Demonstration of a Solution Film Leak Test Technique and Equipment for the S00645 Canister Closure

by

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**DEMONSTRATION OF A SOLUTION FILM LEAK
TEST TECHNIQUE AND EQUIPMENT FOR THE S00645
CANISTER CLOSURE (U)**

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TITLE: DEMONSTRATION OF A SOLUTION FILM LEAK TEST
TECHNIQUE AND EQUIPMENT FOR THE S00645
CANISTER CLOSURE (U)

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DEMONSTRATION OF A SOLUTION FILM LEAK TEST TECHNIQUE AND EQUIPMENT FOR THE S00645 CANISTER CLOSURE (U)

INDTRODUCTION

Canister S00645 nozzle was damaged during processing preparatory to closure welding such that routine closure, using the DWPF Welder (resistance upset welding process), is not feasible¹. A repair plan has been devised to accomplish the final closure using an alternate welding process². A key step in this plan is leak testing of the final closure - the weld must be verified to meet the Waste Acceptance Product Specifications (WAPS) leaktight criterion of 1×10^{-4} atm-cc/sec helium³.

Solution film testing (SFT) has been selected for verifying leaktight integrity of the Canister S00645 final closure. This paper reports the demonstration, as requested by TTR #HLW/ DWPF/TTR-99030⁴, of a solution film test technique capable of detecting the WAPS leaktight acceptance criterion when applied to a DWPF canister nozzle. This work meets requirements of Task Technical and Quality Assurance Plan Number SRT-MTS-99-5004⁵, written in accordance with DOE/RW-0333P criteria.

TECHNIQUE DESIGN AND TEST EQUIPMENT

The solution film test technique (thin-film liquid application) follows the test method specified in ASTM E 515-95, "Standard Test Method for Leaks Using Bubble Emission Techniques"⁶. E 515 identifies this method to be sensitive to the limit of 1×10^{-5} atm-cm³/s. This technique also meets requirements specified in ASME Section V, Article 10, Appendix II, "Bubble Test, Vacuum Box Technique"⁷.

In addition to the general requirements of E 515, several critical parameter values were identified to maximize sensitivity of the technique while retaining ability to visually detect leakage. These parameters include detection fluid (solution), pressure differential (partial vacuum below atmospheric pressure) and test area surface cleanliness. Site Procedure LT-VB⁸ (Bubble test, vacuum box examination), as amended by the specific parameter stipulations identified in Attachment 1, was used for the demonstration testing.

Procedures and personnel utilized in this demonstration were qualified/certified in accordance with standard site quality requirements. Test equipment, gauges, etc., conformed to site calibration (M&TE) standards.

Solution film test equipment consists of a vacuum box, designed specifically to fit a DWPF canister nozzle, vacuum pump, hose and gauge (Figure 1).

DEMONSTRATION

Preparatory to testing, a welded DWPF test nozzle was modified by capping the neck (to facilitate testing) and installing an Accu-Flow⁹ Calibrated leak into the weld plug (Figure 2). The capillary leak, as procured, was set to leak at a rate of 7.5×10^{-5} atm-cc/sec helium. After installation, the test nozzle (capillary leak) was checked using the High-Pressure Lab helium mass spectrometer leak detector to confirm the leak rate. The rate of the installed leak was 4.7×10^{-5} atm-cc/sec helium - within the manufacturers stated tolerance. Figure 3 shows the test equipment and setup used for the demonstration.

Having established a leak rate less than the WAPS acceptance criterion, the test nozzle was fitted with the vacuum box and tested using the SFT method (Figure 4). At a vacuum pressure of 24 inches Hg, a continuous stream of small bubbles was visually observed in the detection fluid, demonstrating the ability to detect leaks smaller than the WAPS criterion. The test nozzle was then re-checked the following day, in the presence of DWPF-Engineering and SRTC QA personnel - results were identical.

Attachment 2 provides a detailed account of the demonstration testing. Attachment 3 is the solution film leak test report (data sheet). Attachment 4 is the helium leak test data sheet certifying the rate of the prepared leak - 4.7×10^{-5} atm-cc/sec helium.

