ENGINEERING PRACTICE STUDY

PROJECT NUMBER: 5961-2008-047

TITLE: REVIEW OF REQUIREMENTS FOR MIL-STD-750 TEST METHOD 1018

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FINAL REPORT

Prepared by:

Kyle Carpenter

ATTCH 1

I. OBJECTIVES: To solicit input from custodians, review activities, users (G-12), and manufacturers (JC-13.1) on this proposal for coordination and concurrence.

II. BACKGROUND: Recent studies presented by JC-13.1 members have suggested that additional requirements be added as failure criteria to MIL-STD-750, Test Method 1018. The following additional requirements are being proposed:

a) A definitions section has been added to improve clarity to the method.
b) The apparatus detection and calibration requirements have been expanded to provide a more accurate and reliable test result.
c) A requirement to retain and archive all raw data has been included.
d) A procedure for vacuum baked devices has been added.
e) The failure criteria section has been expanded to include additional failure criteria.
f) The test sampling procedure has been revised to prohibit c=1 sample criteria.

III. RESULTS: Final results of this effort along with recommendations and conclusions are documented in attachment 1.

IV. CONCLUSIONS: The noted changes (attachment 1) shall be incorporated in the next revision of MIL-STD-750.

V. RECOMMENDATIONS:

a) All essential comments must be accepted or resolved through the DLA Land and Maritime spokesmen to the committees generating the drafts of those industry standards.
b) All suggested comments should be addressed by the industry committees or authors responsible for the draft documents.

METHOD 1018.4

INTERNAL GAS ANALYSIS (IGA)

1. Purpose. . The purpose of this test is to quantitatively measure the gas atmosphere inside a metal or ceramic hermetically-sealed device. Gases inside hermetically sealed devices can impact their long term reliability, Of particular interest is the measurement of the primary sealing gases, the moisture content, the presence of bombing gases that are indicative of non-hermeticity (e.g. helium,O_2 to Ar ratio indicative of room air~ 20 to 1, dissimilar concentration of internally sealed gases (e.g. nitrogen, helium) than originally sealed in the device package, the presence of leak test fluid (i.e. fluorocarbon), and all other gases to determine if the device meets the specified moisture, and other criteria. Also of interest is the measurement of all the other gases since they reflect upon the quality of the sealing process and provide information about the long term chemical stability of the atmosphere inside the device. This test is destructive. The presence of leak test fluorocarbon vapor in the IGA is a destructive indication of failure to meet gross leak test requirements of method 1071.

1.1. Definitions.

a. Signal to Noise Ratio (S/N): A dimensionless measure of the relative strength of an analytical signal (S) to the average strength of the background instrumental noise (N) for a particular sample and is closely related to the detection level. The ratio is useful for determining the effect of the noise on the relative error of a measurement.

b. Accuracy: A combination of the bias and precision of an analytical procedure, which reflects the closeness of a measured value to the true value. For the purposes of laboratory certification, accuracy means the closeness of a measured value to its reference standard.

c. Matrix: The general gaseous makeup of a particular sample.

d. Air burst: A sample of room air used to determine the sensitivity (calibration factor) for Oxygen, Nitrogen & Argon. An air burst reading shall be taken daily (every day testing is performed). Air burst readings shall be performed on 1cc volume size. The results of all air burst measurements shall be recorded, retained & made available to the Qualifying Activity upon request.

2. Apparatus. The apparatus for the internal water-vapor content test shall be as follows:

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~~ 140 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 parts per million volume (ppmv), ~~.01 cc~~, the mass spectrometer shall demonstrate a 50 ~~250~~ ppmv minimum detection limit to moisture, nitrogen, oxygen, argon, carbon dioxide, hydrogen, helium & fluorocarbons (69 & 119) for a package volume of .01 cc). The mass spectrometer shall also be capable of measuring with confidence (signal to noise ratio greater than 20:1) all other gases that are specified in the failure criteria of the part using the smallest volume as the worst case. The mass spectrometer shall also be capable of detecting 50 ppmv of leak test fluorocarbons (LTF) while detecting mass 69 and mass 119. The smallest volume shall be considered the worst case.

