

MIL-STD-202G

METHOD 112E

SEAL

1. PURPOSE. The purpose of this test method is to determine the effectiveness of the seal of a component part which has an internal cavity which is either evacuated or contains air or gas. A defect in any portion of the surface area of a sealed part can permit entrance of damaging contaminants which will reduce its effective life. This test will detect leaks due to the use of inferior sealing materials, or to the manufacturing processes used to form the seal. The degree of completeness of the seal can be verified by testing in the "as received" condition or after submission to other environmental or physical-characteristics tests, such as thermal shock, physical shock, or vibration. This test method does not include the many existing versions of lesser-sensitivity seal tests now appearing in some specifications, which verify such requirements as "water tightness", "moisture proofness", etc. In those lesser-sensitivity seal tests, parts are submerged to various depth in water, heated water, water or alcohol with a vacuum, water with a wetting agent, etc; all these tests depend on observation of bubbles as the criterion for failure.

1.1 Definitions.

- a. Standard leak rate. Standard leak rate is defined as the quantity of dry air at 25°C in atmospheric cubic centimeters flowing through a leak or multiple leak paths per second when the high-pressure side is at 1 atmosphere (760 mm Hg absolute) and the low-pressure side is at a pressure of not greater than 1 mm Hg absolute. Standard leak rate shall be expressed in units of atmospheric cubic centimeters per second (atm cm<sup>3</sup>/s).
- b. Measured leak rate. Measured leak rate ( $R_1$ ) is defined as the leak rate of a given package as measured under specified conditions and employing a specified test medium. Measured leak rate shall be expressed in units of atmospheric cubic centimeters per second (atm cm<sup>3</sup>/s). For the purpose of comparison with rates determined by other methods of testing, the measured leak rates must be converted to equivalent standard leak rate.
- c. Equivalent standard leak rate. The equivalent standard leak ( $L$ ) rate of a given package, with a measured leak rate ( $R_1$ ), is defined as the leak rate of the same package with the same leak geometry, which would exist under the standard conditions of 1.1a. The formula (does not apply to procedure IIIb) in 5.4.3.2.3 represents the  $L/R_1$  ratio and gives the equivalent standard leak rate ( $L$ ) of the package with a measured leak rate ( $R_1$ ) where the package volume and leak test conditioning parameters influence the measured value of ( $R_1$ ). The equivalent standard leak rate shall be expressed in units of atmospheric cubic centimeters per second (atm cm<sup>3</sup>/s).
- d. Sensitivity. (Minimum detectable leak rate). Sensitivity is the smallest leak rate that an instrument, method, or system is capable of measuring or detecting under specified conditions. To compare sensitivities of two or more different test conditions, the sensitivity should be converted to sensitivity under standard conditions by using the appropriate conversion factors.
- e. Ambient background. For a scintillation-crystal counting station, ambient background is the oscillatory reading obtained on the meter readout due to electrical noise plus the reading in counts per minute due to cosmic radiation that penetrates the lead shielding of the closed crystal system. This value must be determined just before device counting.

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## 2. TEST CONDITIONS.

2.1 Selection. There are six test conditions (A, B, C, D, E, and F) in this method. Test conditions A and B utilize oil as a means to detect gross leaks by the observation of bubbles. Both of these tests will detect leaks of a nominal value of  $10^{-5}$  atm  $\text{cm}^3/\text{s}$ . Test condition B is used to test parts which will not withstand the temperature required in test condition A. Test condition C detects fine leaks by using a tracer gas and apparatus to measure a leakage rate ( $R_1$ ) to a nominal of  $10^{-8}$  atm  $\text{cm}^3/\text{s}$ . The apparatus can be calibrated for any leakage rate within its range. Test condition D utilizes a fluorocarbon liquid at  $125^\circ\text{C} \pm 5^\circ\text{C}$  ( $257^\circ\text{F} \pm 9^\circ\text{F}$ ) at ambient pressure and detects gross leaks by the observation of bubbles. This latter test condition provides an alternate gross leak test to test condition A. Test condition E utilizes two fluorocarbon liquids; one under pressure followed by the other at  $125^\circ\text{C} \pm 5^\circ\text{C}$  ( $257^\circ\text{F} \pm 9^\circ\text{F}$ ) at room ambient pressure and detects gross leaks by the observation of bubbles. This test condition provides a measure of leakage rate of a nominal value of  $10^{-5}$  atm  $\text{cm}^3/\text{s}$ . Test condition F utilizes a fluorocarbon liquid and a fluorocarbon vapor detector to detect gross leaks. Test condition F provides a measure of leakage rate of a nominal value of  $10^{-5}$  atm  $\text{cm}^3/\text{s}$ . Both test conditions C and F require more expensive commercial equipment and trained personnel, but the equipment provides automatic and quantitative readings. The selection of a test condition to perform the seal test depends on the state of the art of component part manufacturing, the leakage rate which can be tolerated for the desired reliability, the frequency of testing, range of test conditions, cavity size, and whether the testing is to be on a 100 percent production or on a periodic basis. The individual specification shall specify the test condition letter required. When test condition C is specified, the procedure number (see 5.4) and the degree of leakage rate sensitivity required shall be included. The following is included as a sensitivity and applicability guide:

Test condition A (bubble test - mineral oil or peanut oil at  $125^\circ\text{C} \pm 3^\circ\text{C}$  ( $257^\circ\text{F} \pm 6^\circ\text{F}$ )). Use when a nominal sensitivity of  $10^{-5}$  atm  $\text{cm}^3/\text{s}$  is sufficient.

