

* METHOD 1018.5

* INTERNAL GAS ANALYSIS

1. PURPOSE. The purpose of this test is to measure the water-vapor content of the atmosphere inside a metal or ceramic hermetically-sealed device. It can be destructive (procedures 1 and 2) or nondestructive (procedure 3).

* 2. APPARATUS. The apparatus for the internal gas analysis test shall be as follows for the chosen procedure:

2.1 Procedure 1. (Procedure 1 measures the water-vapor content of the device atmosphere by mass spectrometry.) The apparatus for procedure 1 shall consist of:

a. A mass spectrometer meeting the following requirements:

- (1) Spectra range. The mass spectrometer shall be capable of reading a minimum spectra range of 1 to 100 atomic mass units (AMUs).
- (2) Detection limit. The mass spectrometer shall be capable of reproducibly detecting the specified moisture content for a given volume package with signal to noise ratio of 20 to 1 (i.e., for a specified limit of 5,000 ppmv, .01 cc, the mass spectrometer shall demonstrate a 250 ppmv minimum detection limit to moisture for a package volume of .01 cc). The smallest volume shall be considered the worst case.
- (3) Calibration. The calibration of the mass spectrometer shall be accomplished at the specified moisture limit (± 20 percent) using a package simulator which has the capability of generating at least three known volumes of gas ± 10 percent on a repetitive basis by means of a continuous sample volume purge of known moisture content ± 10 percent. Moisture content shall be established by the standard generation techniques (i.e., 2 pressure, divided flow, or cryogenic method). The dew point analyzer shall be recalibrated a minimum of once per year using equipment traceable to NIST or by a suitable commercial calibration services laboratory using equipment traceable to NIST standards. Calibration records shall be kept on a daily basis. Gas analysis results obtained by this method shall be considered valid only in the moisture range or limit bracketed by at least two (volume or concentration) calibration points (i.e., 5,000 ppmv between .01 - .1 cc or 1,000 - 5,000 ppmv between .01 - .1 cc). A best fit curve shall be used between volume calibration points. Systems not capable of bracketing may use an equivalent procedure as approved by the qualifying activity. Corrections of sensitivity factors deviation greater than 10 percent from the mean between calibration points shall be required.

NOTE: It is recommended that the percentage of water vapor contained in a gas flowing through the gas humidifier be compared to the dew point sensor reading for accuracy of the sensor. The following equation may be used to calculate the percent of water vapor contained in a gas flowing through the gas humidifier.

$$\% H_2O = \frac{100(P_{vmb})}{68.95 \text{ mb/psi } P_g + 1.33 \text{ mb/mmPa}}, \text{ where}$$

P_v = vapor pressure of water in the GPH based on water temperature in degrees centigrade,

P_g = gauge pressure in psi, and

P_a = atmospheric pressure in mm Hg.

- (4) Calibration for other gases. Calibration shall be required for all gases found in concentrations greater than .01 percent by volume. As a minimum, this shall include all gases listed in 3.1c. The applicable gases shall be calibrated at approximately 1 percent concentrations as part of the yearly calibration requirements, with the exception of fluorocarbons, which may use a concentration of approximately 200 ppmv; nitrogen, which may use a concentration of approximately 80 percent or more; helium, which may use a concentration of approximately 10 percent; and oxygen, which may use a concentration of approximately 20 percent.

- (5) Calibration check. The system calibration shall be checked on the day of test prior to any testing. This shall include checking the calibration by in-letting a 5000 ppmv $\pm 20\%$ moisture calibration sample of the required volumes and comparing the result with the calibration sample. The resulting moisture reading shall be within 250 ppmv of the moisture level in the calibration sample. Calibration performed on the day of test prior to any testing may be substituted for the calibration check.
- b. A vacuum opening chamber which can contain the device and a vacuum transfer passage connecting the device to the mass spectrometer of 2.1a. The system shall be maintained at a stable temperature equal to or above the device temperature. The fixturing in the vacuum opening chamber shall position the specimen as required by the piercing arrangement of 2.1c, and maintain the device at $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for a minimum of 10 minutes prior to piercing.

Note: A maximum 5-minute transfer time from prebake to hot insertion into apparatus shall be allowed. If 5 minutes is exceeded, device shall be returned to the prebake oven and the prebake continued until device reaches $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$.

For initial certification of systems or extension of suitability, device temperature on systems using an external fixture shall be characterized by placing a thermocouple into the cavity of a blank device of similar mass, internal volume, construction and size. This shall be a means for proving the device temperature has been maintained at $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ for the minimum ten minutes. This also applies to devices prebaked in an external oven but tested with the external fixture to adjust for any temperature drop during the transfer. These records shall be maintained by the test laboratory.