(3) System calibration. Air bursts shall be performed daily (when testing is to be performed). Air burst readings shall be performed on 1cc volume size. The results of all air burst measurements shall be recorded, retained & made available to the Qualifying Activity upon request. The calibration of the mass spectrometer shall be accomplished quarterly with a moisture level in the 4,500 to 5,500 ppmv range and with a moisture level in the 2,000 to 3,000 ppmv range, and with a moisture level in the 7,000 to 8,000 range using the same sensitivity factor. This calibration needs to be performed for each calibrator volume to demonstrate a linear response and to detect offset. A minimum of three data points for each moisture level shall be collected. Package simulators which have 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 ±5 percent shall be used. Moisture content shall be established by the standard generation techniques (i.e., 2 pressure, divided flow, or cryogenic method). The dew point hygrometer shall be recalibrated a minimum of once per year using equipment traceable to National Institute of Standards and Technology (NIST) or by a suitable commercial calibration services laboratory using equipment traceable to NIST standards. The dew point hygrometer shall be capable of measuring the dew point temperature to an accuracy of $\pm 0.2^{o}$C. The system shall have a pressure sensor to measure the pressure in line with the temperature dew point sensor to an accuracy of ± 0.1 inches of Hg for the range of pressure being used. In addition, the test laboratory shall have a procedure to calculate the concentration of moisture, in units of ppmv, from the dew point temperature measurement and the pressure measurement. 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 to .1 cc or 1,000 to 5,000 ppmv between .01 to .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 deviating 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 dewpoint 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.

$$\%H2O = \frac{100(Pv\text{mb})}{68.95\text{mb/psi}Pg + 1.33\text{mb/mm}Pa}$$

Where:
P_v = vapor pressure of water in the GPH based on water temperature in degrees Celsius. ~~centigrade~~
P_g = gauge pressure in psi.~~, and~~
P_a = atmospheric pressure in mm Hg.

50 ppm for calibration shall be used so that an accurate measurement of the 119 to 69 ratio can be obtained. A LTF compound for calibration shall be used.

(4) Annual 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 3b. The applicable gases shall be calibrated at approximately 1 percent concentrations requirements, with the exception of fluorocarbons, which may use a concentration of approximately 200 ppmv of a leak test fluorocarbon compound by identifying masses 69 and 119; NH_3 which may use a concentration of approximately 200 ppmv; hydrogen, which may use a concentration of approximately 200 ppmv; nitrogen, which may use a concentration of approximately 80 percent; helium, which may use a concentration of approximately 10 percent; and oxygen, which may use a concentration of approximately 20 percent. A minimum detection limit evaluation shall be performed for other gases.

(5) Daily calibration check. The daily calibration check gas standard shall be a similar gas matrix as the parts that will be tested that day. The daily calibration gas shall be a certified commercial gas mixture of the matrix of interest (usually nitrogen or nitrogen/helium mix) that contains nominal 300 ppmv or less of all other gases listed in 3b. The ~~system~~ calibration for moisture shall be checked on the day of test prior to any testing. This shall include checking the calibration by in-letting a sample with a moisture level in the 4,500-5,500 ppmv range at the required volumes and comparing the result with the dew point hygrometer. The resulting moisture reading shall be within 250 ppmv of the moisture level in the calibration sample. NOTE: Equipment error needs to be determined and subtracted from the allowed maximum deviation of 250 ppmv. The calibration check shall be performed using the same conditions used for testing devices (e.g. background pressure, background environment, time between sample inlets, package simulator volume, etc) System calibration for moisture ~~Calibration~~ performed on the day of test prior to any testing may be substituted for this daily calibration check. Calibration records shall be kept on a daily basis.

~~(6) Performed on the day of test prior to any testing may be substituted for this calibration check.~~

(7) Precision tuning shall be performed following significant maintenance or repair of the ion source.

(8) A record of all changes made to the sensitivity factors shall be maintained in addition to a reason for the change.

(9) All raw data, including test parameters used for the spectrometer, background spectra, test spectra, pressure readings, and calibration factors, shall be recorded for each test sample. The IGA lab shall retain the ability to review and recalculate the data in accordance with the archiving retention period requirements .

b. A vacuum opening chamber which can contain the device and a vacuum transfer passage connecting the device to the mass spectrometer of 2.a. A vacuum transfer passage shall efficiently (without significant loss of moisture from adsorption) transfer the gas from the device to the mass spectrometer ion source for measurement.

