



Project Summary

Validation of Emission Sampling and Analysis Test Method for PCDDs and PCDFs II

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The precision and accuracy of the Modified Method 5 sampling and analysis protocols for polychlorinated dibenzo-*p*-dioxins and dibenzofurans in municipal waste combustor stack gas have been determined. This was accomplished using a dynamic spiking system designed to continuously deliver stable isotopic PCDD/PCDF congeners into the MM5 sampling train upstream of the particulate filter during sampling of MWC stack gas. The results from this study indicate that the MM5 sampling train provides quantitative and reproducible measurements of dioxins and furans under the conditions used in this study. Accuracy of the measurements as determined using recovery of the dynamically spiked compounds ranged from 77.6% to 117%. Precision of the measurements was high as evidenced by low relative percent differences between replicate trains. The distribution of native dioxins and furans within the sampling trains was essentially the same as for the dynamically spiked components. The dynamic spiking system is a useful tool in the determination of the accuracy of the measurements. It should be considered as a viable alternative to determine method accuracy during trial burns. It does not affect the determination of native dioxins and furans and does not significantly impact on analytical costs.

This Project Summary was developed by EPA's Atmospheric Research and Exposure Assessment

Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Municipal incineration of waste, coupled with energy recovery, is a practice that is gaining favor in current efforts directed at finding economic, effective and efficient alternatives to land burial. The U.S. Environmental Protection Agency has undertaken to regulate municipal waste incinerators and to characterize their emissions in order to allay public concerns about their emissions. Among pollutants of such concern are the polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs).

A Modified EPA Method 5 (MM5) sampling train, incorporating an ice-water chilled condenser and XAD-2 resin cartridge, has been used to collect medium volatility organic compounds from stack gases for a wide range of combustion sources. The configuration and operating procedures for this system in determining PCDDs and PCDFs in municipal waste combustor (MWC) emissions were standardized by a workshop sponsored by the American Society of Mechanical Engineers, the U.S. EPA, and the U.S. Department of Energy held in September, 1984. The product of the workshop was a draft sampling and analysis procedures document known as the ASME protocol. Recently, the EPA Office of Solid Waste has written a method for sampling and

analysis of semivolatile organic compounds from stationary source emissions. This method is referenced as SW-846 Method 0010.¹

The ASME protocol, and more recently SW-846 Method 0010, have become industry standards for sampling stack gases for PCDDs, PCDFs, PCBs, and many other semivolatile organics. However, low recoveries for specific PCDD isomers spiked into MM5 sampling trains were observed during pilot and field evaluation studies conducted under contract to U.S. EPA's Environmental Monitoring Support Laboratory (EMSL). Since these observations were based on limited experiments, additional data were needed to determine the precision and accuracy of the MM5 system for collecting and recovering PCDD and PCDF emissions.

The overall objective of the current study was to characterize the precision and accuracy of the MM5 sampling system using stable isotopically labeled PCDD and PCDF compounds spiked during MWC flue gas sampling.

Procedures

The general experimental approach was to measure the recoveries of specific ¹³C-labeled PCDD and PCDF congeners dynamically and statically spiked into MM5 trains during incineration stack gas sampling. Selected labeled congeners representing tetra- through octachloro PCDD and PCDF homologs were spiked onto the XAD-2 resin prior to each test (static spike) and continuously throughout the test (dynamic spike). Following completion of each test, the samples were recovered from the trains and analyzed for the spiked compounds and for the native PCDDs and PCDFs. Recoveries were determined for each spiked compound by comparing the amount measured against the amount spiked.

The incinerator selected for this study was a mass burn waste combustor burning municipal refuse. Electrostatic precipitators were used to control particulate emissions. Refuse was fed into a reverse reciprocating stoke grate. In addition to the motion of the grates, the underfire grate assisted in further agitation of the waste. The unit was operated without auxiliary fuel. A total of

26 test runs (including two blank trains sampling ambient air) were conducted during six test days. Ports 1 and 2 were the left- and right-hand liners in one dual probe located at the inlet of the electrostatic precipitator. Ports 3 and 4 were in the second dual probe located at the outlet of the precipitator. Sampling was conducted simultaneously at inlet and outlet sampling ports. The two trains in each probe had closely matched flow rates, while the two pairs were sampled at flow rates about 30% apart.

The MM5 sampling train consists of four main sections (Figure 1): a nozzle/probe assembly (front half), a heated filter assembly with a cyclone for trapping particulates, an ice water-chilled condenser for trapping moisture (back half), and an XAD-2 resin cartridge. During collection of MWC flue gases, the trains were not traversed but rather set up as single, average velocity points. Each of the two average velocity points sampled concurrently were considered to replicate sampling velocity locations.

