

ENGINEERING PRACTICE STUDY

PROJECT NUMBER: 5961–2016–072

TITLE:

MIL–STD–750–1, TEST METHOD 1071, TEST CONDITION B:

PROPOSED PROTOCOL FOR EXTENDING THE DWELL TIME
OF THE DRY RADIOACTIVE TRACER GAS GROSS LEAK TEST

20 OCTOBER 2016

FINAL REPORT

STUDY CONDUCTED BY

DLA LAND AND MARITIME
DOCUMENT STANDARDIZATION DIVISION (VA)
ACTIVE DEVICE BRANCH (VAC)

Prepared by:
David Corbett
Electronics Engineer
Active Devices Branch

Approved by:
Thomas Hess
Chief
Active Devices Branch

I. **OBJECTIVES:** The objective of this Engineering Practice (EP) study was to obtain comments or concurrence from the military services, device manufacturers, laboratories, OEMs, and the user community concerning a proposed post pressurization radioactive tracer gas (⁸⁵Kr) egress retardant protocol (hereafter referred to as "the protocol") intended to be included in a future revision to test condition B of test method 1071 of MIL-STD-750-1. The protocol and a proposed revision to the test condition with the protocol incorporated is intended to extend the dwell time of devices after being exposed to the ⁸⁵Kr. Attachment 1 is the protocol and attachment 2 is the proposed test condition that was included as attachments 1 and 2 of the initial draft.

II. **BACKGROUND:** At the 2016 Jacksonville JEDEC committee meetings, a request was made to follow-up on an EP study covering the same subject as this EP study. The previous EP study was generated as a result of a presentation given at the January 2015 JEDEC Committee meetings. That presentation (see attachment 3) covers the background and principal of the protocol and included results for some packages that were tested. That EP study was coordinated in June of 2015 as project [5961-2015-070](#). The final report for project [5961-2015-070](#) was issued as inconclusive because there was no record of any concurrence or comments either in favor or against the protocol. The coordination for this EP study was different than the June 2015 effort since it included a proposed test condition B revision that incorporated the protocol changes into test condition B for the reviewer's evaluation (attachment 2). After performing a review of the content of the current wording of test condition B of method 1071, additional consistency, editorial, and organization issues were noted and were included in the proposed test condition revision.

III. **RESULTS:** Essential comments were received regarding the proposed changes to test condition B. The two essential comments and one technical comment were received by DLA Land and Maritime are summarized below:

Two commenters (one essential and one technical) accepted the protocol and were in favor of portions of the proposed test condition. These comments, one from military reviewer and another from a test equipment manufacturer, stated that test condition B should not be altered. However, they believe that a new test condition to test method 1071 that incorporates the protocol should be investigated. This new test condition should use most of the content from the proposal in the initial draft (attachment 2) plus new requirements supplied with their comments.

One commenter submitted an essential comment against the protocol and proposed test condition. This military reviewer stated that due to the lack of a technical justification and significant amount of test data being included in the initial draft of the EP study, they are not in favor of adding the protocol into test method 1071.

IV. **CONCLUSIONS:** Based on the essential comments received, no changes will be made to test condition B of test method 1071 of MIL-STD-750-1.

V. **RECOMMENDATIONS:** Investigate if a future EPS to coordinate a proposed new test condition can be undertaken if the dissenting commenter can be convinced that a new test condition allowing DI water as a Kr85 egress retardant has merit.

The point of contact for this final report is Mr. David Corbett, DLA Land and Maritime – VAC, P. O. Box 3990, Columbus, OH 43218-3990. Mr. Corbett can also be reached at 614-692-0526 / DSN 850-0526, or by facsimile at 614-693-642 / DSN 850-1642, or by e-mail to: 750.TestMethods@dla.mil.

ATTACHMENT 1 to Project 5961–2016–072

DWELL TIME EXTENSION PROCEDURE.

1. Device(s) shall be subjected to the pressurization requirements of Test Method 1071, Condition B.
2. Device(s) shall be immersed in deionized (DI) water within one minute upon removal from the pressurization chamber.
3. Device(s) shall remain submerged in the DI water until time for measurement.
4. Once removed from DI immersion, the device(s) shall be tested for leaks within 10 minutes.
5. Device(s) shall not be dried until after leak testing has been completed. Drying the device(s) will negate the effect of the enhancement procedure.

This draft, dated 27 May 2016, prepared by DLA-CC, has not been approved and is subject to modification.

KEY to the changes are as follows: DLA-VAC: Text highlighted in yellow has been added.

