Designation: F 97 – 72 (Reapproved 1997)ε1

Standard Practices for Determining Hermeticity of Electron Devices by Dye Penetration[1]

This standard is issued under the fixed designation F 97; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

ε[1] NOTE—Keywords were added editorially in December 1997.

1. Scope

1.1 These practices cover procedures that will normally detect and locate the sites of gross leaks in electron devices.

1.2 These procedures are suitable for use on selected parts during receiving inspection or to verify and locate leakage sites for production control. They are not quantitative; no indication of leak size can be inferred from the test.

1.3 These procedures are most suitable for use on transparent glass-encased devices; all methods are applicable to transparent parts with an internal cavity. Method A, Penetrant-Capillary, is also applicable to parts, such as terminals, end seals or base assemblies, without an internal cavity, and Method C, Penetrant-Pressure Followed by Vacuum, can be used on opaque parts with an internal cavity. Method B, Penetrant-Pressure, can also be used on opaque parts with an internal cavity if the part is opened after dye penetration and before inspection. Parts that have an internal cavity may either contain gas (such as air, nitrogen, nitrogen-helium mixture, etc.) or be evacuated. These procedures are not suitable for use on grease-filled components.

1.4 Because of the possibility of dye entrapment between the component and an attached part, components with mechanically attached parts, such as a radiator on a power transistor, should be tested before the attachment is made or after it has been removed.

NOTE 1—Alternative methods for determining hermeticity of electron devices may be found in Practices F 98 (see 2.1) and Test Methods F 134 (see 2.1).

1.5 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.6 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.* For specific hazard statement, see Section 8.

2. Referenced Documents

2.1 *ASTM Standards:*

E 165 Practice for Liquid Penetrant Examination[2]

F 98 Practices for Determining Hermeticity of Electron Devices by a Bubble Test[3]

F 134 Test Methods for Determining Hermeticity of Electron Devices with a Helium Mass Spectrometer Leak Detector[4]

2.2 *Other Standards:*

MIL-STD-105 Sampling Procedures and Tables for Inspection by Attributes[5]

MIL-STD-883 Test Methods and Procedures for Microelectronics[6]

3. Summary of Practice

3.1 In these methods, a penetrant dye solution is applied locally to an area of the component to be tested for leaks, or the entire component is immersed in a bath of the penetrant. After contact with the penetrant dye for a specified time, the device is cleaned and visually inspected for dye penetration.

3.2 *Method A, Penetrant-Capillary*—The penetrant dye is applied locally, or the device is immersed in a bath of the dye. This method, in which no external pressure is exerted to force the penetrant dye through the leak, is particularly suited to components and subassemblies without an internal cavity or to subassemblies that cannot be totally immersed.

[1] These practices are under the jurisdiction of ASTM Committee F-1 on Electronics and are the direct responsibility of Subcommittee F01.03 on Metallic Materials.

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[2] *Annual Book of ASTM Standards*, Vol 03.03.

[3] Discontinued; see *1991 Annual Book of Standards*, Vol 10.04.

[4] Discontinued; see *1996 Annual Book of ASTM Standards*, Vol 10.04.

[5] Available from the Superintendent of Documents, Government Printing Office, Washington, DC 20402.

[6] Available from Standardization Documents, Order Desk, Bldg. 4, Section D, 700 Robbins Ave., Philadelphia, PA 19111-5094, ATTN: NPODS.

3.3 *Method B, Penetrant-Pressure*—The component, with an internal cavity, is immersed in a bath of the penetrant dye. The bath is pressurized to force the dye solution through any leaks that may exist.

3.4 *Method C, Penetrant-Pressure Followed by Vacuum*—The component, with an internal cavity, is immersed in a bath of the penetrant dye, which may or may not be pressurized. After being carefully cleaned, the component is placed in a vacuum chamber. Any dye solution that may have entered a leak during immersion is forced out by the internal pressure of the cavity.

3.5 *Selection of Method*—The selection of the method to perform a dye penetration test depends on the nature and physical limitations of the component part (see 1.3). Method B and the pressure option of Method C cannot be used on components or subassemblies when pressure would exceed the rating of the component or subassembly (Note 4).