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 that has been maintained at 100°C ±5°C for the minimum 10 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.b, 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 shall be used to pierce both metal and ceramic packages. For ceramic packages, or devices with thick metal lids, the package lid or cover should be locally thinned by abrasion to facilitate localized piercing.

d. A pressure sensing device ~~located in the transfer passage~~ to measure the pressure rise in the transfer passage during the test. This pressure sensor is used to read a relative pressure change when the device is punctured. This relative pressure change indicates the relative quantity of gas in the device when comparing the test results of one device to another device. The significance of the reading is not intended to be absolute. Although the pressure gauge reading is reported, the pressure gauge is for indication only.

3. Procedure. All devices shall be prebaked for 16 to 24 hours at 100°C ±5°C prior to test. The purpose of prebake is to ensure all internal moisture has reached the vapor phase & is detectable. 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°C ±5°C. Devices whose temperature drops below 100°C ±5°C for more than 1 hour shall undergo another prebake to begin a minimum of 12 hours later. If vacuum bake is used to prebake devices, extending the prebake time shall not be permitted as this can evacuate a significant quantity of the device's internal gas content (skewing the test results). If a power interruption occurs during vacuum bake, the test sequence must stop. For the purposes of Conformance Inspection, additional samples must be submitted.

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 prebake continued until device reaches 100°C ±5°C.

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.c, and maintain the device at 100°C ±5°C for a minimum of 10 minutes prior to piercing.

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. The background vacuum spectra shall be acquired and shall later be subtracted from the sample spectra. After pump down, 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 water-vapor content of the released gases, as a percent by unit volume or ppmv of the total gas content.

b. The proportions (by volume) of ~~the~~ other ~~following~~ gases: N_2, He, Mass 69, and Mass 119 (~~fluorocarbons~~ leak test fluids), O_2, Ar, H_2, CO_2, CH_4, NH_3, and other primary sealing gases or solvents found. ~~, if available~~. Calculations shall be made and reported on all gases ~~present~~ detected and identified with reasonable certainty. 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.

c. The increase in chamber pressure as the gases are released by piercing the device package. A pressure change of ±25 percent from expected 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.

d. The test laboratory should provide comments describing the spectra of unknowns or gases that are present but not in sufficient concentration to be identified or quantified with reasonable certainty.

e. If the test laboratory has reason to believe that the test results may be invalid due to reasons such as improper puncture of the device or equipment malfunction, the results shall be reported as "no test" with additional comments provided. The device may be replaced with another.

f. In the event that the gases in the device have a significantly different gas matrix than the gas matrix used in the daily calibration check, the test laboratory shall conduct a calibration for moisture on the same day using a gas mixture similar to the gases found in the test device and shall recalculate the test results of the test device using the moisture sensitivity factor of that matrix.

NOTE: The device shall be hermetic in accordance with test method 1071 (fine & gross leak conditions) of this standard, and free from any surface contaminants which may interfere with accurate water vapor content measurement. The ~~internal water vapor content~~ IGA laboratory is ~~not~~ required to test for hermeticity (read and record the exact measured air leak rate) in accordance with test method 1071 of this standard. If the parts have ever been exposed to fluorocarbon leak testing, then only the applicable procedure in 1071 may be used to perform a measured leak rate. The IGA lab may read and record the measured air leak rate (if certified),or the IGA lab may contract to any third party certified seal test lab to read and record the measured air leak rate. ~~It is recommended that~~ Samples ~~samples~~ submitted for testing shall include the failure criteria information including ~~about~~ any ~~the~~ prior exposure to fluorocarbon leak testing and information about the manufacturing process, including sealing pressure, sealing gas, free internal cavity volume, lid thickness at puncture site, lid material, and the location of the puncture site.

