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ROUTING OF SHOP DRAWINGS, EQUIPMENT DATA, MATERIAL SAMPLES, OR MANUFACTURER'S CERTIFICATES OF COMPLIANCE FOR APPROVAL

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TO:	USEPA REGION III MAIL CODE 3HW22 841 CHESTNUT BUILDING PHILA., PA. 19107	FROM:	OHM REMEDIAL SERVICES CORPORATION 180 MYRTLE STREET LOCK HAVEN, PA 17745	DATE	NOVEMBER 14, 1996
ATTN:	GREG CRYSTAL	ATTN:	TONY GARCIA		

The attached items listed on ENG Form 4025 are forwarded for approval action

CONTRACT NUMBER	DACW45-93-C-0200	CONTRACTOR	OHM REMEDIAL SERVICES CORPORATION
TRANSMITTAL NUMBERS	11D	PROJECT TITLE AND LOCATION	DRAKE CHEMICAL SUPERFUND SITE, LOCK HAVEN, PA.

COMMENTS (Attach additional sheet, if necessary.)

TRIAL BURN PLAN
REVISION - NOVEMBER 1996

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE	DATE
1 TO EPA 4 TO AREA	ANTHONY T. GARCIA QUALITY CONTROL MANAGER	<i>Anthony T. Garcia</i>	

TO:	FROM:	DATE

COMMENTS (Attach additional sheet, if necessary.)

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE

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The following action codes are given to items listed on ENG Form 4025:

ACTION CODES

- A - APPROVED AS SUBMITTED.
- B - APPROVED, EXCEPT AS NOTED ON DRAWINGS. RESUBMISSION NOT REQUIRED.
- C - APPROVED, EXCEPT AS NOTED ON DRAWINGS. REFER TO ATTACHED SHEET. RESUBMISSION REQUIRED.
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- F - RECEIPT ACKNOWLEDGED
- G - OTHER (specify)

ACTION CODES TO BE INSERTED IN COLUMN G, SECTION I, ENG FORM 4025 (Attach sheets, when required.)

ITEM NO. (Taken from ENG Form 4025)	CODE GIVEN	REMARKS

REMARKS

NO. OF INCL.	TYPED NAME AND TITLE	SIGNATURE

**Revisions and Addendums To
Drake Chemical Superfund Site
Trial Burn Plan dated September 20, 1996**

11/12/96

Revisions:

- 1) Fly ash sampling method in Tables 4-1, 4-2, and G4-1b:

Due to periodic generation of fly ash the sampling procedure included in the above tables is not applicable. The samples will be collected at the end of each run by collecting a minimum of nine random grab samples from the fly ash generated during the run and composite those grabs.

Replacement pages 4-6, 4-8, and G-9.

- 2) Stack THC monitor model number in Table 2-3:

Model number and range have been revised to reflect existing equipment.

Replacement page 2-39.

- 3) Modification of Dioxin and Furan extraction procedure to improve recovery of Internal Quantification Standards.

During the first miniburn low internal quantification standard/surrogate (IQS) recoveries were experienced. To improve these recoveries it is recommended that portions of the Trial Burn Report pertaining to analysis of MM5-SV samples in the Trial Burn and Miniburn be changed, by addition of a second extraction of the filter/XAD with toluene after the initial extraction with MeCl_2 .

The reason for recommending this change, which basically adds a second extraction of the filter/XAD with toluene, is to take corrective action to overcome the problem of low IQS recovery found in the D/F analysis of samples collected during the initial miniburn tests done on October 11 and 12, 1996. The IQS recoveries for those samples ranged from 4-24%. Such problems did not occur in the previous Mini-Risk Burn tests.

One important difference between these tests was that in the Mini-Risk Burn the filter/XAD was extracted with toluene (per EPA Method 23), whereas in the Miniburn the filter/XAD were extracted with MeCl_2 (Method 0010). It is suspected that the low IQS recoveries in the Miniburn may be due wholly, or at least in part, to extraction with MeCl_2 . Unfortunately, the extraction with MeCl_2 is necessary for quantitation of SV-POHCs and SV-PICs so it is not appropriate to use only toluene. As shown in the revised

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figure (page 6-3), one-half the MeCl₂ extract would be analyzed for SV-POHCs/PICs. The other half of the MeCl₂ extract would be combined with one-half the toluene extract for the D/F analysis.

