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Standard Practice for Spacecraft Hardware Thermal Vacuum Bakeout[1]

This standard is issued under the fixed designation E2900; 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.

1. Scope

1.1 This practice establishes methods for thermal vacuum bakeout of spacecraft and spacecraft components.

1.2 This practice defines the equipment, environment, and certification criteria for each type of bakeout.

1.3 The methods defined in this practice are intended to reduce component outgassing rates to levels necessary to meet mission performance requirements of the contamination sensitive hardware. Times, temperatures, and configurations contained in this document have been found to provide satisfactory results. Experienced operators may find that other, similar times, temperatures and configurations have provided satisfactory results. If deviations from these criteria are deemed appropriate, they should be detailed in the bakeout report.

1.4 This practice describes three bakeout methods: Method A, using prescribed time and pressure criteria; Method B, using prescribed QCM stabilization rate criteria; and Method C, which measures the QCM deposition rate.

1.5 Determination of the acceptable molecular outgassing, selection of the bakeout method, and determination of the specific test completion criteria are the responsibility of the user organization.

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.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

E1546 Guide for Development of Fire-Hazard-Assessment Standards

E296 Practice for Ionization Gage Application to Space Simulators

E834 Practice for Determining Vacuum Chamber Gaseous Environment Using a Cold Finger

E1234 Practice for Handling, Transporting, and Installing Nonvolatile Residue (NVR) Sample Plates Used in Environmentally Controlled Areas for Spacecraft

E1235 Test Method for Gravimetric Determination of Nonvolatile Residue (NVR) in Environmentally Controlled Areas for Spacecraft

E1549 Specification for ESD Controlled Garments Required in Cleanrooms and Controlled Environments for Spacecraft for Non-Hazardous and Hazardous Operations

E1559 Test Method for Contamination Outgassing Characteristics of Spacecraft Materials

E1560 Test Method for Gravimetric Determination of Nonvolatile Residue From Cleanroom Wipers

E1731 Test Method for Gravimetric Determination of Nonvolatile Residue from Cleanroom Gloves

E2311 Practice for QCM Measurement of Spacecraft Molecular Contamination in Space

2.2 *Other Standards:*

IEST-STD-CC1246 Product Cleanliness Levels and Contamination Control Program[3]

MIL-STD-1246 Product Cleanliness Levels and Contamination Control Program[4,5]

MIL-P-27401 Propellant Pressurizing Agent, Nitrogen[5]

ISO-14644 Cleanrooms and associated clean environments[3]

FED-STD-209 Federal Standard, Airborne Particulate Cleanliness Classes in Cleanrooms and Clean Zones[5,6]

3. Terminology

3.1 *Definitions:*

3.1.1 *ambient conditions, n*—room temperature and pressure.

3.1.2 *pre-bakeout, n*—to clean or condition, or both, a vacuum chamber prior to its use for flight hardware.

[1] This test method is under the jurisdiction of ASTM Committee E21 on Space Simulation and Applications of Space Technology and is the direct responsibility of Subcommittee E21.05 on Contamination.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from Institute of Environmental Sciences and Technology (IEST), Arlington Place One, 2340 S. Arlington Heights Rd., Suite 100, Arlington Heights, IL 60005-4516, http://www.iest.org.

[4] MIL-STD-1246 may be used in lieu of IEST-STD-CC1246 by mutual agreement of the parties in the contract.

[5] Available from DLA Document Services, Building 4/D, 700 Robbins Ave., Philadelphia, PA 19111-5094, https://assist.daps.dla.mil/quicksearch/

[6] FED-STD-209 may be used in lieu of ISO-14644 by mutual agreement of the parties in the contract.

3.1.3 *bakeout, n*—a process by which volatile molecular contaminants are removed from a spacecraft component or article by exposing it to vacuum and elevated temperature". When the intent is to describe an action, this should be two words: to "bake out".

3.1.4 *cold finger, n*—the device that is used in collecting the sample of the residual gases in an evacuated vacuum chamber.

3.1.5 *cold plate, n*—a vacuum stable metal plate filled with liquid nitrogen used to condense the volatile molecular (or outgassing) contamination generated by the space component undergoing the bakeout.

3.1.6 *cold shroud, n*—the metal lining of the vacuum chamber (usually black painted or black anodized) used as a heating device or when filled with liquid nitrogen, used to simulate deep space.

3.1.7 *cold wall test, n*—a test configuration used to simulate deep space, requiring analytical view factors for the calculation of outgassing rates.

3.1.8 *hot wall test, n*—a test configuration, in which the hardware is isothermal with the surrounding environment. It assumes homogenous mixing for calculating outgassing rates.

3.1.9 *outgassing, n*—the evolution of a gas from a material, usually in a vacuum.

3.1.10 *outgassing rate (g/s), n*—the net rate of mass loss from a material sample as a result of outgassing. Outgassing rate can be normalized per unit sample surface area and expressed as $g \cdot cm^{-2} \cdot s^{-1}$ or it can be normalized per unit initial sample mass and expressed as $g \cdot g^{-1} \cdot s^{-1}$.

3.1.11 *QCM deposition rate stabilization (Hz/hr/hr), n*—the acceleration of the QCM deposition rate.

3.1.12 *quartz crystal microbalance or QCM, n*—a device for measuring small quantities of mass using the properties of a quartz crystal oscillator.

3.1.13 *QCM deposition rate, n*—the QCM output (or beat) frequency change per unit time caused by the mass of a molecular species condensing on the QCM crystal. The QCM deposition rate units may be converted to $g/cm^2/s$ by multiplying by the mass loading constant (m) provided by the vendor for the crystal used (i.e., for a 10 Mhz crystal, m=4.42E-9g/cm^2Hz).

3.1.14 *QCM thermogravimetric analysis or QTGA, n*—a technique in which a QCM is heated at a constant rate to remove a collected deposit.

3.1.15 *temperature stabilization, n*—temperature stabilization has been reached when the unit temperature is within 2°C of the specified temperature and the rate of change is less than 3°C per hour as measured with the unit control thermocouple. This rate can be extrapolated over a 20 min sample time. For example, stabilization has been achieved if two temperature measurements taken 20 min apart are within 1°C of each other.

