SECTION 5

ASSESSMENT OF RESULTS

The data collected from the field sampling and the analytical results are assessed in the following sections.

5.1 WASTE OIL USERS

The waste oil users use the oil to provide fuel to their process, specifically the kilns. In addition, there is an apparent commonality of the plant configurations in that both plants contain kilns to process their products. However, the difference in the products themselves makes the comparison of the two plants somewhat difficult. Site A processes a silica-based material (diatomaceous earth) that is used as a filtering agent and is likely to absorb organic compounds, whereas Site B produces a magnesium/carbonate refractory mix that may chemically bind compounds. The differences in materials processed in Site A as compared to Site B might explain the apparent lower concentrations of organics found in Site A, especially with the high particulate loading at the inlet. It might be postulated that the exhaust gases with the high particulate loading passing through the baghouse may effectively be removing organics from the stream. This may also account for the difference in metals concentration, with Site A having generally lower levels than Site B .

The data from individual runs from Sites A and B have been reviewed to determine any patterns or other points of interest.

Site A. The dioxin and furan emission levels were previously provided in Tables 4-4 and 4-5. The total dioxin emission rates of the inlet and outlet are generally consistent across all three runs. In addition, the outlet emission rates are at least an order of magnitude less than the inlet levels. This trend is also seen with the furan emission rates. The contribution of 2,3,7,8-substituted dioxins/furans to the total emission rate of dioxins/furans follows the same trends as the totals; i.e., it increases at the inlet over consecutive runs, but decreases at the outlet. Also, the destruction of the total dioxins/furans increases over consecutive runs. This may indicate that the plant, recently maintained prior to the sampling, is now beginning to operate more efficiently. Without more data for a comprehensive study, it may not be significant, but the data indicates that dioxins/furans, particularly the 2,3,7,8-substituted dioxins/furans, are more efficiently destroyed under the incinerator

conditions of Run 3. Of particular concern though is the high blank data for dioxins and furans. This precludes more definitive assessment of the sampling data other than to assess general trends which show that inlet data are higher than outlet data.

The PAH emission rates (Table 4-7) show similar trends as the inlet and outlet data for the dioxin emission rates; i.e., the PAH emission rates of the inlet and outlet are remarkably consistent across all runs. In addition, the outlet emission rates are 12 to 19 times lower than the inlet levels and show consistent improvement from Run 1 to Run 3. The data show that all of the metals removal efficiencies were greater than 90% based on difference between inlet and outlet data, but that no general trend in removal efficiencies exists within the operation during this study. The unusual results of the manganese analyses of Runs 1 and 3 are probably due to contamination of the nitric acid impinger by the impinger containing a concentrated solution of permanganate.

Site B. The dioxin and furan emission levels were previously provided in Tables 4-12 and 4-13. The total dioxin emission rates of the inlet and outlet are remarkably consistent across all three runs. In addition, the outlet emission rates are an order of magnitude less than the inlet levels. In contrast, the total furan emissions rates are somewhat similar, but the between-run comparison is less consistent, as are the inlet/outlet rates for the first run, i.e., Run 4. The contribution of 2,3,7,8-substituted dioxins/furans to the total emission rate of dioxins/furans follows the same trend as the totals.

The PAH emission rates (Table 4-15) are remarkably similar, whether comparing inlet versus outlet of a run or even comparison between runs.

For the metals, there is no apparent distinct trend, but one can be inferred. Except for cadmium, the results of the metals train analyses show that for each analyte, removal efficiencies increase or remain constant while inlet amounts were increasing. Cadmium shows a dramatic decrease in removal efficiency with increasing amount at the inlet.

Mercury (Hg) analysis of the rinses and filter was not completed for any of the runs due to holding time exceedance. These results may bias the data low. However, samples collected at hazardous waste incinerators show most of the mercury is collected in the impingers indicating gaseous rather than particulate-based mercury.

In general, it is expected that for Sites A and B the inlet levels would be higher than the outlet emission values. The control device is providing some level of reduction for dioxins/furans, PAHs, and metals emissions. The mechanism for this reduction is not clear. The organic and metal emissions from the kiln may be entrained on particulate matter or they may be in the gaseous phase, or a combina-

tion. The high particulate loading at the baghouses at Sites A and B may have contributed to the removal efficiency.

5.2 DRUM RECONDITIONERS

Site C. There was no pollution control device at this site, and thus assessment of the data is limited to comparison of between-run data. Total dioxin and furan emissions did decrease over consecutive runs, with the last run (Run 9) lower due to lower total furan emissions. The contribution of the 2,3,7,8-substituted dioxins to the total dioxins emissions averaged about 24% except for Run 9, which contributed only 3% to the total. The trend for the substituted furans was more consistent, i.e., 32% (Run 7), 42% (Run 8), and 48% (Run 9).

The total PAH emissions were generally consistent between runs, with the middle run (Run 8) about twice as high because of increased levels of specific PAHs.

The metals emissions were generally consistent between runs, with the exception of lower levels of Cr in Run 9 but higher levels of Cu and Hg. Pb was higher in Run 9.

Site D. There was no pollution control device at this site, and thus assessment of the data is limited to comparison of between-run data. Total dioxin and furan emissions did decrease over consecutive runs. The contribution of the 2,3,7,8-substituted dioxins to the total dioxins emissions was remarkably consistent across all three runs, although slightly increasing, i.e., 35%, 37%, and 42%. The trend for the substituted furans was somewhat consistent although decreasing across all three runs, i.e., 37%, 31%, and 29%.

The total PAH emissions were generally consistent between runs; however, the emission levels are slightly above the blank train levels.

The metals emissions were generally consistent between runs with the exception of higher levels of As, Cr, Cu, and Hg in Run 10 but lower levels of Ni in Run 12.

The differences in organic and metal emission between Sites C and D may be attributed to the contents of the drums burned during each sampling run at each of the plants. Further postulation about the effects of drum contents on emission levels would require knowledge of the contents of each drum, the effect of burning regimes, the process operating conditions at each facility during the time each drum was processed, and the emission levels during the burning of each.

5.3 COMPARISON WITH OTHER STUDIES

The data collected in this study are from waste oil users and drum reconditioner facilities that have not been previously sampled for dioxins and furans, PAHs, or metals. Indeed, their permit requirements have generally required only particulate sampling. Thus a data base to compare each of the specific facilities is not available.

The waste oil users have rotary kilns in their process operations and other features similar to the operations of cement kilns. The dioxin and furan emissions from the waste oil users is compared to the emissions from cement kilns as shown in Table 5-1. Overall, the stack emissions (expressed in lb/hr) from the waste oil users in this study are about 2 orders of magnitude lower than those from the cement kilns.

Metals emissions from the present study and from cement kilns are compared in Table 5-2. There is no apparent difference between the metals emissions from the studies shown.

The drum reconditions have dioxin and furan emissions (Table 5-1) that are higher than the waste oil users and are indeed comparable to those identified in the referenced cement kiln studies. With regard to the metals emissions (Table 5-2), emissions from the drum reconditions are generally comparable with the cement kilns, although As and Cd may be slightly higher in the drum facilities.

TABLE 5-1. COMPARISON OF DIOXIN AND FURAN EMISSIONS[a]

	Waste Oil Users		Drum Reclaimers				
	Site A[b]	Site B[b]	Site C[b]	Site D[b]	Site 1[c]	Site 2[d]	Site 3[e]
Total Dioxins	$1.0x10^{-8}$	$5.75x10^{-8}$	$4.59x10^{-6}$	$7.35x10^{-7}$	$7.05x10^{-6}$	$2.09x10^{-6}$	$1.04x10^{-5}$
Total Furans	$2.39x10^{-8}$	$9.99x10^{-8}$	$8.27x10^{-6}$	$4.52x10^{-7}$	$2.93x10^{-7}$	$4.03x10^{-7}$	$2.02x10^{-6}$
Total PCDD/PCDF emissions	$3.39x10^{-8}$	$15.7x10^{-8}$	$12.86x10^{-6}$	$11.87x10^{-7}$	$7.34x10^{-6}$	$2.49x10^{-6}$	$1.24x10^{-5}$

[a] Emission rates are in lb/hr and were measured at the outlet of emission control devices (if applicable) or at the stack.
[b] Average of three runs.
[c] State of California Air Resources Board, Evaluation of Combustion Processes for Destruction of Organic Wastes, General Portland.
[d] Summary Report on Particulate and Multi-Metals Testing, conducted at Dundee Cement, Clarksville, Missouri.
[e] Source Emissions Survey, Texas Industries, Inc., Midlothian, Texas.

TABLE 5-2. COMPARISON OF METALS EMISSIONS DATA[a]

	Waste Oil Users		Drum Reclaimers				
	Site A[b]	Site B[b]	Site C[b]	Site D[b]	Study 1[c]	Study 2[d]	Study 3[e]
Ag	.0003	.0004	.000592	.000615	.00075		.000212
As	.0004	.001	.00633	.00470	.0017		.000847
Ba	.00004	.0004	.00238	.000606	.0054	.002	.008
Cd	.0004	.001	.00512	.00515	.012		.000163
Cr	.0004	.009	.00148	.00053	.0038	< .001	.000719
Cu	.0002	.0007	.00101	.0082			
Hg	< .000025	.0001	.00808	.000152	.011		.000597
Mn	.23	.0015	.00082	.000321			
Ni	.0009	.047	.103	.084	.026		.000719
Pb	.001	.002	.0047	.00178	.070	.005	.002
Se	.0009	.095	.101	.0246	.018		< .003
Zn	.0006	.001	.000211	.00028			.005

[a] Emission rates are in lb/hr and were measured at the outlet of emission control devices (if applicable) or at the stack.

[b] Average of three runs.

[c] State of California Air Resources Board, Evaluation of Combustion Processes for Destruction of Organic Wastes, General Portland.

[d] Summary Report on Particulate and Multi-Metals Testing, conducted at Dundee Cement, Clarksville, Missouri.

[e] Source Emissions Survey, Texas Industries, Inc., Midlothian, Texas.

SECTION 6

QUALITY ASSURANCE SUMMARY REPORT

QA reviews and audits were conducted throughout the project by the Project QA Coordinator, which are summarized below.

6.1 PRELIMINARY REVIEW OF FIELD DATA

Field records, supporting data, and a draft data summary table were reviewed on November 27, 1990, while samples were being analyzed. Records from Site B, Run 6 were reviewed in detail. The records were evaluated versus the Project QA plan dated June 27, 1990.

The following items were reviewed:

- MM5 equipment calibrations and console checks.
- Run 6 data sheets, which included date/plant ID/personnel/equipment identifiers, nomograph, and isokinetic sampling worksheets, completed field data and sample recovery forms for MM5 inlet/outlet metals and organics. Secondary calculation checks were indicated on the data forms.
- Filter preparations, metal train reagents, CEM standard gas information (concentration, cylinder number, and expiration date), and solvent sources/lot numbers were recorded in LRB 2204.
- Run 6 process data which were recorded on standardized forms by plant personnel.
- Blank train records for metals and organics (one each) for Site B.
- MM5 test data (computer-generated data reduction pages) for Run 6—Inlet metals were audited versus field data sheets and CEM data.
- CEM span checks were verified to be within QC objectives for Run 6—Inlet metals.

Based on this review of the field records and audits of representative test data, the project records were found to be complete, organized, and traceable for metals emissions, organic emissions, and CEMs, with indication of technical review during the data reduction process. Test results which are presented in the draft summary table were found to be correctly represented when verified from supporting data and field records.

6.2 REVIEW OF DIOXINS AND FURANS AND POLYCYCLIC HYDROCARBON DATA

The dioxin/furan and PAH analysis summary tables for 18 sampling trains, 4 blank field trains, 2 laboratory method blanks, and 2 performance audit samples were provided for review in April 1991. The 18 sampling trains represented the following sources:

SAMPLE SOURCES
Site Designation (No. of Test Runs)

Source	A	B	C	D
Waste oil facility	inlet (x3) and outlet	inlet (x3) and outlet		
Drum rec. facility			outlet (x3)	outlet (x3)
Wire rec. facility				

Based on the audit of Run 1 Site A test results, overall QC data, and accuracy determinations from the internal audit samples, the subject dioxin/furan and PAH data (provided as draft tables) were found to be complete within the revised scope of work summarized above, traceable to original GC/MS data, and correctly presented.

6.2.1 Dioxins/Furans Accuracy Checks and QC Results

Two performance audit samples, comprised of a blank XAD/filter and a blank water sample spiked with an independent standard solution, were analyzed concurrently with the field test samples with the following results:

Spiked congener	% Recovery	
	Spiked XAD/ filter	Spiked water
2,3,7,8-TCDD	122	134
1,2,3,7,8-PeCDD	108	109
1,2,3,4,7,8-HxCDD	143	95
1,2,3,4,6,7,8-HpCDD	111	135
OCDD	162	96
2,3,7,8-TCDF	111	121
1,2,3,7,8-PeCDF	99	107
1,2,3,4,7,8-HxCDF	84	98
1,2,3,4,6,7,8-HpCDF	ND[a]	98
OCDF	ND[a]	117

[a] ND = not detected; ion ratios were outside the method-specified window of ±15%. Consistent with method protocol, detection limits (maximum possible concentrations) reported for these two congeners were higher, essentially bracketing the spiked concentrations.

Performance audit samples for PCDD/PCDF did not include all the 2,3,7,8-substituted isomers because it was not considered practical or necessary for a "survey" analysis of this type. In addition, performance audit samples were used to demonstrate the absence of false positive chemical identification.

Emission concentration and toxicity equivalents in the draft summary results for Run 1 from Site A were audited versus the GC/MS data and field sampling summary records. The draft results for this run were found to be traceable to the analysis data and correctly reported. Review of the overall QC results indicated the following:

- Blanks. Three of four field blank trains had significant background levels of 2,3,7,8-TCDD/TCDF and 2,3,7,8-substituted PeCDD/PeCDF congeners (highest toxicity ratings); other lower toxicity-rated congeners were also detected in both the field blanks and the laboratory method blanks. No blank corrections were applied to the test data.

- Surrogate and internal standard recoveries. Field surrogate recoveries were within the 60% to 140% range specified by the ARB method for all sampling trains and blank trains. Internal standard recoveries were all within method criteria of 40% to 120%, except for the labeled congeners for HxCDF, HpCDF, and HxCDF which ranged from about 140% to 160% for several runs. These high internal standard recoveries do not appear to significantly impact the quality of the data.

- GC/MS initial calibration. Precision of the initial 5-point calibration curve established on December 3, 1990, was within 15% RSD (relative standard deviation) for all PCDD/PCDF compounds, with the exception of ^{13}C-1,2,3,6,7,8-HxCDF which was at 25% RSD.

- GC/MS continuing calibration. Continuing calibration data from December 4, 1990, was examined and found to include several compounds outside 30% D (% difference from initial calibration), specifically:

Compound	% Difference
^{13}C-2,3,7,8-TCDF	+106%
^{13}C-1,2,3,7,8-PeCDD	−44%
1,2,3,7,8-PeCDD	+133%
1,2,3,6,7,8-HxCDF	+72%
1,2,3,4,7,8,9-HpCDF	+59%

6.2.2 PAH Accuracy Checks and QC Results

The same two performance audit samples (XAD/filter and water) were also spiked with PAHs from an independent EPA standard solution and extracted with the field test samples. Accuracy results were as follows:

Spiked compound	% Recovery	
	Spiked XAD/ filter	Spiked water
Acenaphthylene	75	62
Phenanthrene	93	77
Fluoranthene	131	111
Benzo[*a*]anthracene	114	135
Benzo[*b*]fluoranthene	82	79
Benzo[*k*]fluoranthene	107	105
Dibenz[*a,h*]anthracene	72	71
Benzo[*g,h,i*]perylene	80	79

Emission concentrations in the draft summary results for Run 1 from Site A was audited versus the GC/MS data and field sampling summary records. The draft results for this run were found to be traceable to the analysis data and correctly reported. Review of the overall QC results indicated the following:

- Blanks. Four field blanks which were reported were found to have no contaminants above the reporting limits except for naphthalene which was slightly above the reporting limit in one train only. The field blank trains show similar levels of naphthalene for all four locations. In comparing emissions from Sites A and B (waste oil users) which had high levels of naphthalene emissions, the blank levels were 7 to 10 times less than the minimum levels in the samples. In contrast, the blank levels were similar to but lower than the emission levels found in Sites C and D. With regard to the emission data, the levels of naphthalene are judged to be real based on the blank levels. The levels of naphthalene quantitated in the blanks were consistent with another study conducted during the same time period, although they are somewhat higher (by a factor of 2 to 3) compared to current blank levels.

- Surrogate and internal standard recoveries. Field surrogate recoveries ranged from 20% to 98%, with the majority (90%) of all surrogate recoveries above 40%. Internal standard recoveries were all within 40% to 100%, except for the d_8-naphthalene which ranged 7% to 59% recovery, and below 40% for a majority (80%) of the samples.

- GC/MS initial calibration. Precision of the initial 5-point calibration curve established on November 30, 1990, was within 27% RSD (relative standard deviation) for all target compounds, surrogates, and internal standards.

- GC/MS continuing calibration. Continuing calibration data from December 4, 1990, were examined and found to be within 12% difference from the initial calibration curve.

6.3 REVIEW OF METALS ANALYSIS DATA

The metals data were provided for review in conjunction with the draft report for Phase II.

For this review the following checks were made:

- Sample traceability and reported values for all metals were verified for Run 1—Inlet from Site A.

- QC data were reviewed, including spike recoveries from the simulated trains.

- Blank train data were reviewed to assure that there was no contamination or background interferences which would significantly impact the test data.

- Summary tables and derived metal emission values were checked for accuracy with the analytical reports and field data (e.g., stack flow, sample volume, etc.).

Based on the above review the metals data were found to be complete, traceable, supported by the QC data, and correctly reported with the following exception:

- As noted in the analyst's report, holding times for mercury were significantly exceeded, which could possibly reduce the reliability of these data.

6.4 REVIEW OF THE FINAL DRAFT REPORT

The final draft report was briefly reviewed. However, final derived values for PAH emissions, metals emissions, and PCDD/PCDF emissions with associated toxic equivalents summations were spot-checked for Run 1 inlet samples in the main report tables. Results from multiple runs were also randomly cross-checked for consistency and reasonableness of the test data.

APPENDIX A

DESCRIPTION OF SAMPLING AND ANALYSIS METHODS

Sampling Procedures
Continuous Emission Monitoring Procedures
PCDD/PCDF Analytical Procedures
Metals Analytical Procedures

SAMPLING PROCEDURES

The sampling and analysis procedures proposed for this test program are described in this section. The published methods and other written procedures to be used are cited here and are appended in Volume III. Details providing clarification and deviations from the published methods are presented in this section. Otherwise, the cited methods will be reasonably followed.

References used in developing this protocol are as follows:

1. Title 17 and 26, California Air Resources Board
2. California AB2588 Emission Inventory Criteria and Guidelines
3. Code of Federal Regulations, Title 40, Part 60 (40 CFR 60)
4. "Quality Assurance Handbook for Air Pollution Measurement Systems, Volume III, Stationary Source Specific Methods," EPA-600/4-77-027b

For all reported test results adjusted to standard conditions from this test program, those standard conditions WERE 68°F (528°R) and 29.92 inHg as specified in the California ARB method.

The largest portion of the scope of work involved California ARB Methods 5, 428, 429, and 436. All of these methods necessitate the use of ARB Methods 1 through 4, which will be discussed first below.

ARB Method 1 was used to determine sampling port placement on the circular and rectangular stacks and for sampling point locations in the gas stream. A check for cyclonic gas flow was conducted in accordance with this method.

ARB Method 2 was used for determining stack gas velocities and volumetric flow rates with Type S pitot tubes. Inclined manometers were used as differential pressure gauges.

Type K thermocouples were used for all temperature measurements site during this test program. All temperature measurement systems were calibrated against ASTM mercury-in-glass reference thermometers for temperatures up to 750°F. For higher temperatures up to 2500°F, an NBS-calibrated reference thermocouple-potentiometer were used.

An aneroid barometer that had been calibrated against an NBS traceable mercury barometer at MRI was used for all barometric pressure readings on-site. The mercury barometer readings are corrected for temperature and gravity.

All Type S pitot tubes are calibrated in a wind tunnel against a hemispherical-tipped standard pitot tube (baseline coefficient of 0.99) designed according to specifications in ARB Method 2, Section 2.7. Pitot tubes to be used for this test program will be calibrated in the range of velocities expected during sampling. The pitot tubes were examined and measured for critical dimensions after the test to ensure that they were not damaged during use. Pitot tube assemblies met the intercomponent spacing requirements described in ARB Method 2, Section 4.1.1.

Stack gas dry molecular weight was determined in accordance with ARB Method 3, and moisture content was determined in accordance with ARB Method 4.

The stack cross-sectional area was determined from MRI's measurements of two diameters.

DIOXINS (PCDDs), FURANS (PCDFs), AND PAHs

One modified Method 5 sampling train and ARB Methods 428 and 429 was used to determine emissions of polychlorinated dibenzo-pdioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), and polycyclic aromatic hydrocarbons (PAHs). A schematic of the sampling train is shown in Figure A-1.

Sampling Procedures

The following describes the deviations from and some clarifications for the sampling procedures in ARB Methods 428 and 429 that were used in the single sampling train combining both of those methods.

General Considerations

Proposed sampling time per sampling run is 180 min with an approximate 3 dscm sample of stack gas being extracted.

Apparatus for Sampling

Stainless steel sampling nozzles were used.

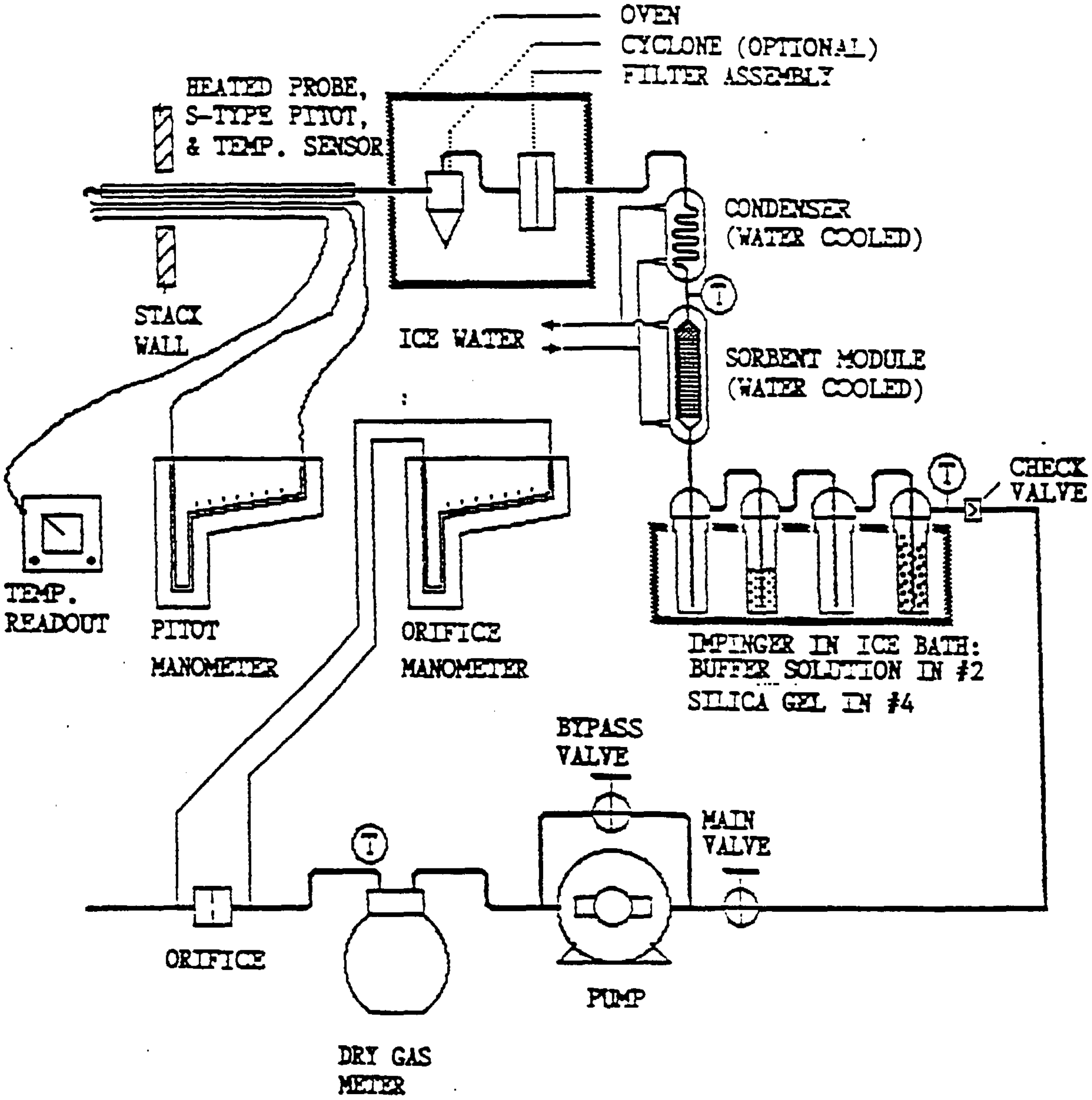


Figure A-1. PCDD and PAH sampling train (from California ARB Method 429).