This change, in MRI's judgment, offers the most plausible corrective action to improve IQS recoveries in the D/F analysis. It involves additions to the specified procedure rather than any deletions. The proposed procedure is analogous to the offsite air monitoring samples which are first extracted with hexane/ether and then with toluene. Then, one-half of the hexane/ether extract is combined with one-half of the toluene extract and the combined extract is analyzed for PCDD/PCDF.

Replacement pages 6-2, 6-3 (figure 6-1), 6-4, 6-12, and G-38.

4) Risk Burn CrVI sample volume modification.

It is requested that the stack gas sample volume for the BIF Cr⁺⁶ Method be changed to from ~100 ft³ to ~35 ft³.

The above change is required by the high moisture content of the stack gas and the limited capacity of the Teflon impingers in the Cr⁺⁶ sampling train. The high moisture content of the gas results in collection of slightly over 1-L of condensate for each 1.0 dscm (35.3 dscf) of gas sampled. Most of the other MM5 type sampling trains can sample up to 3 dscm (105 dscf) by using a 2-L glass impinger in the train. But, even then, the contents of this impinger must be emptied halfway through each 3-hr run (i.e., at port change).

However, the Cr⁺⁶ sampling method requires specialized all-Teflon impingers, which have a capacity of only 0.225 L. This would require very frequent shutdowns of the sampling equipment to empty impingers and replace the KOH solution if sampling at a rate of 1 dscm/hr, which would increase the time for each run by approximately 2 hr. MRI is therefore requesting that the gas sample volume be changed to 1 dscm (35 dscf) so that the Cr⁺⁶ train impingers need be emptied and recharged only once, during port change.

Reducing the sample gas volume actually has little or no effect on the Cr⁺⁶ detection limit, because only one aliquot of the solution in the impingers is analyzed (by ion chromatography) rather than extraction/concentration of the entire amount of the impinger solution. Because of the high moisture content of the stack gas, sampling higher volumes of gas results in higher volumes of condensate, such that the concentration of Cr⁺⁶ in the solution does not increase with increasing sample gas volume. Thus, little or nothing is gained by sampling larger gas volume (e.g., 3 dscm). Sampling at the higher rate would also make it more difficult to maintain the required pH of 8.5 in the KOH impinger solution (a method requirement).

The Cr⁺⁶ EPA sampling method referenced on page G-27 of the Trial Burn Plan does not

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contain any requirement on the volume of gas that must be sampled. Thus the requested change represents a change in the Trial Burn Plan but not a change in the EPA sampling method.

With this change, MRI would sample a gas volume of about 1 dscm over the same 3-hr sampling period as the other sampling trains, by using a lower gas sampling rate on the Cr⁺⁶ train. As stated earlier, this change is necessitated by the high moisture content of the stack gas and the limited capacity of the Teflon impingers in the Cr⁺⁶ sampling equipment.

Replacement page G-10 (Table G4-1b.)

Addendums:

A) Calibration of Continuous Emission Monitors (CEMs)

To clarify acceptable calibration limits for the CEMs an Appendix H is attached.

Addendum A

Calibration of Continuous Emission Monitors (CEMs)

New Appendix H.

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APPENDIX H

CEM System Calibration Acceptable Limits

Table H-1 provides the acceptable calibration limits for the CEMs when calibration gases are introduced at the probe tip of the sampling system.

