STATISTICAL EVALUATION OF MOLECULAR CONTAMINATION DURING SPACECRAFT THERMAL VACUUM TEST

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ABSTRACT

The purpose of this paper is to evaluate the statistical molecular contamination data with a goal to improve spacecraft contamination control. The statistical data was generated in typical thermal vacuum tests at the National Aeronautics and Space Administration, Goddard Space Flight Center (GSFC). The magnitude of material outgassing was measured using a Quartz Crystal Microbalance (QCM) device during the test. A solvent rinse sample was taken at the conclusion of the each test. Then detailed qualitative and quantitative measurements were obtained through chemical analyses. All data used in this study encompassed numerous spacecraft tests in recent years.

INTRODUCTION

Outgassing from polymeric materials is a major molecular contamination source for spacecraft. Rigorous contamination control efforts in the selection of materials, cleaning of sub-assemblies, and integration procedures is necessary in order to minimize material outgassing and accomplish an acceptable overall cleanliness. Among all efforts, high temperature thermal vacuum bakeout is considered as one of the most effective methods in reducing the material outgassing in order to achieve acceptable on-orbit performance.

During the thermal vacuum tests, many monitoring devices were applied to measure the outgassing levels. Devices such as a Residual Gas Analyzer (RGA), pressure gauge, and QCM were commonly used tools. These devices provided valuable "fingerprint" information for the conditions of the chamber and the test components. At the conclusion of each test, cold finger and scavenger plate samples were typically taken for further chemical analyses using infrared spectroscopy and mass spectrometry. Individual test data from the monitoring devices and analyses have been widely applied in evaluating the component conditions and identifying outgassing species for contamination control purposes. However, a statistical approach to correlate the data is not currently available.

An effective approach to spacecraft contamination control may be accomplished by a systematic approach based upon statistical material outgassing data. It is imperative to monitor, evaluate, and correlate the contamination data in order to improve overall spacecraft contamination control. Relevant

test data from Hubble Space telescope (HST) and other satellites will be presented to support the application of a statistical evaluation of molecular outgassing and an effective spacecraft contamination control.

MOLECULAR CONTAMINATION INSTRUMENTATIONS

Quartz Crystal Microbalance

The Thermoelectric Quartz Crystal Microbalance (TQCM) and M-2000 control unit system measures and records condensable mass that deposits on a piezoelectric crystal. Extreme accuracy was obtained by comparing the exposed measurement crystal to an encapsulated reference crystal located in the same TQCM head. A computer controlled thermoelectric device provides a high degree of crystal temperature control, which was vital for accurate frequency measurement. Two TQCM sensing units were used, 10 MHz or 15 MHz. All relevant data were sent from the M-2000 control unit and the personal computer control station to the data acquisition and reduction facility.

The dimension of the TQCM was 5 cm (2 in) diameter x 25.5 cm (10 in) length. The mass sensitivity was 4.43×10^{-9} g/cm^2-Hz for the 10 MHz unit and 1.97×10^{-9} g/cm^2-Hz for the 15 MHz unit. The crystal temperature was operated between -50 and +100 °C within ± 0.1 °C accuracy. One or more TQCM sensing units were installed in a thermal vacuum chamber. The chamber was pumped down to a test pressure of 1.33 mPa (10^{-5} torr) or less, at which point the TQCM was turned on and set for the appropriate operating temperature. As the test articles outgassed and materials condensed on the TQCM sensing crystal, the crystal frequency increased directly proportional to the amount of deposition from test article outgassing.

The function and control of a Cryogenic Quartz Crystal Microbalance (CQCM) was similar to that of a TQCM. The CQCM with mass sensitive piezoelectric crystals were operated between –268 and 127 °C.

Residual Gas Analyzer

The RGA was used to measure the partial pressures of ionized molecules over a mass range of 1 to 300 atomic mass units (AMU) where the best sensitivities were below 100 AMU. Using a combined RF and electrostatic field formed by two metal rods, the RGA scanned the mass range and detected the partial pressures of each element or compound fraction. The RGA probe was located in the thermal vacuum facility and was oriented to maximize the detection of the outgassing species. After the facility pressure reached 13.3 mPa (10^{-4} torr), the instrument was activated. An alternate technique was to use the micro-sampling valve at high chamber pressure. Monitoring and recording of the vacuum environment was via a display meter and printer. The data was stored and recalled upon command. An integrated programmable data system with dual disk drives allowed flexibility in scanning, monitoring, storing, printing, and recalling the data. Three models (UTI 100C , PPT-1A-100FC, and Leybold C100F) were available for various test conditions.