Each sampling train was leak-checked prior to collection of the samples. The stack gases were sampled for 240 minutes. The dynamic spiking system injectors were started 30 minutes after the start of the test and operated for a total of 180 minutes. The MM5 trains were disassembled, each section was capped, and the components taken to a clean area. Each appropriate section of the sampling train was rinsed sequentially with acetone and toluene and the rinsates stored in amber glass bottles with PTFE-lined screw caps. Particulates (from the cyclone and filter) were stored in bottles and wetted with toluene. The XAD cartridge was sealed.

The field static spiking solution, ¹³C-1,2,3,7,8-PeCDF was added to the XAD-2 resin prior to each test. The dynamic spiking solution containing five ¹³C-labeled PCDD/PCDFs (¹³C-1,2,3,4,7,8-HxCDF, ¹³C-OCDF, ¹³C-1,2,3,4-TCDD, ¹³C-1,2,3,7,8,9-HxCDD, and ¹³C-OCDD) was continuously added throughout the test using a dynamic spiking system designed by MRI (Figure 2). The tests were conducted using two spiking levels: 25 and 100 ng. Each level was spiked in duplicate at each sampling location on two of the test days and once on the other test days. The test matrix is shown in Table 1.

Each component of the MM5 sampling train was individually spiked with a Method Internal Standards mix (i.e., surrogates, ¹³C-2,3,7,8-TCDF, ¹³C-2,3,7,8-TCDD, ¹³C-1,2,3,7,8-PeCDD, and ¹³C-1,2,3,4,6,7,8-HpCDD) prior to solvent

extraction. All solvent extractions were conducted using toluene as the extraction solvent. The solid samples (i.e., XAD-2 resin and combined particulates/filter) were Soxhlet-extracted for 16 to 22 hr. XAD-2 resin extracts with free water and all front-half and back-half glassware rinses were back-extracted using reagent grade water. The sample extracts were passed through sodium sulfate to remove residual water. Each extract was rotoevaporated to a final volume of 1-2 mL. A small volume (25 μ L) of tridecane keeper was added to each extract and then further concentrated using a nitrogen evaporator. Hexane was added to the extracts to a final volume of 1 mL.

A 1 x 10 cm tapered column was slurry-packed in hexane with 1 g of silica gel followed by 4 g of 40% (w/w) sulfuric acid modified silica gel. A second column (1 x 30 cm) was also slurry-packed, but with 6 g of acid alumina followed by 1 g of sodium sulfate, also in hexane. The two columns were set such that the silica gel column drained directly into the acid alumina column. The sample extract was applied to the silica gel column and drawn into the packing together with three rinses of the concentrated extract container. The silica gel column was eluted with 45 mL hexane into the alumina column, which was then eluted with 20 mL hexane. A solution of 20% (v/v) dichloromethane/hexane was used to elute the alumina column (20 mL). Finally, 20 mL of a 50% (v/v) dichloromethane/hexane solution was used to elute the column. The three fractions were collected separately and archived. The 20% dichloromethane/hexane fraction was concentrated to 2-3 mL, transferred to a Reaction vial, amended with 25 μ L of tridecane, and the volume reduced to 25 or 100 μ L, depending on the field spiking levels. This cleaned up extract was spiked with a Recovery Internal Standard solution (¹³C-1,2,3,6,7,8-HxCDD) and analyzed for PCDD/PCDF by HRGC/HRMS.

A Carlo Erba MFC-500 gas chromatograph was fitted with a 60-m x 0.25 μ m i.d. DB5 fused silica capillary column using helium carrier gas (20-40 cm/sec). A Grob-type injector in the splitless mode at 270°C was used to inject a 1 μ L portion of the sample extract. After 2 min. at 200°C, the temperature in the GC was increased to 220°C (5°C/min), held for 16 min, increased again to 235°C (5°C/min) and held for 7 min, and finally increased to 330°C (5°C/min). The capillary column was threaded directly into the source chamber of a Kratos MS-50 high

¹ Test Methods for Evaluating Solid Wastes, Physical/Chemical Methods (SW-846), 3rd Edition, Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, DC, 1986.

