



# Quartz Crystal Microbalance Operation and In Situ Calibration

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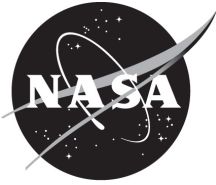
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## LIST OF ACRONYMS

ASTM	American Society for Testing and Materials
CQCM	cryogenic quartz crystal microbalance
QCM	quartz crystal microbalance
TM	Technical Memorandum
VCM	volatile condensable material
VF	view factor

## NOMENCLATURE

$A$	orifice radius
$Ea$	activation energy (cal/mole)
$K$	Clausing factor
$L$	orifice length
$R$	gas constant, 8.3144 kJ/mole-K; 1.9872 cal/K/mole
$r^2$	linear regression coefficient
$R1$	deposition rate measured at $T1$ (moles/cm <sup>2</sup> -s)
$R2$	deposition rate measured at $T2$ (moles/cm <sup>2</sup> -s)
$T$	surface temperature (K)
$T1$	lower effusion cell temperature (K)
$T2$	higher effusion cell temperature (K)
$\tau$	surface dwell time (s)
$\tau_o$	vibration frequency (vibrations/s)



## TECHNICAL MEMORANDUM

### QUARTZ CRYSTAL MICROBALANCE OPERATION AND IN SITU CALIBRATION

#### 1. INTRODUCTION

Quartz crystal microbalances (QCMs) are commonly used to measure the rate of deposition of molecular species on a surface. The usual process is that the deposition rate for a specific material is measured and then mathematically modified to calculate a source term, or outgassing rate, for the material. The source term, which is representative of the material's outgassing rate, can be used as input for computer programs that predict the rate of deposition of the emitted or outgassed material on another surface.

The outgassing process is diffusion controlled, and the rate of release, or outgassing, is temperature dependant. Outgassing measurements are not typically used to measure the release of material due to degradation of the parent material or the material generated by the chemical interactions of the released compounds that result in the production and deposition of a different chemical species.

The deposition process is temperature dependant with the efficiency of the condensation process increasing as the deposition surface temperature becomes increasingly colder than the temperature of the outgassing material.

The species, molecular or atomic, released from a material during the outgassing process are typically quantified and described in two ways: (1) The total amount of material that is lost through the outgassing process is often referred to as the total mass loss for the material, and (2) material released during the outgassing process that will recondense on another surface is often identified as volatile condensable material (VCM). Both quantities can be expressed as a percentage of the original sample mass, but only VCM can also be expressed as an outgassing rate when measured with an instrument such as a QCM.

## 2. QUARTZ CRYSTAL MICROBALANCE TYPES

QCMs typically fall into two major categories and three subcategories. QCMs either have a single quartz crystal or a matched pair of quartz crystals, forming a clear distinction between the two major classes of QCMs. This Technical Memorandum (TM) will only address the QCMs that have a matched pair of quartz crystals. One of the crystals serves as a reference oscillator while the deposition of VCMs occurs on the surface of the other quartz crystal. This class of microbalances can be further subdivided into the following:

- QCMs: These microbalances do not have an active mechanism to control the temperature of the deposition surface. Heating and cooling of the microbalance is typically done by controlling the temperature of the structure on which the QCM is mounted. These are the simplest of the QCMs that have a matched pair of crystals.
- Temperature-controlled QCMs: The temperature of the quartz crystals in these balances is actively controlled. Both cooling and heating is controlled, often by building a Peltier device, a solid state heater/cooler, into the balance.
- Cryogenic QCMs (CQCMs): These microbalances can actively heat the crystal pair, but they rely on passive cooling to control the temperature of the deposition surface. Cryogenic deposition surface temperatures are passively achieved by mounting this microbalance on a cryogen-cooled, typically liquid nitrogen, surface.

Throughout the remainder of this TM, the initialism QCM will be used as the generic label for all three types of QCM.

### 3. MODIFICATION OF THE MEASURED DEPOSITION RATE

The measured deposition rate is simply a measurement of how fast the outgassed materials condense on the deposition surface of the QCM. The difference in oscillation of the reference crystal and the crystal on which material deposits is often referred to as the beat frequency. Using a sensitivity factor specific to the type of QCM being used, the measured change in frequency can be converted into a deposition rate. The sensitivity factor is normally provided by the QCM vendor. The deposition rate is typically reported in units of grams per centimeter squared-second and is calculated from the observed change in the beat frequency with time ( $\Delta\text{Hz/s}$ ).