Cold Fingers

Cold fingers are small stainless steel cylinders which were mounted in the test volume of each thermal vacuum chamber. The size of the cold finger was 5.0 cm diameter x 10.0 cm height with a surface area of 142 cm^2 nominal. The cold finger was operated during the last 8 hours of the test at an operating temperature of -196 °C (-321 °F). At this cold temperature, condensable vapors were collected by the cold finger and analyzed after the test. In some cases, a large cold plate was used to collect the condensable materials. The cold finger was maintained at liquid nitrogen (LN$_2$) temperature during test and maintained until the chamber was backfilled to 80 kPa (600 torr).

The cold finger was thoroughly cleaned before the test. After the thermal vacuum test, the cold finger was warmed and again rinsed with spectroscopic-grade isopropyl alcohol. The rinse sample was collected in a clean bottle and sent to the Materials Engineering Branch for chemical analysis.

Witness Mirrors

Aluminum coated witness mirrors are used primarily to collect outgassed materials in the thermal vacuum chambers and placement within the chamber was not critical in collecting condensable matter. To determine the quantity of material on the mirror, reflective ultraviolet measurements were made prior to test, and then compared to post-test measurements for reflectivity changes. The mirror was placed in the thermal vacuum chamber (or other environment) and allowed to remain in that location for the duration of the test. In most cases, the mirror's temperature followed the thermal vacuum chamber temperature profile. For large accretions (greater than 10% change in reflectivity) the mirror was sent to the Materials Engineering Branch for chemical analysis of the non-volatile residue (NVR).

The mirror size was 5 cm x 5 cm (2 in x 2 in) with 600 to 800Å of aluminum coated on one side and layered with 250Å of Magnesium Fluoride (MgF_2). Typical measurement wavelength ranges recorded were 1216Å, 1608Å, and 2000Å with ± 2% measurement error. The operating temperature range of the mirrors was between -190 and +100 °C (-310 to +212 °F).

Fourier Transform Infrared Spectrometer (FTIR)

The infrared spectrometer instrument was used to obtain Fourier transform data processing to produce transmission spectra. The Materials Engineering Branch FTIR was a Nicolet 710SX research grade instrument used for the identification of organic functional and transmission characteristics of materials from 4000 to 400 cm^{-1} with a resolution of 4 cm^{-1}. Infrared spectra were produced by measuring the amount of infrared radiation which a material absorbed at different wavelengths. These measurements were then compiled and a "fingerprint" spectrum was produced showing all the absorption bands throughout the infrared region.

Gas Chromatography/Mass Spectrometry (GC/MS)

A gas chromatograph interfaced with a quadrupole mass spectrometer was used to analyze the cold finger residue. The GC/MS had an analysis range of 1 to 1000 AMU. By factoring the instrument parameters along with the type and length of column used in the GC, a precise identification of a compound was made. The system that collected most of the cold finger data was a Nermag R10-10C mass spectrometer interfaced with a Varian 3300 gas chromatograph.

CONTAMINATION DATA COLLECTION

The thermal vacuum chambers were operated by the Environmental Test Engineering and Integration Branch at GSFC. The test articles along with their test fixtures were placed in a chamber where a QCM detected the deposition of mass. A Temperature Controlled Witness Mirror (TCWM) and a metallic cylindrical "cold finger" were applied to collect the contaminant for further study.

The QCM and witness mirror were positioned, usually as close as possible to the test article, to fill the QCM's field-of-view. The QCM, the witness mirror, and the walls of the vacuum chamber were kept at specific conditions. At the end of the test, the cold finger was rinsed and the solvent was collected for chemical analysis. The cold finger was evaporated in air and the total amount of residue was determined. FTIR analysis was then performed on the residue. Since the residue was frequently a mixture of various

compounds, the FTIR analysis was used as a screening test to identify the various classes of compounds present in the sample. The residue was analyzed using GC/MS to obtain a more precise identification of each component.

