Comparison of Fourier Transform Infrared (FTIR) and Quadrupole Mass Spectroscopy (QMS) Methods for Determining POU Abatement System Effluent Flow

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Abstract: This report documents comparison testing of quadrupole mass spectroscopy (QMS) and Fourier transform infrared (FTIR) spectroscopy for determining the volumetric flow of point-of-use (POU) abatement system effluent. The EPA Protocol for Measuring Destruction or Removal Efficiency (DRE) of Fluorinated Greenhouse Gas Abatement Equipment in Electronics Manufacturing Version 1 and the International SEMATECH Manufacturing Initiative’s 2009 Guideline for Environmental Characterization of Semiconductor Process Equipment–Revision 2 were used in this testing. The POU abatement system effluent volumetric flow rates as determined by FTIR and QMS were consistent with one another. The FTIR monitoring approach to determine POU abatement system effluent volumetric flow in ISMI’s guideline document provides a suitable alternative to QMS measurements.

Keywords: Flow Rates, Fourier Transform Infrared Spectroscopy, Government Regulations, Point of Use Abatement, Procedures, Quadrupole Mass Spectroscopy

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1 EXECUTIVE SUMMARY

Two methodologies for determining the total volumetric flow (TVF) of various point-of-use (POU) abatement system effluents were studied using three systems in a wafer fab during normal operation. Two of the POU abatement systems were fuel-based thermal oxidation/wet scrubbers (burn/wet); one was an electric heat oxidation/wet scrubber (electric heat/wet). Each of the two methodologies used a tracer release approach to determine the TVF; the main differences were the injection location and analytical instrumentation. The two approaches to determine TVF were as follows:

- Injection of precise volume flow rates of sulfur hexafluoride (SF₆) at the POU abatement system effluent while taking real-time measurements of SF₆ concentrations downstream of the injection point using Fourier transform infrared (FTIR) spectroscopy.
- Injection of precise volume flow rates of krypton (Kr) into the inlet of the POU abatement system while taking real-time measurements of Kr concentration at the POU abatement system effluent using quadrupole mass spectroscopy (QMS).

Determining TVF by QMS was performed according to the Environmental Protection Agency (EPA) Protocol for Measuring Destruction or Removal Efficiency (DRE) of Fluorinated Greenhouse Gas Abatement Equipment in Electronics Manufacturing, Version 1, March 2010 (EPA Protocol) [1]. Determining TVF by FTIR followed the International SEMATECH Manufacturing Initiative’s (ISMI) 2009 Guideline for Environmental Characterization of Semiconductor Process Equipment – Revision 2 (2009 ISMI Guideline) [2]. A test plan (Appendix A) was prepared outlining the testing objectives and submitted to the EPA before the onset of the comparison study.

Mass flow controllers (MFCs) and volumetric flow meters (VFMs) were used to deliver steady and precise flows of each tracer gas. Since neat Kr and SF₆ were the tracer gases used, the MFCs (calibrated for nitrogen) were used to deliver a constant gas flow only. The tracer gas injection flow was determined from a DryCal DC-Lite primary flow meter; a National Institute of Standards and Technology (NIST)-traceable primary standard was used in which a float in the devices’ flow cell was timed between two sensors. (See Appendix B for calibration certifications for all the MFCs and volumetric flow meters).

1.1 POU Abatement System Effluent Flow Determination

The TVF determined at each POU abatement system effluent as calculated according to EPA Protocol equations is summarized in Table 1. The effluent flows were determined by FTIR concentration measurements (120 total data points) of a SF₆ tracer. The TVF for each system’s effluent was determined in triplicate by independently injecting three different controlled flow rates of SF₆ tracer gas (40 data points used for each injection flow rate). Determining the TVF by QMS involved injecting a Kr tracer at four precisely determined flow rates into the abatement system influent while performing QMS monitoring (40 data points used for each injection flow rate) at the effluent. Note that the effluent sampling location was the same for both SF₆ tracer gas and Kr tracer gas studies.

Table 1 gives all the calculated flows in liters per minute (lpm). As is evident from the comparison of flows, the TVFs determined by each methodology were consistent. However, both
the relative error and standard deviation of the flow results was, on average, six times better when using the FTIR approach.

1.2 Summary of Results and Conclusions

- The POU abatement system effluent flow rates as determined by FTIR and QMS were remarkably consistent with one another (9.37% relative percent difference [RPD] at burn/wet abatement system #1, 0.600% RPD at burn/wet abatement system #2 and 1.52% RPD at electric heat/wet abatement system #1).

- The determinations of effluent flow rates were stable and repeatable when determined by tracer release methods (Table 1).

- The tracer release/FTIR monitoring approach in the 2009 ISMI Guideline to determine POU abatement effluent flows resulted in a standard deviation and relative error that was, on average, 5.4 and 5.6 times better than the QMS results, respectively.

- The tracer release/FTIR monitoring approach in the 2009 ISMI Guideline to determine POU abatement effluent flow provided a suitable alternative to performing mass spectroscopy measurements. However, it is imperative to demonstrate that the injected tracer gas is well mixed at the extraction location. This can be done with a simple multipoint stratification test.

1.3 Recommendation

The 2009 ISMI Guideline tracer release/FTIR monitoring approach should be included in the EPA Protocol as an acceptable method for determining POU abatement system effluent flow. However, a concentration stratification should be determined at various points (i.e., 25%, 50%, and 75%) across the effluent duct to ensure homogeneity between the tracer and native effluent.

