UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
RESEARCH TRIANGLE PARK, NC 27711

JAN 1 2 2010

OFFICE OF
AIR QUALITY PLANNING
AND STANDARDS

MEMORANDUM

SUBJECT: Correction to the Dow Request for Method Modifications

FROM: Dr. Conniesue Oldham, Group Leader Connie Oldham
Measurement Technology Group (E143-02)

TO: Dr. Kishor Fruitwala, Section Chief
RCRA Facility Assessment Section (6PD-A)

In a memorandum to you dated November 17, 2009, I approved a number of modifications requested by Dow to several EPA test methods. In the memorandum, there was a minor error. One of the modifications requested by Dow, Item 27 in the attached table, was a request to modify EPA Method 0023A. In my reply, I identified this as a modification to EPA Method 23. Attached is a corrected memorandum identifying Item 27 as a modification to Method 0023A and approving the modification as an alternative to the required procedures in Method 0023A.

If you need further assistance, please contact Gary McAlister at (919) 541-1062.

Attachment

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MEMORANDUM

SUBJECT: Dow Request for Method Modifications

FROM: Dr. Conniesue Oldham, Group Leader Connie Oldham
Measurement Technology Group (E143-02)

TO: Dr. Kishor Fruitwala, Section Chief
RCRA Facility Assessment Section (6PD-A)

This is in response to correspondence dated September 1, 2009, from Dow to you asking for approval of a number of sampling and analytical method deviations that have been proposed for use during the upcoming 40 CFR Part 63 Subpart EEE (Hazardous Waste Combustor MACT) Comprehensive Performance Tests of hazardous waste combustors at numerous Dow facilities located in Texas and Louisiana. Based on our discussions with you, we have decided that some of the modifications are not minor and under EPA's delegation of approval, authority should be reviewed by my office. I am attaching a table summarizing all of the requested modifications. The modifications that my office is reviewing are items 7, 10, 13, 14, 15, 16, 17, 18, 19, 21, 25, 27, 28, 29, 30, 33, 34, 35, and 38.

Items 7 and 10 are modifications to the analytical procedures in Method 29 for measuring metal emissions. Item 7 requests that we allow the use of laboratory reagent water rather than American Society for Testing and Materials (ASTM) Type II water as required by the method. The laboratory reagent water is certified to meet the ASTM Type II specifications for electrical conductivity, which is the important parameter for metals analysis, but is not tested to determine if it meets other Type II specifications such as total organic carbon content. We agree that this is an acceptable modification because the laboratory reagent water will meet the pertinent specifications for ASTM Type II water. Item 10 requests that we allow the laboratory to add boric acid to the front half portion of the Method 29 samples after they have been digested in a microwave oven with hydrofluoric acid. The boric acid will sequester the hydrofluoric acid and prevent it from damaging the laboratory glassware. We agree that it is acceptable to add boric acid to the digested samples because it will not adversely affect the Method 29 analytical results.

Items 13, 14, 15, and 16 are modifications to the analytical procedures in Method 6010 for analysis of metal samples from Method 29 by inductively coupled argon plasma. Items 13 and 14 request that we allow the laboratory to adopt the procedure in EPA CLP ILM4.0 for determining the "specified concentration range around the calibration blank" that is required in Section 4.2.8 of Method 6010 as there is no definition or procedure specified in Section 4.2.8. We agree that the procedure in EPA CLP ILM4.0 is an acceptable procedure for defining the "specified concentration range around the calibration blank." Item 15 requests that we allow the laboratory calibration blank to be prepared in a matrix of five percent nitric acid and five percent hydrochloric acid rather than ten percent hydrochloric acid and two percent nitric acid as specified by the method. We agree that a matrix of five percent nitric acid and five percent hydrochloric acid would better represent the range of sample matrices that arise from the preparation procedures in Method 29 than the specified matrix of ten percent hydrochloric acid and two percent nitric acid from Method 6010. Item 16 requests that we allow the laboratory to consider method blanks acceptable if they are below the reporting limit for the analysis rather than below the method detection limit. In the context of the air samples taken for compliance purposes, having method blank values below the laboratory reporting limit is an acceptable alternative to having method blank values below the method detection limit.