Statistical QCM data

Statistical QCM data were obtained from the thermal vacuum tests between November 1995 and January 1997. The types of the tests were thermal vacuum, thermal balance, and bakeout. About 200 thermal vacuum tests were performed in this time period. There were only 167 tests with complete QCM and cold finger data for this statistical study. Test articles included the flight materials, thermal blanket, solar array, electronic boxes, harness, cables, flight support hardware, instruments, and full spacecraft complements. Major projects such as: HST, Tropical Rainfall Measuring Mission (TRMM), Cassini Composite Infrared Spectrometer (CIRS), Mars Observer Laser Altimeter (MOLA), Geostationary Operational Environmental Satellite (GOES), Fast Auroral Snapshot Explorer (FAST), X-ray Timing Explorer (XTE), and Transition Region and Coronal Explorer (TRACE) were included in the data reduction.

During the thermal vacuum tests, recorded QCM readings were strongly influenced by the collection temperature and environmental parameters of the thermal vacuum chamber. Therefore, the relationships among QCM temperatures, chamber shroud temperatures, and QCM readings were critical elements to be studied. These factors were changed frequently to reflect the test conditions stated in the thermal vacuum test procedures. For a meaningful comparison, only data prior to the activation of the cold finger was compiled. As shown in Table 1, 167 QCM data points were reported for various temperature ranges. Under normal tests, QCMs were kept at –20 °C for easy operations and best readings. For the majority of the thermal vacuum tests (156 tests), QCM temperatures were maintained at –20 °C. For these tests, only regular TQCMs were needed. Less than 7% of the time (11 tests), QCMs were held below –50 °C. Under these occasions, colder QCMs were used to simulate actual hardware conditions in order to understand the potential of the molecular deposition. For the test requiring the temperature as cold as –135 °C, a CQCM was needed.

Table 1   QCM Temperatures

QCM Temperatures (°C)	Number of Tests
-135	1
-50 to -70	10
-20	156
	167

During the thermal vacuum tests, the shroud temperatures greatly affected the temperature profiles and material outgassing. In these 167 tests, the shroud temperatures were maintained between –175 and 100 °C to provide proper temperature conditions for the test articles. As shown in Table 2, there were 42 tests with the shroud temperature below –20 °C, 7 tests with a range of –20 and 25 °C, 65 tests with a range of 25 and 90 °C, and 53 tests above 90 °C. The distribution of the shroud temperature reflected the nature of the tests. Normal thermal vacuum tests were operated between –20 and 90 °C. During the bakeout or chamber certification, the temperature of the shroud was raised to approximately 100 °C to promote the material outgassing and chamber cleaning. Chamber temperatures higher than 100 °C were prohibited to prevent severe outgassing from the chamber wall or test articles. Cold chamber shroud, functioned like a large scavenger plate, and thus isolated chamber outgassing from test articles outgassing.

In general, liquid or gaseous nitrogen was required to cool the shroud for the below room temperature operation.

Table 2   Thermal Vacuum Chamber Shroud Temperatures

Shroud Temperatures (°C)	Number of Tests
< –20	42
–20 to 25	7
25 to 90	65
> 90	53
	167

Both 10 MHz and 15 MHz QCMs were commercially available. Each type of QCM had a unique mass sensitivity depending upon the manufacturer. The baseline for the thermal vacuum tests at GSFC was the 10 MHz QCM. For this study, the values of the 15MHz QCM were converted to the 10 MHz QCM. Table 3 shows the actual or the equivalent readings of a 10 MHz QCM for all 167 tests taken prior to the activation of the cold finger. There were 22 tests with the QCM readings below 1 Hz/hr, which was the minimum sensitivity of a QCM. Two significant factors existed for tests with QCM readings of less than 1 Hz/hr. The negligent QCM readings indicated either the test articles were required to meet a very stringent requirement or the tests were performed at low temperatures. The majority of the QCM readings were within 2 to 250 Hz/hr which illustrated the goal of reducing test article outgassing during the thermal vacuum tests. The tests with QCM readings around 300 Hz/hr represented the bakeout or chamber certification. The QCM value of 300 Hz/hr was a general criterion for a hardware bakeout process that the chamber was guaranteed to meet. For the post chamber certification, the thermal vacuum chambers were required to meet the 300 Hz/hr cleanliness level.