2 INTRODUCTION

To minimize climate change, new and more stringent ESH initiatives and greenhouse gas (GHG) reporting rules for the electronics industry have been issued. Several current semiconductor processes that are suspected of emitting significant quantities of high global warming potential (GWP) gases have had POU abatement systems installed to mitigate ultimate GHG emissions. Very little quantitative information is available about these emissions or the subsequent destruction of some of these gases through POU abatement systems. Because many of the gases, especially those with high GWPs, are under increased scrutiny by regulatory agencies worldwide, the U.S. Environmental Protection Agency published a protocol for the destruction or removal efficiency (DRE) of POU abatement systems. An integral part of this protocol is the measurement of dilution across POU abatement systems, for which accurate determinations of the effluent flow rate are paramount. Since many POU abatement systems incorporate combustion/oxidation gases and purge gases, the process gases entering the abatement system become diluted. According to the EPA Protocol, both POU abatement system influent and effluent flows must be measured. Historically, the small diameter ducts of these systems, the many bends and flow disturbances associated with the ducts, and the general inaccessibility
<table>
<thead>
<tr>
<th>Location</th>
<th>Methodology</th>
<th>Tracer Injection Flow Rates</th>
<th>40-Point Avg. Measured Conc. (ppm)</th>
<th>Calculated Flow (lpm)</th>
<th>Std. Dev. of Calc. Flow (lpm)</th>
<th>Total Volumetric Flow (lpm)</th>
<th>Std. Dev. of Total Flow (lpm)</th>
<th>Relative Error (%)</th>
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<td>QMS</td>
<td>200 — 727 170</td>
<td>727</td>
<td>170</td>
<td>679</td>
<td>746</td>
<td>1.65</td>
<td>0.44</td>
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<td>570</td>
<td>696</td>
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<td>2.25</td>
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<tr>
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<td>1100 — 64.2 31.0</td>
<td>719</td>
<td>16.6</td>
<td>1030 — 2398 436</td>
<td>1530 — 302 102</td>
<td>213</td>
<td>2.77</td>
</tr>
<tr>
<td></td>
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<td>801</td>
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<td>—</td>
<td>1530 — 801 302</td>
<td>213</td>
<td>0.32</td>
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<tr>
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<td>224 — 32.6 679</td>
<td>213</td>
<td>0.32</td>
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</tbody>
</table>

¹ An additional Kr tracer flow was injected, but the results were not consistent with the other three. The cause is unknown but believed to be due to an upset within the abatement or sampling systems.

Note: Shaded columns are TVF and error as calculated according to EPA Protocol equations.
to ports using standard flow equipment made accurate determinations of effluent flow by conventional methodologies (EPA Test Methods 1 and 2) problematic. Consequently, tracer release methods have been adopted, in which a known flow of an inert gas is injected and concentrations are measured of the injection location.

The EPA Protocol currently designates that QMS be used to monitor a released tracer through the POU abatement system to determine effluent flow and FTIR be used to measure abatement system influent flow as well as monitor DRE concentrations of process gases and byproducts. Both the semiconductor industry and the EPA stand to benefit from using a single analytical instrument to determine both concentration and flow. It is more cost-effective and many testing companies are available to collect FTIR-based measurements; very few have QMS capabilities. As described in the 2009 ISMI Guideline, FTIR determination of effluent flow also incorporates the use of a tracer gas release. The main difference in how tracer releases are used in the two methodologies is the FTIR tracer is injected into the effluent upstream of the sample extraction location whereas in the QMS approach it is injected into the POU abatement system inlet. The concern with using the approach incorporating effluent tracer release/FTIR measurements is two-fold:

- The effluent ducts make it difficult to obtain a properly mixed tracer/effluent gas stream at the sample extraction location
- Comparative data about the two approaches are lacking

ISMI contracted with URS Corporation in Austin, TX, to compare tracer release methods (QMS and FTIR) for determining POU abatement system effluent flow rates, which are subsequently used to determine dilution, mass emission rates and destruction or removal efficiencies.

The QMS and FTIR tracer approaches were performed at three different POU abatement systems: two fuel-based thermal oxidation/wet scrubbing (burn/wet) systems and a single electric heat oxidation/wet scrubbing (electric heat/wet) system. The testing was performed at an operational wafer fab on July 27 and 28, 2010. A krypton (Kr) tracer was selected for the QMS-based concentration measurements; sulfur hexafluoride (SF₆) was selected as the tracer for the FTIR-based concentration measurements. Both tracer gases are inert and not native to the gas matrices of the processes/POU abatement systems tested.

3 EXPERIMENTAL SETUP

The EPA Protocol [1], 2009 ISMI Guideline [2], and accepted sample test plan (Appendix A) were followed for this study.

3.1 QMS-based Approach

- On-site QMS calibrations for Kr were performed before testing each day. The first day’s calibration consisted of a zero and five different Kr concentrations that bracketed the Kr tracer concentrations observed during testing. The calibration for day 2 consisted of a zero and three different Kr concentrations. Each Kr calibration curve was fit to a linear regression; the $R^2$ values for each were > 0.99.
• Neat Kr was injected into the inlet of each POU abatement system at precisely known flow rates. Each Kr flow was metered using a MFC and measured using a DryCal primary standard volumetric flow meter. Four different flow rates were injected into each POU abatement system.

• Kr concentrations were determined by QMS measurements of Kr at the POU abatement system effluent. The final 40 concentration measurements for each injected Kr flow were used to determine effluent flow and subsequent statistics.

3.2 FTIR-based Approach

• Effluent emissions were initially analyzed to verify that SF₆ was not native to the process/POU abatement system gas matrix.

• A reference curve consisting of zero and three different references was generated and fit to a linear regression. All observed SF₆ concentrations fell within this calibration curve, and all FTIR references and sample spectra were collected at the same spectral parameters (resolution, apodization, zero filling, etc.). Any slight pressure and temperature differences between the sample and reference spectra were accounted for during sample quantification.

• A neat standard of SF₆ tracer gas was injected across the POU abatement system effluent duct using a multi-hole probe to maximize mixing.

• A sample extraction location was selected at a minimum of eight duct diameters downstream of the tracer injection location.

• A concentration stratification test was performed by FTIR monitoring of SF₆ at three different depths into the duct to ensure homogeneity of the tracer/effluent gas.

• Once homogeneity was verified, the extraction probe was placed into the center of the duct and three different SF₆ flow rates were injected into the effluent gas stream. FTIR spectra were collected every 3 sec; the final 40 spectra (per injected tracer flow rate) were quantified and used to determine effluent flows and subsequent statistics.

Note that the same POU abatement system effluent location was used for QMS and FTIR measurements.

