5.0 Quality Assurance and Quality Control

As part of the Industrial Boiler F-410 trial burn at The Dow Chemical Company, Louisiana Operations facility in Plaquemine, Louisiana, Radian implemented a Quality Assurance/Quality Control (QA/QC) effort tailored to meet the specific needs of this project. This effort was intended to ensure that the sampling and analysis activities were performed under controlled conditions, within specific performance parameters, so that the results of the test program would accurately represent industrial boiler performance. The results of this effort document that the project measurement data are valid, defensible, and useable for evaluating industrial boiler performance and compliance with permit requirements.

The primary objectives of this QA/QC effort were to control, assess, and document data quality. In order to accomplish these objectives, the QA/QC approach consisted of the following key elements:

- Definition of data quality objectives that reflect the overall technical objectives of the project;
- Design of a sampling, analytical, QA/QC, and data analysis system to meet these objectives;
- Evaluation of the performance of the measurement systems; and
- Initiation of corrective action when measurement system performance does not meet the specifications.

This trial burn was conducted in accordance with applicable QA procedures, as described in the Trial Burn Quality Assurance Project Plan (April 1997). These include sampling and analytical procedures, along with specified calibration requirements, QC checks, data reduction and validation procedures, and sample tracking. A discussion of measurement uncertainty, based on results for analysis of QA/QC samples, and any anomalies or limitations in the use of the data are presented in this section. Calibration or standardization data and results for all method QC checks (such as method blanks and laboratory control samples, matrix spiked samples, surrogate

spikes, etc.) are presented in the appendices of this report. Quality records pertaining to field activities, including calibration records for field sampling equipment, field data logs and sample tracking forms are also included in appendices to this report.

Measurement Quality Data Objectives

Overall, the measurement data quality is of acceptable quality to evaluate industrial boiler performance as intended. The first step in obtaining representative measurement data involves collection of representative samples. All sample collection efforts were conducted under strict criteria for acceptability, particularly for collection of stack gas samples, which involved continuous monitoring and control of sample collection conditions, such as temperatures, flow rates, pressures, isokinetic sampling rates, etc. All sampling was conducted in strict accordance with the protocols and criteria for sampling.

Quality control data associated with the analysis of sample indicate that the analytical systems were properly calibrated and operated in a state of control. Of the quality assurance data collected, only a small percentage deviate from the objectives of the program, and there is no evidence of systematic measurement error that would compromise the data or invalidate the conclusions of the test program.

5.1 Sampling Quality Control

Samples were collected according to EPA Reference Methods and procedures detailed in the QAPjP. Samples were handling and stored in segregated areas to minimize cross-contamination.

For stack gas sampling, numerous QC activities were performed to ensure the collection of valid, representative samples. A trial burn task leader supervised activities to ensure adherence to the project QAPjP/Test Plan, including the use of method-specific sampling quality control checks and appropriate documentation procedures With respect to stack gas sampling activities, critical aspects included:

- Pre-sampling preparations, including:
  - Calibration of dry gas meters, temperature sensors, nozzles, pitot tubes, and balances,
  - Preparation of filters and sorbents, including handling, weighing, loading, and identification,
  - Identification of appropriate sampling locations and conditions, including nozzle size, traverse points, sampling rate, etc.;

- Sampling operations, including:
  - Cyclonic flow check (the absence of cyclonic flow was verified by Radian personnel prior to the trial burn),
  - Sample train leak checks (including pitot tubes);
  - Probe handling and plugging of ports during sampling;
  - Temperature controls and documentation;
  - Isokinetic determinations;
  - Minimum sampling times and/or volumes;
  - Completeness of data records; and

- Post-sampling operations, including:
  - Sufficient volume/mass collected,
  - Handling of train to minimize loss or contamination of sample,
  - Determination of isokinetics,
  - Sample recovery,
  - Preparation of field blanks, and
  - Data reduction.

For all sampling activities, recordkeeping procedures were reviewed by the sampling supervisor for completeness, proper and legible transcription of information, making corrections, dating and signing. These records include:

- Master logbooks;
- Stack gas data collection sheets;
- Sampling equipment calibration records;
- Balance calibration records; and
- Sample shipping and tracking forms.

Original sample data sheets and logbooks are presented in the appendices and associated equipment calibration records are also presented in the appendices. These data show that except as discussed in the next paragraph, the method or QAPjP criteria were met for each run,

including, as appropriate, isokinetic sampling rates, sample volume collected, sample train leak rates, and sample train temperatures (e.g., probe liner, filter holder, impinger exit, condenser exit).