EES Job Folder, number 22727 (located in 730-A, Document Control) contains pertinent information associated with this demonstration, including equipment calibration documents, test procedures, etc.

DISCUSSION

The purpose of this effort was to demonstrate that the SFT technique, when adapted to a DWPF canister nozzle, is capable of detecting leaks not meeting the WAPS acceptance criterion.

The ASTM E 515 standard indicates that solution film testing does not provide a quantitative measure of leak rate, but can identify or disclose leaks. In other words, it performs a "pass/fail" function. Sensitivity of SFT testing depends on several factors, but primarily is influenced by detection fluid type, pressure differential across the work piece and test area cleanliness. By following specified procedural requirements, e.g., use of certified materials, certified personnel, maintaining temperature limits, and use of an appropriate detection fluid and test pressure differential, SFT is capable of detecting leaks in the 10^{-5} atm-cc/sec helium range.

CONCLUSIONS

- o The SFT technique and equipment described herein, successfully detected a confirmed leak of 4.7×10^{-5} atm-cc/sec helium. This demonstrates the ability to disclose

a leak in DWPF canister closure welds not meeting the WAPS leak tight acceptance criterion of 1×10^{-4} atm-cc/sec helium.

- o Conditions and environment expected (test piece cleanliness, visual access to test area, lighting, etc.) for testing of the S00645 closure weld, should not be significantly different from that encountered in this demonstration effort.
- o This SFT technique and equipment can be applied to other DWPF canister closure welds requiring leak testing to satisfy WAPS acceptance criteria.

REFERENCES

1. NCR No. 97-NCR-05-0122, *Canister Waste Form #S00645 (U)*, 10/1/1997
2. Kerley, W.D. and Heise, R.B., *Implementation Plan for Canister S00645 (U)*, HLW-DEN-98-0374, 1/13/1999
3. U.S. Department of Energy Office of Environmental Management, *Waste Acceptance Product Specifications for Vitrified High-Level Waste Forms*, Revision 2, December 1996
4. Technical Task Request, Number HLW/DWPF/TTR-99030, Revision 1, *Provide Sensitivity Findings for Vacuum Box to be Used on Canister S00645 Weld*, May 24, 1999
5. Task Technical and Quality Assurance Plan, Number SRT-MTS-99-5004, Revision 0, *Demonstration of a Solution Film Leak Test Technique and Equipment for DWPF Canister Closure Welds*, July 1999
6. American Society for Testing and Materials, Number E 515-94, *Standard Test Method for Leaks Using Bubble Emission Techniques*
7. ASME Boiler and Pressure Vessel Code, Section V, *Nondestructive Examination*, 1998 Edition
8. Bechtel Savannah River, Inc. Nondestructive Examination Standard, Number LT-VB, Revision 4, May 25, 1994
9. "Accu-Flow Calibrated Leak", Manufactured by Vacuum Technology Inc., Oak Ridge, TN

ATTACHMENTS

1. Stipulations for Using Procedure LT-VB on DWPF Canister and Test Nozzle Closure Welds
2. Detailed Account of Demonstration Testing
3. IES Solution Film Leak Test Report
4. IES Proof/Leak Test Data Sheet

Figure 1. Vacuum Box for DWPF Nozzles

Figure 2. Specially Prepared DWPF Test Nozzle

a) Test nozzle connected to helium leak detection unit

b) Bell jar fitted over test nozzle ready for pump down of bell-jar

c) Helium leak detection unit

d) Vacuum box designed to solution film test DWPF nozzles

Figure 3. Test Equipment, Setup and Vacuum Box

Figure 4. Vacuum Box Fitted to Test Nozzle

ATTACHMENT 1
Stipulations for Using Procedure LT-VB
on DWPF Canister and Test Nozzle Closure Welds

Scope: The following stipulations shall be observed when solution-film testing DWPF canister and test nozzle closure welds using procedure "BSRI Nondestructive Examination Standard, Vacuum Box, LT-VB", Revision 4, Dated 5/25/94.

General:

1. The term BSRI NDE personnel shall mean also WSRC NDE personnel.
2. The term BSRI NDE procedures and documents shall include also equivalent WSRC procedures and documents.
3. Use attached "IES Solution Film Leak Test Report" to record pertinent test information.
4. Visual observation for bubbles (detection fluid under test) shall be aided by the use of a flashlight and 5X magnification lens.