The sample transfer line between the filter holder outlet and the condenser inlet was glass or Teflon. The line was heated (248° ± 25°F) and insulated to prevent condensation in the gas stream before entry into the condenser.

The impingers were arranged and modified as described in ARB Methods 428 and 429 and the second impinger was charged with 100 mL of the sodium bicarbonate/sodium carbonate solution described in ARB Method 429, Sections 2.2.8 and 2.3.4.

Gas temperature was monitored at the inlet to the sorbent trap to within 2°F with a thermocouple-pyrometer.

The gas temperature at the outlet of the last impinger (containing silica gel) was monitored to within 2°F with a thermocouple-pyrometer.

Gas temperature at the dry gas meter was averaged from measurements taken at the inlet and the outlet of the meter.

Apparatus for Recovery

Only Teflon wash bottles were used.

All sample storage containers were amber glass bottles with Teflon screw cap liners. Narrow mouth bottles were used for liquid samples. Wide mouth bottles were used for filters. Bottles were procured from I-CHEM Research and will be precleaned according to EPA Standard Cleaning Protocol A. Bottle lots were analyzed by I-CHEM and "Certificates of Analysis" along with QC lot numbers assigned for traceability were provided to MRI. Bottles used for samples to be analyzed for PCDDs, PCDFs, and PAHs were given a final rinse with toluene before use.

Glass caps and plugs with Teflon ferrules compressed with nylon bushings (blank off assemblies) were used to seal sorbent cartridges.

Reagents

The Whatman 934-AH filters to be used for field sampling were not precleaned, but were subjected to a QC check as described in ARB Methods 428 and 429, Section 2.3.1. Blank corrections from the QC check results were not applied to stack sample data.

Toluene (Burdick & Jackson Cat. No. 347) stored in original glass containers were used for recovery.

Cleaning Glassware

All glass, quartz, and Teflon parts of the sampling train that were in contact with the sampled gas and all other applicable labware including water and prepared reagent storage bottles that will be used for setup and recovery in the field were cleaned as described in Section 3A (revised 12/15/79) of the "Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples," EPA-600/8-80-038. The solvents to be used in Step 8 of that section were acetone followed by methylene chloride followed by toluene.

Surrogate Standards

The surrogate standards to be added to the XAD-2 resin in the sorbent cartridges in the preplaboratory prior to shipment are:

For PCDDs and PCDFs	For PAHs
^{37}Cl-2,3,7,8-TCDD	D_{10}-Biphenyl
$^{13}C_{12}$-2,3,4,7,8-PeCDF	
$^{13}C_{12}$-1,2,3,4,7,8-HxCDD	
$^{13}C_{12}$-1,2,3,6,7,8-HxCDF	
$^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF	

Pretest Procedures

In addition to the impingers, the sorbent cartridge were also weighed during setup for the purpose of determining condensed moisture collection. The sorbent cartridges were wrapped with aluminum foil during sampling and shipment to the analytical laboratory to seal out light.

Correcting for Excessive Leak Rates

If a post sampling leak rate in the train was in excess of the allowable rate and the leak did not warrant invalidating the sample, the gas sample volume was corrected according to ARB Method 5, Section 6.3.

Blank Train

One blank train was setup, operated, and recovered as described in ARB Methods 428 and 429, Section 2.6.1. The train was assembled from clean components not previously used during the test. One blank train was assembled and recovered per test site. The probe-sample box was on the roof near the stack during one of the sampling runs.

Sample Recovery

After sampling and the final leak checks were completed, all sampling trains with similar configurations used during this test program were prepared for transfer to the sample recovery areas as follows:

1. External particulate matter near the tip of the sampling nozzle was removed and the nozzle opening was appropriately capped.

2. The probe was disconnected from the sample box. The probe liner outlet was sealed with a capped blank off assembly, and the entire end was covered with a cleaned aluminum foil cap. The inlet to the glass elbow connected to the filter holder inlet was sealed with a plugged blank off assembly.

3. The inlet of the sample transfer line was disconnected from the filter holder outlet, and both openings were sealed with blank off assemblies.

4. The outlet of the sample transfer line was disconnected from the condenser inlet, and both openings were sealed with blank off assemblies.

5. The condenser was removed from the sorbent cartridge and the condenser outlet and sorbent cartridge inlet openings were likewise be sealed.

6. The sorbent cartridge were removed from the inlet of the first impinger, and both openings were sealed with blank off assemblies.

Six sample components in the containers described below were generated for each sampling train and the blank train along with blank samples of appropriate volumes of all reagents and blank filters. All sample bottles were tare weighed with caps and preprinted labels. Gross weights were obtained for all fully recovered liquid samples including their respective rinses. This will enable the recipient laboratory to determine any sample loss during shipment should that occur.

Container No. 1. Sample material collected in the sampling nozzle, probe liner, elbow, and the front half of the filter holder was recovered as described in

ARB Methods 428 and 429, Section 3.4.4, except that the train components will be sequentially rinsed with acetone, methylene chloride, and toluene three times each.

Container No. 2. The filter was recovered as described in ARB Methods 428 and 429, Section 3.4.1.

Container No. 3. The remaining water in the water jacket of the sorbent cartridge was removed and the cartridge reweighed. Condensor condensate was added to the sorbent cartridge before weighing. The sealed cartridge were wrapped in aluminum foil for shipment to the analytical laboratory.

Container No. 4. The filter holder support, the back half of the filter holder, the sample transfer line, and the condenser were sequentially rinsed with acetone, methylene chloride, and toluene three times each.

All four impingers were reweighed for moisture determination before the contents were transferred.

Container No. 5. The contents of the first impinger were transferred to the sample bottle with the aid of a funnel, without separating the impinger top from the impinger bottle, and while rotating and agitating the impinger. The impinger was sequentially rinsed with water, acetone, methylene chloride, and toluene three times each.

Container No. 6. The contents of the second and third impingers were transferred, and those impingers, along with all common connecting components, were rinsed into the sample bottle in the same manner as described for the previous sample component.

All six sample components and reagent blanks were stored at water-ice temperature (4°C) at the test site and during shipment to the analytical laboratory. The filter was not stored on dry ice as described in ARB Method 429, Section 3.5.

Analysis Procedures for Dioxins (PCDDs) and PAHs

The analytical procedures used are described in the subsequent results memo.

MULTIPLE METALS

One modified Method 5 sampling train and the proposed ARB Method 436 was used to determine emissions of total Ag, As, Ba, Cd, Cr, Cu, Hg, Mn, Ni, Pb, Se and Zn. A schematic of the sampling train is shown in Figure A-2.

Sampling Procedures

The following describes the deviations from and some clarifications for the sampling procedures in the proposed ARB Method 436 (undated draft received prior to 9/27/90) that will be used during this test program.

Apparatus for Sampling

Quartz sampling nozzles were used.

Apparatus for Recovery

Polyethylene wash bottles were used to dispense all reagents.

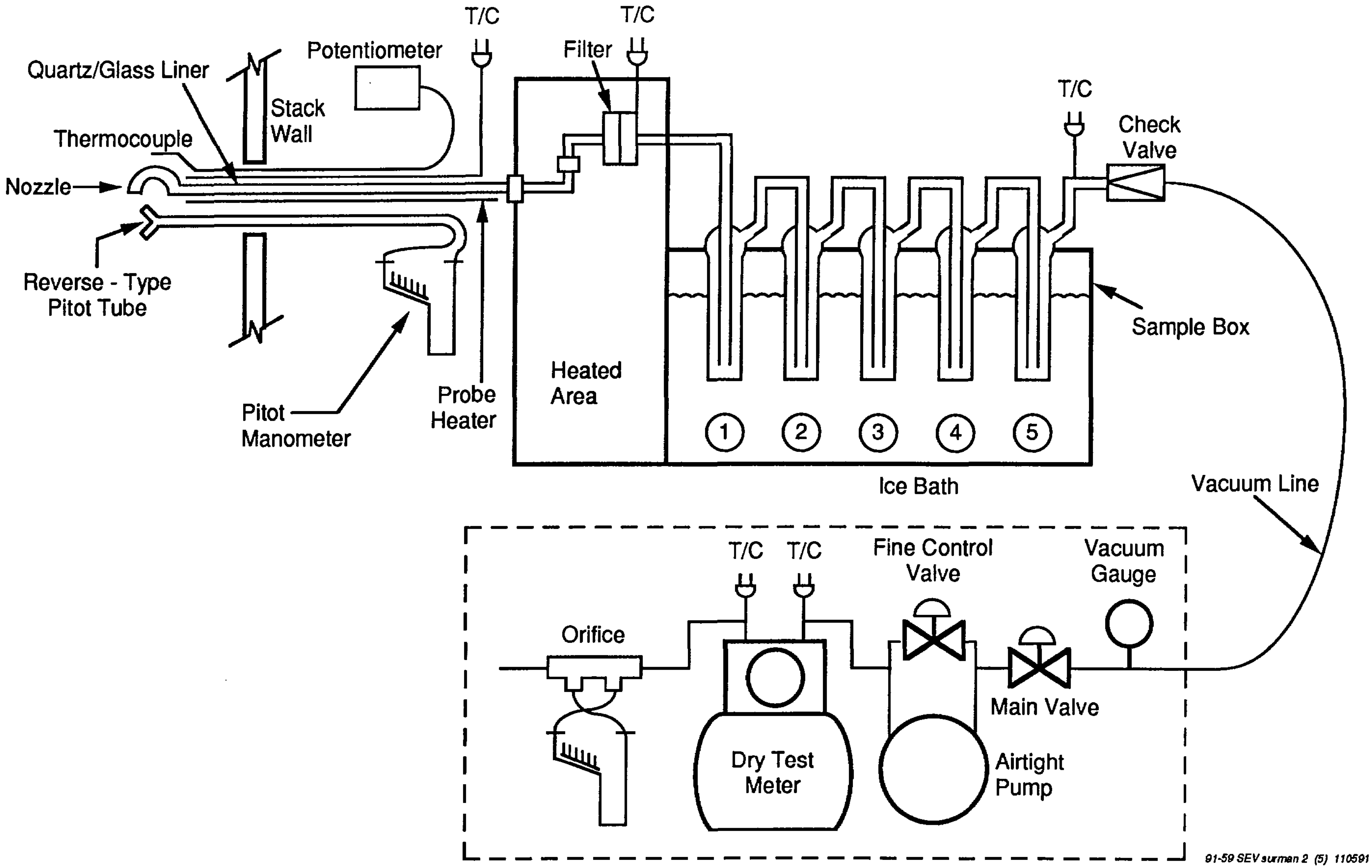
All sample storage containers were the same as those described previously in this plan.

Reagents

Whatman QM-A quartz filters exhibiting a 99.999% efficiency (0.001% penetration) on 0.6-)m particles at 5 cm/s face velocity were used.

Deionized, distilled water meeting ASTM Method D1193-77 specifications for Type I water were used in all sampling and analysis operations.

Nitric acid, concentrated, (Baker Instra-analyzed) was used in all sampling and analysis operations.



91-59 SEV surman 2 (5) 110591

① Modified Greenburg-Smith, 100mL 5% HNO_3 and 10% H_2O_2 Soln
② Greenburg-Smith, 100mL 5% HNO_3 and 10% H_2O_2 Soln
③ Modified Greenburg-Smith, Empty
④ Modified Greenburg-Smith, 100mL Acidic $KMnO_4$
⑤ Modified Greenburg-Smith, Silica Gel

Figure A-2. Multiple metals sampling train.

Cleaning Glassware

All glass, quartz, and Teflon parts of the sampling train that were in contact with the sampled gas and all other applicable labware including water and prepared reagent storage bottles that were used for setup and recovery in the field were cleaned as described in ARB Method 436, Section 5.1.1, and the openings of those items were sealed or covered with parafilm.

Pretest Procedures

The impingers were set up as follows:

1st impinger—100 mL 5% HNO_3 and 10% H_2O_2 solution
2nd impinger—100 mL 5% HNO_3 and 10% H_2O_2 solution
3rd impinger—Empty
4th impinger—100 mL $KMnO_4$
5th impinger—200 g silica gel

The contents of the fourth impinger acted as an acid trap to protect downstream components of the sampling train. The contents of the fourth and fifth impingers was not retained for analysis after recovery.

Blank Train

One blank train was set up, operated, and recovered as described in ARB Method 436, Section 5.1.6 at each test site. The train was assembled from clean components not previously used during the test. The probe-sample box was near the stack during one of the sampling runs.

Sample Recovery

Preparation and transfer of the sampling train components from the sampling location to the recovery areas was as previously outlined in this plan.

Four sample components in the containers described below were generated for each sampling train and the blank train along with blank samples of appropriate volumes of all reagents and blank filters. All sample bottles were tare weighed with caps and preprinted labels. Gross weights were obtained for all fully recovered liquid samples including their respective rinses. This enabled the recipient laboratory to determine any sample loss during shipment. Weighing was conducted during recovery so that weights of recovered samples and the actual weights of their associated rinses could be separately determined.

The total volume of each rinse reagent used for recovery was premeasured gravimetrically in the wash bottles and the entire volume of each was used. This was done to maintain consistent rinse volumes among the stack samples from each run and the blank train samples.

Container No. 1. Sample material collected in the sampling nozzle, probe liner, elbow, and the front half of the filter holder was recovered with acetone as described in ARB Method 436, Section 5.2.2, except that the total amount of acetone used for each rinse was not measured. The final volume was determined from the tare and gross bottle weights obtained during recovery.

Container No. 2. Sample recovery was the same as for the previous sample and as described in ARB Method 436, Section 5.2.3, using 0.1N nitric acid as the the recovery reagent, except that brushing was not employed during this operation since all particulate matter had been removed during the previous operation.

Container No. 3. The filter was recovered as described in ARB Method 436, Section 5.2.1, with the aid of nonmetallic forceps, except that the recovered sample was stored in a sample bottle rather than in a petri dish.

All six impingers were reweighed for moisture determination before the contents were transferred.

Container No. 4. This contained a sample recovered as follows:

1. The contents of the first three impingers were transferred to the sample bottle with the aid of a funnel, without separating the impinger tops from the impinger bottles, and while rotating and agitating each impinger.

2. The sample bottle was weighed.

3. Each impinger was rinsed two times (about 30 mL per impinger rinse) and all connecting components inclusive from the back half of the filter holder (including the filter support) through the third impinger was rinsed two times with 0.1N nitric acid into the sample bottle.

4. The sample bottle was weighed.

Container No. 5.

1. The contents of impingers 3 and 4 were transferred to a sample bottle with the aid of a funnel, without separating the impinger tops from the impinger bottles, and while rotating and agitating the impingers.
2. The sample bottle was weighed.
3. The impingers were rinsed with 15 mL of 8 N HCl three times each.
4. The sample was stored at water ice temperature (4°C) at the test site and during shipment to the analytical laboratory.

The pH of the recovered samples in Container Nos. 2 and 4 was checked with glass rods and indicator strips and was adjusted with concentrated nitric acid.

All five sample components and reagent blanks were stored at water-ice temperature (4°C) at the test site and during shipment to the analytical laboratory.

Reagent Blanks

Reagent blank samples were saved as described in ARB Method 436, Sections 5.2.7 through 5.2.9 and 5.2.12, and will be archived for 6 months after issuance of the final test report. These samples will not be processed and analyzed as described in ARB Method 436, Section 7.1., because the method also requires the employment of a blank train (Section 5.1.6), and for this study the blank train samples were analyzed in place of the reagent blank samples.

Analysis Procedures for Total Metals

The analytical procedures used are described in the subsequent results memo.

Continuous Emission Monitoring

Continuous emission monitoring (CEM) data was collected during all twelve test runs for the CARB Survey from October 17 to November 8, 1990.

At each sampling location (i.e., stack and inlet ducts) sample gas was collected using a single probe with sintered filter. The gas was then extracted through a heated line into a heated manifold. The sample line temperature was

maintained at 250°F. The sample gas temperature was also maintained at 250°F in the manifold until after the moisture was removed. The moisture was removed with the use of a Perma-Pure filter integrated into the heated manifold. The dry sample gas was then transferred to the output of the manifold to each CEM.

The following monitors were used for data collection:

Inlet

Type	Model	Manufacturer
Carbon Monoxide	PIR 2000L	Horiba
Carbon Dioxide	PIR 2000S	Horiba
Oxygen	PMA 200	Horiba
Sulfur Dioxide	721AT2	Western Research
Nitric Oxide	10AR	Thermo Electron

Outlet

Type	Model	Manufacturer
Carbon Monoxide	PIR 2000L	Horiba
Carbon Dioxide	PIR 2000S	Horiba
Oxygen	Portable Oxygen Analyzer	Teledyne
Sulfur Dioxide	721AT2	Western Research
Nitric Oxide	10AR	Thermo Electron

Total CO concentration was determined using a Horiba Model PIR-2000L NDIR. After the CO sample was split from the manifold, it was passed through an ascarite/silica gel cartridge containing approximately 200 g of ascarite and 20 g of silica gel. The ascarite trap removed carbon dioxide, which is an interference to the CO monitor, and the silica gel removed the last traces of moisture prior to the monitor. The sample fraction was then pumped to the NDIR analyzer.

Zero drift was determined by checking the zero calibration before and after each run and comparing the two. Calibration drift was determined by checking the span gas calibration before and after each run. The calibration error (usually referred to as the linearity check) was performed by zeroing and spanning the instrument and then adding a midlevel calibration gas and comparing the instrument value with the real gas value. Zero and calibration drift were less than ±3% of the span value, while the calibration error (linearity check) was less than ±2% of the calibration gas value for each run.

Possible bias on the sample line was checked before the first run at each testing site. Bias check was performed in the same manner as the calibration check, except the calibration gases were introduced into the probe instead of at the analyzer.

All calibration gases were EPA Protocol 1 in date cylinders.

The performance checks for the analyzers are summarized below:

Zero drift: ±3% of span
Span drift: ±3% of span
Linearity checks: ±2% of cylinder gas value
Bias check: ±5%
Leak checks: < 4% of normal flow, before and after each run

Nominal gas concentrations:

Span	Linearity
CO—392.8 ppm	197.9 ppm
CO_2—12.03%	6.06%
O_2—11.89%	5.97%
NO_x—197.2ppm	100.9 ppm
SO_2—401.1ppm	201.0 ppm

The system was leak-checked before and after each run at less than 200 mm Hg. The monitors were zeroed using prepurified nitrogen and spanned using the lowest calibration gas available. Following each run, a final zero and span was performed and the monitors purged for at least 30 min with nitrogen before shutting down.

Raw data from the field CEM printouts were reviewed for completeness and any notations of the operator. Data presented here was collected only during semivolatile sampling on the stack. Invalid data periods due to maintenance activities on the sampling system have also been removed from these data tables.

CEM Data Reduction

Raw data were refined, as follows, to generate final data values (i.e., averages, etc).

- The CEM raw data was first converted from percent of full-scale values to percent (O_2 and CO_2) or ppm CO, NO_x and SO_2 values using a data logging program. This conversion was based upon the average of initial and final zero and span calibration data.

- CO, NO_x and SO_2 were corrected to 12% CO_2 conditions using the CO_2 data collected during each run was carbon monoxide, sulfur dioxide and nitric oxide have been corrected to 12% CO_2. The correction is by the equation:

$$\text{Conc}_{(\text{ppm})} \text{ at } 12\% \ CO_2 = \text{Raw Conc}_{(\text{ppm})} \times \left(\frac{12}{CO_2\% \text{ measured}}\right)$$

STANDARD GASES

Gas	Source	ID No.	Concentration	Expiration Date
NO	Scott Specialty Gases	AAL 9664	197.7 ppm	6-13-91
NO	Scott Specialty Gases	ALM 7095	100.9 ppm	3-16-91
SO_2	Scott Specialty Gases	AAL 935	401.1 ppm	3-18-91
SO_2	Scott Specialty Gases	AAL 19119	201.0 ppm	10-23-91
O_2	Scott Specialty Gases	ALM 4752	11.89%	3-1-91
O_2	Scott Specialty Gases	AAL 1672	5.97%	2-6-92
CO	Scott Specialty Gases	AAL 14318	197.9 ppm	12-12-91
CO	Scott Specialty Gases	ALM 2211	392.8 ppm	1-6-91
CO_2	Scott Specialty Gases	ALM 18638	12.03%	12-11-91
CO_2	Scott Specialty Gases	ALM 4643	6.06%	12-11-91

INTEROFFICE COMMUNICATION

MIDWEST RESEARCH INSTITUTE

April 30, 1991

TO: John Hosenfeld

FROM: Andrés Romeu

SUBJECT: Analytical Results of CARB samples, MRI Project 9420R

Eighteen sample trains and four blank train collected from four sites in California (two waste oil facilities - inlet and outlet, and two drum reconditioning facilities) were analyzed for PAHs and PCDD/PCDFs according to California Air Resources Board Methods 428 and 429.

CRITICAL DATES

Samples were collected during October-November, 1990 and received at MRI within two weeks after sample collection. Table 1 summarizes the critical dates in the collection, preparation, and analysis of these samples.

Table 1. Critical date summary (all dates are for 1990).

Site	Sampling date(s)	Solvent extraction date(s)	GC/MS analysis date(s)	
			PAH	PCDD/F
1		Oct. 31	Dec. 4-5	Dec. 4-7
2	Oct. 24-26	Nov. 7	Dec. 4-5	Dec. 4-7
3	Oct. 30-Nov. 1	Nov. 12	Dec. 4-5	Dec. 4-7
4	Nov. 1-8	Nov. 12	Dec. 4-5	Dec. 4-7

All samples were extracted within 14 days of collection, and analyzed within 30 days of extraction.

SAMPLE PREPARATION

The samples were extracted using ARB Methods 428 and 429 with some modifications.

- Each sample was analyzed for both PAHs and PCDD/PCDFs. This was accomplished by splitting the samples into three equal portions. One portion was archived. A second portion was cleaned up according to ARB Method 429 and analyzed for PAHs (final volume - 250 μL). The last portion was cleaned up according to ARB Method 428 and analyzed for PCDD/PCDFs (final volume - 25 μL).
- Sampling train filters were not solvent extracted prior to sampling. Instead, one filter from a lot was randomly chosen and screened to determined if the lot was contaminated.
- The sampling train filter was not cut into strips because we felt that this might compromise the filter catch.
- The front and back half rinses were back-extracted using the impinger condensate and, when necessary, reagent grade water. These combined samples were extracted with methylene chloride and toluene.
- The sampling train filters and XAD-2 resin were coextracted using toluene.
- The combined front half, back half and condensate extracts were composited with the combined filter/XAD-2 extracts to generate a single extract per sampling train.
- Several PAH internal standards were not available (D_8-acenaphthylene, D_{12}-benzo[b]fluoranthene, D_{12}-benzo[k]fluoranthene, D_{12}-benzo[ghi]perylene, and D_{14}-indeno[1,2,3-cd]pyrene).

- The column performance mix used during the analysis of PCDD/PCDFs differed slightly from that required by ARB Method 428.
- The gas chromatographic conditions used for analysis of PCDD/PCDFs differed slightly from those required by ARB Method 428.
- No confirmation analyses for PCDD/PCDFs were made.

Copies of the MRI Standard Operating Procedures prepared specifically for this project are included in the data packet.