4. Significance and Use

4.1 Contaminants that will reduce the effective life of electron devices may enter the device through small leaks. These leaks are most frequently found at seals between parts of the same or dissimilar materials. Leaks may also result from porosity of a defective portion of the case.

4.2 Dye-penetrant procedures are applicable only to individual gross leaks in a system. The presence of a number of small leaks that may result in a cumulatively unacceptable leak rate when measured by other techniques will not be indicated if each individual leak is below the level of sensitivity of the test.

4.3 There is no general agreement concerning level of leakage which is likely to be deleterious to a particular device. However, since these tests are designed to detect gross leakage, components that exhibit any indication of leakage are normally rejected.

4.4 Since leaks may change in size with different ambient conditions, comparisons between test stations are not conclusive. Therefore these methods are usually employed as go, no-go tests.

5. Interferences

5.1 Since small leaks may become clogged by the dye solution in which the device is immersed, fine leak tests such as a helium mass spectrometer test of Test Methods F 134, if employed, must precede this test.

6. Apparatus

6.1 *Bath*—Container of sufficient volume to allow components or subassemblies to be completely immersed. (Not required for the local-application option of Method A, see 6.5). For use with Method B or the pressure option of Method C it must be possible to increase the pressure over the surface of the bath to meet the requirements of 10.4.1. The bath is maintained at room temperature (20 to 30°C).

6.2 *Light Source*—For use with fluorescent dye penetrants, a 100-W mercury-vapor bulb of the sealed reflector type with a filter with peak transmission at 365 nm and minimum transmission at visible wavelengths. This light source shall have a minimum intensity of 100 fc (1080 lx) at 380 mm (15 in.) from the face of the bulb.

6.3 *Microscope*—Magnification 7× to 20×.

6.4 *Air Jet*.

6.5 *Method A (local-application option only)*:

6.5.1 *Dye Dispenser*, such as an eye dropper, glass rod, swab or brush, for local application of the penetrant dye.

6.6 *Method B (for opaque parts only)*:

6.6.1 Facilities to cut open part.

6.7 *Method C*:

6.7.1 *Vacuum Chamber* capable of being pumped to a pressure not greater than 38 torr (5060 Pa).

7. Materials

7.1 *Penetrant-Dye Solution*, either visible (nonfluorescent) or fluorescent to which between 20 and 25 volume % of a nonionic wetting agent has been added.

NOTE 2—Lack of quantitative data does not permit one to relate penetrant sensitivity to composition or physical properties of the penetrant-dye solution. The choice of penetrant therefore depends on such factors as the sensitivity needed and the interaction of the penetrant with the materials tested. This choice in turn, depends on various features, for example, whether the penetrant base is water washable or not, and the type of inhibitor used. Various types of penetrant-dye solutions are discussed in Practice E 165. The solution chosen shall be agreed upon by the parties to the test. Examples of penetrant-dye solutions that have been found to be suitable may be found in Method 1014 of MIL-STD-883 and in the literature.[7]

7.2 *Penetrant-Dye Developer*, or post emulsifier or both as required by the solution chosen.

7.3 *Dye Cleaner*, as recommended by the supplier of the penetrant dye.

7.4 *Methanol*.

8. Hazards

8.1 Some packages may explode when pressurized or placed in vacuum. Protection against such explosion should be provided in Methods B and C.

8.2 Ultraviolet light with a wavelength less than 360 nm can cause severe injury to the eyes and skin. Protection should be provided if fluorescent dyes are used.

9. Sampling

9.1 Since dye-penetration testing is normally considered destructive, testing performed as part of incoming inspection must be done on a sample basis. The method of sampling shall be agreed upon by the parties to the test and shall be in accordance with acceptable statistical procedures (see MIL-STD-105).

10. Procedure

10.1 General procedures for precleaning, drying, penetrant application, removal of excess penetrants, developing, drying, and inspection are given in Practice E 165.

[7] Guidelines for Hermetic Seal Testing of Semiconductor Devices, Defense Electronics Supply Center, Code ECS, Dayton, OH 45401, pp. 3.1.4 and 4.3.

10.2 In general, precleaning of electron devices prior to penetrant application should be unnecessary. If parts appear to be greasy or dirty or if difficulty is encountered in application of the penetrant-dye solution, clean and dry according to appropriate procedures in 6.3 of Practice E 165.

