**September 18-23, 2016, Kobe, Japan**

## **Special Form Testing of Sealed Source Encapsulation for High-Alpha-Activity Actinide Materials<sup>1</sup>**

**Paper No. 5044** 

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#### **Abstract**

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Beginning in 2008, a new type of sealed-source encapsulation package was developed and tested by Oak Ridge National Laboratory (ORNL). These packages contain high-alpha-activity actinides and are regulated and transported in accordance with the requirements for DOT Class 7 hazardous material. The DOT provides specific regulations pertaining to special form encapsulation designs. The special form designation indicates that the encapsulated radioactive contents have a very low probability of dispersion even when subjected to significant structural events. The use of special form designs simplifies the delivery, transport, acceptance, and receipt processes. It is intended for these sealed-source encapsulations to be shipped to various facilities making it very advantageous for them to be certified as special form. To this end, DOT Certificates of Competent Authority have been sought for the design suitable for containing high-alpha-activity actinide materials. This design consists of the high-alpha-activity material encapsulated within a triangular zirconia canister, referred to as a ZipCan, tile that is then enclosed by a spherical shell. The spherical shell design, with ZipCan tile inside, was tested for compliance with the special form regulations found in

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#### 49 CFR 173.469.

The spherical enclosure was subjected to 9-m impact, 1-m percussion, and 10-min thermal tests at the Packaging Evaluation Facility located at the National Transportation Research Center in Knoxville, Tennessee and operated by ORNL. Before and after each test, the test units were subjected to a helium leak check and a bubble test. The ZipCan tiles and core were also subjected to the tests required for ISO 2919:2012(E), including a Class IV impact test and heat test and subsequently subjected to helium leakage rate tests  $[49 \text{ CFR } 173.469(a)(4)(i)]$ . The impact-tile test unit contained a nonradioactive surrogate; however, the thermal test unit contained a radioactive source. This paper describes the regulatory special form tests and presents detailed impact and leak test results that demonstrate that the sealed source encapsulation designs satisfy the regulatory tests.

#### **Introduction**

Since the 1940s, Oak Ridge National Laboratory (ORNL) has been involved with the early development of regulations pertaining to the transport of radioactive shipments and regulatory testing. A testing capability to develop, support, and comply with transport regulations has been maintained at ORNL from the 1960s to the present day [1]. Early testing at ORNL primarily focused on acquiring information that would support the development of regulations for the International Atomic Energy Agency, as well as assist package designers in meeting radioactive material shipment requirements from the Code of Federal Regulations. For the past 15 years, ORNL package testing has taken place under the Package Testing Program (PTP) at the Package Evaluation Facility (PEF) located at the National Transportation Research Center (NTRC). Package testing and design support have been a key part of the PTP operations for more than 50 years. As a result, the PTP that manages the PEF provides a one-stop shop for design, testing, and certification of a wide variety of packages needed for the shipment of radioactive materials.

Special form materials are limited to those materials which, if released from a package, would present a hazard from direct external radiation and inhalation and ingestion hazards. Normally, a special form material is composed of a material with high physical integrity; therefore, radioactive material contamination is not expected under severe accident conditions. Occasionally a special form material is composed of a nondispersible solid form. However, most often, special form materials are hermetically sealed (encapsulated) into a durable metal capsule. The special form designation indicates that the encapsulated radioactive contents have a very low probability of dispersion even when subjected to significant structural conditions tests. Additionally, a special form certification indicates that the sealed encapsulation has been designed to maintain leak tightness after being subjected to the required impact, percussion, bending, and heat tests.

Beginning in 2008, a new type of sealed-source encapsulation package (SSE) was developed at ORNL. The transport of SSE packages containing radioactive material is subject to regulation by the U.S. Department of Transportation (DOT) with input from the U.S. Nuclear Regulatory Commission [2]. Additionally, the packages must be transported in accordance with the requirements set for DOT Class 7 hazardous material. DOT provides regulations pertaining to specific package contents categorized as special form encapsulation designs. These designs have been shown to simplify the delivery, transport, acceptance, and receipt process, which helps streamline the movement of SSEs to various facilities. Sealed-source encapsulation packages are intended to be shipped to various facilities throughout their lifetimes, making it advantageous for them to be certified as special form designs.