Items 17, 18, 19, and 21 are modifications to the analytical procedures in Method 8260B for the analysis of volatile organic compounds by gas chromatography/mass spectrometry. Item 17 requests that we allow the laboratory to use ion 119 as the quantitation ion for the internal standard chlorobenzene-d_5 for the 25-ml purge tests rather than ion 117 as specified in Method 8260. We agree that it is acceptable to use ion 119 as the quantitation ion for the 25-ml purge tests because it will improve the sensitivity of the analysis. Item 18 requests that we allow an absolute retention time window of 0.2 minutes around the expected retention time for components in the sample rather than a relative retention time (RRT) window of 0.06 RRT units. We agree that setting an absolute retention time window of 0.2 minutes is an acceptable alternative because we do not expect that retention times would shift significantly using the columns and conditions specified in Method 8260B. Item 19 requests the use of alternate ions in the mass spectrum of 12 target analytes to identify and measure those target analytes. These alternate ions are either the base ions in the mass spectrum or replace ions that have interferences. We agree that the alternate ions are acceptable alternatives to the specified ions to identify and measure the 12 target analytes. Item 21 requests that we allow the analyst to substitute a single 30-ml aliquot of methylene chloride to rinse the filter sample container or the front-half rinse sample container when transferring the shipped samples for analysis as a substitute for the three 10-ml aliquots specified by the method. While we believe that the three 10-ml aliquots would be better laboratory practice for transferring sample, we agree that a single 30-ml aliquot would provide adequate recovery of the sample and is an acceptable alternative.

Item 25 requests that we allow continuous liquid-liquid extraction of the combined condensate and condensate rinse as an alternative to the separatory funnel extraction specified in Method 3542. We agree that continuous liquid-liquid extraction is a better extraction procedure than using a separatory funnel and is an acceptable modification to Method 3542.

Item 27 is a modification to Method 0023A sampling procedures. It requests that we allow the tester to use acetone and toluene as solvents for rinsing the sample train components to recover any sample on the glassware surface, and that we allow the solvents to be analyzed together as an alternative to using acetone, methylene chloride, and toluene as the recovery solvents with the toluene rinse analyzed separately as specified in Method 0023A. We already have data to indicate that omitting the methylene chloride and combining the toluene rinse with the other solvents will not affect the measured emissions, so this is an acceptable modification to the Method 0023A sampling procedures.

Items 28 and 29 are modifications to the Method 23 sampling procedure. Together they request that we approve the use of acetone and toluene as solvents for rinsing the sample train components to recover any sample on the glassware surface, and that we allow the solvents to be analyzed together as an alternative to using acetone, methylene chloride, and toluene as the recovery solvents with the toluene rinse analyzed separately as specified in Method 23. We already have data to indicate that omitting the methylene chloride and combining the toluene rinse with the other solvents will not affect the measured emissions, so this is an acceptable modification to the Method 23 sampling procedures.

Items 30, 33, 34, 35 and 38 are modifications to the analytical procedures in Method 23. Items 30, 33, 34, and 35 are modifications to the extraction and preliminary clean-up procedures. Because Method 23 uses isotopically labeled internal standards for calibration, and the proposed modification to the extraction and clean-up procedures occurs after the addition of the internal standards, the calibration procedure will automatically compensate for any modifications to these procedures. Therefore, these are acceptable modifications provided that the results of the analysis meet all of the quality assurance limits in Method 23. Item 38 requests that we allow an alternate acceptable ion abundance ratio range for pentachlorodibenzodioxin. The proposed modification is to allow an ion abundance ratio range of 0.55 to 0.75 for the M/M+2 ratio rather than 1.32 to 1.78 for the M+2/M+4 ratio. We agree that it is acceptable to monitor the M/M+2 ratio as an alternative to the M+2/M+4 ratio and that the range of 0.55 to 0.75 is comparable to the range of 1.32 to 1.78.