One notable problem occurred with collection of the stack gas sampling trains:

- The sampling data sheet from the stack gas chromium (VI) Run 3 of Test Condition 4 was inadvertently misplaced and lost from the time the sample was collected and recovered to the time the data florms were entered into the computer spreadsheets at the Radian facility in Austin, Texas. The average sampling data from runs 1 and 2 of Test Condition 4 were used to calculate the chromium (VI) emission rate from Run 3. A review of all the stack gas chromium (VI) sampling data (5 runs from Test Conditions 1 and 4) in Section 3.0 indicate that all sampling criteria (i.e. isokinetic rates, proper sample volumes) were met for the previous chromium (VI) test runs. The calculated data from Run 3 show comparable results with the previous runs, thus, the use of the average sampling data from runs 1 and 2 do not appear to bias the data to any significant degree.

Quality control activities associated with waste and process sampling activities are described in the QAPjP. These activities include adherence to accepted reference method protocols and use of standardized data recording sheets.

Sampling procedures are described in the QAPjP and in Section 3.0 of this report.

5.2 Analytical Quality Control

The analyses for the industrial boiler test program were performed according to the methods specified in the Trial Burn QAPjP. The analyses followed the technical, operational, and procedural specifications and protocols of the approved methods.

Each method has specific requirements and criteria for controlling and assessing the performance of each analytical method, such as instrument calibration and ongoing calibration verification, analysis of blanks to monitor contamination and carryover, and analysis of known standards that are processed through all the normal sample preparation (such as digestion or extraction, cleanup, etc.) and analysis (such as instrumental analysis by ICPES or GC/MS, etc.)

steps to monitor the accuracy of the entire method in the absence of interferences from unknown sample matrices. Additional QC checks, such as analysis of laboratory spiked samples, were performed to assess the effectiveness of the method in the actual sample matrices with their various often unpredictable interferences.

Data quality objectives were established in the QAPjP as the fundamental indicators of measurement effectiveness. These are based primarily on measures that encompass the entire analytical method as well as the sample matrix, such as analyte recovery in the actual sample matrices (as determined by spike recovery) and measurement variability (as determined by duplicate analysis of samples). Results for these principal data quality indicators are discussed in the following sections, along with a discussion of the results for the routine method performance checks, such as analysis of laboratory control samples and blank samples.

All QC check results are presented in the analytical reports in the appendices.

5.3 Stack Gas Samples

This section presents a summary of measurement data quality for stack gas samples. This summary is based on comparison of the indicators of measurement quality with the data quality objectives established in the QAPjP, review of indicators of control of the analytical processes, and assessment of potential blank effects.

The principal data quality indicators specified in the QAPjP for stack gas samples are matrix spiked samples and surrogate spikes. In addition, laboratory control samples are analyzed as on-going check of analytical method performance, in the absence of sample matrix effects and interferences.

To assess potential low-level bias, blank trains or sample media were collected and analyzed. Field blank trains were prepared during the trial burn by setting up a sampling train at the sampling location, without exposing the train to stack gas. Reagent blanks (e.g., unused filters, XAD resin, and solvents such as HPLC water, methanol, and methylene chloride) were

collected and held for analysis in the event of blank concerns. Method blanks are blanks prepared by the laboratory as a normal sample and included in each sample batch to assess any contamination that could have been introduced in the laboratory. Method blanks are prepared and analyzed using the same equipment, reagents, and procedures as the field samples.

5.3.1 Determination of Volatile POHCs and Volatile Organics: VOST Analys s

VOST analyses were performed according to the prescribed procedure. Quality control data indicate effective and reliable analytical results. The QC data are discussed below.

Laboratory Method Blanks

Laboratory method blanks associated with VOST analyses showed no laboratory contamination problems. Results for all target analytes were below the reporting limits in all laboratory blank samples. Blank results for all compounds are presented in Table 5-1.

Field Blank Samples

Field blank sorbent tubes were collected and analyzed with each sampling run. None of the POHCs were detected in any of the blanks. Results for all compounds are presented in Table 5-2.

Trip Blank Samples

Trip blanks, consisting of unexposed sorbent tubes, were included in each shipment of VOST samples to the laboratory to assess potential contamination from shipping and storage of samples. None of the trip blanks were analyzed.

Surrogate Spikes

Surrogate spike recoveries for Test Conditions 1, 2, 3, and 4 (including Tenax®, Tenax/charcoal) are presented in Tables 5-3, 5-4, 5-5, and 5-6, respectively. As evidenced by the summary table, overall precision and accuracy were very good. The standard deviation for the surrogate recoveries also demonstrate very good precision.