To this end, DOT certificates of competent authority (CoCAs) have been sought for SSE designs that are suitable for containing high-alpha-activity actinide material encapsulated with a triangular zirconia canister, referred to as ZipCan, tile that is then enclosed by a spherical shell. These new designs were tested for compliance with the regulations found in Title 49, Code of Federal Regulations, Section 173.469, *Tests for Special Form Class 7 (Radioactive) Materials (*49 CFR 173.469). All regulatory special form testing activities were conducted at NTRC under the PTP. This paper details the regulatory special form tests and results.

#### **Description of Test Units**

The ZipCan is a type of SSE. It is composed of an equilateral triangle made of various titanium layers (Figure 1). The length of the ZipCan is approximately 2 in. with a thickness of 0.25 in. Radioactive materials are added by depositing drops of a nitrate solution into the wells through the fill tubes. After deposition, the nitrate is converted to an oxide and then covered with potting cement. The final encapsulation is accomplished by welding a plug to the fill tube. Finally, a secondary encapsulation was added to the ZipCan to reduce the high surface alpha activity.



**Figure 1 ZipCan test unit.** 

A rectangular encapsulation geometry, dubbed "ZipCube," was developed to achieve functions not readily achieved by the ZipCan. The ZipCube is a titanium rectangle with two fill holes (Figure 2). The ZipCube has an approximate length of 1.4 in., width of 0.8 in., and an approximate thickness of 0.8 in. Radioactive materials are added by depositing drops of nitrate through the fill tubes. After deposition, the nitrate solution is converted to an oxide. Final encapsulation is accomplished by welding plugs into the fill holes.



**Figure 2 ZipCube test unit.** 

For some SSE designs, there is an outer titanium spherical shell, named "Homer," that serves as the principal encapsulation. The two hemispheres of the sphere are welded together to accomplish final encapsulation (Figure 3). Within this outer spherical shell, there is an internal framework that holds radioactive material (e.g., ZipCans and ZipCubes) in a fixed position. Approximate diameter of the sphere is 6 in.



**Figure 3 Spherical shell test unit.** 

## **Special Form Tests**

Two prototype ZipCans, ZipCubes, and spherical shells were tested to demonstrate compliance with the requirements of 49 CFR 173.469*.* Prototype 1 was loaded with a nonradioactive surrogate weight, and prototype 2 was loaded with radioactive oxide. The special form tests for the spherical shell are as follows:

• Impact Test [49 CFR 173.469 (b)(1)]

*The specimen must fall onto the target from a height of 9 m (30 feet) or greater. The target must be as specified in Sec. 173.465(c)(5).* 

• Percussion Test [49 CFR 173.469 (b)(2)]

*(i) The specimen must be placed on a sheet of lead that 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 drop of 1.4 kg (3 pounds) through 1 m (3.3 feet).* 

*(ii) The flat face of the billet must be 2.5 cm (1 inch) in diameter with the edges rounded off to a radius of 3 mm ±0.3 mm (0.12 inch ±0.012 inch).* 

*(iii) The lead must be of hardness number 3.5 to 4.5 on the Vickers scale and thickness not more than 25 mm (1 inch), and must cover an area greater than that covered by the specimen.* 

• Heat Test [49 CFR 173.469 (b)(4)]

*The specimen must be heated in air to a temperature of not less than 800 °C (1475 °F), held at that temperature for a period of 10 minutes, and then allowed to cool.* 

• Leak Test [49 CFR 173.469 (a)(4)(i)]

*Demonstration of leak tightness of*  $10^4$  *torr-1/s (1.3 x*  $10^4$  *atm-cm<sup>3</sup>/s) based on air at 25 °C (77 o F) and one atmosphere differential for solid radioactive content…* 

Two prototype ZipCubes and ZipCans were tested to demonstrate compliance with the requirements of 49 CFR 173.469 and ISO2919:1999(E), *Radiological protection-Sealed radioactive sources– General requirements and classification.* The special form tests for the Zipcube are as follows:

