Developing a Reliable Fluorinated Greenhouse Gas (F-GHG) Destruction or Removal Efficiency (DRE) Measurement Method for Electronics Manufacturing: A Cooperative Evaluation with Qimonda March 2008



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Table of Contents

Page

Ackno	owledge	ements		2
1.0	Intro	duction.		6
2.0	Expe	rimental	Setup	6
3.0	Data	Analysis	5	8
	3.1	Deterr	nination of Scrubber Dilution	8
	3.2	Scrubł	ber DRE Determinations	11
		3.2.1	DRE Determination During Process Calibration Flows	11
		3.2.2	Determination of DRE during Wafer Processing	14
4.0	Conc	lusion		21

List of Tables

Page

Table I	Data used to determine the total flow emitted from TPU systems on AMZ17 and AMZ18	; 9
Table II	Total chamber and process pump effluent flow from chamber B on AMZ17 and AMZ18	.11
Table III	CF ₄ DRE values determined during process flow calibrations	.13
Table IV	CHF ₃ DRE for AMZ18 scrubber based on data shown in Figure 7	.14
Table V	Process Utilization for etch gases used in contact etch	.17
Table VI	CF ₄ DRE determined for each scrubber by comparing inlet and outlet emission volumes	.19
Table VII	Comparison of two methods used to determine CF_4 DRE on scrubbers for AMZ17 and AMZ18	.21

List of Figures

	Pa	ge
Figure 1	Sampling schematic used for testing TPU PFC DRE	8
Figure 2	QMS Response to ⁸⁴ Kr during calibration	8
Figure 3	Regression analysis of calibration data shown in Figure 2	9
Figure 4	Kr concentration determined for AMZ17 scrubber during spiking experiment	10
Figure 5	CF_4 emission concentration determined from AMZ17 chamber B while CF_4 was flowed through chamber B with RF power off	11
Figure 6	CF ₄ emission profile from AMZ18 chamber B (top) and from TPU (bottom) during proce flow calibration	ess 13
Figure 7	CHF ₃ emissions from process (top) and scrubber (bottom) during CHF ₃ process flow calibration	15
Figure 8	FTIR spectra of emissions from arc etch (top) and contact main etch (bottom)	17
Figure 9	Emission profiles for PFC process gases (top) and plasma by-products (bottom) during contact etching of test wafers on AMZ18 chamber B	19
Figure 10	FTIR spectrum of AMZ18 scrubber emissions during test wafer processing	20
Figure 11	CF ₄ emission profile from AMZ18 scrubber during processing of test wafers on Chamber and during processing of production wafers on multiple chambers	: B, 21
Figure 12	CF_4 emission profile from AMZ17 scrubber during processing of production wafers on Chamber B, and during processing of production wafers on multiple chambers	21

1.0 Introduction

The purpose of this study was to accurately determine the Destruction or Removal Efficiency (DRE) of a Point Of Use (POU) abatement system or scrubber for process emissions containing perfluorinated compounds. A key component in accurately determining DRE was to determine the dilution of process exhaust occurring in the scrubber. This study used an experimental approach to measure the dilution across the scrubber by injecting a chemical tracer that could not react in the scrubber, or be produced as a by-product during scrubber operation. Krypton was used as the chemical tracer as it met the requirements for this application.

Testing was conducted in a fully functional semiconductor manufacturing facility, owned and operated by Qimonda in Richmond VA. Two tools, AMZ17 and AMZ18, each equipped with POU scrubbers were tested. Both tools ran the same process and used the same model of commercially available scrubber. The process evaluated was a contact etch process, which used PFC gases CF_4 , CHF_3 and C_4F_6 .

2.0 Experimental Setup

To carry out the objectives of this study it was necessary to monitor both process and scrubber emissions simultaneously, and determine scrubber dilution using chemical spiking. Process and scrubber emissions data were collected in parallel using Fourier Transform Infrared Spectroscopy (FTIR). Data used to determine scrubber dilution were collected using Quadrupole Mass Spectrometry (QMS). A schematic showing the experimental testing set up is shown in Figure 1.

Two FTIRs were used to determine process and scrubber emissions. Both systems were MKS 2010 Multi Gas Analyzers equipped with liquid nitrogen cooled mercury cadmium telluride (MCT) detectors. One FTIR was equipped with a 10 cm path length single pass gas cell, and was used to sample process effluent. The other FTIR was equipped with a 5.6 m path length multi pass gas cell, and was used to sample scrubber effluent. Both FTIR were operated at 0.5cm⁻¹ resolution. Four scans were co-added for each data point yielding a sampling frequency of 2.2 sec.