3.1.16 *total collection area, n*—the sum of the surface area in the vacuum chamber that is equal to or colder than the QCM crystal temperature.

3.1.17 *visibly clean highly sensitive, VCHS, n*—visual inspection conducted at a distance of 15–50 cm (6–18 in.) with white light of at least 1076 lumens/m^2 (100 fc) intensity. It may be accompanied by ultraviolet (UV) inspection as well.

3.2 *Acronyms:*

3.2.1 *GN2*—Gaseous Nitrogen

3.2.2 *LN2*—Liquid Nitrogen

3.2.3 *MLI*—Multi-Layer Insulation

3.2.4 *NVR*—Nonvolatile Residue

3.2.5 *RGA*—Residual Gas Analyzer

3.2.6 *QCM*—Quartz Crystal Microbalance

3.2.7 *QTGA*—QCM Thermogravimetric Analysis

3.2.8 *TQCM*—Temperature controlled Quartz Crystal Microbalance

3.2.9 *VCHS*—Visibly Clean Highly Sensitive

4. Summary of Practice

4.1 A vacuum chamber is configured in the same manner it would be configured for the hardware bakeout, except that the test article is omitted.

4.2 The empty chamber and its support equipment is cleaned and inspected to VCHS. Then, the chamber is evacuated and pre-baked at a temperature 10°C above the hardware bakeout temperature, using the same procedure used for the component hardware.

4.2.1 For Method A, the chamber is ready for installation of flight hardware after 24 h under vacuum and temperature and a visual inspection.

4.2.2 For Methods B and C, the chamber is ready when the measured QCM deposition rate, witness sample data and visual inspection results are acceptable.

4.3 The spacecraft component to be thermal vacuum baked is exposed to an elevated temperature and a vacuum of 5.0E-5 torr or less for a specified amount of time or until the desired outgassing rate is reached.

4.3.1 *Method A*—The bakeout is terminated at a specified time limit and stabilized chamber pressure.

4.3.2 *Method B*—The bakeout is terminated when the QCM deposition rate stabilizes to a specified level.

4.3.3 *Method C*—There is a bakeout phase and a certification phase. The hardware is exposed to the program specific qualification temperature (usually 10°C above maximum predicted on-orbit operating temperatures) or the maximum tolerable temperature of the component in accordance with Method B. In the certification phase, the temperature is lowered to the predicted maximum on-orbit operating temperatures and the rate is measured. This provides realistic information that can be used to obtain outgassing rates in on-orbit conditions and also provides information about the dependency of the component outgassing rates on temperature. The bakeout is terminated when the QCM deposition rate reaches a specified level.

4.3.4 At the end of the bakeout, witness plates are removed and NVR wipe samples are taken of the cold plate.

5. Apparatus

5.1 *Description*—The bakeout apparatus consists of three main subsystems: a vacuum chamber (including ground support equipment), a temperature control system, and a data acquisition system. Methods B and C require a QCM.

5.1.1 *Vacuum Chamber*—The principle components of the vacuum chamber are the pump, the chamber shrouds and the cryogenic cold plate if needed.

5.1.1.1 The pump should be capable of maintaining the required pressure for a mean free path greater than the largest dimension of the chamber. Diffusion pumps use oil to capture gases and will increase deposition on the QCM. A cold trap between the diffusion pump and vacuum chamber is recommended to reduce backstreaming. Clean, oil-free pumps such as cryogenic, sorption, and turbomolecular are preferred to avoid backstreaming.

5.1.1.2 *High Vacuum Gauge*—An ion gauge or other gauge capable of monitoring pressures below 1e-4 torr. See Practice E296 for guidance in using ionization gauges.

5.1.1.3 *Chamber Shrouds*—The chamber shall be equipped with an inner lining or shroud that provides temperature control which is maintained cold for "cold wall" testing and hot for "hot wall" testing. A bakeout box may be substituted for the shroud for hot wall testing.

(1) Cold wall testing requires the hardware to be heated while the chamber shroud is kept cold, typically at LN_2 temperatures.

(2) Hot wall testing requires an environment that is isothermal with the hardware, this is typically accomplished with a bakeout box or the chamber shrouds. The bakeout box is an enclosed structure which surrounds the hardware and provides uniform heating of components. There are only holes in the box to allow for a small planned vent and a view port for the QCM. The chamber shroud is normally heated with hot GN_2 and the heater plates operate using heater tapes or circulating hot fluid. Whichever heating system is chosen, it should be sufficient to heat the item uniformly. Thermocouples should be placed appropriately to insure uniform heating of the hardware.

5.1.1.4 *Cold Traps*—There are three different types of equipment that can be used to trap contaminants: a LN_2 filled cold wall of the shroud, an LN_2 filled cold plate/cold finger, or the cryopump/diffusion LN_2 trap. The cold trap is kept cold throughout the test and may be analyzed afterward for contaminant identification.

5.1.2 *Temperature Control System*—All temperatures of the bakeout hardware and the QCM are maintained by independently controlled heaters to a precision of ±2°C.

5.1.2.1 *Heating Equipment*—In general, six different types of equipment may be used to heat the component: a bakeout box, heat lamps, resistance bars, heater plates, heater tapes, or the chamber shroud. Methods A and B are independent of the method of creating the environment temperature, while Method C requires either a hot wall or cold wall configuration.

(1) Arrays of heat lamps or resistance bars are commonly used for solar panels.

(2) The chamber shroud or aluminum heater plates are commonly used for electrical components. The chamber shroud is normally heated with hot GN_2 and the heater plates operate using heater tapes or circulating hot fluid.

(3) Heater tapes can be used on the component directly, but heater tape adhesives can bias the results and possibly contaminate the hardware.

5.1.3 *Data Acquisition System*—Data acquisition, storage and manipulation can be accomplished by any method that is capable of monitoring QCM frequencies, QCM temperatures, QCM heater/cooler voltages, hardware temperatures, chamber pressure, and data collection times at specified intervals. The system should be able to store collected data for later retrieval and analysis. An automated, computer operated data collection system is recommended.