Table H-1
CEM Calibration Requirements

Measured Constituent	Instrument Range	Nominal Cal Gas Concentrations	Allowable System Bias or System Accuracy % of Instrument Range	Source of System Accuracy/Bias Ranges	Allowable System Accuracy (ppm or % of measured constituent as appropriate)
CO	0 to 500 ppm	Low - 0 Mid - 250 High - 400	+/-5 % of span	40CFR60, Appendix A, Method 10, Section 4.2.	25 ppm Carbon Monoxide
CO2	0 to 20%	Low - 0 Mid - 10 High - 16	+/-5 % of span	40CFR60, Appendix A, Method 3A, Section 4. Which refers to Method 6C Sections 4.1 through 4.4	1% Carbon Dioxide
O2	0 to 25%	Low - 0 Mid - 12 High - 20	+/-5 % of span	40CFR60, Appendix A, Method 3A, Section 4. Which refers to Method 6C Sections 4.1 through 4.4	1.25% Oxygen
NOx	0 to 1000 ppm	Low - 0 Mid - 250 High - 500	+/-5 % of span	40CFR60, Appendix A, Method 7E, Section 4. Which refers to Method 6C Sections 4.1 through 4.4	25 ppm NOx (Due to high span gas being nominally 500 ppm the 5% was applied to span gas rather than instrument range.)
THC	0 to 400 ppm	Low - 0 Mid - 125 High - 200	+/-5 % of span	40CFR60, Appendix A, Method 25A, Section 5.3.	20 ppm THC

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Revision 1

Fly Ash Sampling

Replacement pages 4-6, 4-8, and G-9.

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Table 4-2. SUMMARY OF SAMPLING AND ANALYSIS PARAMETERS AND METHODS FOR TRIAL BURN

Sample	Sampling frequency for each run	Sample method/size	Analytical parameters	Preparation method ^a	Analytical method ^a
Solid Waste Feed	One grab sample every 15 min composited and split into 3 samples for each run (See note)	Scoop (S007)/ ~ 50 g per grab	Total Solids/ Moisture Content	NA	ASTM D-3173
			Ash Content	NA	ASTM D-3174
			BTU	NA	ASTM D-2015
			Chloride (total)	NA	ASTM E-442 or ASTM D-808/D4327
			Metals ^b	Acid digestion (SW-846-3050A or 3051)	ICP or AA (SW-846 6010A or 7000 series as needed to meet detection limits)
			SV-POHCs ^c	Solvent Extraction (SW-846 3540)	GC/MS (SW-846 8270)
Metal Spike and POHC Spike Material	One grab sample during trial burn	Grab from spike bag, selected at random	NA	NA	Archive
			Bottom Ash		
Fly Ash	Two separate grab samples every 30 min, composited into two separate samples. Each composite sample split into 4 samples for analyses (See note)	Scoop (S007)/ ~ 100 g per grab	SV-POHC ^c , SVOCs ^d	Solvent Extraction (SW-846 3540)	GC/MS (SW-846 8270) and GC/ECD (SW846-8151)
			TOC	NA	SW-846 9060
			VOCs ^d	Dispersion/Purge and Trap (SW-846 8260)	GC/MS (SW-846 8260)
			TCLP Metals ^b	TCLP (EPA Method 1311) Acid digestion per SW-846 3010A/3020A	ICP or AA (SW-846 6010A or 7000 series as needed to meet detection limits). CVAA for Hg (SW-846 7470A).
			SV-POHC ^c , SVOCs ^d	Solvent Extraction (SW-846 3540)	GC/MS (SW-846-8151)
Fly Ash	Two separate grab samples every 30 min, composited into two separate samples. Each composite sample split into 4 samples for analyses (See note)	Scoop (S007)/ ~ 100 g per grab	TOC	NA	SW-846 9060
			VOCs ^d	Dispersion/Purge and Trap (SW-846 8260)	GC/MS (SW-846 8260)
			TCLP Metals ^b	TCLP (EPA Method 1311) Acid digestion per SW-846 3010A/3020A	ICP or AA (SW-846 6010A or 7000 series as needed to meet detection limits). CVAA for Hg (SW-846 7470A).

(continued on next page)