PAH ANALYSIS

A 5-point calibration curve was analyzed on November 30, 1990. The calibration curve passed all of the required criteria oF ARB Method 429. A daily standard was analyzed at the beginning of every 12-hour analysis period and compared to the initial calibration curve. All criteria from ARB Method 429 were met. In addition, DFTPP was analyzed at the beginning of every 12-hour analysis period (before the daily standard). The ion abundance criteria from EPA Method 1625 were used to confirm proper tuning of the mass spectrometer.

Below is a list of the native target compounds and their corresponding labeled compounds which were used to quantitate the sample concentrations.

Internal Standards	Native Compounds
D_8-Naphthalene	Naphthalene
D_{10}-Acenaphthene	Acenaphthylene, Acenaphthene
D_{10}-Fluorene	Fluorene, Fluoranthene
D_{10}-Phenanthrene	Phenanthrene
D_{10}-Anthracene	Anthracene
D_{10}-Pyrene	Pyrene, Retene
D_{12}-Benz(a)anthracene	Benz(a)anthracene, Benzo(k)fluoranthene, Dibenz(a,h)anthracene
D_{12}-Chrysene	Chrysene
D_{12}-Benzo(a)pyrene	Benzo(a)pyrene, Indeno(1,2,3-cd)pyrene, Benzo(ghi)perylene

Two performance audit samples were analyzed. These consisted of a spike into each matrix tested (combined XAD/filter and water). The spike was prepared independently from all standards prepared for this project. The results of the performance audit samples are presented in Table 2.

Table 2. Results of PAH performance audit samples.

Compound	% Recovery	
	XAD/filter	Water
Acenaphthylene	75	62
Phenanthrene	93	77
Fluoranthene	131	111
Benzo[a]anthracene	114	135
Benzo[b]fluoranthene	82	79
Benzo[k]fluoranthene	107	105
Dibenze[a,h]anthracene	72	71
Benzo[ghi]perylene	80	79

Two field surrogates and ten internal standards were added to each sample to monitor sampling and analysis accuracy. Summary statistics can be used to interpret the precision of the sampling and analysis of these samples. Table 3 summarizes the results of surrogate and internal standard recoveries. The results indicate that field surrogate and internal standard recoveries were high and reproducible. D_8-Naphthalene, which has a high vapor pressure, is the sole exception. Without normalization to internal standard recoveries, naphthalene values for the sampling trains might be biased low.

Table 3. Summary of PAH field surrogate and internal standard recoveries.

Compound	Average recovery %	RSD (%)
Field surrogates		
D_{10}-1-Methylnaphthalene	64	24
D_{12}-Perylene	66	32
Laboratory internal standards		
D_8-Naphthalene	37	36
D_{10}-Acenaphthene	72	14
D_{10}-Fluorene	78	12
D_{10}-Phenanthrene	85	11
D_{10}-Anthracene	77	11
D_{10}-Fluoranthene	84	12
D_{10}-Pyrene	84	11
D_{12}-Benz[a]anthracene	81	18
D_{12}-Chrysene	84	16
D_{12}-Benzo[a]pyrene	76	22

Four field blank trains and several method blanks were analyzed. With the exception of one field blank train, none of the target compounds were found in any of the blanks. Naphthalene was found in one field blank train, at a level slightly higher than the reporting limit.

Extract concentrations were calculated against the average RRF obtained from the initial calibration curve. The sample concentrations were calculated using the following equation:

$$\textit{Sample conc.}\ (\mu g) = \textit{Extract conc}\ (\mu g/mL) \times \textit{Extract vol}\ (mL) \times \textit{Split factor} \times \textit{Dilution factor} \tag{1}$$

The upper and lower reporting limits were calculated as above, using the concentration of the most dilute and most concentrated calibration standards included in the initial calibration curve for the extract concentration variable.

PCDD/PCDF RESULTS

A calibration curve was analyzed on December 3, 1990. All of the target compounds met the linearity criterion of 25% RSD or less of ARB Method 428. A daily standard was analyzed at the beginning and end of every 12-hour analysis period and compared to the initial calibration curve. The daily standards were less than 30% different from the initial calibration curve.

Two performance audit samples were analyzed. These consisted of a spike into each matrix tested (combined XAD/filter and water). The spike was prepared independently from all standards prepared for this project. The results of the performance audit samples are presented in Table 4.

Table 4. Results of PCDD/PCDF performance audit samples.

Compound	% Recovery XAD/filter	% Recovery Water
2,3,7,8-TCDD	122	134
1,2,3,7,8-PeCDD	108	109
1,2,3,4,7,8-HxCDD	143	95
1,2,3,4,6,7,8-HpCDD	111	135
OCDD	162	96
2,3,7,8-TCDF	111	121
1,2,3,7,8-PeCDF	99	107
1,2,3,4,7,8-HxCDF	84	98
1,2,3,4,6,7,8-HpCDF	[a]	98
OCDF	[a]	117

[a] Analytes detected, but ion ratios were outside the method-specified window of ±15%. Consistent with method protocol, detection limits (maximum possible concentrations) reported for these congeners were higher, essentially bracketing the spiked concentrations.

Five field surrogates and nine internal standards were added to each sample to monitor sampling and analysis accuracy. The amount of PCDD/PCDF lab and field surrogate added to each sample was 150 μl. The sample was split 3 ways and the final concentration is shown in Table 5.

Table 5. Field and Laboratory Surrogates

Field Surrogate	pg/μL
^{37}Cl-2,3,7,8-TCDD	50
^{13}C-2,3,4,7,8-PeCDF	50
^{13}C-1,2,3,4,7,8-HxCDD	125
^{13}C-1,2,3,6,7,8-HxCDF	125
^{13}C-1,2,3,4,7,8,9-HpCDF	125
Laboratory Surrogate	
^{13}C-2,3,7,8-TCDD	50
^{13}C-2,3,7,8-TCDF	50
^{13}C-1,2,3,7,8-PeCDD	50
^{13}C-1,2,3,7,8-PeCDF	50
^{13}C-1,2,3,6,7,8-HxCDD	125
^{13}C-1,2,3,6,7,8-HxCDF	125
^{13}C-1,2,3,4,6,7,8-HpCDD	125
^{13}C-1,2,3,4,6,7,8-HpCDF	125
^{13}C-OCDD	250

Summary statistics can be used to interpret the precision of the sampling and analysis of these samples. Table 6 summarizes the results of field surrogate and internal standard recoveries. The results indicate that field surrogate and internal standard recoveries were high and reproducible.

Table 6. Summary of PCDD/PCDF field surrogate and internal standard recoveries.

Compound	Average recovery (%)	RSD (%)
Field surrogates		
^{37}Cl-2,3,7,8-TCDD	95	3.6
^{13}C-2,3,4,7,8-PeCDF	93.7	4.2
^{13}C-1,2,3,6,7,8-HxCDF	73.8	5.4
^{13}C-1,2,3,4,7,8-HxCDD	101	4.8
^{13}C-1,2,3,4,7,8,9-HpCDF	90.6	6.9

Laboratory internal standards		
^{13}C-2,3,7,8-TCDF	104	12
^{13}C-2,3,7,8-TCDD	96.8	11
^{13}C-1,2,3,7,8-PeCDF	98.5	14
^{13}C-1,2,3,7,8-PeCDD	97.6	14
^{13}C-1,2,3,4,7,8-HxCDF	124	13
^{13}C-1,2,3,6,7,8-HxCDD	102	12
^{13}C-1,2,3,4,6,7,8-HpCDF	113	15
^{13}C-1,2,3,4,6,7,8-HpCDD	112	14
^{13}C-OCDD	104	15

Below is a list of the native target compounds and their corresponding internal standards which were used to quantify them.

Internal Standards	Native Compounds
^{13}C-2,3,7,8-TCDF	2,3,7,8-TCDF
^{13}C-2,3,7,8-TCDD	2,3,7,8-TCDD
^{13}C-1,2,3,7,8-PeCDF	1,2,3,7,8-PeCDF; 2,3,4,7,8-PeCDF 1,2,3,4,7,8-PeCDF
^{13}C-1,2,3,7,8-PeCDD	1,2,3,7,8-PeCDD
^{13}C-1,2,3,4,7,8-HxCDF	1,2,3,4,7,8-HxCDF; 1,2,3,6,7,8-HxCDF 2,3,4,6,7,8-HxCDF; 1,2,3,7,8,9-HxCDF
^{13}C-1,2,3,6,7,8-HxCDD	1,2,3,4,7,8-HxCDD; 1,2,3,6,7,8-Hx CDD 1,2,3,7,8,9-HxCDD
^{13}C-1,2,3,4,6,7,8-HpCDF	1,2,3,4,6,7,8,-HpCDF; 1,2,3,4,7,8,9-HpCDF
^{13}C-1,2,3,4,6,7,8-HpCDD	1,2,3,4,6,7,8-HpCDD
^{13}C-OCDD	OCDF, OCDD

Four field blank trains and several method blanks were analyzed. Three of the field blank trains had significant background concentrations of tetra- and penta-chlorinated dioxins and furans. Some contamination was found in the all of the field blank trains and in the method blanks. No blank corrections were applied to the test data.

Sample concentrations were calculated using Equation 1.

INTEROFFICE COMMUNICATION

MIDWEST RESEARCH INSTITUTE

July 26, 1991

To:	J. Hosenfeld
From:	E. McClendon
Subject:	Analysis of Metals Train Samples for Test Burns for State of California Air Resources Board; Project 9420-0255

Enclosed are the results of the inductively coupled argon plasma (ICP) and cold vapor atomic absorption (CVAA) analyses performed for Project 9420-0255.

I. Introduction and Request for Analysis

These samples were submitted to determine their metals content to determine the metals emissions of several different incinerators burning different waste streams. The analytes of interest were arsenic (As), barium (Ba), cadmium (Cd), total chromium (Cr), copper (Cu), mercury (Hg), lead (Pb), manganese (Mn), nickel (Ni), selenium (Se), silver (Ag) and zinc (Zn). The analyses were performed on the Jarrell-Ash Model Mark III ICP-AES, Perkin-Elmer Plasma II ICP-AES and the GBC Model 902 atomic absorption spectrometer.

II. Submission of Samples for Analysis and Sample Preparation

A total of 103 samples were received in the Atomic Spectrometry Facility for metals analysis by E. McClendon on October 23, 25, November 6 and 9, 1990. The samples can be split into four groups: front half train samples (comprised of filters, acetone probe rinses and nitric acid probe rinses, both of which may contain excessive particulate); back half train samples (comprised of nitric acid/hydrogen peroxide impingers and their nitric acid rinses); and permanganate impingers and rinses. The back half samples were analyzed for all analytes. The permanganate impingers were analyzed only for Hg. The front half samples from site 1 (approximately one-third of the front half samples) were analyzed for all analytes, while the remaining front half samples were analyzed for all analytes except Hg. Details of the reagents used for sample preparation can be found in the notebook pages.

The back half train samples were prepared for analysis by initially removing an aliquot for the Hg analysis, then reducing the remaining volume to less than 20 mL by heating without boiling. This reduced volume sample was then digested with nitric acid and hydrogen peroxide according to CARB Method 436, with modifications as listed in Appendix A.

The front half train samples were prepared for analysis by resolubilizing the evaporated acetone probe rinses in concentrated nitric acid and combined with the nitric acid probe rinses. In several cases (sites 1 and 2 inlet trains), however, the amount of particulate in the front half acetone and nitric acid probe rinses was very large and thus, this particulate was separated from the liquid portion of the rinse and analyzed separately. See notebook pages for exact details of this procedure. The combined acetone/nitric acid probe rinses were reduced in volume to less than 20 mL and combined with their respective filters in microwave bombs and digested. The digestion followed the one described in the draft protocol, with modifications as listed in Appendix A.

Aliquots of the back half train, permanganate impinger and front half digestates were prepared for Hg analysis by digestion with nitric and sulfuric acids and potassium permanganate.

III. Standard Preparation

Standards for all analyses were prepared at appropriate concentrations from Spex Industries or Solutions Plus Multielement Custom Plasma Standard Analytical Reference Materials. The standards were prepared in 10% (v/v) nitric acid or 0.05 M boric acid/10% nitric acid depending on the samples to be analyzed. The upper instrumental calibration limit was 10 ug/mL for Ag, Ba, Cd, Cr, Cu, Mn, Ni, Pb, and Se by ICP; 5 ug/mL for Zn by ICP; 20 ug/mL for As by ICP and 50 ug/L for Hg by CVAA. A calibration blank consisting of the stock acid or acid mixture was used.

Instrumental check standards for all analyses were prepared at a 5 ug/mL (As), 1 ug/mL (all but As or Hg) or 25 ug/L (Hg) level from custom prepared multielement standards from Solutions Plus or Spex Industries, Inc..

IV. Instrumental Analysis

The samples were analyzed for all analytes except Hg and As on the Jarrell-Ash Model Mark III inductively coupled argon plasma atomic emission spectrometer. Arsenic was analyzed on the Perkin-Elmer Plasma II inductively coupled argon plasma atomic emission spectrometer and Hg was analyzed on the GBC Model 902 atomic absorption spectrometer. The instrument parameters for each analysis can be found in the data packet. The instruments were calibrated according to the manufacturer's suggested technique. The analyses were performed according to CARB Method 436, whose analysis methods parallel EPA SW-846 Methods 6010 and 7470.

V. Sample Analysis Results and Discussion

The samples were analyzed according to CARB Method 436, with modifications as presented in Appendix A. The tables listed below contain the analytical data for this study.

Table	Description
1	Summary of Sample Results
2	Sample Quality Control
3	Instrumental Quality Control
4	ICP Raw Data 4-3-91
4a	ICP Calculated Data 4-3-91
5	ICP Raw Data 4-4-91
5a	ICP Calculated Data 4-4-91
6	ICP Raw Data 4-5-91
6a	ICP Calculated Data 4-5-91
7	ICP Raw Data 4-11-91
7a	ICP Calculated Data 4-11-91
8	ICP Raw Data 4-15-91
8a	ICP Calculated Data 4-15-91
9	Hg CVAA Analysis Raw and Calculated Data 11-6-90
10	Hg CVAA Analysis Raw and Calculated Data 11-7-90
11	Hg CVAA Analysis Raw and Calculated Data 11-14-90
12	Hg CVAA Analysis Raw and Calculated Data 11-15-90
13	Calculation of Front Half Values with Particulate
14	Summary of Impinger Content Bottle Weights

Table 1 contains the analytical results of the analyses. A summary of the sample quality control results is presented in Table 2 and the instrumental quality control data is contained in Table 3. Tables 4 through 14 contain the analytical raw and calculated data.

VI. Internal Quality Control

A. Instrumental Quality Control

The detection limit for the ICP analyses was determined from direct output of the Jarrell-Ash Model Mark III. This detection limit was determined from the calibration blank data generated throughout a sample analysis run and is defined as three times the largest standard deviation of the calibration blank analyses. The detection limit for the atomic absorption and ion chromatography analyses were determined by replicate determinations of a low standard and the detection limit is defined as three times the standard deviation of these analyses divided by the slope of the calibration curve.

Analytical quality check samples were prepared from Custom Multielement Plasma Standard Analytical Reference Materials manufactured by Solutions Plus or Spex Industries, Inc.. The average concentration (ug/mL or ug/L) found for these samples did not deviate from the stated value by more than 10 percent.

For ICP and CVAA, midpoint check standard was analyzed at the beginning, every 10 samples and the end of the analysis. The percent drift calculated from the instrumental check determination is appended in Table 3. Instrument drift was less than 6 percent for all ICP analyses, indicating that the instrument was stable throughout the entire sample analyses. The drift for the atomic absorption analyses was less than 10 percent for all runs.

B. Sample Quality Control

Duplicate determinations and/or serial dilutions were performed for all samples from one run for each site and the data from these analyses is presented in Table 2. Ninety-eight percent (126 of 129) of calculable percent ranges from analysis duplicates or duplicate preparations yielded percent ranges of less than 20 percent. Twenty-nine percent differences were not calculable as one or both of the sample results were less than the detection limit. Serial dilutions were also analyzed for samples which could not have duplicate samples prepared during sample preparation. Percent differences for the serial dilutions were less than 20 percent for 85 percent (95 of 112) of those which were calculable; 34 could not be calculated due to one or both results being less than the detection limit.

Spiked trains were prepared in-house which simulated the field trains. Three trains were prepared: a blank train, which contained reagents only; and two spiked at levels suitable for ICP and CVAA analyses. As with the field trains, these in-house trains were spilt into front and back halves. The front half of each in-house train consisted of approximately the same amounts of acetone and 0.1N nitric acid as were used in the probe rinses of the actual trains and a filter, while the back half of the in-house trains consisted of approximately the same amounts of nitric acid/hydrogen peroxide solution which was used for each field train plus sufficient ASTM Type I water to simulate the maximum condensate collected. The same lots of reagents which comprised the field trains may not necessarily have been used for the in-house trains. The trains were spiked with certified stock standards just prior to the cookdown step of the preparations. Of the resulting 134 spiked determinations, 66 percent (88 of 134) returned values between 80 and 120 percent. Many front half spike recoveries, especially for sites 2, 3 and 4, occurred in the 70-80 percent range. Duplicate percent ranges were less than 20 percent for 66 of 70, or 94 percent of the data. Three of the four percent ranges of more than 20 percent were for silver and silver had poor recoveries for many of the quality control trains, as may be expected due to precipitation as very insoluble silver chloride.

This project was conducted in two parts: one section of the work occurred from approximately late October, 1990 to early December, 1990 and the second from early to mid April, 1991. In the first phase of the work, the samples from site 1 were all prepared for all analyses and analyzed for Hg. The impinger contents from sites 2, 3 and 4 were analyzed for Hg and the impinger contents from site 2 prepared for analysis of other metals. The remaining samples (front half samples from sites 2, 3 and 4) were partly prepared whent the project was stopped in early December. Approximately two-thirds of the front half samples had not been analzyed for Hg at that time. The project was not restarted until early April, 1991, much after the Hg holding times for these samples had passed and thus the samples were not analyzed for Hg. The sample preparation for the other analysis was completed for sites 2, 3 and 4 and the samples analyzed by ICP for all analytes except Hg. Although the quality control of the analyses of this project were very good, it appears that the long delay in completing the sample preparation and analysis may have had a negative impact upon the sample quality control, as evidenced by the unusually low recoveries and poorer duplication of the in-house prepared spiked train samples.

VII. Additional Information

The following raw data accompanies this report. This information is coded by Project 9420-0255 and also contains a part number identifier.

Part Number	Description
9420-0255 1	Photocopies of MRI Laboratory Generated Sample Inventory Sheets
9420-0255 2	Photocopies of Notebook 2097:22-55, 2374:1-9
9420-0255 3	Sample Weighing Sheet - Hg Reserve Aliquots 10-30-90
9420-0255 4	Sample Weighing Sheet - Site 1 Particulates 11-2-90
9420-0255 5	Sample Weighing Sheet - Site 1 Hg Samples 11-5,6-90
9420-0255 6	Sample Weighing Sheet - Hg Reserve Aliquots 11-7-90
9420-0255 7	Sample Weighing Sheet - Site 2 Hg Samples 11-13-90
9420-0255 8	Sample Weighing Sheet - Hg Reserve Aliquots 11-13-90
9420-0255 9	Sample Weighing Sheet - Sites 3 & 4 Hg Samples 11-14-90
9420-0255 10	Sample Weighing Sheet - Site 2 Particulates 11-14-90
9420-0255 11	Atomic Absorption Data Reporting Sheet - 11-6-90
9420-0255 12	CVAA Sample Raw Data - 11-6-90
9420-0255 13	Atomic Absorption Data Reporting Sheet - 11-7-90
9420-0255 14	CVAA Sample Raw Data - 11-7-90
9420-0255 15	Atomic Absorption Data Reporting Sheet - 11-14-90
9420-0255 16	CVAA Sample Raw Data - 11-14-90
9420-0255 17	Atomic Absorption Data Reporting Sheet - 11-15-90
9420-0255 18	CVAA Sample Raw Data - 11-15-90
9420-0255 19	ICP-AES Data Reporting Sheet - 4-3-91
9420-0255 20	Control Table Editor Output - 4-3-91
9420-0255 21	ICP Sample Raw Data - 4-3-91
9420-0255 22	ICP-AES Data Reporting Sheet - 4-4-91
9420-0255 23	Control Table Editor Output - 4-4-91
9420-0255 24	ICP Sample Raw Data - 4-4-91
9420-0255 25	ICP-AES Data Reporting Sheet - 4-5-91
9420-0255 26	Control Table Editor Output - 4-5-91
9420-0255 27	ICP Sample Raw Data - 4-5-91
9420-0255 28	ICP Instrument Parameters and Sample Raw Data 4-11-91
9420-0255 29	ICP Instrument Parameters and Sample Raw Data 4-15-91
9420-0255 30	Photocopies of Field Weighing Sheets

With the exception of parts 9420-0255 1, 2 and 30, the photocopies of the MRI Laboratory Generated Sample Inventory Sheets, notebook 2097:22-55 and 2374:1-9, and field weighing sheets, this file contains the only record of the analyses. This file should therefore be archived as required by the project or as required by MRI policy.

This data has undergone one level of senior review within the Analytical Chemistry Section. In addition, the data has undergone review within the Atomic Spectrometry Facility. The project quality assurance coordinator has yet to reviewed this data.

Approved,

John Stanley, Head
Analytical Chemistry Section

Appendix A

Modifications to CARB Method 436
Atomic Spectrometry Facility - Midwest Research Institute

Paragraph 3.3.4 Beakers and Watchglasses. 250 mL or other appropriate size beakers for sample digestion and volume reduction with watchglasses to cover the tops.

Paragraphs 4.1.2 and 4.3.2 To conform to ASTM Specification D1199.77, Type I (incorporated by reference). Type I water is defined to have a resistance of 17 megohm/cm, which is a measure of the total metals content of the water. Type I water should easily contain less than 1 ng/mL of the target analytes by its nature without requiring further analysis.

Paragraph 4.2 Pretest Preparation for Sampling Reagents. (Lot analyses listed on the label for each reagent can be used to determine if reagents meet the criteria for purity listed for each solution.)

Paragraphs 4.4.15 Multielement Calibration Standards. Procure mixed multielement standards for preparing calibration standards for instrumental analysis. All target analytes should be included in stable mixes plus aluminum and iron. These mixed standards are used as the preparation of mixed standards from individual standards is not cost effective. The concentration of most elements contained in these custom mixes is 100 ug/mL, while the level of elements such as aluminum, iron, sodium, calcium and potassium in another mix is 1000 ug/mL. Quality control standards should be prepared from alternate lots of mixed standards.

Paragraph 4.4.16 Boric Acid, Powder. Baker Analyzed or equivalent.

Paragraph 4.4.17 Boric Acid Solution, 0.5 M. Dissolve 3.019 g boric acid to 100 mL with Type I water. This solution may require some time to dissolve as the solution is nearly saturated.

Paragraphs 4.4.18-4.4.33 These paragraphs are deleted.

Paragraph 4.4.34 Mercury Standards and Quality Control Samples. Prepare fresh daily a 1 ug/mL intermediate mercury standard by diluting 0.25 mL of the 100 ug/mL standard (paragraph 4.4.15) containing mercury to 25 mL with 10% (v/v) nitric acid. The following amounts of this standard and Type I water should be pipetted into 250-mL glass stoppered Erlenmeyer flasks for the calibration curve:

Standard Conc. (ug/L)	0	0.5	1	2.5	5	10	25	50
mL 1 ug/mL Standard	0	0.05	0.1	0.25	0.5	1	2.5	5
mL Type I water	20	20	20	20	19.5	16	17.5	15

A standard for preparing the quality control sample should be prepared by diluting 1 mL of the alternate 100 ug/mL standard to 10 mL with 10% (v/v) nitric acid. The quality control sample should be prepared by pipetting 0.25 mL of this standard and 20 mL of Type I water into a 250-mL glass stoppered Erlenmeyer flask. All standards (calibration and quality control) should be prepared with the samples.

