FINAL REPORT

VOLUME I.

TESTING NON-RECOVERY COKE OVENS FOR STANDARDS DEVELOPMENT

JEWELL COAL & COKE COMPANY
VANSANT, VIRGINIA

EPA CONTRACT NO. 68D90055
EPA PROJECT NO. 91-ME-34
WORK ASSIGNMENT NO. 2-94

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United States Environmental Protection Agency
Research Triangle Park, North Carolina 27711

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DISCLAIMER

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1.0 INTRODUCTION

1.1 SUMMARY OF TESTING PROGRAM

The U.S. Environmental Protection Agency (EPA), Office of Air Quality Planning and Standards (OAQPS), Emission Measurement Branch (EMB) in support of National Emission Standards for Hazardous Air Pollutants (NESHAPS), contracted Entropy Environmentalists, Inc. (Entropy) to conduct a testing program at the Jewell Coal & Coke Company in Vansant, Virginia to determine emissions from a non-recovery coke oven. The testing program was performed from October 28, 1991 through November 1, 1991.

OAQPS/EMB was assisted by the Industrial Studies Branch (ISB). The testing protocol was approved by Office of Research and Development (ORD), Source Method Research Branch (SMRB). The goal of this project was to provide the most comprehensive and reliable data obtainable within a reasonable cost.

The primary purposes of the testing program were to conduct a laboratory and field validation study on the Modified Method 5 sampling train and to perform an emissions characterization evaluation for the following pollutants:

- particulate matter (PM),
- toluene soluble organic compounds (TSO),
- condensible particulate matter (CPM),
- multiple-metals (MMTL), including antimony (Sb), arsenic, (As), barium (Ba), beryllium (Be), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), phosphorus (P), selenium (Se), silver (Ag), thallium (Tl), and zinc (Zn),
- semi-volatile organic quantitative compounds [Modified Method 5 sampling train (MM5)], including benzo-a-pyrene (BaP), cresol, naphthalene, phenol, toluene, and xylenes {ortho (o-), meta (m-), and para (p-)}, plus semi-quantitative and organic qualitative compounds,
- volatile organic quantitative compounds [volatile organic sampling train (VOST)], including benzene, plus semi-quantitative and qualitative organic compounds, and

- continuous emissions monitoring (CEM), including sulfur dioxide (SO_2), nitrogen oxides (NO_x), and carbon monoxide (CO).

1.2 KEY PERSONNEL

Figure 1-1 outlines the organizational scheme that was followed during the field testing program. The EMB Work Assignment Manager was Roy Huntley. The Test Program Contact for Jewell Coal & Coke Company was Rick Waddell. Gene Crumpler, EPA/ISB, Emery Kong, RTI, and Merrill Jackson and Larry Johnson, ORD/SMRB, provided technical guidance. The Entropy Project Director was Helen Yoest; Entropy Project Managers and Field Coordinators were Helen Yoest and Todd Brozell. The QA/QC Officer was Emil Stewart of Entropy. Entropy's Safety Officer for this testing program, was Jared Byer. Entropy subcontracted one outside laboratory, Triangle Laboratories, Inc. (TLI), for the MMTL, TSO, MM5, and VOST sample analyses. Entropy performed the remaining analyses.

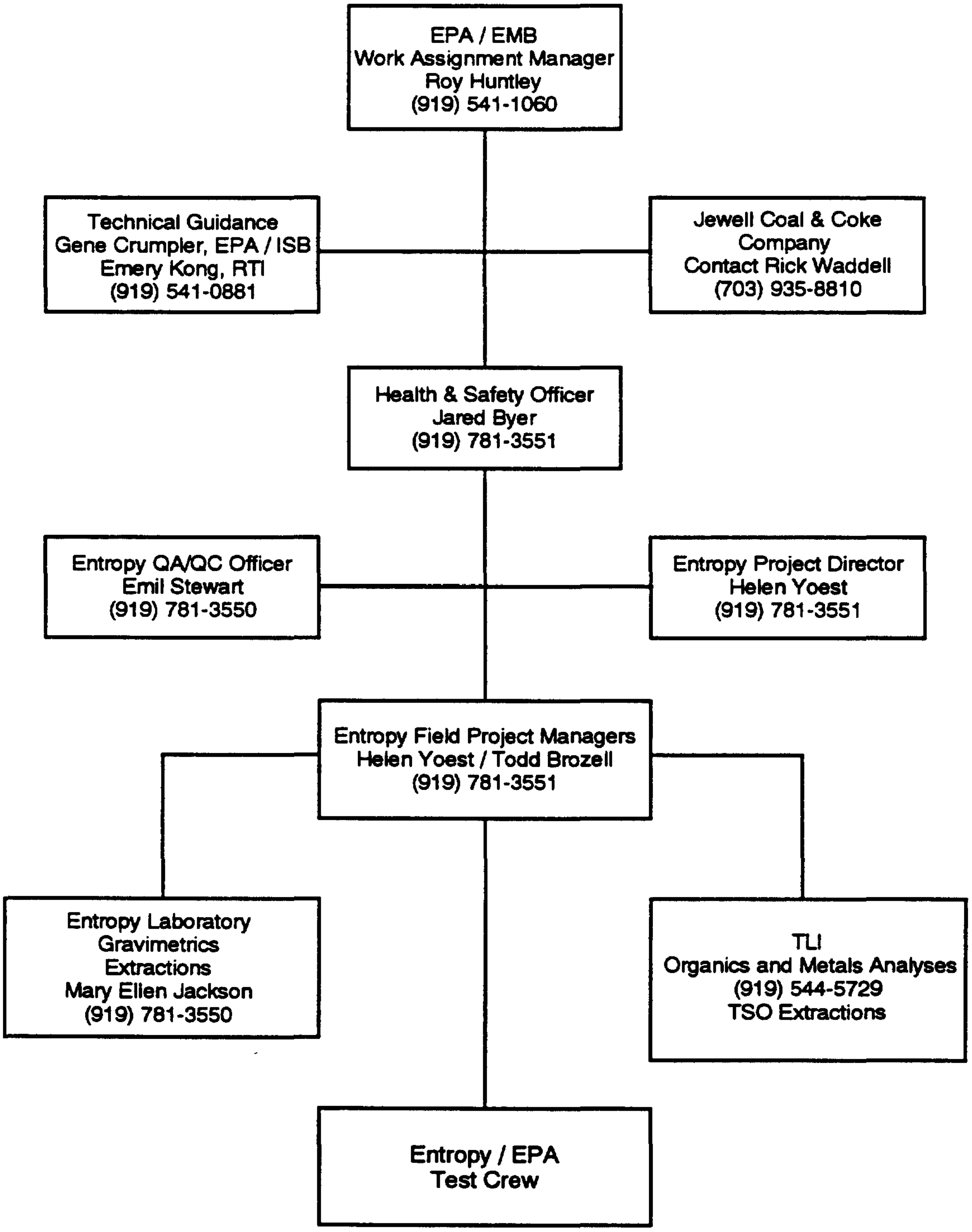

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Figure 1-1. Jewell Coal & Coke Testing Program Organization.

2.0 PLANT AND SAMPLING LOCATION DESCRIPTIONS

2.1 PROCESS DESCRIPTION AND OPERATION

Thirty-six ovens exhaust into four stacks via a common tunnel in Battery 3-C; therefore, each stack approximates the emission's flow rate of nine ovens combined. These ovens were designed with a non-recovery coking technology which uses the volatile fraction driven off the coal as fuel for carbonization - producing heat, carbon dioxide, and water vapor. Coke is the finished product produced by the process which is widely used in the steel industry.

Primary air for combustion is introduced into the oven chamber through one of several ports located on the doors above the charge. These ports are fitted with adjustable dampers to control the amount of air in order to maintain the desired temperature as measured in the oven crown. The partially combusted gas exits the oven chamber through downcomer passages in the oven wall to the sole flues. The sole flues are separated into two sides, each with four passes for combustion of the off-gas prior to exiting the heating system. This design allows the coke side and the pusher side to be controlled individually for more uniform heating. A damper is installed in the second pass of each sole flue to allow outside air to be introduced for complete combustion in the sole flue. Usually additional air is required in the sole flue only during the first hour or two after charging. Spent gas is conducted to a common tunnel through uptake passages in the oven wall. As a further means for controlling oven temperatures, a specially designed uptake damper is used to regulate draft.

The coking process operates on a 48-hour cycle that begins when the even numbered ovens are charged with approximately 40 tons of coal. The heat retained in the coke oven initiates a new carbonization cycle wherein heat is generated when the volatile fraction of the coal is combusted. This coal is left in the ovens to carbonize for 48 hours, after which the coke is pushed out the oven door into a waiting rail car. Twenty-four hours after the charging of the even numbered ovens, the odd numbered ovens are charged with coal and begin their 48-hour cycle. Exceptions to this process schedule, involving Oven 70, are documented in Section 6.0.

Given the process operations, the stack emissions are based on a 24-hour cycle. During this 24-hour cycle, half of the ovens were charged with coal and the other half had coal carbonizing in them.

2.2 SAMPLING LOCATION DESCRIPTIONS

The emissions testing program at the Jewel Coal & Coke Company was conducted on Battery C, Stacks 1-4. The dimensions of the four sampling locations were the same.

The sampling locations and an overview of Battery C are illustrated in Figure 2-1.

2.2.1 Battery C, Stack 1

As shown in Figure 2-2, Stack 1 has two 4-inch ports designated as A and B, located at 90° angles. These ports were used for isokinetic sampling of Methods M5/TSO/202 and MMTL. After determining the stack's inner diameter to be 98.5 inches, and establishing the ports to be 5.5 duct diameters from the nearest upstream disturbance and 0.80 duct diameters from the nearest downstream disturbance (stack exit), a total of 24 sampling points were sampled by each train, 12 points per axis. While one train traversed into the duct of Port A, the other traversed out of Port B.

2.2.2 Battery C, Stack 2

As shown in Figure 2-3, Stack 2 has two 4-inch ports located at 90° angles designated as A and B. These ports were used for the isokinetic sampling of paired MM5 trains. After determining the stack's inner diameter to be 96 inches, and establishing the ports to be 5.2 duct diameters from the nearest upstream disturbance and 0.82 duct diameters from the nearest downstream disturbance (stack exit), a total of 24 points were sampled by the paired trains on one axis due to limitations in the port opening.