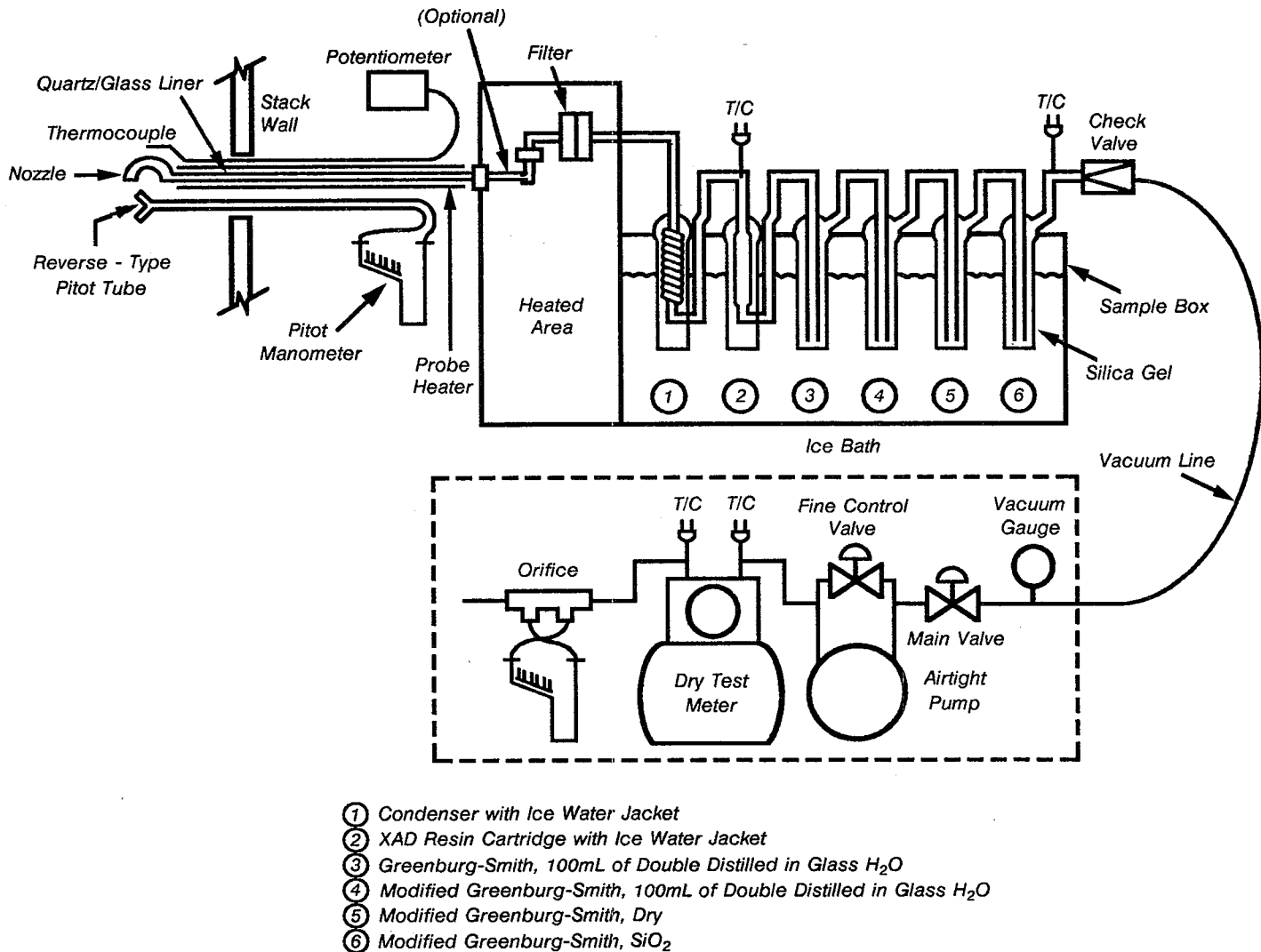


Figure 1. MM5 Sampling train configuration used during the study.

resolution mass spectrometer (transfer line temperature of 280-300°C) scanning from 202-472 amu in one sec or less. The resolution on the mass spectrometer was set to 3,000. All samples and standards were analyzed using the selected ion monitoring (SIM) mode. A three-point calibration curve was analyzed and relative response factors calculated. Daily calibration checks were performed. Table 2 presents the ions monitored during data acquisition.

Results and Discussion

Static Spike Recovery

Results for the recovery of the static spike of a ¹³C-labeled congener added to

the XAD-2 resin prior to sample collection are presented in Table 3. The recoveries of the compound statically spiked onto the XAD-2 resin were somewhat disappointing in the light of the recoveries observed during previous studies.² It is believed that the low recoveries observed, especially for Runs 28 and 29, were anomalous and, in light of the results presented below, do not represent an accurate view of the

²Midwest Research Institute (MRI). 1989. "Validation of Emission Test Methods for PCDDs and PCDFs," Final Report on WA No. 23 prepared for Jimmy C. Pau, Atmospheric Research and Exposure Assessment Laboratory, U.S. EPA, Office of Air Quality Planning and Standards.

sampling accuracy of the MM5 sampling train.