3.3 Analytical Instrumentation

3.3.1 QMS

Traditionally, mass spectrometers have been used as analytic instruments under vacuum applications. Recent advancements in the technology coupled with the development of atmospheric samplers and closed ion sources has enabled atmospheric sampling by QMS. The higher pressure QMS (i.e., residual gas analyzer [RGA]) is smaller, more robust, and more portable than their laboratory predecessors. The RGA used for this testing was a Ferran Scientific Micropole Analyzer (MPA) Sensor System with a 0–100 atomic mass unit (AMU) range. It is mated to electronics produced by its parent company, HoribaSTEC, and is housed in a URS-built differentially pumped vacuum system with an atmosphere-to-analyzer chamber sample interface. This QMS allows sample gas, at or near atmospheric pressure, to leak into the QMS ionizer region under controlled rates through a precision orifice. The QMS can then scan over various AMU ranges or a selected few. The most sensitive Kr response was observed at
Since Kr was the only analyte of interest, AMU 83 was the only mass monitored. Since the QMS analyzer’s response factors over all the mass units depend on the ionizer’s chamber pressure, energy, etc., a careful calibration was performed on-site consistent with the configuration used for all sampling. Krypton calibration curves were generated each day by delivering various concentrations of Kr to the QMS sample interface. Various concentrations of Kr were delivered to the QMS sample interface and calibration curves generated. Figure 1 and Figure 2 correspond to the calibrations on 7/27/10 for the two POU burn/wet systems and 7/28/10 for the POU electric/wet system, respectively. The linear regression equations both resulted in R² values greater than 0.98 as required by the EPA Protocol and were used to calculate all Kr concentrations. Sample was continuously pulled through the extractive system and atmospheric chamber of the QMS at a rate of ~5 lpm, from which a small volume slip stream was pulled into the QMS ionization chamber and analyzed.

A Thermo-Nicolet Magna 550 FTIR bench was used for this testing. The FTIR was optically matched to a 0.1 m gas cell such that the IR beam propagated through the cell a single time. Anti-reflective zinc selenide windows transmitted the IR beam with minimal signal loss. A continuous flow of sample gas was delivered through the cell at ~5 lpm. As the IR beam passes through the sample, the molecules in the sample absorb some of its energy at particular frequencies. After exiting the cell, the beam is directed to a liquid-nitrogen cooled mercury/cadmium/telluride (MCT) detector, a photoconductive device that produces an electrical voltage proportional to the amount of infrared radiation that strikes it. The strength of the absorption at particular frequencies is a measure of the compounds’ concentrations. The total distance traveled by the IR beam inside the cell is the cell path length, an important parameter used in determining sample concentrations.

![Figure 1: QMS Kr Calibration Used for Effluent Flow Determinations of the POU Burn/Wet Systems](image-url)

\[
\text{ppm} = 3794.81X - 149.43 \\
R = 0.99936
\]

**Figure 1** QMS Kr Calibration Used for Effluent Flow Determinations of the POU Burn/Wet Systems
Figure 2  QMS Kr Calibration Used for Effluent Flow Determinations of the POU Electric Heat/Wet System

3.3.2  FTIR Spectrometer

The FTIR sample cell was maintained at room temperature (~23°C) throughout the testing. Instrumental resolution was set to 0.5 cm\(^{-1}\), and signal was averaged over 3-sec periods per data point as suggested in the EPA Protocol. A series of data points was collected at each tracer flow from which the final 40 datapoints were used to calculate the POU abatement system TVF and relative error using EPA Protocol equations.

Figure 3 displays the FTIR calibration curve for SF\(_6\) used for all the qualitative analysis during sampling. The reference curve consists of a zero and three different references that were generated before testing and fit to a linear regression. The co-efficients (also shown in Figure 3) generated from this calibration curve were used in subsequent SF\(_6\) quantitative analysis during sampling. All observed SF\(_6\) concentrations fell within the calibration curve limits, and all FTIR references and sample spectra were collected at the same spectral parameters (resolution, apodization, etc.). Furthermore, sample quantification accounted for the slight temperature and pressure differences between each sample and reference spectra.

A series of URS laboratory and on-site calibration and system checks was performed on the FTIRs and sampling system before testing to ensure data of known quality:

- **Infrared Detector Linearity Checks**

  For best results, the infrared detector must yield a linear response throughout a reasonable absorbance range and all the frequencies in a set of test spectra. Linearity was optimized before shipment and verified just before testing.
Spectral Background
A spectral background is essentially a blank spectrum in that it does not contain any of the target compounds in the sample. It was created by purging the cell with POU abatement system effluent gas, with the process and tracer injection idle, while collecting a spectrum. This spectrum is then used by the analytical software to ratio against each sample spectrum to produce an absorbance spectrum for quantitative analysis. New spectral backgrounds of the non-process type were generated before each of the three POU abatement systems was tested.

SF₆ Challenge
To ensure the accuracy of the FTIR concentration measurements, a one-time challenge was performed. A certified standard of SF₆ was directly injected into the FTIR analysis cell and quantified. The standard was certified at 21.9 ppm; a SF₆ concentration of 22.0 ppm was observed during the challenge. The consistency of the results demonstrates that the FTIR was accurately quantifying SF₆.
3.3.3  **POU Abatement Systems Monitored**

The comparative tests were performed at three different POU abatement systems. All had two stages of abatement. The first two systems tested had a first stage natural gas-fueled thermal oxidation system, followed by a second stage wet scrubbing section. The third system consisted of an electrically heated oxidation section followed by a wet scrubbing section.

3.3.4  **Sampling System and Extraction Locations**

Figure 4 illustrates the sampling system used in this study for both FTIR and QMS systems. Figure 5 to Figure 7 are photographs of the effluent ducts and sample locations for the three POU abatement systems. The QMS and FTIR measurements were not performed simultaneously since the same extraction location was required for both. Although the ductwork of each system varied, the extractive setup was diagrammatically identical: the extraction location was between eight and nine duct diameters downstream of the SF₆ injection location. Sample was extracted from each effluent by a probe positioned in the center of the duct. The sample then was pulled through unheated, ¼-inch perfluoroalkoxy copolymer (PFA) tubing at ~ 5 lpm using Venturi pumps. While the QMS differentially pumped a slipstream of less volume for analysis, the FTIR pulled the entire 5 lpm sample through its 0.1 m cell. A rotometer was attached to the FTIR cell to ensure adequate flow through the system during testing. After passing through the FTIR or QMS, the sample gas mixed with the Venturi pump motive gas before exiting the sampling system through 3/8-inch PFA tubing into facility exhaust.