5.1.3.1 The QCM heater/cooler voltage is used as a diagnostic tool. If there is significant variation in the QCM frequency, it may be related to poor QCM heater/cooler control.

5.1.3.2 Data storage intervals should be short enough to collect inherent variability of the QCM collection device. It has been found that 1 to 5 min between records is satisfactory.

5.1.4 *QCM*—The placement of the QCM has a significant effect on the measurement of outgassing rates. If the QCM views hot chamber surfaces capable of re-emitting contamination, such as it would in a hot box, the readings may be artificially high. If it views a cold shroud, the readings will be too low.

5.1.4.1 The QCM used for this test shall have a sensitivity of at least 1.0E-08 $g \cdot cm^{-2} \cdot Hz^{-1}$. 10 MHz or 15 MHz crystals meet this requirement and are typically used for this application.

5.1.4.2 The QCM shall be thermally connected to a heat sink enabling the QCM to operate in its full temperature range. It may be necessary to cool the heat sink mounting bracket with fluid or gas to keep the temperature stable.

(1) The sink for a TQCM must be maintained at no more than 40°C above the crystal operating temperature (see TQCM manual for details). This ensures that the indium seals will not melt due to internal heat generated by the TQCM. It may be necessary to heat a TQCM if the surrounding area is too cold for its electronics. An alternative is to provide multi-layer insulation to thermally decouple the TQCM electronics from the cold environment.

(2) CQCMs are designed to withstand and perform at cryogenic temperatures as well as any temperature up to the maximum allowed by the manufacturer. This is often 80°C.

5.1.4.3 For a cold wall test, the QCM deposition rate must be monitored by the QCM positioned such that its field of view is completely filled by the item undergoing bakeout. Since the QCM has a field of view between 143° and 150°, this means placing the QCM within several centimetres of the hardware. If

the QCM cannot be placed appropriately, which may be the case due to support equipment or thermal requirements, the rate must be adjusted by modeling and analysis.[7] For this reason it is important to document the exact dimensional location of the QCM with respect to the hardware. Photographs of the test set-up are recommended as well. If several components or parts are baked-out at once, multiple QCMs may be used to get an accurate representation of the outgassing rates. For assembled electronics boxes, the QCM(s) should be placed as close to the box vent as possible, or near a connector if there is no vent.

5.2 *Ideal Chamber Internal Configuration*—Commonly, the outgassing rate data generated by the thermal vacuum bakeout is used as an input to spacecraft system level contamination analyses. For this reason, the ideal chamber configuration is one that closely simulates the component's on-orbit environment and its configuration installed onto the spacecraft.

5.2.1 *Electronic boxes* are generally situated inside of the spacecraft bus, therefore the most likely choice for a vacuum chamber set up would be the "hot box" or to have heated chamber shrouds with a cold plate at one end of the chamber simulating a vent to cold space.

5.2.2 *Electrical harnesses* are notoriously high outgassing items. An often used method is to place them on an aluminum grating plate, heat them with warm chamber shrouds, and collect the contamination with a large cold plate. This allows the harnesses to be heated uniformly and provides a large cold surface area to collect great amounts of contamination that will most likely evolve from the harnesses.

5.2.3 Because the thermal environment for *thermal blankets* can vary so drastically, it is hard to simulate. Therefore, the most efficient way to bakeout thermal blankets and provide uniform heating would be hang them on aluminum or stainless wires inside of a bakeout box or warm shrouds/cold plate configured chamber.

5.3 *Optional Equipment:*

5.3.1 *Cold Finger*—An additional cold plate/finger is often used at the end of the bakeout during the certification phase to determine the identity and relative amount of any contamination remaining after the bakeout phase. Cold finger operations are described in Practice E834.

5.3.2 *Residual Gas Analyzer (RGA)*—An RGA may be installed into the vacuum chamber for chemical identification of outgassing species.

6. Support Equipment/Materials

6.1 *Garments*—Cleanroom smocks, cleanroom snoods or shower caps, and gloves should be worn during cleaning operations and for handling the hardware after the completion of the bakeout. Cleanroom latex, polyethylene, or Nitrile-gloves shall be used.[8] For bakeout items sensitive to particulate contamination, full bunny suits may be required as designated by the contractor.

6.2 *Witness Samples and NVR Sample Wipes*—Witness plates such as those described in Practice E1234 may be used for collecting contamination during the pre-bakeout and the hardware bakeout. Pre-extracted low NVR sample wipes can also be used to verify chamber cleanliness by sampling chamber walls or the cold plate.

6.2.1 *High Purity, Low NVR Solvents* are required for cleaning the chamber and other hardware prior to insertion of the components to be baked-out. Suitable reagent grade solvents include isopropyl alcohol and ethanol (or other low NVR solvents which meet local safety standards). The suitability of these solvents for the specific application must be determined by the user.

6.2.2 *Wipers*—Suitable wipers for cleaning include low NVR cleanroom quality polyester or nylon.[9]

6.3 *HEPA Filtered Portable Clean Tent*—For items undergoing a bakeout that are sensitive to particulate contamination, a clean tent, certified to the required ISO-14644 [Fed-Std-209] cleanliness class, may be used installed over the opening to the chamber.

7. Procedure

NOTE 1—There are three bakeout processes that can be used:

Method A (Time and Temperature) is generally used for components that do not have a line-of-sight to contamination sensitive hardware.

Method B (Outgassing Stabilization) may be used for components that do not have a line-of sight to sensitive hardware, yet have TQCM monitoring requirements.

Method C (Outgassing Measurement) is commonly used for contamination sensitive hardware and components which have a direct line-of-sight to that hardware.

See Table 1 for typical descriptions of test parameters for each method.

