Study on Water Leak-Tightness of Small Leaks

Takashi Miyazawa (1), Yuji Kasai* (2), and Masanori Aritomi (3)
(1) Mitsubishi Materials Corporation, 1-3-25 Koishikawa Bunkyo-ku Tokyo 112-0002 JAPAN
(2) Shibaura Institute of Technology, 307 Tameihara Fukasaku Omiya Saitama 330-0003 JAPAN
(3) Tokyo Institute of Technology, 2-12-1 Ohokayama Meguro-ku Tokyo 152-8550 JAPAN
(*: Presently with East Japan Railway Company)

ABSTRACT

The practical threshold for water leak-tightness of small leaks was determined by experimentation. Small leak samples were made and measurements were taken of the gas leakage rates and water leakage rates for the identical leak samples in order to identify the standardized leakage rate ($Pa{\cdot}m^3/s$ SLR) of a leak sample that did not permit water leakage. The results show that a leak hole corresponding to 10^{-5} $Pa{\cdot}m^3/s$ SLR does not permit water leakage under experimental conditions in this study. In addition, experiments with 1-inch cylinder valve leak samples made by scratching the valve stems were performed. This result assured that the criteria for the preshipment leakage rate test, $1x10^{-3}$ ref-cm^3/s, as prescribed in ANSI N14.5 is an appropriate value from the point of view of water leak-tightness for enriched uranium hexafluoride packages.

INTRODUCTION

Water leak-tightness is an important factor for a package that adopts moderation control such as packages for enriched uranium hexafluoride. For packages containing fissile material, a water immersion test under a head of water of 15 m or of 0.9 m for a period of eight hours is required as a test under accident conditions of transport. The package should not reach criticality by water intrusion under these conditions. Also IAEA regulation ST-1 [Reference 1] para. 677(b)(ii) prescribes tests to demonstrate closure of each package before each shipment for a fissile uranium hexafluoride package. It is possible to demonstrate water leak-tightness by a direct method such as hydrostatic pressure testing when the package is empty. But methods other than such direct demonstration methods are required for packages that have been filled with content. For such situations, a method to determine water leak-tightness by gas leakage measurement can be proposed. In this case, the quantitative criteria of the gas leakage rate is needed, for instance in the form of $Pa{\cdot}m^3/s$ SLR, corresponding to the threshold of water leak-tightness. Theoretically, water leak-tightness can be achieved when the surface tension of water through the leak hole overcomes the differential pressure exerted on the water as the driving force to move fluid. On the other hand, ISO12807 [Reference 2] states, “Because of their relatively high viscosity, liquids are considered not to be able to leak through fine capillaries which a gas might pass under molecular flow.”

In this study, first, leak samples with fine leak holes were made and gas leakage rates of these samples were measured to determine standardized gas leakage rates ($Pa{\cdot}m^3/s$ SLR). Then these leak samples were subjected to water leakage tests to observe if water leakage occurred or not. By these series of tests, the standardized gas leakage rates ($Pa{\cdot}m^3/s$ SLR) of the leak samples that did not permit water leakage could be identified practically, which is the objective of this study. Also in this study, leak samples of “1-inch cylinder valves” were made by scratching the valve stems artificially. The “1-inch cylinder valve” is the valve used for an enriched uranium hexafluoride package. Valve leak samples were subjected to the leakage measurements in the same manner as mentioned above to practically determine the standardized gas leakage rates of the valve leak samples that did not permit water leakage. The cause that determined the threshold of water leak-tightness was also discussed.

EXPERIMENT 1

Four leak samples made by inserting a stainless steel fine wire (diameters: 16, 50, 80 and 100 μm) between a flange and an elastomer O-ring (Figure 1) were subjected to series of the tests. In this leak sample, a hole occurs on either side where the wire goes in and through the O-ring seal. After measuring the air leakage rates of these samples and determining the standardized leakage rates, the leak samples were subjected to water leakage test to see if water leaked or not. The experimental parameters are listed in Table 1.

Gas Leakage test

Gas leakage rates were measured by pressure drop method (Figure 2). When air leakage occurs through the leak sample, the pressure of the inside the air tank decreases. The pressure inside the air tank was measured with the pressure sensor and recorded. The air tank was put in a water bath to keep the tank temperature stable. The experiment room was air-conditioned. The water bath temperature change was kept within 1˚C per day. The background air leakage from the equipment was less than $5\text{x}10^{-6}$ $\text{Pa·m}^3\text{/s}$ when the upstream pressure was 0.8 MPa (absolute). Measured air leakage rates are shown in Figure 3.