Paragraph 4.4.35 ICAP Standards and Quality Control Samples. Calibration and continuing calibration check standards for ICAP analysis should be prepared at appropriate levels by dilution of the 100 and 1000 ug/mL standards (paragraph 4.4.15) with 10% (v/v) nitric acid. An upper calibration limit of 10 ug/mL in 10% (v/v) nitric acid (or other appropriate matrix) is used for most elements, however, the upper calibration limit must be within the linear range of the instrument possibly necessitating lower upper calibration limits. An upper calibration limit of 150 ug/mL may be used for potentially interfering elements such as aluminum, iron, sodium and potassium, if that standard is contained within the linear range for the elemental line used. A minimum of one standard and a blank can be used to form each calibration curve. A separate quality control sample spiked with known amounts of the target metals at a 1 ug/mL (or levels greater than 10 times the instrumental detection limit) should be prepared in 10% (v/v) nitric acid (or other appropriate matrix). An interference check standard should also be prepared in 10% (v/v) nitric acid (or other appropriate matrix) and the interferent levels should reflect those in the samples themselves. The analyte level for the interferent check standard should be 1 ug/mL. All standards should be prepared fresh daily and the standard matrix should match that of the samples.

Paragraph 5.3.1 Container No. 2 (Acetone Rinse). Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the smaple or use methods, subject to the approval of the Administrator, to correct the final results. Transfer the contents to an acid-cleaned 250-mL beaker and evaporate to dryness at ambient temperatures and pressures. When the sample is dry, add 10 mL of concentrated acid, cover the beaker with a watch glass and reflux for 15 minutes on hot plate. Rinse this sample into the beaker containing the sample from Container No. 3 with minimal portions of Type I water.

Paragraph 5.3.2 Container No. 3 (Probe Rinse). The pH of this sample shall be 2 or lower. The sample should be rinsed into an appropriate sized beaker with Type I water, add 10 mL of concentrated nitric acid, and the sample volume should be reduced by heating on a hot plate at a temperature just below boiling. Add the sample resulting from Container No. 2 and its rinses and continue sample volume reduction until the sample is approximately 5-10 mL. Cover this sample with a watchglass between periods of heating. Rinse this combined sample into the microwave bomb containing the filter (from Container No. 1) with minimal portions of Type I water.

Paragraph 5.3.3 Container No. 1 (Filter). Place the entire filter into individual microwave pressure relief vessels. Rinse the container with minimal portions of 10% (v/v) nitric acid if particulate is observed remaining in the container. Add the resulting combined acetone rinse/probe rinse sample and its rinses to the container. Add 5 mL of concentrated nitric acid and 4 mL of concentrated hydorfluoric acid to each vessel. Microwave the samples for approximately 12-15 minutes in intervals of 1-2 minutes at 600 watts. Then cool the samples to room temperature, add 5 mL of a 0.5 M and dilute to a known volume (usually 50 mL) with Type I water. If the sample contains particulate matter, allow the sample to settle prior to analysis. Use a portion of this digestate for the mercury determination (if mercury is a target analyte).

<u>Notes:</u> 1. Suggested micrwave heating times are approximate and are dependent upon the number of samples (and their volumes) being digested. Twelve to 15 minute heating times have been found to be acceptable for simultaneous digestion of up to 12 individual samples. Sufficient heating is evidenced by sorbent reflux within the vessel.

2. This combining procedure has been used on other trial burns with no detrimental effects upon the sample preparation or analysis and has the advantage of better detection limits.

3. If the sampling train uses an optional cyclone, the cylcone catch should be assessed for homogeneity. The sample should be homogenized (i.e., mixing with stirring rods to reduce sample chunks), if necessary and if possible. A portion of the homogenous sample will be separately digested in a microwave bomb and analyzed separately in order to minimize the volume of sample resulting for the front half (Containers 1-3) of the train.

4. Boric acid (0.05M) is added to the samples as a safety procaution due to the hazards of exposure to hydrofluoric acid. Boric acid complexes the fluoride and reduces the hazard of handling these digests.

Paragraph 5.3.4 Container No. 4 (Impingers 1-3). Remove a 50 mL aliquot for mercury analysis if mercury is a target analyte; this aliquot should be prepared and analyzed as described in Section 5.4.3. The remaining sample should be rinsed into an appropriate sized beaker with Type I water and add 10 mL of concentrated nitric acid. The sample volume should be reduced to approximately 5-10 mL by heating on a hot plate at a temperature just below boiling. Cover this sample with a watchglass between periods of heating. Cover the sample with a watchglass, add 10 mL of 50 percent nitric acid and heat for 30 minutes on a hot plate to just below boiling. Add 9 mL of Type I water and 1 mL of 30% hydrogen peroxide and heat for 10 more minutes. Add 10 mL of Type I water and heat the sample for an additional 20 minutes. Cool and dilute to 50 mL with Type I water. If the sample contains particulate matter, allow the sample to settle prior to analysis.

Paragraph 5.4.1 ICAP Analysis. The digestates from Containers 1-3 and Container 4 are analzyed by ICAP using EPA Method 6010. Calibrate the ICAP and set up an analysis program as described in Method 6010. The quality control procedure described in Section 7.3.1 of this method shall be followed. Wavelengths used in the analysis are those contained on the simultaneous unit (Jarrell-Ash Model 1155A); other elements and wavelengths should follow the recommendations in Method 6010.

Prescreen all samples for potential interfering elements such as aluminum and iron. Create an interference check standard as defined in Section 4.4.35. Analyze all samples for the target metals.

Note: When analyzing samples with a hydrofluoric acid/boric acid matrix, the sample introduction system should be rinsed and cleaned with non-ionic detergent as soon as possible after the completion of analysis to minimize damage to the inlet system.

Paragraph 5.4.3 Cold Vapor AAS Mercury Analysis. Portions of the front half digestate, the reserved aliquot of the back half (Container 4) and permanganate impinger (Container 5) should be analyzed for mercury using cold vapor atomic absoprtion spectrometry following the method outlined in EPA Method 7470. Set up the calibration curve as descirbed in Section 4.4.34. These standards must be prepared along with the samples. Add approximately 5-20 mL of each sample to a 250-mL glass stopppered Erlenmeyer flask. Add sufficient Type I water to each sample so that the total volume is 20 mL. Add 5 mL of concentrated sulfuric acid and 2.5 mL of concentrated nitric acid to each sample, mixing after each addition. Add 10 mL of 5 percent potassium permanganate and mix. Add additional potions of 5 percent potassium permanganate, if necessary, unitl the purple color remains in all samples for at least 15 minutes. Add 4 mL of 5 percent potassium persulfate to each sample and heat for 2 hours in a water bath or convection oven maintained at 95 degrees C. Cool and add 3 mL of hydroxylamine hydrochloride solution and mix the sample. Transfer to labelled 100 mL volumetric flasks and dilute to the mark with Type I water. Pipet an appropriate amount of sample, standard or blank into the reaction cell. Attach the cell to the system. Add a predetermined amount of stannous sulfate/chloride solution to the sample and start the recirculating pump. When the absorbance reading levels off (approximately 30-45 seconds), record the value. Then open the valves and vent the system until it returns to a minimum value. Remove the reaction cell, rinse the system where the reaction cell attaches and close the valves. The system is ready for the next sample analysis.

Paragraph 6.3.2 Atomic Absorption Spectrometer - Cold Vapor Analyses. Prepare the standards as outlined in Section 4.4. Calibrate the spectrometer suing these spreapred standards. Calibration procedures are also outlined in the EPA Method 7470. Instrumental drift should be determined after every 10 samples by analyzing a midrange standard. The drift must be less than 20 percent from initial or the instrument must be recalibrated and all samples analyzed since the last acceptable drift check standard reanalyzed.

Paragraph 7.3.1 ICAP Analysis. Follow the quality control shown in Section 8 of Method 6010. For the purposes of a three run test series, these requirements have been modified to include the following: two instrument check standard runs; two calibration blank runs; two interference check samples, one at the beginning and one at the end of the analysis (must be within 25% of the true value or analyze by standard addition); one quality control sample to check the accuracy of the calibration standars (must be within 10% of calibration), and one duplicate analysis (must be within 20 percent of average for values greater than 10 times the detection limit or repeat all analyses).

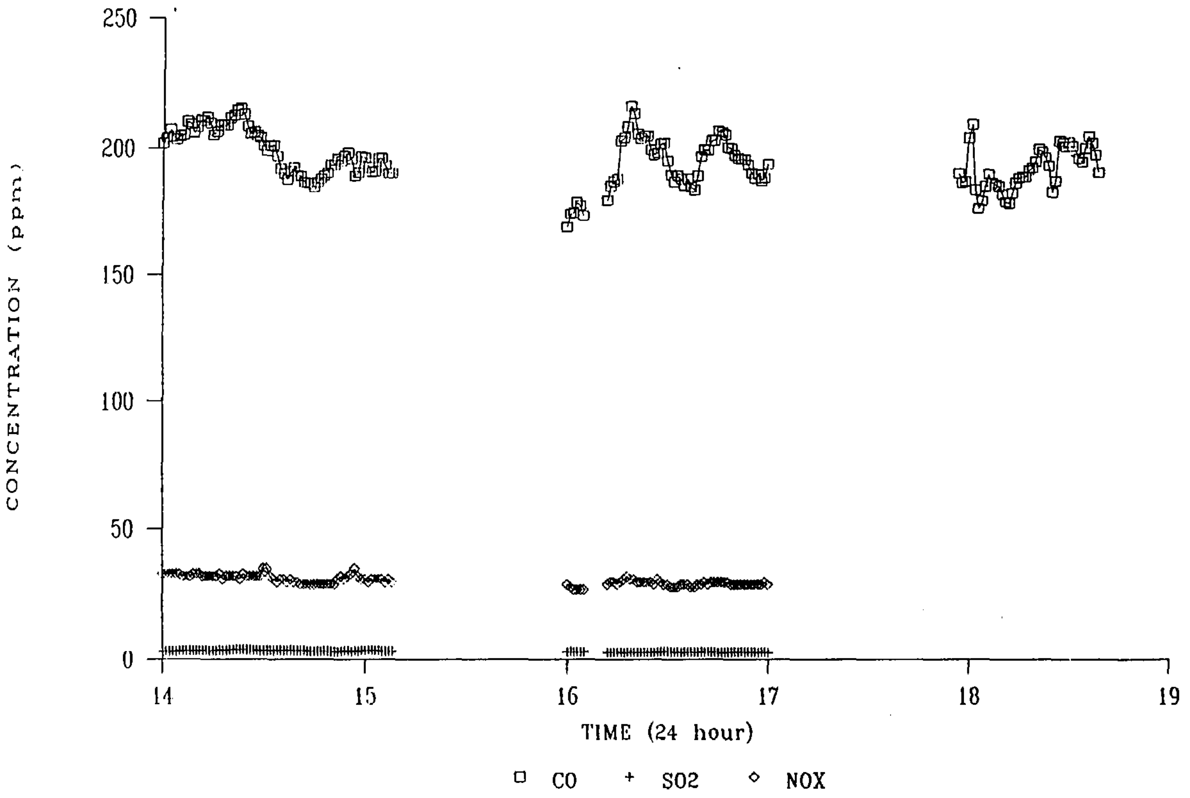
Paragraph 7.3.3 Cold vapor AAS Analysis for Mercury. A quality control sample should be analyezd to check the accuracy of the calibration standards (within 10% or repeat calibration). Perform a matrix spike on one sample from the nitric impinger portion (must be within 25% or samples must be analyzed by the method of standard additions). A serial dilution should also be performed on one sample to assure freedom from other interferences. Additional information on quality control can be obtained from EPA Method 7470.

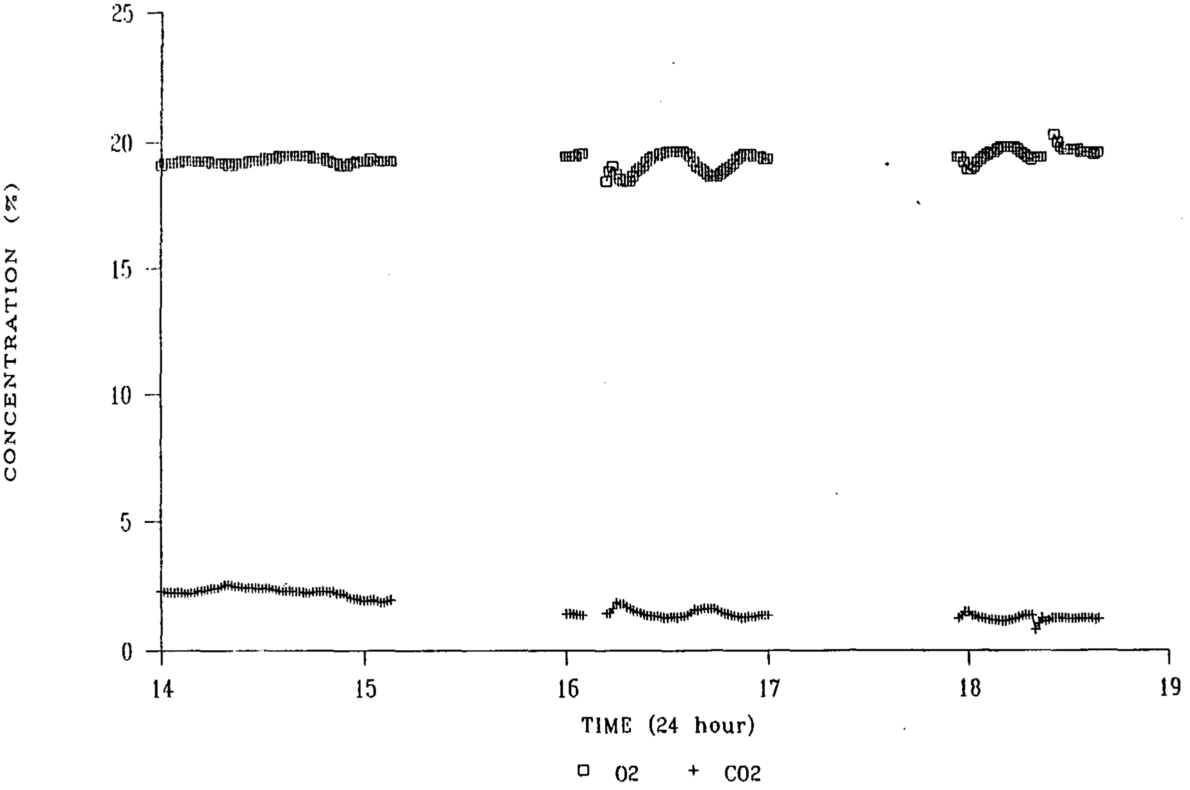
APPENDIX B

CONTINUOUS EMISSION MONITORING DATA (UNCORRECTED)

SITE A

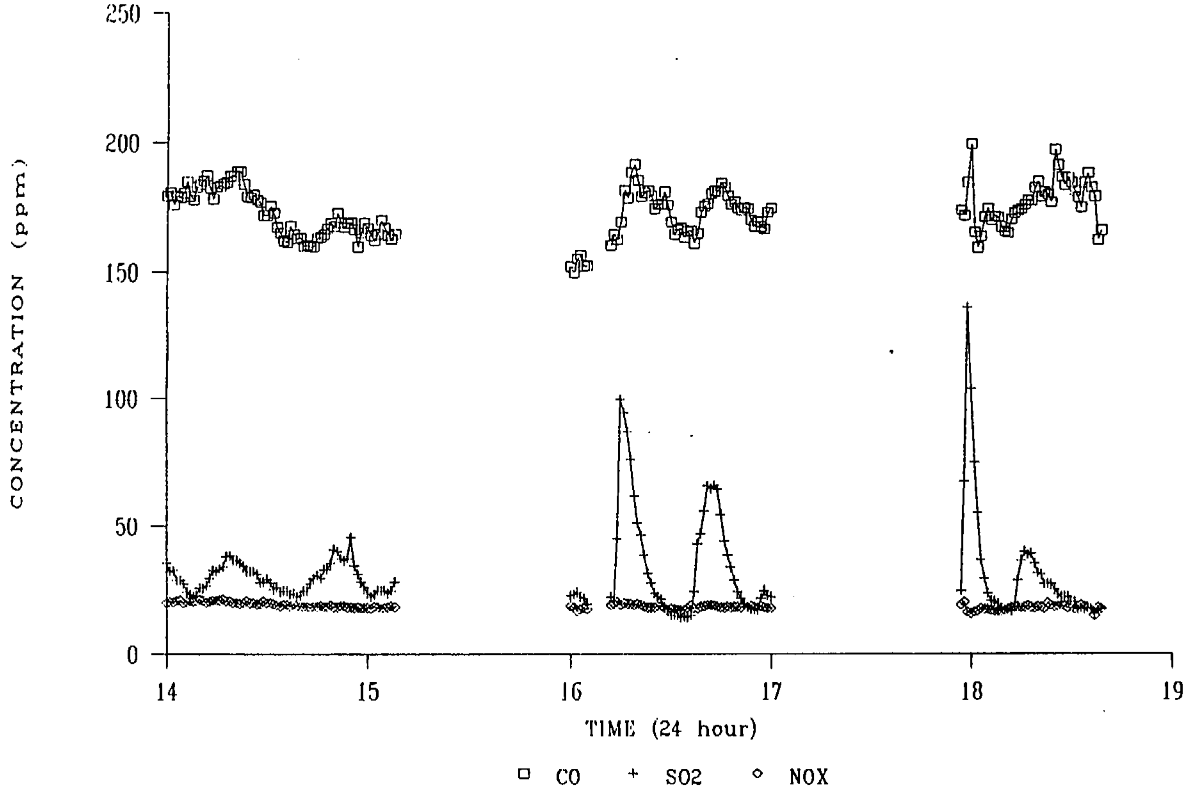
RUN 1-INLET

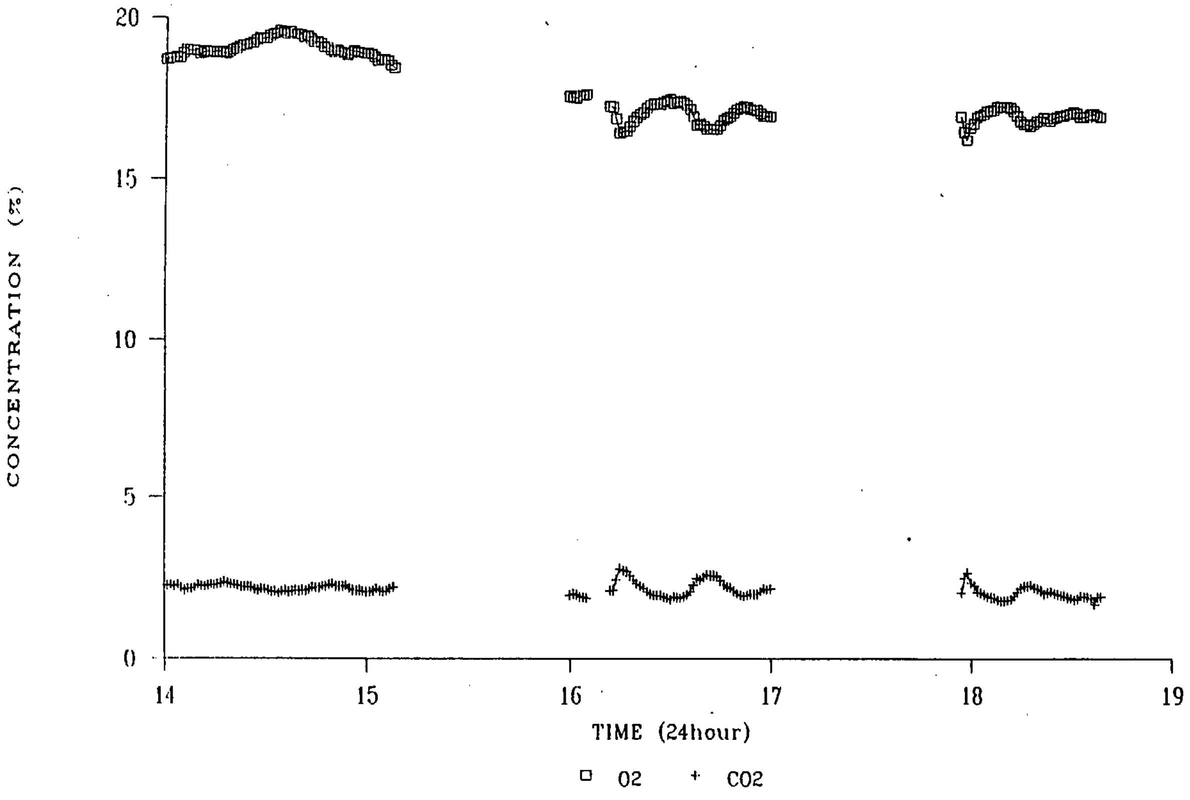




SITE A

RUN 1-OUTLET





SITE A

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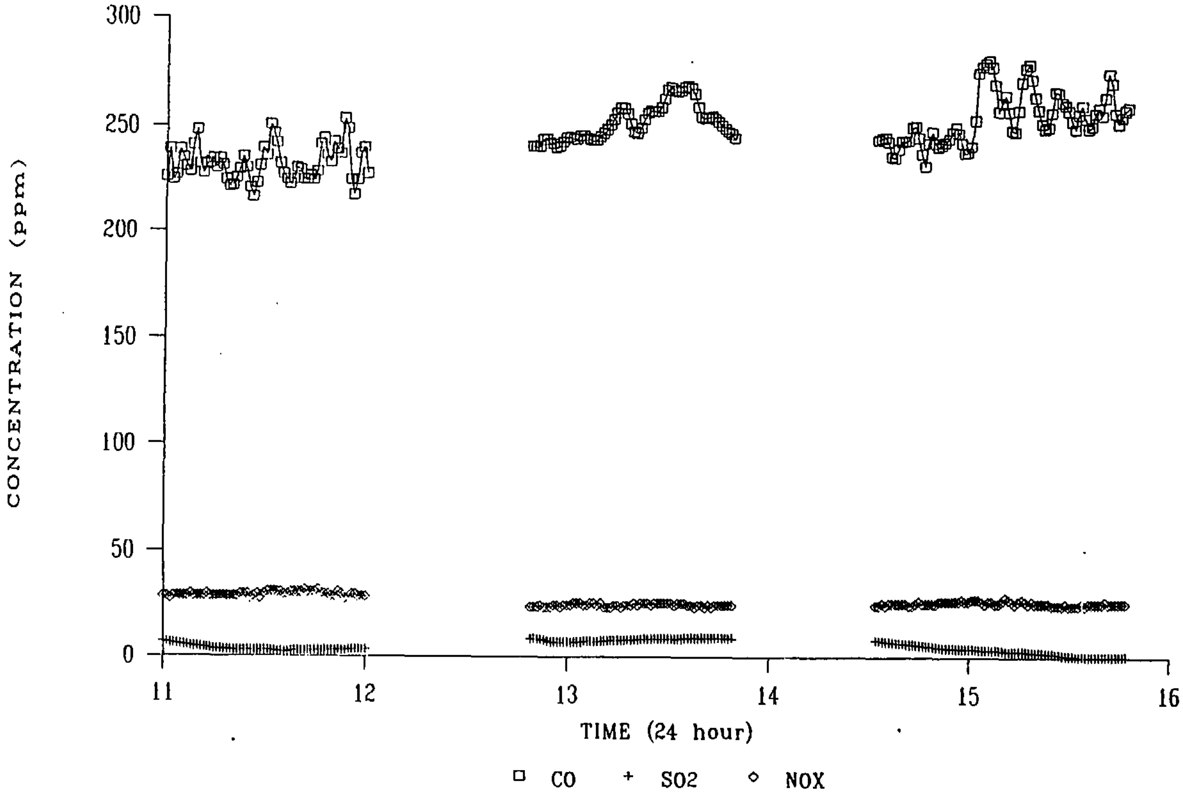