7.1 *Chamber Preparation:*

7.1.1 Chamber preparation includes a chamber cleaning and a pre-bakeout/chamber cleanliness certification.

7.1.2 Don appropriate cleanroom smocks and gloves described in 6.1.

7.1.3 The empty chamber shall be cleaned with non-linting cleanroom wipers per 6.3 and a solvent compatible with the chamber materials of construction and the suspected contaminants to be removed (isopropyl alcohol is generally compatible with vacuum chamber paints, see 6.2).

NOTE 2—Cleaning with solvents in an enclosed space may be hazardous. A special breathing apparatus, such as a respirator with the appropriate solvent cartridge filter may be necessary. All procedures should be reviewed for conformance to local safety requirements.

7.1.4 Solvent wipe clean all support fixtures, cabling, heaters, a cold wall or plate, using a solvent compatible with materials of construction and install into the chamber. Support equipment may be cleaned and verified to an IEST-STD-CC1246 [MIL-STD-1246] level as necessary.

[7] QCM readings may not be accurate unless the QCM field view is completely filled by the bake-out object. Exact measurements of the chamber and its equipment may be made so that it the appropriate view factors and outgassing rates can be calculated using a contamination analysis tools such as TRASYS® and MOLFLUX®. If chamber modeling is used, the source and limits of the model must be given. Method C offers an alternative.

[8] Garments and gloves are available commercially but should be tested for suitability prior to use. Testing may be performed according to Guide E1546 and Test Method E1731 respectively, or according to contractor requirements.

[9] Wipers are available commercially but should be tested prior to use for suitability and NVR per ASTM E1560 or contractor requirements.

TABLE 1 Description of Typical Test Parameters for Bakeout Methods A, B and C

NOTE 1—Unit acceptance or qualification temperature (10°C above maximum on-orbit operating predictions).

NOTE 2—May be adjusted to meet hardware design or program requirements. Because bakeout time and efficiency are dependent on temperature, the chosen temperature should be the highest possible without damage to hardware. Blankets baked above 100°C may shrink several percent.

NOTE 3—A minimum bakeout time may be estimated by 960/T(°C). Specified nominal times can be used for planning purposes or may be adjusted to meet specific program requirements. A minimum bakeout time of 16 to 24 h is recommended.

NOTE 4—Collection at –110°C captures all non-water volatiles while collection at –20°C has been used frequently when no temperature is specified.

NOTE 5—QCM temperature may be adjusted to meet program requirements. Chosen QCM temperature is generally based on the on-orbit predictions for the coldest contamination sensitive surface.

Method(s) Affected	A, B, C	A	B, C	B, C
Bakeout Item	Minimum Bakeout Temp (°C)	Nominal Bakeout Time (hrs) (Note 3)	QCM Temp (°C) (Note 4 and Note 5)	Bakeout Rate Stabilization
Electronic components	Note 1	48	–20 or –110	<3%/hr change in rate
Solar Array	Note 1	48	–20 or –110	<3% change in rate
Thermal Blankets	95 ± 5 (Note 2)	72	–20 or –110	<3% change in rate
Electrical Harnesses	95 ± 5 (Note 2)	96	–20 or –110	<3% change in rate

7.1.5 The QCM shall be chemically cleaned according to instructions provided in Test Method E1559 and placed within the chamber per 5.1.4 as it will be installed for the hardware bakeout.

7.1.6 Visually inspect chamber with white light and ultraviolet light (optional)[10] illumination for complete removal of adhesive residues, grease, oil, particles, metal shavings or other foreign materials. Areas not adequately cleaned shall be re-cleaned.

7.1.7 Install witness plates described in 6.2 into the chamber such that they will have a view to the same equipment or chamber wall that the actual hardware will have during its bakeout.

7.2 *Chamber Pre-Bake:*

7.2.1 For Method A, the pre-bake may or may not be performed depending on the heritage of the chamber. The need for a pre-bakeout must be determined by the user.

NOTE 3—If the final chamber pressure for a proceeding measurement is significantly less than the required final chamber pressure for the bakeout under consideration, indicating a "clean chamber", then consideration can be given to waiving the chamber bakeout portion of the processing cycle. This decision should be recorded in the processing paperwork for the sample being processed and include specific information on the chamber pressure, temperature, and sample identity for the proceeding sample.

7.2.2 For Methods B and C, the pre-bake shall be performed so that the outgassing rate measurements are not biased by high background chamber rates.

NOTE 4—If performing Method A, QCM monitoring during a chamber pre-bakeout certification is not necessary. Therefore, disregard following statements including the QCM if using Method A.

7.2.3 Close chamber door and evacuate chamber to 5.0E-05 torr or better vacuum. Maintain QCM at 25°C during evacuation if present.

7.2.4 Once the desired vacuum level is reached, fill cold plate or other support hardware (i.e. chamber shroud), or both, with LN_2 according to configurations described in 5.2. For Method A, this step is not necessary if a bell jar type chamber without a cold plate is used.

7.2.5 Increase heating element (lamps, hot box, etc) temperature until it stabilizes to 10°C ± 2°C above temperature to be used for the subsequent hardware bakeout.

7.2.6 *For Method A*—Maintain this condition for at least 24 h.

7.2.6.1 Initiate return to ambient by slowly backfilling the chamber with clean dry GN_2 per MIL-P-27401C, Type I, Grade B (99.99 %) pure or better, until a pressure of 400 torr is reached.

7.2.6.2 Warm cold plates or cold shrouds (if filled with LN_2) to ambient and continue to backfill chamber to ambient pressure. Ensure that the hardware temperature remains at least 5°C above the cold surfaces during this process. The warm-up may take over 10 h for cryogenic surfaces and the process should be constantly monitored to ensure that the 5°C temperature differential is maintained. This step may be deleted for chambers not containing a cold plate or cold shroud. **Warning—**Stand clear of chamber when door is opened until the oxygen level has been verified safe with an oxygen analyzer.

7.2.7 *For Method B*—While in vacuum, increase temperature of QCM to at least 80°C for cleaning and maintain this temperature until QCM frequency becomes stable. This may be performed in parallel with 7.2.4 through 7.2.5. It is expected that the QCM heat sink will be set no more than 40°C above the test operating temperature (e.g., <20°C if the QCM crystal temperature is –20°C during the test). This will ensure that the QCM does not overheat during the bake-off.