Specific:

1. Refer to paragraph 4.1.3: Partial vacuum pressure shall be at least 24 inches Hg minimum below atmospheric pressure.
2. Refer to paragraph 4.2.1: Bubble solution shall be Leaktec (American Gas and Chemicals, Inc.), Type OX-315 I.
3. Refer to paragraph 5.4: Immediately prior to application of the bubble solution, the test area shall be rinsed with alcohol and wiped dry with a clean, dry cloth.
4. Refer to paragraph 6.4: With regard to lighting, the following applies: Ambient or artificial illumination shall be considered sufficient when the 1/32 inch graduations on a machinist scale can be resolved at a distance of 24 inches.
5. Refer to paragraph 6.6: The formation of continuous, single small bubbles shall be considered relevant.

ATTACHMENT 2
Detailed Account of Demonstration Testing

Date: August 4, 1999

Place: High Pressure Lab, 723-A

Equipment:

Helium Mass Spectrometer Leak Detection Unit, Make: Varian, Model: 948

Vacuum box designed specifically to test DWPF nozzles

Examiner Certification:

ASNT SNT-TC-1A

Wayne Thompson, Level II (Mass Spectrometer and Bubble Leak Testing)

Procedures:

Mass Spectrometer Testing: L94-8303, Revision 6 "Leak Testing"

Solution Film Testing: LT-VB, Revision 4 "Nondestructive Examination
Standard, Vacuum Box"

1. Prior to this demonstration, a DWPF test nozzle was modified by capping the neck (to facilitate testing) and installing, by welding, an Accu-Flow Open Style Calibrated leak (crimped capillary tube) through the center of the weld plug. The Vendor specified target leak rate of the capillary leak was 7.5×10^{-5} atm-cc/sec helium.
2. The prepared test nozzle was connected to the Mass Spectrometer unit (helium leak tester) and a leak rate of 4.7×10^{-5} atm-cc/sec helium was recorded.

Note: Immediately prior to and after helium leak testing the nozzle, calibration of the helium leak tester was verified by checking against a known standard.
3. The nozzle (immediate area of the installed leak) was wiped down using a clean cloth and alcohol. Detection fluid was applied to the test area and the vacuum box placed on the nozzle and checked for leaks.
4. Within a few seconds, at a vacuum pressure of 24 inches Hg, a continuous stream of tiny bubbles was observed at the leak site. Bubbles were visible with the unaided eye; however, use of a flashlight and magnification lens (5X) significantly improved ability to see the bubbles.
5. The vacuum box was then removed and the test area of the nozzle wiped clean with alcohol. The nozzle was placed under a vacuum to avoid exposure to moisture and other condensables so as to preclude plugging of the leak.
6. The next day, August 5, 1999, DWPF-Engineering personnel were invited to the High-Pressure Lab to witness a repeat of the test. Testing was conducted as described above - results were identical to that of the previous day.

ATTACHMENT 3
IES Solution Film Leak Test Report

Test Date: 8/4/99

WSRC Savannah River Site Operations		IES Solution Film Leak Test Report		Report #: SRT-MTS-99-5004 Report No. 1
Leak Test Identification:				
System/Area: High Pressure Lab		Procedure #: LT-VB^{1/}		Revision: 4
Weld/Test Item: DWPF Test Nozzle N-1		Repair: N/A	Inspection Plan #: SRT-MTS-99-5004	Acceptance Criteria: N/A
Material Type: 304L S/S	Product Form: Test Nozzle	Technique: ☐ Film ☐ Imer ☒ Vbox ☐ Other		
Size/OD/Thickness: N/A	Surface Finish: As-Welded	Preclean: Alcohol		Postclean: Alcohol
Surface Temperature: Before: 26.5 C After: 26.5 C		Temperature Gauge #: M&TE #: EA-1047 Due Date: 4/6/00		
Test Pressure: 24 Inches Hg		Pressure Gauge #: CDS 4791 M&TE #: CDS 4791 Due Date: 12/23/99		
Hold Time: 20 Seconds		Bubble Solution: Brand: Leak-Tec Type: OX-315 Batch #: 980714		
Test Equipment ID (If Used): Special Vacuum Box designed to fit a DWPF Canister or Test Nozzle				
Examination Results				
Item/Weld #:	Acc:	Rej:	Comments:	
Test Nozzle N-1	N/A	N/A	Observed continuous formation of small bubbles at prepared leak area	
Remarks:				
Test Nozzle N-1 was fitted with a "pre-fabricated" leak. The leak rate was identified by helium leak test to be 4.7×10^{-5} atm-cc/sec helium.				
Examiner: <u>Waepe Thompson</u>		Reviewer/Authenticated: <u>[Signature]</u>		Page: <u>1</u> of <u>1</u>
Level: <u>II</u> Date: <u>8-31-99</u> Level: _____ Date: _____		Level: <u>III</u> Date: <u>8/31/99</u>		