3.1 Failure criteria.

a. The ~~Internal gas analysis~~ IGA laboratory shall ~~not~~ classify devices as passed or failed in accordance with the specified failure criteria as applicable. In addition, any device exhibiting gas content or pressure reading indicative of a leak (see Paragraph 1) shall be identified as a leaker and shall fail this test even when the water vapor failure criteria (5,000 ppm for an individual device), or applicable oxygen limit has not yet been achieved.

b. ~~A device being tested in a batch system which exhibits an abnormally low total gas content, as defined in 3.c, shall constitute a hermeticity failure not an IGA failure. 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.~~

b. Sample size for IGA testing shall never allow any c = 1 criteria. Any failure requires investigative and corrective action of the entire production lot to eliminate the root cause of the IGA 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 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 (unless otherwise specified), with the following correction factors permitted, to be used by the manufacturer provided they are documented and shown to be applicable:

a. For packages sealed at partial vacuum, the acceptance criteria could be established per the following chart:~~package volumes less than .01 cc internal free volume which are sealed while heated in a furnace:~~

$$\cancel{C_T = \frac{T_r + 273}{T_s + 273}}$$

~~Where:~~
~~C_T = correction factor (temperature)~~
~~T_r = room temperature (°C)~~
~~T_s = sealing temperature (°C).~~

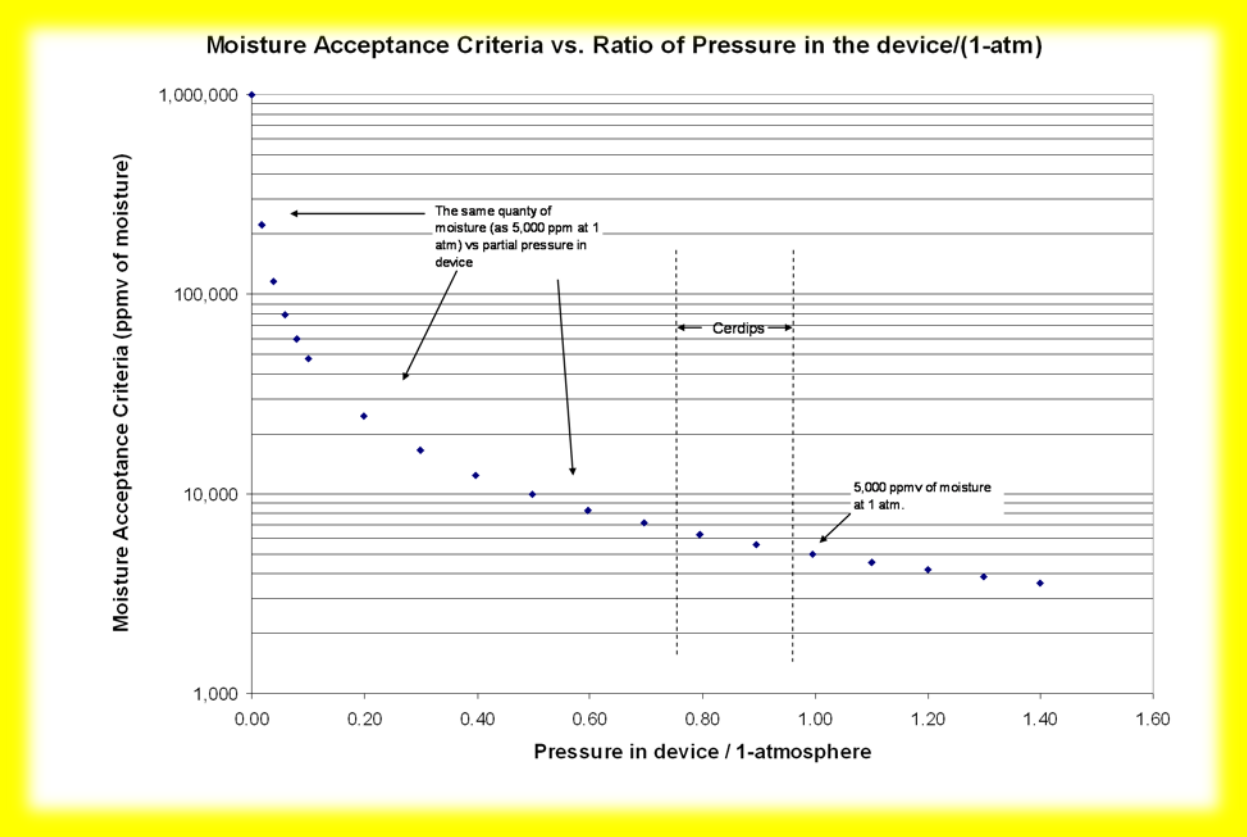
~~b. For package volumes of any size sealed under vacuum conditions:~~