7.2.7.1 Decrease temperature of QCM to –20°C or desired program selected temperature.

NOTE 5—Collection at –110°C captures all non-water volatiles while collection at –20°C has a large heritage baseline.

NOTE 6—If QCM saturates, bakeoff per 7.2.7 then return to the measurement temperature.

[10] The visual inspection should be conducted at a distance of 15 to 50 cm (6 to 18 in). White light illumination should consist of at least 1076 lumens/m^2 (100 fc) intensity. The ultraviolet light should have a 100 W mercury arcspot bulb that produces a beam with minimum intensity of 1500 μW/cm^2. The ultraviolet wavelength should be 3200 to 3800 A.

7.2.7.2 Maintain this condition until an acceptable background QCM deposition rate is reached on the QCM. Generally, for spacecraft applications, a background rate of 3 Hz/hr/hr or less on a 15 MHz QCM is acceptable. Systematic error of up to 10 % of the acceptance rate is considered acceptable.

7.2.7.3 Initiate return to ambient by returning the temperature of the QCM to 25°C and slowly backfill the chamber with clean dry GN_2 per Mil-P-27401C, Type I, Grade B (99.99%) pure or better, until a pressure of 400 torr is reached.

7.2.7.4 Warm cold plates or cold shrouds (if filled with LN_2) to ambient and continue to backfill chamber to ambient pressure. Ensure that the hardware temperature remains at least 5°C above the cold surfaces during this process. For Method A, this step may be deleted for chambers not containing a cold plate or shroud. **Warning—**Stand clear of chamber when door is opened until the oxygen level has been verified safe with an oxygen analyzer.

7.2.7.5 Remove witness plates, package per Test Method E1235, and return to laboratory for analysis. Perform NVR wipe or rinse sampling, as desired.

7.2.7.6 Examine witness plate/NVR wipe data and outgassing rate data to determine its acceptability for the component hardware bakeout.

NOTE 7—For spacecraft applications, 1.0 mg/0.1m^2 or level A per IEST-STD-CC1246 collected on passive witness plates situated in the component hardware bakeout location is generally acceptable.

NOTE 8—Witness plates /NVR wipe samples on the cold plate or shroud will collect greater amounts of contamination. However, this is not an indication of the amount of contamination to which the hardware will be exposed because the hardware will be warm during the bakeout. It is more an indication of the total amount of contamination emitted from the chamber and its equipment during the bakeout. Generally, the NVR residues collected from the cold plate or shroud are analyzed to determine the identity of the largest source(s) of contamination.

7.3 *Method A (Time and Temperature)*—Method A utilizes standard bake-out temperature for a minimum specified amount of time which starts after a stable chamber pressure has been achieved. Outgassing rates are not measured.

NOTE 9—The minimum bakeout period is defined as

$$t = \frac{960}{T}$$

where:

t = time in hours, and
T = temperature in degrees Celsius,

or the nominal period as shown in Table 1, or as defined by the contractor program requirements.

7.3.1 Don appropriate cleanroom smocks and gloves described in 6.1.

7.3.2 Install hardware for bakeout.

7.3.3 Clean the chamber with a HEPA-filtered vacuum cleaner as necessary to remove particulate contamination or other foreign materials.

7.3.4 If required, install witness plates described in 6.2 into the chamber such that they will have a view to hardware undergoing the bakeout. Witness plates may also be placed on the cold plate or chamber shroud.

7.3.5 Close chamber and evacuate chamber to 5.0E-05 torr or less vacuum.

7.3.5.1 If present, fill cold plate or other support hardware with LN_2 once desired vacuum level is reached. Do not fill additional cold plate or finger intended for use at the end of the bakeout.

7.3.6 Increase hardware temperature to the program required level. Use Table 1 as a guideline if program levels are not set.

7.3.7 Maintain this condition for the minimum time required by the program.

7.3.7.1 Use Table 1 as a guideline or set the minimum hours required to 960/T°C if no program time limit exists.

7.3.7.2 During the last five hours, verify that the chamber is less than 1.0E-5 torr and the pressure-time history has stabilized to within a pressure tolerance of ±0.1 of the current decade.

7.3.8 If additional cold plate or cold finger was installed to determine the contamination remaining at the end of the bakeout, fill it with LN_2 during the last 5 hours of the bakeout.

7.3.9 Initiate return to ambient conditions by slowly backfilling the chamber with clean dry GN_2 per Mil-P-27401C, Type I, Grade B (99.99 %) pure or better, until a pressure of 400 torr is reached.

7.3.10 Warm all internal cold surfaces to ambient dew point temperature. Ensure that the hardware temperature remains at least 5°C above the cold surfaces during this process. This step may be deleted for chambers not containing a cold plate or shroud.

7.3.11 Continue to backfill chamber to ambient pressure. **Warning—**Stand clear of chamber when door is opened until the oxygen level has been verified safe with an oxygen analyzer.

7.3.12 Remove witness plates, package per Test Method E1235, and return to laboratory for analysis. Perform NVR wipe sampling, as desired.

NOTE 10—Cold plates or shrouds that have been filled with LN_2 during the chamber pre-bake operation must be solvent wiped with a solvent compatible with contaminant identified during the pre-bake phase. The entire chamber shall be solvent wiped if contamination is found to be dripping off the cold plate.

7.4 *Method B (QCM Deposition Rate Stabilization)*—This method removes the bulk of the outgassing products without having to reach a certain rate. The QCM deposition rate of the article at a standard temperature is monitored with a QCM until specified rate stabilization is reached.

7.4.1 Don appropriate cleanroom smocks and gloves described in 6.1.

7.4.2 Install hardware for bakeout.

7.4.3 Clean the chamber with a HEPA-filtered vacuum cleaner as necessary to remove particulate contamination or other foreign materials.

7.4.4 If required, install witness plates described in 6.2 into the chamber such that they will have a view to hardware undergoing the bakeout. Witness plates may also be placed on the cold plate or chamber shroud.