The source term, or outgassing rate, for a material is normally different than the measured deposition rate and is arrived at mathematically. The calculation involves a view factor (VF) and is further modified by the sample surface area.

The view factor is a mathematical description of the geometry between the emitting surface, the outgassing source, and the deposition surface. If the measurement system employs an effusion cell for heating the sample, then the length and radius of the effusion cell orifice along with the sample surface area must also be considered in the calculation. An additional factor, the Clausing factor, that quantifies the conductance of the orifice (app. A) must also be incorporated into the VF calculation.<sup>1</sup>

For the measurements appearing in this TM, the VF calculation presented in American Society for Testing and Materials (ASTM) method E-1559 has been used to calculate the reported source terms.<sup>2</sup> The ASTM method E-1559 VF is not the only VF calculation employed for the calculation of source terms. The ASTM E-1559 VF method is specific to measurements in which the sample is heated in an effusion cell and should not be used to calculate surface-to-surface VFs.<sup>3</sup>

#### 3.1 Dynamic Deposition Process

A simple assumption often made is that the material being deposited on a surface remains on the surface and that all the condensed species have the same affinity for the deposition surface. This is often not the case, and some deposited material may be lost, or reevaporated, from the deposition surface while the majority of the condensate remains on the deposition surface. Often, the sticking coefficient or factor for a material is set at 1 to simplify the measurement and modeling of the deposition process.

Cooling the outgassing source below the temperature of the deposition surface, effectively turning off the outgassing source, often reveals a slow loss of material from the deposition surface. Reevaporation usually occurs at a significantly lower rate than the measured deposition rate and varies with the deposition surface temperature and composition.

The dwell time of a molecular species on a surface can be calculated if all the required parameters are obtained from laboratory measurements or published references (app. B). However, this level of precision is not needed for the typical applications that the outgassing rate measurements are intended to support.

#### 4. VERIFICATION OF SOURCE-SENSOR RELATIONSHIP

When multiple QCMs are used to measure the outgassing from a common source, the VF for each sensor will be unique to the geometry between the source and the sensor. In some systems employing multiple sensors, a common geometry is used by design, and all the sensors have a common VF. The assumption of a common geometry can be confirmed by comparing the deposition rates that are measured by the sensors while viewing the same outgassing source. For this technique to be effective, the deposition surfaces of the sensors must all be at the same temperature and the sample/QCM geometries must be uniform. Figures 1 and 2 are frequency curves for four CQCMs viewing an effusion cell (fig. 3) containing a sample of adipic acid (Chemical Abstracts Service Registry Number: 124-04-9). The slopes for the curves presented in table 1 were obtained from a linear regression analysis of the individual frequency curves.

