

Testing the Performance of Continuous Emission Monitors for
Measuring Trace Metal and Organic Species Emissions from Incinerators

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16. ABSTRACT In a recently completed test program at the U.S. EPA Incineration Research Facility (IRF), 10 prototype or developing continuous emission monitors (CEMs) for measuring trace metal or trace organic species concentrations were tested. Of the 10 CEMs tested, four measured concentrations of several specific volatile organic compounds, one measured total particulate-bound polynuclear aromatic hydrocarbon concentrations, two measured concentrations of up to 14 trace metals, and three measured mercury concentrations. While the testing consisted of obtaining quantitative measurement data on the four measures of CEM performance checked in a relative accuracy test audit as described in 40 CFR 60 Appendix F-relative accuracy (RA), calibration drift, zero drift, and response time-the primary project objective focused on the RA measurement. The RA measurement was achieved by comparing the monitored analyte concentration reported by the CEM to the concentration determined by the EPA reference method (RM) for the analyte. Four series of tests were performed, each simultaneously testing up to three monitors measuring the same or similar analyte type. Each test series consisted of performing triplicate RM measurements at each of three target flue gas monitored analyte concentrations while the tested CEMs were in operation. All measurements were taken in the wet scrubber exit flue gas from the pilot-scale rotary kiln incineration system at the IRF. The test program results clearly showed the prototype nature of most approaches tested, and the clear need for further development.		
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INTRODUCTION

The U.S. Environmental Protection Agency (EPA) is currently developing more stringent emission standards and considering changes in the way that permits for waste combustion facilities are handled. More public involvement in the process has been proposed. Because the public's apparent perception of incinerators is that high concentrations of hazardous compounds are continually being released from the stacks of the thermal treatment devices, a means by which the "real-time" (defined as ranging from instantaneous to a within-several-hours time frame) organic and metals emissions can be monitored would be of great benefit to both regulators and the regulated community. The ability to have "immediate" knowledge of stack emissions would provide assurances that the thermal treatment device is operating correctly or indicate the change of operating conditions needed to adjust stack emissions. Thus, EPA's Office of Solid Waste (OSW) and the Office of Solid Waste and Emergency Response (OSWER) would like this monitoring capability as a means of responding to and allaying the public's fears by showing that good, safe, and clean combustion practices can be demonstrated.

The immediate needs of the thermal treatment community include the capability of performing "real-time" monitoring of organic compounds and metals as these exit the stack. Conventional procedures usually involve sample collection over an extended period of time and then sample analysis at a later time. "Real-time" monitoring, on the other hand, involves the virtually immediate analysis of trace quantities of pollutants. Several developers have designed monitoring units that they claim will measure various regulated hazardous compounds using a number of different innovative concepts and technologies. The development of these continuous emission monitor (CEM) approaches for both trace metal and trace organic analyte classes has advanced to the state that several candidate approaches are now in the prototype instrument stage. Given this, the general objective of the project reported herein was to test several prototype instruments and establish or estimate for each unit the effectiveness, reliability, accuracy, and detection limit. Support for this project came from both the EPA's National Risk Management Research Laboratory (NRMRL), and the Department of Energy's (DOE's) Office of Technology Development (OTD) through the Savannah River Technical Center (SRTC), and the Education, Research, and Development Association of Georgia Universities (ERDA).

To solicit candidate instruments for testing in the project, an announcement was published in the January 4, 1995 *Commerce Business Daily* (CBD). Several proposals were received in response to this announcement. Proposals addressing both trace metals measurement and trace organic compound measurements were received. The selection of which CEMs to be included in this project was subsequently made taking into consideration the recommendations of a program coordination committee organized by ERDA, with support from SRTC. The selection process resulted in 11 offerings being identified for testing in this program. These are listed in Table 1 by monitored analyte class. As shown, included in the list of CEMs selected for testing in this program are one semivolatile organic constituent (SVOC), four volatile organic constituent (VOC), two multi-metal, and four mercury CEMs.

TEST PROGRAM

The selected approaches were evaluated in this test program, performed in the pilot-scale rotary kiln incineration system (RKS) at EPA's Incineration Research Facility (IRF), located in Jefferson, Arkansas. The testing consisted of obtaining quantitative measurement data on four measures of CEM performance checked in a relative accuracy test audit (RATA) of a CEM as described in 40 CFR 60 Appendix F. These measures are:

- Relative accuracy (RA): the absolute mean difference between the concentrations determined by the CEM and the value determined by the reference method (RM), plus the 2.5 percent error confidence coefficient of a series of tests, divided by the mean of the RM tests
- Calibration drift (CD): the difference in the CEM output reading from the established reference value after a stated period of operation during which no unscheduled maintenance, repair, or adjustment took place; the reference value is established by a calibration standard which has a concentration of nominally 80 percent or greater of the CEM's full scale (span) reading capability
- Zero drift (ZD): the CD where the reference value is 0
- Response time: the time interval between the start of a step change in the concentration of the monitored gas stream and the time when the CEM output reaches 95 percent of the final value

Measuring a CEM's RA requires comparing the monitored analyte concentration reported by the CEM to the concentration determined by the RM for the analyte. In this program, the RM for trace metal (including mercury) monitors was draft Method 29, the EPA multiple metals method documented in the boiler and industrial furnace (BIF) rules.¹ The RM for volatile organic compounds (VOCs) was Method 0030 with analysis using thermal desorption, purge and trap by Method 5040, and quantitation by Method 8015A.² The RM for semivolatile organic compounds (SVOCs) was Method 0010 with analysis by Method 8270B.²

Test Facility

As noted above the test program was conducted in the RKS at the IRF. Figure 1 is a process schematic of the RKS as configured for these tests. The RKS consists of a primary combustion chamber, a transition section, and a fired afterburner chamber. After exiting the afterburner extension, flue gas flows through a quench section that is followed by a primary air pollution control system (APCS). The initial element of the primary APCS for these tests was the venturi scrubber/packed-column scrubber combination shown in Figure 1. This scrubber system removes from the flue gas most of the coarse particles and any acid gas, such as HCl. Following the scrubber system, the flue gas is reheated to about 120°C (250°F) by a 100-kW electric duct heater, then passed through a fabric filter (baghouse). The baghouse removes most of the remaining flue gas particles. Downstream of the baghouse, a backup, secondary APCS, comprised of an activated-carbon adsorber and a high-efficiency particulate air (HEPA) filter is in place.