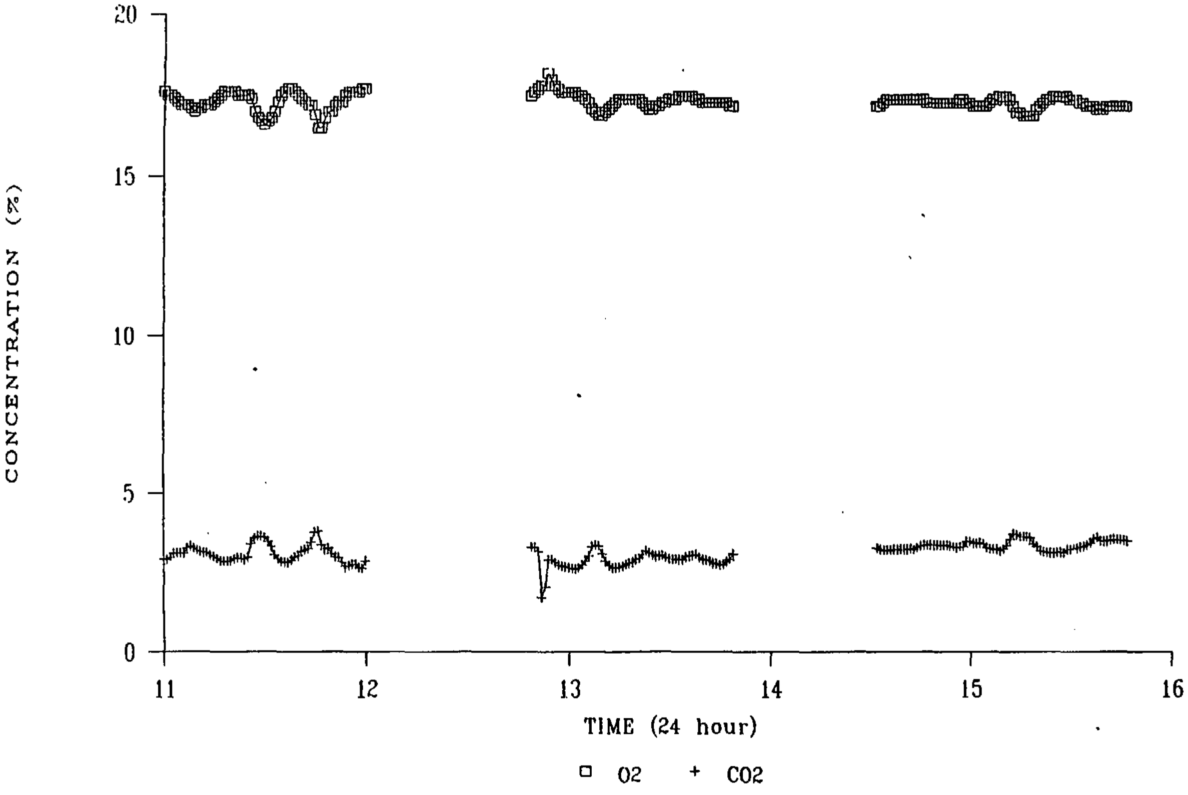

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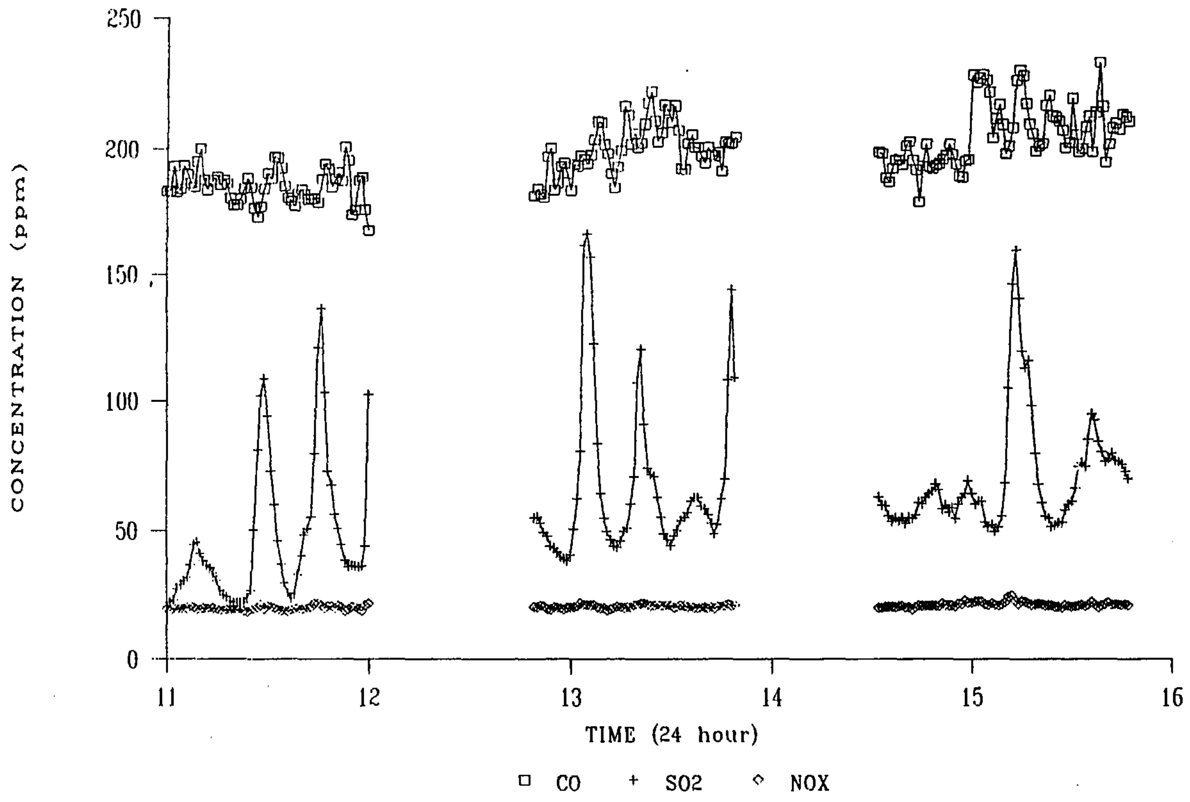
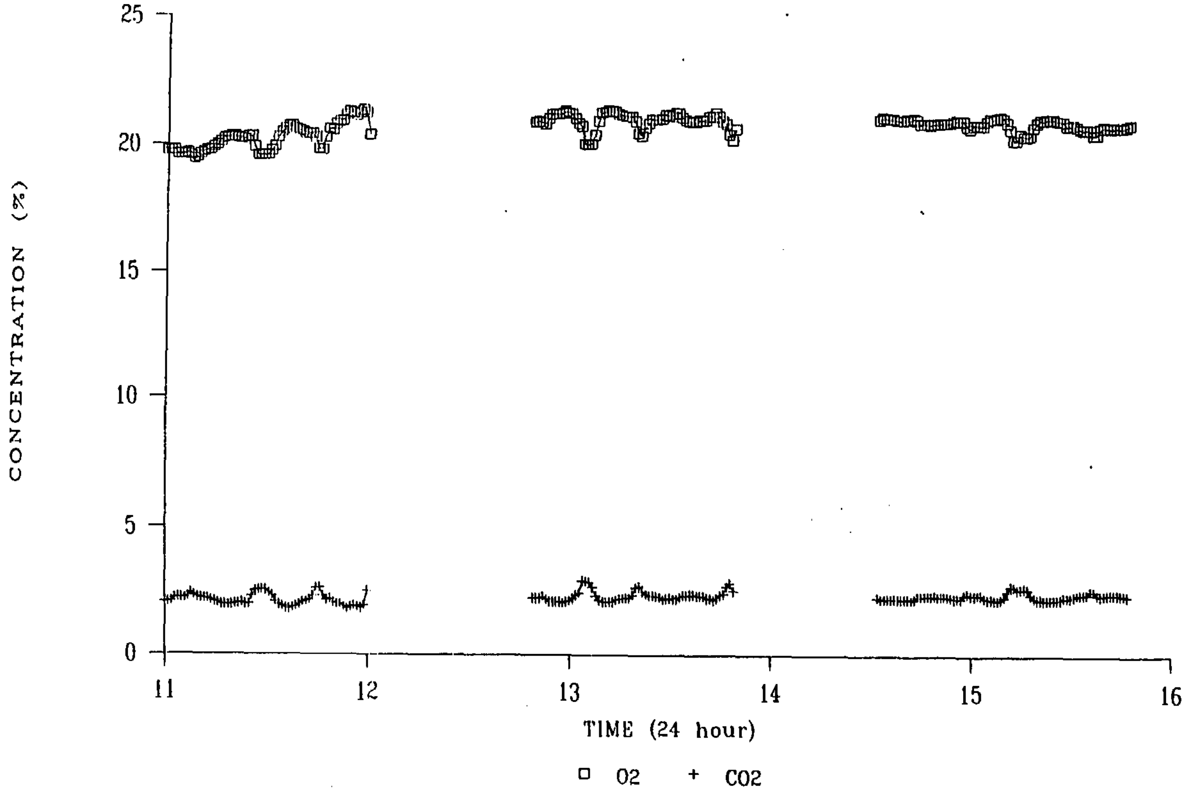
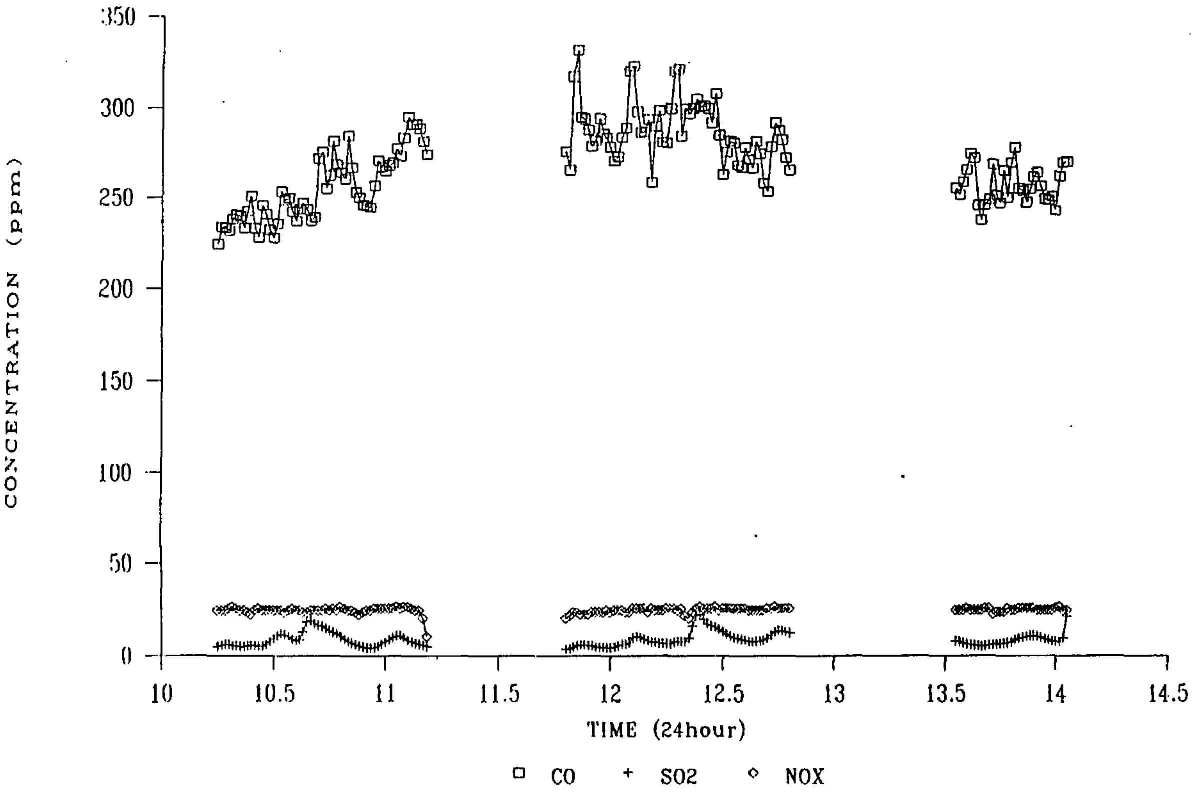
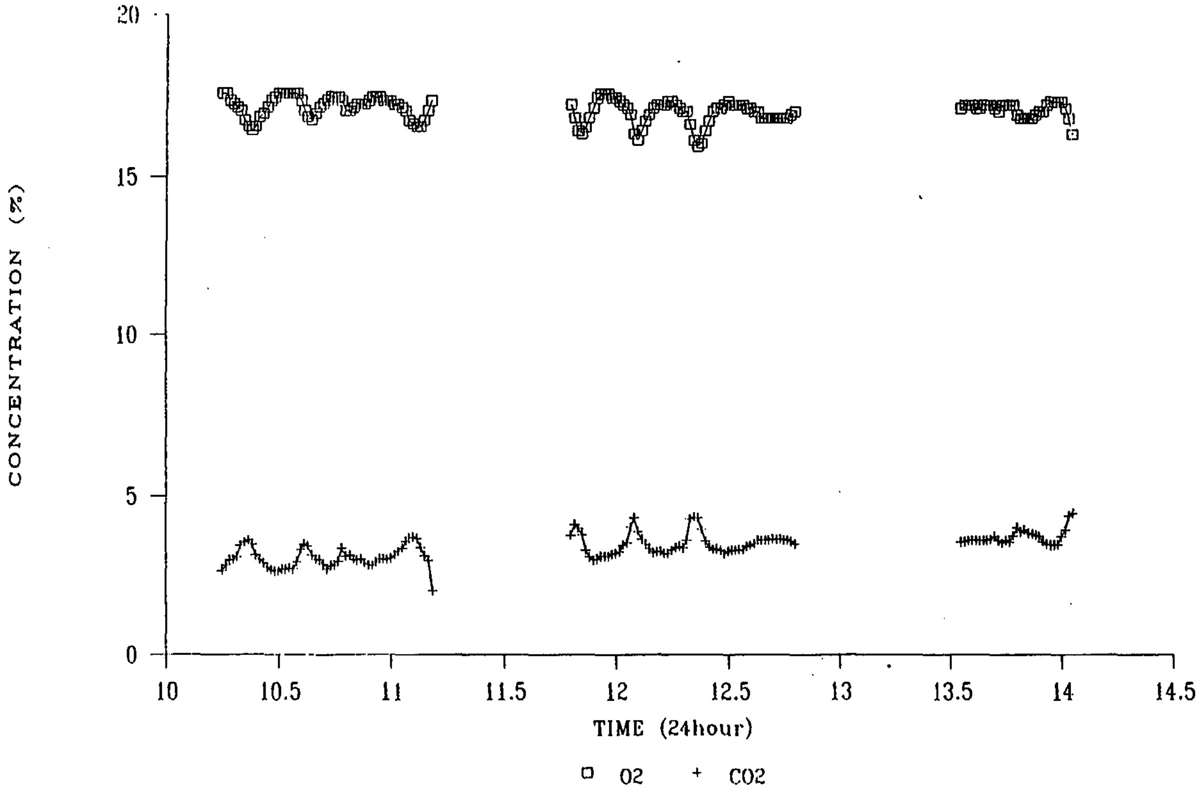




SITE A

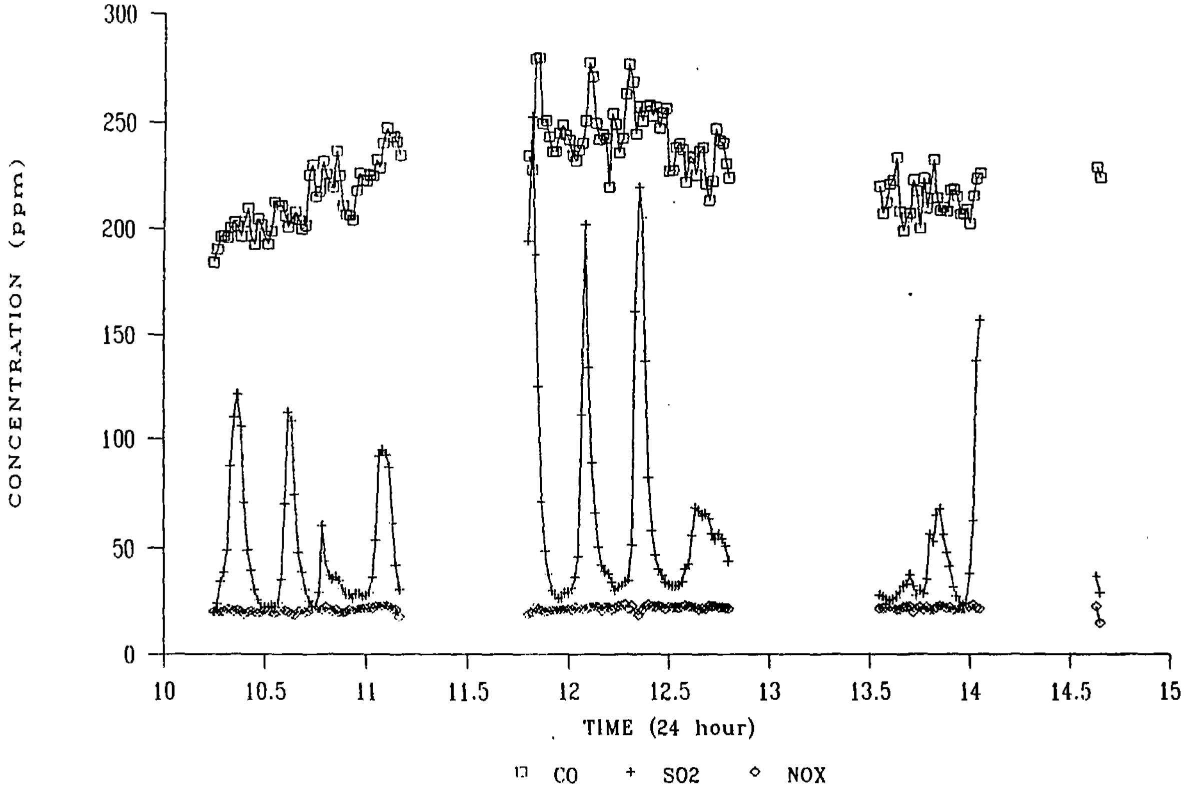
RUN 3-INLET

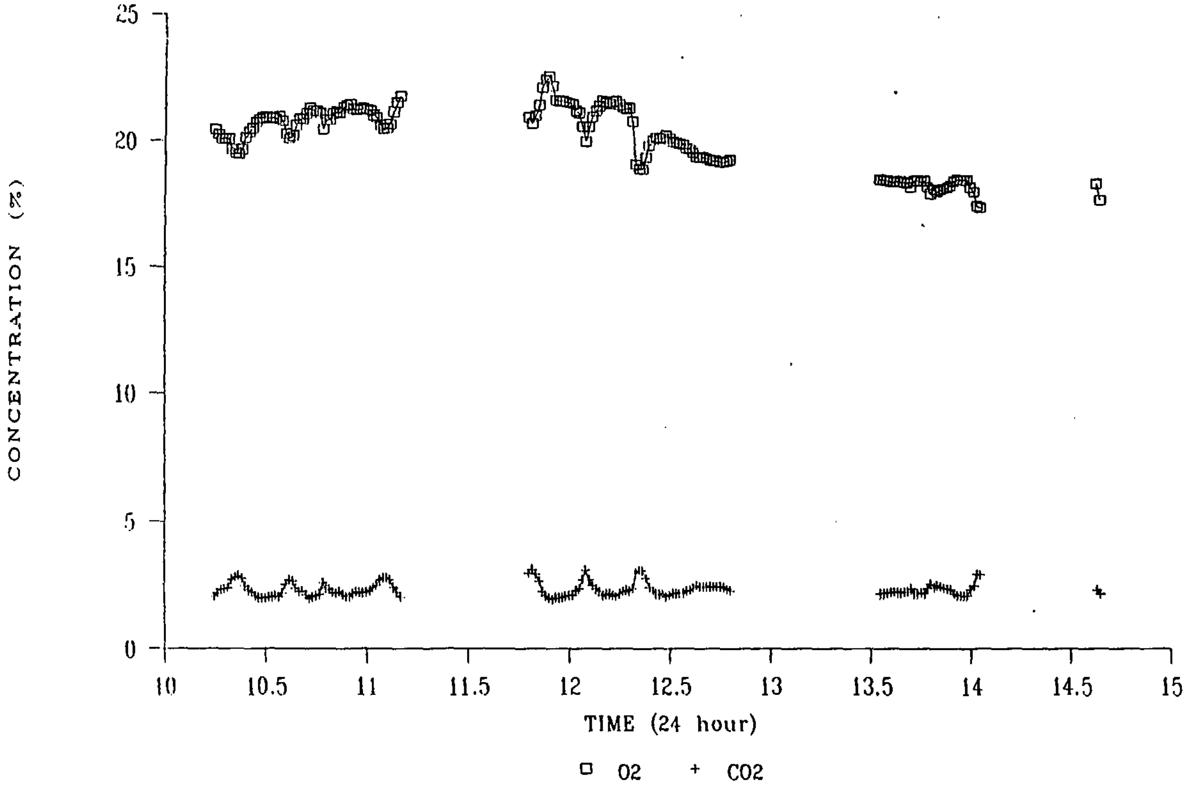




SITE A

RUN 3-OUTLET



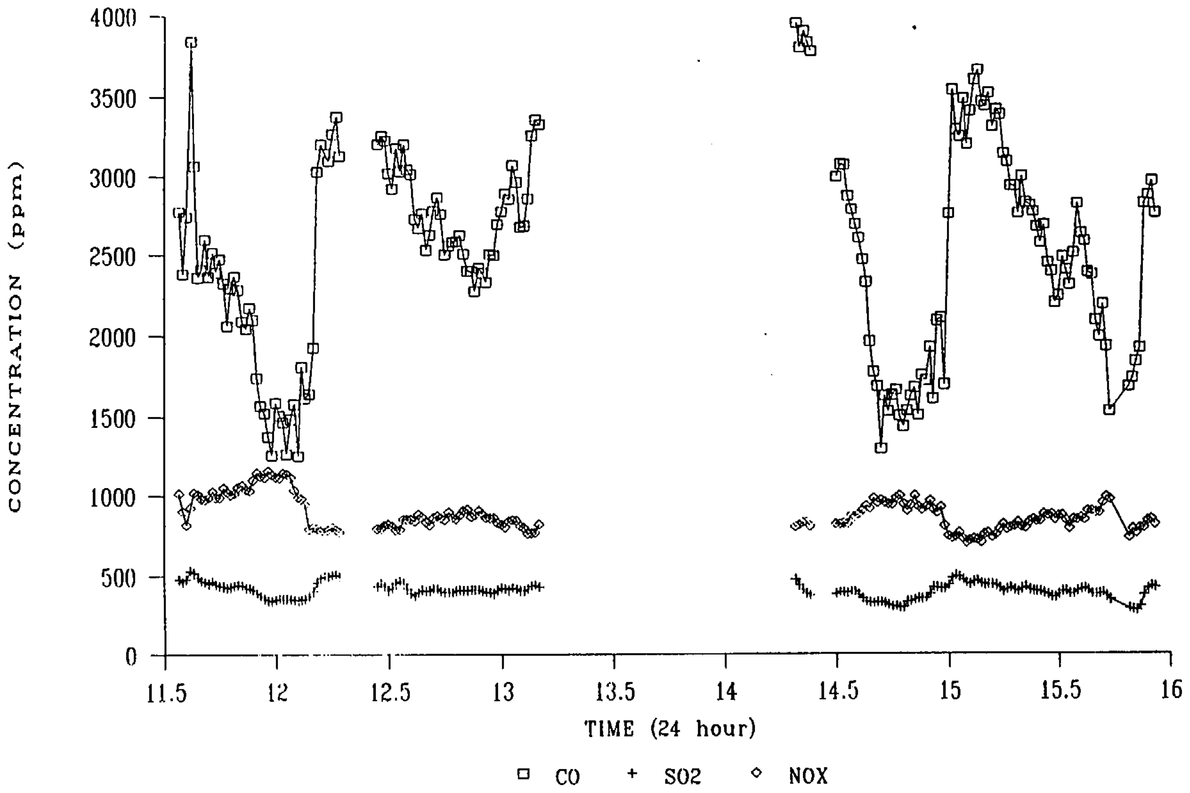


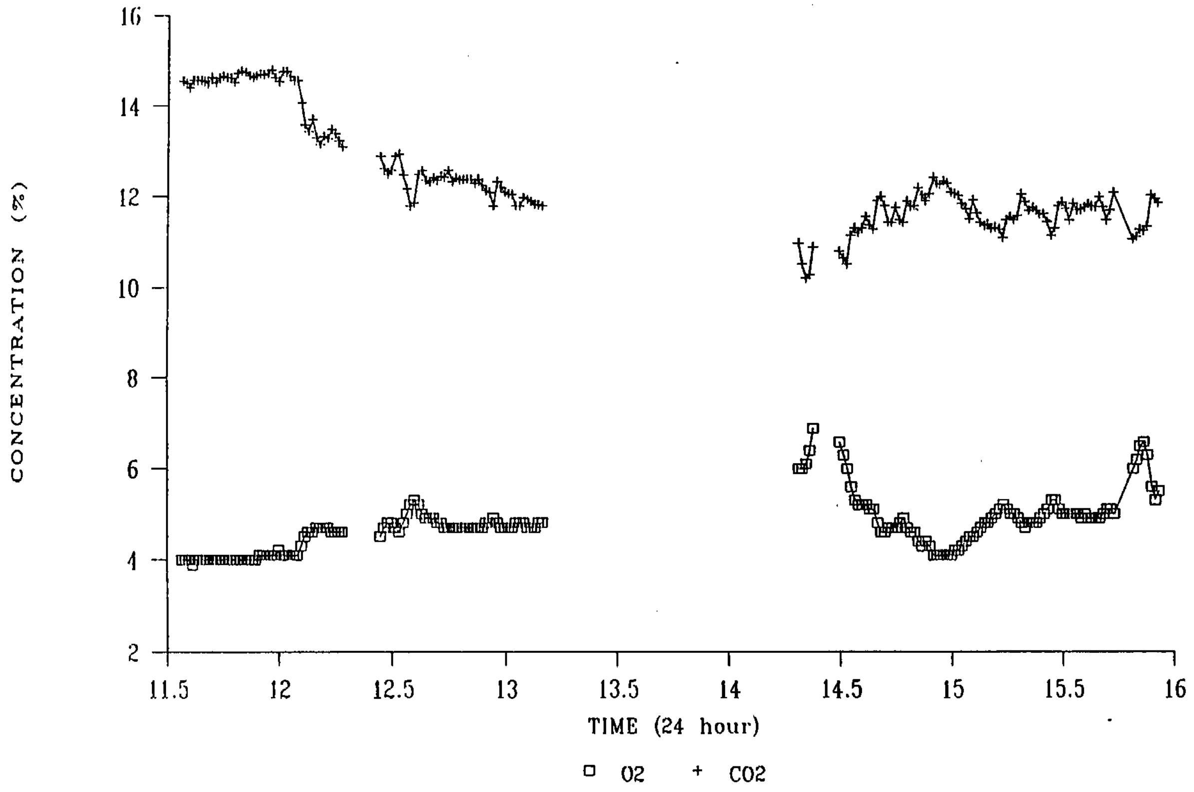
SITE A SUMMARY OF CEM OPERATING PARAMETERS