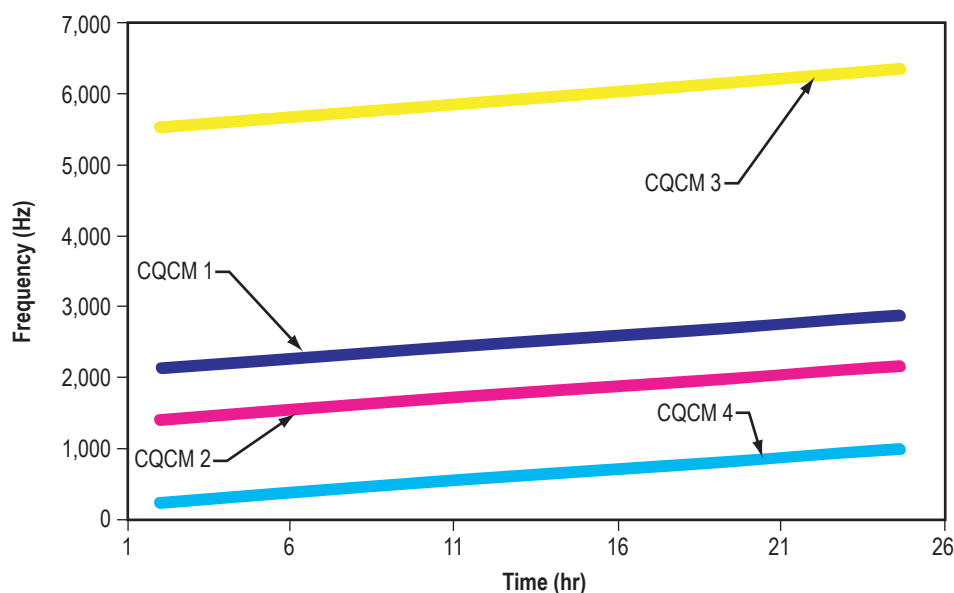


Figure 1. Four CQCMs with a common outgassing source-to-sensor geometry. During the first 2 hr of the measurement, two of the CQCMs were warmed from  $-100$  to  $-62$   $^{\circ}\text{C}$ , and this period was not included in the plot. (Adipic acid, January 27, 2004:  $-62$   $^{\circ}\text{C}$  deposition surface,  $55$   $^{\circ}\text{C}$  effusion cell—from 2 to 24.50556 hr.)

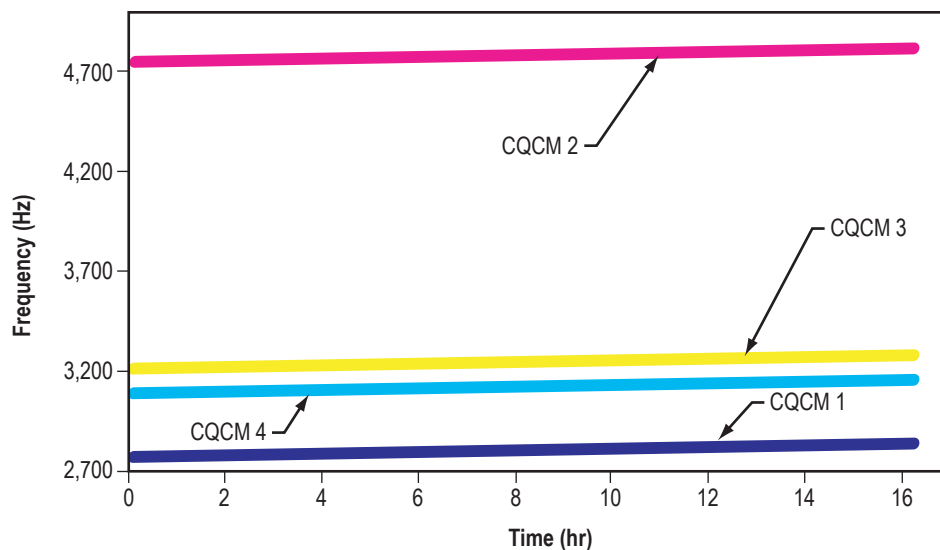


Figure 2. Another example of multiple CQCMs with a common source-to-sensor geometry. (Adipic acid, June 30, 2003:  $-42^{\circ}\text{C}$  deposition surface,  $41^{\circ}\text{C}$  effusion cell—from zero to 16.25 hr.)