We believe that these modifications are acceptable for use at any hazardous waste combustor subject to the emission limits in 40 CFR Part 63, Subpart EEE. Therefore, we will announce on EPA's web site (at http://www.epa.gov/ttn/emc/tmethods.html#CatB) that our approval of these modifications is broadly applicable to all hazardous waste combustors.

If you need further assistance, please contact Gary McAlister at (919) 541-1062.

Attachment

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R=750[9]
1. HCl/Cl_2 Analysis Modifications – KNOX-WC005 based on EPA METHOD 26A (TestAmerica, Knoxville, TN[16])											
1. EPA Method 26A states to analyze a calibration curve both before and after sample analysis. The laboratory will perform a single calibration prior to sample analysis, as described in SW-846 Method 9056.	The laboratory will perform a single calibration prior to sample analysis, as described in SW-846 Method 9056.	Intermediate	√	√	√	√	√	√	√	√	√
2. The matrix matching of calibration standards for H_2SO_4 and NaOH impingers described in EPA Method 26A will not be performed. The impinger solutions are diluted and neutralized during sample collection. Therefore, the concentration of the acid and base in the impingers does not match the initial matrix. Matrix matching is therefore not possible in the final matrix.	The impinger solutions are diluted and neutralized during sample collection. Therefore, the concentration of the acid and base in the impingers does not match the initial matrix. Matrix matching is therefore not possible in the final matrix.	Intermediate	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-760[9]
II. Metals Sampling Modifications – EPA METHOD 29 (URS Corporation, Austin, TX[17])											
3. A Teflon® transfer line will be used between the filter and the first impinger of the sampling train.	This is done to address space limitations on the stack, to allow for ease in probe moving and to minimize potential hazards moving the very large and heavy impinger box.	Minor	√	√	√	√	√	√	√	√	√
4. Approximate rinse volumes will be used in the recovery of the Method 29 sampling train versus the exact volumes prescribed in Method 29. The method modifications proposed include: —Use of approximately (versus "exactly") 100 ml ± 25 ml of 0.1 N HNO_3 to rinse the probe nozzle and liner and front half of the filter housing; —Use of approximately (versus "exactly") 100 ml ± 50 ml of 0.1 N HNO_3 to rinse the transfer line, the HNO_3/H_2O_2 impingers and the back half of the filter housing; —Use of approximately (versus "exactly") 100 ml ± 25 ml of 0.1 N HNO_3 to rinse the empty impinger; and —Use of approximately (versus "exactly") 100 ml ± 50 ml of deionized water to rinse the acidic $KMnO_4$ impingers.	EPA Method 29 states that exactly 100 ml of 0.1 N HNO_3 is to be used to recover the probe nozzle and liner and front half of the filter holder; exactly 100 ml of 0.1 N HNO_3 is to be used to recover the first three impingers (a moisture knock-out if used and two impingers containing HNO_3/H_2O_2); exactly 100 ml of 0.1 N HNO_3 is to be used to recover the empty impinger between the two HNO_3/H_2O_2 impingers and the two impingers containing acidic $KMnO_4$; exactly 100 ml of the acidic $KMnO_4$ solution and 100 ml of water to recover the impingers of acidic $KMnO_4$; and 25 ml of 8N HCl if visible deposits remain in the acidic $KMnO_4$ impingers. The rinse volumes in the method are 1) overly prescriptive, and 2) may be inadequate for sample recovery. The rationale for the method specification to use exactly 100 milliliters of nitric acid in recovery is to ease the blank correction of the data. The data will not be blank corrected; therefore the use of exactly 100 milliliters is not necessary.	Minor	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
III. Metals Analysis Modifications – KNOX-MT-0006 based on EPA METHOD 29 (TestAmerica, Knoxville, TN[16])											
5. The digestates from each fraction will be reduced to final volumes that are lower than the volumes specified in EPA Method 29. This is done to achieve lower reporting limits. The concentrations of the reagents will be maintained at the same concentrations as stated in EPA Method 29.	This is done to achieve lower reporting limit. A lower acid volume is added to a lower final volume to keep the acid concentration constant.	Minor	√	√	√	√	√	√	√	√	√
6. The digestate for the front-half fraction uses 0.4% HCl. The use of HCl is not specified in EPA Method 29, but is necessary to achieve maximum mercury recovery during the microwave extraction.	The use of HCl is not specified in EPA Method 29, but is necessary to achieve maximum mercury recovery during the microwave extraction.	Intermediate	√	√	√	√	√	√	√	√	√
7. Laboratory reagent water is used rather than ASTM Type II water.	The laboratory reagent water meets the ASTM criteria for electrical conductivity, but is not tested for all ASTM Type II criteria. The laboratory uses the term reagent water rather than ASTM Type II water since not all ASTM Type II criteria are evaluated. For example, the reagent water is not tested for Total Organic Carbon since this parameter is not tested for Total Organic Carbon since this parameter is not relevant to the analytical method.	Minor	√	√	√	√	√	√	√	√	√
6. The initial measurement of impinger samples from nitric acid/hydrogen peroxide can be made by weighing to ± 0.5 grams or measuring volume to ± 2.5 mL (± 5 mL for large volume samples).	This information is not used by the laboratory to calculate sample results and is recorded on the laboratory benchmark and provided to the client for information only.	Minor	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R=750[9]
(Continued) III. Metals Analysis Modifications – KNOX-MT-0006 based on EPA METHOD 29 (TestAmerica, Knoxville, TN[16])											
9. The samples are not analyzed in duplicate. Instrument and method precision are measured by analyzing the LCS/LCSD.	TestAmerica Knoxville does not require the reporting of separate duplicate analyses for mercury as specified in Section 9.2.3 of EPA Method 29. Method 29 was written allowing the use of an Atomic Absorption Spectrometer with a CVAAS attachment using a BOD bottle for sample analysis. The reference to this older style instrumentation may have required the duplicate analysis of mercury samples due to the use of BOD bottles for sample preparation and analysis. The use of automated instrumentation used for mercury analysis is referenced in section 11.1.3. Note 2. Is states that "Optionally, Hg can be analyzed by using the CVAAS analysis procedures given by some instrument manufacturer's directions. Upon completion of the digestion described in (1), analyze the sample according to the instrument manufacturer's directions. This approach allows multiple (including duplicate) automated analyses of a digested sample aliquot." The use of automated instrumentation allows for multiple automated analyses and processes the results of multiple readings for each sample to provide a final averaged result for mercury. For example, during a ten second period, the instrument takes a reading every 0.1 second for a total of 100 replicate measurements. The average of these replicate readings to used to determine the absorbance and resulting sample concentration.	Minor	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RK1[6]	F410/420[7]	R-4[8]	R-750[9]