The 49 CFR 173.469 (d) requirement states:

*A specimen that comprises or simulates Class 7 (radioactive) material contained in a sealed capsule need not be subjected to—* 

*(i) The impact test and the percussion test of this section provided that the mass of the special form radioactive material is less than 200 g and it is alternatively subjected to the Class 4 impact test prescribed in ISO 2919, "Sealed Radioactive Sources—Classification"…* 

Since the ZipCan and ZipCubes weigh less than 200 g, they were subjected to a Class 4 impact test only as prescribed in ISO 2919:1999(E) in lieu of the percussion and impact tests.

The ISO 2919:1999(E) impact test is specified below:

## *7.4 Impact Test*

## *7.4.1 Apparatus*

*7.4.1.1 Steel hammer, the upper part of which is equipped with a means of attachment, and the lower part of which shall have an external diameter of (25±1) mm and a flat striking surface with its outer edge rounded to a radius of (3.0±0.3) mm.* 

*The center of gravity of the hammer shall lie on the axis of the circle, which defines the striking surface; this axis itself passing through the point of attachment. The mass of the hammer for each test class is given in Table 1.* 

*7.4.1.2 Steel anvil, the mass of which is at least ten times that of the hammer. It shall be rigidly mounted so that it does not deflect during impact and shall have a flat surface, large enough to support the entire sealed source.* 

According to Table 1 from ISO 2919:1999(E), the weight of the steel hammer for the Class 4 impact test shall be "*2 kg from 1 m or equivalent imparted energy."* Additionally, the ZipCube was subjected to leak test 49 CFR 173.469(a)(4)(i) and heat test 49 CFR 173.469(b)(4).

## **Test Matrix**

The following tables provide the sequence of tests and processes performed on each test unit. The numbers indicate the sequence order in which the process or test was performed on the test unit. The test units that were subjected to an impact test were tested without radioactive material. The test units that were subjected to the heat test were loaded with radioactive material.

<b>Test or Process</b>	<b>Test Unit</b>		
<b>Description</b>	$TU-1$	$TU-2$	
<b>Leak Test</b>			
<b>Impact Test</b> (ISO 2919)		2	
<b>Heat Test</b>			
<b>Leak Test</b>			

**Table 1 Sequence of tests and processes for the ZipCube** 

**Table 2 Sequence of tests and processes for the ZipCan** 

<b>Test or Process</b>	<b>Test Unit</b>		
<b>Description</b>	<b>TU-1</b>	$TU-2$	
<b>Leak Test</b>		1	
(ISO <b>Impact</b> Test 2919)		2	
<b>Heat Test</b>	2		
<b>Leak Test</b>	3	3	

<b>Test or Process</b> <b>Description</b>	<b>Test Unit</b>		
	$TU-1$	$TU-2$	
<b>Helium Leak</b> <b>Test</b>	1,3,5	1,3	
<b>Impact Test</b>	2		
<b>Percussion Test</b>	4		
<b>Heat Test</b>		2	

**Table 3 Sequence of tests and processes for the spherical shell** 

#### **ISO 2919 Impact Tests, ZipCan and ZipCube**

As shown in Table 1 and 2, the ZipCan and ZipCube were subjected to the ISO 2919 impact test. The impact tests were carried out at the indoor drop pad at the NTRC. This drop pad has a total mass of ~13.6 metric tons and meets the specifications for the impact test target (see *Design and Certification of Targets for Drop Testing at the NTRC Package Research Facility Rev. 0,* May 2003, ORNL/NTRC-001. The ZipCan and ZipCube test unit were placed at the center of the indoor drop pad (Figure 4). A 1-in.-diam steel billet, weighing 2.0 kg, was raised to a height of 1 m directly above the vertex using a calibrated meter stick to measure the drop height. When released, the billet impacted the vertex for the ZipCube and impacted the top surface for the ZipCube. The impact of the billet resulted in a slight indentation and deformation, as shown in Figure 5 for the ZipCan and Figure 6 for the ZipCube. After the impact test, the test units were subjected to a helium leakage rate and a bubble test.