![Figure 4 FTIR/QMS Sampling System Diagram](image-url)
Figure 5  Sample and Tracer Injection Location of POU Burn/Wet Abatement System #1

Figure 6  Sample and Tracer Injection Location of POU Burn/Wet Abatement System #2
3.3.5 Tracer Injection System

The injection system for the QMS-based approach, depicted in Figure 8, consisted of a neat Kr standard, a 0–5000 sccm MFC, ¼-inch Teflon tubing, and a volumetric flow meter (VFM). The injection system for the FTIR approach was identical except that SF$_6$ was used, the MFC had a 0–500 sccm range, and a ¾-inch multi-holed stainless steel probe was used to inject the tracer across the effluent duct. Using the MFC to stabilize flow through the calibrated VFM, precise amounts of SF$_6$ were injected through the multi-holed probe across the effluent duct for the FTIR measurements; similarly, precise amounts of Kr were injected into the POU abatement system inlet for the QMS measurements. The SF$_6$ injection port was located ~1 foot downstream of the POU abatement system exit but well upstream of the effluent sample port.
4 EXPERIMENTAL RESULTS AND DISCUSSION

QMS monitored the POU abatement system effluent gas for a Kr tracer that was injected into the POU abatement system influent. Krypton is inert and passes through the POU abatement system unabated. Monitoring was performed in strict accordance with the EPA Protocol. FTIR monitored the effluent downstream of the controlled SF$_6$ tracer release. The sample extraction point was a minimum of eight duct diameters downstream of the tracer injection. Monitoring was performed in strict accordance with the 2009 ISMI Guideline [2]. Table 1 compares the results of these two methods.

Tracer release-based flow measurements were performed by injecting a controlled flow of inert gas, SF$_6$ and Kr, at the POU abatement system effluent and influent, respectively. By measuring the diluted tracer concentrations, the effluent flows can be determined using the following equation from the EPA Protocol (Equation 1) [1],

$$F = \frac{S_f}{C_{ef} \times 10^{-6}}$$  \hspace{1cm} Eq. [1]

where:  
$F =$ TVF based on a single concentration data point  
$S_f =$ Tracer gas flow into the sample duct  
$C_{ef} =$ Measured concentration of the tracer gas

---

**Figure 8** Flow Tracer Injection Dynamics at the POU Abatement System Effluent
This technique is described in both the EPA Protocol [1] and the 2009 ISMI Guideline [2]. The only difference is that the 2009 ISMI Guideline also allows this method to be used to measure POU abatement system effluent flows (Section B.1.5) by injecting the tracer into the POU effluent once it has been verified that the effluent has no native concentration of the tracer. Conversely, the EPA Protocol states that to determine effluent flow the tracer must be injected into the POU abatement system inlet and pass through it with 0% or less than 5% DRE (Table 2 and Section 2.2.3 of the EPA Protocol).

4.1 Statistical Treatment of POU Abatement System Effluent Flow Rates

Equation 1 applies to individual concentration measurements. The SF₆ and Kr concentrations reported in Table 1 (column 5) are simple averages using the last 40 data points corresponding to each injected tracer flow rate (columns 3 and 4). However, the final estimated flow rates listed in column 6 were calculated by averaging the flow rates predicted by each concentration data point using the following equation (Equation 2 of the EPA Protocol), not by plugging the average concentration into Eq. [1],

$$\overline{F}_m = \sum_{i=1}^{n} \frac{F_i}{n}$$  
Eq. [2]

Where:  
- $F_i$ = The effluent flow rate calculated for the $i^{th}$ data point using Eq. [1].
- $\overline{F}_m$ = Simple average of TVF for the $m^{th}$ injected tracer gas flow rate.
- $n = \text{Number of concentration measurements taken while at the } m^{th} \text{ constant tracer gas flow rate.}$

Once the average effluent flow was determined using Eq. [2], a standard deviation for the $m^{th}$ injected tracer gas flow rate was determined using Eq. [3] (Equation 3 of the EPA Protocol). This is a measure of the stability and precision of the sampling system (column 7).

$$\sigma_{\overline{F}_m} = \sqrt{\frac{\sum_{i=1}^{n} (F_i - \overline{F}_m)^2}{n}}$$  
Eq. [3]

where:  
- $\sigma_{\overline{F}_m}$ = Standard deviation of $\overline{F}_m$.

To arrive at a single calculated effluent flow rate, each of the individual flows ($\overline{F}_m$) was used in Eq. [4] (Equation 4 of the EPA Protocol). This equation calculates a variance weighted average of average effluent flow rates calculated using Eq. [2]. Results are presented as the total volumetric flow in the 8th column of Table 1.

$$\hat{F}_g = \frac{\sum_{m=1}^{M} \overline{F}_m}{\sum_{m=1}^{M} \frac{1}{\sigma_{\overline{F}_m}^2}}$$  
Eq. [4]

Where:  
- $\hat{F}_g$ = Variance weighted average TVF for the standard tracer gas (g).
- $M = \text{Number of flow rates used per standard tracer gas (g).}$
Finally, for each effluent flow rate calculated using Eq. [4], a standard error of the mean was calculated using Eq. [5] (Equation 5 of the EPA Protocol). To both simplify and provide the most conservative figure, \( n \) was taken to be constant and equal to the lowest \( n \) encountered in Eq. [2]. Again, this was strictly a measure of precision; it relates only to the mean and that \( \sigma_{F_e} \) will often be lower than any of the \( \sigma_{F_m} \).

\[
\sigma_{F_e} = \left( \sum_{n=\text{const}} \frac{1}{n} \sigma_{F_m}^2 \right)^{1/2} \equiv \frac{1}{n} \times \sqrt{\frac{1}{n} \times \left( \sum_{n=\text{const}} \frac{1}{n} \sigma_{F_m}^2 \right)^{1/2}} \tag{5}
\]

Where: \( \sigma_{F_e} = \) The standard error (uncertainty) of the mean (average) TVF (Table 1, column 9).

Note that Eq. [2]–Eq. [5] were used identically on both the QMS and FTIR data independently. At no time were QMS data used with FTIR concentration data to determine TVF. Relative errors reported for the effluent flow in Table 1, column 10 are based on a 95% confidence interval.

4.2 POU Abatement System Effluent Flow Determination by QMS Method

The EPA Protocol (Section 2.2.6) and 2009 ISMI Guideline (Section A.1) were strictly followed for the QMS-based flow determinations. A neat gas standard of Kr was injected into the POU abatement system inlet at precisely known volumes. During Kr injections, QMS monitored Kr at the effluent sampling location. Krypton was not native to the system; it was assumed that injecting at the POU abatement system inlet would create a well mixed sample stream at the POU abatement system effluent. Four different tracer flows were injected at three different POU abatement systems. An outline of the tracer injection and sample extraction system is diagrammed in Figure 8. The TVFs, corresponding relative errors, and standard deviations from the QMS Method tests are shown in Table 1.