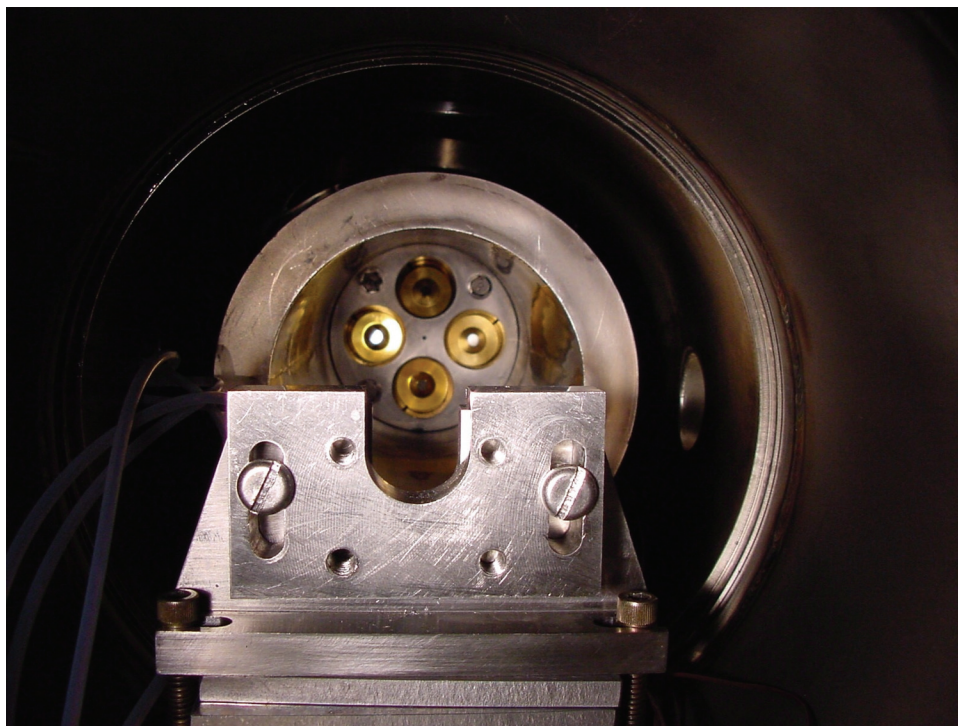


Figure 3. The four CQCMs used to collect the data presented in figures 1 and 2 as they are mounted in the vacuum chamber. The effusion cell, in which the sample is heated, would be mounted on the bracket in the foreground.

Table 1. Frequency data for multiple sensors with a common source-to-sensor geometry.

QCM	Effusion Cell Temperature (°C)	Deposition Surface Temperature (°C)	Frequency (Hz/hr)	Regression Coefficient
January 2004				
CQCM 1	55	−62	31.99	0.9998
CQCM 2	55	−62	33.904	1
CQCM 3	55	−62	36.464	0.9999
CQCM 4	55	−62	33.581	0.9998
June 2003				
CQCM 1	42	−42	4.0657	0.9978
CQCM 2	42	−42	4.2807	0.9977
CQCM 3	42	−42	5.2736	0.9963
CQCM 4	42	−42	3.5931	0.9901

In each instance, perfect agreement between the slopes of the curves was not obtained, suggesting a bias of the common geometry to one of the sensors. The amount of bias that is acceptable for the measurement being made should be taken into account when determining if further modifications to the measurement setup are required or if the measurement setup is adequate to meet the requirements for the accuracy of the data. The performance of the individual sensors can have a large influence on the perceived bias and should also be addressed in the evaluation of a multisensor system.

## 5. INDIVIDUAL SENSOR PERFORMANCE

The quality of the measurement made with each individual QCM can be assessed using deposition measurements made with the QCM to calculate the enthalpy of sublimation for a known condensate species.<sup>4</sup> A series of deposition measurements is made at a constant deposition surface temperature and several source temperatures. The quality of the individual deposition measurements is then assessed using a linear regression analysis of the data to establish the quality of the data and to obtain the slope of the frequency curve. The slope is usually measured in hertz per hour (Hz/hr) and, using the QCM sensitivity factor, converted to a deposition rate, g/cm<sup>2</sup>-s. The deposition rate is then converted to a source term, which is stated in moles/cm<sup>2</sup>-s.

The material selected for the measurements must sublime as a single species and cannot degrade during the outgassing/deposition process.<sup>5</sup> Values for the enthalpy of sublimation and the molecular weight and melting point of the material can be obtained from various chemical references.<sup>6-8</sup> Melting the material is undesirable and will produce erroneous results if the enthalpy of sublimation is calculated using data obtained from a melted sample.

Using the source terms measured for two different effusion cell temperatures, the enthalpy of sublimation for the material can be calculated using the van't Hoff relation:<sup>9</sup>

$$\text{Enthalpy of sublimation} = R[T_2 - T_1 / T_2 \times T_1] \times \ln[R_1 / R_2] \quad , \quad (1)$$

where

$R$  is 8.3144 kJ/mole-K, the gas constant

$T_1$  is the lower effusion cell temperature (K)

$T_2$  is the higher effusion cell temperature (K)

$R_1$  is the deposition rate measured at  $T_1$  (moles/cm<sup>2</sup>-s)

$R_2$  is the deposition rate measured at  $T_2$  (moles/cm<sup>2</sup>-s).