Table 3   QCM Readings

QCM Readings (Hz/hr)	Number of Tests
< 1	22
2 to 50	65
50 to 250	24
250 to 300	37
> 300	19
	167

Statistical cold finger chemical analysis data

In addition to the statistical QCM data, the statistical chemical analysis data were also obtained. Upon receiving the cold finger sample, the Materials Engineering Branch's laboratory performed the chemical analyses using the FTIR and GC/MS techniques. NVR results from these chemical analyses provided the quantity of the molecular contaminants, identified various outgassing species, and in some cases confirmed contamination sources. Table 4 shows the quantity of the NVR collected during the cold finger period. The amount of the NVR was directly proportional to the molecular outgassing inside the thermal vacuum chamber after the activation of the cold finger. There were 31 tests with the NVR values of less than 0.1 mg. A small NVR (<0.1 mg) always indicated a low outgassing nature of the test articles. For example, some HST hardware were required to be baked out to below 1 Hz/hr. Most of the NVR

values after normal thermal vacuum tests were below 0.5 mg. Typical NVR values after the chamber bakeout and certification were around 1.0 mg because of the high temperature.

NVR values of 2.0 mg and higher suggested problems associated with the test articles and/or thermal vacuum chambers. One example was that a high concentration of hydrocarbons was constantly detected when the temperature of the chamber shroud was 100°C or higher. These hydrocarbons were associated with the lubricating oils, machining oils, and greases used on various parts of the thermal vacuum chamber. The other example was due to the malfunction of the cooling system of the cold finger. The cold finger sample was inadvertently activated at the beginning of the thermal vacuum test, and exceeded the normal 8-hour operation period.

Table 4   Cold Finger Results

NVR (mg)	Number of Tests
< 0.1	31
0.1 to 0.5	52
0.5 to 1.2	49
> 1.2	35
	167

Cold finger chemical species

Besides the NVR values, principal chemical species were identified from the chemical analysis. The chemical species with their frequency of occurrences are shown in Table 5. There were more than 40 identifiable chemical species commonly detected during the thermal vacuum tests at GSFC.

A plasticizer is a high-boiling molecular compound or softening agent, usually liquid, added to a polymer to facilitate processing or to increase flexibility or toughness. Some commercial plasticizers use complex mixtures of phthalate esters. Di-(2-ethylhexyl) phthalate (DEHP) is used primarily as one of several plasticizers in polyvinyl chloride (PVC) polymers to make them flexible. Some PVC can contain up to 50% by weight of plasticizers. DEHP was the most observed plasticizer of GSFC's cold finger samples. Dibutyl phthalate (DBP) and Butyl benzyl phthalate (BBP) also function as plasticizers for many rubbers and a wide range of other synthetic resins. In addition, di-(2-ethylhexyl) adipate, di-(2-ethylhexyl) azelate, and di-(2-ethylhexyl) sebacate were also frequently outgassing compounds from the polymeric materials. Triphenyl phosphate (TPP) and 2-butoxy ethanol phosphate 3:1 are common flame retardant compounds added to the polymeric materials either as plasticizers or as an anti-wear additive. TPP was found to be outgassed from the flight hardware as the additive to the TRMM thermal blankets.

Room-temperature vulcanizing (RTV) silicone adhesives and sealants are designed for a wide range of sealing, bonding, and protection for spacecraft applications. Therefore, adhesive material of silicones were easily detectable during the thermal vacuum tests. There were 88 occurrences of either methyl silicones, methyl phenyl silicones, fluorinated silicones, or silicones. On one sample, methyl silicones was detected because a piece of pink polyester tape with a silicone adhesive was inadvertently placed on the thermal blanket. This blanket was removed from a test fixture hardware, the pink polyester tape was removed, and the fixture was cleaned with a solvent prior to installing a new thermal blanket on the fixture. Methyl silicones were also identified to be released from silicone grommets used in the assembly of the electrical connectors, from silicone foam, from Furon Chorlastic R-10404, or from Thermobond used for the heat sink on the circuit boards. Fluorosilicone material, ZZ-R-765, was