Test condition B (bubble test - silicone oil at room ambient temperature  $25^\circ\text{C} \pm 2^\circ\text{C}$  ( $77^\circ\text{F} \pm 3.6^\circ\text{F}$ ), with a vacuum resulting in an absolute pressure of not greater than 1.5 inches of mercury (not greater than 38.1 torr). Use to test parts which cannot withstand the temperature of test condition A, and when a nominal sensitivity of  $10^{-5}$  atm  $\text{cm}^3/\text{s}$  is sufficient.

Test condition C (tracer gas test). Use when a nominal sensitivity of  $10^{-8}$  atm  $\text{cm}^3/\text{s}$  is sufficient.

Procedure I - (For parts having an evacuation tube which is unsealed and is to be connected to the leak detector for test purposes.)

Leak from outside of part to inside. Check the seal of evacuation tube by backfilling with air or gas. Then subject to procedure IIIa, IIIb, IIIc or IV.

Procedure II - (For parts having an evacuation tube which is unsealed to permit pressurizing with a tracer gas.)

Leak from inside of part to outside. Check the seal of evacuation tube by backfilling with air or gas. Then subject to procedure IIIa, IIIb, IIIc, or IV.

Procedure III - (For parts which are to be tested without breaking their seals.)

Fixed method (IIIa) - Part not filled with tracer gas. Force gas into leaks, then detect its escape. Then subject to test condition A, B or D (see 5.4.3.2.1 permitting use of water), as applicable, to check for gross leaks.

IIIb - Part not filled with tracer gas. Force radioactive gas into leaks, then use a radioactivity counter to determine gas that has entered part. Then subject to test condition A, B, or D (see 5.4.3.2.2.4 permitting use of water), as applicable, to check for gross leaks.

Flexible method (IIIc). Part not filled with tracer gas. Force gas into leaks then detect its escape. Then subject to test condition A, B or D (see 5.4.3.2.3 permitting use of water), as applicable, to check for gross leaks.

Procedure IV - (For parts which are to be tested without breaking their seals.) Parts backfilled with tracer gas as normally supplied.

Leak from inside of part to outside, then subject to test condition A, B or D (see 5.4.4 permitting use of water), as applicable, to check for gross leaks.

Test condition D (gross leak bubble test - Fluorocarbon liquid at 125°C ±5°C (257°F ±9°F). Use as an alternate when test condition A is specified as a gross leak test.

Test condition E (bubble test - two fluorocarbon liquids - one at pressure followed by immersion in a second liquid at 125°C ±5°C (257° ±9°F). Use when a nominal sensitivity of 10<sup>-5</sup> atm cm<sup>3</sup>/s is sufficient.

Test condition F (fluorocarbon vapor detection test). Use when a nominal sensitivity of 10<sup>-5</sup> atm cm<sup>3</sup>/s is sufficient.

2.2 Substitution. The individual specification should specify the lowest sensitivity test condition which is practical; a higher sensitivity test which can be calibrated shall be permitted, i.e., if a test is specified such as test condition A requiring a sensitivity of 10<sup>-5</sup> atm cm<sup>3</sup>/s, test condition C may be used since it can be calibrated to read this leakage rate. Substitution of test condition B or C should be made to test condition A when parts are rated at a temperature lower than 130°C (266°F). Test condition D may be substituted for test condition A.

### 3. TEST CONDITION A.

#### 3.1 Materials.

3.1.1 Mineral oil or peanut oil. The oil used for the bath shall be clear mineral oil or peanut oil having a universal Saybolt viscosity of 175 to 190 seconds when tested at 38°C (100°F).

#### 3.2 Apparatus.

3.2.1 Heated oil container. The container for the oil bath shall be of sufficient depth to immerse the uppermost portion of the enclosure or seal to be tested to a depth of 1 inch (25.4 mm) below the surface of the bath. The container shall be capable of maintaining the oil at the required temperature.

3.3 Precautions. This condition shall not be used for parts rated at a temperature lower than 130°C (266°F). It shall not be used for parts which contain seals made of materials which will outgas due to the temperature of the bath.

3.4 Preparation of specimens. Immediately before immersion it shall be determined that the specimen is at room ambient temperature and free of any foreign (including labels) matter. If applicable, the critical side of the specimen shall be determined. The critical side is defined as the side having the greatest number of seals or length of seal.

3.5 Procedure. This test consists of one immersion of the specimen or groups of specimens into a bath of clear mineral oil or peanut oil (see 3.1.1) maintained at a temperature of  $125^{\circ}\text{C} \pm 3^{\circ}\text{C}$  ( $257^{\circ}\text{F} \pm 6^{\circ}\text{F}$ ). The specimen shall be placed in the oil bath with the critical side (or side of special interest) in a horizontal position facing up. If the specimen has no critical side, it shall be placed in the bath with its major axis in a horizontal position. The specimen shall be completely submerged in the bath, with the uppermost portion of the enclosure or seal at a depth of 1 inch (25.4 mm), and left in that position for a minimum duration of 1 minute. The specimen shall be carefully observed during the entire duration of the immersion for indication of a poor seal as evidenced by a continuous stream of bubbles emanating from the specimen. After the test is completed, the specimen shall be cleaned in a suitable degreaser and permitted to dry thoroughly before any additional tests are performed.

