Executive Summary

Dow Silicones Corporation, a subsidiary of Dow, operates a chemical manufacturing facility in Midland, Michigan. The facility uses a liquid nitrogen cooled cryogenic condenser to control emissions from multiple emission groups. The purpose of this compliance test was to demonstrate compliance with VOC and benzene emission limits as found in table FG304VENTRECOVERY of Renewable Operating Permit (ROP) No. MI-ROP-A4043-2019. Please note the emission limits apply to the outlet of the 304 vent recovery system prior to mixing with any other vents streams. Sampling was conducted in the vent header rather than an exhaust stack

The compliance test measuring emissions of VOC and Benzene emissions was completed on November 19th, 2019. Emissions for both VOC and Benzene were significantly less than their corresponding pound per hour limits. As discussed with Chris Hare and Gina McCann on November 5, 2019, EU502-02 was not running during the test since the process was down and was not expected to be operational until the end of Q1 2020. EU502-02 represents a small amount of emissions treated by the 304 vent recovery system. EGLE requested the emissions from this emission unit be included in the test report as calculated emissions. Therefore, compliance with the VOC and Benzene emission limits has been determined based upon the emission test results and calculated emissions from EU502-02.

AQD has published a guidance document entitled "Format for Submittal of Source Emission Test Plans and Reports" (February 2008). The following is a summary of the emissions test program and results in the format suggested by the aforementioned document.

The results of the test results are summarized in the tables below.

Emission Results VOC and Benzene

Sample Type	Test Method	Sampling Time (Min/Run)	Allowable Emission Rate	Actual Emission Rate*
VOC Emissions (lb/hr)	EPA Method 25A	60	30.0 lb/hr	12.7 + 0.042** = 12.742 lb/hr
Benzene Emissions (lb/hr)	EPA Method 18	60	0.46 lb/hr	< 0.01 + 0.009 = <0.019** lb/hr

* Emissions based on average of three one-hour runs.

**EU502-02 Calculated Emissions on Outlet of 304 Vent Recovery

Sample Type	Calculated Emission Rate
VOC Emissions	0.042 lb/hr
Benzene Emissions	0.009 lb/hr

Testing Run Data

PARAMETER	RUN 1	RUN 2	RUN 3	AVERAGE
Run Date	11/19/2019	11/19/2019	11/19/2019	N/A
Run Times	1430/1530	1620/1720	1736/1836	N/A
Stack Gas Flow Std Cond (scfm)	158.45	192.68	107.11	152.75
Conc. THC as Propane (ppmv)	12641	11994	11609	12081
THC as Propane Emissions (Lb/Hr)	**13.8**	**15.9**	**8.5**	**12.7**
Conc Benzene (ppmv)	3.93	1.71	2.77	2.80
Benzene Emissions (Lb/Hr)	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**

Operational Rates

Run	Run Time	Total Vent Recovery Flow (lb/hr)	HX1 2044 Exit Gas Outlet Temp (Deg C)	HX2 2044 Exit Gas Outlet Temp (Deg C)
Run 1	1430/1530	339	-77	-77
Run 2	1620/1720	416	-80	-80
Run 3	1736/1836	238	-79	-80
Average	**N/A**	**331**	**-79**	**-79**

1. Summary of Test Program/Introduction

Dow Silicones Corporation, a subsidiary of Dow, operates a chemical manufacturing facility in Midland, Michigan. The facility uses a liquid nitrogen cooled cryogenic condenser to control emissions from multiple emission groups. The purpose of this compliance test was to demonstrate compliance with VOC and benzene emission limits as found in table FG304VENTRECOVERY of Renewable Operating Permit (ROP) No. MI-ROP-A4043-2019. Please note the emission limits apply to the outlet of the 304 vent recovery system prior to mixing with any other vents streams. Sampling was conducted in the vent header rather than an exhaust stack.