5. Test condition B – radioisotope dry gross leak. This test condition covers a procedure for detecting gross leaks in sealed devices by the use of a radioactive tracer gas. This condition is identified as "dry" because the device package is not subjected to any solutions before the radioactive tracer gas exposure.

5.1 Test condition applicability. This test condition can be used to test devices that internally contain some ⁸⁵Kr absorbing or adsorbing medium, such as electrical insulation, organic, or molecular sieve, or approved gettering material. If the device does not contain any absorbing or adsorbing medium, then this test condition can only be used on devices with an internal free volume of 0.02 cc or larger, or after being subjected to the requirements of a and b, that it can comply with the requirements of c:

- a. A .005 inch (0.13 mm) diameter hole shall be made in a representative unit of the devices to be tested.
- b. The device shall be subjected to 5.3.1 of this test condition and removed from the test chamber of the radioactive tracer gas pressurization console immediately after the test chamber is vented to atmosphere, and measured in the counting station.
- c. A net reading indicating 500 cpm or greater is considered a reject. The device shall remain a reject for a minimum of 10 minutes after removal from the test chamber of the radioactive tracer gas pressurization console. If the device does not fail, then this test condition shall not be used.

5.2 Apparatus. The apparatus required for test condition B shall be as follows:

- a. Radioactive tracer gas pressurization console containing ⁸⁵Kr/air mixture.
- b. Counting station. The equipment of the counting station shall consist of a scintillation crystal, photomultiplier tube, preamplifier, ratemeter, and ⁸⁵Kr reference standards. The counting station shall be of sufficient sensitivity to determine through the device package wall the radiation level of the ⁸⁵Kr tracer gas present within the device.
 - (1) A "well type" counting station with a minimum sensitivity of 10,000, cpm per μ Ci of ⁸⁵Kr tracer gas and a minimum detectable count rate of 500 cpm above background level.
 - (2) A "flat top" counting station with a minimum sensitivity of 4,500, cpm per μ Ci of ⁸⁵Kr tracer gas and a minimum detectable count rate of 500 cpm above background level.
 - (3) A "tunnel" counting station with a minimum sensitivity of 4,500, cpm per μ Ci of ⁸⁵Kr tracer gas and a minimum detectable count rate of 500 cpm above background level.
- c. A tracer gas consisting of a mixture of ⁸⁵Kr and air. The concentration of ⁸⁵Kr in air shall be no less than 100 μ Ci per atm-cc. This value shall be determined at least once each 30 days, following manufacturer's procedure, and recorded in accordance with the calibration requirements of the general test method standard MIL-STD-750. The specific activity may be measured automatically by the equipment during cycling of the equipment. However, it is recommended that an analytical sample of the gas be measured annually for low use systems and semiannually for high use production systems.
- d. ESD protective tubes (plastic or aluminum) shall be utilized to ensure the system is ESD safe when using the "well type" counting station.
- e. All calibration records (e.g. daily, monthly, voltage crystal plateau graphs, and Certificates of Conformance for ⁸⁵Kr reference standards, specific activity, etc.) shall be maintained and made available to the qualifying activity.
- f. The crystal voltage plateau graph shall be performed and documented semiannually. Care should be taken when performing crystal voltage plateau measurements. Potential exposure to high voltage and potentiometer disturbance must be taken into consideration. Disturbance of the potentiometers may require the ratemeter to be repaired.
- g. Deionized (DI) water.

This draft, dated 27 May 2016, prepared by DLA-CC, has not been approved and is subject to modification.
KEY to the changes are as follows: DLA-VAC: Text highlighted in yellow has been added.

5.3 Procedure.

5.3.1 Evacuation and pressurization. The devices shall be placed into the test chamber of the radioactive tracer gas pressurization console with aluminum filler blocks placed in the test chamber to displace all unneeded free volume. The test chamber shall be evacuated to a pressure not to exceed 66.7 Pa (0.5 torr). The devices shall then be subjected to a minimum of 30 psia (207 kPa) of ⁸⁵Kr/air gas mixture for the time specified in 5.3.1.1 or 5.3.1.2, as applicable. For devices that have pressure limits less than 30 psia (207 kPa), the pressure limit shall be specified and not exceeded. After the bombing time has been reached, the gas mixture shall then be transferred to the storage tank until a pressure of 266.7 Pa (2.0 torr) maximum exists in the test chamber. This gas transfer shall be complete in 3 minutes maximum. The test chamber shall then be backfilled with air and the devices shall then be removed from the test chamber.

