SimulTek Research

PROGRESS IN ENVIRONMENTAL SIMULATION TECHNOLOGIES

ASTM E1559 Outgassing System

from Simultek Research Co.

"Every Space Materials Engineer, Surface Physicist, Spacecraft Contamination Engineer and Spacecraft Contamination Effects Analyst looking at outgas testing essentially requires a Contamination Test Facility, designed to meet the new standard in it's entirety."

ASTM E1559 Outgassing System

Responding to the growing demand from International community, Simultek Research Co. designed an Outgassing System for testing the outgassing properties of materials in accordance with the ASTM E595/E1559 test methods.

Test ASTM E1559 uses a method that allows the total mass loss to be determined through the use of three Quartz Crystal Microbalances (QCMs) set to various temperatures. Two methods (A and B) may be used for determining the outgassing kinetics. In method A, specific QCM and specimen temperatures as well as geometry of the source/receiver are required, whereas in method B there is considerable flexibility in the temperatures that can be used and even in the test geometry. Test Method A requires that three of the QCMs be maintained at 90 K (-183°C), 160 K (-113°C), and 298 K (25°C). Although an effusion cell temperature of 125° C (398 K) typically is used for Method A, other temperatures can be used in Method B that may be more closely related to the actual operational material temperatures.

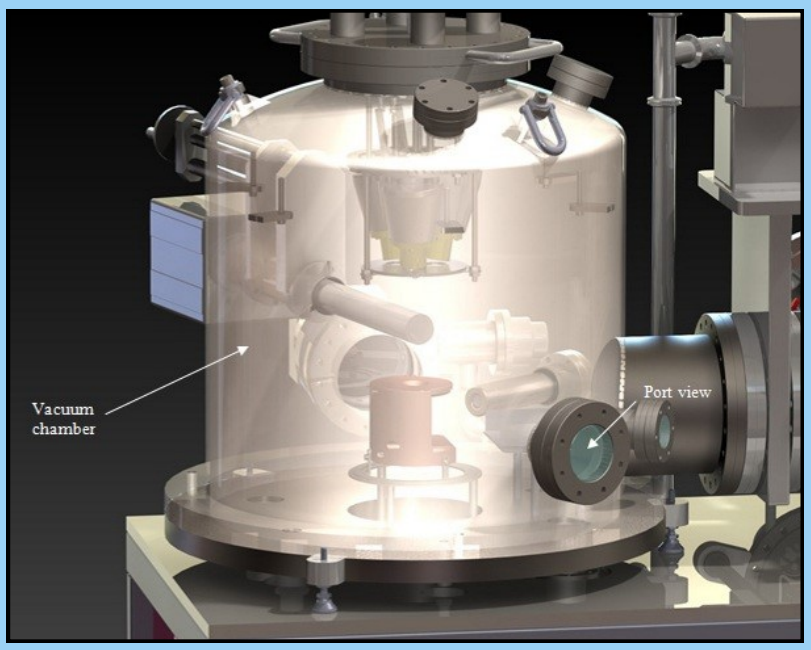
The ASTM E1559 requires that the QCMs consist of two crystals (one for mass collection and one for reference) and that they have a sensitivity of at least 1×10^{-8} g/cm^2/Hz at 298 K. This is typically accomplished with crystals that have natural frequencies of 10 MHz to 15 MHz. The E1559 test facility uses QCMs with 15 MHz crystals that have a sensitivity of 1.73×10^{-9} g/cm^2/Hz. The crystals are 1.27 cm in diameter and optically polished and plated with gold. The pair of crystals is designated as a sensor crystal and a reference crystal.