	ZERO DRIFT (% of span)	SPAN DRIFT (% of span)	LINE BIAS (% of span)	ANALYZER LINEARITY DEVIATION (% of span)
RUN 1 INLET				
O2	1.04	2.03	1.4	1.1
CO2	1.20	1.02	4.3	0.25
CO	0.98	0.46	0.13	0.64
SO2	0.13	2.47	0.52	1.3
NOx	0.80	0.81	2.4	N/A
RUN 1 OUTLET				
O2	0.30	0.06	1.77	0.25
CO2	0.21	0.09	0.5	0.33
CO	2.03	1.55	1.45	1.58
SO2	1.57	2.36	0.92	0.15
NOx	0.04	3.61	0.3	N/A
RUN 2 INLET				
O2	2.51	2.96		
CO2	2.17	3.01		
CO	0.74	1.23		
SO2	0.14	9.59		
NOx	0.98	1.07		
RUN 2 OUTLET				
O2	0.68	0.29		
CO2	0.39	0.32		
CO	1.32	0.90		
SO2	0.39	0.58		
NOx	0.96	1.89		
RUN 3 INLET				
O2	2.47	1.86		
CO2	0.73	2.00		
CO	3.38	2.25		
SO2	1.89	2.72		
NOx	0.13	1.58		
RUN 3 OUTLET				
O2	2.4	1.97		
CO2	0.12	0.15		
CO	2.04	0.42		
SO2	0.19	0.77		
NOx	0.48	0.71		

SITE B

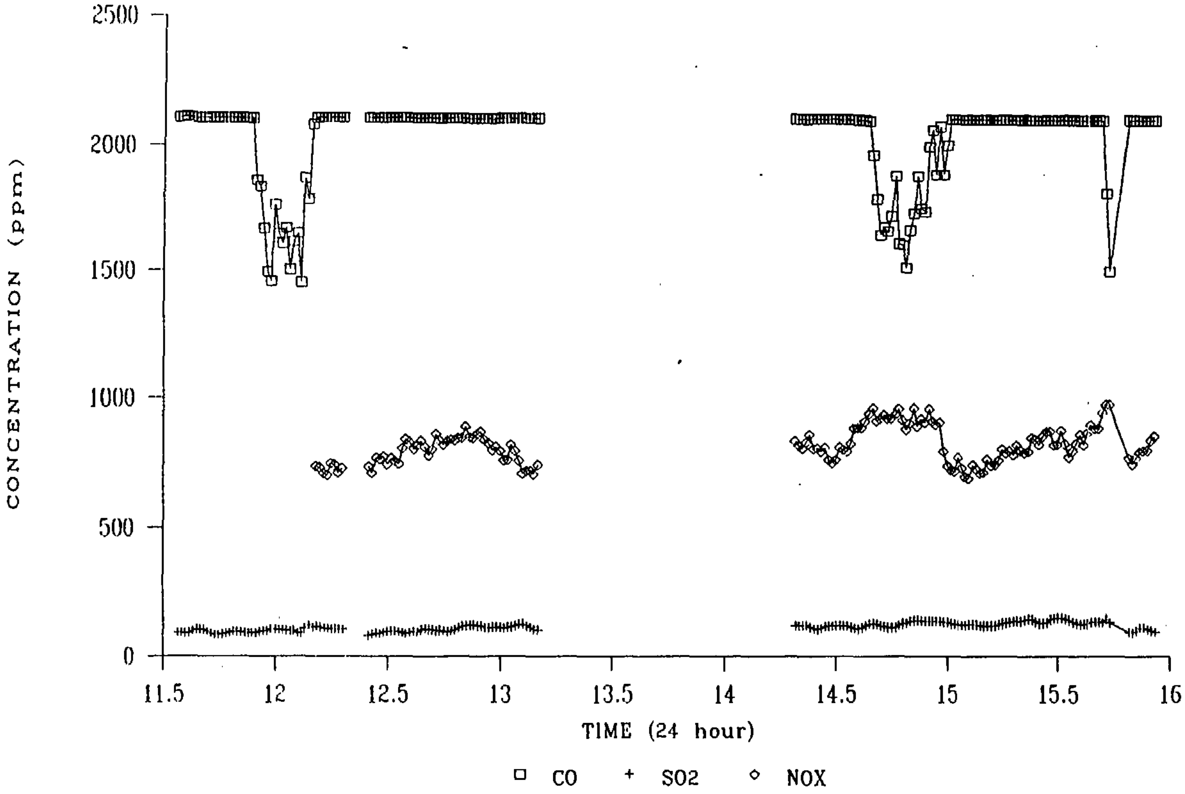
RUN 4-INLET

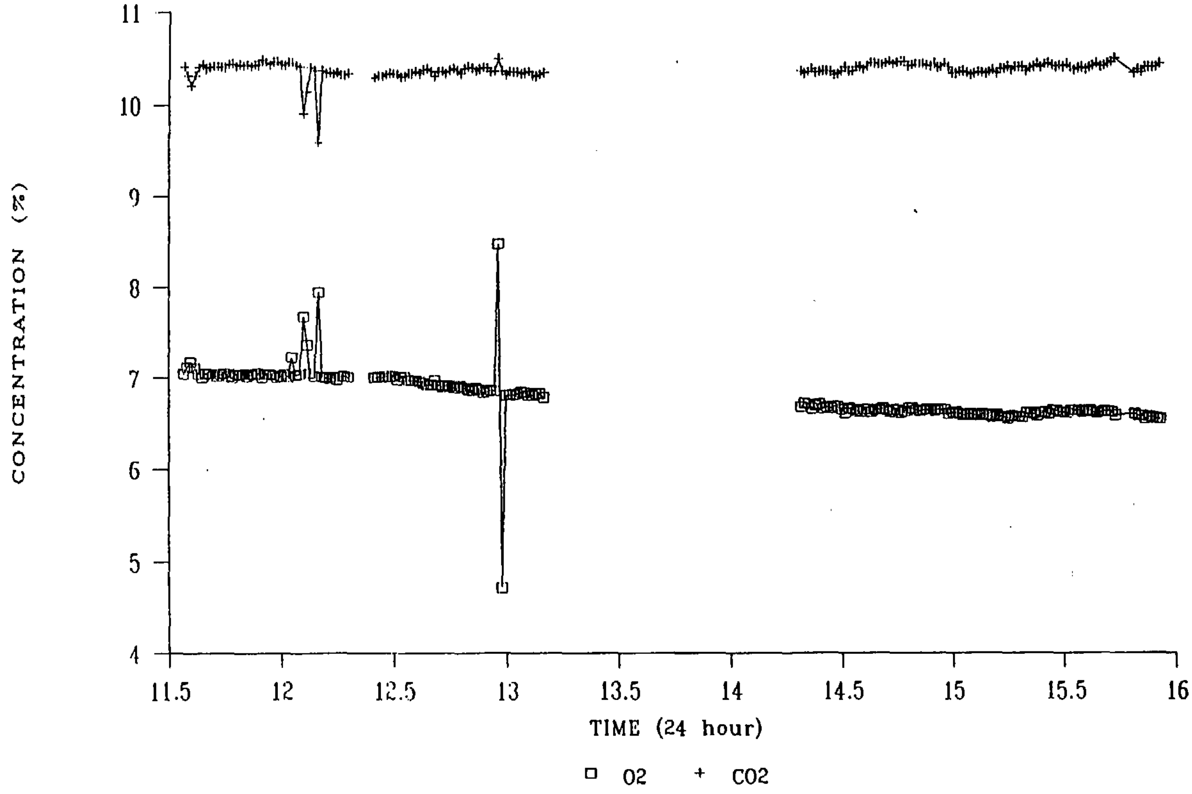




SITE B

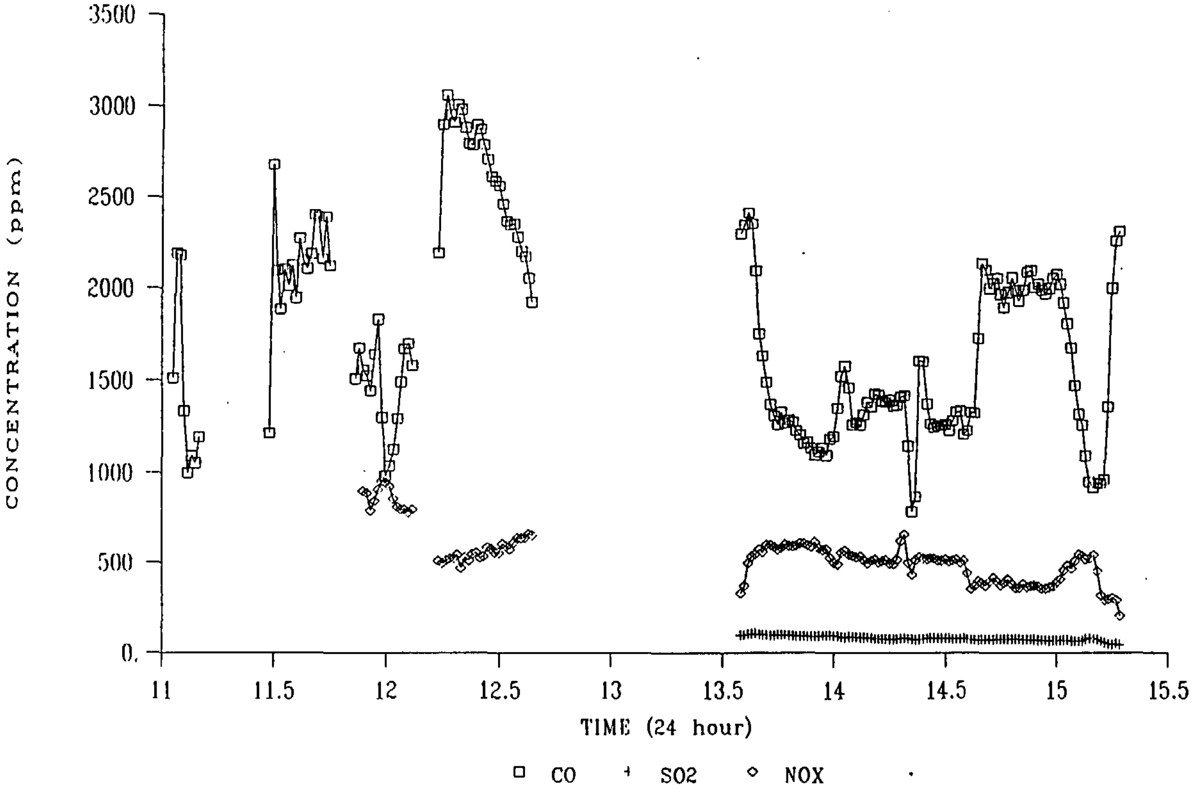
RUN 4-OUTLET

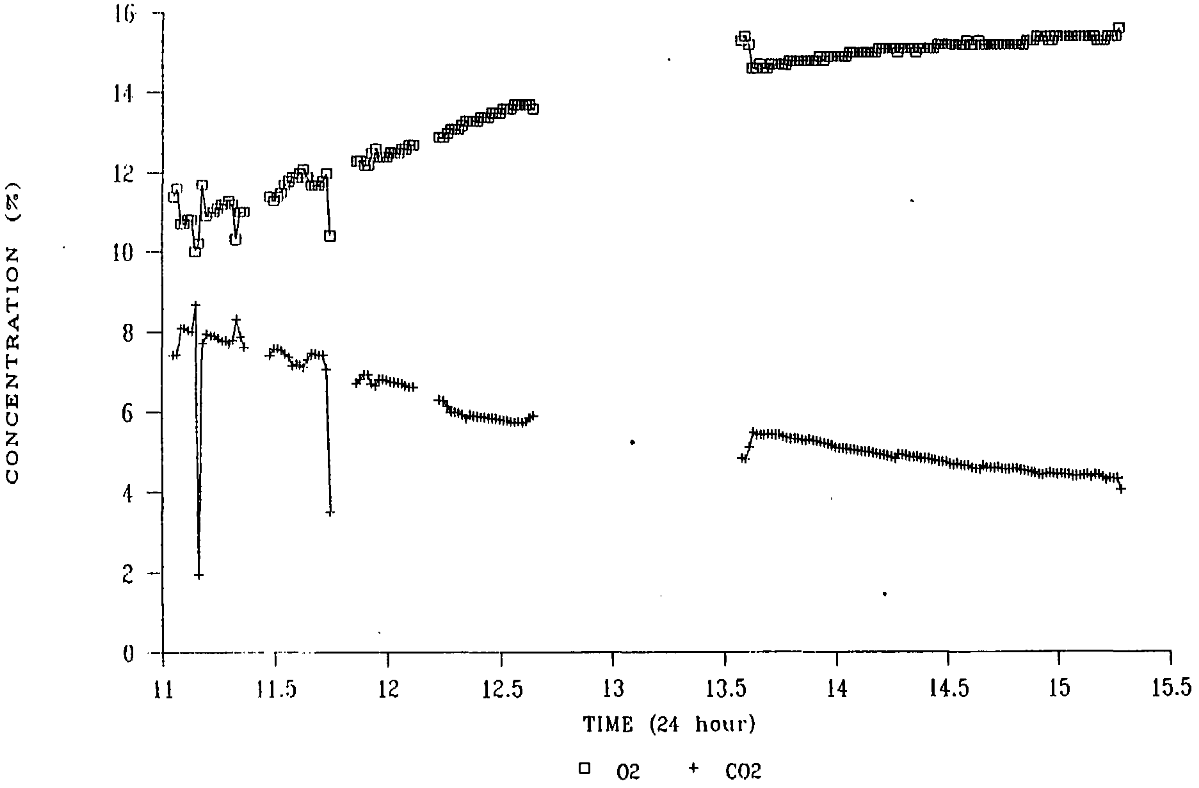




SITE B

RUN 5-INLET





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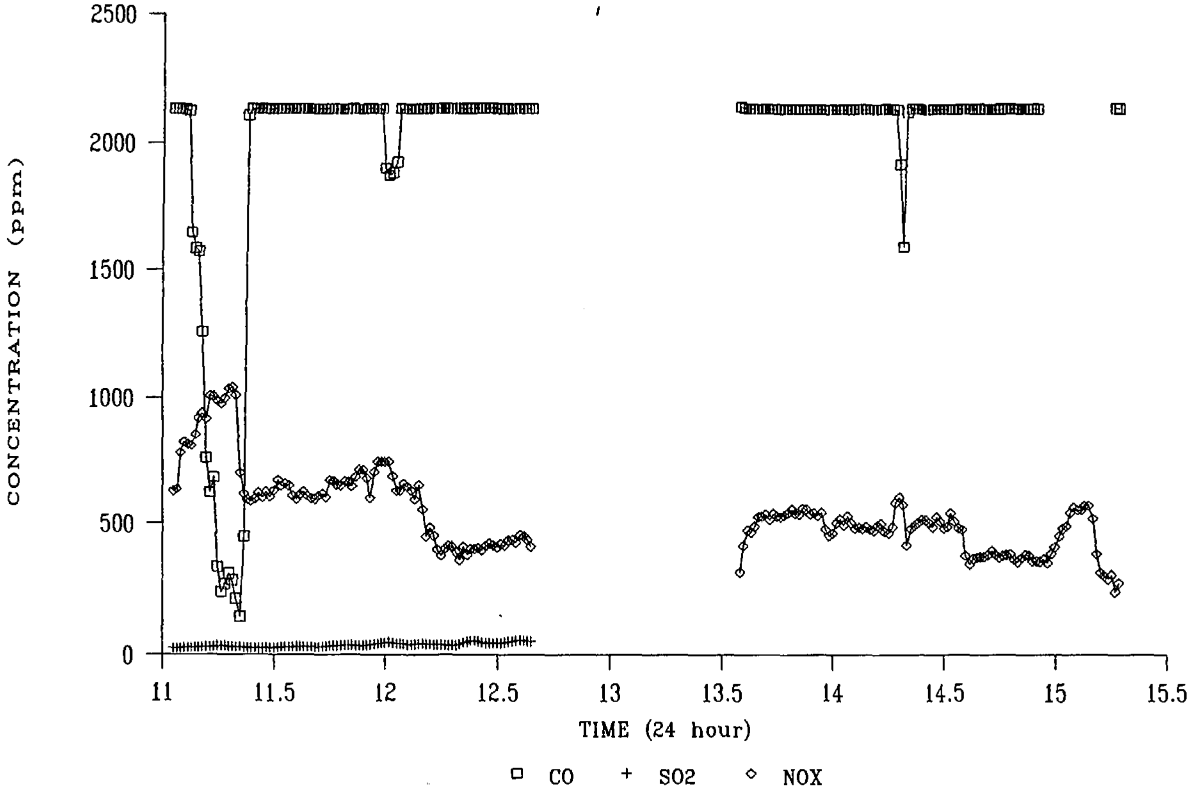
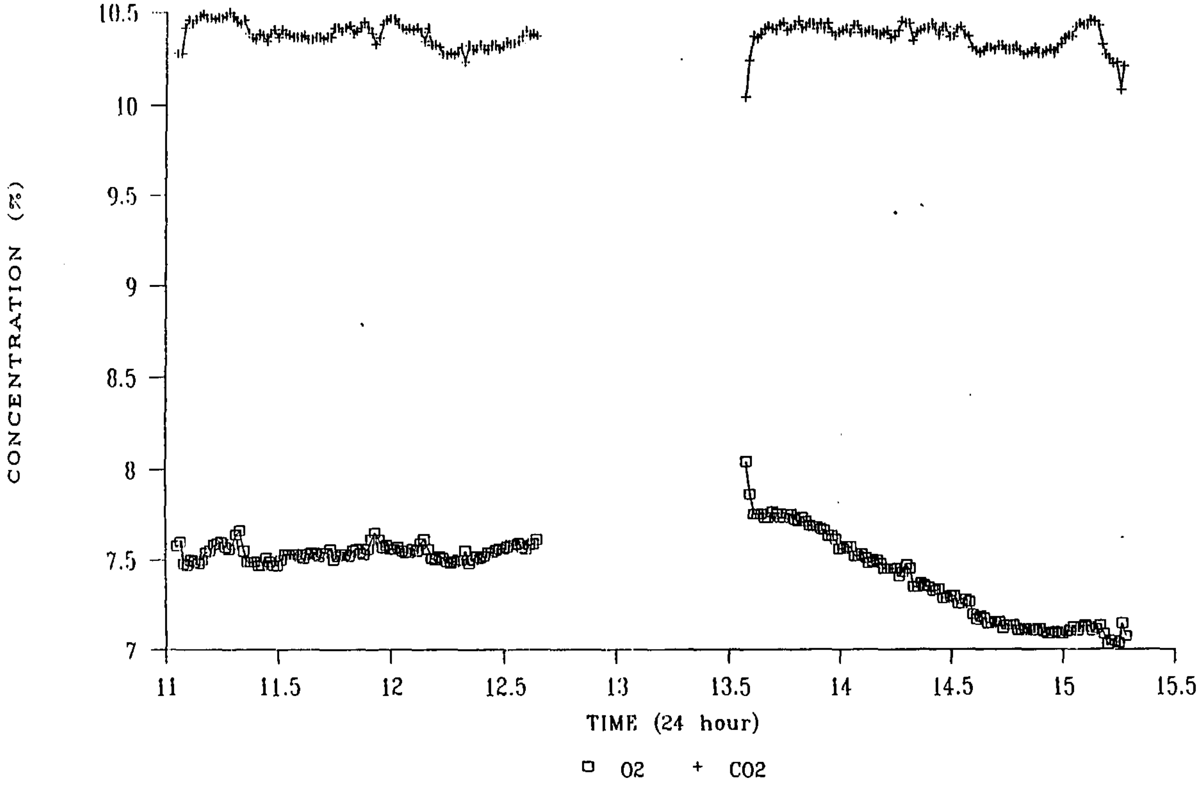
RUN 5-OUTLET