A Balzers QMS system was used to sample scrubber effluent during dilution determination. The QMS was operated in Selective Ion Monitoring (SIM) mode and a secondary electron multiplier was used to enhance sensitivity. A 1 sec sampling frequency was used for each data point. To account for potential changes in QMS sensitivity, ion signals were normalized to the signal obtained for the nitrogen fragment (N+), which is formed during electron impact ionization of N_2 .

Sampling of effluent streams was done using metal bellows sampling pumps that were located after the instruments. The sample flow rate was controlled using adjustable flow rate valves. The sample line pressure for both FTIRs and the QMS were monitored using capacitance manometers. A filter was installed in the sample line used for monitoring scrubber emissions to ensure that particulate emissions from the scrubber would not coat the FTIR internal optics, or

the pressure reducing orifice used for the QMS. Since the scrubber DRE determination and the scrubber dilution determination were independent events, it was possible to use the same sample line for both operations. This was accomplished by switching the instrument inlet sample fitting from the FTIR to the QMS.

The QMS was calibrated to determine its response to Kr on site using a dynamic dilution blending system. Test atmospheres containing Kr, were created by blending a calibration standard containing 1% of Kr in N_2 with N_2 diluent. The QMS response to ⁸⁴Kr during calibration is shown in Figure 2. From regression analyses of these data a calibration curve was generated and is shown in Figure 3. This calibration was repeated for both tools tested.



Figure 1: Sampling schematic used for testing TPU PFC DRE



Figure 2: QMS Response to ⁸⁴Kr during calibration.





Figure 3: Regression analysis of calibration data shown in Figure 2

3.0 Data Analysis

3.1 Determination of Scrubber Dilution

One of the primary goals of this study was to accurately determine the dilution that occurs when gas emitted from the process chamber passes through the scrubber. Dilution can occur from many sources including effluents from other chambers, combustion gases and by-products added to and generated within the scrubber, vapors added as the gas stream passes through the water scrubber portion of the system, in-board leaks, and back diffusion from main headers. The method of determining dilution in this study was to use a purely experimental approach where a chemical was spiked into the gas stream entering the scrubber at a known flow rate, and determined in the scrubber effluent stream. From the determined concentration and the controlled flow rate added to the process exhaust duct, a total flow from the scrubber could be calculated:

$$TF = S_{f}/(C_{an} X 10^{-6})$$
(1)

Where S_f represents the spike gas flow and is reported in liters per minute, and C_{an} represents the analyte concentration reported in ppmv.

The experiment conducted to determine dilution for the scrubbers on AMZ17 and AMZ18 consisted of using the calibration system shown in Figure 1 to add calibration gas into the process effluent through the FTIR sample line where the process effluent was monitored. While calibration gas was being added, the QMS was used to sample scrubber effluent. The flow of calibration gas was controlled with a 0-5 slm Mass Flow Controller (MFC) that was calibrated for nitrogen. Five flow rates were added to the scrubber: 1, 2, 3, 4 and 5 slm. The concentration profile for Kr determined from QMS data during this experiment are shown in Figure 4 for AMZ17.



Figure 4: Kr concentration determined for AMZ17 scrubber during spiking experiment. Flow rates of calibration gas are indicated on the graph.

Applying Eq. 1 to the data obtained for the scrubbers on AMZ17 and AMZ 18 yielded the total flows contained in Table 1. The average total flow for each scrubber is also contained in Table 1.

AMZ17 System	Equivalent Kr	Kr Concentration	Total Flow
Total Cal Gas Flow	Flow (slm)	measured at TPU	(slm)
(slm)		Outlet (ppmv)	
1.0	0.010	15.1 ± 1.4	662 ± 61
2.0	0.020	27.1 ± 2.7	738 ± 74
3.0	0.030	40.8 ± 1.8	735 ± 32
4.0	0.040	55.5 ± 1.9	723 ± 25
5.0	0.050	69.5 ± 2.0	719 ± 21
AMZ18 System			Ave. Total Flow
Total Cal Gas Flow			for AMZ17 System
(slm)			721 ± 2
1.0	0.010	14.1 ± 1.6	709 ± 80
2.0	0.020	25.5 ± 1.7	784 ± 52
3.0	0.030	38.8 ± 1.7	773 ± 34
4.0	0.040	52.2 ± 1.9	766 ± 28
5.0	0.050	65.8 ± 2.4	760 ± 28
			Ave. Total Flow
			for AMZ18 System
			765 ± 2

Table I: Data used to determine the total flow emitted from TPU systems on AMZ17 andAMZ18.