The effusion cell is a cylindrical container approximately 65±5 mm inside diameter by 50±5 mm deep, and is machined from aluminum for high thermal conductivity. The integrated heater ensures uniform temperature distribution throughout the inner cavity. The effusion cell has a detachable cover plate which allows for insertion of material samples. This lid has a cylindrical orifice of 3.0±0.1 mm in diameter by 3.0±0.1 mm long providing the appropriate flux distribution. The cell has an integral temperature sensor and heater, and control system which permits operating the cell from ambient to 398-425 K with a precision of ±0.5 K or better. The effusion cell is positioned inside the test chamber at 150±1 mm from the surface of the sensing crystal in each QCM so that the center of the cell orifice exit plane coincides with the intersection point of the QCM axes. The cell orifice thus has the same view factor of 415.08 cm^2 to the all QCMs.

The sample, on the order of 10 to 20 g, is placed inside the effusion cell and mounted in the interlock chamber. After the interlock chamber has been evacuated to a level of approximately10^{-9} Torr, the gate valve is opened and the effusion cell is moved manually into the test chamber using the transfer mechanism. Once the location has been established, the effusion cell temperature controller is set to the desired temperature, usually to 125°C (398 K), and the QCM frequencies recorded as a function of time with a 1 min time period. The time required for completion of an outgassing test varies depending on the needs of the user. In many instances outgassing times of up to 5 days are used in order to determine the long-term outgassing rates.

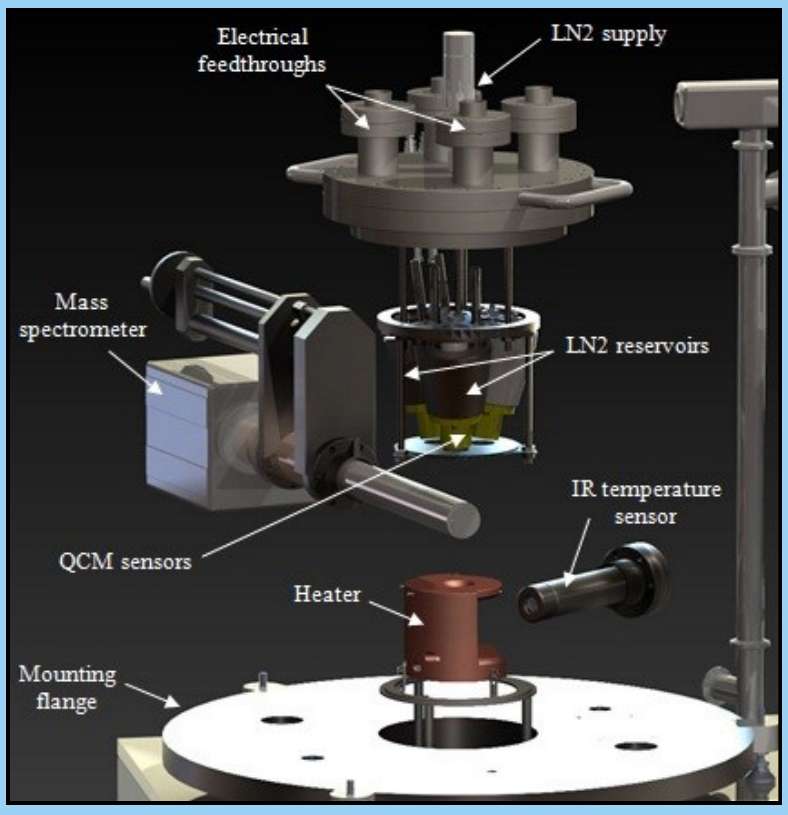
The Outgassing System features oil-free pumping with a scroll pump and turbo pumps and the test operation is made fully computer-controlled. This catalogue describes a general design outgassing facility that accommodates the set-up for the ASTM E595/E1559 test methods. The details on the technical design of the vacuum system, sample set-up, QCM system, and internal configuration of the outgassing facility are briefly presented. When ordering, the system design and the final specifications will be tuned to the needs of the customer.