AQD has published a guidance document entitled "Format for Submittal of Source Emission Test Plans and Reports" (February 2008). The following is a summary of the emissions test program and results in the format suggested by the aforementioned document.

a) Identification, location and dates of tests

A compliance test measuring emissions of VOC and Benzene was completed on November 19th, 2019 on the 304 Vent Recovery System in Midland Michigan.

b) Purpose of testing

The purpose of this test was to demonstrate compliance with the regulations for the 304 Vent Recovery System at Dow Silicones Corporation, a wholly owned subsidiary of Dow in Midland, Michigan. The specific objectives were:

- Determine Benzene and VOC emissions.

c) Brief Description of source

The 304 Vent Recovery System comprised of two interchangers (HX1 2040 and HX2 2040) and two condensers (HX1 2044 and HX2 2044) which operate in series to remove air contaminates from process exhaust. The 304 vent recovery system receives process exhaust from several emission units on-site (i.e., EU502-07, EU508-01 and EURULE290 (EU502-02)). According to the ROP, EU502-01 is allowed to vent to FG304VENTRECOVERY, but it does not currently vent to the 304 vent recovery system. FG304VENTRECOVERY is a CAM subject emission unit subject to the requirements of 40 CFR Part 64. The condensers are CAM subject devices for VOC.

d) Test program contacts

The contact for the source and test report is:

Ms. Jenny Kraut, Air Specialist
Dow
1400 Building
Midland, Michigan 48674
989-496-7133

Names and affiliation of personnel including their roles of the test program is summarized below.

Role	Role Description	Name	Affiliation
Process Focal Point	• Coordinate plant operation during the test • Ensure the unit is operating at the agreed upon conditions in the test plan • Collect any process data required • Provide all technical support related to process operation	Matt Weber	Dow
Environmental Focal Point	• Ensure all regulatory requirements and citations are reviewed and considered for the testing	Brandon Bishop	Dow
Test Plan Coordinator	• Leadership of the sampling program • Develop the overall testing plan • Determine the correct sample methods.	Chuck Glenn	Dow
Test Plan Coordinator Back-up	• Leadership of the sampling program • Develop the overall testing plan • Determine the correct sample methods.	Spencer Hurley	Dow
Technical Reviewer	• Completes technical review of the test data	Michael Abel	Dow
Field Team Leader	• Ensures field sampling meets the quality assurance objectives of the plan	James Edmister	AECOM
Sample Project Leader	• Ensures data generated meets the quality assurance objectives of the plan	Daniel Nuñez	AECOM
Analytical Project Manager	• Oversees laboratory analysis • Ensures data generated meets the quality assurance objectives of the plan	Ashley Miller	Enthalpy

2. Summary of Results

a) Operating Data – See Appendix B for Raw Data

Operational Rates

Run	Run Time	Total Vent Recovery Flow (lb/hr)	HX1 2044 Exit Gas Outlet Temp (Deg C)	HX2 2044 Exit Gas Outlet Temp (Deg C)
Run 1	1430/1530	339	-77	-77
Run 2	1620/1720	416	-80	-80
Run 3	1736/1836	238	-79	-80
Average	**N/A**	**331**	**-79**	**-79**

b) Applicable permit number, State Registration Number (SRN) and Emission Unit ID or designation for the source.

- MI-ROP- A4043-2019
    - FG304VENTRECOVERY

c) Results expressed in units consistent with the emission limitation applicable to the source and comparison with emission regulations

Testing Run Data Benzene and VOC

PARAMETER	RUN 1	RUN 2	RUN 3	AVERAGE
Run Date	11/19/19	11/19/19	11/19/19	N/A
Run Times	1430/1530	1620/1720	1736/1836	N/A
Vent Flow Rate from Process (lb/hr)	339	416	238	331
Molecular Weight (g/mol)	13.7	13.9	14.3	14.0
Vent Flowrate (scfm-wet)	158.45	192.68	107.11	133.14
Conc. THC as Propane (ppmv)	12641	11994	11609	12081
THC as Propane Emissions (Lb/Hr)	**13.8**	**15.9**	**8.54**	**12.7**
Conc. Benzene (ppmv)	3.93	1.71	2.77	2.80
Benzene Emissions (Lb/Hr)	**< 0.01**	**< 0.01**	**< 0.01**	**< 0.01**

VOC limits - 30.0 lbs/hr & 22.5 tons/yr

- Emission limits apply to the outlet of the 304 vent recovery system prior to mixing with any other vent streams. Emissions testing was conducted in the vent header rather than at an exhaust stack.
- The emission rates in the table above do not include emissions from EU502-02. Please refer to the executive summary for these emissions.
- In a violation notice from EGLE dated October 16, 2019, Dow Silicones Corporation (DSC) was cited in non-compliance with the 22.5 ton/yr VOC emission limit. This citation was based on a 2013 emissions test report for FG304VENTRECOVERY. The results of recent testing also indicate non-compliance with the ton/yr VOC limit. As a result, DSC has committed to repermitting to correct the violation. DSC does not believe there is any harm to the environment due to the alleged violation.