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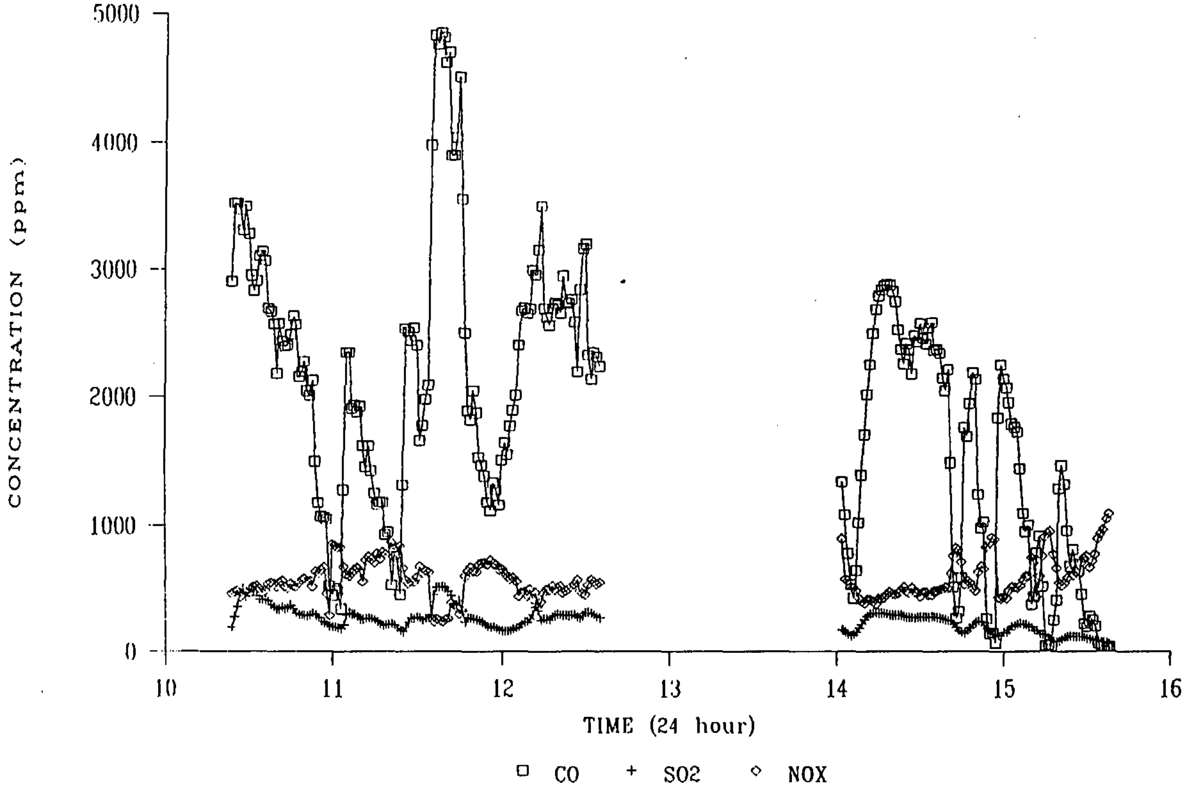
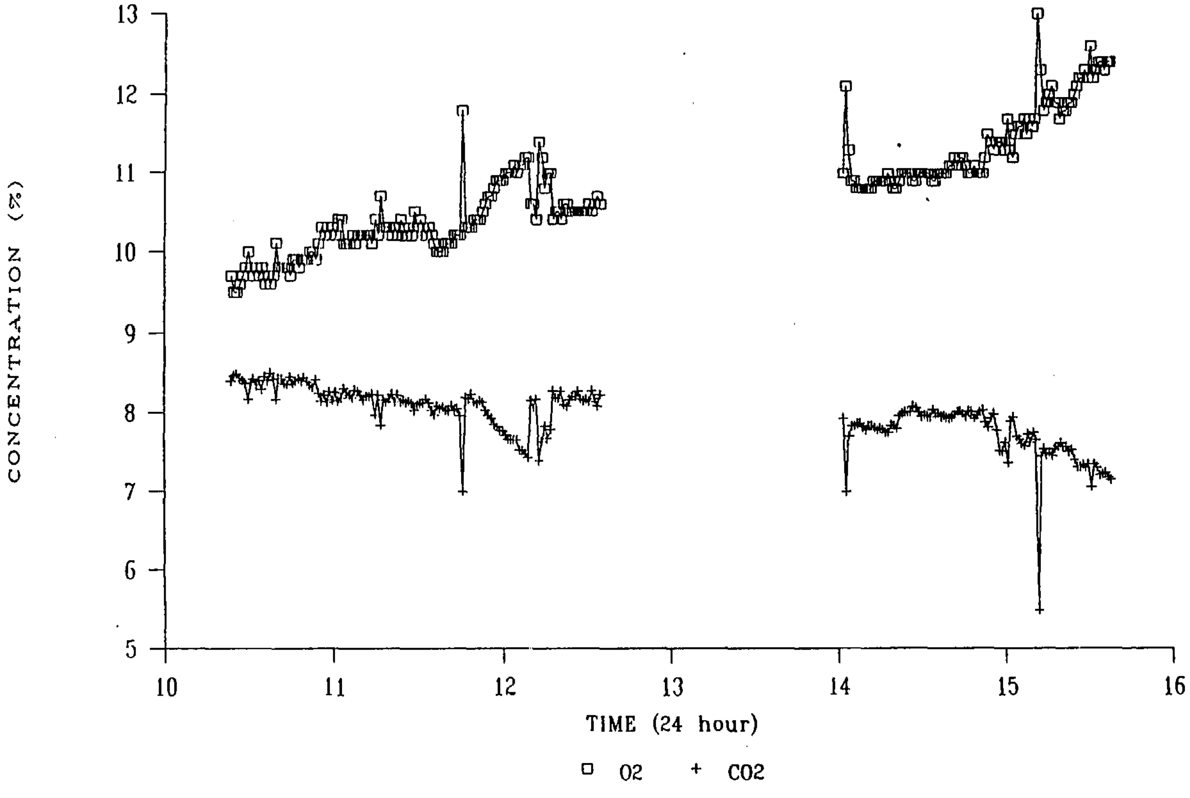
RUN 6-INLET





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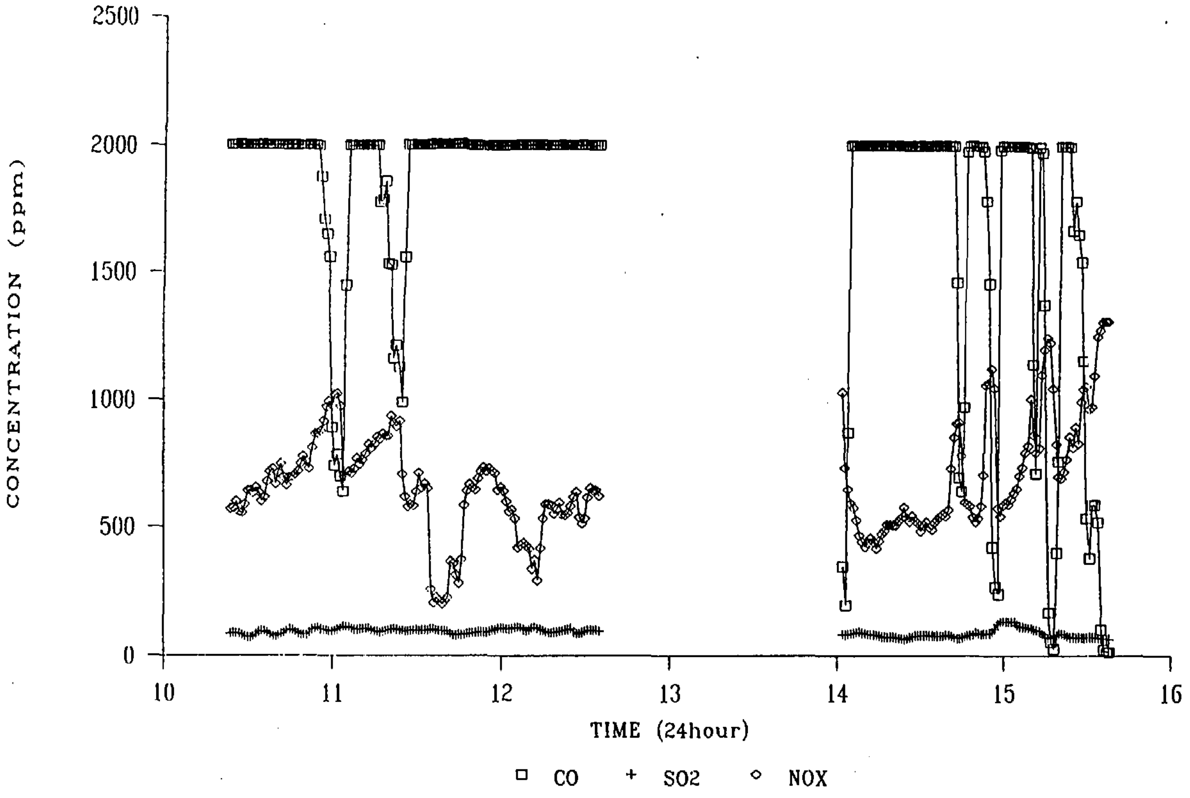
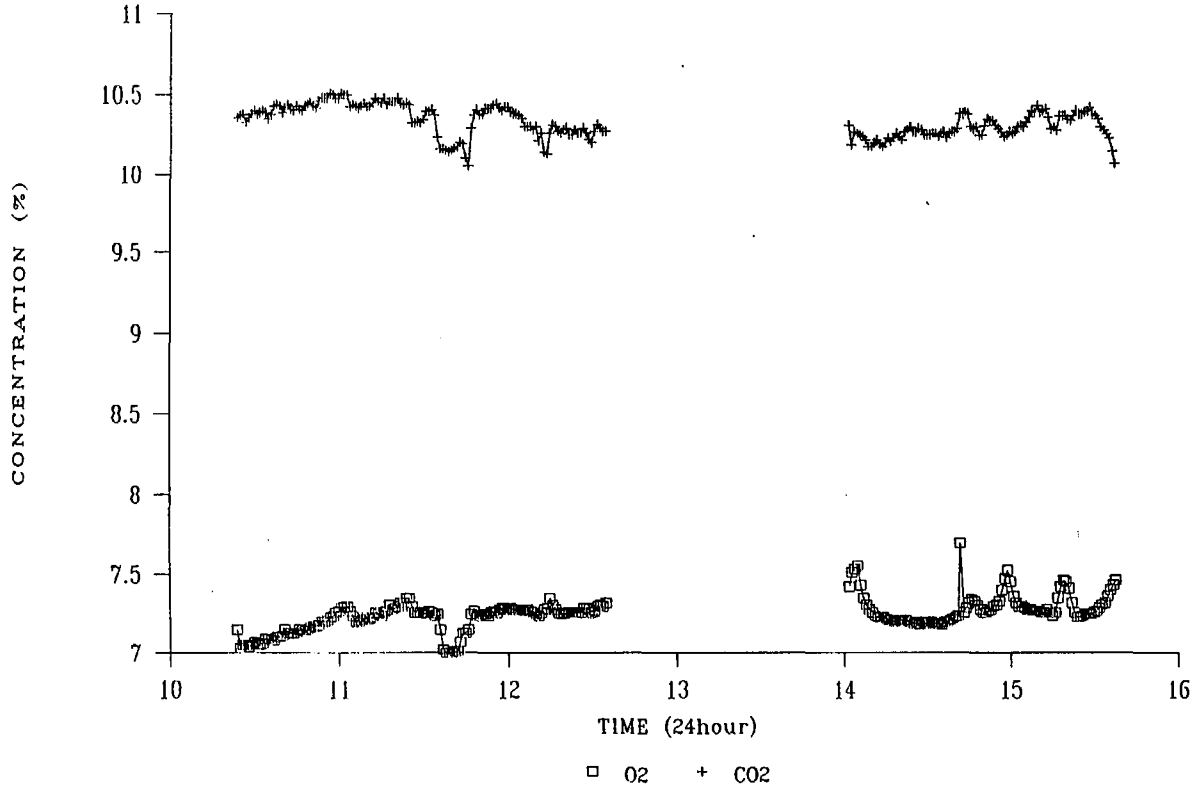
RUN 6-OUTLET





SITE B SUMMARY OF CEM OPERATING PARAMETERS

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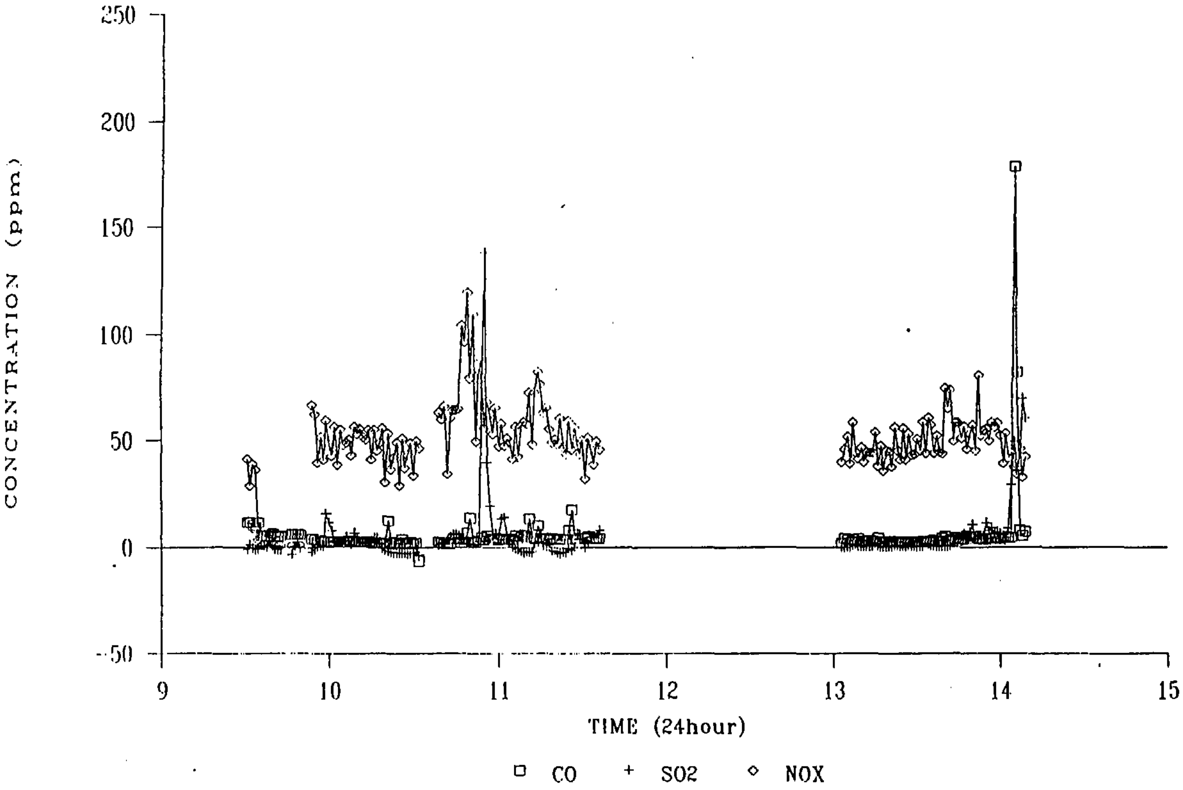
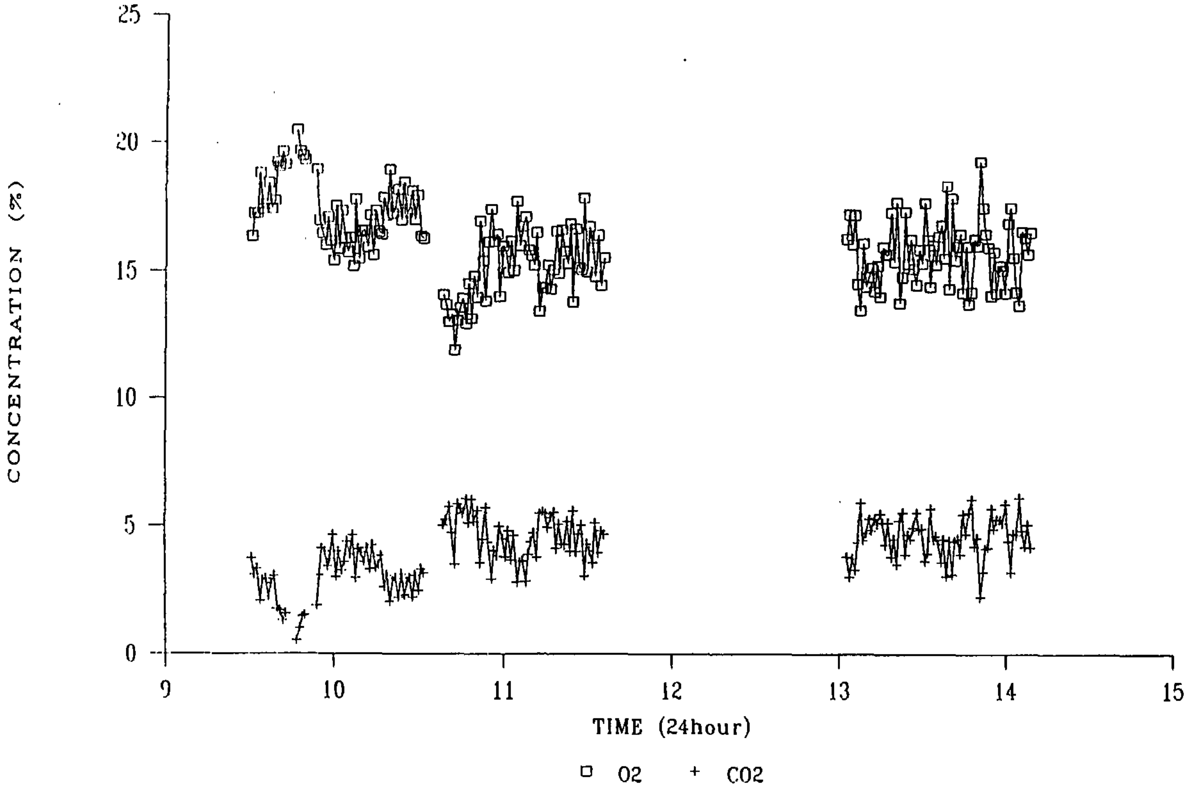
RUN 4 INLET				
O2	1.06	0.84	0.5	2.7
CO2	0.79	0.22	0.5	0.3
CO	2.51	2.38	0.6	1.8
SO2	1.35	8.26	1.09	1
NOx	1.15	3.37	0.9	0.05
RUN 4 OUTLET				
O2	0.09	2.68	0.5	1.4
CO2	0.17	0.23	0.2	0.8
CO	1.07	0.36	0.6	0.5
SO2	0.35	0.35	0.5	0.8
NOx	0.43	3.62	0.5	0.2
RUN 5 INLET				
O2	0.35	0.24		
CO2	1.63	1.52		
CO	2.53	0.25		
SO2	0.96	0.92		
NOx	2.48	5.35		
RUN 5 OUTLET				
O2	1.76	4.49		
CO2	0.26	0.31		
CO	1.31	0.59		
SO2	1.57	0.00		
NOx	0.08	0.70		
RUN 6 INLET				
O2	2.37	3.13		
CO2	2.91	2.63		
CO	1.5	0.25		
SO2	0.28	1.14		
NOx	1.25	2.65		
RUN 6 OUTLET				
O2	0.72	0.3		
CO2	0.09	0.92		
CO	0.61	0.74		
SO2	0	1.74		
NOx	0.78	1.64		