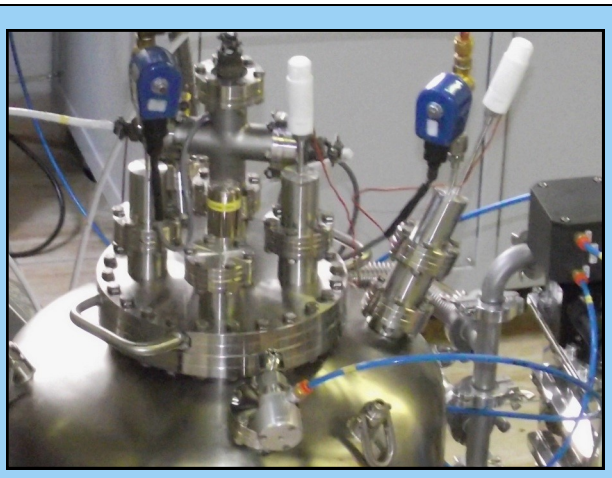
INTERNAL CONFIGURATION OF THE ASTM E1559 TEST CHAMBER

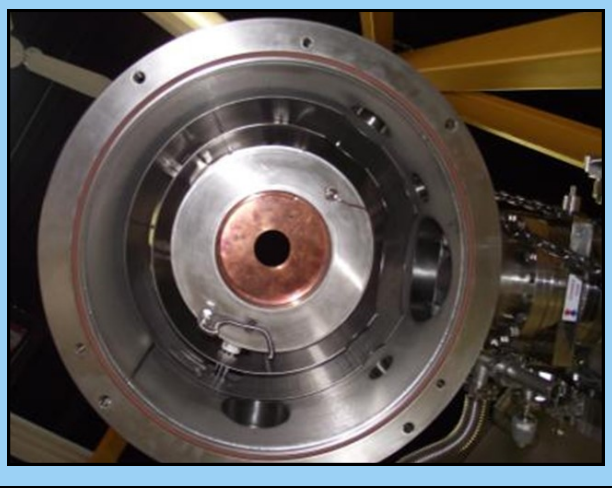


INSTRUMENTATION

- High-vacuum test chamber (Pressure: ≤ 5×10^{-9} Torr)
- Test chamber diameter: 467 mm
- Test chamber length: 563 mm
- Air-lock chamber (Pressure: ≤ 5×10^{-8} Torr)
- Air-lock chamber diameter: 150 mm
- Air-lock chamber length: 333 mm
- Tri-Scroll 300 fore pump, 250 L/min pumping speed
- Varian V301 test chamber turbo pump, 250 L/s pumping speed (N_2)
- Varian V81 air-lock chamber turbo pump, 77 L/s pumping speed (N_2)
- CrystalTek Corp. Cryo-QCM sensors
- OMEGA Non-Contact Infrared Temperature Sensor
- SRS RGA300 Mass Spectrometer (Mass range: 1-300 AMU)