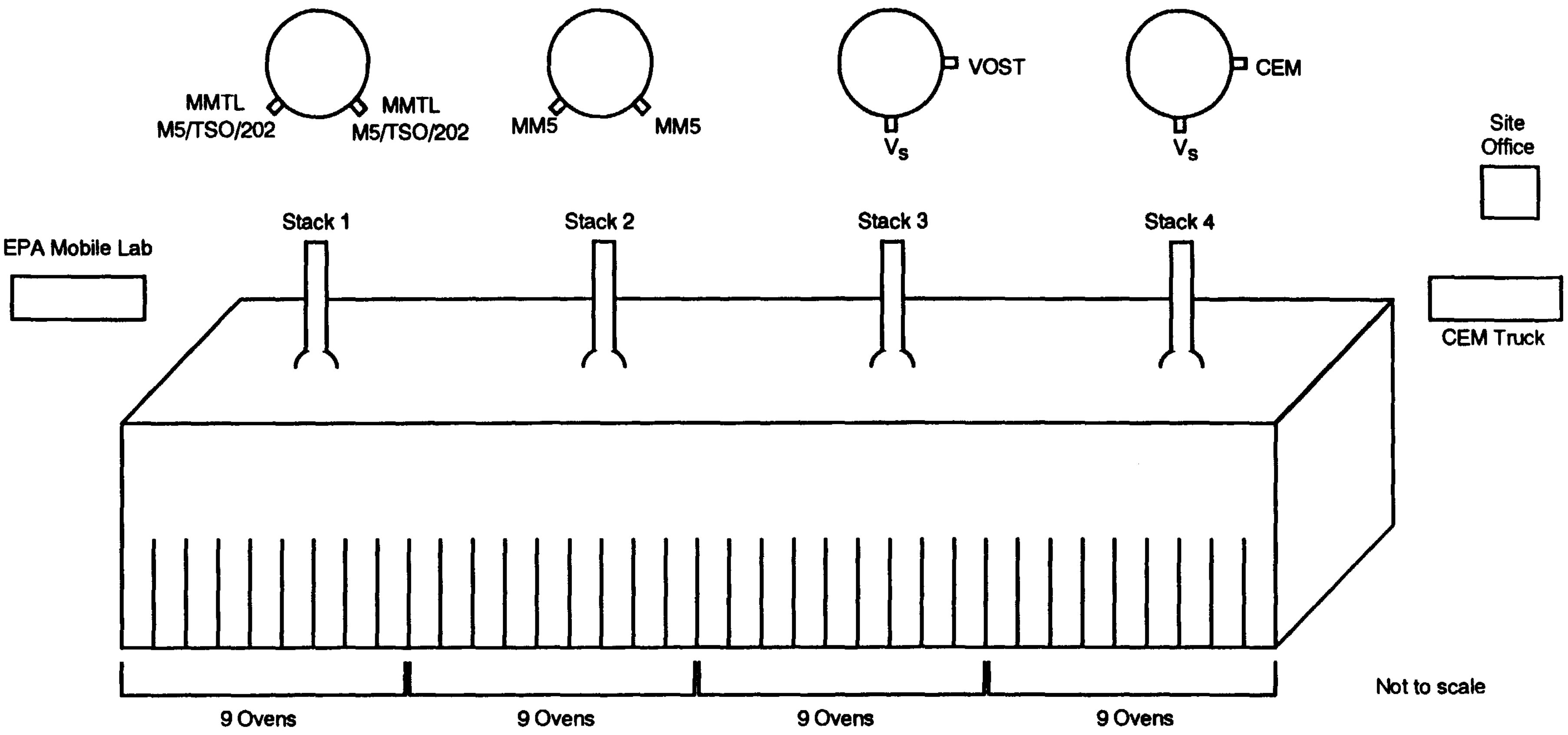
2.2.3 Battery C, Stack 3

As shown in Figure 2-4, Stack 3 has two 4-inch ports designated as A and B at 90° angles and an inner diameter of 98 inches. A manual EPA Method 2 traverse of Port A was conducted to determine a point of average velocity; the continuous EPA Method 2 probe was then placed at this point to monitor flue gas velocity and temperature. A VOST train was operated in Port B. Both methods were single point and non-isokinetic.

2.2.4 Battery C, Stack 4

As shown in Figure 2-5, Stack 4 has two 4-inch ports designated as A and B at 90° angles; the stack's inner diameter was measured and found to be 96.125 inches. A continuous EPA Method 2 probe placed at the point of average velocity monitored Port A, while a continuous emission monitoring (CEM) probe for Methods 3A, 6C, 7E, and 10 was operated in Port B. The CEM methods were single point and non-isokinetic.

Non-Recovery Coke Oven
Battery C
Test Method and Port Orientation



MMTL - Multi-metals Train
M5 - Method 5, Particulate Matter
TSO - Toluene Soluble Organics
202 - Condensibles
MM5 - Semi-volitile Organics
VOST - Volatile Organics
V_s - Velocity
CEM - Continuous Emission Monitoring

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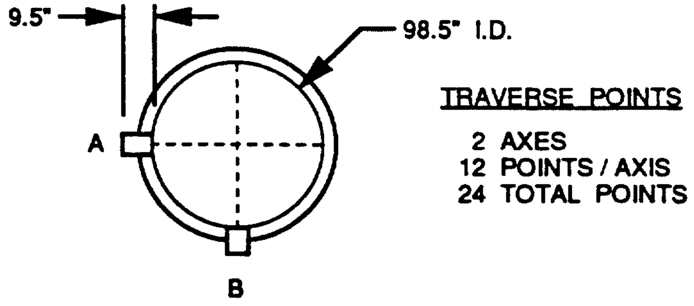
Figure 2-1. Overview of Battery C.



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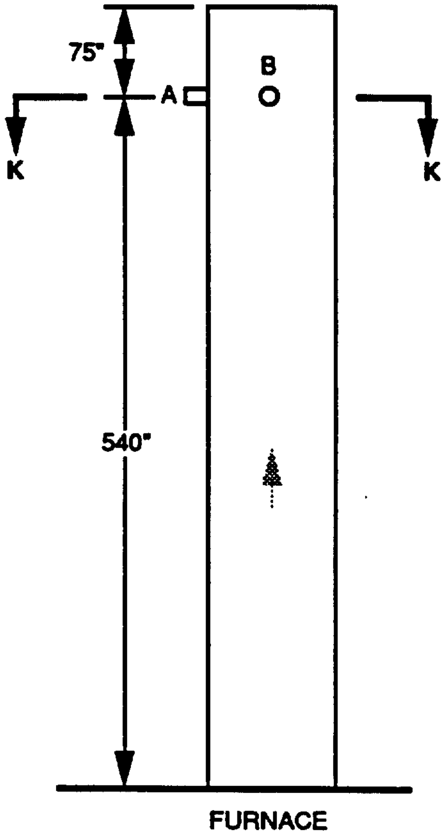
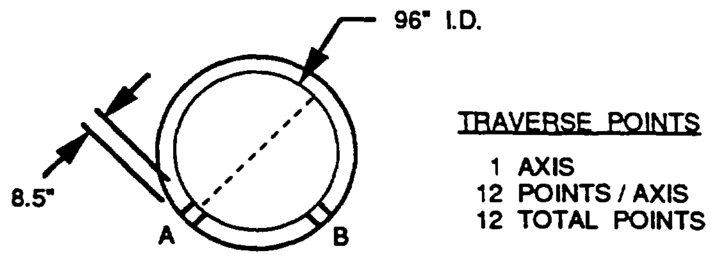


Figure 2-2. Stack 1 Test Location



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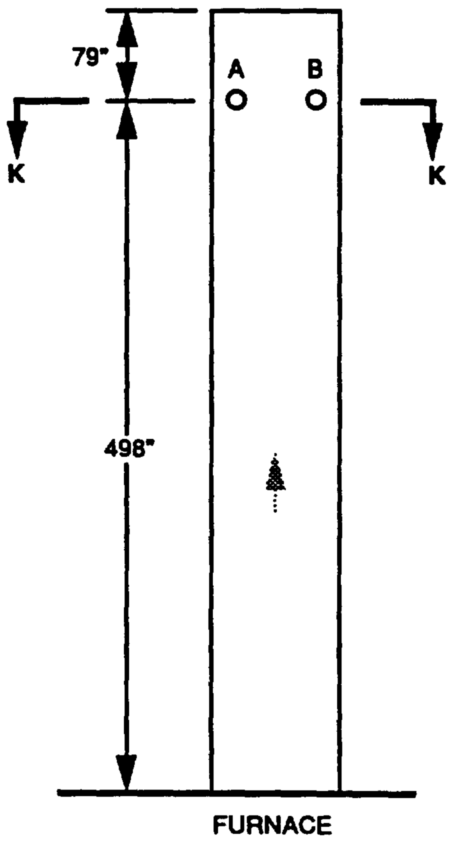
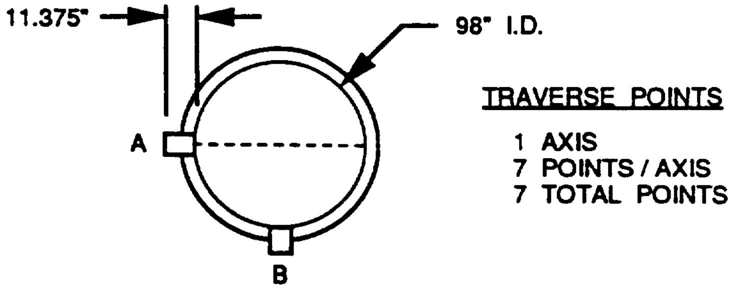


Figure 2-3. Stack 2 Test Location



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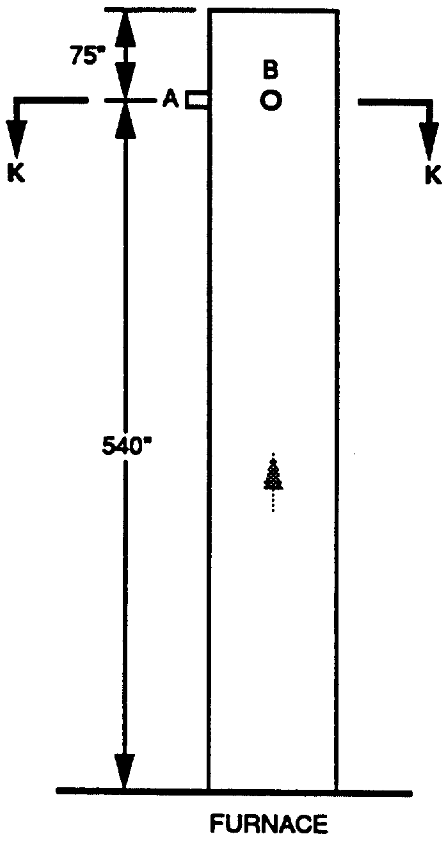
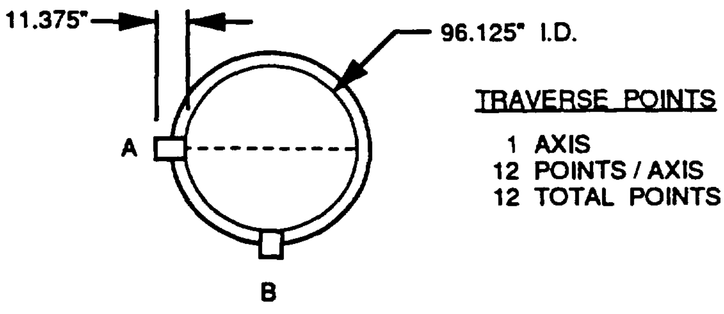