5.3.1.1 Pressurization time for optional pre-acceptance testing. An optional pre-acceptance pressurization test protocol intended to conserve the ⁸⁵Kr/air gas mixture may be performed prior to the gross leak test of 5.3.1.2 to remove devices exhibiting significant leakage. The pre-gross leak test shall implement all required bombing conditions as stated in 5.3.1, except that the soak time may be reduced to 36 seconds (.01 hours) minimum.

5.3.1.2 Pressurization time for product acceptance. All devices to be evaluated for compliance to this test condition must be subjected to a minimum pressurization time of 2 minutes (.04 hours).

5.3.2 Evaluation. There are 2 protocols for evaluating devices once they have been removed from the test chamber of the radioactive tracer gas pressurization console. They are as follows:

- (1) The dry evaluation protocol (see 5.3.2.1).
- (2) An optional semi-dry evaluation after wet storage protocol (see 5.3.2.2). The intent of the semi-dry evaluation protocol is to extend the post pressurization dwell time beyond the 10 minute limit of the dry evaluation protocol.

5.3.2.1 Dry evaluation.

5.3.2.1.1 Measurement. The devices shall be measured in the counting station within 10 minutes after completion of the test chamber air backfill cycle of 5.3.1.

5.3.2.1.2 Re-pressurization. If the devices are not all measured before the end of 10 minutes from removal from the test chamber of the radioactive tracer gas pressurization console, the unevaluated devices shall be returned to the test chamber and re-pressurized in accordance with 5.3.1, and then measured at the counting station within 10 minutes.

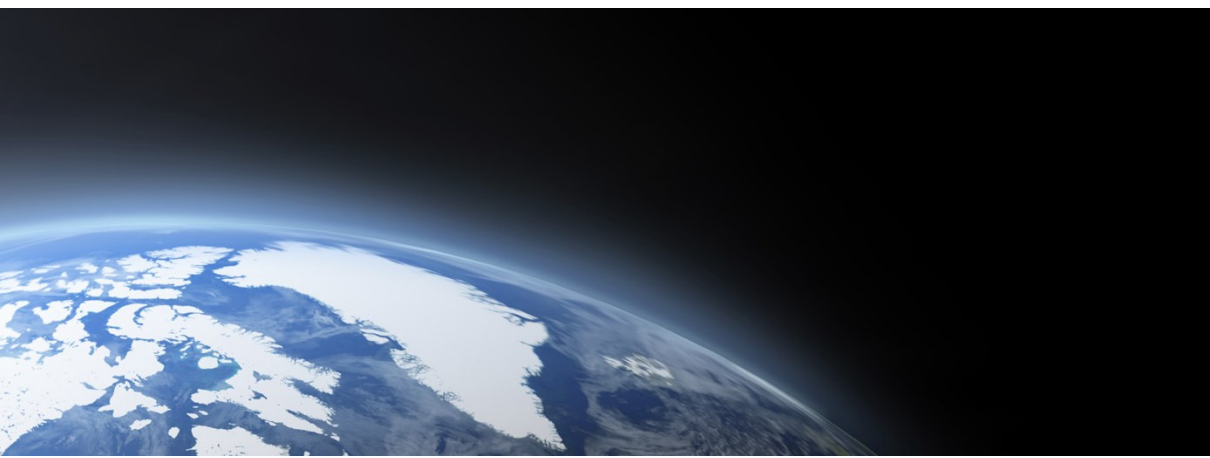
5.3.2.2 Semi-dry evaluation after wet storage (optional).

5.3.2.2.1 Dwell time extension. If the optional semi-dry evaluation protocol is to be used, then the devices shall be immersed in deionized (DI) water within one minute upon removal from the test chamber of the radioactive tracer gas pressurization console. The devices shall remain submerged in the DI water until time for measurement at the counting station.

5.3.2.2.2 Measurement. Once removed from the DI immersion, the devices shall be measured in the counting station within 10 minutes. Device(s) shall not be dried until after measuring has been completed. Drying the device(s) will negate the effect of the dwell time extension protocol.

5.3.2.2.3 Re-pressurization. Re-pressurization is not applicable for the semi-dry evaluation protocol.

5.4 Failure criteria. Any device indicating 500 cpm, or greater, above the ambient background of the counting station shall be rejected as a gross leak failure.



Mil-Std-750-TM1071.9 Condition B Radioisotope Dry Gross Leak Dwell Enhancement Procedure

Bill Pelletier
Process Engineer
RSO
1/07/2015

Radioisotope Dry Gross Leak has Issues with Escapes from Large leaks.