**Figure 4 Impact billet 1 m above the ZipCan and ZipCube.** 



**Figure 5 ZipCan vertex impact test result.** 



Before Impact



After Impact

## **Figure 6 ZipCube ISO impact test result.**

#### **Heat Test Results, ZipCan and ZipCube**

Since the heat test unit contained radioactive materials, the thermal tests were completed in a glove box in a radiological facility at ORNL (Figure 7). A tube furnace was used for the heat test. The furnace was preheated for a period of 3 h at 850 °C to ensure full thermal soak. After the 3-h preheat, the furnace door was opened and the 10-min special form test was started (Figure 8 and Figure 9). After the 10-min period, the test unit was removed from the furnace and allowed to cool under ambient conditions (see Figure 10 and Figure 11). The test unit was subjected to a helium leak test and bubble after the heat test.



**Figure 7 Muffle furnace used in the heat test.** 







**Figure 9 Thermal test temperature profile for ZipCube.** 



**Figure 10 ZipCan post heat test.**



**Figure 11 ZipCube post heat test.** 

#### **9-Meter Impact Test, Homer**

Test Unit 1 of the Homer design was subjected to the 49 CFR 173.469(b)(1) impact test. This test was carried out at an indoor facility with a 9-m pit located at ORNL. A steel plate was placed on top of a concrete slab and used as the unyielding impact target. The weight of the steel plate was 510 kg, and it meets the specifications for the impact test target. There was no specified orientation for the impact test since the test unit is a sphere. A 30 ft plumb bob was used to measure the drop height. The unit impacted the steel plate target, bounced, and re-impacted the steel plate target a second time. Subsequent inspection of the test unit showed minor scuff marks at the impact location (Figure 12). After the impact test, the test unit was subjected to a helium leakage rate test and a bubble test.



**Figure 12 Homer 9-m impact test.** 

#### **Percussion Test, Homer**

Test unit 1 was subjected to the 49 CFR 173.469 percussion test. The percussion test was carried out at an indoor facility with a 9-m pit located at ORNL. The mass of the floor was assumed to be an unyielding surface because an engineering judgment was made that the target mass to test unit mass ratio was greater than 10:1. A  $12 \times 12 \times 1$ -in.-thick sheet of pure lead (ASTM B749-97) was placed on the indoor drop pad, and TU-1 was placed on top of the lead sheet. A 1-in.-diam steel billet, weighing 3.14 lb (1.402 kg), was raised to a height of greater than 1 m (3.3 ft) directly above TU-1 and released such that the billet directly impacted TU-1. The measurement of the height of the billet above TU-1 for the percussion test is shown in Figure 13.

When released, the billet appeared to impact TU-1 squarely on the sphere. The impact of the billet resulted in a slight blemish and deformation of the sphere outer shell (see Figure 14). After the percussion test, TU-1 was subjected to a helium leakage rate and a bubble test.



**Figure 13 Homer percussion test setup.** 



**Figure 14 Homer percussion test impact result.** 

#### **Heat Test Results, Homer**

The 49 CFR 173.469(b)(4) Heat Test was performed on TU-2 in a small bench-top industrial furnace located inside a fume hood at ORNL (see Figure 15). The test was performed according to the procedure outlined in the *Test Plan for the Special Form Qualification Testing of the Nitrate Deposition Sphere (NDS) Radiation Signature Training Device*, ORNL/NTRC-062, and testing activities and results were recorded on Test Forms 2, 3, and 4 from the test plan. Two calibrated Type K thermocouples were inserted into the working space within the furnace, and these thermocouples were monitored through the use of a calibrated Fluke 52 K/J Thermometer, property number M211562, and their temperature readings were manually logged.



**Figure 15 Heat test furnace with test unit.** 

The furnace was preheated at 1025  $\degree$ C for 10 h prior to the test to ensure that the furnace was fully heat soaked and would remain above the required test temperature of 800 °C when the test unit was inserted. The temperature in the furnace was recorded three times in the hours just prior to the test to ensure steady readings near the set point had been reached. One hour prior to testing the temperature within the furnace had stabilized, with the two thermocouples reading  $1024 \degree C$  and  $1024 \degree C$ . The temperature within the furnace was again recorded just prior to the opening of the furnace for insertion of TU-2. The temperatures recorded at that time were 1024 °C and 1024 °C (see Figure 16).