#### 4. TEST CONDITION B.

##### 4.1 Materials.

4.1.1 Silicone oil. The oil used for the bath shall be clear silicone oil having a viscosity of 20 centistokes at  $25^{\circ}\text{C}$  ( $77^{\circ}\text{F}$ ).

##### 4.2 Apparatus.

4.2.1 Reduced pressure vessel. The vessel for the oil bath shall be of sufficient depth to immerse the uppermost portion of the enclosure or seal to be tested to a depth of 1 inch (25.4 mm) below the surface of the bath, over which can be drawn a vacuum resulting in an absolute pressure of not greater than 1.5 inches of mercury (not greater than 38.1 torr).

4.2.2 Vacuum pump. The vacuum pump shall be capable of evacuating and holding a vacuum resulting in an absolute pressure of not greater than 1.5 inches of mercury (not greater than 38.1 torr) for a minimum duration of 1 minute in the reduced pressure vessel (see 4.2.1).

4.2.3 Degassing of silicone oil. The silicone oil shall be placed in the test fixture and a pressure of 1 inch (25.4 mm) of mercury or less attained over the fluid for as long as is necessary to degas the fluid. Such degassing is complete when no further bubbling or frothing is present in the fluid. Throughout the test, components shall be lowered gently in the fluid to prevent aeration of the fluid. The fluid shall not be poured from one container to another without first being degassed again before testing.

##### 4.3 Preparation of specimens. As specified in 3.4.

4.4 Procedure. This test consists of one immersion of the specimen or group of specimens into a bath of clear silicone oil (see 4.1.1) maintained at room ambient temperature. The specimen shall be placed in the oil bath with the critical side (or side of special interest) in a horizontal position facing up. If the specimen has no critical side, it shall be placed in the bath with its major axis in a horizontal position. The specimen shall be completely submerged in the bath with the uppermost portion of the enclosure or seal at a depth of 1 inch (25.4 mm). A vacuum resulting in an absolute pressure of not greater than 1.5 inches of mercury (not greater than 38.1 torr) shall be drawn and held over the bath for a minimum duration of 1 minute. The specimen shall be carefully observed during the entire duration of the immersion for indication of a poor seal as evidenced by a continuous stream of bubbles emanating from the specimen. After the test is completed, the specimen shall be cleaned in a suitable degreaser and permitted to dry thoroughly before any additional tests are performed.

4.5 Precaution. This test condition should not be used either as a separate test or a gross leak test for component parts which are to be subsequently attached to printed circuit board assemblies. Since complete removal of silicone oil residues is difficult, the oil will be transferred unknowingly to other parts during assembly processes. Traces of silicone can result in poor solder wettability of component part leads, poor adhesion to sealants, and mealing of the conformal coating on the printed circuit board.

## 5. TEST CONDITION C.

5.1 Materials.

5.1.1 Tracer gases. When performing tests in accordance with procedures I, II, and IV of this condition, tracer gases, i.e., helium, argon, or other rare gas, or a mixture of a gas with nitrogen (such as 90 percent nitrogen and 10 percent helium) shall be used. The tracer gas used in procedures IIIa and IIIc shall be helium. The tracer gas used in procedure IIIb shall be the radioactive gas, krypton 85.

5.2 Apparatus. For all the procedures of this test condition, the test apparatus, exclusive of pressurization equipment, shall be calibrated using a diffusion type calibrated standard leak at least once each working shift.

5.2.1 Mass-spectrometer-type leak detector. For procedures I, II, IIIa, IIIc, and IV of this test condition, a commercially available mass-spectrometer-type leak detector, preset to read a tracer-gas content, shall be used to measure the leakage rate of gas through a faulty seal. Another instrument may be used if it can be demonstrated to the Government that the instrument, properly calibrated to read tracer-gas content, has the required leakage-detection sensitivity (see 2.1).

5.2.1.1 Chambers. Depending on which procedure is used (see 5.4), suitable pressure or vacuum chambers are required.

5.2.1.2 Pumps. Depending on which procedure is used (see 5.4), suitable pressure or vacuum pumps are required.

5.2.2 Radioactive-gas detection apparatus. Apparatus for procedure IIIb shall consist of:

- a. Radioactive tracer gas activation console.
- b. Counting equipment consisting of a scintillation crystal, photomultiplier tube, preamplifier, ratemeter, and krypton 85 reference standards. The counting station shall be of sufficient sensitivity to determine through the device wall the radiation level of any krypton 85 tracer gas present within the device. The counting station shall have a minimum sensitivity, in c/m per  $\mu\text{Ci}$ , corresponding to a leak rate of  $10^{-9}$  atm  $\text{cm}^3/\text{s}$  of krypton 85 and shall be calibrated at least once every working shift using krypton 85 reference standards and following the equipment manufacturer's instruction.
- c. A tracer gas consisting of a mixture of krypton 85 and dry nitrogen. The concentration of krypton 85 in dry nitrogen shall be no less than 100 microcuries per atmospheric cubic centimeter. This value shall be determined at least once each 30 days and recorded in accordance with the calibration requirements of this standard.

