact public health and safety. A special form radioactive material can be either a qualified solid material, such as irradiated hardware, or a qualified sealed capsule containing a radioactive material, such as an encapsulated radiation source. Special form radioactive materials have many uses, which include:

- Provide radiation sources for:
  - calibration of radiation diagnostic equipment
  - nondestructive tests of materials
  - treatment of tumors (oncology)
- Increase the quantity of a radioactive material that can be shipped in a Type A package.
- Provide part of the containment in Type B packages.

Thus there are two types of issues for special form radioactive material: (1) the acceptance criteria for qualification of the material itself, and (2) the acceptance criteria necessary for special form radioactive material to be accredited for part of the containment in Type B packages. This paper describes both types of issues.

**QUALIFICATION OF A SPECIAL FORM RADIOACTIVE MATERIAL**

Necessary, but not sufficient conditions for qualifying a special form radioactive material are given in 10 CFR 71.75. To qualify the material requires:

- (a) Evaluation of the contents of a single package for qualification as special form must include a determination of the effect on a specimen of those contents of the tests specified in § 71.77
  - (1) Specimens (solid radioactive material or capsules) to be tested must be as normally prepared for loading in a single package, with the radioactive material duplicated as closely as practicable.
  - (2) A different specimen may be used for each of the tests.
- (b) The specimen must not break or shatter when subjected to the impact, percussion, or bending tests.
- (c) The specimen must not melt or disperse when subjected to the heat test.

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For a solid radioactive material, the indispersibility of the tested specimen must conform to 10 CFR 71.75(d):

(d) After each test, leak-tightness or indispersibility of the specimen must be determined by a method no less sensitive than the following leaching assessment procedure. For a capsule resistant to corrosion by water and which has an internal void volume greater than 0.1 ml ( $6.10 \times 10^{-3}$  in<sup>3</sup>), an alternative to the leaching assessment is a demonstration of leak-tightness of  $10^{-4}$  torr-l/s ( $1.03$  lb-in/s) (based on air at 25°C (77°F) and one atmosphere differential pressure) for solid radioactive content, or  $10^{-6}$  torr-l/s ( $3 \times 10^{-4}$  lb-in/s) for liquid or gaseous radioactive content.

- (1) The specimen must be immersed for 7 days in water at ambient temperature. The water must have a pH of 6-8 and a maximum conductivity of 10 µmho/cm at 20°C (68°F). Encapsulated material is not subject to the 7-day requirement.
- (2) The water with specimen must then be heated to a temperature of 50°C ±5°C (122°F ±9°F) and maintained at this temperature for 4 hours.
- (3) The activity of the water must be determined at that time.
- (4) The specimen must then be stored for at least 7 days in still air of humidity not less than 90 percent and a temperature not less than 30°C (86°F).
- (5) The specimen must then be immersed in water having a pH of 6-8 and a maximum conductivity of 10 µmho/cm at 20°C, and the water with specimen heated to 50° ±5°C (122° ±9°F) and maintained at this temperature for 4 hours.
- (6) The activity of the water must be determined at that time.
- (7) The activities determined in paragraphs (d)(3) and (d)(6) of this section must not exceed 0.0541 µCi (2 kBq).

Both IAEA Safety Series No. 6 and 49 CFR 173.469 allow an alternative to the above leach test method for an encapsulated material. This method consists of performing steps (d)(5) through (d)(7) above followed by performing steps (d)(4) through (d)(7). Another option to 10 CFR 71.75(d) for sealed encapsulated radioactive material is 10 CFR 71.75(e) given below:

(e) A specimen that comprises or simulates radioactive material contained in a sealed capsule need not be subjected to the leak-tightness procedure specified in this section provided it is alternatively subjected to any of the tests prescribed in the International Organization for Standardization document ISO/TR4826-1979(E), "Sealed radioactive sources - Leak test methods", which are acceptable to NRC.

The tests prescribed in ISO/TR 4826-1979(E) (International Organization for Standardization 1979) include:

- wipe (smear) test
- dry wipe test
- immersion test (similar to 10 CFR 71.75(d)(2))
- boiling immersion test
- gaseous emanation test
- liquid scintillant emanation test
- vacuum bubble test
- hot-liquid bubble test
- gas pressurization bubble test

- helium test
- helium pressurization test
- water pressurization test

Note that not all of the above test methods are applicable or appropriate to all sealed sources. It is necessary to select from them the most suitable test or combination of tests for the particular situation. For the above leak tests, the sealed source is considered to be leak-free if the measured activity is less than 5 nCi (185 Bq). Additional guidance on leak testing is given in ANSI N14.5 (American National Standards Institute 1987).