The QMS data for each set of injected Kr flows consisted of the final 40 Kr concentration measurements, which were used to calculate the effluent flow for each injected tracer flow. Note that one of the four injected tracer flows at the burn/wet abatement system #2 resulted in a calculated effluent flow that was inconsistent with the other three and was omitted from the TVF determination. The cause of this inconsistency is unknown. Since the EPA Protocol states a criterion of three tracer injections per effluent flow determination, the TVF of the burn/wet abatement system #2 still complies with the protocol.

Another minor issue with the QMS sampling occurred in the calibration curve associated with the POU electric heat/wet abatement system #1 tests. The highest Kr concentration observed during the testing was outside the range of the QMS calibration curve. The average Kr concentration during the high flow tracer injection was ~30% greater than the highest concentration of the calibration curve for that day. The calibration curve is fit to a linear regression and was extrapolated to include this point. TVF and relative error calculations for this POU abatement system also included this data point. If this were removed, the TVF for the POU electric heat/wet abatement system #1 would not change significantly (0.47 lpm less or 0.1% RPD).

The compilation of the effluent flows determined from each tracer injection was then used to calculate the TVF of each of the three POU abatement systems. Figure 9 through Figure 11 plot the total QMS data sets for each injected tracer flow at each POU abatement system.
Figure 9  QMS Kr Measurements at POU Burn/Wet Abatement System #1

Figure 10  QMS Kr Measurements at POU Burn/Wet Abatement System #2
4.3 POU Abatement System Effluent Flow Determination by FTIR Method

A controlled SF$_6$ tracer release was used simultaneously with the FTIR concentration measurements to determine flow at each POU abatement system effluent. Sections A.2 and B.1.5 of the 2009 ISMI Guideline were followed without deviation as they pertained to the testing. The SF$_6$ tracer was not native to any of the processes/POU abatement systems used for this study. Three different flow rates of SF$_6$ were injected into each system effluent ~1 foot above the POU abatement system housing. The injection system is depicted in Figure 8. For each POU abatement system, the sample gas was extracted from a port between eight and nine duct diameters downstream from the SF$_6$ injection location. Before TVF measurements, a series of stratification tests was performed by monitoring the SF$_6$ at various depths (25%, 50%, and 75%) into the duct to demonstrate homogeneity of the tracer/effluent gas. The results from the stratification tests, summarized in Table 2, are consistent in concentration at each point across the ducts, demonstrating homogeneity of the effluent and SF$_6$ gases for each system.

Table 2 Stratification Traverse Results

<table>
<thead>
<tr>
<th>POU Abatement System</th>
<th>Traverse Position % into Duct</th>
<th>SF$_6$ Conc. (ppm)</th>
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</thead>
<tbody>
<tr>
<td>Burn/Wet #1</td>
<td>25</td>
<td>58.7</td>
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<tr>
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<td>50</td>
<td>60.8</td>
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<tr>
<td></td>
<td>75</td>
<td>58.3</td>
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<tr>
<td>Burn/Wet #2</td>
<td>25</td>
<td>57.5</td>
</tr>
<tr>
<td></td>
<td>50</td>
<td>57.1</td>
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<tr>
<td></td>
<td>75</td>
<td>61.2</td>
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<tr>
<td>Electric/Wet #1</td>
<td>25</td>
<td>108.9</td>
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<tr>
<td></td>
<td>50</td>
<td>107.5</td>
</tr>
<tr>
<td></td>
<td>75</td>
<td>109.3</td>
</tr>
</tbody>
</table>
The low standard deviations in Table 1 show that the SF₆ concentrations were extremely consistent throughout monitoring. Figure 12 through Figure 14 plot the FTIR SF₆ concentration results for each of the injected flows into each POU abatement system effluent.

The SF₆ flow was set and controlled by the MFC, measured by the VFM, and injected across the POU abatement system effluent flow using a multi-point probe. All reported tracer gas flows in Table 1 were taken from the VFM, not the MFC, as the MFC was calibrated for nitrogen and was not as precise as the VFM. The repeatability of these experiments demonstrates that the FTIR method can produce accurate results and is applicable for measuring flows such as these.

![Figure 12](image-url) FTIR SF₆ Measurements at POU Burn/Wet Abatement System #1
Figure 13  FTIR SF₆ Measurements at POU Burn/Wet Abatement System #2

Figure 14  FTIR SF₆ Measurements at POU Electric Heat/Wet Abatement System #1
4.4 Comparative Analysis of Tracer Release QMS and FTIR Methods

Table 1 compares measurements using both QMS and FTIR tracer approaches. Each methodology collected concentration data sets at specific tracer gas injection rates that were then used to calculate POU abatement system effluent flow rates. For each of the three different POU abatement systems studied, the TVF determined by QMS and FTIR agreed favorably. The relative percent difference (RPD) in effluent flows determined by each methodology was 9.37% at burn/wet abatement system #1, 0.600% at burn/wet abatement system #2, and 1.52% at electric heat/wet abatement system #1. The remarkable consistency between the two methodologies suggests that either approach is acceptable for determining POU abatement system effluent flow. However, when analyzing the data scatter and error of each methodology, FTIR clearly provided much more stable and precise results. Comparison of the relative error (a report requirement of the EPA Protocol) shows a 5.1, 8.6, and 3.1 times improvement in FTIR over QMS data for the burn/wet abatement system #1, burn/wet abatement system #2, and electric heat/wet abatement system #1, respectively. In short, the relative error as calculated according to the EPA Protocol requirements is on average 5.6 times lower when using the FTIR-based approach. This is easily seen in Figure 15 for POU abatement system burn/wet #2; a plot of both the QMS and FTIR flows over the entirety of a 120-point test shows that although their TVFs agreed, the FTIR data had much less variability.

Figure 15 TVF Determinations of QMS and FTIR Data (POU Burn/Wet Abatement System #2)
5 SUMMARY OF RESULTS AND CONCLUSIONS

- The POU abatement system effluent flow rates as determined by FTIR and QMS were remarkably consistent with one another (9.37% RPD at burn/wet abatement system #1, 0.600% RPD at burn/wet abatement system #2, and 1.52% RPD at electric heat/wet abatement system #1).

- The determinations of POU abatement system effluent flow rates were stable and repeatable when determined using tracer release methods (Table 1).

- The 2009 ISMI Guideline tracer release/FTIR monitoring approach to determine effluent flows resulted in a standard deviation and relative error that was on average 5.4 and 5.6 times better than the QMS results, respectively.

- The 2009 ISMI Guideline tracer release/FTIR monitoring approach provided a suitable alternative to performing mass spectroscopy measurements. However, the injected tracer gas must be well mixed at the extraction location. A simple multipoint stratification test can verify this.