The total scrubber flow data can be combined with the flow from the process chamber and pump, which go into the scrubber and is referred to as the total process flow, to determine the dilution that occurs as the process effluent passes through the scrubber. This calculation requires measuring the dilution that occurs as gases from the etch chamber are pumped out of the chamber and fore line and sent into the corrosive scrubber exhaust. The experiment to measure the dilution of AMZ17 and AMZ18 chamber effluent consisted of flowing CF_4 into the chamber with the RF power in the chamber turned off at several flow rates. The determined CF_4 concentration in the effluent could be used to calculate the total process flow entering the scrubber from the following equation:

$$TPF = PG_{f}/(C_{PG} \times 10^{-6})$$
(2)

Here the total process flow (TPF) is determined from the ratio of the process gas flow (slm) divided by the measured concentration (C_{PG}) in ppmv. The values obtained for CF₄ on AMZ17 are shown in Figure 5. From these data the total process exhaust flows for both AMZ17 and AMZ18 were calculated and are contained in Table II.



CF4 Emissions from ETCAMZ17 Chamber B during CF4 Chamber Flows

Figure 5: CF₄ emission concentrations determined from AMZ17 chamber B while CF₄ was flowed through chamber B with RF power off.

Table II: Total chamber and process pump effluent flow from chamber B on AMZ17 and AMZ18. Values determined from the average concentration measured during each CF_4 flow using Eq. 2

ETCAMZ17			ETCAMZ18		
CF ₄ Flow	CF ₄ Conc.	Total	CF₄ Flow	CF ₄ Conc.	Total
(slm)	(ppmv)	Effluent	(slm)	(ppmv)	Effluent
		Flow			Flow
		(slm)			(slm)
0.200	11,211 ±	17.8 ± 0.3	0.200	$12,134 \pm$	16.5 ± 0.3
	183			195	
0.150	8780 ± 55	17.1 ± 0.2	0.150	9652 ± 121	15.5 ± 0.2
0.100	5784 ± 109	17.3 ± 0.3	0.100	6477 ± 48	15.4 ± 0.1
0.050	3024 ± 43	16.5 ± 0.2	0.050	3297 ± 32	15.2 ± 0.2
0.025	1554 ± 48	16.1 ± 0.5	0.025	1681 ± 61	14.9 ± 0.5
			•		

The data in Table II yielded an average total flow of 17.0 ± 0.7 slm for AMZ17 and 15.5 ± 0.6 slm for AMZ18. The process flow into the scrubber combined with the total flow from the scrubber yielded the experimentally measured dilution for each scrubber:

System Dilution = TF_{out}/TF_{in}		(3)
For AMZ17:	Dilution = $721 \pm 2/17.0 \pm 0.1 = 42.4 \pm 2$	
For AMZ18:	Dilution = $765 \pm 2/15.5 \pm 0.1 = 49.4 \pm 2$	

Equipped with these dilution factors and total flows into and out of the scrubbers it is now possible to determine the scrubbers DRE for the gases used and by-products formed during wafer processing.

3.2 Scrubber DRE Determinations

Determination of the scrubber performance was done using two different testing conditions. The first was to measure the scrubber effluent of the etch process gases during the total process flow calibrations and the second was to measure the scrubber effluent during wafer processing.

3.2.1. DRE Determination During Process Calibration Flows

The scrubber DRE for CF_4 , CHF_3 and C_4F_6 was determined during total process flow calibrations by comparing the steady-state inlet and outlet concentrations during the flow of each gas, and adjusting for the scrubber dilution. From these data a direct calculation of the scrubber DRE could be made. Figure 6 shows the scrubber inlet and outlet CF_4 concentrations determined for AMZ18 while CF_4 was flowing through the chamber. (These were the data used to calculate the AMZ18 process dilution.) These data were used to calculate the scrubber DRE for CF_4 using the following equation:

$$DRE = 1 - ((CF_{4out}X Dilution)/CF_{4in})$$
(4)

Here CF_{4out} and CF_{4in} represent the average CF_4 concentrations determined for each flow shown in Figure 6. Using this method of comparing concentrations into and out of the scrubber, CF_4 DRE values for AMZ17 and AMZ18 are tabulated and contained in Table III.