Table 5-1
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn
Summary of Laboratory Blank Results for VOST

Analyte	092197 (µg)	092297 (µg)	092697 (µg)	092697 (µg)	092797 (µg)	092997 (µg)	093097 (µg)	100297 (µg)	100497 (µg)
Dichlorodifluoromethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Chloromethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Vinyl Chloride	ND	ND	ND	ND	NA	NA	NA	NA	NA
Bromomethane	0.017	0.044	ND	ND	NA	NA	NA	NA	NA
Chloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Trichlorofluoromethane	ND	0ND	ND	ND	NA	NA	NA	NA	NA
1,1-Dichloroethene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Methylene Chloride	0.002	0.002	0.004	0.005	NA	NA	NA	NA	NA
Trans-1,2-Dichloroethene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,1-Dichloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
2,2-Dichloropropane	ND	ND	ND	ND	NA	NA	NA	NA	NA
cis-1,2-Dichloroethene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Chloroform	ND	ND	ND	ND	NA	NA	NA	NA	NA
Bromochloromethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,1,1-Trichloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Carbon tetrachloride	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,1-Dichloropropene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Benzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,2-Dichoroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Trichlroethene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,2-Dichloropropane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Dibromomethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Bromodichloromethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
cis-1,3-Dichloropropene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Toluene	0.005	0.006	0.008	0.009	NA	NA	NA	NA	NA
Trans-1,3-Dichloropropene	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,2-Trichloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Tetrachloroethene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,3-Dichloropropane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Dibromochloromethane	ND	ND	ND	ND	NA	NA	NA	NA	NA

Table 5-1
(Continued)

Analyte	092197 (µg)	092297 (µg)	092697 (µg)	092697 (µg)	092797 (µg)	092997 (µg)	093097 (µg)	100297 (µg)	100497 (µg)
1,2-Dibromoethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Chlorobenzene	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Ethylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
m-/p-Xylene	ND	ND	ND	ND	NA	NA	NA	NA	NA
o-Xylene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Styrene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Bromoform	ND	ND	ND	ND	NA	NA	NA	NA	NA
Cumene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,1,2,2-Teterachloroethane	ND	ND	ND	ND	NA	NA	NA	NA	NA
Bromobenzene	0.001	ND	ND	0.003	NA	NA	NA	NA	NA
1,2,3-Trichloropropane	ND	ND	ND	ND	NA	NA	NA	NA	NA
n-Propylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
2-Chlorotoluene	ND	ND	ND	ND	NA	NA	NA	NA	NA
4-Chlorotoluene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,3,5-Trimethylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Tert-Butylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,2,4-Trimethylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
Sec-Butylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
p-Cymene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,3-Dichlorobenzene	ND	ND	ND	0.003	NA	NA	NA	NA	NA
1,4-Dichlorobenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
n-Butylbenzene	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,2-Dichlorobenzene	ND	ND	ND	0.004	NA	NA	NA	NA	NA
1,2-Dibromo-3-chloropropane	ND	ND	ND	ND	NA	NA	NA	NA	NA
1,2,4-Trichlorobenzene	0.020	0.015	0.006	0.025	NA	NA	NA	NA	NA
Hexachlorobutadiene	0.006	0.012	0.002	0.006	NA	NA	NA	NA	NA
Naphthalene	0.035	0.027	0.010	0.045	NA	NA	NA	NA	NA
1,2,3-Trichlorobenzene	0.047	0.036	0.013	0.050	NA	NA	NA	NA	NA

ND — Not Detected
NA — Not Analyzed

Table 5-2
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn
Summary of Field Blank Results for VOST

Analyte	TC1 Run 1 (µg)	TC1 Run 2 (µg)	TC1 Run 3 (µg)	TC2 Run 1 (µg)	TC2 Run 2 (µg)	TC2 Run 3 (µg)	TC3 Run 1 (µg)	TC3 Run 2 (µg)	TC4 Run 1 (µg)	TC4 Run 2 (µg)	TC4 Run 3 (µg)
Dichlorodifluoromethane	NA	NA	NA	NA	NA	NA	0.010	0.032	NA	NA	NA
Chloromethane	NA	NA	NA	NA	NA	NA	0.014	ND	NA	NA	NA
Vinyl Chloride	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Bromomethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Chloroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Trichlorofluoromethane	NA	NA	NA	NA	NA	NA	ND	0.048	NA	NA	NA
1,1-Dichloroethene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Methylene Chloride	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Trans-1,2-Dichloroethene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,1-Dichloroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
2,2-Dichloropropane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
cis-1,2-Dichloroethene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Chloroform	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Bromochloromethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,1,1-Trichloroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Carbon tetrachloride	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,1-Dichloropropene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Benzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2-Dichoroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Trichlroethene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2-Dichloropropane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Dibromomethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Bromodichloromethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
cis-1,3-Dichloropropene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Toluene	NA	NA	NA	NA	NA	NA	0.005	ND	NA	NA	NA
Trans-1,3-Dichloropropene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,1,2-Trichloroethane	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
Tetrachloroethene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,3-Dichloropropane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Dibromochloromethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2-Dibromoethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Chlorobenzene	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND	ND
1,1,1,2-Tetrachloroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA

Table 5-2
(Continued)

Analyte	TC1 Run 1 (µg)	TC1 Run 2 (µg)	TC1 Run 3 (µg)	TC2 Run 1 (µg)	TC2 Run 2 (µg)	TC2 Run 3 (µg)	TC3 Run 1 (µg)	TC3 Run 2 (µg)	TC4 Run 1 (µg)	TC4 Run 2 (µg)	TC4 Run 3 (µg)
Ethylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
m-/p-Xylene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
o-Xylene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Styrene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Bromoform	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Cumene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,1,2,2-Teterachloroethane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Bromobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2,3-Trichloropropane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
n-Propylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
2-Chlorotoluene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
4-Chlorotoluene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,3,5-Trimethylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Tert-Butylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2,4-Trimethylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Sec-Butylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
p-Cymene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,3-Dichlorobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,4-Dichlorobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
n-Butylbenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2-Dichlorobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2-Dibromo-3-chloropropane	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2,4-Trichlorobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Hexachlorobutadiene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
Naphthalene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA
1,2,3-Trichlorobenzene	NA	NA	NA	NA	NA	NA	ND	ND	NA	NA	NA

ND — Not Detected

NA — Not Analyzed

Table 5-3
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 1
Summary of VOST Surrogate Recovery Percentages

	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/29/97**		**9/29/97**		**9/29/97**		**9/29/97**	
Run 1								
Dibromofluoromethane	104	104	105	107	103	106	108	109
Toluene-d_8	102	101	101	102	100	102	101	102
4-Bromofluorobenzene	119	113	111	113	110	118	112	105
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/30/97**		**10/2/97**		**9/30/97**		**9/29/97**	
Run 2								
Dibromofluoromethane	94	104	104	106	95	106	102	105
Toluene-d_8	106	116	101	101	100	101	100	101
4-Bromofluorobenzene	136	136	115	108	117	115	114	110
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/30/97**		**9/30.97**		**9/30/97**		**9/29/97**	
Run 3								
Dibromofluoromethane	98	107	93	106	98	109	109	106
Toluene-d_8	102	102	103	100	101	100	101	100
4-Bromofluorobenzene	121	118	119	111	118	118	116	112
Sample No:	**092997**		**093097**					
Date:	**9/29/97**		**9/30/97**					
Lab Blanks [a]								
Dibromofluoromethane	105		108					
Toluene-d_8	102		102					
4-Bromofluorobenzene	108		105					

T — Tenax Tube
T/C — Tenax/Charcoal Tube

[a] Lab blanks were not analyzed separately.

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Table 5-4
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 2
Summary of VOST Surrogate Recovery Percentages

	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**10/1/97**		**10/1/97**		**10/1/97**		**9/30/97**	
Run 1								
Dibromofluoromethane	106	100	106	99	104	105	106	108
Toluene-d_8	102	102	102	101	101	101	101	102
4-Bromofluorobenzene	118	121	118	115	121	116	116	106
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**10/1/97**		**10/1/97**		**10/1/97**		**9/30/97**	
Run 2								
Dibromofluoromethane	102	109	102	110	106	107	106	108
Toluene-d_8	100	102	102	102	101	102	101	102
4-Bromofluorobenzene	113	114	117	118	117	112	118	115
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**10/1/97**		**10/1/97**		**10/1/97**		**9/30/97**	
Run 3								
Dibromofluoromethane	104	110	100	108	104	107	100	108
Toluene-d_8	100	102	100	100	101	101	101	102
4-Bromofluorobenzene	115	115	110	113	112	118	112	122
Sample No:	**100197**							
Date:	**10/1/97**							
Lab Blanks [a]								
Dibromofluoromethane	109							
Toluene-d_8	101							
4-Bromofluorobenzene	113							

T — Tenax Tube
T/C — Tenax/Charcoal Tube

[a] Lab blanks were not analyzed separately.

Table 5-5
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 3
Summary of VOST Surrogate Recovery Percentages

	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/21/97**		**9/22/97**		**9/22/97**		**9/23/97**	
Run 1								
Dibromofluoromethane	104	108	108	113	104	106	138	140
Toluene-d_8	100	102	102	106	102	102	104	108
4-Bromofluorobenzene	109	105	112	115	112	105	80	80
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/26/97**		**9/26/97**		**9/26/97**		**9/23/97**	
Run 2								
Dibromofluoromethane	109	108	104	108	107	106	132	143
Toluene-d_8	102	103	101	102	102	104	105	110
4-Bromofluorobenzene	118	120	107	114	116	119	80	80
Sample No:	**1**		**2**					
Date:	**9/26/97**		**9/26/97**					
Run 3								
Dibromofluoromethane	102	108	106	109				
Toluene-d_8	102	102	101	102				
4-Bromofluorobenzene	111	120	116	119				
Sample No:	**092197**		**092297**		**092397**		**092697**	
Date:	**9/21/97**		**9/22/97**		**9/23/97**		**9/26/97**	
Lab Blanks [a]								
Dibromofluoromethane	112		110		146		108	
Toluene-d_8	100		101		105		102	
4-Bromofluorobenzene	118		119		79		124	

T — Tenax Tube
T/C — Tenax/Charcoal Tube

[a] Lab blanks were not analyzed separately.