Table 5 Occurrence Frequency of Chemical Species

Chemical Species	Number of Occurrences
Plasticizer	
Di-(2-ethylhexyl) phthalate	159
Dibutyl phthalate	31
Butyl benzyl phthalate	98
Didecyl phthalate	19
Dioctyl phthalate	10
Phthalate ester	46
Di-(2-Ethylhexyl) adipate	74
Di-(2-Ethylhexyl) azelate	66
Di-(2-Ethylhexyl) sebacate	70
2-Mercaptobenzothiazole (MBT)	6
Triphenyl phosphate (TPP)	40
2-butoxy ethanol phosphate 3:1	18
Azelate ester	4
Butylbenzenesulfonamide	1
Elastomers	
Methyl silicones	52
Methyl phenyl silicones	25
Fluorinated silicones	2
Silicones	9
Polymers and Adhesives	
Bis-phenol-A(Epoxy)	45
Methylene diphenol (Primer 9924)	12
Aromatic amine (uralane)	21
Methyl-benzenediamine (epoxy)	13
Unknown amine (epoxy)	9
Polyurethane curing agent	5
Unknown phthalimide (epoxy)	14
Aliphatic amine (epoxy)	7
Acrylic adhesive	1
Other Compounds	
Saturated & unsaturated hydrocarbons	167
Detergent derivatives	67
Palmitic acid	114
Myristic acid	18
Stearic acid	51
Lauric acid	3
Benzoic acid	4
Calcofluor (Fluorescent tracking compound)	20
Tetraethylene glycol di-2-ethylhexanoate	3
DC704 diffusion pump oil	20
Phthalic anhydride	14
2-Hydroxy 4-methoxy benzophenone (antioxidant)	2
Erucamide	1
Tri-(allyl) cyanurate	1

identified as part of the assembly of the electric connectors (the insulator material) used on the flight hardware.

Caprolactam (Nylon-6 bagging contaminate) did not appear in any cold fingers while Tri-(allyl) cyanurate was only detected once. These highly volatile compounds were normally vaporized before the activation of the cold finger. As a result, these compounds were detected in large quantities in the scavenger plate samples which were activated earlier to preserve volatile chemical species. In contrast, Bis-phenol-A was less volatile and was only found in certification samples because it was derived from the epoxy chamber coatings at high temperature. Bis-phenol-A was a major outgassing species from the TRMM beta cloth. Methylene diphenol most likely came from Chemglaze 9924 wash primer used with polyurethane coating materials on the flight hardware.

Occasionally an aromatic amine from the curing agent in various Uralanes was detected from the test samples. For example, Uralane 5753LV with CAB-O-SIL was used as a staking compound on a piece of flight hardware. Final analysis could not define whether the curing agent was excessively added to the mixture or whether the polyurethane was not cured completely before the thermal vacuum test.

Saturated & unsaturated hydrocarbons were the most common chemical species. The butoxy compound, possibly from the detergent used for cleaning the chambers, was detected when the temperature of the chamber was high and the amounts of other residues were low. Fatty acids such as palmitic acid, myristic acid, stearic acid, and lauric acid were frequently found in the thermal vacuum test. They were often natural organic oils commonly used in polymers, antistatic agents, and general handling contaminants (including fingerprints). They were soluble in alcohol and were easily removed by a simple solvent cleaning.

Calcofluor, a fluorescent tracking compound, was observed 20 times during all tests. This was an additive usually found in polyurethane conformal coatings. The percentages added to the conformal coating usually varied from tenths of a percent to a few percent. DC704 diffusion pump oil occurred 20 times during the evaluation period. Specifically there were 11 times for chamber 237, 5 times for chamber 243, and 4 times for chamber 238. Even though DC704 was no longer used at GSFC, traceable quantities of the compound still appeared in the cold finger analysis. The presence of DC704 was a strong indication that the hardware was contaminated during previous thermal vacuum tests conducted at vendor facilities because GSFC's Chambers 237, 238, and 243 used Santovac diffusion pump oil. This indicated that thermal vacuum chambers equipped with diffusion pumps and DC704 were still common in the aerospace industry. Tetraethylene glycol di-2-ethylhexanoate was a unique chemical species which was detected only in Chamber 290.

Major cold finger chemical species

In each chemical analysis, as high as 15 chemical species were identified and listed according to their relative quantities within the test sample. In general the top 3 chemical species represented the most significant outgassing components during the thermal vacuum tests. A summary of 11 common chemical species were found to be among the top 3 of all identified chemical compounds (refer to Table 6). As shown in Table 6, saturated & unsaturated hydrocarbons (164 times) and DEHP (140 times), butoxy compound (34 times), and methyl silicones (22 times) were major chemical species. Even though the butoxy compound and methyl silicones were less frequent in the overall samples surveyed, i.e. Table 5, they were identified as significant chemical species as shown in Table 6. Therefore, the summary list in Table 6 identified the significance and relative quantity of the common chemical species.