CF4 Emissions from TPU During Process Flow Calibration



Figure 6: CF_4 emission profile from AMZ18 chamber B (top) and from TPU (bottom) during process flow calibration. Each level of concentration is equivalent to a specific CF_4 flow rate thru the process chamber.

Table III: CF₄ DRE values determined during process flow calibrations.

AMZ	217			
CF ₄ Flow	Ave Process	Ave Scrubber	Dilution	DRE
(slm)	Emiss. Conc.	Emiss. Conc.	Adjusted	(%)
	(ppmv)	(ppmv)	Concentration	
			(ppmv)	
0.200	$11,121 \pm 183$	191 ± 2.5	8098	$\textbf{27.8} \pm \textbf{0.6}$
0.150	8783 ± 55	151 ± 1.5	6402	$\textbf{27.1} \pm \textbf{0.4}$
0.100	5811 ± 109	104 ± 0.6	4410	23.6 ± 0.5
0.050	3028 ± 43	51 ± 0.4	2162	28.5 ± 0.5
0.025	1551 ± 48	27 ± 0.5	1145	26.3 ± 1.0
AMZ	218			
0.200	$12,134 \pm 195$	200 ± 2.0	9880	18.6 ± 0.4
0.150	9652 ± 121	162 ± 0.8	8005	17.1 ± 0.2
0.100	6477 ± 48	113 ± 0.5	5582	13.8 ± 0.2
0.050	3297 ± 32	59 ± 0.3	2915	11.6 ± 0.2
0.025	1681 ± 61	30 ± 0.2	1482	11.8 ± 0.5

From the data contained in Table III the scrubber CF_4 DRE appears higher for the AMZ17 scrubber relative to the AMZ18 scrubber. Results for CHF_3 indicated that AMZ17 abated CHF_3 sufficiently to yield a concentration below the detection limit of the FTIR equipped with a 5.6m cell, while relatively low emissions of CHF_3 were detected from AMZ18 scrubber. The CHF_3 inlet and outlet profiles determined during CHF_3 flows through chamber B on AMZ18 are shown in Figure 7. Both scrubbers abated C_4F_6 sufficiently to yield an outlet concentration not detected by the FTIR.



CHF3 Emissions from AMZ18 Chamber B during Process Flow Calibration

CHF3 Emissions from Scrubber during CHF3 Process Flow Calibration



Figure 7: CHF₃ emissions from process (top) and scrubber (bottom) during CHF₃ process flow calibration.

From the data shown in Figure 7, the AMZ18 scrubber DRE was calculated for CHF_3 during the total process flow calibration and is contained in Table IV. These data indicate a relatively high DRE for CHF_3 on AMZ18 scrubber. The AMZ17 scrubber had a higher DRE as CHF_3 was not detected in the effluent during the CHF_3 flow calibration.

CHF₃ Flow	Ave Process	Ave Scrubber	Dilution	DRE
(slm)	Emiss. Conc.	Emiss. Conc.	Adjusted	(%)
	(ppmv)	(ppmv)	Concentration	
			(ppmv)	
0.200	$12,820 \pm 170$	3.1 ± 0.1	153	98.8
0.100	6538 ± 74	1.4 ± 0.06	69	98.9
0.025	1601 ± 15	0.4 ± 0.01	20	98.8

Table IV: CHF₃ DRE for AMZ18 scrubber based on data shown in Figure 7.

The DRE for C_4F_6 was high for both AMZ17 and AMZ18 scrubbers. The estimated detection limit for C_4F_6 was 0.5 ppmv with the 5.6m gas cell based on a signal to noise ratio of 3. Thus, based on a 0.100 slm process flow (0.100 slm was the maximum flow possible with the installed MFC), which yielded an average process emission of 6474 ± 59 ppmv on AMZ18, the minimum DRE would be 99.6 %.