1/ Stipulations in Attachment 6.2, SRT-MTS-99-5004 were applied

General:

- o Lighting in demonstration area sufficient to resolve 1/32 inch graduations on a machinist scale at a distance of 24 inches
- o Visual examination was aided by use of a flashlight and 5X magnification lens

ATTACHMENT 4
IES Proof/Leak Test Data Sheet

Attached is the High-Pressure Lab (IES) mass spectrometer helium leak test report certifying the leak rate of the installed leak device in the DWPF test nozzle, N-1. In addition, calculation M-CLC-A-00150, "Verification of IES Proof/Leak Test Software, Version 1.9" is attached.

** ACCEPTABLE **

08/04/99
PAGE 1 OF 2
Record : 1374

Basis No.: SRT-IES-99-204
RETENTION: LIFE

CC: GARY CANNELL
Laboratory Copy
File Room, 730-A
SRL Records, 773-A

730-A

IES PROOF/LEAK TEST DATA SHEET (U)

Test Item: VTI CALIBRATION LEAK
Test Item Serial Number: 06564
Test Date: 08/04/99
Body: Leakage Rate, std cc He/sec: 4.7×10^{-5}

Proof Pressure (psig): NA
Leak Test Pressure (psig): 1 ATMOS

Acceptable Maximum Leak Rates, std cc He/sec:
Body: 9.0×10^{-5}

IES Test Engineer: D. J. TRAPP Phone: 725-8307

Reviewed by: *C. J. Allen II* 8/10/99

Certified L.T. *Wayne Thompson II* Date: 8-4-99
Signature/Level

COMMENTS

ACTUAL PROOF TEST PRESSURE NA
ACTUAL LEAK TEST PRESSURE 1 ATMOS.
ACTUAL BURST TEST PRESSURE NA

PAGE 2 OF 2
Record : 1374

TEST DESCRIPTION

Technician: WAYNE THOMPSON
Procedure No.: OP:L9.4-8303
Revision No.: 7
Proof Test Time (min):
Proof Test Fluid:
Leak Test Method: BELL JAR
Leak Test Time (min): 3
Leak Test Fluid: HE
% Concentration: 100

TEST EQUIPMENT

M&TE Description	No.	Cal?	Expires
Leak Detector	8-1135	Y	B/A
Strip Chart Recorder	12853	N	NA
Pressure Gauge/Transducer	3-1068	Y	01-05-00
Thermometer	EA-1047	Y	04-06-00

PRELIMINARY CALIBRATION

Preliminary Standard Leak Rate, std cc He/sec: 9.61×10^{-5}
Standard Leak M&TE #: 3-1452 Expires: 01/22/00
Preliminary Standard Leak Cal. Temp, °C: 26.6
Room Temperature, °C: 23.8
Preliminary Temp Corrected Standard Leak Rate, std cc He/sec:
 8.8×10^{-5}
Preliminary Cal Background, Divisions: 6.5
Preliminary Cal Meter Reading, Divisions: 85000.0
Preliminary Sensitivity (std cc He/sec/Division): 1.0×10^{-9}

FINAL CALIBRATION

Final Standard Leak Rate, std cc He/sec: 9.61×10^{-5}
Standard Leak M&TE #: 3-1452 Expires: 01/22/00
Final Standard Leak Cal Temp, °C: 26.6
Final Temp Corrected Standard Leak Rate, std cc He/sec: 8.8×10^{-5}
Final Cal Background, Divisions: 35.0
Final Cal Meter Reading, Divisions: 80000.0
Final Sensitivity (std cc He/sec/Division): 1.1×10^{-9}