Dynamic Spike Recovery (Inlet)

Table 4 presents the percent recovery of the compounds dynamically spiked at 25 ng into the sampling trains at the inlet sampling ports (Runs 28, 29, 30 and 31). The average of all total recovery values for the individual congeners spiked into each of the sampling trains analyzed was 95.1% (n=30, 28% RSD). This average value was obtained from two sets of replicate sampling trains and two sets of individual, non-replicate sampling trains. The overall average indicates that the sampling train quantitatively collected the

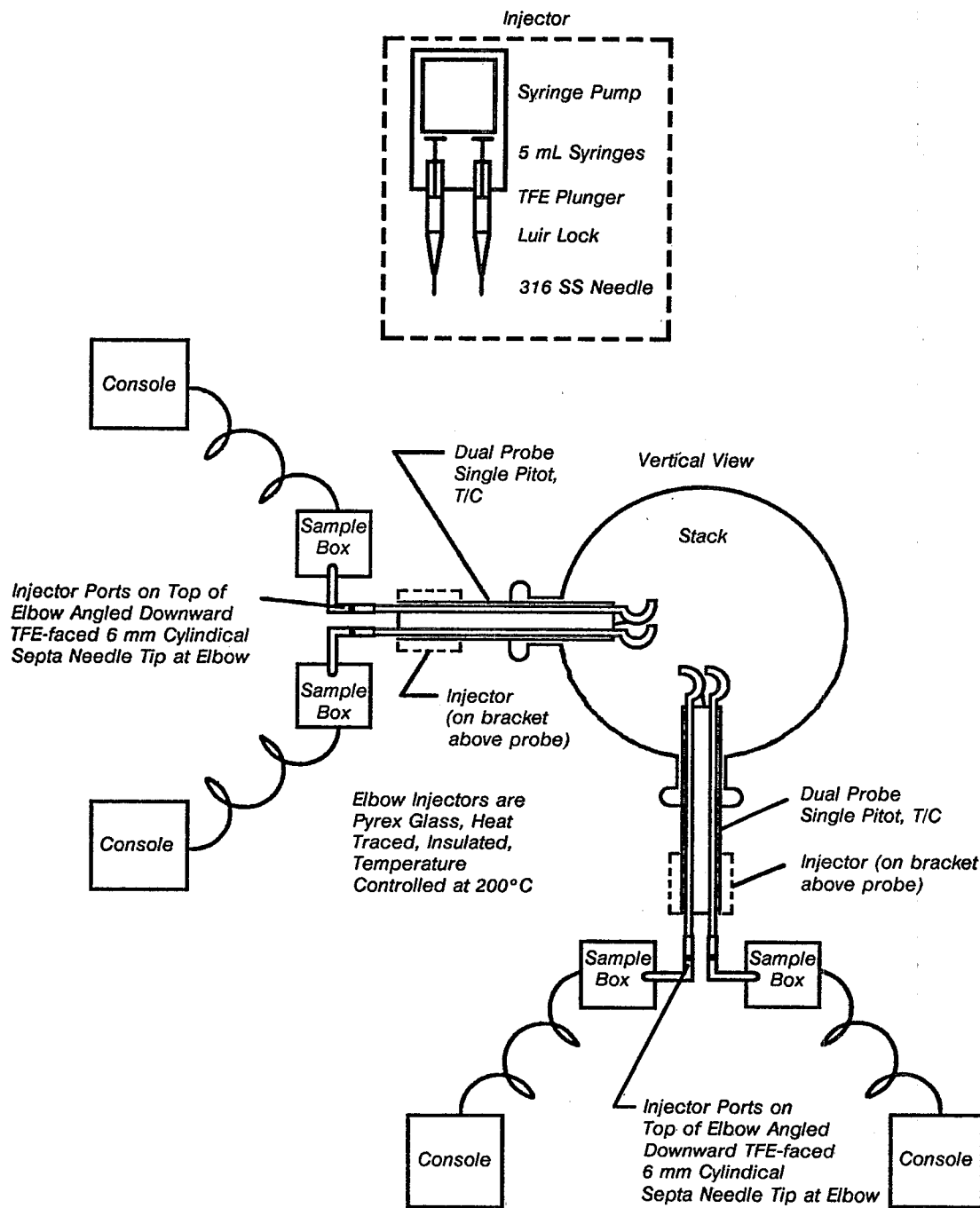


Figure 2. Dynamic spiking system configuration relative to the MM5 sampling train.

dynamically spiked compounds added during the sampling runs.