For this test program, an afterburner exit flue gas partial quench system was installed in the afterburner extension so that the afterburner exit flue gas temperature could be decreased to the 360° to 427°C (680° to 800°F) range prior to completely quenching to saturation. The partial quench capability was needed for flue gas spiking of the VOC and SVOC analytes to be monitored as discussed below. This partial quench system consisted of a water spray nozzle inserted into one of the first ports in the afterburner extension. The system was designed to ensure complete evaporation of the water spray occurred prior to entering the full quench device.

All CEMs tested in this test program sampled flue gas at the scrubber exit location. The length of scrubber exit ductwork accessible for testing had four sets of ports, each set comprised of four individual ports at 90° increments in the duct circular cross section.

Testing Procedures

The test program consisted of four series of tests; each series tested one set of CEMs, generally monitoring the same analyte set. Up to three CEMs were tested at the same time during each of the four test series. Thus, each CEM had access to one of the four sets of ports at the sampling location. The fourth set of ports was dedicated to the RM sampling performed by the IRF staff.

The major portion of the test program consisted of performing three sequential RM measurements, while the tested CEMs were in operation, at each of three flue gas concentrations of monitored analytes. Thus, each test series was designed to supply nine sets of parallel RM and CEM reading data, three at each of three analyte concentrations. These nine sets of parallel RM and CEM data supported the calculation of each CEM's RA. Thus, up to three RAs were calculated for each CEM, one at each of the three flue gas concentrations tested. Other test efforts discussed below supported the measurements of CD, ZD, and response time. To ensure that the sets of RM/CEM concentration data were indeed parallel and comparable, the developers were notified of the start and stop times of each RM procedure so that they could report an average analyte concentration that corresponded directly to the RM measurement period.

Performing one RM measurement of the flue gas constituent concentrations can require a 2- to 2.5-hour flue gas sampling period. With contingency for sampling train filter changes and other sampling procedure delay events, a 3-hour time period was normally allotted to completing one RM test. Thus, the three sequential RM tests were targeted for completion over a 9-hour period of continuous, steady RKS operation at a nominally constant scrubber exit flue gas monitored analyte concentration. This nominally 9-hour period was termed 1 test day. Testing at three different scrubber exit flue gas concentrations, thus, required 3 test days comprising each test series.

At the beginning of each test day, the RKS was brought to steady operation at the desired incineration conditions firing natural gas. After the RKS combustion gas CEMs were calibrated and all RM sampling preparations completed, test waste feed was initiated and steady RKS operation reestablished. During this time, each CEM developer was given the opportunity to calibrate his instrument. This calibration included zero and span checks. The day's test, the three sequential RM sampling efforts, began after all CEMs had completed zero and span checks. The first of the three sequential RM sampling efforts began after the CEMs being tested had been calibrated, provided that at least 1 hour of waste feeding had elapsed. At the end of the test day (after completion of the third

RM sampling period), up to two successive step changes (increases and decreases) in flue gas analyte concentrations were induced. Measurements of CEM responses to these step changes gave data on instrument response time.

After these step change/response observation exercises, test waste feed was stopped and each developer was given the opportunity to check the calibration of his instrument. These post-test checks yielded the measures of CD and ZD. The RKS continued to operate, firing natural gas, until the kiln was visually clear of bottom ash, or for 2 hours, whichever time was longer. After this time period, the RKS was set to an unattended operating condition firing natural gas in preparation for the next test day.

The four test series were completed over the 8-week period beginning July 24, 1995, and ending September 15. Table 2 summarizes the participants and test dates for each test. Of the 11 instruments selected for testing, 10 were able to obtain some data. The Euramark mercury monitor was damaged in transport to the IRF and repair efforts by the developer were unsuccessful. The Marine Shale Processors (MSP) VOC monitor malfunctioned during the initial test in Series 1 and could not be brought into reliable operation. MSP was given the opportunity to return and participate in Series 4, the last test series completed, which they did.

Test Waste Feed

The incinerator feed material was a synthetic hazardous waste comprised of an attapulgite clay solid sorbent combined with a mixture of 14 trace metals and VOCs. The mixture of VOCs added to the sorbent base contained 76 percent toluene by weight, with 12 percent each of chlorobenzene and tetrachloroethene. This mixture was combined with the clay sorbent in the ratio of 1.0 kg of organic constituent mixture to 2.4 kg of clay. The resulting organic compound/clay mixture, thus, contained nominally 22.4 percent of toluene and 3.5 percent each of chlorobenzene and tetrachloroethene. Its chlorine content was nominally 4.1 percent and its heating value nominally 10.7 MJ/kg (4,590 Btu/lb). The mixture was a free-flowing solid with no free-standing liquid. Thus, for all tests the mixture was continuously fed to the RKS via a screw feeder system.

For all tests, the target clay/organic mixture feedrate was 68 kg/hr (150 lb/hr). The target kiln exit gas temperature was 870°C (1,600°F), and the target afterburner exit gas temperature was 1,065°C (1,950°F). Combustion system temperatures were maintained by controlling the auxiliary fuel (natural gas) firing rates to the system combustion chambers. The kiln rotation rate was set to give a kiln solids residence time of about 1 hour.

For all tests, the RKS scrubber system was operated at its design operating conditions, with the exception of the venturi scrubber pressure drop. For these tests the adjustable-throat venturi was opened to its widest setting so that the venturi pressure drop was at a minimum, with the corresponding particulate collection efficiency also at a minimum. The desire was to produce scrubber exit flue gas metal-containing particulate concentrations in the 100 mg/dscm range.

Multi-Metal and Mercury CEM Tests

The trace metals of interest to this test program are those being considered for regulation by OSW and other EPA Program Offices. These are listed in Table 3. Table 3 also notes the program target scrubber exit flue gas concentrations of each metal for the tests of multi-metals CEMs. For the mercury CEMs tests in Test Series 3, the low concentration targets were at half the levels noted in the low-concentration column in Table 3. The intermediate concentration targets were at those noted in the low-concentration column in Table 3, and the high-concentration targets were those noted in the intermediate-concentrations column in Table 3. This change was incorporated at the request of the mercury CEM developers.

Trace metals were added to the RKS, to result in scrubber exit flue gas levels, via two routes. Both routes used an aqueous spike solution of the metals. The composition of the most concentrated spike solution used is given in Table 4. The most concentrated solution was added for the multi-metal CEM test days at the high target flue gas metals concentration. This solution was diluted 10-fold and 40-fold for the multi-metal CEM test days at the intermediate and low target concentrations. The concentrated solution was diluted 10-fold, 40-fold, and 80-fold for the mercury monitor tests at the high, intermediate and low target concentrations. The entire complement of metals was retained in the aqueous spike solution for the mercury CEM tests so that any potential interferences with CEM readings due to the presence of the other metals could be assessed.