LEAK MEASUREMENTS

Body Sample Background, Divisions: 6.5
Sample Meter Reading, Divisions: 43000.0

INFORMATION ONLY

Calculation Cover Sheet

Project N/A		Calculation Number M-CLC-A-00150		Project Number N/A	
Title Verification of IES Proof/Leak Test Software, Version 1.9		Functional Classification GS		Sheet 1 of <u>5</u>	
		Discipline Mechanical Engineering			
☐ Preliminary ☒ Confirmed					
Computer Program No. ☒ N/A		Version/Release No.			
Purpose and Objective This calculation compares IES Proof/Leak Test Software, Version 1.9 leak test results with manual calculations per IES Procedure L9.4-8303 Rev. 7, Section 5.5.1.					
Summary of Conclusion The IES Proof/Leak Test Software, Version 1.9 calculates leak test results correctly as described in IES Procedure L9.4-8303 Rev. 7, Section 5.5.1.					
Revisions					
Rev No.	Revision Description				
1	Initial issue				
Sign Off					
Rev No.	Originator (Print) Sign/Date	Verification/ Checking Method	Verifier/Checker (Print) Sign/Date	Manager (Print) Sign//Date	
1	Donald J. Trapp 8/10/99 <i>Donald J. Trapp</i>	DOCUMENT REVIEW	Michael L. Restivo <i>Michael L. Restivo</i>	John C. Whitehead 8/20/99 <i>John C. Whitehead</i>	
Classification Unclassified					

Calculation Continuation Sheet

Calculation No. M-CLC-A-00150	Sheet No. 3/2 2/7 8/10/99	Rev. 0
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References: IES Manual L9.4-8303, Rev. 7, "Proof/Leak Test Procedure", Section 5.5, Calculations for Helium Leak Tests
 IES Proof/Leak Test Software, Version 1.9
 IES Proof/Leak Test Data Sheet for "VTI Calibration Leak", Serial No. 06564, BASIS No. SRT-IES-99-204,
 8/4/99

The step numbers for the Calculations below correspond to the step numbers in L9.4-8303, Rev. 7, Section 5.5.1

Verification of IES Proof/Leak Test Software, Version 1.9

Test Parameters for "VTI Calibration Leak", Serial No. 06554, tested on 8/4/99:

Helium Belljar Test with helium gas at atmospheric pressure inside the test item

Room Temperature = 23.8°C

% Helium = 100%

Preliminary Calibration:

Standard Leak M&TE Number: 3-1452, Expires 1/22/00

Standard Leak Rate = 9.61×10^{-5} std cc He/sec

Calibration Temperature = 26.6°C

Calibration Background = 6.5 Divisions

Calibration Meter Reading = 85,000 Divisions

Final Calibration:

Standard Leak M&TE Number: 3-1452, Expires 1/22/00

Standard Leak Rate = 9.61×10^{-5} std cc He/sec

Calibration Temperature = 26.6°C

Calibration Background = 35 Divisions

Calibration Meter Reading = 80,000 Divisions

Leak Measurements

Sample Background = 6.5 Divisions

Sample Meter Reading = 43,000 Divisions

Computer Generated Results:

Preliminary Temperature Corrected Standard Leak Rate = 8.8×10^{-5} std cc He/sec

Preliminary Sensitivity = 1.0×10^{-9} std cc He/sec/Division

Final Temperature Corrected Standard Leak Rate = 8.8×10^{-5} std cc He/sec

Final Sensitivity = 1.1×10^{-9} std cc He/sec/Division

Leakage Rate = 4.7×10^{-5} std cc He/sec/Division

Calculation Continuation Sheet

Calculation No. <i>M-CLC-A-00150</i>	Sheet No. <i>23</i> <i>8/14/99</i>	Rev. <i>Φ</i>
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Manual Calculations

- A. Temperature Correction Factor (For Both Preliminary and Final Calibrations)

$$CL = C - [(T_c - T_r) * .03 * C]$$

CL = Actual Calibration Leakage Rate

C = Stated Leakage rate of Calibrated Leak = 9.61×10^{-5} std cc He/sec

T_c = Temperature at which calibrated leak was calibrated = 26.6°C

T_r = Room Temperature = 23.8°C

$$CL = 9.61e^{-5} - [(26.6 - 23.8) * .03 * 9.61e^{-5}] = 8.80e^{-5} \text{ std cc He/sec}$$

This matches the computer results.