(Continued) III. Metals Analysis Modifications – KNOX-MT-0006 based on EPA METHOD 29 (TestAmerica, Knoxville, TN[16])											
	TestAmerica Knoxville provides relevant mercury precision data for the method by performing one of the following depending on the train fraction: LCS/LCSD, MS/MSD or PDS/PDSD.										
10. Boric acid is added after the HF microwave digestion of the front-half samples. Hydrofluoric acid is sequestered by the addition of boric, acid, protecting glassware and instrumentation.	Hydrofluoric acid is sequestered by the addition of boric acid, protecting glassware and instrumentation. Do you have any data showing that the Boric acid addition does not negatively impact the metals analysis? Yes, We have MDL data and Demonstration of capability Data.	Minor	√	√	√	√	√	√	√	√	√
11. The aliquot for mercury analysis of the nitric acid/hydrogen peroxide impingers is taken from the sample after it is reduced in volume to 100 mL. This is done to provide lower reporting limits.	This is done to provide lower reporting limit for mercury by taking an aliquot after sample concentration.	Minor	√	√	√	√	√	√	√	√	√
IV. Metals Analysis Modifications – KNOX-MT-0007 based on SW-846 METHOD 6010B (TestAmerica, Knoxville, TN[16])											
12. Mixed calibration standard solutions are purchased from approved vendors.	For ICP Atomic Emission Spectroscopy, a vendor prepared mixed standard containing the analytes of interest is used to calibrate the instrument rather than using individual solutions of the elements.	Minor	√	√	√	√	√	√	√	√	√
13. SW-846 Method 6010B states that if the correction routine is operating properly, the determined apparent analyte(s) concentration from analysis of each interference solution should fall within a specific concentration range around the calibration blank. In determining inter-element correction factors, the laboratory uses the procedure in EPA CSP ILMO4.0.	In determining IEC's, because of lack of definition in Method 6010B, the laboratory has adopted the procedure in EPA CLP ILM4.0 for the "concentration range around the calibration blank."	Minor	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
(Continued) IV. Metals Analysis Modifications – KNOX-MT-0007 based on SW-846 METHOD 6010B (TestAmerica, Knoxville, TN[16])											
14. ICSA (interference check sample A) results from the non-interfering elements must fall within ± 1 RL from zero. If this is not achieved the field sample data must be evaluated.	This is a clarification of the criteria used by the laboratory since it is not specified in the reference method.	Minor	√	√	√	√	√	√	√	√	√
15. The calibration blank is prepared in 5% nitric acid and 5% hydrochloric acid, rather than 2% nitric acid and 10% hydrochloric acid.	The matrix used by the laboratory provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.	Minor	√	√	√	√	√	√	√	√	√
16. Method blanks are considered acceptable if they are below the reporting limit, rather than the method detection limit.	Method 6010B does not list air as one of the applicable matrices although method 29 references 6010. The method blank criteria have been set to support the reporting limits provided for the air matrix and is qualified to the laboratory MDL.	Minor	√	√	√	√	√	√	√	√	√
V. Volatile POHC Analysis Modifications – KNOX-MS-0015 based on SW-846 METHOD 8260B (TestAmerica, Knoxville, TN[16])											
17. Ion 119 is used as the quantitation ion for chlorobenzene-d_5, for 25-ml purge tests.	This alternate ion is used for quantitation to provide improved relative response of associated target analytes when a larger purge volume is required.	Minor		√	√	√	√		√	√	√
18. A retention time window of 0.2 minutes is used for all components, since these alternate ions data systems do not have the capability of using the relative retention time units specified.	0.2 minutes was chosen to reflect a window comparable to the Method Specified 0.06 RRT window specified in Method 8260B. The use of capillary column chromatography provides reliable and reproducible target analyte retention times	Minor		√	√	√	√		√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
VI. Volatile POHC Analysis Modifications – KNOX-MS-0011 based on SW-846 METHOD 8260B (TestAmerica, Knoxville, TN[16])											
19. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846. The following is a list of the analytes that have different quantitative ions assigned in the laboratory SOP than those ions listed in Method 8260B. The order is presented as analyte name, Method 8260B quantitative ion, and TestAmerica Knoxville quantitative ion, respectively. 1. 1,2-dichloroethane-d4 (surrogate), 102, 65, (65 is the base ion) 2. Trichlorofluoromethane, 151, 101, (151 does not exist, so we use the base) 3. Acetone, 58, 43, (43 is the base) 4. 2-butanone, 72, 43, (43 is the base) 5. Trichloroethene, 95, 130, (interferences with 95) 6. 1,1,2-trichloroethane, 83, 97, (97 is the base) 7. Toluene, 92, 91, (91 is the base) 8. 4-methyl-2-pentanone, 100, 43, (43 is the base) 9. Ethylbenzene, 91, 106, (106 is used to be consistent with xylenes) 10. 1,2-dibromo-3-chloropropane, 75, 157, (157 is the base) 11. Cis-1,4-dichloro-2-butene, 75, 88 (interferences with 75) 12. 1,2,3-trichloropropane, 75, 110 (interferences with 75)	Except where otherwise noted, where the base ion is used, these alternate ions provide for greater sensitivity for the target analyte.	Minor		√	√	√	√		√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
VII. Semivolatile POHC Analysis Modifications – PREP METHOD SW-846 METHOD 3542 (TestAmerica, Knoxville, TN[16])											
20. Rather than spiking the filter in a petri-dish on the bench, the filter will be transferred to the soxhlet extraction apparatus, and all spiking material will be added there.	Spiking the filter in the Petri dish on the bench results in the loss of the more volatile components present in the spike solution for this semivolatile organic analysis. The filter is spiked in the soxhlet apparatus providing improved recovery of the more volatile spiked analytes.	Minor	√								
21. A single 30-mL aliquot of methylene chloride may be used to rinse the Petri dish containing the filter or the container holding the front half rinse; the method specifies the use of 3 rinses with 10 mL.	The same volume of methylene chloride is used with fewer transfers resulting in greater efficiency and equivalent rinsing.	Minor	√								
22. The front-half rinse is not filtered as specified; the use of continuous extraction on the filter and liquid portion of the sample eliminates the need for filtering the particulate material.	The use of continuous extraction on the filter and liquid portion of the sample eliminates the need for filtering the particulate material.	Minor	√								
23. If the sample is not concentrated immediately after extraction, it is stored at 4°C in the dark, and not transferred to another container.	The elimination of an additional container reduces the chance for sample contamination.	Minor	√								