The two routes of metals addition were incorporated into the clay/organic mixture and atomized into the kiln main burner flame. The solid waste feed route was effected by metering the aqueous spike solution into the clay/organic liquid mixture at the screw feeder just prior to feed introduction into the kiln. A gear pump was used to inject the spike solution at a flowrate of 2 L/hr. The burner flame atomization route was effected by spraying the aqueous spike solution through the liquid feed nozzle of the kiln dual fuel main burner at a rate of 6 L/hr.

The solution of the VOCs and SVOCs used to establish the target flue gas VOC and SVOC concentrations for the organic CEM test was also injected into the partially quenched afterburner extension flue gas at the intermediate injection concentration for the multi-metal and mercury CEM tests, again to assess any potential interferences.

VOC and SVOC CEM Tests

The list of VOCs present in the scrubber exit flue gas for all tests is given in Table 5. This list contains many of the VOC species currently being considered for regulation. The target flue gas concentrations of the compounds were in the 1 to 2, 10 to 20, and 160 to 240 $\mu\text{g}/\text{dscm}$ ranges (low, intermediate, and high concentrations) for all VOCs except carbon tetrachloride and chloroform. For these two VOCs, the target flue gas concentrations were doubled to 2 to 4, 20 to 40, and 320 to 480 $\mu\text{g}/\text{dscm}$. Naphthalene, phenanthrene, and pyrene were the SVOCs introduced into the flue gas for all tests. The target flue gas concentrations for these were 1 to 2, 10 to 20, and 160 to 240 $\mu\text{g}/\text{dscm}$.

The VOCs and SVOCs were introduced into the flue gas by metering a solution of the spiking compounds in methanol through a length of fine bore stainless steel tubing into the afterburner extension at its centerline. As noted above, the afterburner exit flue gas was partially quenched to a

temperature of between 360° to 427°C (680° and 800°F) by a water spray introduced into the first port (nearest the afterburner proper) of the afterburner extension. The VOC/SVOC solution was introduced through a port midway along the length of the afterburner extension. This arrangement afforded time for the flue gas to cool prior to VOC/SVOC solution injection as well as time for the VOC/SVOC solution to evaporate and fully mix in the flue gas prior to entering the water quench section of the RKS.

The concentrated organic spiking solution prepared consisted of 0.4 g/L of carbon tetrachloride and chloroform, 0.2 g/L of each of the other 8 constituents listed in Table 5, and 0.2 g/L of the three SVOCs in methanol. The concentrated solution was used for the high target flue gas VOC and SVOC CEM tests. The solution was metered into the partially quenched afterburner extension flue gas at a feedrate in the 800 to 1,200 mL/hr range. The concentrated organic solution was diluted with methanol 10-fold and 100-fold for the intermediate and low target flue gas concentration tests, respectively, and fed at the same 800 to 1,200 mL/hr feedrate. Trace metals were also fed into the kiln in the same manner used for the multi-metals and mercury CEM tests. The multi-metal CEM test intermediate feed solution concentrations were used.

TEST RESULTS

VOC CEM Tests

Tables 6 through 8 present the results of the three sequential RM measurements, along with the Oak Ridge National Laboratory (ORNL) and EcoLogic CEM results, for each of the three VOC concentrations tested in Test Series 1. The EcoLogic CEM data for the first day of testing at the low VOC concentration were not reported in EcoLogic's test report due to operator error which resulted in CEM readings that were inflated and incorrect. MSP was an original participant in Test Series 1. However, during this series, they were unable to bring their instrument into operation. Because access space was available for another CEM in Test Series 4, MSP was afforded the opportunity to return and participate in this series.

RAs were calculated using the RM and CEM data in Tables 6 through 8. These are summarized in Table 9. For the cases noted above where the ORNL CEM measured concentrations were less than the reporting limit, no RA calculation was performed when two or more of the three CEM concentrations on a test day were less than the reporting limit. In cases for which only one of the three CEM concentrations for the day was less than reporting limit, the RA was calculated by using the reporting limit as the CEM concentration. The data in Table 9 show that the calculated RAs for the ORNL CEM ranged from 123 to 305 percent at the low test concentration, with an average of 196 percent over the seven compounds reported. ORNL CEM RAs were improved at the intermediate test concentration, at 113 to 278 percent, with an average of 154 percent over the nine compounds reported. Further improvement is seen at the high test concentration, with an RA range of 84 to 144 percent, and an average of 105 percent over all 10 compounds reported. In fact, the RA for all VOCs reported uniformly improved as the test concentration increased.

Because EcoLogic did not report CEM concentrations for the low concentration test day, no RA calculation was possible. For the intermediate test concentration, the RAs of the EcoLogic CEM ranged from 65 to 7,320 percent, with an average of 2,520 percent. Much improved performance

was seen at the high test concentration, for which the RA ranged from 27 to 283 percent and averaged 121 percent. As seen for the ORNL CEM, the RAs for nine of the 10 VOCs reported were improved at the high test concentration compared to the intermediate concentration.

Tables 10 through 12 present the results of the three sequential RM measurements, along with the EPA/APPCD and MSP CEM results, for each of the three VOC concentrations tested in Test Series 4. The tables indicate that, out of the nine sampling periods, MSP obtained data for only two. The MSP CEM failed during the first test day. The failure was caused by moisture condensation in the instrument's sampling system which, in turn, caused pressure fluctuations at the MS inlet. These pressure fluctuations persisted throughout the rest of the test series. The MSP team was unable to effect system repairs in the time to participate in any subsequent testing.

RAs corresponding to the RM/CEM concentration data given in Table 10 through 12 are summarized in Table 13. Because the MSP system only operated on the first test day at the low VOC concentration, only this one set of RAs is noted in the table. However, even this RA calculation is not strictly appropriate because it is based on only two pairs of RM and CEM measurements. All EPA CEM performance specifications require a minimum of three pairs of RM and CEM measurements for an RA calculation.