3.2.2 Determination of DRE during Wafer Processing

Determining the scrubber DRE under wafer processing conditions can be much more challenging, particularly if the PFC effluent concentration does not reach a steady state condition (here a steady state condition is defined as dC/dt = 0, where the concentration (C) is not changing over a relatively short period of time, as shown in Figure 6). Under these conditions it may be

necessary to numerically integrate the PFC concentration over time to yield an emission volume, which can be compared to the integrated process emission volume entering the scrubber for a given analyte. To convert measured concentrations into volumes, the following equation was used:

$$V_{\rm EM} = \sum C_i T_f \Delta t \tag{5}$$

Where the total emission volume (V_{EM}) is the summation of each FTIR data point where the concentration of analyte C is determined during time interval Δt and multiplied by the total flow (T_f). The summation of the entire emission profile provides an emission volume for a given analyte during the process. During this study, these calculations were performed using standard spreadsheet software (Microsoft Excel). Use of this technique reinforces the importance of accurately determining the total process and scrubber flows as described in the sections above.

The process tested on AMZ17 and AMZ18 was a dielectric etch process that had two distinct etch steps. The approximate process recipes were as follows:

Step 1 Arc Etch: 160 sccm CF₄; 100 sccm CHF₃; 150 sccm Ar; 20 sccm O₂ 35 sec Step 2: Main Etch: 60 sccm C₄F₆; 1000 sccm Ar; 45 sccm O₂ 65 sec

In addition to the etch times listed above, up to 5 sec of additional chamber stabilization time is required to turn the process gases on and set the chamber pressure prior to turning on the process plasma.

During the etch processes etch gases CF_4 , CHF_3 and C_4F_6 could be detected in the process effluent. Many etch by-products were also detected. These included SiF_4 , HF, COF_2 , C_2F_4 , C_2F_6 and CO. Figure 8 shows the FTIR spectrum of process emissions for each etch step.





Figure 8: FTIR spectra of emissions from arc etch (top) and contact main etch (bottom)

Typical emission profiles for the etch process are shown in Figure 9. These data were acquired on AMZ18 chamber B during the etching of test wafers. The top graph shows PFC process gas emissions during both etch steps for three wafers. The bottom graph shows the etch by-products

formed in the plasma during the process. The spikes observed in the process gas emissions are attributed to the stabilization flow at the beginning of the etch process, and the chamber purge of residual gases after the plasma has been turned off at the conclusion of etching.

The PFC emissions data shown in Figure 9 (top) were integrated to determine the total emission volume for each gas, and used to calculate the process utilization for each molecule. Using the process recipe shown above the following PFC plasma utilization values were obtained for CF_4 , CHF_3 and C_4F_6 :

Table V: Process Utilization for etch gases used in contact etch. The volumes reported as being used were calculated from the process recipe and assumed a stabilization flow of 5 seconds. Data reported per wafer processed.

CF4 Used for Process	CF₄ Emitted from process	CF ₄ Process Utilization
(sl)	(sl)	(%)
0.107	0.088	18
CHF ₃ Used for Process	CHF ₃ Emitted from	CHF₃ Process Utilization
(sl)	process	(%)
	(sl)	
0.067	0.032	52
C ₄ F ₆ Used for Process	C ₄ F ₆ Emitted from	C ₄ F ₆ Process Utilization
(sl)	process	(%)
	(sl)	
0.070	0.005	93

The data contained in Table V include emissions for both etch steps of the process. Approximately 10% of the total CF_4 emission was observed during the C_4F_6 process step, where CF_4 was formed as a by-product.



17

By-product Emissions from Contact Etch on AMZ18



Figure 9: Emission profiles for PFC process gases (top) and plasma by-products (bottom) during contact etching of test wafers on AMZ18 chamber B. The ARC and Main etch portions are labeled in the figures.

Emissions from the scrubber during wafer processing included primarily CF₄. Figure 10 shows an FTIR spectrum obtained during test wafer processing on AMZ18. Low level CHF₃ and C₂F₆ emissions were detected. The CF₄ emission profile from the scrubber is shown in Figure 11. Only the data for the test wafers was used to calculate a DRE as emissions from multiple chambers confound the DRE determination if the scrubber loading from other chambers is not accounted for. CF₄ emissions from the test wafers were integrated and used to calculate DRE by comparing to the integrated process emissions reported above. Figure 12 shows CF₄ emissions from AMZ17 scrubber. Again multiple chambers were being used on the tool. CF₄ emissions for the last 9 wafers were integrated to yield total CF₄ emission for 9 wafers and compared to the integrated process emissions for the same 9 wafers. The results for both scrubbers are contained in Table VI below:

Table VI: CF₄ DRE determined for each scrubber by comparing inlet and outlet emission volumes.