7.4.5 Close chamber and evacuate chamber to 5.0E-05 torr or less vacuum.

7.4.5.1 If present, fill cold plate or other support hardware with LN_2 once desired vacuum level is reached. Do not fill additional cold plate or finger intended for use at the end of the bakeout.

7.4.6 Increase the temperature of the QCM to at least 80°C for cleaning and maintain this temperature until QCM frequency becomes stable. This may be performed in parallel with 7.4.5.

7.4.7 Increase hardware temperature to the program required level. Use Table 1 as a guideline if program levels are not set.

7.4.8 Decrease temperature of QCM to the desired program selected temperature.

NOTE 11—Collection at –110°C captures all non-water volatiles while collection at –20°C has a large heritage baseline.

NOTE 12—If QCM saturates, bake off the QCM by increasing the temperature of the QCM crystal to at least 80°C and maintaining this temperature until the slope of the curve of QCM frequency versus time becomes constant.

NOTE 13—Slight background fluctuation in the frequency of the QCM is normal.

7.4.8.1 Use Table 1 as a guideline if no program specified temperature exists.

7.4.8.2 Maintain this condition until the required program outgassing rate stabilization is met and held for at least 5 h.

7.4.9 If additional cold plate or cold finger was installed to determine the contamination remaining at the end of the bakeout, fill it with LN_2 during the last 5 h of the bakeout.

NOTE 14—This time is in addition to the time to reach the 5-h QCM rate stabilization.

7.4.10 Initiate return to ambient conditions by slowly backfilling the chamber with clean dry GN_2 per Mil-P-27401C, Type I, Grade B (99.99 %) pure or better, until a pressure of 400 torr is reached.

7.4.11 Warm all internal cold surfaces to above the ambient dew point temperature. Ensure that the hardware temperature remains at least 5°C above the cold surfaces during this process. This step may be deleted for chambers not containing a cold plate or shroud.

7.4.12 Continue to backfill chamber to ambient pressure. **Warning—**Stand clear of chamber when door is opened until the oxygen level has been verified safe with an oxygen analyzer.

7.4.13 Remove witness plates, package per Test Method E1235, and return to laboratory for analysis. Perform NVR wipe sampling, as desired.

NOTE 15—Cold plates or shrouds that have been filled with LN_2 during the chamber pre-bake operation must be solvent wiped with a solvent compatible with contaminant identified during the pre-bake phase. The entire chamber shall be solvent wiped if contamination is found to be dripping off the cold plate.

7.5 *Method C (Outgassing Measurement)*—For Method C, there is a bakeout phase and a certification phase. The bakeout is terminated based on reaching the required bakeout and certification rates. There is no time limit for this method.

7.5.1 Don appropriate cleanroom smocks and gloves described in 6.1.

7.5.2 Install hardware for bakeout.

7.5.2.1 For a cold wall test, verify QCM placement (distance and viewing) is consistent with analytical view factor model.

7.5.2.2 For a hot wall test, verify the cross sectional area of all vents and collecting surfaces.

7.5.3 Clean the chamber with a HEPA-filtered vacuum cleaner as necessary to remove particulate contamination or other foreign materials.

7.5.4 If required, install witness plates described in 6.2 into the chamber such that they will have a view to hardware undergoing the bakeout. Witness plates may also be placed on the cold plate or chamber shroud.

7.5.5 Close chamber and evacuate chamber to 5.0E-05 torr or less vacuum.

7.5.5.1 If present, fill cold plate or other support hardware with LN_2 once desired vacuum level is reached. Do not fill additional cold plate or finger intended for use at the end of the bakeout.

7.5.5.2 For cold wall testing, the pressure should be sufficiently low that the mean free path is greater than the largest chamber dimension.

7.5.6 Increase the temperature of the QCM to at least 80°C for cleaning and maintain this temperature until QCM frequency becomes stable. This may be performed in parallel with 7.5.5.

7.5.7 Increase hardware temperature to the program required level. Use Table 1 as a guideline if program levels are not set.

7.5.8 Decrease temperature of QCM to the desired program selected temperature.

NOTE 16—Collection at –110°C captures all non-water volatiles while collection at –20°C has a large heritage baseline.

NOTE 17—If QCM saturates, bake off the QCM by increasing the temperature of the QCM crystal to at least 80°C and maintaining this temperature until the slope of the curve of QCM frequency versus time becomes constant.

NOTE 18—Slight background fluctuation in the frequency of the QCM is normal.

7.5.8.1 Use Table 1 as a guideline if no program specified temperature exists.

7.5.8.2 Maintain this condition until the specified outgassing rate stabilization is met and held for at least 5 h.

7.5.9 Decrease the hardware temperature to the program required hardware certification level. If no program requirement exists, use Table 1 as a guideline to set one.

7.5.10 Increase the temperature of the QCM to at least 80°C for cleaning and maintain this temperature until the slope of the curve of QCM frequency versus time becomes constant. This may be performed in parallel with 7.5.9.

NOTE 19—Slight background fluctuation in the frequency of the QCM is normal.

7.5.11 Decrease the QCM temperature to the required program temperature.

7.5.11.1 This step will not be started until the hardware is within ±2°C of the hardware certification temperature.

7.5.11.2 If QCM saturates, bake off the QCM by increasing the temperature of the QCM crystal to at least 80°C and

maintaining this temperature until the slope of the curve of QCM frequency versus time becomes constant.

NOTE 20—Slight background fluctuation in the frequency of the QCM is normal.

7.5.12 Record outgassing rate at certification temperature for a minimum of 5 h or until the rate has stabilized.

7.5.12.1 If the specified program certification is not met, the bakeout may continue at the certification temperature or return to the bakeout temperature for at least 8 h.

7.5.12.2 An outgassing stabilization rate need not be achieved at this point since it was reached already. Repeat 7.5.6 and 7.5.7.

7.5.12.3 After a minimum of 8 h, repeat 7.5.8 through 7.5.12.

7.5.13 If an additional cold plate or cold finger was installed to determine the contamination remaining at the end of the bakeout, fill it with LN_2 during the last 5 h of the bakeout.