NINE GRAB SAMPLES COLLECTED FROM THE FLY ASH PILE DEPOSITED DURING THE RUN,

Table G4-1b. RISK BURN SAMPLING AND ANALYSIS PROTOCOLS

Sample	Sampling Frequency	Sample Method	Analytical Parameters	Preparation Method	Analysis Method
Waste Feed	Sampling and analysis to be done by OHM in accordance with procedures in the CQMSP	per CQMSP	per CQMSP (See Tables G4-1c and G4-1d)	per CQMSP	per CQMSP (See Table G4-1c)
Combined Klin, Cyclone and SCC ashes	One grab sample every 30 min, composited. The composite sample will be split into 5 separate samples for analysis.	Scoop (S007) ~ 50 g/grab sample	Volatiles (See Table G4-2)	Dispersion Purge and Trap (SW846-8260)	GC/MS SW846-8260
			Semivolatiles (See Table G4-3)	Solvent Extraction (SW846-3540)	GC/MS SW846-8270
			β -Naphthylamine		HRGC/HRMS with SIM
			Herbicides (See Table G4-4)	Per SW846-8151	GC/ECD SW846-8151
Fly Ash	One grab sample every 30 min, composited. The composite sample will be split into 5 separate samples for analysis. <i>Nine grab samples collected from the flyash pile deposited during the run.</i>	Scoop (S007) ~ 50 g/grab sample	TCLP Metals (See Table G4-5)	EPA 1311 extraction. Acid digestion per SW846-3010A/3020A	ICP or AA SW846-6010A or 7000 series. CVAA for Hg (SW846-7470A)
			Dioxins and Furans (See Table G4-6)	Per SW846-8290	HRGC/HRMS SW846-8290
			Volatiles (See Table G4-2)	Dispersion Purge and Trap (SW846-8260)	GC/MS SW846-8260
			Semivolatiles (See Table G4-3)	Solvent Extraction (SW846-3540)	GC/MS SW846-8270
Scrubber Water	One grab sample every 30 min, composited to produce one sample per run.	Sample tap	β -Naphthylamine		HRGC/MS with SIM
			Herbicides (See Table G4-4)	Per SW846-8151	GC/ECD SW846-8151
			TCLP Metals (See Table G4-5)	EPA 1311 extraction. Acid digestion per SW846-3010A/3020A	ICP or AA SW846-6010A or 7000 series. CVAA for Hg (SW846-7470A)
			Dioxins and Furans (See Table G4-6)	Per SW846-8290	HRGC/HRMS SW846-8290
			TCLP Metals (See Table G4-5)	EPA 1311 extraction. Acid digestion per SW846-3010A/3020A	ICP or AA SW846-6010A or 7000 series. CVAA for Hg (SW846-7470A)

Revision 2

Stack THC Monitor

Replacement page 2-39

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Table 2-3 (Continued)

Parameter	Instrument manufacturer or equivalent	Model No. and instrument type	Instrument range	Instrument accuracy	Calibration procedures	Calibration frequency
Stack CO ₂	Rosemount or equivalent	880A or equivalent (NDIR)	0%-15%	1% of span	Set and verify zero with CO ₂ free gas standard. Set instrument span with known gas standard. Set span to equal gas standard concentration. Verify instrument linearity with a midrange gas standard. During calibration, the last recorded measurement of stack gas will be sent to the DCS (for the purpose of calculating rolling average).	Daily
Stack NO _x	Rosemount or equivalent Thermo Environmental	951A (chemillu-minescent) 10AR (chemillu-minescent)	0-500 ppm	1% of span	Set and verify zero with NO _x free gas standard. Set instrument span with known gas standard. Set span to equal gas standard concentration. Verify instrument linearity with a midrange gas standard. During calibration, the last recorded measurement of stack gas will be sent to the DCS (for the purpose of calculating rolling average).	Daily
Stack THC	Rosemount or equivalent	702 400A or equivalent (FID)	0-250 400 ppm	1% of span	Verify instrument linearity with a midrange gas standard. During calibration, the last recorded measurement of stack gas will be sent to the DCS (for the purpose of calculating rolling average). Set and verify zero with hydrocarbon free gas standard. Set instrument span with known propane standard. Set span to equal gas standard concentration. Verify instrument linearity with a midrange gas standard. During calibration, the last recorded measurement of stack gas will be sent to the DCS (for the purpose of calculating rolling average).	Weekly

Revision 3

Dioxin/Furan Extraction Procedure

Replacement pages 6-2, 6-3 (figure 6-1), 6-4, 6-12, and G-38.