6 RECOMMENDATION

Results indicate that the 2009 ISMI Guideline tracer release/FTIR monitoring approach should be included in the EPA Protocol as an acceptable method of determining POU abatement system effluent flow. However, a concentration stratification determination must be performed at various points (i.e., 25%, 50%, and 75%) across the effluent duct to ensure homogeneity between the tracer and native effluent.

7 REFERENCES


Appendix A – ISMI POU Abatement Device Volumetric Flow Rate Methodology
Comparison Test Plan

A.1 Introduction
ISMI will be conducting comparison testing at a wafer fab between two tracer-based methodologies for determining POU abatement device total outlet volumetric flow rate:

- Using a non-reactive gas at POU abatement device outlet; and
- Quadrupole mass spectroscopy (QMS) and noble gas method (using krypton) through POU abatement device.

This comparison testing will provide data that is requested in the EPA's Protocol for Measuring Destruction or Removal Efficiency (DRE) of Fluorinated Greenhouse Gases Abatement Equipment in Electronics Manufacturing (Version 1) (EPA Protocol), Page 31: "While the Post-abatement Flow Tracer Method is not permitted in the Protocol, EPA is interested in learning more, through in-fab testing, about the validity of this method." The testing will provide technical data for EPA to support addition of the volumetric flow determination methodology of utilizing non-reactive gas at POU abatement device outlet to next version of EPA protocol. The dates for this testing are Tuesday, July 27, 2010 and Wednesday, July 28, 2010, and will occur in the Phoenix, Arizona area.

A.2 Testing Scope
The testing will directly compare two methodologies for determining POU abatement device outlet volumetric flow rate using semiconductor production processes and POU abatement devices located at a wafer fab. The two methodologies for determining outlet volumetric flow rate are:

- Injecting non-reactive gas (SF₆) at POU abatement device outlet while utilizing real-time concentration FTIR measurements downstream of the injection location; and
- Injecting krypton through POU abatement device while monitoring the POU abatement device outlet for krypton using QMS.

This testing will be conducted in accordance with pertinent sections of the 2009 Guideline for Environmental Characterization of Semiconductor Equipment (ISMI Guideline) and EPA's Protocol for Measuring Destruction or Removal Efficiency (DRE) of Fluorinated Greenhouse Gases Abatement Equipment in Electronics Manufacturing (Version 1) (EPA Protocol). Sherer Consulting Services, Inc. (SCS) will be the testing coordinator, working directly with ISMI, wafer fab, and testing company URS Corporation. EPA and their representatives are welcome and encouraged to attend this testing. Safety and additional subfab training will be provided for all personnel involved, as required by the wafer fab company.

SCS will work with wafer fab personnel to ensure that site is prepared for testing. URS Corporation personnel will review testing locations when they arrive Monday morning (see later schedule).
It is planned at this time that the following three processes and corresponding POU abatement devices will be selected for the testing.

- CVD process with POU thermal oxidation (using fuel)/wet scrubbing system (one inlet).
- CVD process with POU thermal oxidation (using fuel)/wet scrubbing system (two inlets).
- CVD process with POU electric heat oxidation/wet scrubbing system (two inlets).

Two planned POU abatement devices will have two process inlets and one planned POU abatement device will have one process inlet. Each inlet will have volumetric flow rate determination conducted. The injection of krypton will be done into one of the two inlets for each two-inlet POU abatement device. This should allow for good mixing in POU abatement device and subsequent krypton measurement at outlet of POU abatement device. The same POU abatement device outlet location will be utilized for all QMS and FTIR measurements for volumetric flow rate determination; the QMS and FTIR measurements will not be performed simultaneously as each methodology should be conducted separately.

Any deviation from methodologies stated in EPA Protocol and ISMI Guideline will be approved by SCS and URS Corporation before use. The testing will be conducted in series for each process/POU abatement device tandem as follows:

1. Determine each POU abatement device total inlet volumetric flow rate using a chamber clean gas with the tool plasma off.
2. QMS analysis of krypton through POU abatement device.
3. FTIR analysis of SF$_6$ tracer gas at POU abatement device outlet.

Using SF$_6$ tracer gas at POU abatement device outlet methodology is presented below from ISMI Guideline, with an addition of the upstream diameter from any exhaust lateral connection. This method will accurately flow SF$_6$ into POU abatement device outlet at known volumetric flow rates using mass flow meter while measuring the SF$_6$ concentration downstream (by FTIR). This methodology requires the following criteria to be utilized:

- The POU abatement device outlet must be sampled to ensure that the non-reactive gas is not present.
- SF$_6$ must be injected across the POU abatement device outlet duct. This can be accomplished by using an injection probe with equally spaced holes.
- The location selected for downstream non-reactive gas analysis must be at least eight duct diameters downstream from SF$_6$ tracer gas injection location. In addition, this location will be at least two duct diameters upstream from an exhaust lateral connection.
- The location selected for downstream SF$_6$ tracer gas measurements must also be used for QMS and noble gas method measurements.
- It is recommended that the downstream tracer gas analysis location duct be traversed with the sampling probe at a few locations to ensure the tracer gas is well mixed. For example, if the duct is 4 inches, the duct can be traversed in 1-inch increments (25%, 50% and 75%) to prove that the concentration across the duct is uniform.
A.3 Proposed Schedule

Monday: URS Corporation to arrive on-site for safety orientation, review testing locations, and set-up equipment at first testing location.

Tuesday: EPA and their representatives arrive on-site by 7:30 AM for safety orientation. POU abatement flow rate comparison testing at first POU abatement device is scheduled to begin at 9:00 AM, and second POU abatement device at 2:00 PM. Move to third POU abatement device.

Wednesday: POU abatement volumetric flow rate comparison testing at third POU abatement device at 9:00 AM; testing completed by noon.
Appendix B – Equipment Certifications

Brooks Instrument

Manufacturing Router for Elastomer Sealed Devices (Excluding 5853/63 & MF53/63)

It is the responsibility of everyone working on this MFC to comply with following the process exactly and to put your clock number (No initials) and date in the spaces provided. If a required step doesn’t have a clock number and date, don’t accept the MFC. If a step is not required place a “NFR” in the clock number space. When performing multiple steps, it is permissible to draw a line from the prior step checked to the last step performed. Any deviations from this process must be approved and signed off by Manufacturing Engineering.