If the flow regime through the leak hole is viscous flow, the gas leakage rate can be expressed in the following equation.

$$Lu = \frac{\pi(P_u^2 - P_d^2)D^4}{256\mu a} \qquad (\text{Pa·m}^3\text{/s}) \qquad \ldots \text{ Equation 1}$$

(Pu: Upstream pressure (Pa), Pd: Downstream pressure (Pa), D: Diameter of leak hole (m), a: Length of leak hole (m), μ: Viscosity of gas (Pa·s))

Equation 1 can be rewritten as Equation 2 to express "Leak Path Characteristic Value" D^4/a.

$$\frac{D^4}{a} = \frac{256\mu L_u}{\pi(P_u^2 - P_d^2)} \qquad (\text{m}^3) \qquad \ldots \text{ Equation 2}$$

By substituting the measured leakage rate, the upstream and downstream pressure and the viscosity of gas, the leak path characteristic value D^4/a can be calculated by Equation 2. With this calculated D^4/a value, the leakage rate through the identical leak hole under another condition can be calculated by Equation 1. Since the leakage rate varies with upstream and downstream pressure, temperature and the kind of gas, "standardized leakage rate" (unit: $\text{Pa·m}^3\text{/s}$ SLR), i.e. the dry air at 298K leakage rate at upstream pressure of $1.013\text{x}10^{-5}$ Pa and downstream pressure 0 Pa is generally used to express the magnitude of a leak. Figure 4 shows the leak path characteristic values D^4/a calculated from measurement results shown in Figure 3. The reason D^4/a values decrease with differential pressure increase is considered to be because of deformation of the elastomer O-ring with upstream pressure increase. For calculating standardized leakage rates, D^4/a values at the pressure condition of Pu:0.2MPa (absolute) and Pd:0.1MPa (absolute) were adopted since the differential pressure of this condition is the same as that of standardized leakage rate condition (Pu:0.1MPa (absolute) and Pd:0MPa (absolute)). Calculated standardized leakage rates of leak samples are listed in Table 2. Equivalent diameters listed in Table2 were calculated by Equation 2 and the assumption that its leak hole length is equal to the width of O-ring groove, 3.2mm.

Water Leakage Teat

After determining the standardized leakage rates, leak samples were subjected to water leakage tests (figure 5). The air in the buffer tank was compressed and set at the upstream pressure. When water leaks through the leak sample, a head of water in pipe 1 connected to pipe 3 decreases while a head of water in pipe 2 does not change. The differential pressure between pipe 1 and pipe 2 is measured and recorded. The tests were carried out for at least 24 hours. The detection limit of this measurement was $7\text{x}10^{-13}$ m^3/s ($0.06cm^3$/24hours). The result of the water leakage test is shown in Table 2. Water leakage did not occur from the leak sample with a 16μm wire. The leak sample with a 50μm wire permitted water leakage when the differential pressure was over 0.5 MPa. The leak samples with 80μm and 100μm wires permitted water leakage under any pressure conditions.

In addition, two leak samples made of glass capillaries which had about the same magnitudes of standardized leakage rates as the 50μm wire – leak sample were subjected to water leakage tests in the same manner. Furthermore, stainless steel orifice samples that had about the same diameters as capillary samples were subjected to water leakage tests in the same manner. These test results were shown in Table 3. The 20μm diameter-capillary permitted water leakage under any pressure conditions. But the 10μm diameter-capillary did not permit water leakage under any pressure conditions. Stainless steel orifice leak samples (Diameter: 10.3μm and 19.3μm, thickness: 50μm) did not permit water leakage under any pressure conditions.

Findings

Under the upstream pressure of 0.2 – 0.8MPa (absolute) and the downstream pressure of atmospheric pressure, water leakage did not occur from the leak hole corresponding to $1.1\text{x}10^{-5}$ $Pa{\cdot}m^3/s$ SLR. The threshold of water leak-tightness is considered to exist between $1.1\text{x}10^{-5}$ $Pa{\cdot}m^3/s$ SLR and $1.7\text{x}10^{-4}$ $Pa{\cdot}m^3/s$ SLR under this experimental condition. In addition, the orifice leak sample with a diameter of 19.3μm did not permit water leakage. In this case, the standardized leakage rate was as much as an order of 10^{-3} $Pa{\cdot}m^3/s$ SLR. On the other hand, from the point of view of opening size, the threshold of water leak-tightness seems to lie in the range of around 10 – 20μm diameter opening according to the results with these three kinds of leak samples in Experiment 1.