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1.1 MM5-SV Samples

Upon receipt in the laboratory, the MM5-SV samples will be removed from the insulated containers and will be placed in cold storage (<4°C). The MM5-SV samples will be in the following fractions:

1. Filter
2. XAD-2 resin trap
3. Methanol/methylene chloride rinsate
4. Toluene rinsate
5. Condensate (impinger contents)

Figure 6-1 presents a diagram of the analytical scheme to which MM5-SV sampling train samples are subjected. The particulate filter and XAD-2 resin will be combined for extraction, first with methylene chloride (MeCl₂), and second with toluene. The MeOH/MeCl₂ rinses and condensate will be extracted with MeCl₂ and combined with the first MeCl₂ extract of the filter/XAD. Likewise, the toluene rinse (after volume reduction) will be combined with the above extracts. This combined extract will be reduced in volume by K-D or Rotovap and split in half. One half will proceed through analysis for SV POHCs/PICs as shown in Figure 6-1. The other half will be combined with half the toluene extract for the second extraction of the filter/XAD, for PCDD/PCDF analysis.

Only the combined particulate filter/XAD-2 resin will be spiked with surrogate compounds. The SV-POHC surrogates used will be *d*₈-naphthalene and *d*₄-1,4-dichlorobenzene. The PCDD/PCDF surrogates are ¹³C-labeled tetra- through octa-CDDs and ¹³C-labeled tetra- through hepta-CDFs, as specified in EPA Method 23. Solvent extraction methods for MM5 sampling trains are consistent with SW-846 Method 0010 with the exception of the sampling train condensates, which are extracted according to Method 3510 (or 3250, if emulsions are present). All extracts are combined for analysis rather than separately analyzing the extract from each component as specified in EPA Method 0010 because of the cumulative result of increasing the detection limit. It is for this reason that only the filter/XAD will be spiked with the POHC and PCDD/PCDF surrogates to