The tests for qualifying a special form radioactive material given in 10 CFR 71.77 include:

- (a) *Impact test.* The specimen must fall onto a flat, horizontal, essentially unyielding surface from a height of not less than 9 m (29.5 ft).
- (b) *Percussion test.* The specimen must be placed on a sheet of lead which is supported by a smooth solid surface and struck by the flat face of a steel billet so as to produce an impact equivalent to that resulting from a free fall of 1.4 kg (3.00 lbs) through 1 m (39.4 in). The flat face of the billet must be 25 mm (0.984 in) in diameter with the edges rounded to a radius of  $3 \pm 0.3$  mm (0.118 ±0.0118 in). The lead, of hardness number 3.5 to 4.5 on the Vickers scale and not more than 25 mm (0.984 in) thick, must cover an area greater than that covered by the specimen. A fresh surface of lead must be used for each impact. The billet must strike the specimen so as to cause maximum damage.
- (c) *Bending test.* The test is applicable only to long, slender sources with both a minimum length of 10 cm (3.94 in) and a length to minimum width ratio not less than 10. The specimen must be rigidly clamped in a horizontal position so that one half of its length protrudes from the face of the clamp. The orientation of the specimen must ensure that the specimen will suffer maximum damage when its free end is struck by the flat face of a steel billet. The billet must strike the specimen so as to produce an impact equivalent to that resulting from a free vertical fall of 1.4 kg (3.09 lb) through 1 m (39.4 in). The flat face of the billet must be 25 mm (0.984 in) in diameter with the edges rounded off to a radius of  $3 \pm 0.3$  mm (0.118 ±0.0118 in).
- (d) *Heat test.* The specimen must be heated to a temperature of not less than 800°C (1472°F) in an atmosphere which is essentially air, and held at that temperature for a period of 10 minutes and must then be allowed to cool.

In lieu of 10 CFR 71.77(a)-(d) a sealed encapsulated radioactive material can be tested as follows:

- (e) A specimen that comprises or simulates radioactive material contained in a sealed capsule need not be subjected to the following:
  - (1) The impact test and the percussion test of this section provided it is alternatively subjected to the Class 4 impact test prescribed in the International Organization for Standardization document ISO 2919-1980(e), "Sealed radioactive sources - Classification"; and
  - (2) The heat test of this section provided it is alternatively subjected to the Class 6 temperature test specified in the International Organization for Standardization document ISO 2919-1980(E), "Sealed radioactive sources - Classification."

For the ISO-2919-1980(E) (International Organization for Standardization 1980) Class 4 impact test, a 2 kg hammer with a flat striking surface diameter of 55 mm and edges rounded to a radius of

3 mm drops from a height of 1 meter on the specimen placed on a >20 kg anvil. The anvil has a flat surface large enough to take the whole of the specimen and is rigidly mounted so that it does not deflect during impact.

For the ISO Class 6 temperature test, the specimen is cooled from ambient to a temperature of -40°C in less than 45 minutes and held at -40°C for 20 minutes. The specimen is heated from ambient to a temperature of 800°C in less than 70 minutes and held at 800°C for an hour. A thermal shock test must also be performed on either the specimen used previously for the temperature test, or on a second specimen. The specimen must be heated from ambient to a temperature of 800°C in less than 70 minutes, held at 800°C for at least 15 minutes, and transferred in a maximum time of 15 s to water at a temperature of 20°C. The water, if flowing, must have a flow rate of at least 10 times the specimen volume per minute, or, if stationary, must have a volume at least 20 times the source volume.

The impact test prescribed by ISO 2919-1980(E) is more severe than the percussion test prescribed by 10 CFR 71.77(b). However, for specimen masses greater than 0.222 kg, the impact test of 10 CFR 71.77(a) will produce a higher kinetic energy at impact than will the alternate test prescribed by ISO 2919-1980(E).

The heat test prescribed by ISO 2919-1980(E) is more severe than that prescribed by 10 CFR 71.77(d).