5.3 Supplementary tests. When parts to be tested are normally evacuated through a tube and are sealed in some manner prior to delivery, procedures I and II (see 5.4.1 and 5.4.2) will require a separate verification of the seal of the evacuation tube in conjunction with this test method, using the mass-spectrometer-type leak detector (see 5.2.1). The verification may be accomplished by backfilling the specimen with air or gas at a specified pressure and then submitting the specimen to either procedure IIIa, IIIb, IIIc, or IV.

5.4 Procedures. This test condition consists of five procedures (see 5.4.1 to 5.4.4, inclusive), the choice of which must be specified in the individual specification. Procedure IV is the preferred method of performing this test for parts that are not evacuated.

5.4.1 Procedure I. The mass-spectrometer-type leak detector shall be coupled to the unsealed evacuation tube of the specimen, and a vacuum created within the specimen. It is extremely important that the coupling connections between the specimen and the leak detector be perfectly sealed. The specimen shall then be subjected to a gas atmosphere either by surrounding the specimen with the gas or by spraying the specimen thoroughly with a jet of the gas. If a defect exists in the specimen, an amount of gas that depends upon the size of the defect will be drawn through it and passed into the leak detector, which will read the leakage rate. When this portion of the procedure is completed, the specimen shall be filled with air or gas at a specified pressure and having a known percentage of tracer gas. The evacuation tube shall then be pinched off and sealed. After sealing, the seal of the tube shall be verified by either procedure IIIa, IIIb, IIIc or IV, if filled with tracer gas.

5.4.2 Procedure II. The mass-spectrometer-type leak detector shall be coupled to the chamber. The specimen shall be placed in the chamber, and a tube leading from the gas source and having a known percentage of tracer gas shall be connected to the unsealed evacuation tube of the specimen. It is extremely important that the coupling connections between the specimen and the leak detector be perfectly sealed. The chamber shall then be evacuated and the gas forced under pressure into the specimen. If a leak exists, the gas passes through the specimen into the chamber and then into the leak detector, which will read the leakage rate. If pressure cycling is required, the individual specification shall specify the number of times that a pressure cycle shall be applied. When this portion of the procedure is completed, the specimen shall be filled with air or gas at a specified pressure and having a known percentage of tracer gas. The evacuation tube shall then be pinched off and sealed. After sealing, the seal of the tube shall be verified by either procedure IIIa, IIIb, IIIc or IV, if filled with a tracer gas.

5.4.3 Procedure III. The tests shall be conducted in accordance with procedure IIIa, or IIIc. When bomb pressure specified exceeds the device capability, alternate pressure, exposure time and dwell time may be used provided they satisfy the leak rate pressure, time relationships which apply and provided no less than 30 psig bomb pressure is applied in any case.

#### 5.4.3.1 Testing precautions.

- a. These precautions are applicable to procedures IIIa, IIIb, and IIIc. To avoid spurious indications, contaminants and surface coatings that may conceal existing leaks, and tracer gas that may be absorbed or adsorbed on the surfaces of the specimen, shall be removed. These test procedures shall not apply to organic-coated parts.
- b. Test procedure IIIa is a "fixed" method with specified conditions per table I that will insure the test sensitivity necessary to detect the required leak rate (R1).
- c. Test procedure IIIc is a "flexible" method that allows the variance of test conditions in accordance with the formula of 5.4.3.2.3 to detect the specified equivalent standard leak rate at a predetermined leak rate (R1).

5.4.3.2 General procedure. For IIIa and IIIc the completed device(s) shall be placed in a sealed chamber which is then pressurized with a tracer gas of 100 +0, -5 percent helium for the required time and pressure. The pressure shall then be relieved and each specimen transferred to another chamber or chambers which are connected to the evacuating system and a mass-spectrometer-type leak detector. When the chamber(s) is evacuated, any tracer gas which was previously forced into the specimen will thus be drawn out and indicated by the leak detector as a measured leak rate (R1). The number of devices removed from pressurization for leak testing shall be limited such that the test of the last device can be completed within 60 minutes for procedure IIIa or within the chosen value of dwell time ( $t_z$ ) for procedure IIIc.

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5.4.3.2.1 Procedure IIIa. The device(s) shall be tested using the appropriate conditions specified in table I for the internal cavity volume of the package under test. The time (t) is the time under pressure and time (t<sub>z</sub>) is the maximum time allowed after release of pressure before the device(s) shall be read. This method shall not be used if the maximum equivalent standard leak rate limit given in the procurement document is less than the limits specified herein for procedure IIIc. Upon completion of this procedure, the specimen shall be checked for gross leaks by subjecting the specimen either to test condition A, B, or D. Water, at room ambient temperature and a pressure of 2.5 inches (63.5 mm) of mercury, may be used in place of silicone oil, if test condition B is used to verify gross leaks.

TABLE I. Fixed conditions procedure IIIa.