NOTE 21—This time is in addition to the time to reach the 5-h QCM rate stabilization.

7.5.14 Initiate return to ambient conditions by slowly backfilling the chamber with clean dry GN_2 per Mil-P-27401C, Type I, Grade B (99.99 %) pure or better, until a pressure of 400 torr is reached.

7.5.15 Warm all internal cold surfaces to above the ambient dew point temperature. Ensure that the hardware temperature remains at least 5°C above the cold surfaces during this process. This step may be deleted for chambers not containing a cold plate or shroud.

7.5.16 Continue to backfill chamber to ambient pressure. **Warning—**Stand clear of chamber when door is opened until the oxygen level has been verified safe with an oxygen analyzer.

7.5.17 Remove witness plates, package per Test Method E1235, and return to laboratory for analysis. Perform NVR wipe sampling, as desired.

NOTE 22—Cold plates or shrouds that have been filled with LN_2 during the chamber pre-bake operation must be solvent wiped with a solvent compatible with contaminant identified during the pre-bake phase. The entire chamber shall be solvent wiped if contamination is found to be dripping off the cold plate.

8. Data Processing

8.1 *QCM Measurements*—QCM frequencies must be evaluated over a period of time to be equated to a QCM deposition rate. It is frequently necessary to allow the TQCM to stabilize before using data for analysis. The QCM temperature must remain stable (±0.5°C) over the measurement period since the QCM frequency is impacted by temperature.

8.1.1 QCM deposition rate stabilization determination: Outgassing rates can appear to be unstable if they are near the sensitivity limit of the QCM. A linear or nonlinear regression can be performed to factor out the systematic error. Data reported over a 1- to 5-min time increment can be used effectively to evaluate outgassing in an hour or two. The user should at least wait until the QCM heater voltage stabilizes. An unstable heater voltage causes very small changes in adsorption and desorption that are manifested in unstable mass readings on the QCM.

8.1.1.1 A commonly used stabilization value is 3 %/h. Two consecutive QCM deposition rates are divided by the intervening time in hours. Multiply the result by 100 to provide a value in %/h.

8.1.2 *Outgassing Rate Determination*—The difference between consecutive readings is divided by the time between measurements to provide a QCM deposition rate in Hz/unit time. An alternative is to use a nonlinear regression program to determine the relationship between frequency and time. Differentiation will yield the QCM deposition rate. Caution is required when using this method, as some nonlinear equations are not monotonic. These equations can also increase in slope near the end of the monitoring period, which is an artifact of the regression equation, not the test. This is especially true of a polynomial series approximation.

8.2 *Chamber Model*—If QCM deposition rates in Hz/unit time are converted to test article outgassing rates in grams/unit time, it is necessary to model the vacuum chamber. An analytical outgassing model provides corresponding view factors for the hardware and the QCM, which are essential for evaluating cold wall test results.

8.2.1 *Determination of Source Outgassing Rate*—The QCM deposition rate is converted from Hz/unit time to a deposition rate in g/cm^2/unit time based on the crystal sensitivity. A cold wall test assumes only line of sight molecular flux, therefore the reciprocity of view factors may be implemented.

$$OG_s = R_{QCM} * S_{QCM} * (VF_{QCM->HW} / VF_{HW->QCM}) \quad (1)$$

where:

OG_s = source outgassing rate,
R_{QCM} = QCM deposition rate (Hz/unit time),
S_{QCM} = QCM sensitivity (e.g. 4.42e-9 g/cm^2/Hz for a 10 MHz crystal),
$VF_{HW->QCM}$ = analytical view factor of hardware to QCM, and
$VF_{QCM->HW}$ = analytical view factor of QCM to hardware.

NOTE 23—R_{QCM} is the deposition rate of the QCM with test hardware after subtracting the background rate without test hardware in the chamber.

NOTE 24—See Appendix X1 for simple methods to transform QCM deposition rates to outgassing rates.

9. Report

9.1 The report should include all of the background data used to determine the endpoint. All reports shall include documentation of the equipment and instrumentation used, the configuration of the vacuum chamber contents, and dimensional documentation of the locations of the QCM(s) with respect to the component baked.

9.2 *Method A (Time and Temperature):*

9.2.1 State the hardware baked in this process.

9.2.2 Define the temperature used in the bakeout, including tolerances.

9.2.3 State the time the hardware was held at the bakeout temperature and how the time specification was determined.

9.2.4 Provide a plot of hardware temperature and chamber pressure versus time.

9.2.5 If NVR witness plates are used, report the mass collected and the size of the plate. Also report chemical

analysis results of the NVR if available. If NVR analysis is not done, explain why not.

9.2.6 Report visual inspection results as the hardware is removed from the vacuum chamber. Describe all visible deposits and changes in appearance or physical condition. If MLI is baked, report shrinkage or warping if present.

9.3 *Method B (Outgassing Stabilization):*

9.3.1 State the hardware baked in this process.

9.3.2 Define the temperature used in the bakeout, including tolerances.

9.3.3 State the time the hardware was held at the bakeout temperature.

9.3.4 Provide a plot of the hardware and shroud temperature and chamber pressure versus time, include cold plate and cold finger temperatures if applicable.

9.3.5 Report the QCM temperature, including tolerances, and the data collection interval.

9.3.6 Report the QCM rate acceleration requirement and the time span that the hardware met or exceeded this rate.

9.3.7 Provide a plot of the QCM frequency versus time, include hardware and QCM temperatures.

9.3.8 If NVR witness plates are used, report the mass collected and the size of the plate. Also report chemical analysis results of the NVR if available. If NVR analysis is not done, explain why not.

9.3.9 Report visual inspection results as the hardware is removed from the vacuum chamber. Describe all visible deposits and changes in appearance or physical condition. If MLI is baked, report shrinkage or warping if present.

9.4 *Method C (Outgassing Measurement):*

9.4.1 State the hardware baked in this process.

9.4.2 Define the temperature used in the bakeout, including tolerances.

9.4.3 State the time the hardware was held at the bakeout temperature.

9.4.4 Report the QCM temperature, including tolerances.

9.4.5 Provide a plot of the hardware and environment temperature and chamber pressure versus time, include cold plate and cold finger temperatures if applicable.