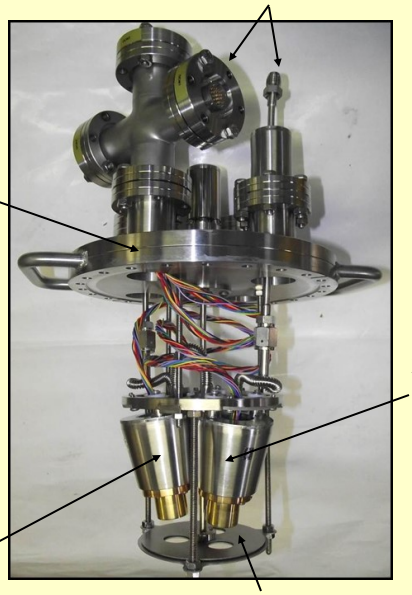


CRYSTAL TEK CORPORATION

Quartz Crystal Microbalances

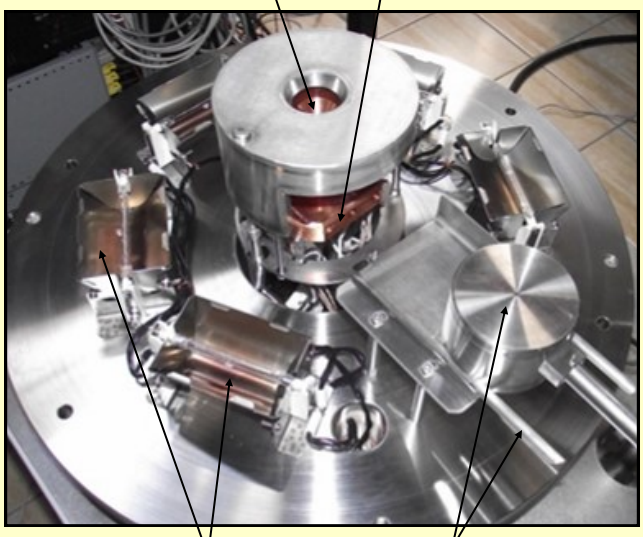
QCM SENSOR SPECIFICATIONS

- CrystalTek Model 36S/C Cryo-QCM sensor
- CrystalTek Model 26E/C Cryo-electronics
- CrystalTek Model 66T QCM controller
- Sensor dimensions: ∅3.05 cm × 3.18 cm length
- Sensor crystals: Two AT cut, gold plated, optically polished quartz crystals
- Natural frequency: 15 MHz
- Aperture diameter: 0.64 cm
- Temperature range: 77-400 K
- Sensor sensitivity: 1.73×10^{-9} g/cm^2 Hz
- Accuracy of mass measurement: 0.2%
- Dynamic range: Non-solid films: 10-20 kHz; Solid films: > 50 kHz



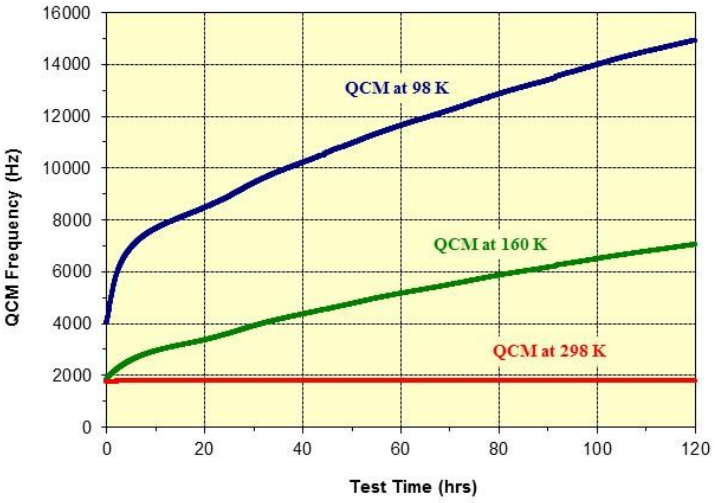
SOURCE EFFUSION CELL

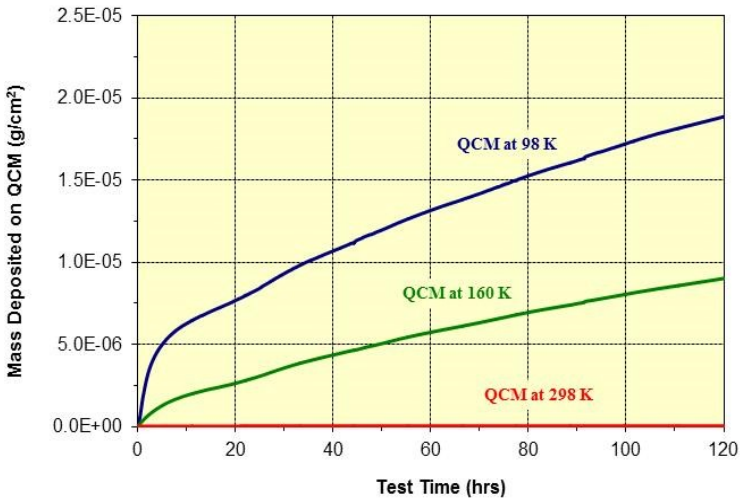
- Inside diameter: 65±5 mm
- Depth: 50±5 mm
- Volume 0.1659 mm^3
- Orifice diameter: 3.0±0.1 mm
- Cell temperature range: 398-425 K
- Capable of heating effusion cell to 425 K in 20 min
- Distance between the orifice and QCM sensors: (50-250)±1 mm
- Effusion cell transfer mechanism for insertion into the test chamber