Figure 2-4. Stack 3 Test Location



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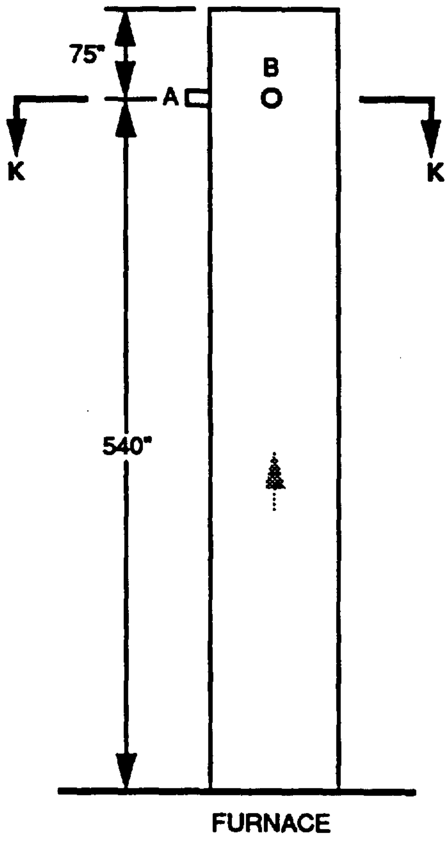

Figure 2-5. Stack 4 Test Location

3.0 OBJECTIVES AND OVERALL TEST MATRIX

3.1 OBJECTIVES

The primary objective of the testing program was to collect emissions data to characterize the emissions from the Jewell Coal and Coke Company non-recovery coke oven in support of NESHAPS.

Not all of the sampling methods used in the testing program were validated. Hence, laboratory and field validations were performed during this testing program for the quantitative semi-volatile organic compounds. For purposes of this document, PM, metals, benzene, and CEM were considered validated from a combustion source.

At Stack 1, PM, TSO, CPM, and MMTL were measured using EPA Method 5 (M5) for PM, an unnumbered method for TSO, Method 202 for CPM, and a draft EPA method for MMTL. Semi-volatile organic compounds were measured from Stack 2 using EPA Office of Solid Waste (SW-846) 846 Method 0010. Volatile organic compounds were measured from Stack 3 using SW-846 Method 0030; O_2 and CO_2, SO_2, NO_x, and CO were measured instrumentally from Stack 4 using Methods 3A, 6C, 7E, and 10, respectively.

The testing program objectives are summarized below:

- Determination of the levels of PM, CPM, metals, O_2, CO_2, SO_2, NO_x, CO, and organic compounds (volatile, semi-volatile, and TSO) from a non-recovery coke oven.
- Laboratory validation of semi-volatile organic compounds including BaP, cresol, naphthalene, phenol, toluene, and xylenes (o-, m-, p-).
- Field validation of semi-volatile organic compounds, including BaP, cresol, naphthalene, phenol, toluene, and xylenes (o-, m-, p-) from paired MM5 sampling trains.

All test runs were performed while the plant was operating under normal conditions. Four conditions were selected for each 24-hour cycle. It was desired to characterize emissions during the charging cycle; therefore, the charging cycle was selected as Condition I. The other three conditions were selected in order to facilitate the testing. Condition I was isolated during the charging cycle. The testing began at the beginning of the charge and continued two and a half hours into the charge. Condition II began where Condition I ended, with just enough down time for train turnarounds. Condition II testing was six hours in duration. Conditions III and IV followed the same format as Condition II, i.e., beginning the next run with just enough down time for turnarounds.

Testing was performed concurrently at each of the testing locations. Run durations varied with parameter and condition. Condition I M5/TSO/202, MM5, and MMTL sampling lasted approximately two hours; CEM sampling lasted approximately three hours. Sampling during Conditions II, III, and IV for the same parameters lasted approximately three hours. VOST for Condition I, Runs 1 and 4, lasted 20 minutes. The sampling time for Runs 2 and 3 was 40 minutes. All VOST runs during Conditions II, III, and IV lasted 40 minutes. All valid runs collected ~20 liters of sample. Any exceptions to the ideal run times are addressed in each parameter described in Sections 7.0 through 11.0.

Quality assurance/quality control (QA/QC) procedures, as specified by the applicable methods, were adhered to and test logs were maintained. The EPA Work Assignment Manager was consulted prior to any deviations from the procedures set forth in the test protocol.

3.2 TEST MATRIX

The sampling and analytical matrix for the testing program is presented in Table 3-1. Both manual and instrumental methods were used to determine the pollutant concentration emission rates. Each method is briefly described below. For more detailed descriptions of the testing program's sampling and analytical procedures, refer to Sections 7.0 through 11.0.

Particulate matter (PM and CPM) and TSO were determined using a combined train of M5 for PM, TSO, and EPA Method 202 for CPM. The PM, TSO, and CPM determinations were made from Stack 1.

Gravimetric analyses were performed on the front-half catch for PM, the back-half catch was analyzed for CPM, and the filter and front-half dry downs were then used for TSO analyses. EPA Method 3 was performed in conjunction with the particulate sampling for measuring O_2 and CO_2, while tests were performed to determine the flue gas molecular weight and the stack gas flow rate.

MMTL testing for the 16 metals previously mentioned was conducted using a draft EPA method. In cases where a choice was given for an analytical procedure, the procedure with the lowest detection limit was used.

Organic compound testing was conducted using the MM5 train of SW-846 Method 0010 for semi-volatile organic compounds, and the Volatile Organic Sampling Train (VOST) of SW-846 Method 0030 for volatile organic compounds. From an integrated sample taken at a single point from Stack 2, gas constituents were measured using EPA Method 3. Stack 3 gas constituents were determined from a proportion of the results from Stacks 1, 2, and 4.

Effluent gas samples were collected to determine the O_2 and CO_2, SO_2, NO_x, and CO using Methods 3A, 6C, 7E, and 10, respectively. All CEM measurements were taken from Stack 4.

Flow rate data for Stacks 1 and 2 were determined from the manual testing methods performed at the locations; for Stack 3, the flow rate data were obtained using a pressure transducer and thermocouple which fed information directly to the data acquisition system. Stack 4 flow rate data were supposed to be obtained in a similar procedure to that used for Stack 3; however, the data are suspect. Therefore, the data were analyzed in an effort to provide the case for using an average flow rate from Stacks 1, 2 and 3. More details concerning sampling and analyses can be found in Sections 7.0 through 11.0.

Figures 3-1, 3-2, and 3-3 graphically display time logs of each of the test methods.

TABLE 3-1. TEST MATRIX
JEWELL COAL AND COKE COMPANY

Sampling Location	Condition No.	No. of Runs	Sample/Type Parameter	Sampling Method	Sample Run Time (min)	Analytical Method	Analytical Laboratory
Stack 1	1	3	M5/TSO/202	M202	120	PM-M5/TSO, CPM-M202	PM/CPM - Entropy
	2	3	M5/TSO/202	M202	180	PM-M5/TSO, CPM-M202	TSO - TLI
	3	3	M5/TSO/202	M202	180	PM-M5/TSO, CPM-M202	
	4	3	M5/TSO/202	M202	180	PM-M5/TSO, CPM-M202	
Stack 1	1	3	MMTL	Draft EPA	120	ICAP/AAS/GVVS	TLI
	2	3	MMTL	Draft EPA	180	ICAP/AAS/GVVS	TLI
	3	3	MMTL	Draft EPA	180	ICAP/AAS/GVVS	TLI
	4	3	MMTL	Draft EPA	190	ICAP/AAS/GVVS	TLI
Stack 1	1	3	O_2/CO_2	M3	120	Orsat	Entropy
	2	3	O_2/CO_2	M3	180	Orsat	Entropy
	3	3	O_2/CO_2	M3	180	Orsat	Entropy
	4	3	O_2/CO_2	M3	180	Orsat	Entropy
Stack 2	1	3	MM5	SW-846-0010	120	HRGC/LRMS (M8270), HPLC	TLI
	2	3	MM5	SW-846-0010	180	HRGC/LRMS (M8270), HPLC	TLI
	3	3	MM5	SW-846-0010	180	HRGC/LRMS (M8270), HPLC	TLI
	4	3	MM5	SW-846-0010	180	HRGC/LRMS (M8270), HPLC	TLI
Stack 2	1	3	O_2/CO_2	M3	120	Orsat	Entropy
	2	3	O_2/CO_2	M3	180	Orsat	Entropy
	3	3	O_2/CO_2	M3	180	Orsat	Entropy
	4	3	O_2/CO_2	M3	180	Orsat	Entropy
Stack 3	1	4/per cycle	VOST	SW 846-0030	20/40**	HRGC/LRMS (M5040 & M8240)	TLI
	2	3/2*/per cycle	VOST	SW-846-0030	40	HRGC/LRMS (M5040 & M8240)	TLI
	3	2/1*/per cycle	VOST	SW-846-0030	40	HRGC/LRMS (M5040 & M8240)	TLI
	4	2/1*/per cycle	VOST	SW-846-0030	40	HRGC/LRMS (M5040 & M8240)	TLI
Stack 3	1	3	Velocity	M2	180	Differential Pressure	Entropy
	2	3	Velocity	M2	360	Differential Pressure	Entropy
	3	3	Velocity	M2	360	Differential Pressure	Entropy
	4	3	Velocity	M2	360	Differential Pressure	Entropy
Stack 4	1	3	CEM (O_2,CO_2,SO_2,NO_x,CO)	M3A,6C,7E,10	180	O_2 fuel cell	Entropy
	2	3	CEM (O_2,CO_2,SO_2,NO_x,CO)		360	CO_2 NDIR	Entropy
	3	3	CEM (O_2,CO_2,SO_2,NO_x,CO)		360	NO_x chemiluminescent	Entropy
	4	3	CEM (O_2,CO_2,SO_2,NO_x,CO)		360	SO_2, UV	Entropy
Stack 4	1	3	Velocity	M2	180	Differential pressure	Entropy
	2	3	Velocity	M2	360	Differential pressure	Entropy
	3	3	Velocity	M2	360	Differential pressure	Entropy
	4	3	Velocity	M2	360	Differential pressure	Entropy

*3 on Cycle 1, day 1; 2 on Cycles 2 and 3; one sample per cycle will be archived.

**The first and fourth of each are 20 minutes in duration. The second and third of each are 40 minutes in duration.

Figure 3-1.