The data in Table 13 show that the RAs for the EPA CEM ranged from 71 to 3,190 percent, and averaged 638 percent, for the low test concentrations. The relatively high average RA was driven by the two very high RAs for 1,2-dichloroethane and 1,1-dichloroethane, however. The median RA for the low concentration test at a much improved 113 to 137 percent, removes the dominant influence of the two compounds for which the CEM did poorly. Two values are noted for the median in Table 13 because 10 compounds are reported; thus, the median represents the fifth and sixth lowest RAs noted. The RAs for the EPA CEM were improved at the intermediate test concentration, ranging from 29 to 1,130 percent and averaging 213 percent. Poor performance in quantitating 1,2-dichloroethane and 1,1-dichloroethane again accounts largely for the high average RA. Again, the median RA at 83 to 98 percent better reflects the mean performance of the CEM by removing the dominant influence of the RAs for the two VOCs poorly quantitated. Further improved performance of the EPA CEM was seen at the high test concentration, with an RA range from 34 to 133 percent and an average RA of 73 percent. In fact, at the high test concentration, the RAs for two compounds poorly quantitated at the low and intermediate test concentrations are more in line with those calculated for the other eight compounds. For this reason, the median RA at 53 to 70 percent is comparable to the average RA.

The calculated RAs based on the two available CEM/RM measurement pairs for the MSP CEM were quite large, ranging from 315 to 412,000 percent and averaging 54,600 percent. Even the median RAs for the MSP CEM, at 2,840 to 6,480 percent, are quite high.

SVOC CEM Tests

The SVOC CEM tests were performed during Test Series 4 of the test program, simultaneously with the second set of VOC CEM tests. Table 14 presents the results of the three sequential RM measurements performed each test day, and compares these to the EcoChem CEM results for the test days at the low and intermediate SVOC concentrations. Due to problems in the flue gas conditioning (moisture removal) system, the EcoChem CEM could not be brought into operation on the last day of testing at the high SVOC concentration. Because EcoChem was the only SVOC CEM participating

in the test program, SVOC RM measurements were stopped after the first RM on this test day because there would be no CEM reading for comparison. The single RM measurement is given, for completeness, in Table 14. In addition, no CEM data were obtained during the first RM period on the intermediate concentration test day because the EcoChem CEM was not in operation, again due to problems with the flue gas moisture removal system.

Table 14 also notes the RA of the EcoChem PAH CEM for the two test days the CEM was in operation. As was done for the MSP VOC CEM discussed above, an RA was calculated for the intermediate concentration test day using only the two pairs of RM and CEM data, despite the universal performance specification (PS) requirement that at least three pairs of data be used in a true RA determination.

Table 14 indicates that the RAs for the EcoChem CEM were 527 and 99 percent. As was seen in the VOC CEM tests, the RA at the higher test flue gas concentration was improved in comparison to the lower test concentration.

Multi-Metal CEM Tests

Tables 15 through 17 summarize the results of the three sequential RM measurements performed each test day and compares these to the Sandia National Laboratories (SNL) and Metorex CEM measurements. As indicated in the tables, the Metorex instrument did not measure beryllium or mercury. The SNL CEM did not detect any of the test trace metals on the low concentration test day, only arsenic, barium, and lead were reported on the intermediate concentration test day, and only antimony, arsenic, barium, and lead for one or more RM periods were reported on the high concentration test day.

The RAs corresponding to the measurement pair data in Tables 15 through 17 are summarized in Table 18. Neither beryllium or mercury is included in the tables because neither CEM tested measured these two metals. In addition, results for silver are not included in the table. Spike recovery from QA samples was poor, so silver concentrations as measured by the RM are highly suspect.

The data in Table 18 show that the RAs for the SNL CEM ranged from 64 to 1,560 percent for the three metals reported on the intermediate concentration test day, and from 65 to 188 percent for the two metals reported on the high concentration test day. RAs for the Metorex CEM ranged from 88 to 236 percent, with an average of 129 percent and a median of 116 percent for the low concentration test. Corresponding RAs for the intermediate-concentration test were 72 to 467 percent, with an average of 168 percent and a median of 135 percent, and, for the high-concentration test, 93 to 177 percent, with an average of 129 percent and a median of 121 percent. The RAs for the Metorex CEM were comparable for each test concentration. No marked improvement as flue gas concentration increased, as observed for the VOC CEMs, is seen in the Metorex CEM data.

Mercury CEM Tests

Table 19 summarizes the results of three sequential RM measurements performed each mercury CEM test day and compares these to the corresponding three mercury CEM measurements. Calculated RAs for each CEM are also given in the table for the three test days, each representing a different flue gas mercury concentration.

The table indicates several periods during which the Perkin Elmer and the Senova CEMs were not in operation. In the Senova case, a critical part of the Senova CEM was broken when packing the CEM for shipment to the IRF. The time required to locate and secure a replacement part caused a delay in the Senova team's arrival at the IRF such that the first test day, at the intermediate flue gas mercury concentration, was missed. The Senova CEM was in operation on the second test day, at the high flue gas mercury concentration. However, a malfunction that caused unstable sample gas flow to the analyzer prevented Senova from obtaining valid data on the third and last day of testing at the low flue gas mercury concentration. Thus, the Senova team was able to obtain only one day of test results.

The Perkin Elmer team was unable to obtain reliable results during the second RM period on the first test day (intermediate concentration) because of a buildup of a white powder which clogged the probe sintered metal filter. On the second day of testing (high concentration) the Perkin Elmer CEM developed a defect at the sample drain pump and a broken probe fitting, so no data were obtained for the first two RM periods on this test day. As a result, no RA could be calculated for the Perkin Elmer CEM for the high concentration test, and the RA in Table 19 for the intermediate concentration test is based on only two pairs of RM/CEM measurements.

The data in Table 19 show that the EcoChem CEM had an RA of about 60 percent for both the low and the high concentration tests. The RA at the intermediate concentration was increased, at 92 percent. The RA of the Perkin Elmer CEM was 602 percent at the low mercury concentration and 1,150 percent (based on two measurement pairs) at the intermediate mercury concentration. The RA of the Senova CEM was 186 percent at the one test concentration having data.

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REFERENCES

1. 40 CFR Part 266, Appendix IX.
2. "Test Methods for Evaluating Solid Waste: Physical/Chemical Methods," EPA SW-846, Third Edition, Revision 2, September 1994.

Table 1. Participants in the CEM test programs.