Benzene limit – 0.46 lbs/hr

- See comments above.

3. Source Description

a) Description of process, including operation of emission control equipment

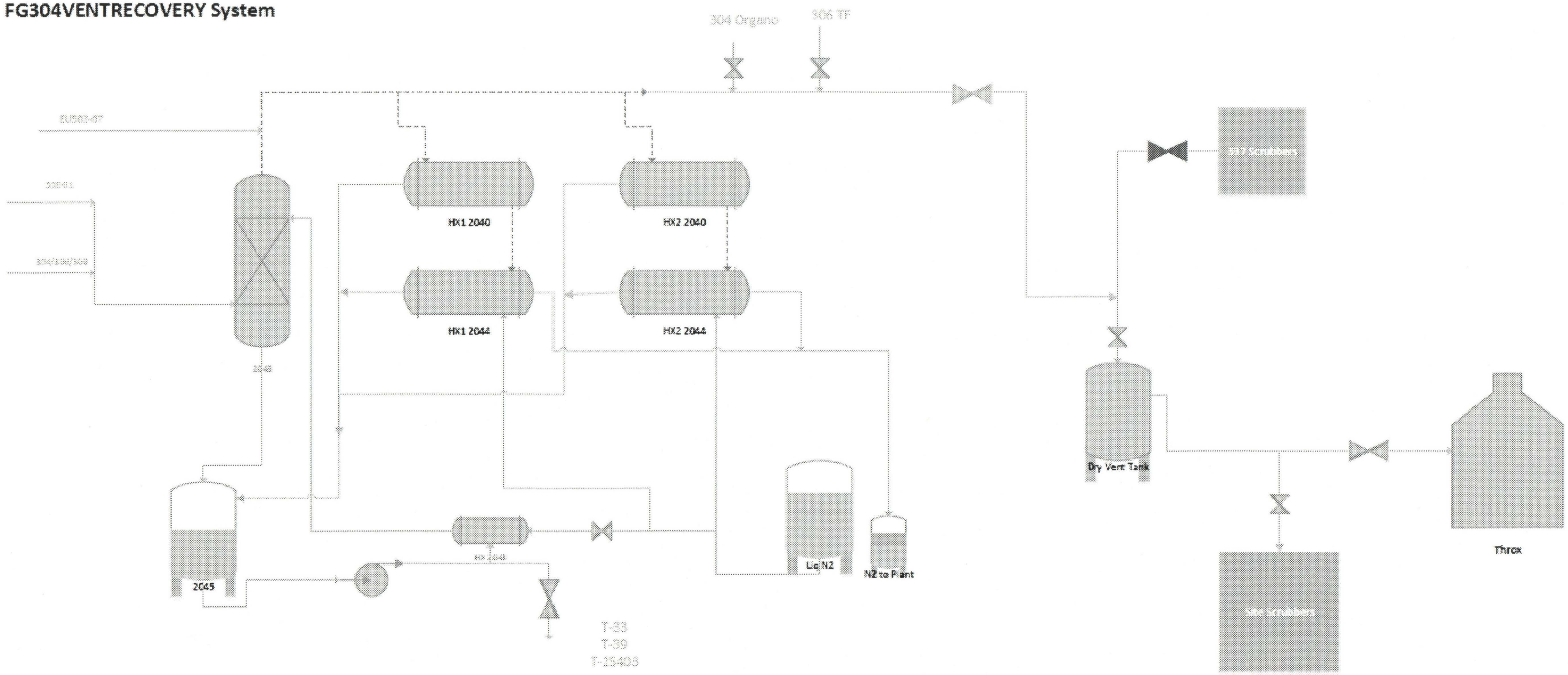
The 304 Vent Recovery System comprised of two interchangers (HX1 2040 and HX2 2040) and two condensers (HX1 2044 and HX2 2044) which operate in series to remove air contaminates from process exhaust. The 304 vent recovery system receives process exhaust from several emission units on-site (i.e., EU502-07, EU508-01 and EURULE290 (EU502-02)). According to the ROP, EU502-01 is allowed to vent to FG304VENTRECOVERY, but it does not currently vent to the 304 vent recovery system. FG304VENTRECOVERY is a CAM subject emission unit subject to the requirements of 40 CFR Part 64. The condensers are CAM subject devices for VOC.