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
(continued) VII. Semivolatile POHC Analysis Modifications – PREP METHOD SW-846 METHOD 3542 (TestAmerica, Knoxville, TN[16])											
24. For extraction of the probe and nozzle rinse, the laboratory will have the flexibility to select whether to raise or lower the pH first.	This modification listed in the laboratory SOP actually concerns the order of pH adjustment of the condensate fraction. As mentioned below, the continuous liquid-liquid extraction provides for improved recoveries of target analytes as the use of this extraction technique provides a longer solvent/sample contact time. The sample pH is adjusted to both a low and a high pH as indicated in the method, and this pH adjustment order works well for this alternate extraction technique.	Minor	√								
25. Continuous liquid-liquid extraction of the combined condensate and condensate rinse is used rather than the separatory funnel extraction specified in the method.	The continuous liquid-liquid extraction technique provides improved recoveries of target analytes.	Minor	√								
26. The final extracts may be concentrated to 1 milliliter before analysis, rather than the 5 milliliters specified in the method.	The use of a 1 mL final volume improves sensitivity of the method.	Minor	√								
VIII. PCDDs/PCDFs Sampling Modifications – SW-846 METHOD 0023A (URS Corporation, Austin, TX[17])											
27. Method 0023A calls for a combination of the acetone, methylene chloride, and toluene rinses in the field. The methylene chloride rinse is being eliminated.	These changes will ease compliance with shipping regulations, and the elimination of methylene chloride is supported by guidance documentation from EPA for the determination of PCDDs/PCDFs by EPA Method 23.	Minor	√	√	√	√	√		√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
IX. PCDDs/PCDFs Sampling Modifications – EPA METHOD 23 (URS Corporation, Austin, TX[17])											
28. EPA Method 23 calls for the use of acetone, methylene chloride, and toluene rinses in the field. Only acetone and toluene will be used to recover the train.	This is considered a minor deviation to a method and is explicitly discussed on EPA's website (www.epa.gov/ttn/emc/methods/method23.html).	Minor						√			
29. EPA Method 23 calls for separate analysis of the toluene rinse fraction. This fraction will be combined with all other fractions for extraction and analysis.		Minor						√			
X. PCDDs/PCDFs Analysis Modifications – SW-846 METHOD 8290 (Vista Analytical Laboratories, El Dorado, CA[18])											
30. No silica gel will be added to the soxhlet apparatus.	Modifications have been made to reduce interference and/or improve sensitivity	Minor	√	√	√	√	√	√	√	√	√
31. All provided solvent rinses are combined concentrated, and added to the resin/filter components before soxhlet extraction.		Minor	√	√	√	√	√	√	√	√	√
32. Prior to clean-up, the extract is dried with sodium sulfate and cleanup recovery standard is added.		Minor	√	√	√	√	√	√	√	√	√
33. The extract is split for archiving. Tetradecane is added to the remaining half and the extract is concentrated to the tetradecane.		Minor	√	√	√	√	√	√	√	√	√
34. The extract undergoes acid/base silica gel column and florisil column clean-up. Basic alumina and AX-21 carbon/celite 545 columns are not used.		Minor	√	√	√	√	√	√	√	√	√