- c. A piercing arrangement functioning within the opening chamber or transfer passage of 2.1b, which can pierce the specimen housing (without breaking the mass spectrometer chamber vacuum and without disturbing the package sealing medium), thus allowing the specimen's internal gases to escape into the chamber and mass spectrometer.

NOTE: A sharp-pointed piercing tool, actuated from outside the chamber wall via a bellows to permit movement, should be used to pierce both metal and ceramic packages. For ceramic packages, the package lid or cover should be locally thinned by abrasion to facilitate localized piercing.

2.2 Procedure 2. (Procedure 2 measures the water-vapor content of the device atmosphere by integrating moisture picked up by a dry carrier gas at 50°C .) The apparatus for procedure 2 shall consist of:

- a. An integrating electronic detector and moisture sensor capable of reproducibly detecting a water-vapor content of 300 ± 50 ppmv moisture for the package volume being tested. This shall be determined by dividing the absolute sensitivity in micrograms H_2O by the computed weight of the gas in the device under test, and then correcting to ppmv.
- b. A piercing chamber or enclosure, connected to the integrating detector of 2.2a, which will contain the device specimen and maintain its temperature at $100^{\circ}\text{C} \pm 5^{\circ}\text{C}$ during measurements. The chamber shall position the specimen as required by the piercing arrangement. The piercing mechanism shall open the package in a manner which will allow the contained gas to be purged out by the carrier gas or removed by evacuation. The sensor and connection to the piercing chamber will be maintained at a temperature of $50^{\circ}\text{C} \pm 2^{\circ}\text{C}$.

2.3 Procedure 3. (Procedure 3 measures the water-vapor content of the device atmosphere by measuring the response of a calibrated moisture sensor or an IC chip which is sealed within the device housing, with its electrical terminals available at the package exterior.) The apparatus for procedure 3 shall consist of one of the following:

- a. A moisture sensor element and readout instrument capable of detecting a water-vapor content of 300 ± 50 ppmv while sensor is mounted inside a sealed device.
- b. Metallization runs on the device being tested isolated by back-biased diodes which when connected as part of a bridge network can detect 2,000 ppmv within the cavity. The chip shall be cooled in a manner such that the chip surface is the coolest surface in the cavity. The device shall be cooled below dew point and then heated to room temperature as one complete test cycle.

NOTE: Suitable types of sensors may include (among others) parallel or interdigitated metal stripes on an oxidized silicon chip, and porous anodized-aluminum structures with gold-surface electrodes.

Surface conductivity sensors may not be used in metal packages without external package wall insulation. When used, the sensor shall be the coolest surface in the cavity. It should be noted that some surface conductivity sensors require a higher ionic content than available in ultraclean CERDIP packages. In any case, correlation with mass spectrometer procedure 1 shall be established by clearly showing that the sensor reading can determine whether the cavity atmosphere has more or less than the specified moisture limit at 100°C.

- * 3. PROCEDURE. The internal gas analysis test shall be conducted in accordance with the requirement of procedure 1, procedure 2, or procedure 3. All devices shall be prebaked for 16-24 hours at 100°C ±5°C prior to hot insertion into apparatus. External ovens shall have a means to indicate if a power interruption occurs during the prebaking period and for how long the temperature drops below 100 ±5°C. Devices baked in an external oven which loses power and whose temperature drops below 100 ±5°C for more than 1 hour shall undergo another prebake to begin a minimum of 12 hours later.

Note: It is recommended that samples submitted to the labs shall include information about the manufacturing process including sealing temperature, sealing pressure, sealing gas, free internal cavity volume, lid thickness at puncture site, lid material, and the location of the puncture site.

- * 3.1 Procedure 1. The device shall be hermetic in accordance with test method 1014, and free from any surface contaminants which may interfere with accurate water-vapor content measurement. The internal gas analysis laboratory is not required to test for hermeticity in accordance with Test Method 1014 of MIL-STD-883.

After device insertion, the device and chamber shall be pumped down and baked out at a temperature of 100°C ±5°C until the background pressure level will not prevent achieving the specified measurement accuracy and sensitivity. After pumpdown, the device case or lid shall be punctured and the following properties of the released gases shall be measured, using the mass spectrometer:

- a. The increase in chamber pressure as the gases are released by piercing the device package. A pressure rise of less than 50 percent of normal for that package volume and pressurization may indicate that (1) the puncture was not fully accomplished, (2) the device package was not sealed hermetically, or (3) does not contain the normal internal pressure.
- b. The water-vapor content of the released gases, as a percent by unit volume or parts per million volume (ppmv) of the total gas content.
- c. The proportions (by volume) of the other following gases: N₂, He, Mass 69 (fluorocarbons), O₂, Ar, H₂, CO₂, CH₄, NH₃, and other solvents, if available. Calculations shall be made and reported on all gases present greater than .01 percent by volume. Data reduction shall be performed in a manner which will preclude the cracking pattern interference from other gas specie in the calculations of moisture content. Data shall be corrected for any system dependent matrix effects such as the presence of hydrogen in the internal ambient.