The 304 vent recovery system is comprised of two interchangers (HX1 2040 and HX2 2040) and two liquid nitrogen cooled cryogenic condensers (HX1 2044 and HX2 2044) which operate in series to remove air contaminates from process exhaust.

Sampling was conducted in the vent header rather than an exhaust stack. A Dow micro motion sensor was used to determine exhaust gas flowrates. VOC/benzene samples were collected from separate sample trains at a single stack sampling point.

b) Process flow sheet or diagram

FG304VENTRECOVERY System



c) Type and quantity of raw and finished materials processed during the tests

Total vent recovery flow to THROX was monitored and recorded during testing (FT-23854). The proposed flow rate during testing was 200 – 400 lbs/hr. The measured flow rate during testing is depicted in the table below.

Run	**Run Time**	**Total Vent Recovery Flow (lb/hr)**
Run 1	1430/1530	339
Run 2	1620/1720	416
Run 3	1736/1836	238
Average	**N/A**	**331**

d) Maximum and normal rated capacity of the process

Parameter	Design Maximum Operating Rate	Proposed Operating Rate	Normal Operating Rate
FT-23854	1,000 lb/hr	200 to 400 lb/hr	200 to 1,000 lb/hr

e) A description of process instrumentation monitored during the test

Parameter	Design Maximum Operating Rate	Proposed Operating Rate	Normal Operating Rate
FT-23854	1,000 lb/hr	200 to 400 lb/hr	200 to 1,000 lb/hr
Control Devices Below			
TT-1662_AVE	-76 C	-90 to -76 C	-110 C
TT-25420_AVE	-76 C	-90 to -76 C	-110 C

4. Sampling and Analytical Procedures

a) Description of sampling train(s), field procedures, recovery and analytical procedures

EPA Method 25A was utilized to determine total THC as propane concentrations during each run on the outlet. Based on previous sample data, the VOC make-up is primarily propane (1.7 %), pentane (0.5 %), isobutane (775 ppmvw) and ethylene (492 ppmvw). All are excellent responders to FIA.

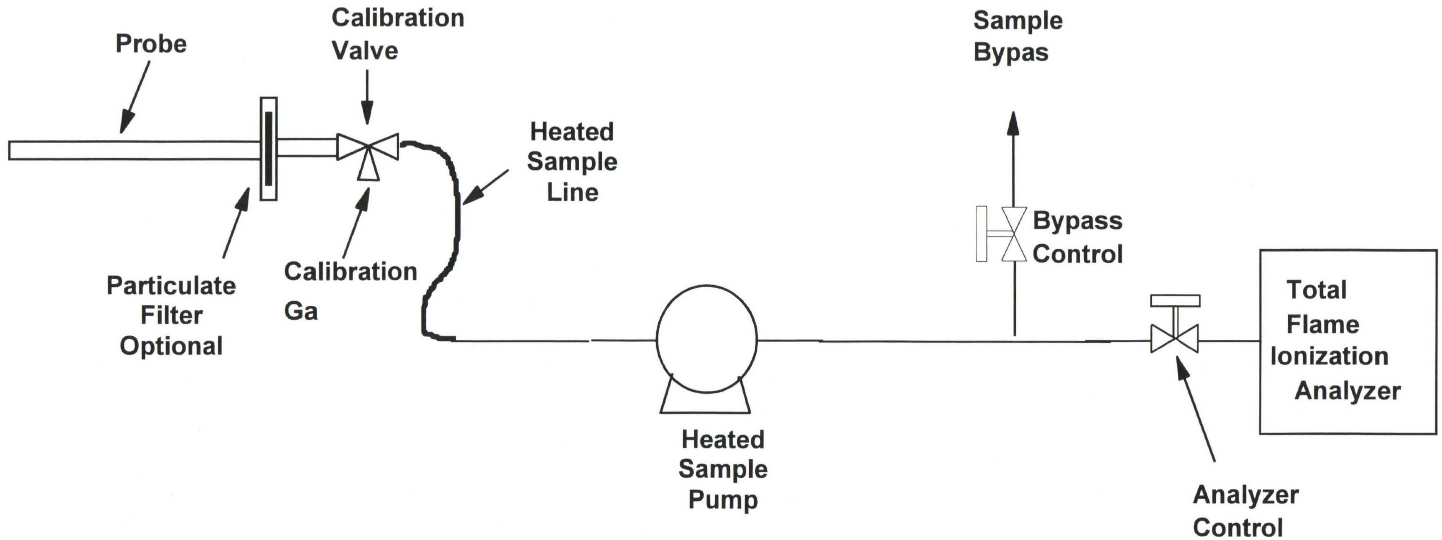
A gas sample was extracted from the source through a heated line to a flame ionization analyzer (FIA). Results are reported as volume concentration equivalent to propane.