Table 5-6
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 4
Summary of VOST Surrogate Recovery Percentages

	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)	T (%)	T/C (%)
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**9/27/97**		**9/27/.97**		**9/27/97**		**10/2/97**	
Run 1								
Dibromofluoromethane	103	106	98	103	102	104	105	109
Toluene-d_8	102	102	101	102	103	102	100	102
4-Bromofluorobenzene	NA	NA	NA	NA	NA	NA	102	100
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**10/4/97**		**10/2/97**		**10/4/97**		**10/2/97**	
Run 2								
Dibromofluoromethane	98	104	95	108	99	105	108	109
Toluene-d_8	98	100	100	99	96	100	102	100
4-Bromofluorobenzene	101	107	98	103	104	104	105	98
Sample No:	**1**		**2**		**3**		**Field Blank**	
Date:	**10/4/97**		**10/2/97**		**10/4/97**		**10/2/97**	
Run 3								
Dibromofluoromethane	99	101	66	105	98	106	104	108
Toluene-d_8	92	90	117	100	95	99	100	100
4-Bromofluorobenzene	104	102	159	111	103	103	99	100
Sample No:	**092797**		**100297**		**100497**			
Date:	**9/27/97**		**10/2/97**		**10/4/97**			
Lab Blanks [a]								
Dibromofluoromethane	104		108		100			
Toluene-d_8	102		100		95			
4-Bromofluorobenzene	NA		108		108			

T — Tenax Tube
T/C — Tenax/Charcoal Tube
NA — Not Analyzed
[a] Lab blanks were not analyzed separately.

Breakthrough D termination

Breakthrough in VOST traps was assessed by analyzing the front- and back-half tubes separately from each pair of traps collected. The objective for breakthrough is that the back half should have less than 30% of the analyte mass measured on the front half, if the amount on the back half is greater than 75 ng. During Test Condition 3, only trichlorofluoromethane, 1,2,4-trichlorobenzene, naphthalene, and 1,2,3-trichlorobenzene from the first sample pair of Run 1, Test Condition 3 had breakthrough exceeding the objective. The two POHCs showed no breakthrough. Table 5-7 presents the breakthrough data.

Table 5-7
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 3, Run 1
Summary of VOST Breakthrough Data

Compound (µg)	Front	Back	Break-through	Front	Back	Break-through	Front	Back	Break-Though
Run 1	Sample 1			Sample 2			Sample 3		
1,2,4-trichlorobenzene	0.038	0.105	276	NA	NA	NA	NA	NA	NA
Naphthalene	0.080	0.141	176	NA	NA	NA	NA	NA	NA
1,2,3-trichlorobenzene	0.063	0.120	190	NA	NA	NA	NA	NA	NA
Trichlorofluoromethane	NA	NA	NA	0.008	0.464	5,800%	NA	NA	NA

ND — Not Detected

Trichlorofluoromethane is a non-critical parameter or PIC. Breakthrough of the compound was isolated to the one sample pair, and corresponds with the very high volatility of the species.

The other three compounds that exhibited breakthrough in the one VOST saple pair are typically classified as semivolatile organic compounds. A discussion of the applicability of VOST to determine these compounds and others similar in organic properties follows

VOST Audit

There was not an EPA VOST Audit gas standard submitted by the Agency for sampling on-site.

Hold Tim

All VOST tube pairs were analyzed within the specified hold time.

Applicability of VOST

The VOST samples collected during Test Condition 3, i.e., the Risk Burn, were analyzed for an extended analyte list to support the preparation of an indirect risk assessment. There are several concerns regarding a portion of the VOST results.

The extended VOST analyte list is based on the calibration "cocktail" of SW-846 Method 8260. Method 8260 includes a number of compounds with boiling points above 150°C. VOST, as originally conceived, is for the measurement of organic compounds with boiling points ranging from 30°C to 100°C. The applicability of VOST has been extended. As an example, monochlorobenzene has a boiling point of 131-132°C. Chlorobenzene is included in VOST audit gas cylinders, and measured accurately,

However, the applicability of VOST for compounds with boiling points above 150°C has not been validated. SW-846 Method 8260 is an EPA-validated method, and presumably, has been shown to be accurate for all compounds in the Method 8260 analyte list. Although Method 8260 may be applicable for analysis of waters for compounds with boiling points, VOST is not applicable. The high boiling point compounds will most likely not make it to the Tenax resin traps, condensing out prior to the traps. And obviously, the VOST train is not rinsed by a solvent, so any compounds dropping out of the sample gas before the Tenax traps would not be completely recovered by a water resin.