Monitored analyte	Developer	Approach
SVOCs	EcoChem	Photoionization of aerosol-bound polycyclic aromatic hydrocarbons
VOCs	EcoLogic	Continuous chemical ionization mass spectrometry
	Marine Shale Processors (MSP)	Continuous online mass spectrometry
	Oak Ridge National Laboratory (ORNL)	Direct sampling ion trap mass spectrometry
	EPA, Air Pollution Prevention and Control Division (APPCD)	On line gas chromatography with dual flame ionization, electron capture detection
Multi-metals	Sandia National Laboratory (SNL)	Laser induced plasma spectroscopy
	Metorex	Extractive beta gauge particulate monitor with x-ray fluorescence metals analysis
Mercury	Perkin-Elmer	Gold trap amalgamation collection, cold vapor atomic absorption spectroscopy analysis
	Euramark	Cold vapor atomic absorption spectroscopy
	Senova	Noble metal film solid state chemical microsensor
	EcoChem	Cold vapor atomic absorption spectroscopy

Table 2. CEM test summary.

Test series	CEM tested	Monitored analyte	Test completion dates		
			Low analyte concentration	Intermediate analyte concentration	High analyte concentration
1	EcoLogic	VOC	7/31/95	8/2/95	8/4/95
	Marine Shale Processors ^a	VOC			
	Oak Ridge National Laboratory	VOC			
2	Sandia National Laboratory	Multi-metals	8/14/95	8/16/95	8/18/95
	Metorex	Multi-metals			
	Euramark ^b	Hg			
3	Perkin-Elmer	Hg	9/1/95	8/28/95	8/30/95
	Senova	Hg			
	EcoChem	Hg			
4	EcoChem	SVOC	9/11/95	9/13/95	9/15/95
	EPA/APPCD	VOC			
	Marine Shale Processors	VOC			

^aMarine Shale Processors was unable to bring their system into operation. They returned during the last test series.

^bEuramark was unable to bring their system into operation.

Table 3. Test trace metals and target flue gas concentrations.

Metal	Target flue gas concentration, $\mu\text{g}/\text{dscm}$		
	Low	Intermediate	High
Antimony	10	40	400
Arsenic	5	20	200
Barium	50	200	2,000
Beryllium	0.5	2	20
Cadmium	5	20	200
Chromium	20	80	560
Cobalt	10	40	400
Lead	50	200	2,000
Manganese	5	20	200
Mercury	20	80	800
Nickel	10	40	400
Selenium	50	200	2,000
Silver	5	20	200
Thallium	5	20	200

Table 4. Concentrated aqueous spike solution composition.

Metals	Metal concentration, g/L	Compound	Compound concentration, g/L
Antimony	0.32	$\text{C}_4\text{H}_4\text{KO}_7\text{Sb}$	0.85
Arsenic	0.18	As_2O_3	0.24
Barium	6.3	$\text{Ba}(\text{NO}_3)_2$	12.0
Beryllium	0.06	10,000 ppm Be standard	NA ^a
Cadmium	0.075	$\text{Cd}(\text{NO}_3)_2 \cdot 4 \text{H}_2\text{O}$	0.21
Chromium	1.5	$\text{Cr}(\text{NO}_3)_3 \cdot 9 \text{H}_2\text{O}$	11.2
Cobalt	1.6	$\text{Co}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$	7.78
Lead	1.40	$\text{Pb}(\text{NO}_3)_2$	2.24
Manganese	0.52	$\text{Mn}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$	2.72
Mercury	0.26	$\text{Hg}(\text{NO}_3)_2 \cdot \text{H}_2\text{O}$	0.44
Nickel	1.90	$\text{Ni}(\text{NO}_3)_2 \cdot 6 \text{H}_2\text{O}$	9.41
Selenium	0.70	SeO_2	0.98
Silver	0.11	AgNO_3	0.17
Thallium	0.075	$\text{Ti}(\text{NO}_3)_3 \cdot 3 \text{H}_2\text{O}$	0.16

^aNA = Not applicable.

Table 5. VOC spiked into flue gas.

Benzene	1,1-Dichloroethene
Carbon tetrachloride	Tetrachloroethene
Chlorobenzene	Toluene
Chloroform	1,1,1-Trichloroethane
1,2-Dichloroethane	Trichloroethene

Table 6. Measured flue gas concentrations for the test of the ORNL and EcoLogic CEMs at the low VOC concentration.

Compound	Concentration, $\mu\text{g/dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	ORN			ORN			ORN		
	RM	L	EcoLogic	RM	L	EcoLogic	RM	L	EcoLogic
Benzene	32.4	1.3	NO ^a	41.9	1.6	NO	59.6	1.6	NO
Carbon tetrachloride	31.2	<0.4	NO	34.2	<0.4	NO	38.0	0.92	NO
Chlorobenzene	55.6	0.76	NO	49.2	1.2	NO	86.7	5.6	NO
Chloroform	40.8	0.56	NO	47.3	0.4	NO	41.6	3.6	NO
1,2-Dichloroethane	2.4	2.5	NO	3.3	1.5	NO	2.6	6.8	NO
1,1-Dichloroethene	86.4	3.2	NO	35.6	<0.4	NO	16.9	11.0	NO
Tetrachloroethene	89.9	1.7	NO	73.9	1.3	NO	126	4.3	NO
Toluene	352	9.2	NO	316	9.2	NO	462	16	NO
1,1,1-Trichloroethane	2.5	<0.4	NO	4.6	<0.4	NO	6.4	<0.4	NO
Trichloroethene	6.9	<0.4	NO	5.9	<0.4	NO	3.9	<0.4	NO

^aNO = CEM not operational.

Table 7. Measured flue gas concentrations for the test of the ORNL and EcoLogic CEMs at the intermediate VOC concentration.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	ORN			ORN			ORN		
	RM	L	EcoLogic	RM	L	EcoLogic	RM	L	EcoLogic
Benzene	32.7	12.0	97	28.7	<2.3	820	36.4	5.5	870
Carbon tetrachloride	46.9	10.1	7.9	41.7	3.8	24	58.5	6.4	16
Chlorobenzene	59.8	25.8	81	46.3	7.2	81	74.3	27.6	98
Chloroform	57.1	23.9	140	56.7	9.6	230	66.3	18.4	170
1,2-Dichloroethane	17.4	43.3	210	12.3	16.6	340	15.3	29.5	290
1,1-Dichloroethene	24.0	55.3	320	20.5	26.7	430	14.3	40.5	370
Tetrachloroethene	81.4	11.1	120	64.1	2.5	770	101	7.4	710
Toluene	342	147	210	218	71.8	120	413	103	250
1,1,1-Trichloroethane	13.8	<2.3	800	13.3	<2.3	910	12.9	<2.3	840
Trichloroethene	19.4	4.6	420	18.6	0.9	770	20.4	1.8	510