AMZ17		
Integrated CF₄ Process	Integrated CF ₄ Scrubber	Scrubber CF ₄ DRE
Emissions for 9 wafers	Emissions for 9 wafers	(%)
(sl)	(sl)	
0.920	0.716	22
AMZ18		
Integrated CF ₄ Process	Integrated CF ₄ Scrubber	Scrubber CF ₄ DRE
Emissions for 5 test wafers	Emissions for 5 test wafers	(%)
(sl)	(sl)	
0.445	0.425	4

Emissions of C_2F_6 and CHF_3 from the AMZ18 scrubber during test wafer processing were integrated and used to estimate minimum DRE values of > 98 % for C_2F_6 and > 98.7 % for CHF_3 . Actual DRE values are presumed to be higher. The minimum DRE for C_2F_6 is based on an integrated per wafer process emission of 0.010 sl and an integrated scrubber emission of < 0.001 sl. The minimum DRE for CHF_3 is based on an integrated per wafer process emission of 0.033 sl and an integrated scrubber emission of < 0.001 sl. CHF_3 and C_2F_6 were not detected in the effluent of the scrubber on AMZ17.



Figure 10: FTIR spectrum of AMZ18 scrubber emissions during test wafer processing.





Figure 11: CF₄ emission profile from AMZ18 scrubber during processing of test wafers on Chamber B, and during processing of production wafers on multiple chambers.



CF4 Emissions from TPU During AMTZ17 Wafer Processing

Figure 12: CF₄ emission profile from AMZ17 scrubber during processing of production wafers on Chamber B, and during processing of production wafers on multiple chambers.

III: Comparison of CF₄ DRE Determinations:

Both methods of determining the scrubber DRE for CF_4 on AMZ17 and AMZ18 are compared in Table VII. Both methods are in reasonable agreement and both indicate a relatively low DRE for CF_4 on the scrubbers tested. Higher DRE were obtained for etch gases CHF_3 and C_4F_6 , and process by-product C_2F_6 . The AMZ17 scrubber appears to be more effective at abating all PFCs. It was noted during testing that the AMZ17 scrubber was operating at a higher reported temperature than the AMZ18 scrubber, however, temperature data were not collected.¹

¹ Observations of temperature during the study indicated that AMZ17 was running at approximately 885 to 905 °C, whereas, AMZ18 was running at approximately 800 to 815 °C. It is believed that the noted temperatures refer to the external wall temperature of the combustion chamber within the abatement device.

Table VII: Comparison of two methods used to determine CF₄ DRE on scrubbers for AMZ17 and AMZ18

Scrubber	CF4 DRE determined from Process Gas flow Calibrations (average of 5) (%)	CF4 DRE from Integrated Process Emissions (%)
ETCAMZ17	27	22
ETCAMZ18	15	4

4.0 Conclusion

Determination of the DRE of PFCs by POU scrubbers has been conducted for a contact etch process at Qimonda. Two scrubbers of the same make and model were tested. The processes tested were the same on each tool. The results indicated very high DRE for most PFC process gases and PFC by-products. Only CF_4 was determined to have a low DRE on both systems.

One key element of this study was to accurately determine the dilution that occurs to process effluent as it passes through the scrubber into the corrosive scrubber exhaust duct. In this study, the dilution was determined by using a chemical tracer, Kr, which was injected into the scrubber inlet of one process chamber, and subsequently determined in the scrubber effluent. The effluent Kr concentration was determined by using a QMS, which was calibrated on-site for its response to Kr. The choice of Kr for chemical spiking was dictated by the need for a tracer that would not react in or be produced by the scrubber.

Two methods of determining process gas DRE were investigated. Use of a continuous flow of PFC gases permitted a steady state emission of PFC from both the process chamber and scrubber (provided the DRE was < 100%). This allowed direct comparison of emission concentrations from tool and scrubber after scrubber effluent data were corrected for dilution. Wafer processing emissions were also used to calculate DRE by integrating the emission concentrations over time and total flow to yield emission volumes into and out of the scrubber. The comparison of these methods yielded DRE values that were in reasonable agreement. The latter method could be needed in cases where the effluent data do not appear to reach a steady state, particularly for the scrubber effluent. The fundamental difference between the two methods is that etch by-products would be present in method 2, whereas they would not present in method 1. This would include any particulates that may be present in the etch effluent stream.