Jewel Coal and Coke Time Log
Cycle 1, Day 1

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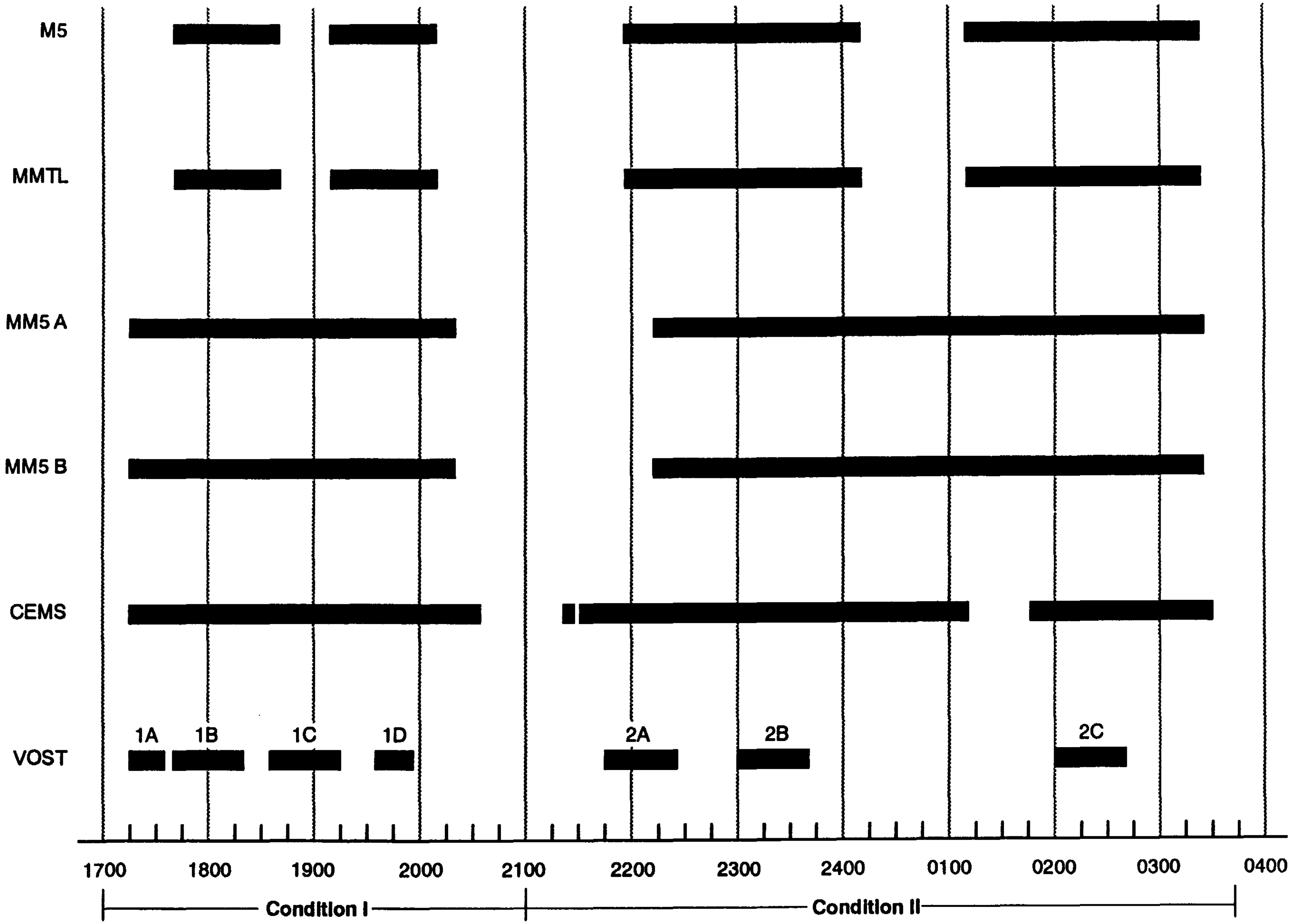
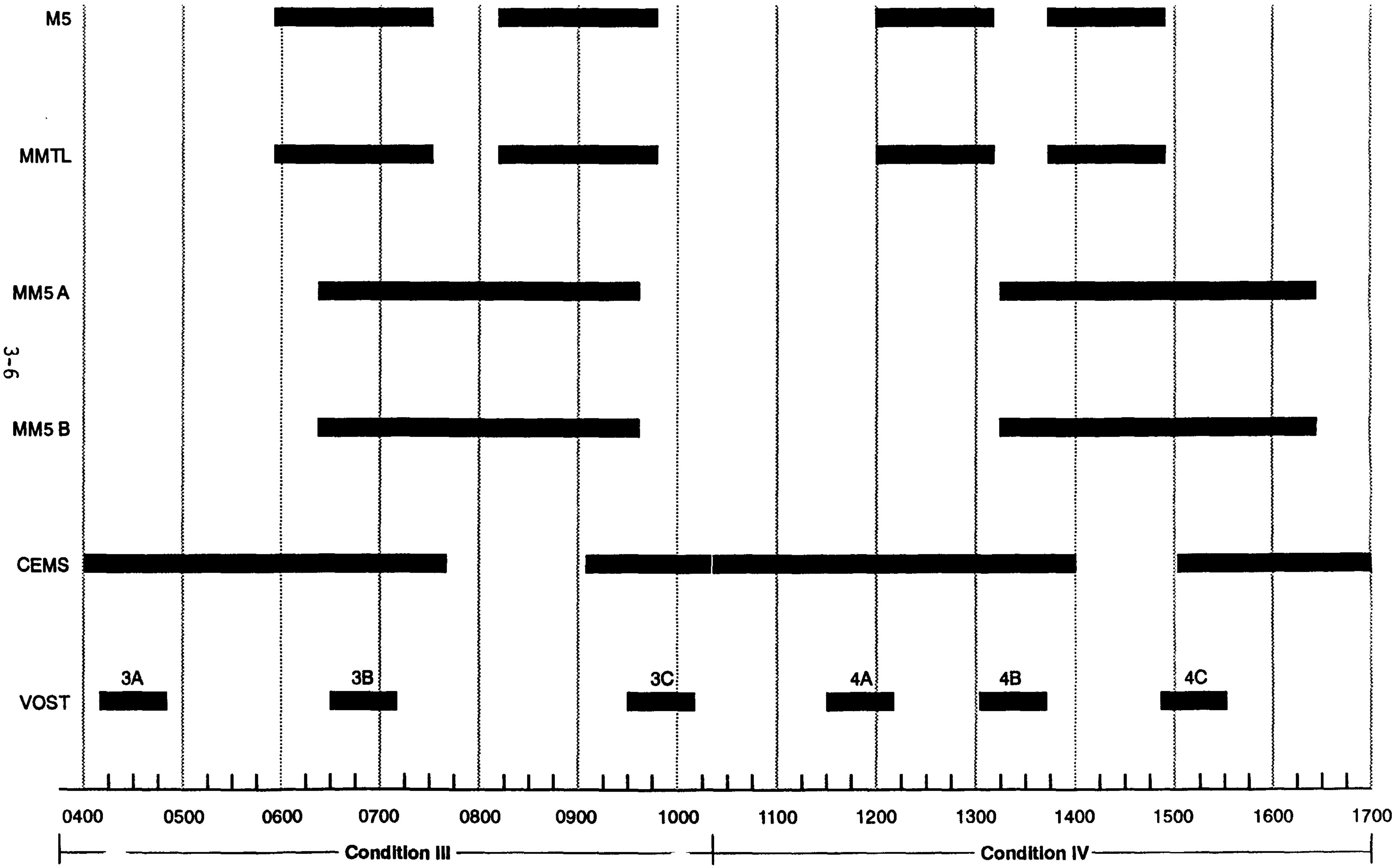


Figure 3-1. (continued)



Jewel Coal and Coke Time Log
Cycle 1, Day 1

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Figure 3-2.

Jewel Coal and Coke Time Log
Cycle 2, Day 2

Page 1 of 2

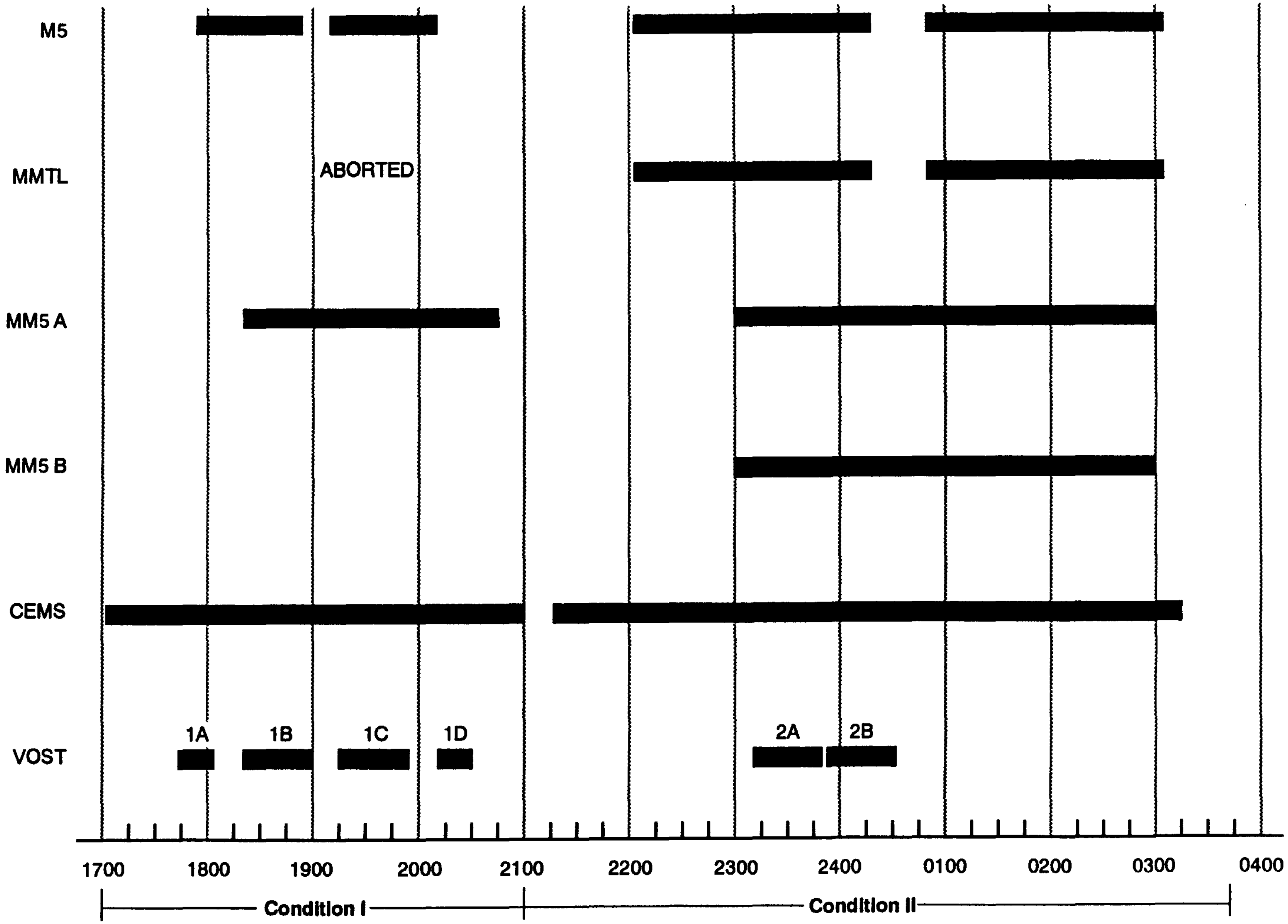

Figure 3-2. (continued)