EXPERIMENT 2

As mentioned earlier, since the enriched uranium hexafluoride package adopts moderation control, water leak-tightness is a very important issue. The containment boundary of the enriched uranium hexafluoride package is a 30B cylinder, whose containment ability is assured strictly on occasions of fabrication, washing, repairing, and periodical inspection in compliance with tests prescribed in ANSI N14.1 [Reference 4]. However, the matter of interest is the water leak-tightness of the valve after filling the 30B cylinder with content. In this study, leak samples of the 1-inch cylinder valves (Figure 6) used for the enriched uranium hexafluoride packages were made by scratching the valve stems artificially and were subjected to leakage measurements in the same manner as in Experiment 1 to practically determine the water leak-tightness threshold. After a scratch was made on the valve stem (Figure 7), the stem was screwed into the valve body with a torque of 55 foot-pounds prescribed in ANSI N14.1. Four valve leak samples were prepared in this manner. After air leakage rates were measured with these valve leak samples, they were subjected to water leakage tests.

Gas Leakage Test

Measured gas leakage rates are shown in Figure 8. Leak path characteristic values D^4/a were calculated (Figure 9) with these measured results and Equation 2. D^4/a values of Sample #1 and Sample #2 are almost constant regardless of differential pressure. This explains that air flow regime is viscous flow expressed in Equation 1. On the contrary, D^4/a values of Sample #3 and Sample #4 decrease with differential pressure increase. Figure 10 shows the volumetric flow rates at the upstream pressure converted from measured gas leakage rates shown in Figure 8. The volumetric flow rates of Sample #3 and Sample #4 at upstream pressure reach constant values as upstream pressure increases. This explains that the flow regime transits from viscous flow to choked flow. In order to express this flow regime strictly, the flow model of "viscous flow + exit loss" is needed [Reference 6]. Therefore, the "viscous flow + exit loss" flow model was adopted to calculate standardized leakage rates for valve samples #3 and #4.

Water Leakage Test

The water leakage test results of valve leak samples are shown in Table 4. No water leakage from Sample #1 and Sample #2 was detected. Water leakage from Sample #3 and Sample #4 occurred under every pressure condition.

Findings

From this experiments, water leakage was discovered not to occur from the valve leak sample with a standardized leakage rate is $8.9\text{x}10^{-5}$ Pa·m^3/s SLR under the upstream pressure condition of 0.2 – 0.8MPa (absolute) and the downstream pressure of atmospheric pressure. $8.9\text{x}10^{-5}$ Pa·m^3/s SLR ($8.8\text{x}10^{-4}$ ref-cm^3/s) is almost the same value as the criteria for the preshipment leakage rate test, $1\text{x}10^{-3}$ ref-cm^3/s, as prescribed in ANSI N14.5 [Reference 7].

DISCUSSION

As stated earlier, if the mechanism of water leak-tightness is due to just the water surface tension, pressurization as little as 0.03MPa should theoretically make water leak from a circular leak hole with a cross section diameter of 10µm. However, in this study, water leakage did not occur from leaks with a cross section diameter of 10µm even under the upstream pressure of 0.8MPa (absolute).

After water leakage tests were performed, the valve leak samples #1 and #2 that did not allow water leakage were dried in a desiccator for 24 hours and subjected to the gas leakage test again. But not even the gas leakage occurred anymore. The same result was also observed for the orifice leak sample that did not allow water leakage. These results suggest that a small amount of impurity (tiny particles) in the water plugged the leaks. The water used in the experiment was filtered and purified by ion-exchange resin before being supplied to the test section. But the dust in the laboratory room might have entered the test section when the leak sample was replaced and corrosion products from the piping or a tank might have also entered the water. The collection efficiency of the filter used in the experiments was 30-50% about 10µm-particles according to its specification. Then water was sampled from the test section and filtered with polycarbonate membrane filters that have 0.8µm pores. Particles collected on the membrane filter were observed with a microscope. Roughly 1 particle of 10-100µm range and roughly 10 particles of 1-10µm range per 1cm^3 of water were observed. Besides those particles, many tiny particles (which seem to be rust from the piping) less than the order of sub-micron size also existed. These sub-micron particles plugged polycarbonate membrane filter pores quickly too. However, neither those particles nor the large ones were at all visible to the naked eye.