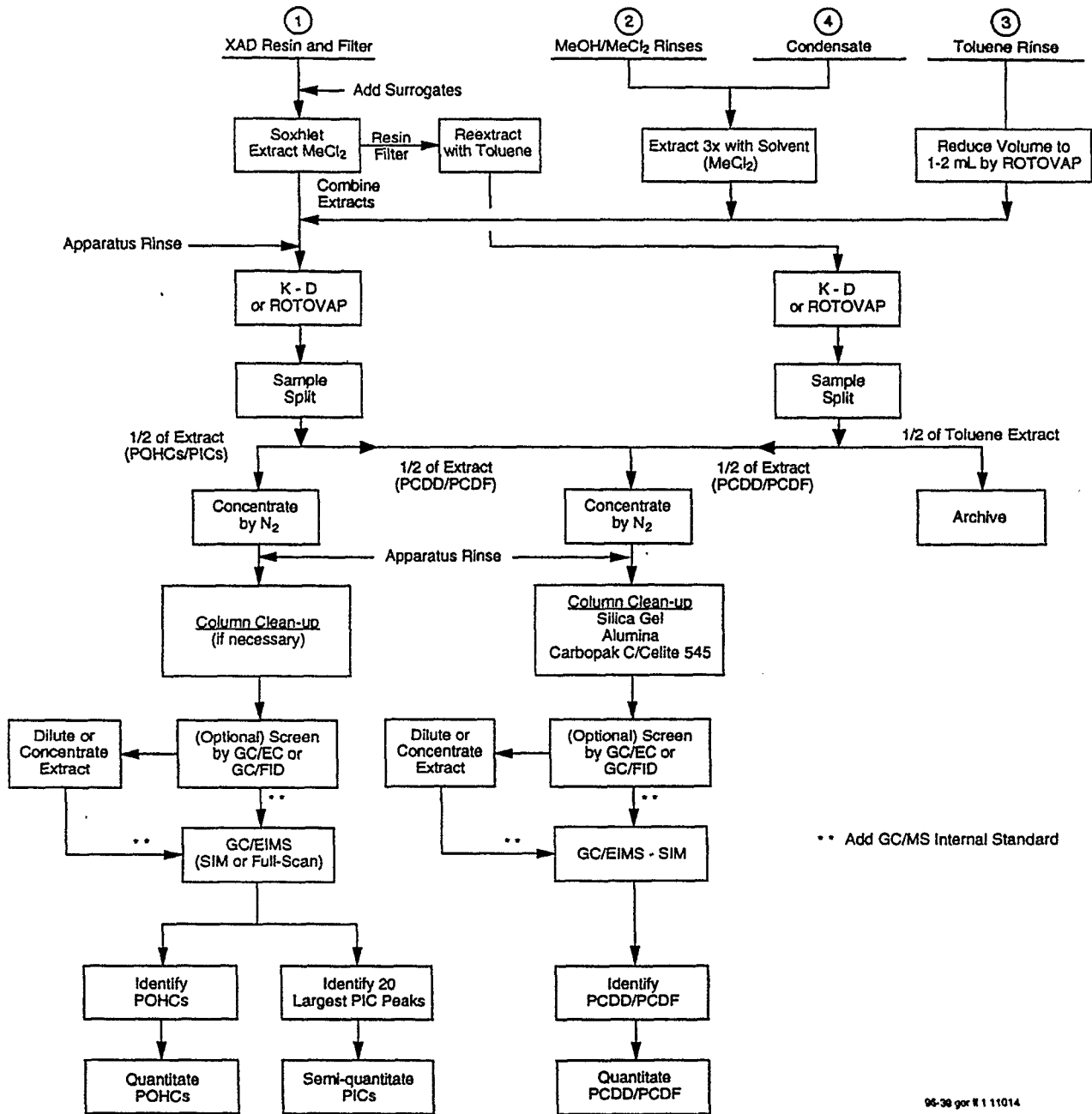


Figure 6-1. POHC, PIC, and PCDD/PCDF analysis scheme for MM5-SV train components.

determine their recovery efficiency, since most if not all of the analytes are expected to be captured in the filter/XAD.

MM5-SV sampling train extract composites will be split, with one-half of the MeCl₂ extract being analyzed for the SV-POHCs and SV-PICs. The other half of the MeCl₂ extract will be combined with one-half the filter/XAD toluene extract for PCDD/PCDF analysis.

Composited extract analysis for the SV-POHCs and PICs will be conducted by SW-846 Method 8270. Although these samples generally do not need to undergo column cleanup, if GC/MS analysis indicates that there are significant interferences present in the sample extract, the extracts will be subjected to a silica gel column cleanup (consistent with SW-846 Method 3630). Composited extracts for analysis of PCDD/PCDFs will be subjected to the cleanup scheme described in EPA Method 23 and analyzed by HRGC/HRMS (high resolution gas chromatography/high resolution mass spectrometry), also per EPA Method 23, with a few minor modifications as listed in Table 6-1.

6.1.2 Metals Emission Samples (MM5-MM)

The probe/front-half nitric acid wash and filter fractions for the MM5-MM sample trains from all tests will be submitted for metal analysis along with the impinger samples. Field reagent blanks will be submitted, combined, and analyzed in the same manner as the train samples. These samples will be analyzed for As, Be, Cd, Cr, and Pb.

Figure 6-2 presents a schematic of the analytical scheme for the metals samples from the MM5-MM train.

Surrogates appropriate for the analytes in each matrix will be spiked into the samples prior to extraction/analysis.

Labeled surrogates (d_8 -naphthalene and d_4 -1,4-dichlorobenzene) will be used for the SV-POHC and SV-PIC analysis of MM5 samples. Unlabeled surrogates may be used on other types of samples (e.g., soil feeds) when appropriate. In these cases, the appropriate unlabeled surrogates will be used, or one sample of each type will be spiked with the actual analyte at 3 times the expected concentration to assess recovery.

6.2.1.1 Modified Method 5 Sample Analysis

Analysis for MM5 SV-POHCs and SV-PICs will be by GC/MS using a 30-m DB-5 fused silica capillary column. MRI normally carries out analysis methods in accordance with SW-846 procedures which includes Method 0010 that specifies separate analysis of the component extracts. This will be modified by combining all the component extracts for one analysis. Therefore, only the XAD-2/filter will be spiked with labeled surrogate compounds prior to extraction. Combining all the components for a single analysis yields a single result and avoids adding together the results for individual components, which may be below the detection limit. Another modification is that XAD/filter will be re-extracted with toluene after the first MeCl_2 extraction. One-half of each of these extracts will be combined for PCDD/PCDF analysis as was described in Section 6.1.1.