Table 6 Occurrence Frequency of Major Cold Finger Chemical Species

Chemical Species	Top 3
1. Saturated & unsaturated hydrocarbons	164
2. Di-(2-ethylhexyl) phthalate	140
3. Butoxy compound (detergent derivatives)	34
4. Methyl silicones	22
5. Palmitic acid	21
6. Phthalate ester	16
7. Methyl phenyl silicones	14
8. Di-(2-ethylhexyl) adipate	13
9. Bis-phenol-A (epoxy)	10
10. Triphenyl phosphate (TPP)	9
11. Di-(2-ethylhexyl) azelate	9

COMPARISON WITH EARLY CHEMICAL ANALYSIS DATA

Extensive chemical analyses were performed at GSFC from 1970 to 1978. A total of 1163 data points were reported by Colony[1] in 1979. In the report, individual materials were incorporated into their generic classes along with the frequency and percentage. Esters were the leading generic class of compounds found in the early survey, followed by hydrocarbons and methyl silicones. As shown in Table 7, the chemical classes of the 70s consisted of 36% esters, 23% aliphatic hydrocarbons, 16 % methyl silicones, 5.7% aromatic silicones, 4.7% antioxidants, 2.8% aromatic hydrocarbons, 2.2% polyurethane derivatives, 1.1% organic acids, and 9% other materials. Within the classes, DEHP was the most often found individual species in large outgassing samples. The compound TPP appeared in 1972-1973 and then not again until 1977. The use of di-(2-ethylhexyl) azelate increased radically in 1976 and 1977 and disappeared after 1977.

Table 7 GSFC Chemical Analysis Data Comparison

Chemical Classes	Percentage (%)		
	1970-1978	1995-1997	
		All Occurrences	Top 3
1. All esters	36	47.2	41.4
2. Aliphatic hydrocarbons	23	12.4	32.7
3. Methyl silicones	16	4.5	4.8
4. Aromatic silicones	5.7	1.9	2.8
5. Antioxidants	4.7	0.1	0.0
6. Aromatic hydrocarbons	2.8	0.0	0.0
7. Polyurethane derivatives	2.2	2.5	0.8
8. Organic acids	1.1	14.1	5.2
9. Other materials	9	17.3	12.3

Based upon all occurrences, the chemical classes of 1995-1997 consisted of 47.2% esters, 12.4% aliphatic hydrocarbons, 4.5 % methyl silicones, 1.9% aromatic silicones, 0.1% antioxidants, 2.5% polyurethane derivatives, 14.1% organic acids, and 17.3% other materials. However, based upon the top 3 species, the chemical classes of the 90s changed to 41.4% esters, 32.7% aliphatic hydrocarbons, 4.8 % methyl silicones, 2.8% aromatic silicones, 0.8 polyurethane derivatives, 5.2% organic acids, and 12.3%

other materials. Esters and hydrocarbons remained as the leading generic classes. However, the usage of methyl silicones was reduced. Antioxidants and aromatic hydrocarbons did not appear on the top 3 list indicating the reduction of the usage in spacecraft materials. DEHP continued to be the most often found individual species in outgassing samples. Both TPP and di-(2-ethylhexyl) azelate appeared frequently and showed 9 times each on the top 3 list.

In summary, the esters, aliphatic hydrocarbons, and methyl silicones accounted for 75% of the outgassing problems in the 70s. In comparison, these 3 species account for 64% (based upon all occurrences) or 79% (based upon top 3) of the outgassing problems in the 90s. This comparison also demonstrated that even though more polymeric materials were incorporated into spacecraft now than in the 70s, they were pre-treated to prevent high concentrations of outgassing species. This actually reduced the levels of species that were seen in the cold finger samples (For example, the decrease in methyl silicone levels).