To ensure that the required testing temperature of 800 °C was maintained, the set point used for the preheat (1025 °C) was maintained for the duration of the test. After the final pre-heat temperature reading, the furnace door was opened, TU-2 was inserted into the furnace in a ceramic pan, and the furnace door was closed. The ceramic pan was made of alumina, which was chosen specifically for its low thermal mass, ensuring it would not act as a heat sink during testing. Inside the alumina pan was a transparent quartz ring that TU-2 rested upon. Figure 17 shows TU-2 just prior to insertion into the furnace for the heat test. The furnace remained above the test temperature of 800 °C after test unit insertion, so the 10-min test duration began immediately once the furnace door was closed. Figure 18 shows TU-2 in the furnace for the 10-min heat test. Temperature readings from both thermocouples were manually logged every 30 s for the 10-minute test duration.

After the heat test was completed, the furnace door was opened, and the pan containing TU-2 was removed from the furnace. It was observed that there was no apparent distortion of the test unit due to the heat test. TU-2 was subsequently allowed to cool naturally. Figure 19 shows TU-2 after removal from the furnace. TU-2 did appear to have a darker color after the heat test likely due to minor oxidation of its titanium surface. After the thermal test, TU-2 was subjected to a helium leakage rate test as described.



**Figure 16 Homer thermal test temperature profile.** 



**Figure 17 Homer test unit inserted into the furnace.** 



**Figure 18 Homer test unit removed from the furnace.** 



**Figure 19 Homer post heat test passive cooling.** 

## **Leak Rate Testing**

#### Evacuated Envelope (with Back Pressurization)

Leak rate tests that meet the test requirements of 49 CFR 173.469 (a)(4)(i) were performed individually on each test unit after each test. The leak rate tests were performed using American National Standards Institute (ANSI) N14.5-1997, *American National Standard for Radioactive Materials–Leakage Tests on Packages for Shipment*, Table A.1, Test Description A.5.5, *Evacuated Envelope (with back pressurization)* and Test Description A.5.6 *Gas bubble techniques*. The ANSI document indicates that the back pressure method "is ideal for welded capsules from very small sizes up to the sizes limited by the dimensions of the pressurizing chamber," that the "nominal test

sensitivity =  $10^{-3}$ --  $10^{-8}$  ref-cm/s," and that the bubble test method is used for hermetically sealed test specimens.

Section A.5.5 of ANSI N14.5-1997, *Evacuated Envelope with Helium Back Pressure*, of the ANSI document references ASTM E493, *Standard Test Methods for Leaks Using the Mass Spectrometer Leak Detector in the Inside-Out Testing Mode*. This standard provides the method for converting a measured leak rate using the evacuated envelope with helium back-pressure method into the standardized leak rate that must be compared with the pass/fail criteria stated in 49 CFR 173.469(a)(4)(i), which is 10<sup>−</sup>4 torr-L/s (1.3 × 10−4 atm-cm3/s).

The equation provided in Sect. 11.1.9 of ASTM E493 is:

$$
S_1 = (P_e/P_a) * (1 - e^{(-3600^*a^*T)}) * (e^{(-a^*t)}) * L \quad , \tag{1}
$$

where:



Since we are measuring  $S_1$  and wish to solve for L, the equation requires an iterative solver to find the solution. The equation was solved using spreadsheet iteration. Note that the ASTM International standard uses the term "bombing," whereas the ANSI standard uses the term "back pressure." These terms have the same meaning. To solve Equation 1, the internal volume (i.e., void space) within the test units must be known. For the test units, this internal volume consisted of accessible internal void spaces. The internal void was measured from the computer-aided design models.