## METHODS FOR QUALIFYING A SPECIAL FORM RADIOACTIVE MATERIAL

A necessary, but not sufficient condition for qualifying a special form radioactive material is to perform the tests on the material specified in 10 CFR 71.77 and verify by the tests specified in 10 CFR 71.75 that the radiation releases of 0.05  $\mu$ Ci from the damaged specimen has not been exceeded. Additional specifications for the conduct of the tests given in 10 CFR 71.77 are given in ISO 2919-1980(E) as well as in IAEA Safety Series No. 37 (International Atomic Energy Agency 1987). Also, additional specifications for the conduct of the tests given in 10 CFR 71.75 to determine the radioactive release from the damaged specimens are given in ISO/TR 4826-1979(E) as well as in IAEA Safety Series No. 37.

Testing may not be the most desirable method to qualify a special form radioactive material especially if a small number of units of the special form are to be fabricated and/or shipped. An option for qualifying of a special form radioactive material by analysis may be desirable. Qualification of special form by analysis is allowed in 49 CFR 173.476(c)(3), but is not addressed in 10 CFR 71. Some of the issues in determining the method of qualification (by test or by analysis) include the degree of knowledge of the properties of the material for the mathematical simulation of the tests prescribed in 10 CFR 71.77 as well as the ability to translate the results of the analysis into the acceptance criteria defined by 10 CFR 71.75(b) through (e). These issues are discussed below.

### Material Property Issues

Radioactive materials are usually produced by bombarding a material composed of stable nuclei with neutrons (usually from a fission source) or with energetic ions. The fission neutrons and alpha particles from (n, $\alpha$ ) reactions will cause atom displacements within the material. The effect of energetic particle irradiation is to radiation harden the material by increasing the yield strength and tensile strength of a metal. However, the yield strength increases more than the tensile strength which results in the embrittlement of the material. For body center cubic materials, the nil-ductility temperature will increase with the neutron fluence which results in the material becoming brittle at higher temperatures. In addition, the neutrons can cause void formation in the material which causes swelling of the material.

Helium produced by the decay of alpha emitting radioisotopes or by the capture of neutrons by (n, $\alpha$ ) reactions may also damage the special form radioactive material. The helium production in special form radioactive material may cause swelling and embrittlement of, and internal stresses in the material. In addition, neutron capture in the special form radioactive material may cause the formation of new radionuclides in the material.

The production and decay of radionuclides in the special form radioactive material will result in a change in the chemical composition of the material. This change in the material chemical composition can cause a change of the physical and mechanical properties of the material, as a function of time. For example,  $^{137}\text{CsCl}$  is frequently encapsulated and qualified as a special form radioactive material to be used as a gamma radiation source. The  $^{137}\text{Cs}$ , with a half-life of 30 years, undergoes radioactive decay to  $^{137}\text{Ba}$ . Because of the radioactive decay of the  $^{137}\text{Cs}$ , the composition of the source will change as a function of time. The quantity of  $\text{CsCl}$  will decrease, while the quantity of  $\text{BaCl}_2$  will increase. As the source composition changes, the physical properties of the source material will change. For example, the melting point will decrease with a decrease in the percentage of  $\text{CsCl}$  of the source contents until a eutectic composition of the  $\text{Cs}$ ,  $\text{Ba}$ , and  $\text{Cl}$  atoms is achieved. This change in the melting point can be substantial.

The storage environment of a special form radioactive material can also affect the material properties. Special form radioactive material may chemically and/or electro-chemically react with the storage environment. These reactions, over a period of time, may change the composition of the containment portion of the special form radioactive material, with resultant changes in the mechanical and physical properties of the material. Chemical processes include the formation of stable compounds and the selective leaching of constituent alloying materials from the special form radioactive material. Electro-chemical processes include the interaction of different materials, or different regions of the same material, coupled by an electrically conducting environment, causing pitting on the surface of a material. Examples of the types of corrosion include, but are not limited to, intergranular corrosion, such as the attack by liquid metals on austenitic stainless steels; stress corrosion; and hydrogen embrittlement. Corrosion often decreases a material's strength and ductility, as well as its functional lifetime. Corrosion rates are not only a function of the materials and the environment, but also a function of the thermodynamic state system, such as the temperature, stress level, and heat treatment of the component materials.

For example,  $^{137}\text{CsCl}$  is frequently encapsulated in a stainless steel and qualified as special form radioactive material. When properly stored at room temperature, the corrosion rate of the  $\text{CsCl}$  contents on the stainless steel may be negligibly small. However, if the special form radioactive material is transported or operated so that the temperature increases several hundred degrees, the corrosion rate increases significantly, and in the extreme, may disqualify the special form radioactive material in a short period of time compared to storage at room temperature.