The orifice leak sample that did not even let gas leak after being subjected to the water leakage test was observed with a microscope. Yellow plugging was observed. It is supposed that tiny rust particles and other kinds of particles plugged the leak quickly before the detectable change of head of water was observed in a water leakage test.

Water leak-tightness for packages containing fissile material is required under accident conditions of transport (IAEA regulation ST-1 para. 677). Water assumed under accident condition of transport is dirty water such as river water, swamp water, rain, and fire extinguishing water. In comparison to those kinds of water, the water used in the experiment was clean enough. Therefore, even though the mechanism of water leak-tightness is due to plugging, experiment results in this study are considered to be still more conservative than the case supposed on an accidental occasion.

However, even though there was a leak hole of 1 inch in diameter on the cylinder containing UF_6 and the cylinder was put in water, the leak hole would be plugged by insoluble material of monohydrated UO_2F_2 and metallic products. This results from the reaction of the UF_6 and water and the reaction of the resultant HF with the metal [Reference 8].

CONCLUSION

The practical threshold for water leak-tightness of a small leak was determined by experimentation. The results show that a leak hole corresponding to 10^{-5} $Pa\cdot m^3/s$ SLR does not permit water leakage. From the experiment with 1-inch cylinder valve leak samples made by scratching the valve stems, a leak corresponding to 8.9×10^{-5} $Pa\cdot m^3/s$ SLR did not permit water leakage. The mechanism of water leak-tightness is considered to be plugging by the tiny particles existing in water. In an actual case of accidental conditions, water is expected to contain many more particles than the water used in experiments in this study. Therefore, leak holes larger than those determined to be water leak-tight in this study can be assumed to prevent water leakage in the case of accidental conditions. In addition, it was assured that the criteria for preshipment leakage rate test, 1×10^{-3} ref-cm^3/s prescribed in ANSI N14.5 is an appropriate value from the point of view of water leak-tightness for enriched uranium hexafluoride packages.

Table 1 Leakage Test Experimental Parameters

Working Fluid	Air, Water (Room temperature)
Upstream Pressure (MPa (absolute))	0.2, 0.3, 0.4, 0.5, 0.6, 0.7, 0.8
Downstream Pressure (MPa (absolute))	0.1 (Atmospheric pressure)

Table 2 Water Leakage Test Results (1)

Inserted Stainless Steel Wire Diameter (μm)	Standardized Leakage Rate per Single Leak Hole ($Pa\cdot m^3/s$ SLR)	Leakage/ No Leakage	Equivalent Diameter per Single Leak Hole (μm)
16	8.9×10^{-6}	No Leakage	8
50	2.7×10^{-4}	Leakage Pu-Pd ≧ 0.5MPa	19
80	9.2×10^{-4}	Leakage Pu-Pd ≧ 0.1MPa	26
100	1.53×10^{-3}	Leakage Pu-Pd ≧ 0.1MPa	29

Table 3 Water Leakage Test Results (2)

Leak Sample	Standardized Leakage Rate ($Pa\cdot m^3/s$ SLR)	Leakage/ No Leakage
Capillary Diameter: 20μm Length: 7mm	1.7×10^{-4}	Leakage Pu-Pd≧0.1MPa
Capillary Diameter: 10μm Length: 7mm	1.1×10^{-5}	No Leakage
Orifice Diameter: 19.3μm Thickness: 50μm	5.9×10^{-3} (*)	No Leakage
Orifice Diameter: 10.3μm Thickness: 50μm	1.7×10^{-3} (*)	No Leakage

(*) Standardized leakage rate was calculated as a choked flow [Reference 3].