The appropriate method for the higher boiling point compounds in Modified Method 5 sampling train with analysis by SW-846 Method 8270. Six compounds (trichlorobenzene, the dichlorobenzenes, and hexachlorobutadiene) reported on VOST samples, shown in Tables 4-9A and 4-9B, are also analyzed and reported as semivolatile organic compounds in Tables 4-10A and 4-10B, sampled by Modified Method 5 and analyzed by SW-846 Method 8270. The results reported in Tables 4-10A and 4-10B are most applicable.

VOST Result Concerns

All VOST to be sample pairs, i.e. Tenax and Tenax/charcoal, were analyzed separately to assess breakthrough. As stated above, breakthrough acceptance criteria were met for all analytes with the exception of four compounds from Run 1 of Test Condition 3. The VOST samples from Test Condition 3 were analyzed for an extended analytes list.

VOST results are presented in Tables 4-9A (not blank corrected) and Table 4-9B (blank corrected). There appears to be inordinate results for many of the higher boiling point compounds, especially in Run 1. These results were reviewed, and a problem was identified in the analysis of the VOST samples.

The VOST analytical results are presented in Appendix F. Reviewing these result it is noted that there are a series of analytical "hits", i.e. an actual result, above the method detection limit, for the higher boiling compounds. Moreover, the analytical "hits" for the higher boiling point compounds occurred on the second VOST tube, and not the first.

The VOST analytical laboratory, Triangle Labs was contacted, and these data were reviewed. The most likely scenario is that during calibration of the GC/MS with the Method 8260 calibration "cocktail" that the higher boiling point compounds (>150°C) in the calibration standard, "dropped out" in a "cold", i.e. unheated, portion of the calibration system. Then during subsequent analyses, these compounds, "bled" into the GC/MS, appearing as actual sample "hits". The impact of this occurrence is that the calibration curve would be biased low for the higher boiling point analytes, since the anticipated concentration of an analyte did not make it to the detector. And subsequently, analytical results for the higher boiling point compounds was biased high. This has a double impact in that the mass detected may well have not come from the VOST sample but from the loss of the analyte during the calibration of the GC/MS.

The Modified Method 5/Method 8270 results should be used for these higher boiling point compounds, i.e. >150°C.

5.3.2 D termination of Semivolatile POHCs in Stack Ga

Semivolatile organic compounds in stack gas were determined by analysis using GC/MS Method 8270B. The measurements results are valid and reliable. No significant problems were identified.

The stack gases were sampled for semivolatile organic compounds using the Modified Method 5 sampling train, SW-846 Method 0010, with analysis by SW-846 Method 8270B. Method 0010 calls for the recovery of three separate components: combined XAD and filter, impinger, and solvent rinses. The three components of the Modified Method 5 samples were extracted separately, and the solvent extracts were combined for a single analysis.

The solvent extracts were combined to provide the lowest method detection limit. The mass of any analyte can be distributed among the three components. Although the total mass of the analyte could be above the analytical detection, that mass distributed among the three components may be below the analytical detection limit in each of the three component extracts.

The benefits of analysis of the combined solvent extracts are:

- Higher likelihood to acquire an actual analytical result;
- Provide the lowest method detection limit; and
- Eliminate the need to mathematically combine the results of analysis of three component extracts, i.e. adding the analytical detection limits for three components, or adding one or more analytical results with one or more detection limits.

Laboratory Method Blanks

Laboratory method blanks were analyzed with each batch of trial burn samples. The blank results, presented in the appendices, showed no contamination concern.

Field Blank Train

A field blank train was prepared and analyzed during the trial burn for Test Condition 3. Results for analysis of the train showed no contamination concerns. Results were presented in Section 4.0 with the actual sample results to facilitate blank corrections.

Laboratory Control Samples

Laboratory control sample results associated with Method 8270B analysis of stack gas samples show reliable method performance. Results for the laboratory control sample are presented in the appendices.

Surrogate Spike Samples

Spike sample recoveries show accurate, precise recoveries in all samples. The precision objective of 50% RSD (relative standard deviation) was easily achieved. These results are presented in the appendices.

5.3.3 Determination of Polychlorinated Dibenzodioxins and Dibenzofurans in Stack Gas

Samples of stack emissions collected according to EPA Method 23 were analyzed for polychlorinated dibenzodioxins and dibenzofurans. Quality control data demonstrate effective, accurate analyses. The Method 23 sampling train toluene rinses were recovered and analyzed separately from the other train components.