10.3 *Method A*:

10.3.1 Apply the penetrant-dye solution at room temperature to the area of the part under investigation by immersion in the bath or by localized application with the dye dispenser. Allow the penetrant-dye solution to remain in contact with the component for a minimum of 3 h. For increased sensitivity, a contact duration of at least 16 h is preferred.

10.3.2 Carefully remove excess penetrant-dye solution from the part by washing in the dye cleaner; for some solutions use of a post-emulsifier may be required. Rinse with methanol; then dry with an air jet. Avoid ultrasonic agitation as it may remove the penetrant from the leak sites.

10.3.3 *Inspection*:

10.3.3.1 *Terminals, Seals, and Other Parts Without an Internal Cavity*—Coat the surface opposite to that on which the solution was applied with the appropriate dye developer (5.5 of Practice E 165) if required by the penetrant-dye solution. Illuminate the surface opposite to that on which the penetrant-dye was applied with daylight or 100-W incandescent bulb or equivalent, minimum illumination 1080 1x if coated with visible dye or the filtered mercury bulb (see 7.2 and Note 3) if coated with fluorescent dye and inspect under the microscope.

NOTE 3—**Precaution:** Protect eyes and skin from direct exposure to ultraviolet light source (see 8.2).

10.3.3.2 *Transparent Parts with an Internal Cavity*—Illuminate the part with daylight or mercury bulb (**Precaution**—Note 3) as specified in 10.3.3.1 and inspect under the microscope. View the part so that light passes through the part toward the microscope.

10.4 *Method B*:

10.4.1 Immerse the part in a bath of the penetrant dye at room temperature. Pressurize the bath, at 620 kPa (90 psig, gage) minimum, for a minimum of 2 h. For increased sensitivity, application of pressure for at least 16 h is preferred. **Precaution**—Use eye protection to avoid injury from packages that might explode when pressurized.

NOTE 4—When the specified bath pressure exceeds the package capability, lower pressures may be used provided that the time of exposure is increased to maintain a time-pressure product of at least 12 atm·h (1240 kPa·h or 180 psig·h). The effect of this change on the sensitivity of the test has not yet been established; any deviations from specified test conditions must be agreed to by parties to the test.

10.4.2 Clean the part in accordance with 10.3.2.

10.4.3 Inspect transparent parts in accordance with 10.3.3.2. For opaque parts, cut open the cavity and inspect in accordance with 10.3.3.1.

10.5 *Method C*:

10.5.1 Immerse the part in a bath of the penetrant dye at room temperature. If no external pressure is applied, maintain the part in the bath for a minimum of 3 h. If the bath is pressurized as in 10.4.1, maintain the part in the bath for a minimum of 2 h. In either case, a period of at least 16 h is preferred for increased sensitivity. **Precaution**—Use eye protection to avoid injury from packages that may explode when pressurized.

10.5.2 Clean the part as described in 10.3.2 to remove all external traces of the dye. Inspect the surfaces coated with penetrant-dye in accordance with 10.3.3.1 to establish that all external traces of the dye have been removed.

10.5.3 Place the part in the vacuum chamber and reduce the pressure to a maximum of 5060 Pa (38 torr). **Precaution**—Use eye protection to avoid injury from packages that may explode when placed in vacuum. Hold under vacuum for 5 to 30 min.

10.5.4 Inspect the outside surfaces of the part as required in 10.3.3.1.

11. Interpretation of Results

11.1 Evidence of dye penetration to the opposite side of the part or to the interior of the cavity shall be taken as the indication of the presence of a leakage site in Methods A and B. Evidence of dye on the exterior of the part shall be taken as the indication of the presence of a leakage site in Method C.

12. Report

12.1 Report the following information:

12.1.1 Identification of the specimen,

12.1.2 Method used,

12.1.3 Identification of the penetrant,

12.1.4 Duration of contact with the penetrant,

12.1.5 Pressure (for Method B and the pressure option of Method C),

12.1.6 Vacuum pressure and duration (for Method C),

12.1.7 Method of inspection, and

12.1.8 Qualitative description and sketch of the leakage sites. If no leakage sites were found, report that the component does not leak when tested in accordance with this procedure.

13. Keywords

13.1 dye penetrant; electron devices; hermetic seals; visual inspection