To illustrate the process, the enthalpy of sublimation for adipic acid,  $129.3 \pm 2.5$  kJ-mole<sup>-1</sup>, has been calculated and is presented in table 2.<sup>8</sup>

Exact agreement between the calculated enthalpy of sublimation and published values may not always be achieved, and a decision on the accuracy needed for the measurements will have to be made by those making the measurement. Often the published values for the enthalpy of sublimation will be a value plus or minus some uncertainty, which makes exact agreement between the calculated and those published values even more difficult to achieve (app. C). The objective is to show that the data collected with each QCM as an indicator of QCM performance are accurate and reasonable over a range of source temperatures.



Table 2. Enthalpy of sublimation calculated from adipic acid deposition measurements.

CQCM No.	Deposition Surface Temperature (°C)	T1 (°C)	T2 (°C)	R1 (moles/cm <sup>2</sup> -s)	R2 (moles/cm <sup>2</sup> -s)	Enthalpy of Sublimation (%)
1	-62	33	55	$1.9 \times 10^{-11}$	$5.2 \times 10^{-10}$	97.3
1	-62	37	55	$3.47 \times 10^{-11}$	$5.2 \times 10^{-10}$	98.7
1	-62	40	55	$6.26 \times 10^{-11}$	$5.2 \times 10^{-10}$	93.5
1	-62	44	55	$9.67 \times 10^{-11}$	$5.2 \times 10^{-10}$	102.7
2	-62	33	55	$2.14 \times 10^{-11}$	$5.45 \times 10^{-10}$	95.3
2	-62	37	55	$3.72 \times 10^{-11}$	$5.45 \times 10^{-10}$	97.6
2	-62	44	55	$1.01 \times 10^{-10}$	$5.45 \times 10^{-10}$	102.3
2	-62	50	55	$2.8 \times 10^{-10}$	$5.45 \times 10^{-10}$	91
3	-102	33	55	$2.5 \times 10^{-11}$	$7.11 \times 10^{-10}$	98.6
3	-102	37	55	$3.98 \times 10^{-11}$	$7.11 \times 10^{-10}$	105
3	-102	44	55	$1.28 \times 10^{-10}$	$7.11 \times 10^{-10}$	104.6
3	-102	33	50	$2.5 \times 10^{-11}$	$4.39 \times 10^{-10}$	107.5

## 6. MEASUREMENT DURATION

The number of the data points collected during each deposition measurement should lend itself to good statistical analysis. Data collection intervals of 5 to 300 s over a period of 10 hr or more will provide an adequate amount of data with good resolution for deposition calculations (fig. 4). In addition, the random QCM frequency data variations from fluctuations in the sample temperature, small variations in the deposition surface temperature, and disturbances from cryo-panel fill cycles will have a less pronounced effect when data are collected for reasonably long periods (fig. 5).