Method Blanks

Results for analysis of laboratory method blanks indicated no laboratory contamination concerns for PCDD/PCDF analyses. Filters and associated rinses were extracted and analyzed with filter blanks; XAD and associated rinses were analyzed with the XAD blanks. The laboratory blank results are presented in the appendices.

Fi ld Blank Train

A field blank train was collected and analyzed during Test Condition 3. The field blank looks at potential contamination from the overall sampling plus analysis perspective. Results for analysis of the blank samples point to no significant contamination concerns. The field blank train results are summarized in Section 4.0 with the actual sample results to facilitate any blank correction.

Laboratory Control Samples

Laboratory control sample results for Method 23 analyses indicate a well-controlled measurement process. The results, presented in the appendices, show good precision and accuracy.

Internal Standards Spike Samples

Internal standard compounds were added to each sample to determine overall recovery efficiency in each sample. The results are summarized in Table 5-8. As indicated by the recovery data, the measurements were very consistent and showed good recoveries.

5.3.4 Determination of Total Organic Emissions in Stack Gas

Total organic emissions (TOE) in the stack gas were determined during Test Condition 3 using the TOE sampling train outlined in the TOE guidance document. QA/QC activities involved the collection of a field blank and spiking the Tedlar bag with a known analyte concentration to determine recovery efficiency for the C_1 through C_6 compounds.

Field Blank Train

A field blank train was collected during the trial burn for Test Condition 3. The field blank consisted of a Tedlar bag condensate blank (water), nitrogen in a Tedlar bag blank, and a MM5 sampling train blank for TCO and GRAV analyses. The results of the TOE field blank are presented in Section 4.0 with the actual sample results to facilitate blank corrections. The GRAV analysis of the field blank indicated an extremely high background level comparable to the actual sample values. The other portions of the TOE field blank indicates no concerns form background contamination.

Table 5-8
The Dow Chemical Company, Louisiana Operations
Vinyl II Industrial Boiler F-410 Trial Burn — Test Condition 3
Stack Gas — PCDD/PCDF Internal Standard Recoveries

Compound	TLI Blank S976384 (%)	Run 1 (%)	Run 2 (%)	Run 3 (%)	Field Blank (%)
$^{13}C_2$-2378-TCDF	81.3	92.7	98.5	89.4	86.6
$^{13}C_2$-2378-TCDD	68.3	79.3	78.9	70.9	74.2
$^{13}C_2$-PeCDF 123	68.5	77.8	78.8	69.8	67.1
$^{13}C_2$-PeCDD 123	68.6	79.6	77.3	71.6	72.3
$^{13}C_2$-HxCDF 678	74.7	79.2	81.7	75.5	76.4
$^{13}C_2$-HxCDD 678	73.7	80.1	78.6	74.2	77.5
$^{13}C_2$-HpCDF 678	70.4	68.3	69.5	64.0	66.9
$^{13}C_2$-HpCDD 678	88.4	84.4	87.9	92.0	82.3
$^{13}C_2$-OCDD	90.7	75.5	95.7	94.3	88.6

Spiked Sampl

During Run 1 of Test Condition 3, the Tedlar bag of the TOE sampling train was spiked with 100 mL of 2,496 ppmv propane to yield expected concentration of 7.03 ppmv in the final Tedlar bag volume. The measured concentration recovered from the bag was 7.38 ppmv or a recovery percentage of 104.9 percent. Table 5-9 presents these results.

Table 5-9
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 3
TOE Method 0040 Spike Recovery Results

Run Identification	Metered Volume (dsl)	Propane Spike Volume (dsl)	Propane Expected Concentration (ppmv)	Propane Actual Concentration (ppmv)	Recovery (%)
TC3-Run 1	35.533	0.100	7.03	7.38	104.9

5.3.5 Determination of Hydrogen Chloride and Chlorine in Stack Gas

Hydrogen chloride (HCl), chlorine (Cl_2), and particulate matter in stack emissions were determined on a combined EPA Method 0050 sample train. HCl is captured in a sulfuric acid impinger which allow Cl_2 to pass through. The Cl_2 is subsequently trapped in NaOH impingers. Particulate matter is captured on the filter and in the probe/nozzle rinse. HCl and Cl_2 are both determined as chloride using ion chromatography. Particulate matter is determined gravimetrically.

Laboratory Method Blanks

Laboratory method blanks showed no chloride contamination. The method blank results are presented in the appendices.

Field Blank Trains

Field blank trains were collected and analyzed during each test condition to evaluate overall contamination potential. The blank results were all below the detection limits and are presented in the appendices.

5.3.6 D terminati n of Particulate Mass Loading in Stack Gas

The key QC samples for particulate matter determinations was the field blank.

Field Blank Train

Field blank train results for the probe/nozzle rinse and filter were less than 2 mg and did not significantly impact the sample results.