- B. Preliminary Calibration

$$PCLR = \frac{CL}{(MR - BG)}$$

PCLR = Preliminary Test Sensitivity

CL = Actual Calibration Leakage Rate = 8.8×10^{-5} std cc He/sec

MR = Meter Reading with calibrated leak opened to the leak detector =
85,000 Divisions

BG = Meter Reading after calibrated leak has been closed = 6.5 Divisions

$$PCLR = \frac{8.8e^{-5}}{(85000 - 6.5)} = 1.03e^{-9} \text{ std cc He/sec/Division}$$

This matches the computer results of 1.0×10^{-9} std cc He/sec/Division for Preliminary Sensitivity.

Calculation Continuation Sheet

Calculation No. <i>M-CLC-A-00150</i>	Sheet No. <i>4</i>	Rev. <i>φ</i>
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C. Final Calibration

$$ACLR = \frac{CL}{(MR - BG)}$$

ACLR = Preliminary Test Sensitivity

CL = Actual Calibration Leakage Rate = 8.8×10^{-5} std cc He/sec

MR = Meter Reading with calibrated leak opened to the leak detector =
80,000 Divisions

BG = Meter Reading after calibrated leak has been closed = 35 Divisions

$$ACLR = \frac{8.8e^{-5}}{(80000 - 35)} = 1.10e^{-9} \text{ std cc He/sec/Division}$$

This matches the computer results of 1.1×10^{-9} std cc He/sec/Division for Final Sensitivity.

D. Measured Leak Rate:

$$MLR = ACLR * (MR - BG) * (\%He/100)$$

MLR = Measured Leak Rate of the test item

ACLR = Final test sensitivity = $1.10e^{-9}$ std cc He/sec/Division

MR = Meter Reading with the test item filled with helium = 43,000
Divisions

BG = Meter Reading after helium has been vented = 6.5 Divisions

%He = the percent concentration of helium in the tracer gas = 100%

$$MLR = 1.1e^{-9} * (43000 - 6.5) * (100/100) = 4.73e^{-5} \text{ std cc He/sec}$$

This matches the computer results of 4.7×10^{-5} std cc He/sec for Leakage Rate.

Calculation Continuation Sheet

Calculation No. <i>M-CLC-A-00150</i>	Sheet No. <i>5</i>	Rev. <i>φ</i>
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E. Minimum Detectable Leak

$$MDL = ACLR * .02 * \text{Full_Scale_Reading_of_Background_Scale}$$

MDL = Minimum Detectable Leakage Rate

ACLR = Final Test Sensitivity = $1.10e^{-9}$ std cc He/sec/Division

Full Scale Reading of the Background Scale: The background for the test was 6.5 divisions. The full scale reading for the leak detector is 10 Divisions.

$$MDL = 1.1e^{-9} * .02 * 10 = 2.2e^{-10} \text{ std cc He/sec}$$

This matches the computer results of 2.2×10^{-10} std cc He/sec for Minimum Detectable Leak Rate. This number only appears on the computer screen and is not printed out on the datasheet.

F. Reported Leak Rate

Measured Leak Rate = 4.73×10^{-5} std cc He/sec

Minimal Detectable Leak Rate = 2.2×10^{-10} std cc He/sec

Final Sensitivity = 1.10×10^{-9} std cc He/sec/Division

The Reported Leak Rate is the largest of the three numbers listed above, which would be the Measure Leak Rate = 4.73×10^{-5} std cc He/sec.

This matches the computer results, which reports a leak rate of 4.7×10^{-5} std cc He/sec.

Conclusion:

The IES Proof/Leak Test Software calculated the leak test results exactly as specified in L9.3-8303, 5.1 *5.5.1 01/18/09*