6.2.1.2 Analysis of Ash Samples for SV-POHCs, SVOCs, and VOCs

Ash sample extracts will be analyzed for the SV-POHCs and SVOCs selected for this trial burn by EPA Method 8270 with the same GC/MS column as

G.6.1.2 Analysis of MM5-SV Samples

The MM5-SV samples will be analyzed for the semivolatile organic compounds listed in Table G4-8a (which includes β -naphthylamine) and for pesticides (Table G4-8b). This will be done using the methods and procedures described in Sections 6.1.1 and 6.2.1.1 of the Trial Burn Plan, with the exception that the MM5-SV samples will not be analyzed for PCDD/PCDF because a separate EPA Method 23 train will be used for that purpose as described below in Section G.6.1.3. Thus the MM5-SV samples will not be re-extracted with toluene as was discussed in Section 6.1.1.

In addition, as part of the GC/MS analysis for semivolatile organics, the 20 largest peaks will be determined. Those peaks which are not compounds in Table G4-8a will be tentatively identified and semiquantitated. In this regard, it must be noted that tentative identification means, in some cases, that a specific compound cannot be identified. For example, the peak might be identified only as "C₈ alkane".

G.6.1.3 Analysis of MM5-D/F Samples

The MM5-D/F samples will be analyzed for the congeners listed in Table G4-6 using EPA Method 23 procedures as described in Sections 6.1.1 and 6.2.2 of the Trial Burn Plan, including minor exceptions given in Table 6-1. Total PCDD/PCDF will also be determined.

G.6.1.4 Analysis of MM5-PCB Samples

The MM5-PCB samples, collected in accordance with EPA Method 23 sampling procedures, will be prepared and cleaned up using EPA Method 23 analysis (e.g., deletion of alumina cleanup step that removes PCBs). Samples will be analyzed by EPA draft Method 1668, modified (expanded) to include quantitation of all PCB homolog groups, as listed in Table G4-10. This HRGC/HRMS method provides low detection limits as shown in Table G4-10.

G.6.1.5 MM5-MM Train Analysis for Metals

MM5-MM samples will be analyzed for the metals listed in Table G4-7a. This analysis will be done using the procedures, and modifications, described in Sections 6.1.2 and 6.2.6 of the Trial Burn Plan but will include analysis of the permanganate impingers for Hg (see Section G5.1.2).

Revision 4

Risk Burn CrVI Sample Volume

Replacement page G-10 (Table G4-1b.)

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Table G4-1b (Continued)

Sample	Sampling Frequency	Sample Method	Analytical Parameters	Preparation Method	Analysis Method
Stack Gas	3-hr composite per test run	Method 0050 ~ 100 ft ³	Particulate	Desiccation	Gravimetric (EPA Method 5)
	3-hr composite per test run	Draft Method 29, ~ 100 ft ³	HCl/Cl ₂ Metals (See Table G4-7a)	NA	Ion Chromatography
	3-hr composite per test run	BIF C ¹⁶ Method	Hexavalent Chromium (See Table G4-7b)	Preconcentration (BIF Guidance)	IC/PCR (BIF Guidance)
	3-hr composite per test run	Method 0010, ~ 100 ft ³	Semivolatiles +20 (Including β-naphthylamine) and pesticides (See Tables G4-8a and G4-8b)	Soxhlet extraction	GC/MS and GC/MS-SIM
	Four-trap pair at 20 min per pair	VOST Train A SW846-0030	Volatiles +20 (See Table G4-9)	Thermal desorption (SW846-5041)	SW846-5041 GC/MS full scan
	Three trap pair at 20 min per pair (Train B)	VOST Train B SW846-0030	Volatile (See Table G4-9)	Thermal desorption (SW846-5041)	SW846-5041 GC/MS-SIM
	3-hr composite per test run	Method 23	Dioxins/Furans (See Table G4-6)	per Method 23	GC/MS per Method 23
	3-hr composite per test run	per Method 23	PCBs (See Table G4-10)	Modified Method 23	GC/MS per Method 166B
	Continuous by MIRI	Heated Teflon Line	SO ₂	per Method 6C	per Method 6C
	Continuous by OHM	Heated Teflon Line	CO, NO _x , THC	OHM's PYROX 8212 CEM system will be used to provide this data	NA