Modification	Justification	Modification Category [15]	Dow Freeport						Dow Plaquemine		
			B-901/2/3[1]	B-824[2]	F-210[3]	FTB-603[4]	F-2820[5]	B-33 RKI[6]	F410/420[7]	R-4[8]	R-750[9]
(continued) X. PCDDs/PCDFs Analysis Modifications – SW-846 METHOD 8290 (Vista Analytical Laboratories, El Dorado, CA[18])											
35. After clean-up, the elate is concentrated, added to a conical vial containing tetradecane and recovery standard solution, and concentrated again to a final volume 20 ul.		Minor	√	√	√	√	√	√	√	√	√
36. The internal standard mix includes ^{13}C-OCDF. The concentration of the ^{13C}C-OCDF is 200 pg/ul. OCDF is quantified against ^{13C}C-OCDF.		Minor	√	√	√	√	√	√	√	√	√
37. The initial calibration curve includes six levels; the concentration of the lowest level is at one half the low point of the curve specified in Table 23-2 of EPA Method 23.		Minor	√	√	√	√	√	√	√	√	√
38. For pentachlorodibenzodioxin, the acceptable range for abundance ratios is as follows: Use M+2/M+4, the theoretical ration is 0.65, and the control limits are 0.55 and 0.75.		Minor	√	√	√	√	√	√	√	√	√