CONTAMINATION DATA CORRELATION

At the end of the thermal vacuum tests, the QCM readings were used to provide test termination criteria. These readings illustrated the dynamic outgassing behavior of the test articles and thermal vacuum chamber conditions. However, the QCM was not capable of determining the outgassing species unless an extensive Thermogravimetric Analysis (TGA) procedure was performed. This may not always be possible with long chain hydrocarbons or high AMU species. During the thermal vacuum tests, however, the TGA was applied only for very specific test conditions. On the contrary, the NVR values from the cold finger tests was not transient. The NVR values provided a collected information of the quantity and chemical constituents of the cold finger sample. Utilizing the combined QCM and NVR information helped provide a better understanding of the thermal vacuum tests. In addition, the correlation of the QCM readings from thermal vacuum tests and NVR values from the cold finger tests helped provide a trend of all thermal vacuum tests.

As shown in Figure 1, the NVR values are plotted against the QCM values. Data with QCM values higher than 350 Hz/hr or NVR values larger than 3.0 mg were not selected. These data points were excluded due to incompleteness, inconsistency, chamber failure, or abnormal high outgassing condition. All QCM readings were reasonably separated into two major groups according to their thermal vacuum shroud temperatures. Large variations of some data points may be caused by the locations of QCMs. Generally, the QCM values of the cold shroud tests (−20 °C or below) were less than 50 Hz/hr and the NVR values were below 1.0 mg. For the warm shroud tests (above room temperature), the main range of the NVR values was between 0.1 and 2.0 mg. And the majority of the QCM values were below 300 Hz/hr. For data point with the same QCM value, the cold shroud tests generated a higher NVR value than that of the warm shroud.

At GSFC, the QCM requirement of the post-test chamber certification was 300 Hz/hr. As shown in Figure 1, only a few of the certifications have the QCM values higher than 300 Hz/hr, i.e. non-certified. However, for those non-certified chambers, the 300 Hz/hr was achieved prior to the next thermal vacuum test cycle. The range of the NVR for all certified chambers was between 0.3 to 2.5 mg. It was safe to assume that the chamber was certified if the NVR was below 1.0 mg limit.

CONCLUSIONS

To understand material outgassing under thermal vacuum conditions, it was advantageous to continuously monitoring the chamber condition by the QCM and cold finger. The QCM and cold finger
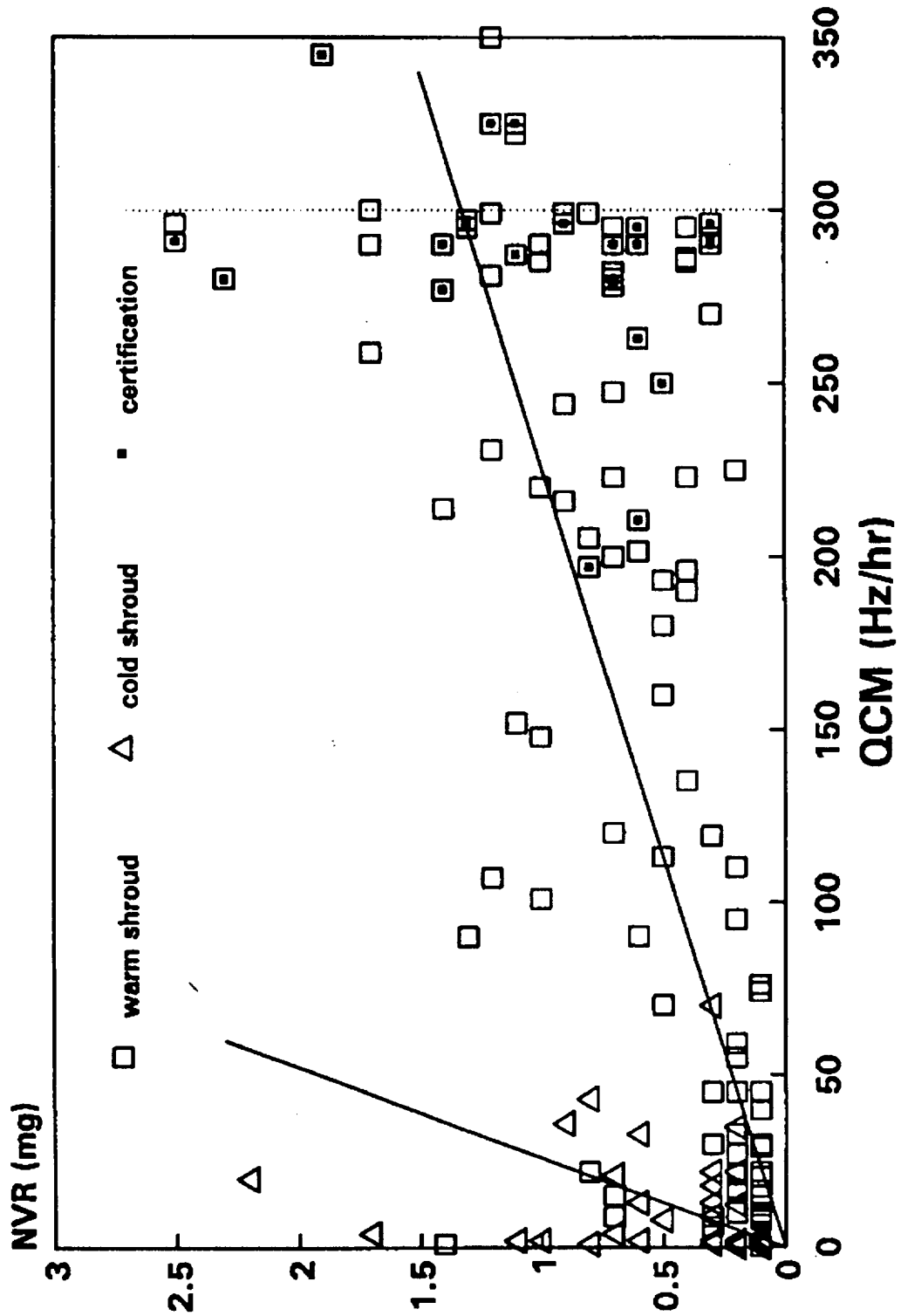
Figure 1 Thermal vacuum QCM values and chemical analysis NVR results