Volume of package (cm <sup>3</sup> )	Bomb condition			R1 Reject limit (atm cm <sup>3</sup> /s He)
	1bf/in <sup>2</sup> gage	Exposure time hours	Maximum dwell hours	
V < 0.40	60 ±2	2 +0.2, -0	1	5 x 10 <sup>-8</sup>
V ≥ 0.40	60 ±2	2 +0.2, -0	1	2 x 10 <sup>-7</sup>
* V ≥ 0.40	30 ±2	4 +0.4, -0	1	1 x 10 <sup>-7</sup>

5.4.3.2.2 Procedure IIIb.

5.4.3.2.2.1 Activation parameters. The activation pressure and soak time shall be determined in accordance with the following equation:

$$Q_s = \frac{R}{skTPt}$$

The parameters of equation (1) are defined as follows:

- Q<sub>s</sub> = The maximum calculated leak rate allowable, in atm cm<sup>3</sup>/sKr, for the devices to be tested.
- R = Counts per minute above the ambient background after activation if the device leak rate were exactly equal to Q<sub>s</sub>. This is the reject count above the background of both the counting equipment and the component, if it has been through prior radioactive leak tests.
- s = The specific activity, in microcuries per atmospheric cubic centimeter, of the krypton 85 tracer gas in the activation system.
- k = The overall counting efficiency of the scintillation crystal in counts per minute per microcurie of krypton 85 in the internal void of the specific component being evaluated. This factor depends upon component configuration and dimensions of the scintillation crystal. The counting efficiency shall be determined in accordance with 5.4.3.2.2.2.
- T = Soak time, in hours, that the devices are to be activated.
- $\bar{P}$  = P<sub>e</sub><sup>2</sup> - P<sub>i</sub><sup>2</sup>, where P<sub>e</sub> is the activation pressure in atmospheres absolute and P<sub>i</sub> is the original internal pressure of the devices in atmospheres absolute. The activation pressure (P<sub>e</sub>) may be established by specification or if a convenient soak time (T) has been established, the activation pressure (P<sub>e</sub>) can be adjusted to satisfy equation (1).
- t = Conversion of hours to seconds and is equal to 3,600 seconds per hour.

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5.4.3.2.2.2 Determination of counting efficiency (k). The counting efficiency (k) of equation in 5.4.3.2.2.1 shall be determined as follows:

- a. Five representative units of the device type being tested shall be tubulated and the internal void of the device shall be backfilled through the tubulation with a known volume and known specific activity of krypton 85 tracer gas and the tubulation shall be sealed off.
- b. The counts per minute shall be directly read in the shielded scintillation crystal of the counting station in which the devices are read. From this value, the counting efficiency, in counts per minute per microcurie, shall be calculated.

5.4.3.2.2.3 Evaluation of surface sorption. All device encapsulations consisting of glass, metal, and ceramic or combinations thereof, including coatings and external sealants, shall be evaluated for surface sorption of krypton 85 before establishing the leak test parameters. Representative samples of the questionable material shall be subjected to the predetermined pressure and time conditions established for the device configuration as specified by 5.4.3.2.2.1. The samples shall then be counted every 10 minutes, with count rate noted, until the count rate becomes asymptotic with time. (This is the point in time at which surface sorption is no longer a problem.) This time lapse shall be noted and shall determine the "wait time" specified in 5.4.3.2.2.4.

5.4.3.2.2.4 Specific procedure IIIb. The devices shall be placed in radioactive tracer gas activation tank. The activation chamber may be partially filled with inert material to reduce pumpdown time. The tank shall be evacuated to 0.5 torr. The devices shall be subjected to a minimum of 2 atmospheres absolute pressure of krypton 85/dry nitrogen mixture for the time necessary to satisfy the equation. Actual pressure and soak time shall be determined in accordance with 5.4.3.2.2.1. The R value in counts per minute shall be not less than 600 above ambient background. The krypton 85/dry nitrogen gas mixture shall be evacuated to storage until 0.5 torr vacuum exists in the activation tank. This evacuation shall be completed within 3 minutes maximum. The activation tank shall then be backfilled with air (air wash). The devices shall then be removed from the activation tank and leak tested within 1 hour after gas exposure with a scintillation-crystal-equipped counting station. Device encapsulations that come under the requirements of 5.4.3.2.2.3 shall be exposed to ambient air for a time not less than the "wait time" determined by 5.4.3.2.2.3. In no case will the time between removal from the activation chamber and test exceed 1 hour. This exposure shall be performed after gas exposure but before determining leak rate with the counting station. Device encapsulations that do not come under the requirements of 5.4.3.2.2.3 may be tested without a "wait time". (The number of devices removed from pressurization for leak testing shall be limited such that the test of the last device can be completed within 1 hour.) The actual leak rate of the component shall be calculated with the following equation:

$$Q = \frac{(ACTUAL READOUT IN NET COUNTS PER MINUTE) \times Q_S}{R}$$

Where Q = Actual leak rate in atm cm<sup>3</sup>/s, and Q<sub>S</sub> and R are defined in 5.4.3.2.2.1.

Unless otherwise specified, devices that exhibit a leak rate equal to or greater than 1 x 10<sup>-8</sup> atmospheric cubic centimeters of krypton 85 per second shall be considered a failure.

Upon completion of this procedure, the specimen shall be checked for gross leaks by subjecting the specimen either to test condition A, B, or D. Water, at room ambient temperature and a pressure of 2.5 inches (63.5 mm) of mercury, may be used in place of silicone oil, if test condition B is used to verify gross leaks.