Jewel Coal and Coke Time Log
Cycle 2, Day 2

Page 2 of 2

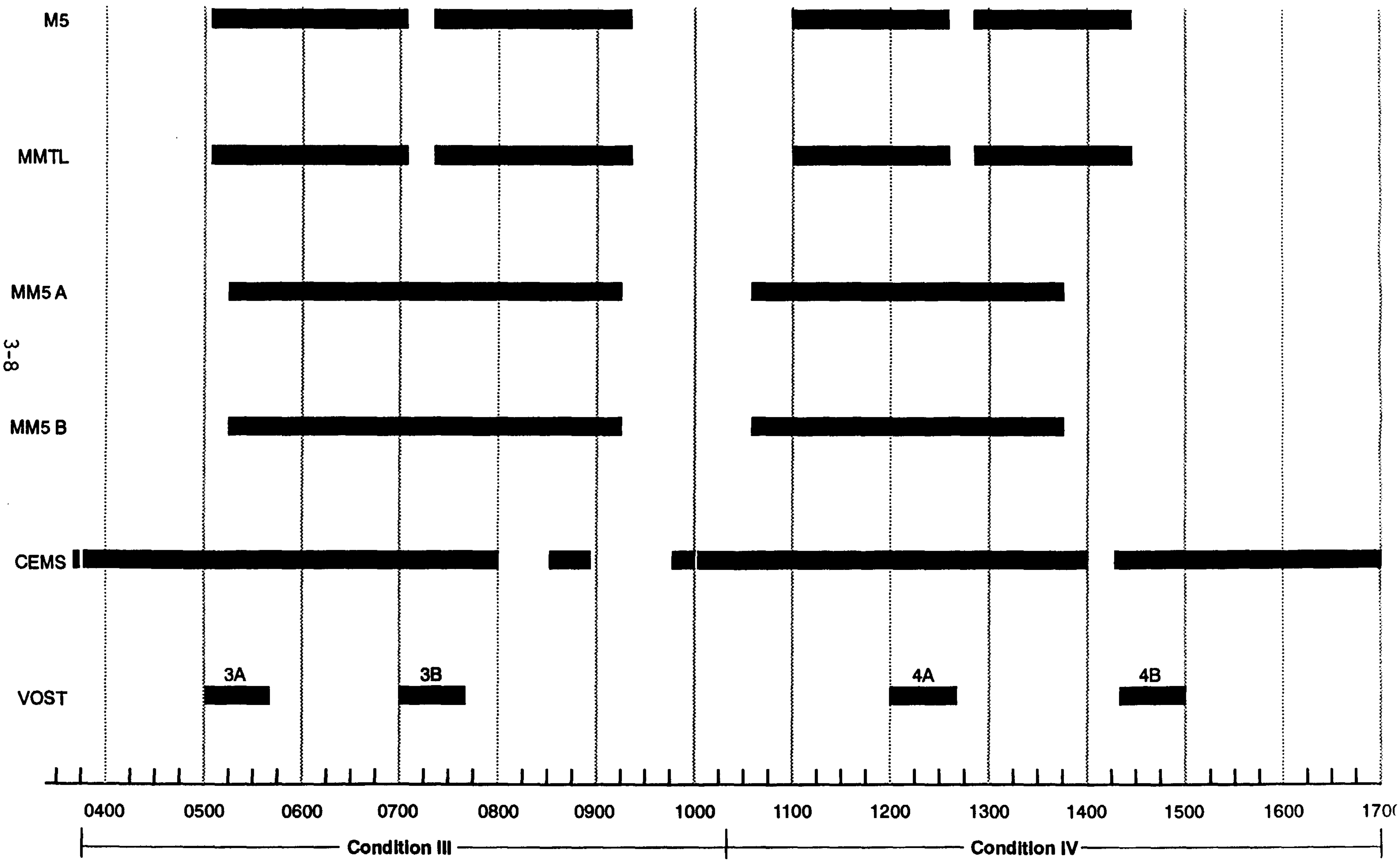

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Figure 3-3.

Jewel Coal and Coke Time Log
Cycle 3, Day 3

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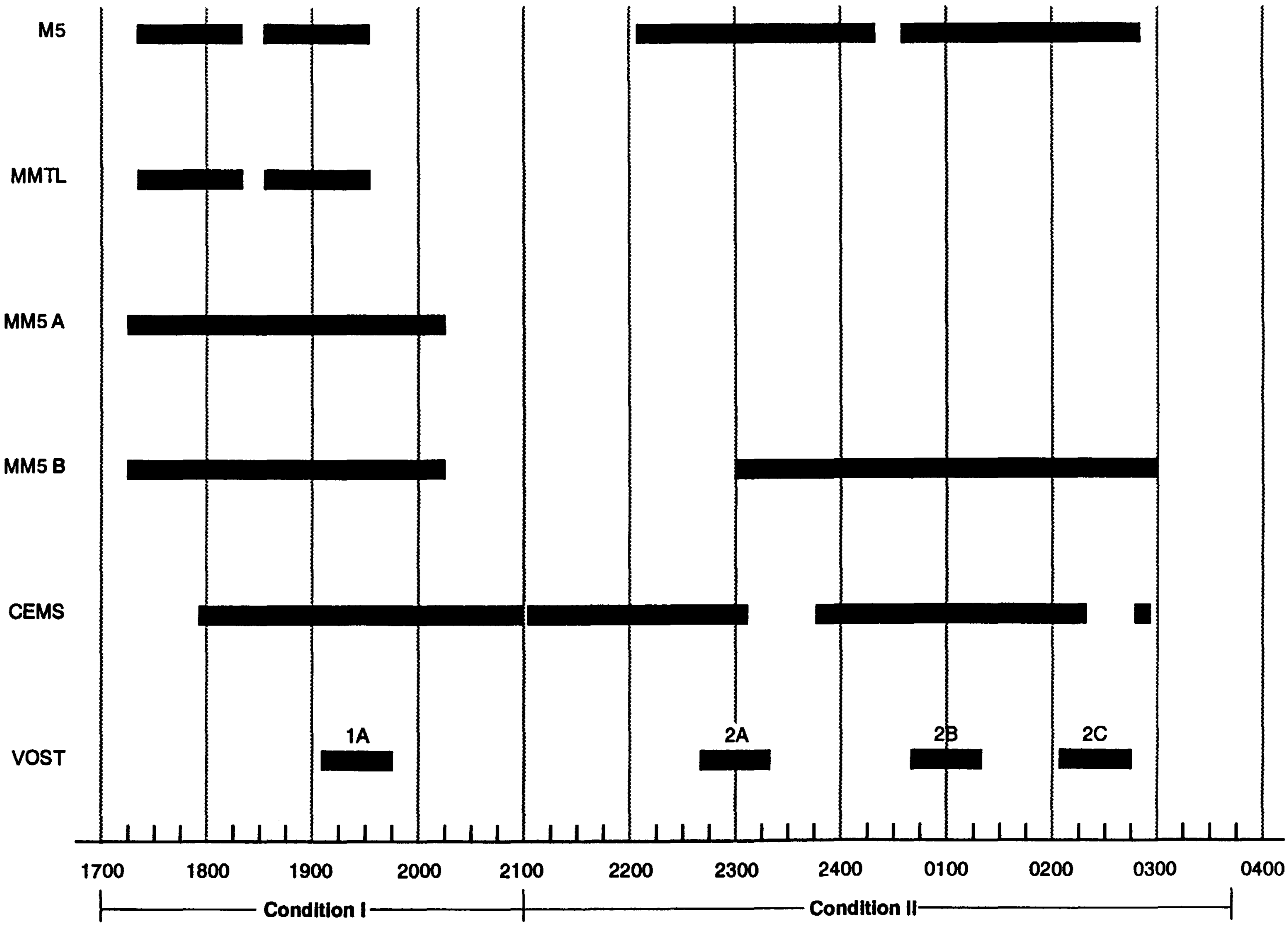
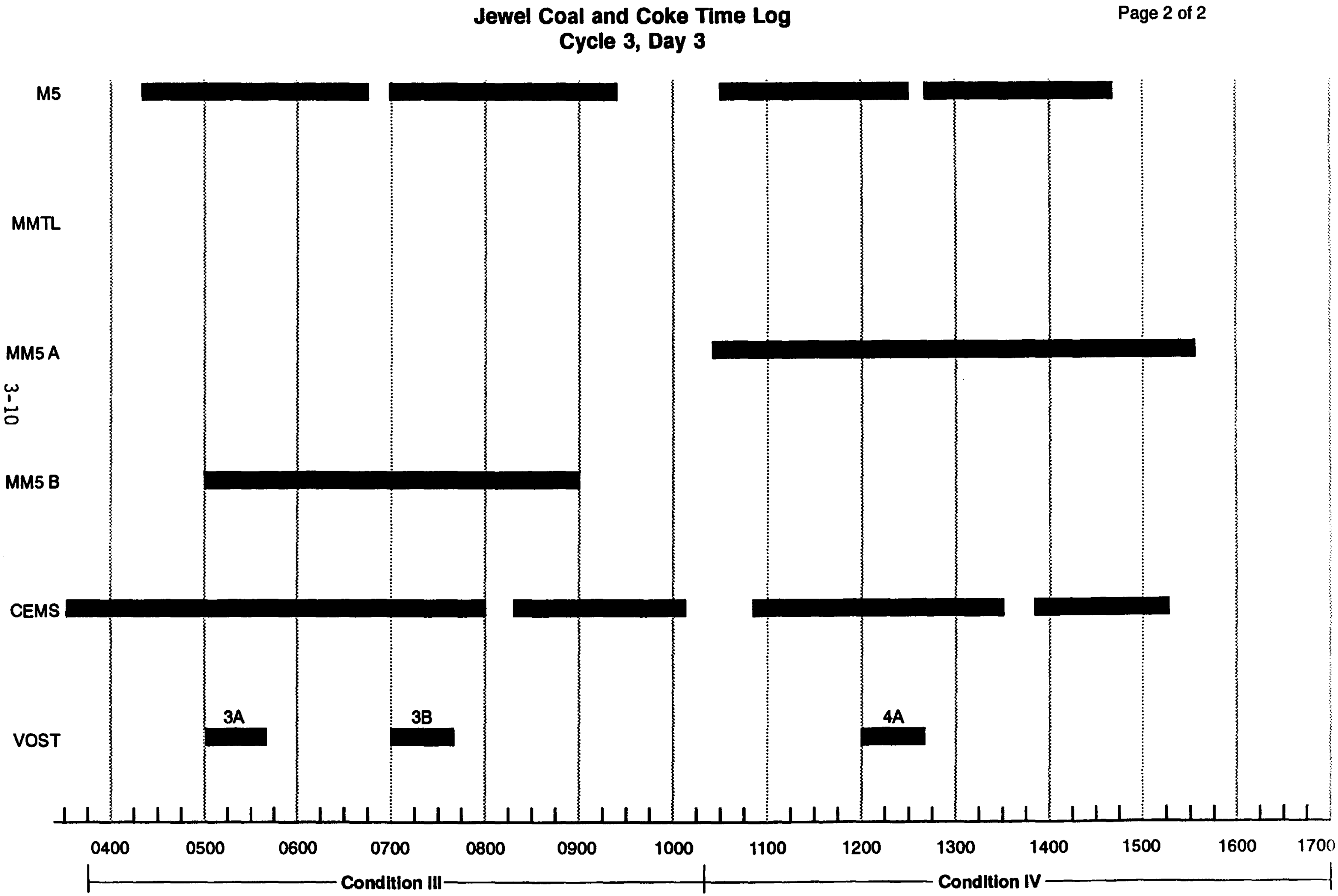