provided complementary information for both qualitative and quantitative contamination assessments. Chemical analysis also provided interesting information such as either the unique evidence of contamination transfer (DC704 diffusion pump oil) or unique signature of a chamber (Tetraethylene glycol di-2-ethylhexanoate).

The conditions of the chamber shroud were very important for a successful thermal vacuum test. Test conditions were influenced by the shroud temperatures. For bakeout or chamber certification, the shroud temperature was raised to a maximum temperature to expedite outgassing. On the contrary, the cold shroud served as a cold sink that could provide a favorable clean environment for the test articles. Therefore, it was important to understand the status of the chamber conditions and monitor by way of the QCM and cold finger.

During the thermal vacuum test, the QCM and chamber shroud temperatures strongly affected the QCM readings. The placement of the QCMs in the thermal vacuum chamber was also an important factor of QCM results. QCMs performed adequately for the majority of the thermal vacuum tests when maintained at a stable temperature of -20 °C. The QCM value of 300 Hz/hr was a general criterion for the bakeout and chamber certification at GSFC. Typical NVR values after the chamber bakeouts and certifications were around 1.0 mg. Therefore, from a statistical point of view, it was reasonable to assume that the chamber was certified if the NVR was below 1.0 mg limit. A small NVR (<0.1 mg) always indicated the low outgassing nature of the test articles. A high NVR (>2.0 mg) indicated problems associated with the test articles and/or thermal vacuum test chambers.

The chemical analysis was not only effective in determining the outgassing potential, but also useful in assessing the conditions of the facility. The variation of NVR collected from a particular chamber provided the long-term conditions of the thermal vacuum chamber and its accessories. The continuous appearance of high NVR values and/or specific chemical species indicated the need for chamber recertification.

DEHP, hydrocarbons, and silicones were the most common chemical species in outgassing samples in the 70s as well as in the 90s. The evidence of these phthalate esters and hydrocarbons indicated their extensive use in many polymers and lubricants. The detergent derivative of butoxy compound appeared often whenever the temperature of the chamber shroud was raised above 70°C. Due to its wide applications, silicones, continue to be detected in the cold finger samples. The comparison of current and previous GSFC chemical analysis data showed the variations of material usage, material development, process changes, test concepts, and test approaches.

The statistical approach allowed for a parametric correlation of QCM and NVR values obtained from thermal vacuum tests. As expected from the correlation, the reduction in outgassing has been observed on the QCM and supported by NVR results. The correlation is not definitive, but it helps in determining the degree of success of an established contamination control approach. A precise determination is difficult to derive because of the dynamic changes in materials used in the manufacturing processes that affect outgassing and NVR results.

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