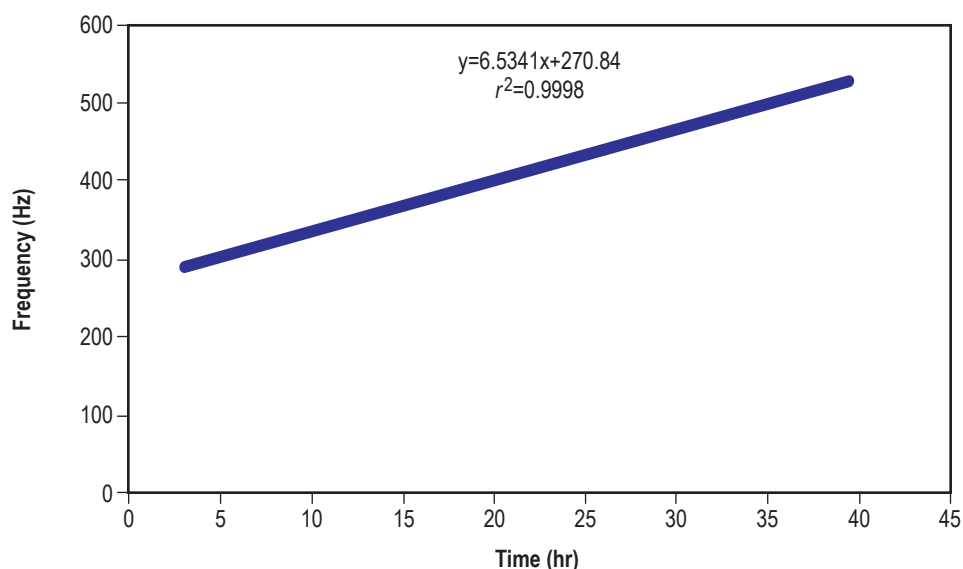


Figure 4. Linear regression analysis of frequency data collected with a CQCM. The slope for the curve is x-value multiplier (6.5341 Hz/hr) and the  $r^2$  value (0.9998) is the linear regression coefficient. (Adipic acid, CQCM 2, January 24, 2004:  $-62^\circ\text{C}$  deposition surface,  $44^\circ\text{C}$  effusion cell.)

Linear regression analysis is a good method for the evaluation of frequency data. It provides the slope of the frequency curve in addition to a linear regression coefficient. Typically, data with a correlation coefficient  $> 0.99$  are of high enough quality to produce good deposition data and are adequate for the accurate calculation of the enthalpy of sublimation.

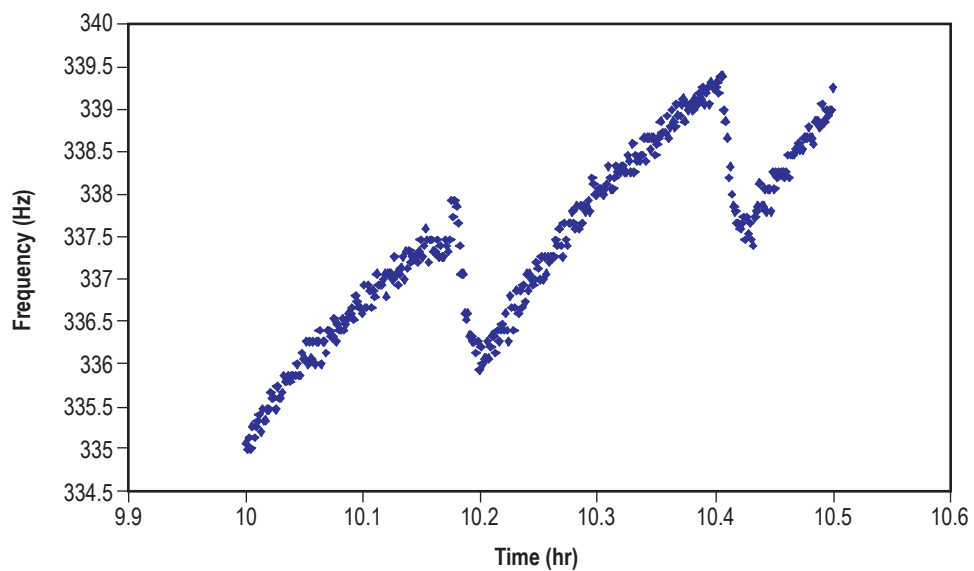


Figure 5. Time period from 10 to 10.5 hr of the frequency data presented in figure 4. The fluctuation in the frequency data primarily comes from fluctuations in the effusion cell heater temperature and a slight fluctuation in the deposition surface temperature. (Adipic acid, CQCM 2, January 24, 2004:  $-62^{\circ}\text{C}$  deposition surface,  $44^{\circ}\text{C}$  effusion cell—from 10 to 10.5 hr.)

## **7. DEPOSITION SURFACE REGENERATION**

The typical beat frequency observed for a clean QCM crystal pair will fall between 500 and 2,000 Hz. Condensation of material on the QCM crystal will cause a linear increase in the beat frequency until the deposition of material on the crystal begins to degrade the measurement being made. As a guideline for the user, the capacity—a maximum frequency value or microbalance loading—of each QCM is typically provided by the vendor. The deposition surface can be regenerated by raising the temperature of the crystal pair, thereby thermally reversing the deposition process. At the end of the crystal cleaning process, some hysteresis, or residual mass, which will be observed as a slight increase in the baseline frequency when the crystal is cooled, may remain on the crystal.