5.4.3.2.2.5 Personnel precautions. A Nuclear Regulatory Commission (NRC) license is necessary for possession and use of the krypton 85 leak-test equipment. In the use of gas, code of Federal regulations Nuclear Regulatory Commission Rules and Regulations, Title 10, Chapters 1, 20, 30, 31, and 32 should be followed and the maximum permissible tolerance levels prescribed by the National Committee on Radiological Protection should be observed.

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5.4.3.2.3 Procedure IIIc. Values for bomb pressure exposure time and dwell time shall be chosen such that actual measured tracer gas leak rate ( $R_1$ ) reading obtained for the device under test (if defective) will be greater than the minimum detection sensitivity capability of the mass spectrometer. The devices shall be subjected to a minimum of 2 atmospheres absolute of helium atmosphere. If the chosen dwell time ( $t_2$ ) is greater than 60 minutes, graphs shall be plotted to determine an  $R_1$  value which will assure overlap with the selected gross leak test condition. The chosen values, in conjunction with the value of the internal volume of the device package to be tested and the maximum equivalent standard leak rate (L) limit (as shown below or as specified in the applicable procurement document), shall be used to calculate the measured leak rate ( $R_1$ ) limit using the following formula:

$$R_1 = \frac{LP_E}{P_O} \left( \frac{M_A}{M} \right)^{1/2} \left\{ 1 - e^{-\left[ \frac{Lt_1}{VP_O} \left( \frac{M_A}{M} \right)^{1/2} \right]} \right\} e^{-\left[ \frac{Lt_2}{VP_O} \left( \frac{M_A}{M} \right)^{1/2} \right]}$$

- $R_1$  = The measured leak rate of tracer gas (He) through the leak in atm cm<sup>3</sup>/s He.
- L = The equivalent standard leak rate in atm cm<sup>3</sup>/s air.
- $P_E$  = The pressure of exposure in atmospheres absolute.
- $P_O$  = The atmospheric pressure in atmospheres absolute (1 atmosphere).
- $M_A$  = The molecular weight of air in grams (28.7 grams).
- M = The molecular weight of the tracer gas (helium) in grams (4 grams).
- $t_1$  = The time of exposure to  $P_E$  in seconds.
- $t_2$  = The dwell time between release of pressure and leak detection in seconds.
- V = The internal volume of the device package cavity in cubic centimeters.

Unless otherwise specified, devices with an internal cavity volume of 0.01 cm<sup>3</sup> or less shall be rejected if the equivalent standard leak rate (L) exceeds  $5 \times 10^{-8}$  atm cm<sup>3</sup>/s. Devices with an internal cavity volume greater than 0.01 cm<sup>3</sup> and equal to or less than 0.4 cm<sup>3</sup> shall be rejected if the equivalent standard leak rate (L) exceeds  $1 \times 10^{-7}$  atm cm<sup>3</sup>/s. Devices with an internal cavity volume greater than 0.4 cm<sup>3</sup> shall be rejected if the equivalent standard leak rate (L) exceeds  $1 \times 10^{-6}$  atm cm<sup>3</sup>/s.

Upon completion of this procedure, the specimen shall be checked for gross leaks by subjecting the specimen either to test condition A, B, or D. Water, at room ambient temperature and a pressure of 2.5 inches of mercury, may be used in place of silicone oil, if test condition B is used to verify gross leaks.

5.4.4 Procedure IV. The specimen, which is backfilled and sealed during production with a known percentage of tracer gas, shall be placed in a chamber connected to the mass-spectrometer-type leak detector, and the chamber evacuated. The internal pressure of the specimen shall be one atmosphere or greater. If a leak exists, the gas passes through the specimen into the chamber and then into the leak detector which will read the leakage rate. If specified, the specimen shall be perforated to determine if a gas is actually present. Upon completion of this procedure, the specimen shall be checked for gross leaks by subjecting the specimen either to test condition A, B, or D. Water, at room ambient temperature and a pressure of 2.5 inches (63.5 mm) of mercury, may be used in place of silicone oil, if test condition B is used to verify gross leaks.

6. TEST CONDITION D.

6.1 Materials.

6.1.1 Fluorocarbon liquid. The fluid shall be D02, D02-TS, D03, FC-40 1/ or FC-43 2/.

6.2 Apparatus.

6.2.1 Heated fluid container. The container for the fluid shall be made of pyrex glass and shall be sufficient size to hold the test specimen in the fluid and to maintain a temperature of 125°C ±5°C (257°F ±9°F).

6.2.2 Filter. A filter shall be used which is capable of removing particles greater than 1 micron in size from the fluid.

6.2.3 Magnifier. A 3X minimum magnifier or a stereo zoom microscope shall be used for observation of bubbles emanating from the test specimens when immersed in the indicator fluid.

6.3 Precautions. The following precautions shall be observed:

- a. Fluorocarbon liquids shall be filtered as specified in 6.2.2. Bulk filtering and storage is permissible. Leak indicator fluids, which have accumulated observable quantities of particulate matter during use, shall be discarded or reclaimed by filtration for reuse. Leak detecting fluids shall not be used for more than one eight-hour shift without being refiltered.
- b. The observation container shall contain sufficient fluid to assure coverage of the test specimen to a minimum depth of two inches.
- c. Illumination shall be a lighting source capable of providing at least 15,000 foot candles at the test specimen position. The lighting source shall not require calibration but the light level shall be verified at the test specimen position. The background behind the glass observation container should be a dull, non-reflective black material.
- d. The observation container should be covered at all times when not in use to minimize evaporation losses and moisture adsorption.
- e. Test specimens to be tested should be free from foreign material on the surface, including conformal coatings and markings which may contribute to erroneous test results.