Figure 3-3. (continued)



4.0 LABORATORY VALIDATION

Entropy subcontracted Triangle Laboratories, Inc. (TLI) to evaluate the semi-volatile organic compound Method SW-846 0010 using isotope dilution for the quantification of specific analytes. The analytes targeted were toluene, xylene, phenol, cresol, naphthalene, and BaP. The experiment was designed according to the criteria of proposed Test Method 301, "Protocol for the Field Validation of Emission Concentrations from Stationary Sources." All laboratory procedures met the specifications of this method.

Proposed test Method 301 is to be used whenever a test method is proposed to meet U. S. Environmental Protection Agency requirements. This Method includes procedures for determining the bias and precision of the proposed method based on the measured concentrations.

A method's bias, or systematic error, may be determined by comparing the measured concentrations to a reference value. Significant bias may be eliminated by multiplying all measurements by a correction factor which is calculated during the validation procedure. If a proposed method has a bias correction factor outside the range 0.7 to 1.3, the method is unacceptable. The range is 0.9 to 1.1 for validated method to proposed method comparisons.

A method's precision, or random error, is determined by the percent relative standard deviation. The precision must be at most 50% relative standard deviation for the method to be acceptable. For validated method to proposed method comparison, the proposed method must be at least as precise as the validated method.

For this testing program the isotopic spiking method of validation was followed since there was no validated method for the comparison approach. The deuterated compounds ethylbenzene-d_{10}, 2-chlorophenol-d_4, acenaphthene-d_{10}, and benzo-e-pyrene-d_{12} were pre-spiked prior to field sampling. The sampling was accomplished with paired Modified Method 5 trains as specified by proposed Method 301. The bias and precision were determined according to the calculations presented in Section 4.2. The procedures and results of the field validation are presented in greater detail in Section 5.

4.1 EXPERIMENTAL DESIGN

The six target analytes were analyzed versus their respective labeled internal standard (SOLN C1); surrogate compounds (SOLN 2) were then quantitatively compared to the internal standards. For the actual field study, these surrogates served as pre-spike compounds prior to field sampling. XAD resin was also spiked with the pre-spike compound terphenyl-d14.

Laboratory samples were prepared for extraction by fortifying each fraction front-half, back-half, and aqueous impinger solution separately with deuterium-labeled compounds identical to the analytes of interest, excluding the isotopic label. For purposes of this report, analysis fractions followed the matrix employed by Entropy during field sampling and recovery, so that the "front half" fraction included the filter and probe rinse, the "back half" fraction included the XAD resin and condenser rinse, and the "aqueous impinger" fraction included the reagent and related catch recovered from the sampling train impingers. Prior to GC/MS analysis, the three extracted constituents were separately fortified with deuterium-labeled compounds as internal recovery standards (SOLN C3).

Table 4-1 lists the analytes, pre-spike compounds or surrogates, internal standards, and recovery standards. Table 4-2 presents the relationships between the analytes, internal standards, and surrogates. Using a pre-determined mass (QM) for each compound to generate selected ion current profiles (SICP) for quantification, each compound was measured against a respective standard (INTID). This comparison allowed TLI to correct for analyte or surrogate (C2) losses due to laboratory handling. In the same manner, pre-spiked terphenyl-d14 and laboratory spiked standards (C1) recovery were quantified relative to the recovery internal standards (C3).

All labeled internal standards were verified by GC/MS analysis and determined to be of 98% or greater purity.

Calibration standards were prepared by a trained TLI chemist with the assistance of a witness to verify the correct solutions and amounts of solutions, measurements, and procedures used.

GC/MS analysis was performed using a VG Trio-1 mass spectrometer with a HP 5890-II GC and a J&W DB-625 30m x 0.32 mm GC column. All GC/MS procedures followed the specifications detailed by the appropriate subsections of proposed Method 301.

Initial calibration curves generated by GC/MS analysis of calibration solutions yielded relative response factors for the analytes with a relative standard deviation (%RSD) of less than 30%. One deviation of note: because some of the analytes showed saturated mass spectra at the lower points of the calibration curve, the lower range points were excluded from the calibration curve in order to maximize sensitivity. Continuing calibration was demonstrated by analyzing the mid-point of the calibration curve and comparing the response factors to those of the initial calibration; all deviation percentages were within QC criteria for all analytes.

All laboratory spiking and extraction procedures were performed as per the appropriate section of proposed Method 301 and are shown in Scheme 1 of Appendix D. The only change from the protocol was that the final extract volume was 10 mL instead of 5 mL.

An aliquot of each sample extract was taken, and SOLN C3 was added to the aliquot to attain a final concentration of 50 ug/mL. A one-microliter aliquot of this solution was analyzed. Individual analytes and standards were identified using retention time information reference and library mass spectra databases. Quantification of the samples was accomplished by generating a selected ion current peak (SICP) for the quantitation mass (QM) and integrating this peak to find the resultant peak area. See Appendix D for conversion equation from area to amount (μg).

4.2 REQUIRED CALCULATIONS

The following calculations are required by proposed test Method 301 in order to validate a proposed method by means of isotopic spiking.

The sample mean is calculated using the following equation:

$$SM = \frac{\Sigma x_i}{n}$$

where:

SM = sample mean
x_i = ith measurement
n = sample size.

Then the bias is calculated by:

$$B = SM - CS$$

where:

B = bias
SM = sample mean
CS = amount spiked.

Next, the sample standard deviation is calculated using the following equation:

$$SD = \sqrt{\frac{\Sigma(x_i - SM)^2}{n - 1}}$$

where:

SD = sample standard deviation
x_i = ith measurement
SM = sample mean
n = sample size.

The standard deviation of the mean is calculated by:

$$SDM = \frac{SD}{\sqrt{n}}$$

where:

SDM = standard deviation of the mean

SD = sample standard deviation
n = sample size.

Then the bias of the sample is tested for significance by calculating:

$$t = \frac{|B|}{SDM}$$

and comparing this calculated t-value to the critical t-value for a two-sided test at the .05 level of significance, with n-1 degrees of freedom. If the calculated t is larger than $t_{.025}$, the bias is considered significant and a correction factor must be calculated using the following:

$$CF = \frac{1}{1 + \frac{B}{CS}} = \frac{CS}{SM}$$

where:

CF = correction factor
B = bias
CS = amount spiked
SM = sample mean

Finally, the precision, as measured by the percent relative standard deviation, is calculated by:

$$\%RSD = \frac{SD}{SM} \times 100$$

where:

%RSD = percent relative standard deviation
SD = sample standard deviation
SM = sample mean.

4.3 LABORATORY VALIDATION RESULTS

In the following subsections, laboratory validation for each fraction is addressed. For comparison, analyte and surrogate results are reported both with and without blank correction. Proposed test Method 301 specifies that 12 samples be analyzed; however, due to certain constraints, the EPA authorized the use of only three samples per fraction. The percent relative standard deviations and correction factors were calculated as illustrated in 4.2.

4.3.1 Front-Half

Without blank correction, all of the analytes and C2 surrogates passed the validation requirements. The %RSD's ranged from 1.88% to 6.43%. The analytes toluene, and benzo-a-pyrene, as well as the surrogates 2-chlorophenol-d_4 and benzo-e-pyrene-d_{12}, required calculation of correction factors of 0.81, 1.30, 1.17, and 1.15, respectively. Benzo-a-pyrene, with a correction factor of 1.30, just met the bias requirement.

With blank correction of the data, the %RSD's ranged from 1.88% to 7.25%, all meeting the requirement of less than 50%. 2-chlorophenol-d_4, benzo-e-pyrene-d_{12}, and benzo-a-pyrene required correction factors which were 1.17, 1.15, and 1.46, respectively. Thus, benzo-a-pyrene failed proposed Method 301, which specifies that correction factors fall within 0.7 to 1.30, inclusive.

Table 4-3 presents the validation parameters for the front-half fraction, and Table 4-4 presents the validation results. Both tables present the results for the analytes and the surrogates for both uncorrected and corrected data.

4.3.1.1 Conclusions

- Toluene, phenol, cresol, and naphthalene passed validation with and without blank corrections.
- Benzo-a-pyrene passed validation if data were not blank corrected; BaP failed validation if data were blank corrected.
- All surrogates passed validation for both uncorrected and corrected data.

4.3.2 Back-Half

Without blank correction, naphthalene fails validation with a %RSD of 53.56%. The other %RSD's ranged from 1.43% to 9.11%. Toluene also failed validation with a correction factor of 0.61.

With blank correction, naphthalene still fails with a %RSD of 58.86%, which is higher than when the data are uncorrected. However, toluene now meets both the precision and bias requirements.

Table 4-5 presents the validation parameters for the back-half, and Table 4-6 presents the validation results. Each table contains results for both uncorrected and corrected data.

4.3.2.1 Conclusions

- Xylene, phenol, cresol, and BaP pass validation both with and without blank correction.
- Naphthalene fails validation in the back-half both with and without blank corrections.
- Toluene fails validation if data are not blank corrected; however, it passes validation if data are blank corrected.
- All surrogates passed validation for both uncorrected and corrected data.