5.4 Waste Feed and Process Samples

An evaluation of data quality for analysis of waste feed and process samples is presented below.

5.4.1 Determination of Volatile Organic Compounds in Waste Feed and Process Streams

Concentrations of volatile organic compounds in waste feed and process samples were determined according to the procedures given in SW846 GC/MS Method 8260. Quality control data indicate that the analyses were performed under controlled conditions, as specified in the methods, and that the analytical methods were effective in the various sample matrices.

Laboratory Method Blanks

Laboratory method blanks were analyzed with each batch of field samples. None of the target compounds were reported above the detection limit. The results are presented in the appendices.

Laboratory Control Samples

Laboratory control sample results for volatile organic compounds are presented in the appendices. These results show consistent, accurate analyses. Precision, expressed as the RPD for the LCS/LCSD pairs, was very good.

Surrogate Spike Samples

Surrogate compounds were spiked in each sample analyzed for volatile organic compounds. The results are presented in the appendices. These results show good, repeatable recoveries in every sample matrix.

Matrix Spik Sampl s

Matrix spike results are presented in the appendices. These results show acceptable recoveries for the Method 8260 criteria.

Duplicate Sample Analysis

Results for duplicate analysis of one sample from Test Condition 2, Run 3, are summarized in Table 5-10. Repeatability of results was good.

Table 5-10
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler R-410 Trial Burn — Test Condition 1, Run 3
Liquid Waste Feed — Duplicate Analysis Results for Volatile POHCs

Compound	Sample Analysis (µg/mL)	Duplicate Analysis (µg/mL)	RPD (%)
Chlorobenzene	15,300	15,600	0.98
Trichloroethane	246,000	245.000	0.20

5.4.2 Determination of Semivolatile Organic Compounds in Waste Feed and Process Streams

Concentrations of semivolatile organic compound (SVOC) in waste feed and process samples were determined during Test Condition 3 according to the analytical procedures described in SW-846 Method 8270B. Following calibration and tuning as specified in Method 8270B, the key estimators of accuracy for SVOCs in the actual sample matrices relates to the assessment of surrogate compound and matrix spike sample analyte recoveries. The routine Method 8270A surrogates were measured. Recoveries for these matrix spike samples was used as another estimator of accuracy.

Results from blank sample analyses was examined in assessing potential sample contamination. The laboratory analyzed analytical method blanks as part of this project.

Laboratory M thod Blanks

Laboratory method blanks were prepared and analyzed with the samples. No SVOCs were detected in any of the laboratory blanks.

Laboratory Control Sample Recoveries

Duplicate laboratory control samples were analyzed with the samples. The LCS/LCSD results demonstrate good method performance, expressed in terms of percent recovery and the relative percent difference (RPD) between duplicate measurements. The recoveries are presented in the appendices.

Surrogate spike recoveries are presented in the appendices. These results demonstrate effective measurement performance for the critical measurement parameters.

Matrix Spike Sample Recoveries

Duplicate samples were fortified with known quantities of standard matrix spikes to assess analyte recovery. Recoveries for each matrix type are presented in the appendices. These results indicate good recovery and repeatability for both the SVOC surrogates in all sample matrices.

5.4.3 Determination of Metals in Waste Feed Samples

Samples of the liquid waste feed were analyzed for eleven metals using SW846 ICPES Method 6010A. Mercury was analyzed by SW846 CVAA Method 7471A and 7470A. The quality control data indicate effective measurement results.

Laboratory Method Blanks

Laboratory method blanks were prepared and analyzed with the samples. The blank results indicated no significant impact on regular sample measurement results. These results are presented in the appendices

Laboratory Control Sampl Recov ri s

Duplicate aliquots control samples were analyzed with each batch of samples. The LCS/LCSD results demonstrated good method performance, expressed in terms of percent recovery of the metals and the relative percent difference (RPD) between duplicate measurements. These results are presented in the appendices.

5.4.4 Determination of Physical and Miscellaneous Parameters in Waste Feed

Quality control data associated with determination of physical parameters indicate reliable and effective analyses.

Duplicate Analyses

Results for duplicate analysis of liquid waste feed samples for chlorine, heating value, and ash are summarized in Table 5-11, showing generally good precision. These results show excellent analytical repeatability, with small RPDs.

Table 5-11
The Dow Chemical Company, Louisiana Operations
Vinyl II, Industrial Boiler F-410 Trial Burn — Test Condition 3, Run 3
Liquid Waste Feed — Duplicate Analysis Results for
Chlorine, Heating Value, and Ash

Compound	Sample Result	Duplicate Result	RPD (%)
Chlorine (%)	65.4	65.9	0.4
Heating Value (Btu/lb)	6,400	6,400	0.0
Ash (wt. %)	<0.01	<0.01	NA

NA — Not Applicable