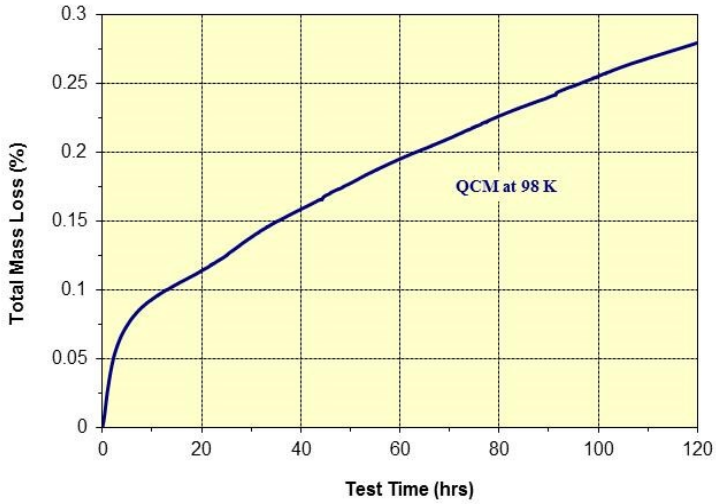


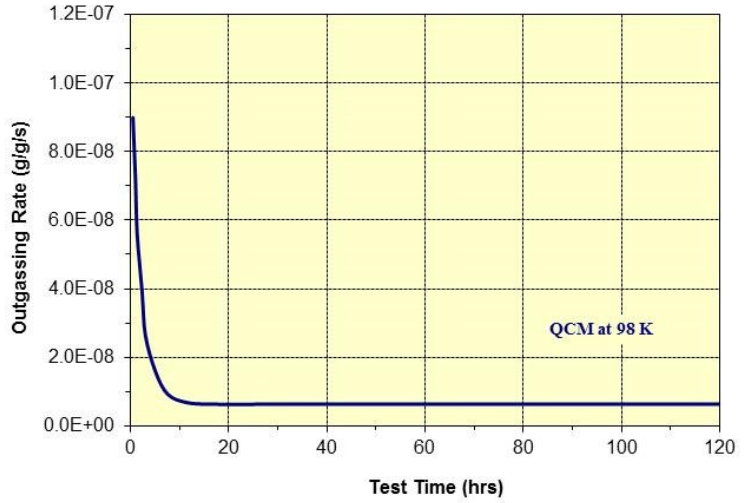
TML MEASUREMENTS

Typical Total Mass Loss and Outgassing Rate measurement by QCM at 90 K and at a given material source temperature.



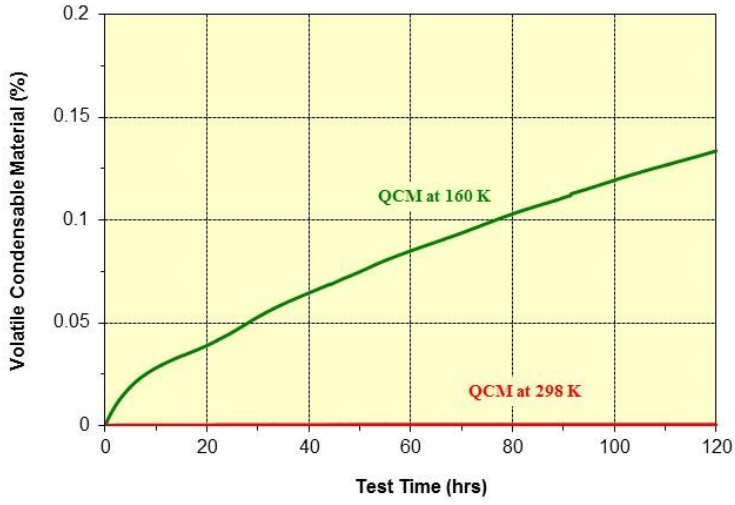






CVCM MEASUREMENTS

Typical Collected Volatile Mass Loss given by QCM at various temperatures.