Table 4 Water Leakage Test Results (3)

Valve Leak Sample	Standardized Leakage Rate ($Pa\cdot m^3/s$ SLR)	Leakage/ No Leakage
Sample #1	2.1×10^{-5}	No Leakage
Sample #2	8.9×10^{-5}	No Leakage
Sample #3	3.3×10^{-3}	Leakage Pu-Pd≧0.1MPa
Sample #4	3.0×10^{-2}	Leakage Pu-Pd≧0.1MPa

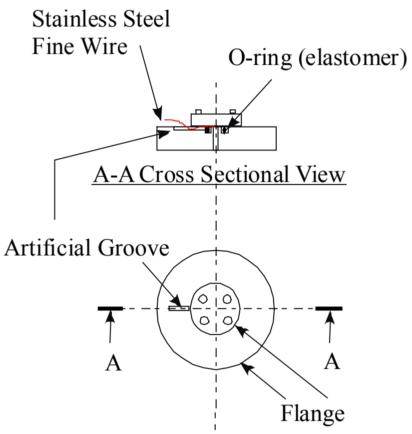

Figure 1 Schematic Diagram of Leak Sample Made with Elastomer O-ring and Stainless Steel Fine Wire

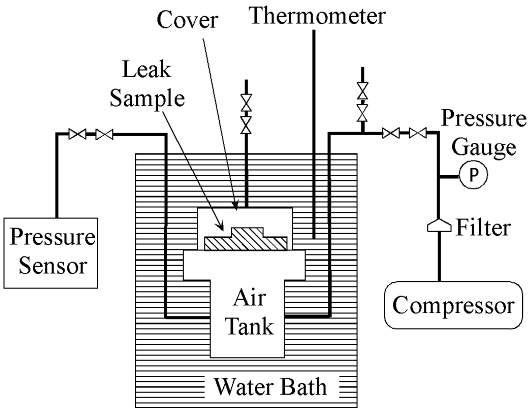

Figure 2 Schematic Diagram of Gas Leakage Test Equipment by Pressure Drop Measurement

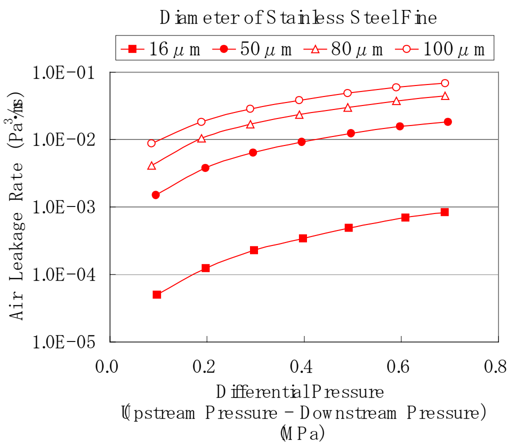

Figure 3 Measured Air Leakage Rates

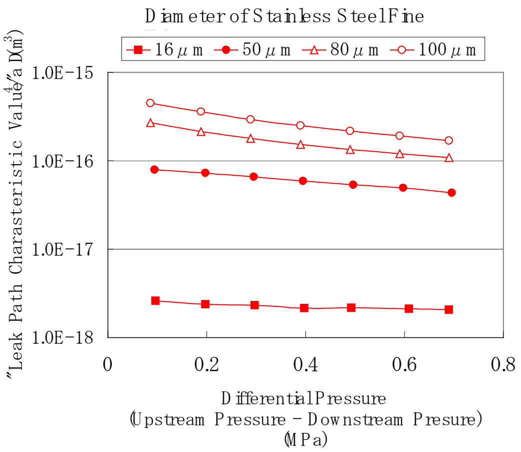

Figure 4 " Leak Path Characteristic Values " D^4/a

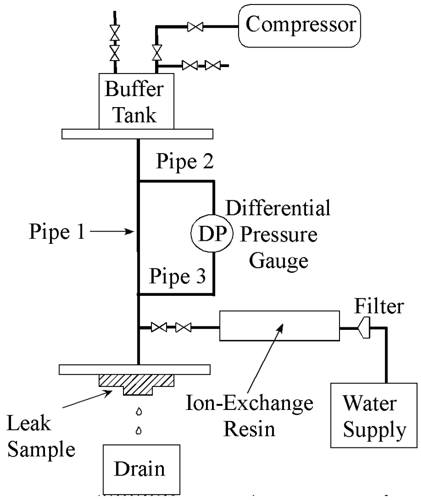

Figure 5 Schematic Diagram of Water Leakage Test Apparatus

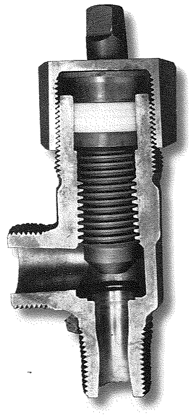

Figure 6 Cutaway View of 1-inch Cylinder Valve for 30B Cylinder [Reference 5]