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<th>Clock No.</th>
<th>Date</th>
<th>Process Step</th>
<th>O-ring type</th>
<th>O-ring type</th>
<th>O-ring type</th>
<th>Seal Type</th>
<th>Restrictor Size</th>
<th>O-ring type</th>
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<td>Torque valve screws to J-834-Z-905</td>
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<td>Install &amp; tighten restrictor. (Inspect restrictor cavity for debris)</td>
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<td>Install &amp; torque and block part J-834-Z-905 (site 1 bodies only)</td>
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<td>Pressure test per J-834-C-024 &amp; PLT-014 (Except 5866). Record result in Pressure Test Log and place check mark on sensor label.</td>
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<td>Assemble coil and tighten nut.</td>
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<td>Assemble Printed Circuit Board(s) and plug in coil</td>
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<td>Helium leak test per ASY-216. Place check mark on Sensor label.</td>
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<td>Calibration or Function Check (for uncalibrated devices) per CAL-005</td>
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<td>Set Final Jumpers Settings Per Order</td>
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<td>For RS-232 or RS-489 communications board, verify baud rate are set correctly in software and hardware.</td>
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<td>Perform functional check for SLA series SMART-II devices with HART functionality as per ASY-286.</td>
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<td>Response time adjustment per CAL-001</td>
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<td>Torque valve screws J-834-Z-905 (Only required for N.O. or if N.C. was opened)</td>
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<td>Pressure test per PLT-021 (for PCs) or J-834-C-024 &amp; PLT-014 (Only required for N.O., if N.C. was opened)</td>
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<td>Helium leak test per ASY-216 (For 5866, SLA5810/20 &amp; SLAMFI10/20 only required if device was opened).</td>
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<td>QC Check (If Required)</td>
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<td>Verify jumper settings per order</td>
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<td>Push digital valve nut a little using hand nut tool or spinner wrench on digital devices only.</td>
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<td>Button Up (Check Proper Labeling) Per ASY-221</td>
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<td>Check S V Reference (Ground Only) per ASY-184</td>
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<td>Perform Digital Communications and Valve Function Check per ASY-256 (DeviceNet) or ASY-261 (Profi Bus)</td>
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<td>Perform RT function Check Per CAL-009 (Delta RTs Only).</td>
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<td>Perform power cycle test that device turns on.</td>
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If failure occurs, list step number and repair work done on back of form.

File this router with the calibration data sheet and copy of the customer order in the customer order file.

From ASY703-01 (Rev A) 30/7/2006
LAMINAR TECHNOLOGIES, INC.
CERTIFICATE OF CALIBRATION

**DUT STATISTICS**

| Manufacturer: BROOKS | RECOMMENDED RE-CERTIFICATION DATE: 6/14/2008 |
| Customer P/N: | Fitting In: SW |
| Model: 5650E | Connector: 15PIN |
| Serial Number: 9709-A-95151/1 | Fitting Out: SW |
| WIP #: 75228 | Response: F |
| Range (SCCM): 500 SCCM | Jet: 32 |
| Process Gas: N2 | Auto Zero: N |
| Calibration Gas: N2 | Sensor: NA |
| Mounting: H | A-Shutoff: N |
| Press.In: 25.00 | Seals: V |
| Press.Out: A | Valve: C |
| DUT Output | DUT Error |

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<th>DUT Error (sccm)</th>
<th>DUT Error (% F.S.)</th>
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<td>0</td>
<td>0.00</td>
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<td>375</td>
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**Calibration Test Points**

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<td>0.50</td>
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<td>0.00</td>
<td>0.14</td>
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<td>-0.36</td>
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<tr>
<td>-1.00</td>
<td>-0.36</td>
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</table>

**K-Factor:** 1

**Cal Date:** 6/14/2007

**Operator:** AC

**Ambient Temperature:** 21.22 °C

**Reference Temperature:** °C

**Ambient Pressure:** 762.3 mmHg

**Reference Pressure:** 760 mmHg

*All Calibrations are performed in a controlled class 100 environment by qualified personnel using instrumentation and methods which guarantee that specifications claimed are reliable. The above data was measured on a primary standard traceable to the National Institute of Standards and Technology and conform to ANSI/NCSL Z540-1 which supercedes MILSTD 45692A. Helium leak detection performed to at least 1x10⁻⁶ or better.*

www.laminartech.com 1-800-229-4700 Document Control #LTI107

ISMI Technology Transfer #10095115A-ENG
Calibration Certificate

Certificate No. 46904
Product DryCat DC-Lite Medium High
Serial No. 4989
Cal. Date 4/16/2010
Sold to: URS Corporation - Austin
9400 Amberglen Blvd.
Austin, TX 78729
USA

All calibrations are performed in accordance with ISO 17025 at Bios International Corporation, 10 Park Place, Butler, NJ 07405, 800.463.4977, an ISO 17025:2005 accredited laboratory through NVLAP. This report shall not be reproduced except in full without the written approval of the laboratory. Results only relate to the items calibrated. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the Federal Government.

All units tested in accordance with Bios International Corporation test number PR95-2 or PR91-10 using high-purity bottled nitrogen or dry filtered laboratory air.

As Received Calibration Data

<table>
<thead>
<tr>
<th>Technician</th>
<th>Lab. Pressure</th>
<th>Lab. Temperature</th>
</tr>
</thead>
<tbody>
<tr>
<td>David Strathearn</td>
<td>763 mmHg</td>
<td>22.7 °C</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Instrument Reading</th>
<th>Lab Standard Reading</th>
<th>Deviation</th>
<th>Allowable Deviation</th>
<th>As Received</th>
</tr>
</thead>
<tbody>
<tr>
<td>269.8ccm</td>
<td>269.15ccm</td>
<td>0.32%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>504.5ccm</td>
<td>504.85ccm</td>
<td>-0.02%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>1700.0ccm</td>
<td>1700.5ccm</td>
<td>-0.06%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
</tbody>
</table>

Bios International Standards Used

<table>
<thead>
<tr>
<th>Description</th>
<th>Standard Serial Number</th>
<th>Calibration Date</th>
<th>Calibration Due Date</th>
</tr>
</thead>
</table>

Bios International • 10 Park Place Butler, NJ 07405 • 800.463.4977 • www.biosint.com

Page 1 of 2
As Shipped Calibration Data

Certificate No. 46004
Technician David Strathearn
Lab. Pressure 760 mmHg
Lab. Temperature 22.7°C