The test units were leak tested at ORNL by a certified American Society for Nondestructive Testing Level II and Level III leak tester before and after each special form test. The test apparatuses used employed a mass spectrometer configured to detect helium, a calibrated helium leak to calibrate the system, and two separate vessels: one for helium back pressurization and one for the subsequent helium leakage rate testing under vacuum conditions. Figure 20 shows a schematic of the system used for helium back pressurization, and Figure 21 shows a schematic of the system used for the helium leakage rate test.



## **Figure 20 Diagram of the helium back pressurization system.**



## **Figure 21 Diagram of the helium leak testing system.**

#### Gas bubble technique

The gas bubble test was done using the methods described in ANSI N14.5-1997, *American National Standard for Radioactive Materials–Leakage Tests on Packages for Shipment,* Table A.1 test description A.5.6 (b), Vacuum Bubble. The method involves immersing the test unit in a liquid and then producing a vacuum above the liquid (e.g., water/glycol or isopropyl alcohol) in which the test item is submerged. A leak is indicated by a stream of bubbles (see Figure 22). This method is applicable to welded capsules. The nominal test sensitivity is  $10^{-3}$  ref-cm<sup>3</sup>/s ( $10^{-4}$  Pa-m<sup>3</sup>/s).



**Figure 22 Vacuum bubble test.** 

#### Leak Test Results, ZipCube

The ZipCube test unit was subjected to a helium leak test and a bubble leak test after each regulatory test to ensure that the welds had not failed. Table 4 list the results of the helium leak test and bubble test. For the ZipCube leak test, the following values were used:







## Leak Test Results, Homer

The Homer test unit was subjected to a helium leak test and a bubble leak test after each regulatory test to ensure that the welds had not failed. Table 5 list the results of the helium leak test and bubble test. For the Homer leak test, the following values were used:

The He leak testing the following values were used:



 $V = 607$  cc



#### **Table 5 HOMER leak rate test variables and results for TU-1 and TU-2**

#### Leak Test Results, ZipCan

The ZipCan test unit was subjected to a helium leak test and a bubble leak test after each regulatory test to ensure that the welds had not failed. Table 6 and Table 7 list the results of the helium leak test and bubble test. For the ZipCan leak test, the following values were used:

	<b>Test Unit</b>			
<b>Parameter</b>	$TU-2$		$TU-1$	
	<b>Leak Test 1</b>	<b>Leak Test 2</b>	<b>Leak Test 1</b>	<b>Leak Test 2</b>
Void Space - V	1.057	1.057	1.057	1.057
(cc)				
<b>Bombing Pressure - Pe</b>	30.0	50.0	50.0	30.0
(psig)				
<b>Atmospheric Pressure - Pa</b> (psia)	14.69	14.69	14.69	14.69
<b>Bombing Time - T</b> (h)	0.5	$\mathbf{1}$	$\mathbf{1}$	0.5
Time between bombing and $testing - t$ (s)	$<$ 3,600	< 1,800	<15,300	3,600
Measured leak rate $(cc/s) - S1$ (atm-cc He/s)	$2.0 \times 10^{-7}$	$5.0 \times 10^{-9}$	$< 4.6 \times 10^{-6}$	$<$ 3.0 $\times$ 10 <sup>-6</sup>
$a = L/V$ $(s^{-1})$	$< 9.46 \times 10^{-5}$	$< 9.46 \times 10^{-8}$	$< 9.46 \times 10^{-5}$	$< 9.46 \times 10^{-5}$
<b>Standardized Leak Rate - L</b> (atm-cc He/s)	$< 1.0 \times 10^{-4}$	$< 1.0 \times 10^{-7}$	$< 1.0 \times 10^{-4}$	$< 1.0 \times 10^{-4}$

**Table 6 ZipCan leak rate test variables and results for TU-1 and TU-2** 



# **Table 7 ZipCan bubble test variables and results for TU-1 and TU-2**

#### **Conclusions**

Two prototype ZipCan test units, a ZipCube test unit, and a spherical shell test unit were subjected to the tests specified in 49 CFR 173.469, and ISO 2919:1999(E). All test units surpassed the leak rate criteria following each special form test. The special form testing process has shown that the design of the ZipCan, Homer, and the ZipCube meets the special form criteria.

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