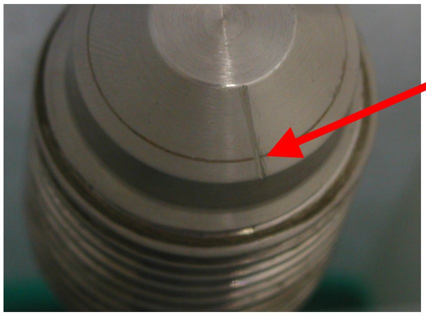

Figure 7 Artificial Scratch on 1-inch Cylinder Valve Stem

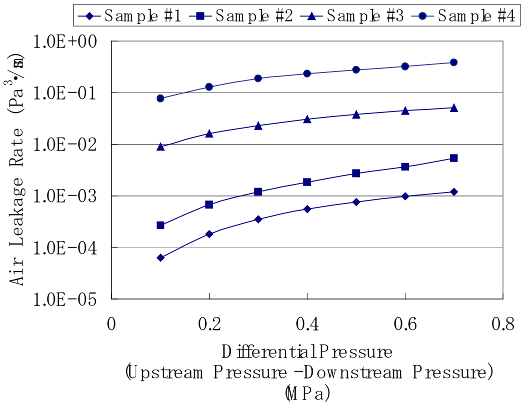

Figure 8 Measured Air Leakage Rates

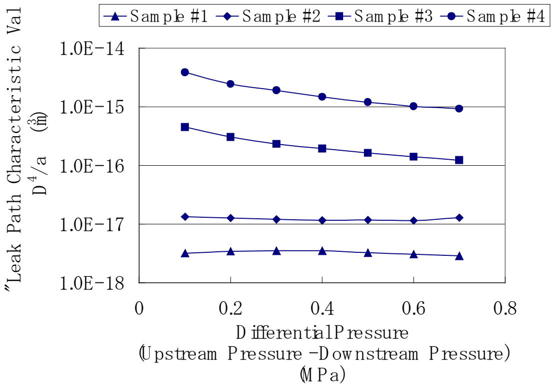

Figure 9 "Leak Path Characteristic Values" D^4/a

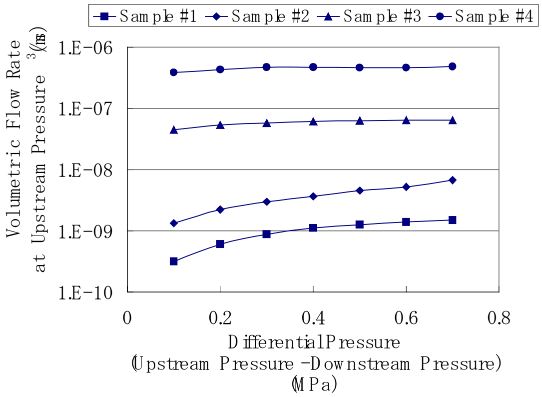

Figure 10 Volumetric Flow Rates at Upstream Pressure

REFERENCES

[1] Safety Standards Series No.ST-1 ”Regulation for the Safe Transport of Radioactive Material” 1996 Edition

[2] ISO 12807:1996(E) “Safe transport of radioactive materials – Leakage testing on packages”, pp67, 1996

[3] ANSI N14.5-1987 “American National Standard for Radioactive Materials - Leakage Tests on Packages for Shipment”, pp20-22

[4] ANSI N14.1-1995 “American National Standard for Nuclear Materials - Uranium Hexafluoride - Packaging for Transport”, pp19-20

[5] USEC-651 "The UF6 Manual - Good Handling Practices for Uranium Hexafluoride" Revision 8 January 1999, pp.44

[6] Donald J. Santeler, “Exit Loss in Viscous Tube Flow”, J. Vac. Sci. Technol. A 4(3) May/June pp348-352, 1986

[7] ANSI N14.5-1997 “American National Standard for Radioactive Materials - Leakage Tests on Packages for Shipment”, pp9-10

[8] “Water Immersion Tests of UF6 Cylinders with Simulated Damage”, K-D-1987, A. J. Mallet, November 7, 1967