9.4.6 Report the QCM rate acceleration requirement and the time span that the hardware met or exceeded this rate, if used.

9.4.7 State the hardware outgassing certification temperature, including tolerances.

9.4.8 State the outgassing acceptance rate for the test hardware.

9.4.9 State the QCM temperature during certification, including tolerances, and the data collection interval.

9.4.10 Report all factors used to convert the deposition rate (Hz/unit time) to an outgassing rate.

9.4.10.1 *Cold Wall Test*—Report QCM location, distance to hardware, and view factors, and include a summary of analytical view factors and the code used to produce them.

9.4.10.2 *Hot Wall Test*—Report the geometric factors used in the evaluation, including the effective area of the pump, the surface area of the scavenger plate, and any other vents or collection areas. Also report the conversion factor calculated from these areas.

9.4.11 Report the final QCM deposition rate.

9.4.12 Report the final hardware outgassing rate calculated from the QCM deposition rate. Also report the time that the hardware outgassing rate was less than the acceptance rate.

9.4.13 Provide a plot of the outgassing rate versus time, include hardware and QCM temperatures.

9.4.14 If NVR witness plates are used, report the mass collected and the size of the plate. Also report chemical analysis results of the NVR if available. If NVR analysis is not done, explain why not.

9.4.15 Report visual inspection results as the hardware is removed from the vacuum chamber. Describe all visible deposits and changes in appearance or physical condition. If MLI is baked, report shrinkage or warping if present.

10. Precision and Bias

10.1 Precision and bias depend upon the specific test methods used, instruments used, and the precision and bias of the particular test methods and procedures. Please refer to specific individual test methods for precision and bias of that test.

11. Keywords

11.1 contamination; contamination sensitivity; NVR; outgassing; outgassing rate; QCM; spacecraft testing; thermal vacuum testing; thermal vacuum bakeout; TQCM; vacuum bakeout

APPENDIX

(Nonmandatory Information)

X1. METHODS FOR TRANSFORMING QCM DEPOSITION RATES TO OUTGASSING RATES

INTRODUCTION

The following information provides simple methods for transforming QCM deposition rates to outgassing rates. It should be noted that view factors are extremely important and need to be considered before undertaking the transformation. QCM placement needs to be unbiased to make these methods work properly. Yet, the QCM needs to have a substantial view of the test article. The analyst that will use the calculated outgassing rates should be consulted to ensure that the data derived from these tests are acceptable. The three approaches below are commonly used first order approaches, but are not the only methods that can be used.

X1.1 Homogenous Mixing Model—This is best done with a hot wall test where the QCM has a diffuse view of the test article and the environment. A cold finger or cold plate is also required to calculate the effective pump area.

X1.1.1 *Determination of the Effective Pump Area*—It can be shown that:

$$\frac{OG_{NS}}{OG_S} = \frac{A_{QCM}+A_P+A_S}{A_{QCM}+A_P} \quad \text{(X1.1)}$$

where:

OG_{NS} = outgassing rate without scavenger plate,
OG_S = outgassing rate with scavenger plate,
A_{QCM} = exposed surface area of QCM crystal,
A_P = effective surface area of the high vacuum pump, and
A_S = surface area of the scavenger plate.

The QCM crystal area is inconsequential area compared to the areas of the scavenger plate and the effective area of the pump. The effective area of the pump is:

$$A_P - \frac{A_S}{\frac{OG_{NS}}{OG_S} - 1} \quad \text{(X1.2)}$$

Thus, the effective area of the pump can be determined by simply achieving a stable background rate in an empty chamber without the cold plate activated and then reaching a stable deposition rate after the cold plate is activated. A minimum deposition rate of 50 Hz/h is recommended to reduce the inherent noise level of this estimation.

X1.1.2 *Determination of Total Outgassing Rate*—A summation of areas is used to convert the QCM deposition rate to an outgassing rate. A QCM deposition rate is reported in Hz/unit time. Multiplying this by the mass sensitivity (grams/cm^2-Hz) will transform the deposition rate to grams/cm^2-unit time. Multiplying by the surface area of the collector or vent provides a mass flux in grams/unit time. The material collected on the QCM is a portion of the amount outgassed, and it is much smaller than the amount passing through the pump or condensed on a cold plate if present; the QCM surface area can be ignored in this calculation.

$$OG_{HW} = R_{QCM} * S_{QCM} * A_{collect} \quad \text{(X1.3)}$$

where:

OG_{HW} = outgassing rate of test article,
R_{QCM} = deposition rate of QCM (Hz/unit time)
S_{QCM} = QCM sensitivity (e.g. 4.42e-9 g/cm^2/Hz for a 10MHz crystal), and
$A_{collect}$ = summation of total collection area, includes effective pump area, QCM, coldplates, etc.

This calculates the outgassing rate of the hardware in grams/unit time.

X1.2 QCM Thermogravimetric Analysis—Estimation of deposition rates for QCM temperatures warmer than measured can be interpolated from a QTGA. This method is fully described in Test Method E1559.

X1.2.1 The frequency is normalized across the range of temperatures, from base collection temperature to 80°C, during QCM bakeoff. The normalized frequency at any given temperature provides a scalar for the interpolation of the outgassing rate at a higher collection temperature. For example, if an estimate of the deposition rate at –30°C was desired from a test collection of –110°C:

$$R_{-30} = R_{-110} * F_{norm-30} \quad \text{(X1.4)}$$

where:

R_{-30} = outgassing rate for –30°C collection,
R_{-110} = deposition rate of QCM –110°C, and
$F_{norm-30}$ = $(F_{-30}–F_{min})/(F_{max}–F_{min})$; frequency at –30°C normalized over QTGA range.

X1.2.2 Individual QCM frequency response to temperature varies and should be determined independently. Starting with sufficient mass collection (>1000 Hz) can minimize the effects of temperature dependency.