$$\cancel{C_P = \frac{P_s}{P_a}}$$

~~C_P = correction factor (pressure)~~
~~P_s = sealing pressure~~
~~P_a = atmospheric pressure (pressures may be in 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.~~



b. If we assumed that a device having 1 atmosphere of gas pressure and a moisture concentration of <=5,000 ppmv was an good acceptance criteria, then a device that was sealed under a partial vacuum "but the moisture content (in moles) remained the same", then the equivalent acceptance criteria would be per the above graph. This graph could be used by DLA LAND AND MARITIME and the manufacturer to establish an acceptance criteria for a specific product line [provided, however, that the product continued to be manufactured at the same, or lower pressure as agreed to and there was no outgassing in the cavity of the device. The graph applies to product sealed under positive pressure also. It would be difficult to determine the pressure ratio on a device to device basis because you would have to compare the data to a similar device known to have 1 atm, but it could be established for a product line with some error limits established.

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 ~~Defense Supply Center~~ DLA LAND AND MARITIME-VQE, P.O. Box 3990, Columbus, OH 43218-3990 or e-mail vqe.chief@dla.mil.

5. Surrogate monitors. Surrogate monitors are only applicable for packages less than .01 cc to evaluate the process baseline. Surrogate monitors will be subject to IGA testing in accordance with method 1018 herein. A production lot will be validated by the performance of its monitors. It is well known and established that pre-seal bake and storage conditions of packaging materials will severely impact the levels of moisture detected in almost any package type. The use of the surrogate monitors without a controlled and disciplined manufacturing line is of questionable value. The proposed test is not, nor is it intended to be, a direct measurement of small packaged product internal moisture. However, it is a quantifiable indicator that the process and controls used are consistent. This is an improvement over the existing situation in which there is a requirement for control of internal moisture and no accurate and repeatable method of measurement.

5.1. Requirements. Surrogate monitors are to be procured from the same manufacturer and be manufactured in the same technology as the production headers, using the same materials, plating, processing, and technology. For example, the UB packages: Kyocera header, multilayer cofired ceramic technology; SemiAlloys lid, Alloy 52, nickel underplate, and gold plate.

a. The device manufacturer shall use the same preconditioning on surrogate monitors and production product, i.e. vacuum bake time and temperature, storage conditions, die attach materials and process.

b. Surrogate monitors shall be sealed at the same time and using the same process as the production parts.

c. To optimize the effect of preconditioning, the transit time from the oven to the seal furnace shall be controlled and minimal.

d. A typical process would include:

(1) Batch high-vacuum bake headers and lids.

(2) Store baked material in dry nitrogen.

(3) Second vacuum bake overnight (a minimum of 12 hrs) just prior to seal.

(4) Minimize the post second bake exposure to atmosphere.

e. Surrogate monitor packages will be under baseline documentation control. Full traceability from procurement to utilization shall be maintained.

f. Initially, the surrogate monitors will be used at the beginning of the seal operation and at 2 hour intervals. A minimum of six monitors must be processed for each seal lot (a "seal lot" may consist of multiple production lots if they go through sealing without interruptions (other than the scheduled breaks) and have identical traceability of headers and lids).

g. It is expected that it will take approximately 6 months for a manufacturer to collect enough lots and data to establish a baseline. Later modifications of the preconditioning process will be evaluated against this baseline.

h. The device manufacturer will submit to DLA LAND AND MARITIME the results from a minimum of three seal lots to establish the effectiveness of the process baseline. Additional testing will be retained and available to DLA LAND AND MARITIME upon request.

6. Summary. The following details shall be specified in the applicable acquisition document: The maximum allowable water vapor content if other than 5,000 ppmv; All other gas contents that represent a specific part failure.