Benzene sampling was conducted on the sample points using EPA Method 18 Bag sampling methodology. Three runs of approximately one-hour duration were conducted for each test. *Retained bags from each of the primary bags are either held or shipped separately to the contract lab to reduce the potential of loss of a sample run due to a bag leak during transit.* The primary samples were shipped to a contract lab for analysis (Enthalpy Analytical). The lab followed the spike and recovery requirements found in EPA M18.

Molecular weight sampling was conducted on the sample points using EPA Method 18 Bag sampling methodology. Three runs of approximately one-hour duration were conducted for each test. *Retained bags from each of the primary bags are either held or shipped separately to the contract lab to reduce the potential of loss of a sample run due to a bag leak during transit.* The primary samples were shipped to a contract lab for analysis (Enthalpy Analytical). This approach was used due to the vent stream make-up. The analysis completed was ASTM D1946. This provided concentration make-up of H_2, O_2, N_2, CO, CH_4 and CO_2.

Volumetric flowrate data was generated by Dow using the in-plant micro motion flow detector.

FIGURE 4.2: SAMPLING TRAIN FOR VOC (M25A) - Glass Wool Filter not used



5. Test Results and Discussion

a) Detailed tabulation of results including process operating conditions and flue gas conditions

Detailed results can be found in the executive summary and section 2(c).

b) Discussion of significance of results relative to operating parameter and emission regulations

All pound per hour air permit limits were achieved during sampling. VOC emissions were less than 50% of the pound per hour allowance. Benzene emissions were seen at negligible levels. These results are similar to previous test events.

c) Discussion of variations from normal sampling procedures or operating condition which could have affected the results.

Bag samples were collected in independent bags for analysis. The oxygen and nitrogen results indicate the run 1 bag collected for molecular weight appeared to have a slight leak. Results were corrected to the levels the O2 was for runs two and three. Doing so brought the molecular weight to levels similar to runs two and three and reports at the worst case for VOC and benzene emissions.

d) Discussion of any process or control equipment upset condition which occurred during test

There were no process or control equipment upset conditions during testing.

e) Description of any major maintenance performed on the air pollution devices during the three month period prior to testing

No maintenance has been performed on air pollution devices in the three month period prior to testing.

f) In the event of a re-test, a description of any changes made to the process or air pollution devices since the last test.