6.4 Procedure. The test specimens, mounted on specified fixtures to hold them in the fluid, shall be immersed, with the uppermost portion at the minimum depth of 2 inches below the surface of the fluid, lid downward, one at a time (or in such a configuration that a single bubble from a single specimen out of a group under observation may be clearly observed as to its occurrence and source). The fluid shall be maintained at a temperature of 125°C ±5°C (257°F ±9°F). The specimens shall be observed against a dull non-reflective black background through the magnifier (see 6.2.3) from the instant of immersion until 20 seconds after immersion. Leakers will be identified by a single bubble or a stream of bubbles. Specimens from which a single bubble is observed is considered to be a reject.

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1/ D02, D02-TS, and D03 are the registered trade mark of Ausimont (Division of Montedison).

2/ Minnesota Mining Co. (3M) registered trade name.

7. TEST CONDITION E.

7.1 Materials.

7.1.1 Fluorocarbon liquid. The fluids shall be D/80, FC-72 or PP-1 fluorocarbon detector fluids, and D02, D02-TS, D03, FC-40, FC-43, PP-7 or PP-9 fluorocarbon indicator fluids.

7.2 Apparatus.

7.2.1 Heated fluid container. The container for the fluid shall be made of pyrex glass and shall be of sufficient size to hold the test specimen in the fluid and to maintain a temperature of  $125^{\circ}\text{C} \pm 5^{\circ}\text{C}$  ( $257^{\circ}\text{F} \pm 9^{\circ}\text{F}$ ).

7.2.2 Filter. A filter shall be used which is capable of removing particles greater than 1 micron in size from the fluid.

7.2.3 Magnifier. A magnifier with a magnification in the range between 3X to 30X for observation of bubbles emanating from devices when immersed in the indicator fluid.

7.2.4 Chamber. A vacuum/pressure chamber for the evacuation and subsequent pressure bombing of device up to  $75 \text{ lbf/in}^2$  up to 10 hours.

7.2.5 Lighting source. A lighting source capable of producing at least 15,000 foot candles in air at a distance equal to that which the most distant device in the bath will be from the source.

7.2.6 Instruments. Suitable calibrated instruments to indicate the test temperature pressures and times are as specified.

7.2.7 Fixtures. Suitable fixtures to hold the device(s) in the indicator fluid.

7.3 Precautions. The following precautions shall be observed.

- a. Fluorocarbon liquids shall be filtered as specified in 7.2.2. Bulk filtering and storage is permissible. Leak indicator fluids, which have accumulated observable quantities of particulate matter during use, shall be discarded or reclaimed by filtration for reuse. Leak detecting fluids shall not be used for more than one eight-hour shift without being refiltered.
- b. The observation container shall contain sufficient fluid to assure coverage of the test specimen to a minimum depth of two inches (50.8 mm).
- c. Illumination shall be a lighting source capable of providing at least 15,000 foot candles at the test specimen position. The lighting source shall not require calibration but the light level shall be verified at the test specimen position. The background behind the glass observation container should be a dull, non-reflective black material.
- d. The observation container should be covered at all times when not in use to minimize evaporation losses and moisture adsorption.
- e. Test specimens to be tested should be free from foreign material on the surface, including conformal coatings and markings that may contribute to erroneous test results.
- f. Precautions should be taken to prevent operator injury due to package rupture or violent evolution of bomb fluid when testing large packages.

7.4 Procedure. The test specimens mounted on specified fixtures to hold them in the leak detecting fluid shall be inserted into the combination vacuum-pressure vessel and the applied ambient pressure shall be reduced to less than 5 torr for 1 hour. Then without breaking vacuum, a sufficient quantity of leak detecting fluid (see 7.1.1) to cover the test specimens, shall be drawn into the vacuum/pressure vessel by inserting a transfer tube from the vacuum/pressure vessel into a container of leak detecting fluid and opening a valve in the tube. For test specimens with an internal cavity volume of  $0.1 \text{ cm}^3$  or less, the pressure in the vessel shall be increased to  $90 \text{ lbf/in}^2$  and maintained at that pressure for 3 hours minimum. For test specimens with an internal volume in excess of  $0.1 \text{ cm}^3$ , the pressure shall be increased to  $50 \text{ lbf/in}^2$  and maintained at that pressure for 3 hours minimum. After pressurization, the pressure shall be released from the pressure vessel and the test specimens shall be removed from the pressure vessel, and retained in a bath containing the leak detecting fluid. Then they shall be dried for  $3 \pm 1$  minutes in air prior to immersion in the indicator fluid. The test specimens shall be immersed with the uppermost portion at a minimum depth of 2 inches (50.8 mm) below the surface of the indicator fluid, lid downward, one at a time (or in such a configuration that a single bubble from a single specimen out of a group under observation may be clearly observed as to its occurrence and source. The leak indicator fluid shall be maintained at a temperature of  $125^\circ\text{C} \pm 5^\circ\text{C}$  ( $257^\circ\text{F} \pm 9^\circ\text{F}$ ). The test specimens shall be observed against a dull non-reflective black background through the magnifier (see 7.2.3) from the instant of immersion until 20 seconds after immersion. Leaks will be identified by a single bubble or a stream of bubbles. Specimens from which a single bubble is observed is considered to be a reject.