Table 8. Measured flue gas concentrations for the test of the ORNL and EcoLogic CEMs at the high VOC concentration.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	ORN			ORN			ORN		
	RM	L	EcoLogic	RM	L	EcoLogic	RM	L	EcoLogic
Benzene	102	36.8	140	89.1	50.7	160	91.3	28.6	190
Carbon tetrachloride	423	101	380	409	119	350	446	76.4	360
Chlorobenzene	337	138	250	299	170	270	269	97.6	280
Chloroform	417	101	330	411	168	350	413	91.2	390
1,2-Dichloroethane	184	88.4	450	174	114	500	183	6.3	540
1,1-Dichloroethene	116	38.7	350	140	44.2	440	162	35.9	480
Tetrachloroethene	429	62.6	690	374	61.7	740	324	37.8	690
Toluene	1,760	847	1,300	1,393	921	120	1,024	460	760
1,1,1-Trichloroethane	175	24.9	170	164	37.8	190	182	20.3	210
Trichloroethene	189	15.7	340	176	19.3	300	185	14.7	360

Table 9. Relative accuracies of the ORNL and EcoLogic CEMs.

Compound	RA, %					
	ORNL			EcoLogic		
	Test concentration			Test concentration		
	Low	Intermediate	High	Low	Intermediate	High
Benzene	173	119	98	NC	5,020	154
Carbon tetrachloride	NC ^a	129	100	NC	135	27
Chlorobenzene	164	93	84	NC	74	52
Chloroform	123	105	97	NC	396	33
1,2-Dichloroethane	305	278	144	NC	2,890	239
1,1-Dichloroethene	299	277	115	NC	2,520	283
Tetrachloroethene	162	142	113	NC	1,640	128
Toluene	145	131	88	NC	65	143
1,1,1-Trichloroethane	NC	NC	110	NC	7,320	36
Trichloroethene	NC	113	103	NC	5,140	116
Average ^b	196	154	105	NC	2,520	121
Median ^b	164	129	100, 103	NC	1,640, 2,520	116, 128

^aNC = Not calculated.

^bAverage and median excludes NC entries.

Table 10. Measured flue gas concentrations for the test of the EPA/APPCD and MSP CEMs at the low VOC concentration.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	RM	EPA/		RM	EPA/		RM	EPA/	
		APPCD	MSP		APPCD	MSP		APPCD	MSP
Benzene	8.2	21.31	795	5.9	29.93	707	8.4	22.21	NO ^a
Carbon tetrachloride	13.9	9.68	118	11.9	5.99	126	13.3	4.99	NO
Chlorobenzene	21.6	18.56	143	20.8	29.16	60.2	16.0	18.8	NO
Chloroform	15.8	16.95	3,439	18.4	14.89	1,515	15.8	9.25	NO
1,2-Dichloroethane	1.8	43.21	73.7	1.5	39.18	78.5	1.6	30.33	NO
1,1-Dichloroethene	2.0	76.34	322	6.3	90.23	271	5.1	84.49	NO
Tetrachloroethene	32.7	15.65	124	26.9	31.7	107	20.6	8.56	NO
Toluene	160.9	131.92	1,308	149.4	221.38	814	97.1	57.85	NO
1,1,1-Trichloroethane	2.1	2.21	3.6	1.9	2.22	4.1	1.8	3.53	NO
Trichloroethene	2.6	2.18	3,022	2.9	2.71	1,602	3.0	1.74	NO

^aNO = Not operational.

Table 11. Measured flue gas concentrations for the test of the EPA/APPCD and MSP CEMs at the intermediate VOC concentration.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	RM	EPA/ APPCD	MSP	RM	EPA/ APPCD	MSP	RM	EPA/ APPCD	MSP
Benzene	33.9	35.8	NO ^a	32.9	42.41	NO	33.6	50.34	NO
Carbon tetrachloride	53.5	31.45	NO	57.8	37.58	NO	64.1	40.55	NO
Chlorobenzene	29.5	24.86	NO	64.6	54.15	NO	75.0	40.73	NO
Chloroform	43.2	26.33	NO	62.1	31.61	NO	63.5	43.31	NO
1,2-Dichloroethane	20.7	27.97	NO	18.1	34.9	NO	16.6	55.71	NO
1,1-Dichloroethene	14.2	47.14	NO	11.3	54.48	NO	9.9	101.19	NO
Tetrachloroethene	39.3	22.87	NO	92.7	58.32	NO	96.8	30.3	NO
Toluene	143	90.22	NO	498.5	306.1	NO	551.8	163.48	NO
1,1,1-Trichloroethane	17.3	13.68	NO	16.2	14.33	NO	17.4	14.54	NO
Trichloroethene	22.9	16.04	NO	20.6	16.63	NO	19.1	15.74	NO

^aNO = Not operational.

Table 12. Measured flue gas concentrations for the test of the EPA/APPCD and MSP CEMs at the high VOC concentration.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	RM	EPA/ APPCD	MSP	RM	EPA/ APPCD	MSP	RM	EPA/ APPCD	MSP
Benzene	102.6	96.33	NO ^a	129.5	98.73	NO	117.7	88.66	NO
Carbon tetrachloride	222.5	209.55	NO	266.2	205.53	NO	283.7	135.72	NO
Chlorobenzene	104.8	119.78	NO	146.5	113.78	NO	127.2	126.99	NO
Chloroform	229.4	190.9	NO	243.8	199.06	NO	241.1	178.42	NO
1,2-Dichloroethane	93.7	95.44	NO	121.2	107.96	NO	114.9	90.81	NO
1,1-Dichloroethene	65.9	113.67	NO	65.5	124.81	NO	71.9	144.06	NO
Tetrachloroethene	112.8	150.65	NO	162.6	161.66	NO	132.2	131.62	NO
Toluene	176.6	191.23	NO	445.4	213.8	NO	261.5	217.23	NO
1,1,1-Trichloroethane	97.8	92.89	NO	106.8	87.31	NO	103.5	57.93	NO
Trichloroethene	98.2	98.46	NO	114.3	91.81	NO	113.1	70.04	NO

^aNO = Not operational.

Table 13. Relative accuracies of the EPA/APCCD and MSP CEMs.