N/A

g) Results of any quality assurance audit sample analysis required by the reference method

N/A

h) Calibration sheets for the dry gas meter, orifice meter, pitot tube and any other equipment or analytical procedure that require calibration

N/A

i) Sample calculations of all formulas used to calculate the results

Stack Gas Volumetric Rates

$$Q_{SW} = \left(\frac{Flow\ lb}{hr}\right)\left(\frac{hr}{60\ min}\right)\left(\frac{453.6\ g}{lb}\right)\left(\frac{mol}{MW\ g}\right)\left(\frac{22.4\ l}{mol}\right)\left(\frac{scf}{28.32\ l}\right)$$

Run #1 Example

$$Q_{SW} = \left(\frac{339\ lb}{hr}\right)\left(\frac{hr}{60\ min}\right)\left(\frac{453.6\ g}{lb}\right)\left(\frac{mol}{13.74\ g}\right)\left(\frac{22.4\ l}{mol}\right)\left(\frac{scf}{28.32\ l}\right) = \underline{158.45\ scfm}$$

Analyzer Calibration Error Calculations

The calibration error test consisted of challenging each reference monitor at three measurement points against known calibration gas values. Calibration error for the reference is calculated using the following equation:

$$CE_{RM} = \frac{|Analyzer\ Response - Calibration\ Gas\ Value|}{Span\ of\ Analzyer} \times 100$$

Run #1 Example Low Gas

$$CE_{RM} = \frac{|681.0\ ppmv - 684.4\ ppmv|}{2233.0\ ppmv} \times 100 = \underline{0.7\ \%}$$

System Calibration Bias Calculations

The system bias calibration test consisted of challenging the reference sample system at two measurement points against the local calibration values. Calibration bias calculations for the reference sample system are calculated using the following equation:

$$CB_{RM} = \frac{|System\ Calibration\ Response - Analzyer\ Calibration\ Response|}{Span\ of\ Analzyer} \times 100$$

Run #1 Example Zero Gas

$$CB_{RM} = \frac{|2.6\ ppmv - 0.0\ ppmv|}{2233.0\ ppmv} \times 100 = \underline{0.1\ \%}$$

Calibration Drift Calculations

The calibration drift tests were conducted at the beginning and end of each run. Analyzer maintenance, repair or adjustment could not be completed until the system calibration response was recorded. Calibration drift for the reference is calculated using the following equation:

$$CD_{RM} = \frac{|Final\ System\ Cal\ Response - Initial\ System\ Cal\ Response|}{Span\ of\ Analzyer} \times 100$$

Run #1 Zero Example

$$CD_{RM} = \frac{|3.1\ ppmv - 2.6\ ppmv|}{2233.0\ ppmv} \times 100 = \underline{0.0\ ppmv}$$

System Calibration Drift Correction

The gas concentrations are corrected for the system calibration bias. The concentrations are calculated using the following equations:

$$C_{Gas} = (\overline{C} - C_O)\left(\frac{C_{MA}}{C_M - C_O}\right)$$

where: C_{Gas} = Effluent Concentration, dry ppm or %
$\overline{C}$ = Average Analyzer Concentration, ppm or %
C_O = Average Initial and Final System Calibration Responses for Zero Gas, ppm or %
C_M = Average Initial and Final System Calibration Responses for Upscale Calibration Gas, ppm or %
C_{MA} = Actual Concentration of Upscale Calibration Gas, ppm or %

THC Outlet Emission Rate

$$E_{THC} = \frac{(THC_{Gas})(Q_{SW})(Gas_{MW})(28.32\ L/ft^3)}{(10^6\ ppmv)(24.056\ L/mol)(453.6\ g/lb)}$$

where: E_{THC} = Emission of THC, (lb/hr)
THC_{Gas} = Concentration of THC Gas, $(wet\ ppmv)$
Q_{SW} = Stack Gas Flow @ Std. Conditions, wet basis (scf/hr)
Gas_{MW}= Molecular Weight of Gas $(g/g\ mol)$ Where:
VOC_{MW} = Molecular Weight of VOC as Propane $(44.1\ g/g\ mol)$

Run 1 THC Run 1 Examples

$$E_{THC} = \frac{(12698\ ppmv)(158.45\ scfm)(44.1\ g/mol)(28.32\ L/ft^3)}{(10^6\ ppmv)(24.056\ L/mol)(453.6\ g/lb)} = \underline{13.8\ lb/hr}$$

j) Copies of all the field sheets, cyclonic flow checks including pre-testing, aborted tests and/or repeated attempts

All data sheets can be found in Appendix A.

k) Copies of all laboratory data including QA/QC

All laboratory data can be found in Appendix C.