Not all QCMs measure the deposition process as an increase in the beat frequency. Some QCMs measure the increase in deposited material as a decrease in the measured beat frequency. Either approach to the deposition measurement process is acceptable as long as the frequency change observed is consistent with the proper functioning of the QCM.

## 8. CONCLUSION

QCMs are widely used by the aerospace community, as well as other communities interested in the rate at which material condenses on contamination-sensitive surfaces, to measure deposition rates. Simple methods can be employed to evaluate the relation between the sensor and the source of the condensing species. The in situ assessment of individual QCM performance can be made using physical constants such as the enthalpy of sublimation, which is available in chemical handbooks and other references.

This TM describes the calibration of QCMs installed in the Marshall Space Flight Center Outgassing Facility, which is operated by the Environmental Effects Group (ED31). The deposition rate measurements for a substance that sublimates as a single molecular species demonstrated that the facility provides quality outgassing measurements and that the microbalances can be monitored for both performance and sensitivity. The techniques presented in this TM are also recommended for the in situ calibration of QCMs installed in other vacuum chambers.

## APPENDIX A—CALCULATION OF THE CLAUSING FACTOR FOR AN ORIFICE OR SHORT TUBE

The Clausing factor,  $K$ , is a unitless number that describes the conductance of material through short tubes or the orifice of an effusion cell. This factor was developed by Clausing, and a good description of the factor and the theoretical background for the calculation can be found in Dushman:<sup>1</sup>

$$K=1/[1+(3/8)(L/A)] \quad , \quad (2)$$

where  $L$  is the orifice length and  $A$  is the orifice radius.

## APPENDIX B—CALCULATION OF THE DWELL TIME OF CONTAMINANTS ON SURFACES

Surface dwell time,  $\tau$  (s), for a contaminant can be calculated if the vibration frequency of the species, the activation energy of the species, and the temperature of the surface are known:

$$\tau = \tau_o \exp(Ea/RT) \quad , \quad (3)$$

where

$\tau_o$  is the vibration frequency (vibrations/s)

$Ea$  is the activation energy (cal/mole)

$R$  is the gas constant (1.9872 cal/K/mole)

$T$  is the surface temperature (K).

The activation energy and characteristic vibration frequency of a molecule or atom for a species can be obtained from publications, chemical handbooks and databases, or measurements made in the laboratory.<sup>8</sup>

## **APPENDIX C—NOTES ON USING THE VAN'T HOFFT RELATION TO CALCULATE THE ENTHALPY OF SUBLIMATION**

Screening the measured frequency data using linear regression can quickly eliminate poor quality data and help guide the measurement process before attempting to calculate the enthalpy of sublimation. A range of acceptable error in the calculated enthalpy of sublimation; e.g.,  $\pm 10$  percent, should be identified as providing measurements with the accuracy required for the application that the measurements are intended to support.

The efficiency of the deposition process increases as the deposition surface temperature becomes increasingly colder than the source temperature. The data collected for the hottest source temperature and not eliminated by the linear regression screening process would be a good candidate to select as one of the two data sets needed for the enthalpy of sublimation calculation. This data set can be considered to be an informal reference point for the exercise, against which the data collected at other source temperatures can be evaluated.

The apparent error or disagreement between the calculated enthalpy of sublimation and the published value can become very large when using data collected at two very similar source temperatures, typically a difference of  $<5$  °C. Some of the error is from instabilities in the sample heater, instabilities in the temperature of the deposition surface, and the fill cycles of the cryogenic surfaces. Repeating the calculation and substituting a deposition rate measured at a slightly higher or lower source temperature than the previously selected, informal reference may reduce the error.

Once the enthalpy of sublimation has been calculated for a range of source temperatures, the agreement or disagreement in the calculated values can be used to evaluate the performance of the microbalance.



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13. ABSTRACT (Maximum 200 words)  Quartz crystal microbalances (QCMs) are commonly used to measure the rate of deposition of molecular species on a surface. The measurement is often used to select materials with a low outgassing rate for applications where the material has a line of sight to a contamination-sensitive surface. A quantitative, in situ calibration of the balance, or balances, using a pure material for which the enthalpy of sublimation is known, is described in this Technical Memorandum. Supporting calculations for surface dwell times of deposited materials and the effusion cell Clausing factor are presented along with examples of multiple QCM measurements of outgassing from a common source.				
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