Compound	RA, %			
	EPA/APPCD			MSD
	Test concentration			Test concentration
	Low	Intermediate	High	Low
Benzene	429	83	48	18,300
Carbon tetrachloride	86	45	95	1,340
Chlorobenzene	87	98	53	2,840
Chloroform	76	71	34	86,000
1,2-Dichloroethane	3,190	334	40	6,480
1,1-Dichloroethene	2,040	1,130	133	15,500
Tetrachloroethene	137	134	50	526
Toluene	113	158	138	2,560
1,1,1-Trichloroethane	150	29	73	315
Trichloroethene	71	45	70	412,000
Average	638	213	73	54,600
Median	113, 137	83, 98	53, 70	2,840, 6,480

Table 14. Measured flue gas concentrations for the tests of the EcoChem PAH CEM.

	Concentration, $\mu\text{g}/\text{dscm}$			RA, %
	1st daily RM	2nd daily RM	3rd daily RM	
Low Concentration Test				
Naphthalene	1.7	1.8	1.7	
Phenanthrene	1.3	1.2	1.3	
Pyrene	1.0	0.8	0.9	
Total PAH	4.0	3.8	3.9	
EcoChem CEM	6.9	14.8	15.5	527
Intermediate Concentration Test				
Naphthalene	17.5	10.9	15.8	
Phenanthrene	15.7	10.1	15.3	
Pyrene	9.1	19.6	9.7	
Total PAH	42.3	40.6	40.8	
EcoChem CEM	NO ^a	33.2	39.0	99
High Concentration Test				
Naphthalene	97.0	NP ^b	NP	
Phenanthrene	91.4	NP	NP	
Pyrene	68.2	NP	NP	
Total PAH	256.6	NP	NP	
EcoChem CEM	NO	NO	NO	NC ^c

^aNO = Not operational.

^bNP = Not performed.

^cNC = Not calculated.

Table 15. Measured flue gas metals concentrations for the test of the SNL and Metorex CEMs at the low metals concentrations.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	1st daily RM			2nd daily RM			3rd daily RM		
	RM	SNL	Metorex	RM	SNL	Metorex	RM	SNL	Metorex
Antimony (Sb)	4.5	ND ^a	ND	5.1	ND	ND	4.5	ND	5.13
Arsenic (As)	4.4	ND	3.65	3.8	ND	0.83	3.6	ND	1.19
Barium (Ba)	11.7	ND	ND	15.8	ND	ND	18.6	ND	6.23
Beryllium (Be)	0.1	ND	NM ^b	0.1	ND	NM	0.1	ND	NM
Cadmium (Cd)	9.7	ND	2.63	12.1	ND	ND	13.2	ND	10.02
Chromium (Cr)	22.3	ND	2.49	23.5	ND	0.56	28.0	ND	22.29
Cobalt (Co)	7.8	ND	12.11	7.1	ND	ND	7.1	ND	14.68
Lead (Pb)	101	ND	11.51	85.6	ND	9.06	110	ND	12.36
Manganese (Mn)	21.8	ND	5.89	29.2	ND	ND	31.6	ND	19.43
Mercury (Hg)	14.9	ND	NM	17.2	ND	NM	11.2	ND	NM
Nickel (Ni)	39.6	ND	27.52	29.1	ND	6.15	42.4	ND	21.87
Selenium (Se)	11.4	ND	1.51	12.3	ND	1.47	12.3	ND	3.62
Silver (Ag) ^c	2.9	ND	0.98	4.8	ND	0.91	4.5	ND	0.99
Thallium (Tl)	1.1	ND	ND	1.5	ND	ND	1.7	ND	ND

^aND = Not detected.

^bNM = Not measured.

^cRM data for silver not reliable due to low spike recoveries.

Table 16. Measured flue gas concentrations for the test of the SNL and Metorex CEMs at the intermediate metals concentrations.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	Reference Method 1			Reference Method 2			Reference Method 3		
	RM	SNL	Metorex	RM	SNL	Metorex	RM	SNL	Metorex
Antimony (Sb)	11.0	ND ^a	39.73	11.6	ND	22.00	9.5	ND	8.49
Arsenic (As)	11.1	63	11.92	10.8	42	0.74	8.7	115	6.61
Barium (Ba)	78.0	251	44.37	80.0	199	11.87	49.2	463	9.92
Beryllium (Be)	0.6	ND	NM ^b	0.6	ND	NM	0.4	ND	NM
Cadmium (Cd)	14.0	ND	7.22	15.0	ND	26.93	14.2	ND	10.09
Chromium (Cr)	54.7	ND	56.48	59.5	ND	72.51	50.3	ND	25.68
Cobalt (Co)	32.3	ND	14.72	33.9	ND	20.79	27.4	ND	9.79
Lead (Pb)	141	144	107.07	141	93	51.80	136	106	40.86
Manganese (Mn)	24.2	ND	61.4	24.6	ND	55.58	18.2	ND	31.25
Mercury (Hg)	54.3	ND	NM	83.7	ND	NM	75.3	ND	NM
Nickel (Ni)	59.9	ND	26.48	61.2	ND	21.26	52.6	ND	15.76
Selenium (Se)	43.2	ND	29.34	54.5	ND	21.27	53.2	ND	18.12
Silver (Ag) ^c	5.0	ND	20.90	7.9	ND	12.77	6.9	ND	6.05
Thallium (Tl)	11.1	ND	12.96	11.2	ND	4.49	12.4	ND	2.72

^aND = Not detected.

^bNM = Not measured.

^cRM data for silver not reliable due to low spike recoveries.

Table 17. Measured flue gas metals concentrations for the test of the SNL and Metorex CEMs at the high metals concentrations.

Compound	Concentration, $\mu\text{g}/\text{dscm}$								
	Reference Method 1			Reference Method 2			Reference Method 3		
	RM	SNL	Metorex	RM	SNL	Metorex	RM	SNL	Metorex
Antimony (Sb)	114	233	27.32	75.7	186	18.35	43.5	131	6.59
Arsenic (As)	82.2	75	21.82	64.8	86	13.28	54.8	65	4.68
Barium (Ba)	331	650	207.37	484.3	ND	111.07	285	ND	27.29
Beryllium (Be)	12.1	ND ^a	NM ^b	6.8	ND	NM	4.0	ND	NM
Cadmium (Cd)	88.0	ND	33.58	60.9	ND	31.73	88.7	ND	22.18
Chromium (Cr)	425	ND	129.33	299	ND	91.85	241	ND	34.70
Cobalt (Co)	357	ND	100.16	229	ND	67.47	248	ND	37.62
Lead (Pb)	1,650	ND	297.20	1,082	ND	282.71	2,176	54	167.04
Manganese (Mn)	179	ND	52.89	89.9	ND	35.25	95.6	ND	16.56
Mercury (Hg)	625	ND	NM	451	ND	NM	581	ND	NM
Nickel (Ni)	550	ND	160.10	347	ND	111.42	429	ND	67.89
Selenium (Se)	421	ND	102.52	399	ND	96.70	383	ND	39.92
Silver (Ag) ^c	5.0	ND	33.60	8.1	ND	25.60	7.1	ND	14.88
Thallium (Tl)	114	ND	32.29	94.3	ND	28.10	113	ND	16.84

^aND = Not detected.