4.3.3 Impingers

Without blank correction, all analytes and C2 surrogates pass the %RSD requirement, with values ranging from 0.73% to 25.36%. Toluene and benzo-e-pyrene-d_{12} both failed validation with correction factors of 0.27 and 1.31, respectively.

With blank correction, toluene still fails validation with an absolute %RSD of 109.21%. The correction factor for benzo-e-pyrene-d_{12} is still 1.31 which is just outside the range specified in proposed Method 301.

Table 4-7 presents the validation parameters for the impingers, and Table 4-8 presents the validation results. Both tables present the results for uncorrected and corrected data for all analytes and surrogates.

4.3.3.1 Conclusions

- Xylene, cresol, phenol, and naphthalene passed validation.
- Toluene fails validation in the impingers.
- Ethylbenzene-d_{10}, 2-chlorophenol-d_4, and acenaphthene-d_{10} unconditionally passed validation in the impingers.
- Benzo-e-pyrene-d_{12} is borderline with a correction factor of 1.31 for both uncorrected and corrected data.

4.4 DISCUSSIONS AND CONCLUSIONS

The method was judged successful without qualification for the analytes xylene, phenol, cresol along with their corresponding surrogates ethylbenzene-d_{10} and 2-chlorophenol-d_4. However, a difference was found between benzo-a-pyrene and its corresponding surrogate benzo-e-pyrene-d_{12} in the front-half and in the impingers. In the front-half, BaP failed validation with a correction factor of 1.46 when the data were blank corrected. However, BeP-d_{12} passed validation unconditionally in the front-half. This discrepancy between BaP and its surrogate BeP-d_{12} suggests that some interference may have been occurring during the GC/MS analyses. In the impingers, benzo-e-pyrene-d_{12} failed validation completely; however, benzo-a-pyrene passed validation without qualification. BeP-d_{12} had a correction factor of 1.31 with both uncorrected and corrected data. The correction factor for BaP in both cases was 1.26. Since the two correction factors are very close to one another (a difference of 0.05) and the correction factor for BeP-d_{12} fell just outside the interval [0.70, 1.31], it appears that benzo-e-pyrene-d_{12} is an appropriate surrogate for benzo-a-pyrene and that BeP-d_{12} may be considered to have passed validation.

Toluene failed validation completely in the impingers; while naphthalene failed validation completely in the back-half. Anticipated background contamination made naphthalene and toluene analyses results unreliable and inconclusive. XAD resin inherently contains naphthalene which is difficult to completely remove. Although contamination levels usually fall below 30 micrograms, TLI has observed periods of higher levels and views this experiment as having occurred during such a period. Toluene is commonly used as an extraction solvent in the TLI laboratories and was singled out in the planning period as a possible contaminant source. Every effort was made to reduce all laboratory evaluation samples and instrumentation exposure to toluene; however, this was judged by TLI to have been only moderately successful. This contamination may explain why the surrogates ethylbenzene-d_{10} and acenaphthene-d_{10} unconditionally passed validation in all three fractions, while their corresponding analytes, toluene and naphthalene, did not completely pass validation in the laboratory.

TABLE 4-1.

SEMI-VOLATILES - COKE OVEN
ANALYTES, CORRESPONDING INTERNAL STANDARDS, AND SURROGATE COMPOUNDS

	Quantitation Mass	Identification	Internal Standard Identification
Unlabeled Analytes			
Toluene	91	AN	IS5
Xylene	91	AN	IS6
Phenol	94	AN	IS7
Cresol	108	AN	IS8
Naphthalene	128	AN	IS9
Benz-a-pyrene	252	AN	IS10
Labeled Standards			
Prespike			
Terphenyl-d_{14}	244	SUR1	IS3
Prespike Solution C2			
Ethyl benzene-d_{10}	116	SUR2	IS5
2-Chlorophenol-d_4	132	SUR3	IS7
Acenaphthene-d_{10}	164	SUR4	IS9
Benz-e-pyrene-d_{12}	264	SUR5	IS10
Internal Standards Solution C1			
Toluene-d_8	98	IS5	IS1
o-Xylene-d_{10}	116	IS6	IS1
Phenol-d_5	99	IS7	IS1
o-Cresol-d_7H	115	IS8	IS1
Naphthalene-d_8	136	IS9	IS2
Benz-a-pyrene-d_{12}	264	IS10	IS4
Recovery Standards Solution C3			
Di-chlorobenzene-d_4	152	IS1	
Phenanthrene-d_{10}	188	IS2	
Chrysene-d_{12}	240	IS3	
Perylene-d_{12}	264	IS4	

AN = Analyte
IS = Internal Standard
SUR = Surrogate

TABLE 4-2.

ANALYTES OF INTEREST WITH CORRESPONDING SURROGATES AND INTERNAL STANDARDS

ANALYTE	PRESPIKE SURROGATE	LABELED INTERNAL STANDARD
Toluene	Ethylbenzene-d10	Toluene d8
Xylene	Ethylbenzene-d10	o-Xylene d10
Phenol	2-Chlorophenol-d4	Phenol d5
Cresol	2-Chlorophenol-d4	m-Cresol d7H
Naphthalene	Acenaphthene-d10	Naphthalene d8
Benzo-a-pyrene	Benzo-e-pyrene-d12	Benzo-a-pyrene d12

TABLE 4-3.
LABORATORY VALIDATION PARAMETERS

FRONT HALF - UNCORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	97.76	99.23	101.47	99.49	-0.51	1.87	1.88	1.08	0.48		4.30
2-Chlorophenol-d4	88.18	83.87	85.35	85.80	-14.20	2.19	2.55	1.26	11.23	1.17	
Acenaphthene-d10	97.75	99.76	103.54	100.35	0.35	2.94	2.93	1.70	0.21		
Terphenyl-d14											
Benzo-e-pyrene-d12	88.55	84.73	87.79	87.02	-12.98	2.02	2.32	1.17	11.12	1.15	
Analyte											
Toluene	123.97	117.37	131.21	124.18	24.18	6.92	5.57	4.00	6.05	0.81	
Xylene	95.32	92.67	104.52	97.50	-2.50	6.22	6.38	3.59	0.70		
Phenol	89.83	90.78	99.27	93.29	-6.71	5.20	5.57	3.00	2.23		
Cresol	98.68	105.52	109.32	104.51	4.51	5.39	5.16	3.11	1.45		
Naphthalene	85.46	87.88	95.26	89.53	-10.47	5.10	5.70	2.95	3.55		
Benzo-a-pyrene	74.59	74.04	82.89	77.17	-22.83	4.96	6.43	2.86	7.97	1.30	

FRONT HALF - CORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	97.8	99.2	101.5	99.50	-0.50	1.87	1.88	1.08	0.46		4.30
2-Chlorophenol-d4	88.2	83.9	85.3	85.80	-14.20	2.19	2.56	1.27	11.21	1.17	
Acenaphthene-d10	97.7	99.8	103.5	100.33	0.33	2.94	2.93	1.70	0.20		
Terphenyl-d14											
Benzo-e-pyrene-d12	88.5	84.7	87.8	87.00	-13.00	2.02	2.32	1.17	11.13	1.15	
Analyte											
Toluene	103.1	96.5	110.3	103.30	3.30	6.90	6.68	3.98	0.83		
Xylene	95.3	92.7	104.5	97.50	-2.50	6.20	6.36	3.58	0.70		
Phenol	89.8	90.8	99.3	93.30	-6.70	5.22	5.60	3.01	2.22		
Cresol	98.7	105.5	109.3	104.50	4.50	5.37	5.14	3.10	1.45		
Naphthalene	85.5	87.9	95.3	89.57	-10.43	5.11	5.70	2.95	3.54		
Benzo-a-pyrene	66.0	65.4	74.3	68.57	-31.43	4.97	7.25	2.87	10.95	1.46	

TABLE 4-4.
LABORATORY VALIDATION RESULTS

FRONT HALF UNCORRECTED	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	5.57%	Pass	0.81	Pass
Xylene	6.38%	Pass	na	Pass
Phenol	5.57%	Pass	na	Pass
Cresol	5.16%	Pass	na	Pass
Naphthalene	5.70%	Pass	na	Pass
Benzo-a-pyrene	6.43%	Pass	1.30	Pass
SURROGATE				
Ethylbenzene-d10	1.88%	Pass	na	Pass
2-Chlorophenol-d4	2.55%	Pass	1.17	Pass
Acenaphthene-d10	2.93%	Pass	na	Pass
Benzo-e-pyrene-d12	2.32%	Pass	1.15	Pass

FRONT HALF CORRECTED	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	6.68%	Pass	na	Pass
Xylene	6.36%	Pass	na	Pass
Phenol	5.60%	Pass	na	Pass
Cresol	5.14%	Pass	na	Pass
Naphthalene	5.70%	Pass	na	Pass
Benzo-a-pyrene	7.25%	Pass	1.46	Fail
SURROGATE				
Ethylbenzene-d10	1.88%	Pass	na	Pass
2-Chlorophenol-d4	2.56%	Pass	1.17	Pass
Acenaphthene-d10	2.93%	Pass	na	Pass
Benzo-e-pyrene-d12	2.32%	Pass	1.15	Pass

na - correction factor not necessary

TABLE 4-5.
LABORATORY VALIDATION PARAMETERS

BACK HALF - UNCORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	97.47	90.77	97.91	95.38	-4.62	4.00	4.19	2.31	2.00		4.30
2-Chlorophenol-d4	84.54	82.53	84.67	83.91	-16.09	1.20	1.43	0.69	23.22	1.19	
Acenaphthene-d10	102.46	97.32	102.93	100.90	0.90	3.11	3.08	1.80	0.50		
Terphenyl-d14	60.89	60.94	65.39	62.41	-37.59	2.58	4.14	1.49	25.20	1.60	
Benzo-e-pyrene-d12	91.43	86.76	90.34	89.51	-10.49	2.44	2.73	1.41	7.44	1.12	
Analyte											
Toluene	150.72	158.86	179.54	163.04	63.04	14.86	9.11	8.58	7.35	0.61	
Xylene	101.11	97	101.9	100.00	0.00	2.63	2.63	1.52	0.00		
Phenol	96.75	93.09	96.86	95.57	-4.43	2.15	2.25	1.24	3.58		
Cresol	110.14	97.5	99.51	102.38	2.38	6.79	6.63	3.92	0.61		
Naphthalene	117.57	317.59	415.61	283.59	183.59	151.90	53.56	87.70	2.09		
Benzo-a-pyrene	80.56	77.94	81.13	79.88	-20.12	1.70	2.13	0.98	20.49	1.25	