The ionizing radiation from the special form radioactive material can cause chemical changes in a fluid storage environment by breaking chemical bonds (radiolysis) or, in the case of neutrons, by transmutation of the atoms that make up the fluid. In addition, the excitation of electrons in the storage environment may exacerbate the chemical activity between the environment and the special form radioactive material, increasing corrosion rates in the system. Thus for long-term storage, the material's mechanical properties must be known after a long exposure to the synergistic effects of the storage environment and ionizing radiation from the radioactive material, as well as in the "as-received" conditions.

Fabrication processes can influence the properties of a special form radioactive material. Fabrication of radioactive material to produce special form radioactive material may require special processes due to the activity of the material. Machining of a candidate special form radioactive material that emits short-ranged particles, such as alpha particles, is often performed in glove boxes to prevent the ingestion of radioactive particulates. For health and safety considerations, all machining, forming, joining, and inspecting processes on intense sources of penetrating radiation may be required to be performed remotely using master-slave manipulation. Remote handling processes increase the risk of damage to a special form radioactive material. Remote fabrication processes increase the potential for the production of defective parts. Remote inspection processes increase the potential for failure to detect defective parts. Thus, the manufacturer of a special form

radioactive material must be subjected to a comprehensive quality assurance program including, but not limited to, fabrication, handling, and inspection processes.

#### Mathematical Simulation Results and Compliance with 10 CFR 71.75

The mathematical simulation of the tests prescribed in 10 CFR 71.75 and described in 10 CFR 71.77 can be performed with digital computers using finite element/finite difference computer codes. These codes are used to analyze Type B packages for conformance to the accident conditions prescribed in 10 CFR 71.51 and described in 10 CFR 71.73.

The mathematical simulation of the tests described in 10 CFR 71.77 must use linear elastic analysis to compute the material stress response to the dynamic loading from the specified tests. The steady state thermal stresses due to the temperature gradients within the material should also be calculated using linear elastic theory.

The material properties used in the mathematical simulation of the tests and the acceptance criteria should be listed in ASME (American Society of Mechanical Engineers) Boiler and Pressure Vessel Codes, Section III, Appendix I and presented in detail in Section II, Parts A, B, and C. For materials not specified in the Code or irradiated materials, property data submitted substantially in accordance with the requirements of Code Section III, Appendix IV-1400 may be suitable for use.

The maximum allowable release of 0.05  $\mu\text{Ci}$  of material follow each test on a special form radioactive material per 10 CFR 71.75 is closer to the maximum of  $10^{-6}$   $\text{A}_2$  per hour release rates for a Type B material under normal conditions than the  $\text{A}_2$  per week releases for a Type B material under accident conditions as specified in 10 CFR 71.51. Thus the design criteria for special form radioactive material should be the same as given in Reg. Guide 7.6 (U.S. Nuclear Regulatory Commission 1978) for a shipping container for a Type B quantity of radioactive material under normal conditions. This design criteria is similar to the criteria for Level A Service Limits of Section III of the ASME Boiler & Pressure Vessel Code.

#### RECOMMENDATIONS TO MINIMIZE THE ISSUES FOR QUANTIFYING A SPECIAL FORM RADIOACTIVE MATERIAL

Radioactive materials are usually produced by bombarding a material composed of stable nuclei with neutrons (usually from a fission source) or with energetic ions. A radioactive material may have a chemical composition identical to an unirradiated material, but because of the radiation damage to the material during production, the radioactive material may have substantially different mechanical properties than the unirradiated material. The decay of radionuclides in the special form radioactive material will result in a change in the chemical composition of the material. This change in the material chemical composition can cause a change of the physical and mechanical properties of the material, as a function of time. Since the mechanical properties of an irradiated material as a function of radiation dose (or damage) and decay time are usually unknown, it is prudent to encapsulate a radioactive material with a well-characterized material to create a special form radioactive material.

Unencapsulated irradiated material may qualify as a special form radioactive material for shipment in Type B containers. To qualify as such, irradiated materials must demonstrably meet the special form test conditions because unirradiated material with identical chemical composition can have substantially different physical properties. Also, corrosion products or crude may be part of the irradiated material and must be evaluated in the testing.