<table>
<thead>
<tr>
<th>Instrument Reading</th>
<th>Lab Standard Reading</th>
<th>Deviation</th>
<th>Allowable Deviation</th>
<th>As Shipped</th>
</tr>
</thead>
<tbody>
<tr>
<td>208.8ccm</td>
<td>200.5ccm</td>
<td>0.15%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>5018ccm</td>
<td>5145.9ccm</td>
<td>0.64%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>17020ccm</td>
<td>17035.5ccm</td>
<td>-0.09%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
</tbody>
</table>

Bios International Standards Used

<table>
<thead>
<tr>
<th>Description</th>
<th>Standard Serial Number</th>
<th>Calibration Date</th>
<th>Calibration Due Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>ML_500_10</td>
<td>113778</td>
<td>1/13/2009</td>
<td>11/13/2010</td>
</tr>
<tr>
<td>ML_500_44</td>
<td>110104</td>
<td>5/20/2009</td>
<td>5/20/2010</td>
</tr>
</tbody>
</table>

Calibration Notes

Bios is an ISO 17025-accredited metrology laboratory. Each Bios primary gas flow standard is dynamically verified by comparing it to one of our laboratory standards, which is a Proven DryCal® Technology volumetric piston prover of much higher accuracy (±0.2% or better) but of similar operating principles. For this purpose, a flow generator of ±0.03% stability is used. Our laboratory standards are qualified by direct remeasurement of their dimensions (diameter, length and time) using NIST-traceable precision gauges and instruments, such as depth micrometers and laser micrometers. NIST numbers for these gauges and instruments are available upon request. Rigorous analyses of our laboratory standards' uncertainties have been performed, in accordance with The Guide to the Expression of Uncertainty in Measurement (the GUM), assuring their traceable accuracy.

Technician Notes:

Harvey Fadden, President and Chief Metrologist
# Calibration Certificate

**Certificate No.**  46005  
**Product**  DryCal DC-Lite Low  
**Serial No.**  4914  
**Cal. Date**  4/15/2010

**Sold to:**  URS Corporation - Austin  
**Address:**  9400 Amberglen Blvd.  
**City, State:**  Austin, TX 78729  
**Country:**  USA

All calibrations are performed in accordance with ISO 17025 at Bios International Corporation, 10 Park Place, Butler, NJ, 07405, 800-665-4977, an ISO 17025:2005 - accredited laboratory through NVLAP. This report shall not be reproduced except in full without the written approval of the laboratory. Results only relate to the items calibrated. This report must not be used to claim product certification, approval, or endorsement by NVLAP, NIST, or any agency of the Federal Government.

All units tested in accordance with Bios International Corporation test number PR01-2 or PR01-10 using high purity bottled nitrogen or dry filtered laboratory air.

## As Received Calibration Data

<table>
<thead>
<tr>
<th>Instrument Reading</th>
<th>Lab Standard Reading</th>
<th>Deviation</th>
<th>Allowable Deviation</th>
<th>As Received</th>
</tr>
</thead>
<tbody>
<tr>
<td>30.26ccm</td>
<td>30.06ccm</td>
<td>0.73%</td>
<td>1.0%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>191.1ccm</td>
<td>190.05ccm</td>
<td>0.65%</td>
<td>1.0%</td>
<td>Out of Tolerance</td>
</tr>
<tr>
<td>516.9ccm</td>
<td>500.65ccm</td>
<td>3.25%</td>
<td>1.0%</td>
<td>Out of Tolerance</td>
</tr>
</tbody>
</table>

**Lab. Pressure**  763 mmHg  
**Lab. Temperature**  22.5°C

## Bios International Standards Used

<table>
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</thead>
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Bios International  
- 10 Park Place Butler, NJ 07405  
- 800.665.4977  
- [www.biosint.com](http://www.biosint.com)
### As Shipped Calibration Data

<table>
<thead>
<tr>
<th>Instrument Reading</th>
<th>Lab Standard Reading</th>
<th>Deviation</th>
<th>Allowable Deviation</th>
<th>As Shipped</th>
</tr>
</thead>
<tbody>
<tr>
<td>30.5 ccm</td>
<td>30.7 ccm</td>
<td>-0.6%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>100.8 ccm</td>
<td>100.05 ccm</td>
<td>0.75%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
<tr>
<td>508.6 ccm</td>
<td>508.1 ccm</td>
<td>0.6%</td>
<td>1.00%</td>
<td>In Tolerance</td>
</tr>
</tbody>
</table>

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<tr>
<th>Description</th>
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<th>Calibration Date</th>
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</tr>
</thead>
<tbody>
<tr>
<td>ML-S00-10</td>
<td>113778</td>
<td>11/13/2009</td>
<td>11/13/2010</td>
</tr>
</tbody>
</table>

### Calibration Notes

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### Technician Notes:

![Signature]

Harvey Padden, President and Chief Metrologist
CERTIFICATE

SOLD TO: Praxair
PO Box 9224
Des Moines, IA 50306

SHIP TO: Praxair Dale Atchley
2801 Montopolis Drive
Austin, TX 78741

DATE: 10/05/09
P.O. # 2580263
ORDER # 100509A

ANALYTICAL REPORT: PRODUCT CERTIFICATION

<table>
<thead>
<tr>
<th>CYLINDER #</th>
<th>CONSTITUENT</th>
<th>CONCENTRATION</th>
<th>NOMINAL</th>
<th>ACTUAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>CC53242</td>
<td>Formaldehyde</td>
<td>CH₃O</td>
<td>60.0 ppm</td>
<td>59.0 ppm</td>
</tr>
<tr>
<td></td>
<td>Sulfur Hexafluoride</td>
<td>SF₆</td>
<td>20.0 ppm</td>
<td>21.9 ppm</td>
</tr>
<tr>
<td></td>
<td>Nitrogen</td>
<td>N₂</td>
<td>Balance</td>
<td>Balance</td>
</tr>
</tbody>
</table>

Blend Tolerance +/- 10%
Analytical Accuracy ≤ 5%

PRESSURE: 2000 PSIA
Valve: CGA-350
ANALYSIS DATE: 10/05/09
EXPIRATION DATE: 10/05/10
Compressed Gas N.O.S.
(Formaldehyde, Nitrogen) 2.2
UN 1956
Emergency Phone # 1 800 535 5053

ANALYST: [Signature]
Date: 10/5/09
International SEMATECH Manufacturing Initiative
Technology Transfer
2706 Montopolis Drive
Austin, TX 78741

http://ismi.sematech.org
e-mail: info@sematech.org