Runs 28 and 29 were conducted using replicate sampling trains at both inlet ports. Evaluation of the replicate data from a qualitative point of view suggests

that for Run 28, reproducibility was good, but for Run 29, the recoveries from Port No. 1 were about one-half of that seen for Port No. 2. It is suspected that during Run 29, the syringe used for dynamic spiking into Port No. 1 leaked at the Luer-Lok fitting, thereby directing an

unascertained portion of the dynamic spike into the sampling train. This could not be confirmed from field observations, but is a plausible explanation of the results obtained. This conclusion is supported by the results generated from the analysis of native dioxins and furans.

Table 1. Test Matrix for Field Sampling, Runs 28 to 33

Test Day	Run No.	Location				Blank
		Inlet ^a		Outlet ^b		
		Port No. 1	Port No. 2	Port No. 3	Port No. 4	
1	28	L ^c	L	L	L	None
2	29	L	L	L	L	Outlet
3	30	L	H ^d	L	H	None
4	31	H	L	H ^e	L ^e	None
5	32	H	H	H	H	Inlet
6	33	H	H	H	H	None

^a The individual MM5 sampling trains at Ports No. 1 and 2 of the inlet sampling location represent replicate trains sampling the flue gas simultaneously.

^b The individual MM5 sampling trains at Ports No. 3 and 4 of the outlet sampling location represent replicate trains sampling the flue gas simultaneously.

^c L indicates that the dynamic and static spiking levels for this run were at the low level (i.e., 25 ng).

^d H indicates that the dynamic and static spiking levels for this run were at the high level (i.e., 100 ng).

^e These dynamic (but not the static) spiking levels were inadvertently switched by the field crew. Thus, Port No. 3 was spiked at the low level, and Port No. 4 was spiked at the high level. The effects of this overview were negligible.

Table 5 presents the results of the recovery of the labeled compounds dynamically spiked into the inlet sampling trains at 100 ng (Runs 30, 31, 32, and 33). The average of all total recovery values for the individual congeners spiked into each sampling train was 77.6% (n=30, 24% RSD). This average value was obtained from two sets of replicate sampling trains and two sets of individual, non-replicate sampling trains. The overall average for the sampling trains dynamically spiked at 100 ng was approximately 20% lower than those spiked at 25 ng.

Dynamic Spike Recovery (Outlet)

The percent recovery results for compounds dynamically spiked at 25 ng into the MM5 trains in the outlet sampling ports are presented in Table 6 (Runs 28, 29, 30 and 31). The average of all total recovery values for the individual congeners spiked into each of the sampling trains analyzed was 112% (n=30, 12% RSD). This average value was obtained from two sets of replicate sampling trains and two individual, non-replicate sampling trains. The overall average indicates that the sampling trains quantitatively collected the dynamically spiked compounds added during the sampling runs.

Table 7 shows the recoveries of the labeled compounds dynamically spiked into the outlet sampling trains at 100 ng. The average of all sums of recovery values for these sampling trains was 117% (n=30, 13% RSD). This average value was obtained from two sets of replicate sampling trains and two individual, non-replicate sampling trains. The overall average indicates that the sampling trains quantitatively collected the dynamically spiked compounds added during the sampling runs.

Native Dioxins and Furans

Figures 3 and 4 present the distribution of the dynamically spiked components and native dioxins and furans collected during all of the runs for this study. The bars represent the normalized overall average of all compounds collected in all sampling trains for each separate component of the MM5 trains. The distribution of spiked components and for the native dioxins and furans for the inlet sampling locations are essentially the same, with the distribution heavily favoring the filter assembly. The distribution in the outlet sampling trains for the spiked compounds and for the natives are also very similar, but in this case, the distribution favored the filter assembly and the XAD-2 trap almost equally. This is expected, since particulate loading at the outlet sampling

port is decreased, thereby allowing compounds in the vapor phase to break through to the back part of the sampling train. However, based on the high percentage of compounds captured in the filter assembly, it appears that particulate loading at the outlet was relatively high for this set of sampling runs. Particulate material in the flue adsorbs the gas phase dioxins and furans (native or spiked) when it impinges on the filter assembly, thereby effectively trapping these compounds in the filter assembly of the MM5 sampling train.

Statistical Analysis

A number of analysis of variance models were run on the data generated during this study. The variables used to conduct these tests were run number (demonstrative of day-to-day variations), dynamic spike level, sampling location, replicate train recoveries, and specific dynamically spiked compounds. Several analysis of variance models were considered and, after each model, the factors or combination of factors which did not have a significant effect on the result were removed and an additional model fitted.