A prudent approach for qualification of a special form radioactive material is to perform the tests specified in the Federal Regulations if the candidate radioactive material is unencapsulated or encapsulated by an irradiated material. Design by the mathematical simulation of the tests specified by the Federal Regulations is most appropriate if the radioactive material is encapsulated in a well characterized, unirradiated material. Design by mathematical simulation may also be appropriate for the cases where the encapsulation material has been irradiated or for the unencapsulated

material, if, and only if, the properties of the irradiated material are well known for the irradiated state of the material, or can be calculated with a high enough degree of confidence to satisfy a review of the design per Fisher et al (1988).

#### SPECIAL FORM AND TYPE B PACKAGES

A qualified special form radioactive material will contain its radioactivity in normal or accident conditions. Thus the containment issues are similar to those of the packaging of radioactive material for shipment or storage, especially if the special form radioactive material consists of a radioactive material encapsulated by an unirradiated material.

The current 10 CFR 71 and IAEA Safety Series No. 6 standards for Type B quantities of radioactive material do not address the implications of special form radioactive material on the containment requirements of the package. Thus, as current regulations stand, there is no differentiation between the containment requirements for a package containing a Type B quantity of a normal radioactive material vis-a-vis the same quantity of a special form radioactive material. However, a special form radioactive material can satisfy the containment requirements for a Type B package if it can be demonstrated that its properties will satisfy the test requirements specified in 10 CFR 71.71 and 10 CFR 71.73 at temperatures as low as  $-29^\circ\text{C}$ , with the activity release rate less than that specified in 10 CFR 71.51.

For credit to be given to the containment structural requirements for a Type B package with special form radioactive material contents, the following acceptance criteria are necessary, but not sufficient.

- The tests on the package specified in 10 CFR 71.71 and 10 CFR 71.73 shall not produce static or dynamic structural loads, or cause such loads to be produced in the special form radioactive material greater than those specified in 10 CFR 71.77. The static and dynamic structural response of the special form radioactive material to the loads applied to the special form by the package response to the loads of 10 CFR 71.71 and 10 CFR 71.73 must not exceed the yield strength of the special form material, or cause either brittle fracture or, in the case of a special form radioactive material formed by encapsulation, leak-before-break at stresses below the yield strength.
- The thermal tests on the package specified in 10 CFR 71.71 and 10 CFR 71.73 shall not produce thermal loads, or cause such loads to be produced in the special form radioactive material greater than those specified in 10 CFR 71.77(d). The response of the special form radioactive material to the thermal loads applied from the package response to the thermal loads of 10 CFR 71.71 and 10 CFR 71.73 must not exceed the temperature that would be produced by the application of the heat test of 10 CFR 71.77(d) to the unpackaged special form radioactive material.

If the package design and contents conform to the above acceptance criteria the only requirement on the package containment is that, under the test of 10 CFR 71.71 and 71.73, the package cannot lose one of the units of the special form radioactive material contents.

No credit for the form of the radioactive contents can be taken on the containment requirements of the package if above criteria cannot be demonstrated. For this case the package containment must conform to the requirements of 10 CFR 71.51, without consideration of the form of the contents.

#### CONCLUSIONS

A radioactive material may be qualified as a special form radioactive material if it complies with 10 CFR 71 or 49 CFR 173. The qualification may be performed by either tests and/or analysis. Because the properties of irradiated materials are not well known as a function of radiation dose and vary as a function of time after irradiation, it is prudent to encapsulate the radioactive material in a

well-characterized material to create the special form radioactive material. The tests prescribed in 10 CFR 71 can be performed or mathematically simulated on the encapsulated material. The acceptance criteria for the tested material are specified in 10 CFR 71. The acceptance criteria for the mathematical simulation are based on a linear elastic analysis using the criteria given in Reg. Guide 7.6 for normal operating conditions.

Unencapsulated irradiated material may also be qualified as a special form radioactive material by analysis (if the properties of the irradiated material are well known for the degree of radiation damage) or by test. However, the half life of the irradiated material should be long relative to the expected life of the special form material, or the concentration of the radioisotopes in the material is low to minimize the change in material properties with time.

Credit can be given to the containment structural/thermal requirements for a Type B package with special form radioactive material contents under certain conditions. These conditions include the restriction that the response of the package to the tests in 10 CFR 71.51 will not cause the contents to exceed the test conditions prescribed by 10 CFR 71.75.

#### ACKNOWLEDGEMENTS

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