SITE C

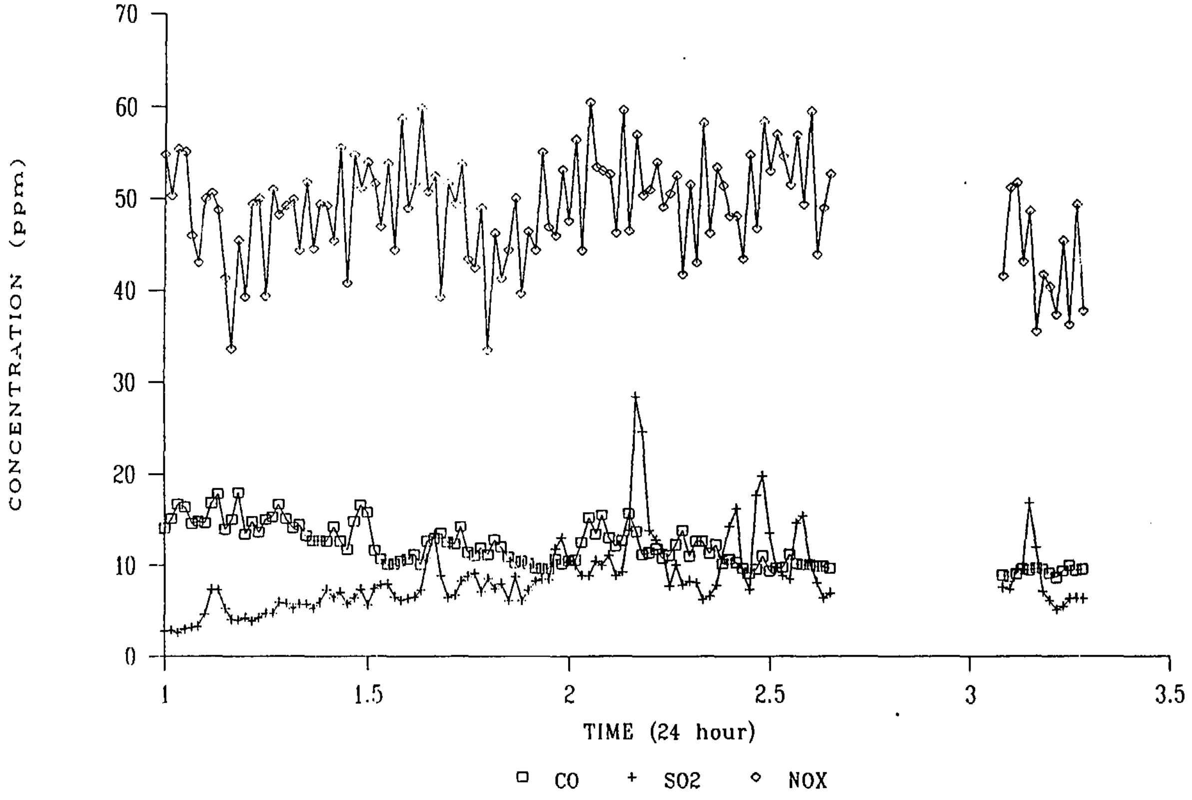
RUN 7-OUTLET

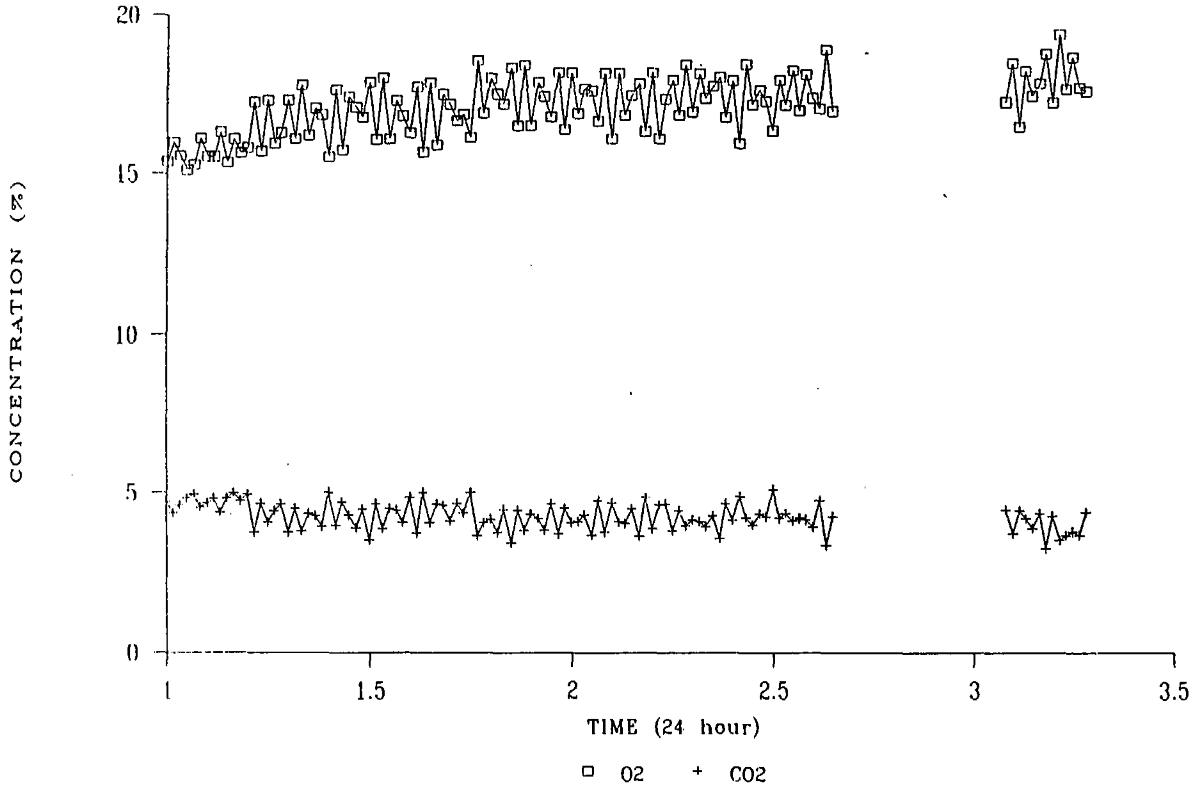




SITE C

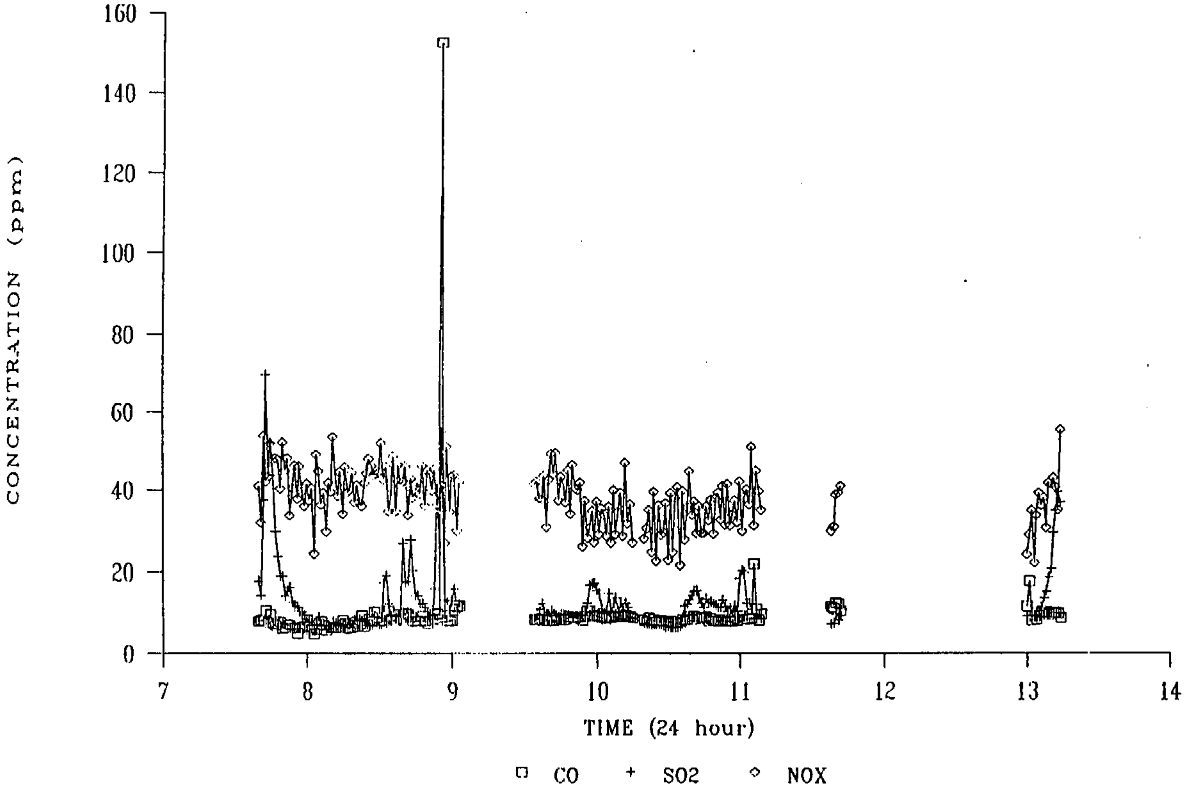
RUN 8-OUTLET

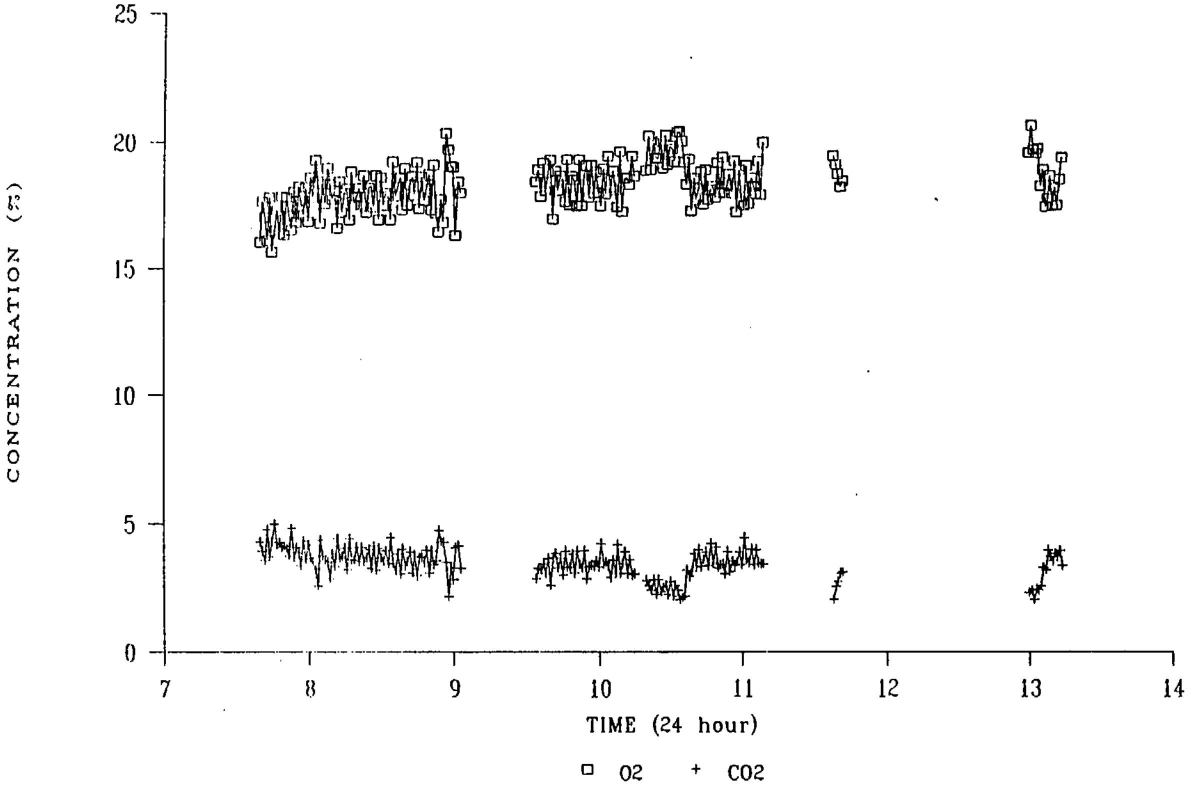




SITE C

RUN 9-OUTLET



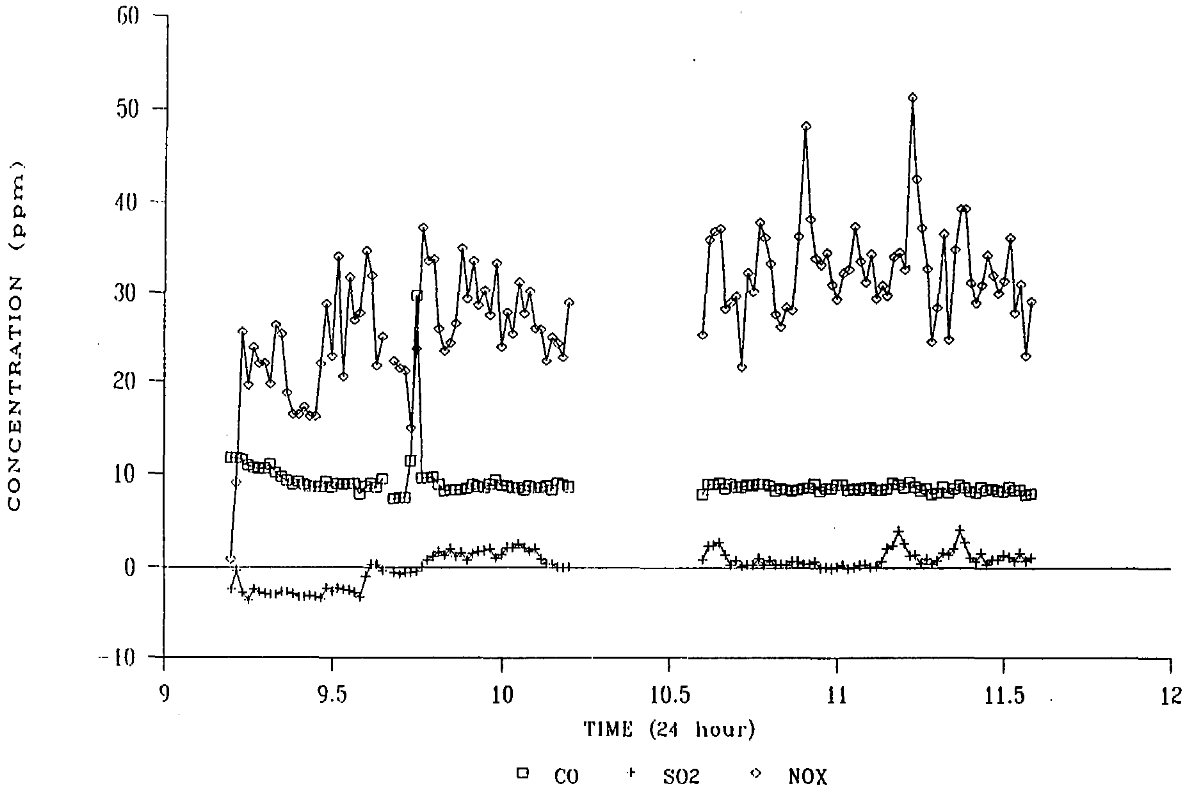


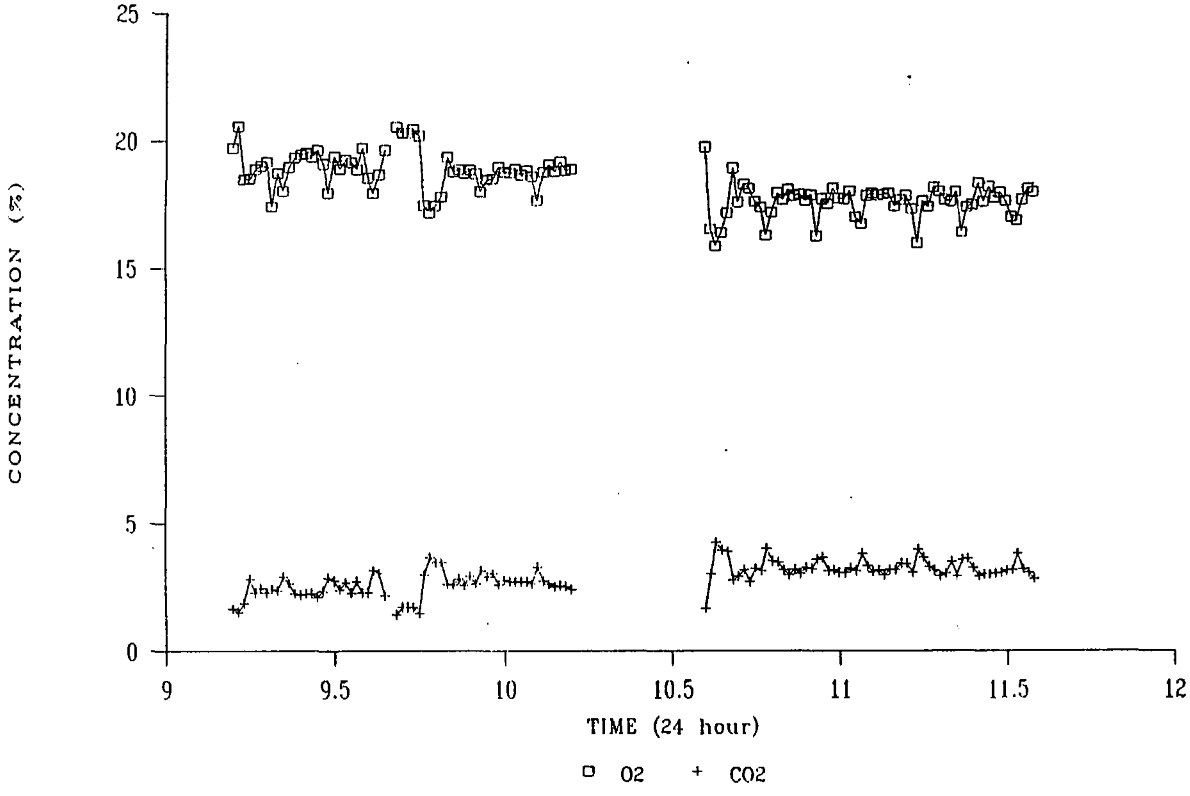
SITE C SUMMARY OF CEM OPERATING PARAMETERS

	ZERO DRIFT (% of span)	SPAN DRIFT (% of span)	LINE BIAS (% of span)	ANALYZER LINEARITY DEVIATION (% of span)
RUN 7 OUTLET				
O2	0.47	0.59	1.26	1.35
CO2	0.37	0.64	0.75	0.8
CO	0.56	1.14	0.2	0.89
SO2	0.17	1.33	1.82	0.95
NOx	1.48	2.19	0.15	0.7
RUN 8 OUTLET				
O2	0.3	0.18		
CO2	0.13	0.27		
CO	2.26	2.50		
SO2	0.41	2.30		
NOx	1.34	0.52		
RUN 9 OUTLET				
O2	0.48	2.63		
CO2	0.2	0.59		
CO	1.65	0.93		
SO2	0.4	3.55		
NOx	0.84	0.15		

SITE D

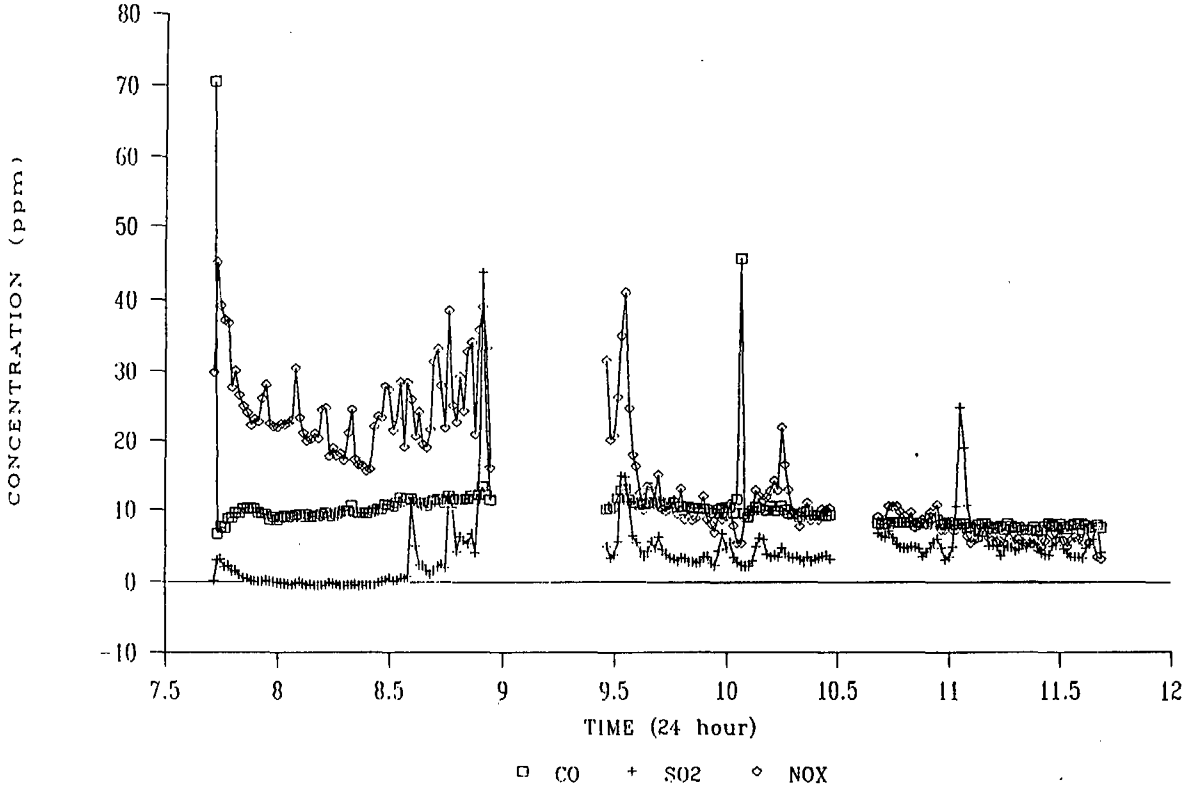
RUN 10 OUTLET

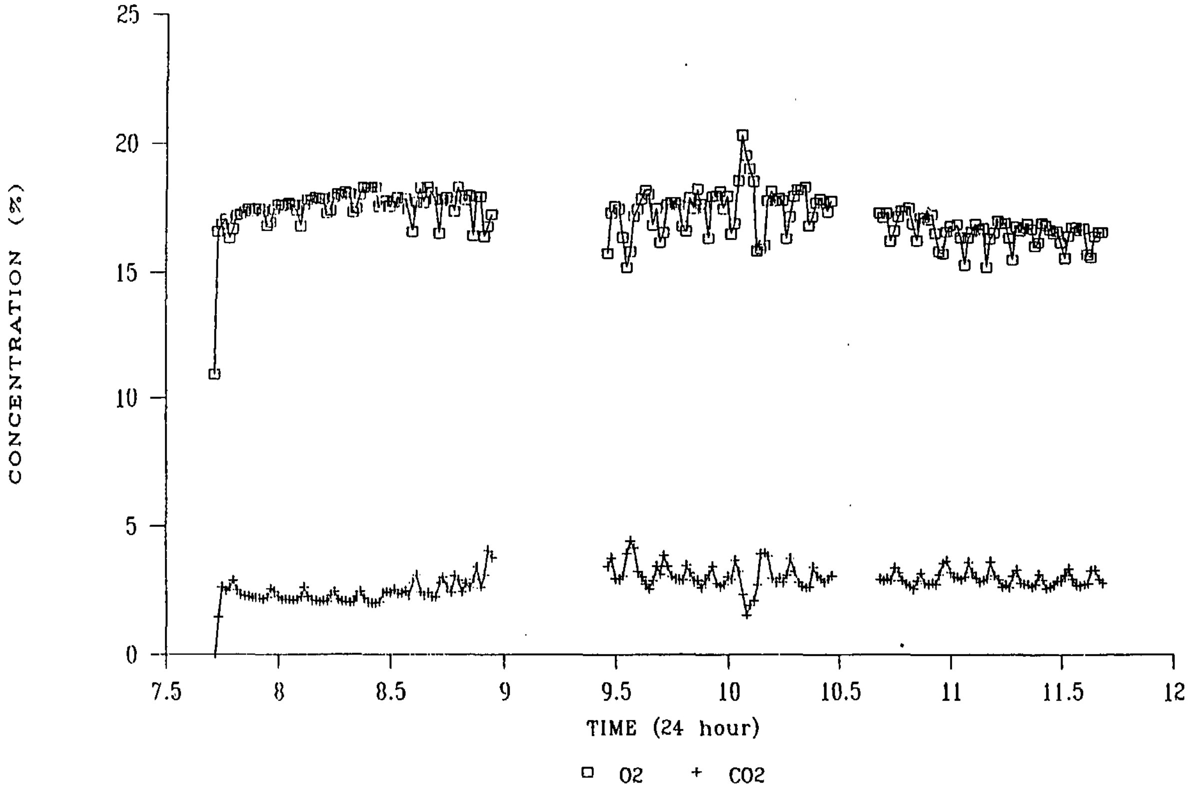




SITE D

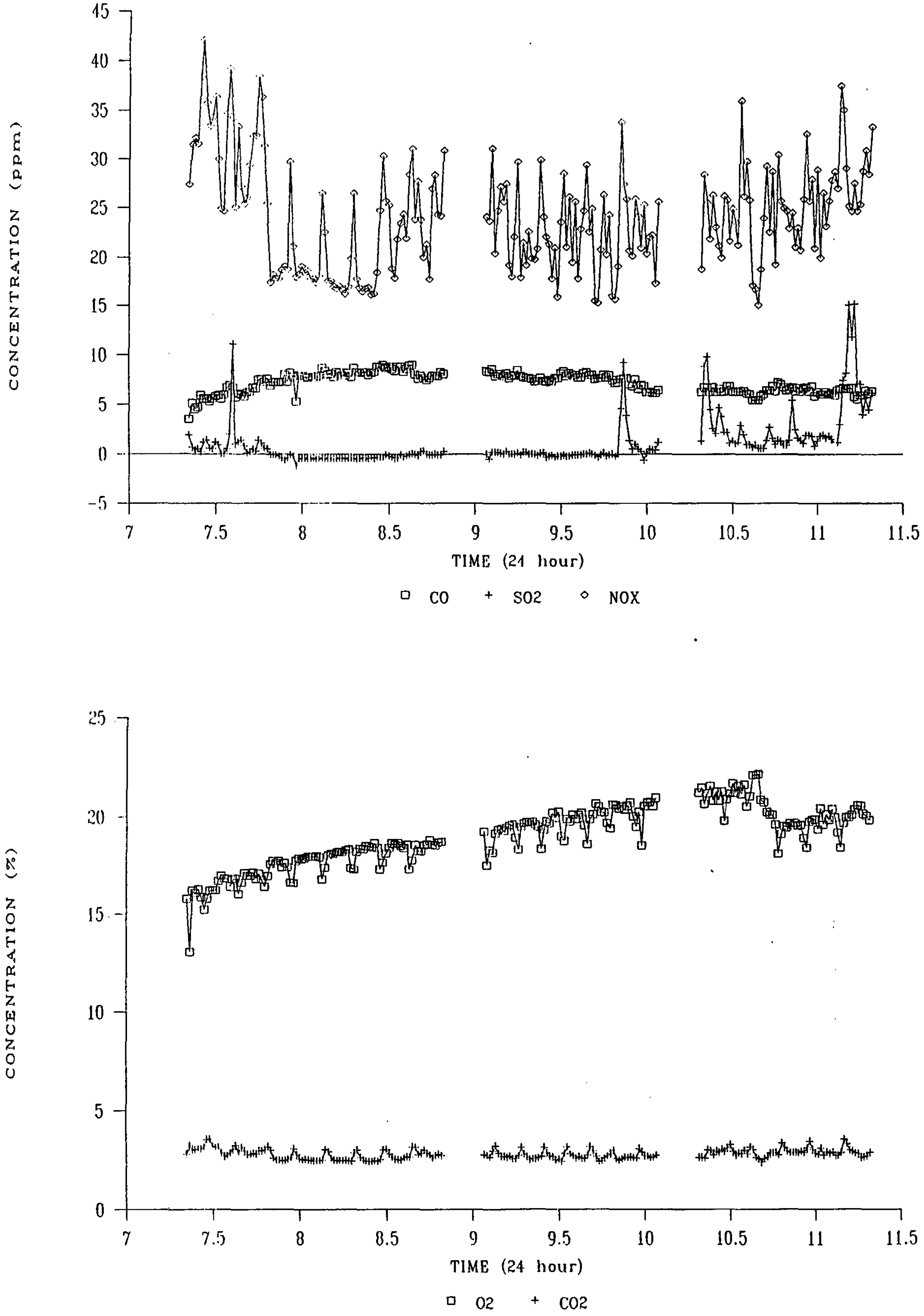
RUN11--OUTLET





SITE D

RUN 12-OUTLET



SITE D SUMMARY OF CEM OPERATING PARAMETERS

	ZERO DRIFT (% of span)	SPAN DRIFT (% of span)	LINE BIAS (% of span)	ANALYZER LINEARITY DEVIATION (% of span)
RUN 10 OUTLET				
O2	0.47	0.63	0.6	1.6
CO2	0.44	1.02	1.16	0.1
CO	1.63	1.56	0.9	0.6
SO2	0.00	0.39	0.6	0.3
NOx	1.08	0.19	0.1	0.3
RUN 11 OUTLET				
O2	0.69	0.64		
CO2	0.66	0.82		
CO	1.1	1.65		
SO2	0.79	3.23		
NOx	0.21	3.16		
RUN 12 OUTLET				
O2	0.37	0.51		
CO2	2.11	1.48		
CO	0.75	1.31		
SO2	0.73	10.69		
NOx	0.6	1.13		

APPENDIX C

WASTE OIL ANALYSES PROVIDED BY SITE FACILITIES A AND B

SITE A: WASTE OIL COMPOSITION (Obtained from Facility)

Analyte	Concentration (ppm)[a]
As	0.2
Cd	0.2 -2
Cr	0.5 - 2
Pb	1.5 - 25

[a] Data provided by plant for waste oil analysis conducted during February - April 1990.

SITE B: WASTE OIL COMPOSITION (Obtained from Facility)

Analyte	Result (mg/Kg)[a]	Reporting Limit (mg/Kg)	EPA Method
Antimony	ND	2.5	6010
Arsenic	ND	1.3	6010
Barium	0.90	0.25	6010
Beryllium	ND	0.35	6010
Cadmium	ND	0.25	6010
Chromium (total)	0.26	0.25	6010
Cobalt	ND	0.25	6010
Copper	5.7	0.5	6010
Lead	4.7	1.3	7420
Mercury	ND	0.1	7471
Molybdenum	2.3	0.25	6010
Nickel	6.8	0.25	6010
Selenium	ND	1.3	6010
Silver	ND	0.5	6010
Thallium	ND	2.5	6010
Vanadium	11	0.5	6010
Zinc	73	0.25	6010

[a] Data provided by plant for waste oil analysis on sample submitted on 9/5/90.