## 8. TEST CONDITION F.

### 8.1 Materials.

8.1.1 Fluorocarbon liquid. The fluid used shall be FC-84 or D/80 fluorocarbon detector fluid.

### 8.2 Apparatus.

8.2.1 Chamber. A vacuum/pressure chamber capable of the evacuation and subsequent pressure bombing of devices up to 75 psig and maintaining pressure up to 12.5 hours.

8.2.2 Filter. A filtration system capable of removing particles greater than 1 micron in size from the fluorocarbon fluid.

8.2.3 Detector. A fluorocarbon vapor detection system with a nominal sensitivity of  $10^{-5} \text{ atm cm}^3/\text{s}$ . The sensitivity of the detector shall be better than 14 micrograms of FC84 or D/80. The detector shall have a linear response to 280 micrograms and a response time of better than 1 second.

8.2.4 Instruments. Suitable calibrated instruments to indicate that test, pressure times and temperatures are as specified. The detection system shall be calibrated using a calibration source of fluorocarbon at least once each shift to an accuracy of at least  $\pm 15$  percent.

8.3 Precautions. The following precautions shall be observed in conducting the fluorocarbon gross leak test.

- a. Fluorocarbons shall be filtered through a filter system capable of removing particles greater than 1 micrometer prior to use. Bulk filtering and storage is permissible. Liquid that has accumulated observable quantities of particulate matter during use shall be discarded or reclaimed by filtration for re-use. Precautions shall be taken to prevent contamination.
- b. Precautions should be taken to prevent operator injury due to package rupture or violent evolution of bomb fluid when testing large packages.

8.4 Procedure. The devices shall be placed in a vacuum/pressure chamber and the pressure reduced to 5 torr or lower and maintained for 30 minutes minimum. A sufficient amount of FC-84 or D/80 detector fluid shall be admitted to the pressure chamber to completely cover the devices. The fluid shall be admitted after the 30 minute minimum vacuum period but before breaking the vacuum. The devices shall then be pressurized in accordance with table II. Upon completion of the pressurization period, the pressure shall be released and the devices removed from the pressure chamber without being removed from the detector fluid for more than 20 seconds. When the devices are removed from the fluid for testing, they shall be air dried for a minimum of 20 seconds and a maximum of 5 minutes prior to the test cycle. The devices shall then be tested with a fluorocarbon vapor detector in accordance with the system operating instructions and as follows. Devices shall be admitted to the purge chamber and purged (heated to remove any remaining surface fluorocarbon and to initiate vaporization of any internal fluorocarbon fluid). Purge time shall be in accordance with table III. Test time shall commence immediately after purge either in the same chamber (single chamber system) or an adjoining test chamber (dual chamber system). Test time shall be a minimum of 3.5 seconds (unless the device is rejected earlier) with the fluorocarbon vapor detector purge and test chambers at a temperature of 125°C ±5°C or 2.5 seconds minimum with the purge and test chambers at 150°C ±5°C. During test time, the amount of fluorocarbon outgassing is measured and the total integrated value is compared to the alarm level.

NOTE: Air dry, purge, and test time limits for each device shall be complied with in all cases, whether the device is handled individually or in plastic or metal tubes.

8.4.1 Failure criteria. A device shall be rejected if the detector instrumentation indicates more than 280 micrograms of fluorocarbon gas.

TABLE II. Test pressure and duration for condition F.

Test pressure	Duration
75 psig	.5 hr
60 psig	1.0 hr
30 psig	2.5 hr
15 psig	12.5 hr

TABLE III. Purge time for condition F.

Package with internal free volume (cm <sup>3</sup> )	Purge time <sup>1/</sup> (seconds)
≤ 0.01	3 1/3-5
>0.01 ≤0.10	6-9
>0.10	8 2/3-13

<sup>1/</sup> With purge chamber at 150°C, minimum and maximum purge times should be reduced by 1/3.

NOTE: If necessary, due to device construction or materials, maximum purge time can be determined by cycling a device with a 0.02 to 0.05 inch diameter hole and measuring the maximum time that can be used without permitting the device to escape detection during the test cycle.

9. Measurements. Upon completion of the test, measurements shall be made as specified.
10. Summary. The following details are to be specified in the individual specification.
  - a. Test condition letter (see 2.1).
    - (1) For test condition C.
      - (a) Procedure number and accept and reject leak rate.
      - (b) Backfilling pressure when verification of seal is required (see 5.3, 5.4.1 and 5.4.2).
      - (c) Number of pressure cycles if applicable (see 5.4.2).
      - (d) For procedure IV.
        1. Reduced pressure of the chamber and duration of pressurization (see 5.4.4).
        2. Whether perforation of specimen is required (see 5.4.4).
      - (e) For procedures III and IV.
        1. Applicable gross leak conditions.
    - (2) For test conditions D and E.
      - (a) Test mounting fixtures (see 6.4 and 7.4).
        1. Measurements after test (see 9).