^bNM = Not measured.

^cRM data for silver not reliable due to low spike recoveries.

Table 18. Relative accuracies of the SNL and Metorex CEMs.

Compound	RA, %					
	SNL			Metorex		
	Test concentration			Test concentration		
	Low	Intermediate	High	Low	Intermediate	High
Antimony	NC ^a	NC	188	NC	467	158
Arsenic	NC	1,560	65	125	174	101
Barium	NC	905	NC	NC	135	153
Cadmium	NC	NC	NC	89	177	123
Chromium	NC	NC	NC	158	94	113
Cobalt	NC	NC	NC	236	72	118
Lead	NC	64	NC	115	112	177
Manganese	NC	NC	NC	116	261	146
Nickel	NC	NC	NC	88	77	121
Selenium	NC	NC	NC	104	113	93
Thallium	NC	NC	NC	NC	171	111
Average ^b	—	843	127	129	168	129
Median ^b	—	905	65, 188	116	135	121

^aNC = Not calculated.

^bAverage and median excludes RAs not calculated (NC).

Table 19. Measured flue gas concentrations and RAs for the mercury CEM tests.

Test	Mercury concentration ($\mu\text{g}/\text{dscm}$)			
	RM	EcoChem CEM	Perkin-Elmer CEM	Senova CEM
Low Mercury Concentration				
RM 1	21	22	78	NO ^a
RM 2	16	20	42	NO
RM 3	13	19	11	NO
RA, %		60	602	NC ^b
Intermediate Mercury Concentration				
RM 1	56	83	61	NO
RM 2	34	43	NO	NO
RM 3	40	56	125	NO
RA, %		92	1,150	NC
High Mercury Concentration				
RM 1	119	137	NO	232
RM 2	94	81	NO	116
RM 3	86	62	405	165
RA, %		61	NC	186

^aNO = Not operational.

^bNC = Not calculated.

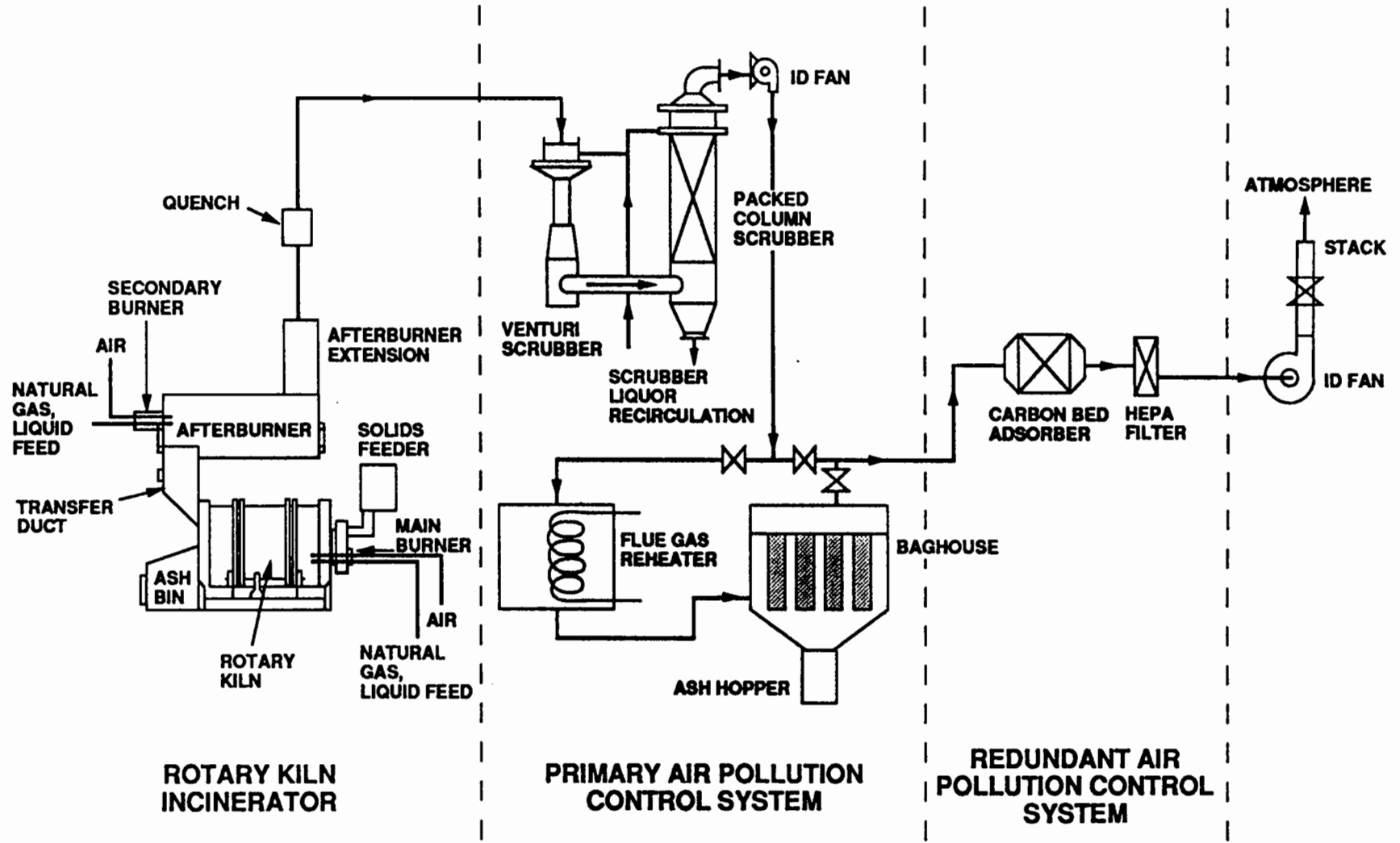


Figure 1. Schematic of the IRF rotary kiln incineration system.