ASTM E1559 TEST METHOD FOR MATERIALS OUTGASSING/DEPOSITION KINETICS

PREDICTS Outgassing and Deposition Kinetics at selected source temperatures and selected surface temperatures

ALLOWS effective contamination control is essential for the success of most technological applications

MEASURES TML, CVCM, and QTGA

TML

TOTAL MASS LOSS (TML): The prepared material total mass loss at a set source temperature is deposited by known molecular flux onto the coldest (90 K) QCM. The QCM responds to the collecting mass as a frequency shift with test time. This shift can be related lo the mass Joss percentage and by differentiation, the evaporation rate.

CVCM

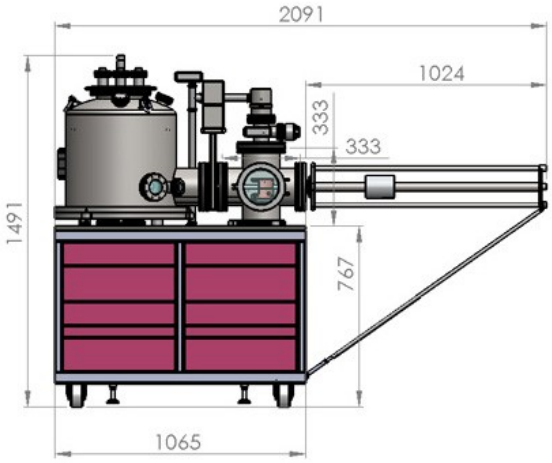
COLLECTED VOLATILE CONDENSABLE MATERIALS (CVCM): Setting the temperature of the collecting QCMs individually the volatile materials portion that is condensable at each receiver temperature can be measured by the QCMs.

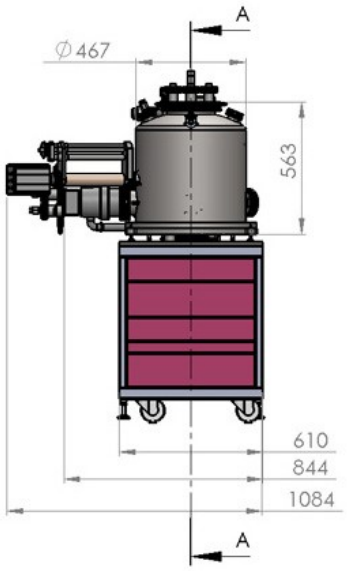
MATERIALS OUTGASSING

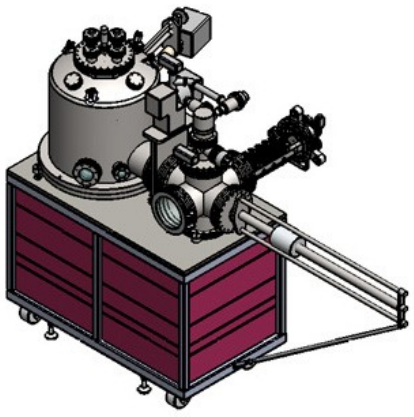
- Prepared materials are subjected to environmental temperatures in an effusion cell
- Effusion cell temperatures range from 293 K to 425 K

QTGA

QCM THERMOGRAVIMETRIC ANALYSIS (QTGA): QCMs can be used to identify the molecular species. Use of an *in-situ* heater in each QCM allows the re-evaporation of the deposited mass sequentially with the temperature, and thus the identification from vapor-pressure characteristics.







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