To test the null hypothesis that the MM5 sampling trains did not produce reproducible (precise) results, the dynamic spike recovery was used as the dependent variable. Differences between

Table 2. Specific PCDDs and PCDFs Analyzed and Corresponding Ions Monitored During HRGC/HRMS Analysis

Compound	Ion (relative intensity)		
	Primary	Secondary	Ratio Range
<u>Statically spiked compound</u>			
¹³ C-1,2,3,7,8-PeCDF	351.9005(100)	349.9035(61)	0.49-0.73
<u>Dynamically spiked compounds</u>			
¹³ C-1,2,3,4,7,8-HxCDF	385.8615(100)	387.8586(87)	0.66-0.98
¹³ C-OCDF	455.7806(100)	453.7386(87)	0.70-1.04
¹³ C-1,2,3,4-TCDD	333.9344(100)	331.9373(76)	0.61-0.91
¹³ C-1,2,3,7,8,9-HxCDD	401.8564(100)	403.8535(82)	0.66-0.98
¹³ C-OCDD	471.7755(100)	469.7785(87)	0.70-1.04
<u>Native PCDD/PCDFs</u>			
2,3,7,8-TCDF	305.8987(100)	303.9016(76)	0.61-0.91
2,3,7,8-TCDD	321.8936(100)	319.8965(76)	0.71-0.91
1,2,3,7,8-PeCDF	339.8597(100)	337.8627(61)	0.49-0.73
1,2,3,7,8-PeCDD	355.8546(100)	353.8576(61)	0.49-0.73
2,3,4,6,7,8-HxCDF	373.8207(100)	375.8178(82)	0.66-0.98
1,2,3,7,8,9-HxCDD	389.8156(100)	391.8127(82)	0.66-0.98
1,2,3,4,6,7,8-HpCDF	407.7817(100)	409.7787(98)	0.78-1.18
1,2,3,4,6,7,8-HpCDD	423.7767(100)	425.7737(99)	0.79-1.19
OCDF	443.7398(100)	441.7428(87)	0.70-1.04
OCDD	459.7347(100)	457.7377(87)	0.70-1.04
<u>Surrogates (method internal standards)</u>			
¹³ C-2,3,7,8-TCDF	317.9395(100)	315.9424(76)	0.61-0.91
¹³ C-2,3,7,8-TCDD	333.9343(100)	331.9373(76)	0.61-0.91
¹³ C-1,2,3,7,8-PeCDD	367.8954(100)	365.8984(61)	0.47-0.73
¹³ C-1,2,3,4,6,7,8-HpCDD	435.8175(100)	437.8145(99)	0.79-1.19
<u>Internal standard (recovery internal standard)</u>			
¹³ C-1,2,3,6,7,8-HxCDD	401.8564(100)	403.8535(82)	0.66-0.98

the aggregate of all replicate sampling trains were found to be nonsignificant ($P=0.103$). This was also true for all but one of the individual replicate runs (Run 29). The factor that was found to have the most influence on sampling precision was sampling location (i.e., inlet or outlet), and day-to-day variability, the specific dynamically spiked compound, and the interaction between run and sampling location had lower but approximately equal influence. These variables accounted for 74% of the variability observed. These observations indicate that for any given day, a pair of MM5 sampling trains sampling a particular portion of a stack effluent for any given compound will produce results of acceptable precision.

The precision of the dynamic spike sampling and analysis for each compound was estimated and is expressed as the pooled standard deviation of the difference in recovery between each set of replicates:

- ¹³C-HxCDF 12.3%
- ¹³C-OCDF 7.01%
- ¹³C-TCDD 8.27%
- ¹³C-HxCDD 7.18%
- ¹³C-OCDD 5.77%

Thus, for ¹³C-HxCDF, the difference between replicate sampling trains is estimated to have a standard deviation of 12.3% recovery about two-thirds of the time.

The absolute difference between the dynamic spike recoveries and 100% was used as the dependent variable for fitting additional analysis of variance models. Run number, sampling location, replication, and the interaction between run and sampling location significantly affected the results in 40% of the measurements. The variability in the balance of the measurements may be attributed to random fluctuations, unknown variables, or to a skewed distribution of the measurements. If no variability had occurred, the predicted difference between 100% and the actual

dynamic spike recovery was estimated to be 18% (i.e., 82 to 118% recovery). This range increased to somewhat less than 30% when all other factors influencing variability were considered.

Conclusions and Recommendations

The results generated during this study indicate that the MM5 sampling train provides quantitative and reproducible measurements of dioxins and furans under the conditions used in this study. Accuracy of the measurements as determined using dynamic spike recoveries ranged from 77.6% to 117%. Precision of the measurements was high as evidenced by low relative percent differences between replicate sampling trains (with one exception) during the measurement of dynamically spiked dioxins and furans. The distribution of the native and dynamically spiked dioxins and furans within the sampling trains was essentially the same.