[1] Phase II (Boilers) Units: B-901, B-902, and B-903, Dow, Freeport, Texas.

[2] Phase II (HAF) Unit: B-824, Dow, Freeport, Texas.

[3] Phase II (HAF) Unit: F-210, Dow, Freeport, Texas.

[4] Phase II (HAF) Unit: FTB-603, Dow, Freeport, Texas.

[5] Phase II (Boiler) Unit: F-2820, Dow, Freeport, Texas.

[6] Phase I (Incinerator) Unit: B-33 Rotary Kiln Incinerator, D

[7] Phase II (Boilers) Units: F-410 and F-420 (Vinyls), Dow Plaquemine, Louisiana.

[8] Phase II (Boiler) Unit: R-4 (Glycols), Dow Plaquemine, Lo

[9] Phase II (Boilers) Units: R-750 (Chlorinated Methanes [CMP]), Dow, Plaquemine, Louisiana.

[10] Phase II (Boiler) Unit: B-53, UCC, Texas City, Texas.

[11] Phase I (Incinerator) Unit: VA-5, UCC, Texas City, Texas.

[12] Phase II (Boilers) Units: Unit 6 & 7, Angus, Sterlington, L

[13] Phase I (Incinerator) Unit: MN-460, Dow (formerly Celanese), Clear Lake, Texas.

[14] Phase II (Boiler) Unit: HT-1, Rohm & Haas, Deer Park, T

[15] Deviations labeled as "Minor" have been submitted to EPA Region 6 for approval. Deviations labeled as "Intermediate" have been submitted to EPA RTP for approval and approval was received via other similar facilities in this source category. See http://www.epa.gov/ttn/emc/approalt/ALT053.pdf for a copy of the approval letter.

[16] TestAmerica Laboratories, Knoxville, TN maintains the following certifications, approvals, and accreditations: Louisiana DEQ Cert. #03079 and Texas CEQ.

[17] URS Corporation, Austin, TX (Austin General Engineering) sampling laboratory maintains the following accreditation: Louisiana DEQ Cert. # 84341.

[18] Vista Analytical Laboratories, El Dorado, CA maintains the following certifications, approvals, and accreditations: Louisiana DEQ Cert. #01977 and Texas CEQ T104704189-08-TX.