3.1.1 Failure criteria.

- a. A device which has a water-vapor content greater than the specified maximum value shall constitute a failure.
- b. A device which exhibits an abnormally low total gas content, as defined in 3.1a, shall constitute a failure, if it is not replaced. Such a device may be replaced by another device from the same population; if the replacement device exhibits normal total gas content for its type, neither it nor the original device shall constitute a failure for this cause.

3.2 Procedure 2. The device shall be hermetic in accordance with test method 1014, and free from any surface contaminants which may interfere with accurate water-vapor content measurement.

After device insertion into the piercing chamber, gas shall be flowed through the system until a stable base-line value of the detector output is attained. With the gas flow continuing, the device package shall then be pierced so that a portion of the purge gas flows through the package under test and the evolved moisture integrated until the base-line detector reading is again reached. An alternative allows the package gas to be transferred to a holding chamber which contains a moisture sensor and a pressure indicator. System is calibrated by injecting a known quantity of moisture or opening a package of known moisture content.

3.2.1 Failure criteria.

- a. A device which has a water-vapor content (by volume) greater than the specified maximum value shall constitute a failure.
- b. After removal from the piercing chamber, the device shall be inspected to ascertain that the package has been fully opened. A device package which was not pierced shall constitute a failure, if the test is not performed on another device from the same population; if this retest sample or replacement is demonstrated to be pierced and meets the specified water-vapor content criteria, the specimen shall be considered to have passed the test.
- c. A package which is a leaker in the purge case will be wet and counted as a failure. In the case of evacuation, a normal pressure rise shall be measured as in 3.1a.

3.3 Procedure 3. The moisture sensor shall be calibrated in an atmosphere of known water-vapor content, such as that established by a saturated solution of an appropriate salt or dilution flow stream. It shall be demonstrated that the sensor calibration can be verified after package seal or that post seal calibration of the sensor by lid removal is an acceptable procedure.

The moisture sensor shall be sealed in the device package or, when specified, in a dummy package of the same type. This sealing shall be done under the same processes, with the same die attach materials and in the same facilities during the same time period as the device population being tested.

The water-vapor content measurement shall be made, at 100°C or below, by measuring the moisture sensor response. Correlation with procedure 1 shall be accomplished before suitability of the sensor for procedure 3 is granted. It shall be shown the package ambient and sensor surface are free from any contaminating materials such as organic solvents which might result in a lower than usual moisture reading.

3.3.1 Failure criteria. A specimen which has a water-vapor content greater than the specified maximum value shall constitute a failure.

4. IMPLEMENTATION. Suitability for performing method 1018 analysis is granted by the qualifying activity for specific limits and volumes. Method 1018 calibration procedures and the suitability survey are designed to guarantee ±20 percent lab-to-lab correlation in making a determination whether the sample passes or fails the specified limit. Water vapor contents reported either above or below the (water vapor content - volume) range of suitability are not certified as correlatable values. This out of specification data has meaning only in a relative sense and only when one laboratory's results are being compared. The specification limit of 5,000 ppmv shall apply to all package volumes, with the following correction factors permitted to be used, provided they are documented and shown to be applicable:

For package volumes less than .01 cc internal free volume which are sealed while heated in a furnace:

$$C_T = \frac{T_r + 273}{T_s + 273}, \text{ where } C_T = \text{correction factor (temperature), } T_r = \text{room temperature (}^\circ\text{C), } T_s = \text{sealing temperature (}^\circ\text{C).}$$

For package volumes of any size sealed under vacuum conditions:

$$C_P = \frac{P_s}{P_a}, \text{ } C_P = \text{correction factor (pressure), } P_s = \text{sealing pressure, } P_a = \text{atmospheric pressure (pressures may be in either}$$

Torr or mm Hg).

The correction factor, if used, shall be applied as follows:

Water Vapor (Corrected) = Water Vapor (Measured) x C_x , where C_x is the applicable correction factor.

The range of suitability for each laboratory will be extended by the qualifying activity when the analytical laboratories demonstrate an expanded capability. Information on current analytical laboratory suitability status can be obtained by contacting DSCC-VQ.

5. SUMMARY. The following details shall be specified in the applicable acquisition document:
 - a. The procedure (1, 2, or 3) when a specific procedure is to be used (see 3).
 - b. The maximum allowable water-vapor content falling within the range of suitability as specified in test method 5005, 5008, or 5010, or general specifications MIL-PRF-38534 or MIL-PRF-38535.