Table 3. Percent Recovery of Statically Spiked ¹³C-PeCDF on XAD Resin Component of MM5 Sampling Trains.

Run No.	Spike Level (ng)	Sampling Location	Sampling Port	Percent Recovery on XAD Resin
28	25	Inlet	1	55.1
			2	34.8
		Outlet	3	57.3
			4	38.8
29	25	Inlet	1	51.4
			2	48.8
		Outlet	3	57.0
			4	63.3
30	25	Inlet	1	52.2
	100		2	78.5
	25	Outlet	3	66.1
	100		4	78.3
31	100	Inlet	1	73.8
	25		2	70.9
	100 ^a	Outlet	3	85.8
	25 ^a		4	75.8
32	100	Inlet	1	76.7
			2	86.3
		Outlet	3	74.5
			4	67.1
33	100	Inlet	1	75.8
			2	77.3
		Outlet	3	80.3
			4	80.8

^a The dynamic spiking levels on these samples were inadvertently switched during the test run, but the static spike levels were as planned.

The dynamic spiking system is a useful tool in the determination of the accuracy of the measurements. It should be considered as a viable alternative to determine sampling accuracy and precision during trial burns. Some additional validation may be required to determine if the dynamic spiking system causes the spontaneous *in situ* formation of additional PCDDs and PCDFs by comparing replicate trains, one of which has the dynamic spiking system.

It is recommended that during trial burns, selected MM5 trains be equipped with the dynamic spiking system in order to assess the accuracy of the measurements. This modification should have minimal impact on sampling and analysis costs.

Table 4. Percent Recovery of Dynamically Spiked PCDDs and PCDFs Spiked at 25 ng at the Inlet Sampling Locations

Run No.	¹³ C-Labeled Analyte	Average Total Recovery (%)	Relative Percent Difference
28 ^a	HxCDF	125	16
	OCDF	108	2.0
	TCDD	126	19
	HxCDD	92.6	11
	OCDD	111	4.0
29 ^a	HxCDF	80.1	76
	OCDF	69.5	64
	TCDD	72.7	50
	HxCDD	64.2	64
	OCDD	71.8	73
30 ^b	HxCDF	83	NA
	OCDF	109	NA
	TCDD	102	NA
	HxCDD	74.4	NA
	OCDD	95.3	NA
31 ^b	HxCDF	137	NA
	OCDF	122	NA
	TCDD	94.9	NA
	HxCDD	92.0	NA
	OCDD	126	NA

^a Duplicate sampling trains were collected and analyzed for Runs 28 and 29.

^b Single sampling trains were collected and analyzed for Runs 30 and 31.

Table 5. Percent Recovery of Dynamically Spiked PCDDs and PCDFs Spiked at 100 ng at the Inlet Sampling Locations

Run No.	¹³ C-Labeled Analyte	Average Total Recovery (%)	Relative Percent Difference
30 ^a	HxCDF	102	NA
	OCDF	80.4	NA
	TCDD	91.9	NA
	HxCDD	83.2	NA
	OCDD	95.1	NA
31 ^a	HxCDF	114	NA
	OCDF	103	NA
	TCDD	99.7	NA
	HxCDD	97.7	NA
	OCDD	120	NA
32 ^b	HxCDF	70.9	30
	OCDF	52.5	6.0
	TCDD	65.5	11
	HxCDD	58.1	23
	OCDD	60.9	11
33 ^b	HxCDF	80.7	2.0
	OCDF	61.6	1.0
	TCDD	84.7	5.0
	HxCDD	63.8	2.0
	OCDD	71.8	1.0

^a Single sampling trains were collected and analyzed for Runs 30 and 31.

^b Duplicate sampling trains were collected and analyzed for Runs 32 and 33.

Table 6. Percent Recovery of Dynamically Spiked PCDDs and PCDFs Spiked at 25 ng at the Outlet Sampling Locations

Run No.	¹³ C-Labeled Analyte	Average Total Recovery (%)	Relative Percent Difference
28 ^a	HxCDF	124	16
	OCDF	102	2.0
	TCDD	121	7.0
	HxCDD	105	17
	OCDD	105	3.0
29 ^a	HxCDF	140	4.0
	OCDF	96.7	9.0
	TCDD	124	1.0
	HxCDD	113	3.0
	OCDD	106	6.0
30 ^b	HxCDF	115	NA
	OCDF	104	NA
	TCDD	124	NA
	HxCDD	102	NA
	OCDD	120	NA
31 ^b	HxCDF	127	NA
	OCDF	153	NA
	TCDD	139	NA
	HxCDD	118	NA
	OCDD	165	NA

^a Duplicate sampling trains were collected and analyzed for Runs 28 and 29.