- Changes to the MIL-STD-750F with test method 1071.9, Condition A volume from <0.02 to < 0.05 has left the industry with a gross leak testing procedure that has potential weaknesses.
- The current expectation with these changes is that the Radioisotope tests will cover the entire range of leaks and cavity sizes.
- Condition B has been changed to exclude cavity sizes $<0.1\text{cc}$ unless they pass 10 min dwell with 5 mil hole.
- A dwell enhancement procedure was needed for condition B to ensure the gas will not escape from large leaks during regular testing intervals.
- This dwell enhancement procedure should be utilized to cover the cavity sizes not meeting the requirements of Paragraph 5,a,b.
- This dwell enhancement procedure is not intended to replace condition A at this time. Condition A relies on gettering Krypton internally and is needed for devices $<0.02\text{cc}$.
- This procedure was developed to cover the ranges $>0.02\text{cc}$ thru 0.2cc or larger.

Condition B: Radioisotope Dry Gross Leak Criteria for Acceptance.

■ **From Condition B, 1071.9:**

- 5. This test shall be used to test devices that internally contain some ^{85}Kr absorbing medium. If the device does not contain any absorbing medium this can be used on parts with 0.1cc internal free volume or larger, or that can demonstrate that the following requirements are met.
 - a. A 5 mil diameter hole shall be made in a representative unit of the device to be tested.
 - b. The device shall be subjected to this test condition and removed from the pressurization tank immediately after the tank is vented to atmosphere, and measured in the counting station. A net reading of 500 cpm or greater is considered a reject. The device shall remain a reject for a minimum of 10 minutes after removal from the pressurization tank. If the device does not fail, this test may not be used.

Condition A. is the recommended replacement test.

5 mil hole test results

Device	Cavity	Dwell	Result
TO-18	0.0258cc	2 minutes	Fail
16 DIP	0.074cc	4 minutes	Fail
SMD.5	0.089cc	6 minutes	Fail
DO-4	0.170cc	6 minutes	Fail
TO-39	0.212cc	13 minutes	Pass
TO-254	0.450cc	45 minutes	Pass

- All devices were tested to the current revision of 1071.9
- All the devices tested did not have getters inside
- Specific activity is 300 uCi ⁸⁵Kr/atm-cc
- Bomb pressure was 60 psia
- Bomb time was 2.4 minutes
- Devices must indicate for 10 minutes minimum to pass per 1071.9
- Tests results indicate that devices less than 0.2cc will not pass

Devices that don't meet the criteria for acceptance

Device type	Cavity size
TO-46	0.0099cc
TO-71	0.0194cc
TO-18	0.0258cc
TO-72	0.0265cc
14 and 16 lead flatpacks	@0.032cc
20-LCC	0.041cc
14 and 16 lead dips	@0.075cc
U3 and SMD.5	0.089cc
TO-78	0.150cc
DO-4	0.170cc

Dwell Enhancement Procedure

- Enhancing the dwell time for condition B or G2 is simple.
- Theory is that ^{85}Kr is inert and will not mix with water.
- Therefore any gas in the device will stay in the device.
- Bomb the devices in the usual way for condition B or G2.
- Immerse the devices in DI water within 1 min after removing from the bomb.
- As long as the devices remain immersed in the DI water the Krypton gas has no place to go, and will stay in the device.
- The devices need to stay wet for testing but not necessarily immersed; drying the devices before testing will negate the benefit of the water trapping the gas within the cavity.
- Once the rejects have been culled from the lot the hermetic devices can be dried.

Proposed changes to the standard

- Mil-Std-750 TM1071 Section 5.c (condition B requirements).
- Devices that do not meet the requirements of 5,a,b shall utilize the dwell enhancement procedure herein or perform Condition A.
- Or we can insert a new gross leak condition (TBD) for devices 0.02cc to 0.2cc utilizing the condition B bombing criteria.

5 Mil hole Dry Vs. Enhanced

The OLD Way

The NEW Way

Device	Cavity	Dry Dwell Time	Result	DI Water Dwell time	Result
TO-18	0.0258cc	2 minutes	Fail	Unlimited	Pass
16 DIP	0.074cc	4 minutes	Fail	Unlimited	Pass
SMD.5	0.089cc	6 minutes	Fail	Unlimited	Pass
DO-4	0.170cc	6 minutes	Fail	Unlimited	Pass
TO-39	0.212cc	13 minutes	Pass	Unlimited	Pass
TO-254	0.450cc	45 minutes	Pass	Unlimited	Pass