^b Single sampling trains were collected and analyzed for Runs 30 and 31.

Table 7. Percent Recovery of Dynamically Spiked PCDDs and PCDFs Spiked at 100 ng at the Outlet Sampling Locations

Run No.	¹³ C-Labeled Analyte	Average Total Recovery (%)	Relative Percent Difference
30 ^a	HxCDF	124	NA
	OCDF	102	NA
	TCDD	113	NA
	HxCDD	105	NA
	OCDD	118	NA
31 ^a	HxCDF	127	NA
	OCDF	87.2	NA
	TCDD	114	NA
	HxCDD	98.8	NA
	OCDD	102	NA
32 ^b	HxCDF	119	24
	OCDF	109	22
	TCDD	105	14
	HxCDD	107	10
	OCDD	127	15
33 ^b	HxCDF	118	1.0
	OCDF	108	2.0
	TCDD	114	6.0
	HxCDD	103	2.0
	OCDD	118	0.0

^a Single sampling trains were collected and analyzed for Runs 30 and 31.

^b Duplicate sampling trains were collected and analyzed for Runs 32 and 33.

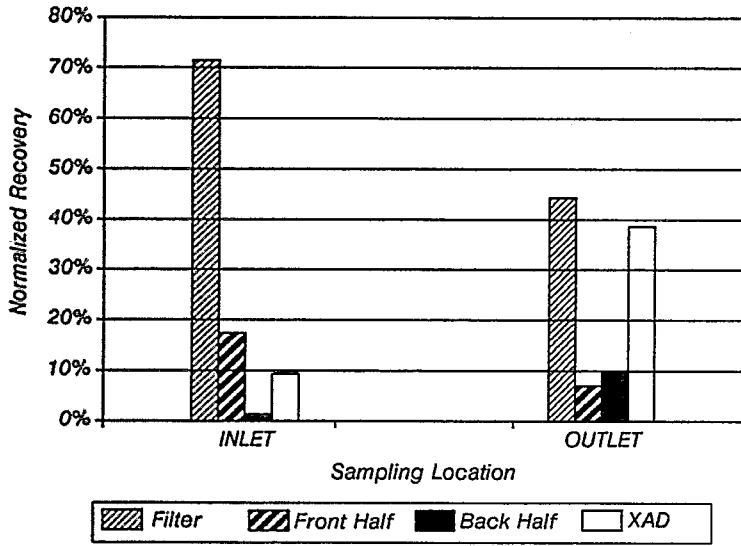


Figure 3. Distribution of dynamically spiked compounds in the MM5 sampling train.

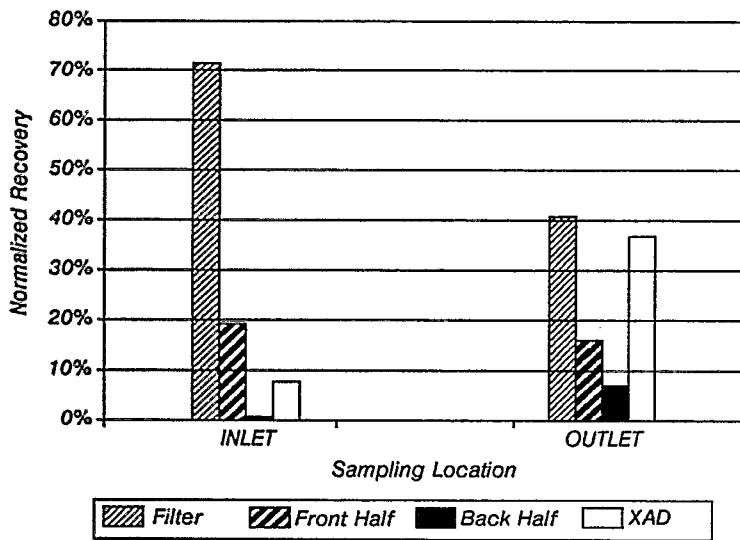


Figure 4. Distribution of native PCDDs and PCDFs in the MM5 sampling train.

The EPA author, Jimmy C. Pau (also the EPA Project Officer, see below), is with Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, NC 27711; and Andrés A. Romeu, Marilyn Whitacre, and John T. Coates, Jr. are with Midwest Research Institute, Kansas City, MO 64110.

The complete report, entitled "Validation of Emission Sampling and Analysis Test Method for PCDDs and PCDFs," (Order No. PB90-235 847/AS; Cost: \$17.00, subject to change) will be available only from:

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