BACK HALF - CORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	97.5	90.8	97.9	95.40	-4.60	3.99	4.18	2.30	2.00		4.30
2-Chlorophenol-d4	84.5	82.5	84.7	83.90	-16.10	1.22	1.45	0.70	22.92	1.19	
Acenaphthene-d10	102.5	97.3	102.9	100.90	0.90	3.12	3.10	1.80	0.50		
Terphenyl-d14	60.9	60.9	65.4	62.40	-37.60	2.60	4.16	1.50	25.07	1.60	
Benzo-e-pyrene-d12	91.4	86.8	90.3	89.50	-10.50	2.40	2.68	1.39	7.57	1.12	
Analyte											
Toluene	78.8	86.9	107.6	91.10	-8.90	14.85	16.30	8.57	1.04		
Xylene	101.1	97.0	101.9	100.00	0.00	2.63	2.63	1.52	0.00		
Phenol	96.8	93.1	96.9	95.60	-4.40	2.17	2.27	1.25	3.52		
Cresol	110.1	97.5	99.5	102.37	2.37	6.77	6.61	3.91	0.61		
Naphthalene	92.1	292.1	390.2	258.13	158.13	151.92	58.86	87.71	1.80		
Benzo-a-pyrene	80.6	77.9	81.1	79.87	-20.13	1.72	2.16	0.99	20.26	1.25	

TABLE 4-6.
LABORATORY VALIDATION RESULTS

BACK HALF	PRECISION		BIAS	
UNCORRECTED	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	9.11%	Pass	0.61	Fail
Xylene	2.63%	Pass	na	Pass
Phenol	2.25%	Pass	na	Pass
Cresol	6.63%	Pass	na	Pass
Naphthalene	53.56%	Fail	na	Pass
Benzo-a-pyrene	2.13%	Pass	1.25	Pass
SURROGATE				
Ethylbenzene-d10	4.19%	Pass	na	Pass
2-Chlorophenol-d4	1.43%	Pass	1.19	Pass
Acenaphthene-d10	3.08%	Pass	na	Pass
Benzo-e-pyrene-d12	2.73%	Pass	1.12	Pass

BACK HALF	PRECISION		BIAS	
CORRECTED	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	16.30%	Pass	na	Pass
Xylene	2.63%	Pass	na	Pass
Phenol	2.27%	Pass	na	Pass
Cresol	6.61%	Pass	na	Pass
Naphthalene	58.86%	Fail	na	Pass
Benzo-a-pyrene	2.16%	Pass	1.25	Pass
SURROGATE				
Ethylbenzene-d10	4.18%	Pass	na	Pass
2-Chlorophenol-d4	1.45%	Pass	1.19	Pass
Acenaphthene-d10	3.10%	Pass	na	Pass
Benzo-e-pyrene-d12	2.68%	Pass	1.12	Pass

na - correction factor not necessary

TABLE 4-7.
LABORATORY VALIDATION PARAMETERS

IMPINGERS - UNCORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	89.16	95.2	86.54	90.30	-9.70	4.44	4.92	2.56	3.78		4.30
2-Chlorophenol-d4	129.56	131.3	131.1	130.65	30.65	0.95	0.73	0.55	55.76	0.77	
Acenaphthene-d10	101.4	102.48	97.77	100.55	0.55	2.47	2.45	1.42	0.39		
Terphenyl-d14											
Benzo-e-pyrene-d12	80.57	77.13	72.04	76.58	-23.42	4.29	5.60	2.48	9.45	1.31	
Analyte											
Toluene	483.16	328.33	310.79	374.09	274.09	94.86	25.36	54.77	5.00	0.27	
Xylene	101.34	99.35	95.47	98.72	-1.28	2.99	3.02	1.72	0.74		
Phenol	99.64	97.81	96.21	97.89	-2.11	1.72	1.75	0.99	2.13		
Cresol	108.94	107.2	102.98	106.37	6.37	3.06	2.88	1.77	3.60		
Naphthalene	107.22	92.77	89.93	96.64	-3.36	9.27	9.59	5.35	0.63		
Benzo-a-pyrene	80.92	78.58	78.82	79.44	-20.56	1.29	1.62	0.74	27.66	1.26	

IMPINGERS - CORRECTED

	M1	M2	M3	Mean	Bias	SD	RSD	SDM	t-value	CF	Critical Value
Surrogate											
Ethylbenzene-d10	89.2	95.2	86.5	90.30	-9.70	4.45	4.93	2.57	3.77		4.30
2-Chlorophenol-d4	129.6	131.3	131.1	130.67	30.67	0.93	0.71	0.54	57.17	0.77	
Acenaphthene-d10	101.4	102.5	97.8	100.57	0.57	2.46	2.44	1.42	0.40		
Terphenyl-d14											
Benzo-e-pyrene-d12	80.6	77.1	72	76.57	-23.43	4.32	5.65	2.50	9.39	1.31	
Analyte											
Toluene	22.2	-132.6	-150.1	-86.83	-186.83	94.83	-109.21	54.75	3.41		
Xylene	101.3	99.4	95.5	98.73	-1.27	2.96	2.99	1.71	0.74		
Phenol	99.6	97.8	96.2	97.87	-2.13	1.70	1.74	0.98	2.17		
Cresol	108.9	107.2	103	106.37	6.37	3.04	2.86	1.75	3.63		
Naphthalene	107.2	92.8	89.9	96.63	-3.37	9.27	9.59	5.35	0.63		
Benzo-a-pyrene	80.9	78.6	78.8	79.43	-20.57	1.27	1.60	0.74	27.96	1.26	

TABLE 4-8.
LABORATORY VALIDATION RESULTS

IMPINGERS UNCORRECTED	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	25.36%	Pass	0.27	Fail
Xylene	3.02%	Pass	na	Pass
Phenol	1.75%	Pass	na	Pass
Cresol	2.88%	Pass	na	Pass
Naphthalene	9.59%	Pass	na	Pass
Benzo-a-pyrene	1.62%	Pass	1.26	Pass
SURROGATE				
Ethylbenzene-d10	4.92%	Pass	na	Pass
2-Chlorophenol-d4	0.73%	Pass	0.77	Pass
Acenaphthene-d10	2.45%	Pass	na	Pass
Benzo-e-pyrene-d12	5.60%	Pass	1.31	Fail

IMPINGERS CORRECTED	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
ANALYTE				
Toluene	-109.21%	Fail	na	Pass
Xylene	2.99%	Pass	na	Pass
Phenol	1.74%	Pass	na	Pass
Cresol	2.86%	Pass	na	Pass
Naphthalene	9.59%	Pass	na	Pass
Benzo-a-pyrene	1.60%	Pass	1.26	Pass
SURROGATE				
Ethylbenzene-d10	4.93%	Pass	na	Pass
2-Chlorophenol-d4	0.71%	Pass	0.77	Pass
Acenaphthene-d10	2.44%	Pass	na	Pass
Benzo-e-pyrene-d12	5.65%	Pass	1.31	Fail

na - correction factor not necessary

5.0 FIELD VALIDATION

Entropy subcontracted Triangle Laboratories, Inc. (TLI) to evaluate the semi-volatile organic compound Method SW-846 0010 using isotope spiking for the quantification of the analytes toluene, xylene, phenol, cresol, naphthalene, and BaP. The field experiment was designed according to the criteria of proposed Test Method 301, "Protocol for the Field Validation of Emission Concentrations from Stationary Sources." All field and laboratory procedures met the specifications of this method.

5.1 EXPERIMENTAL DESIGN

The labeled compounds (surrogates) ethylbenzene-d_{10}, 2-chlorophenol-d_4, acenaphthene-d_{10}, and benzo-e-pyrene-d_{12} were chosen to be pre-spiked prior to field sampling because they are chemically similar to the analytes of interest. The XAD resin for all samples was pre-spiked with 50 μg of each of the surrogates. The recoveries of these surrogate compounds are intended to demonstrate the ability of the entire methodology to achieve the proposed test Method 301 requirements. The surrogate compounds were introduced directly by a methylene chloride solution into the inlet end of the XAD trap. This was accomplished by means of a syringe immediately before sealing and packing the traps for shipping to the field.

Field samples were prepared for extraction by fortifying each fraction (front-half, back-half, and impingers) separately with 50 μg each of deuterium-labeled internal standards (solution C1) identical to the analytes of interest, excluding the isotopic label. The analytes were measured relative to these internal standards and any loss due to laboratory handling was corrected during the analytical calculations. Prior to GC/MS analysis, the separate fractions were fortified with deuterium-labeled compounds (solution C3) used as recovery standards. The recovery of the laboratory spiked internal standards (solution C1) are measured versus these recovery standards. Table 4-1 (Section 4.0) lists the analytes, surrogates, internal standards (solution

C1), and recovery standards (solution C3). Table 4-2 (Section 4.0) presents the relationship between the analytes of interest and their corresponding surrogates and internal standards.

GC/MS analysis was performed using a VG Trio-1 mass spectrometer with a HP 5890-II GC and a J&W DB-625 30m x 0.32 mm GC column. All GC/MS procedures followed the specifications detailed in the appropriate subsections of proposed test Method 301. The analytes were prepared in solutions ranging from 5 μg/mL to 200 μg/mL. The internal standards were injected at a constant concentration of 50 μg/mL. Two initial calibrations for the analytes were performed on two separate GC/MS instruments, F and G. All of the analytes were within QC criteria with a percent relative standard deviation (%RSD) of the response factor less than 30%. The relative difference between the means of the two initial calibrations were calculated to demonstrate the stability of the response factors. There was a difference between response factors for toluene-d_8; however, toluene-d_8 is absorbed early. Since ethylbenzene-d_{10} is measured relative to toluene-d_8, any change to the response factor for toluene-d_8 results in a change in the response factor for ethylbenzene-d_{10}. Additionally, six continuing calibrations were conducted. All response factors were within the QC criteria of ± 30% difference from the initial calibration.

5.2 PAIRED TRAINS

In order to apply EPA proposed test Method 301 procedures for validation, the amounts (μg) of spiked surrogates that were recovered were calculated based on the corrected percent recovery and the amount of labeled compounds spiked (50 μg). The bias and percent relative standard deviation requirements specified by proposed test Method 301 were determined in two ways: (1) using valid paired train data only and (2) using all valid data, excluding the field blanks. Proposed test Method 301 specifies that paired trains be used; however, since the validation calculations do not involve the use of pairs, the validation protocol was applied to all the valid data to determine if different conclusions would be reached.

There were seven pairs of data (14 measurements) for all surrogates except 2-chlorophenol-d_4, for which there were six pairs of data (12 measurements). Proposed Test Method 301 specifies that at least six pairs of data be collected. Method 301 specifies that the %RSD must be less than 50%. The %RSD's for the surrogates ranged from 5.56% to 36.86%. Therefore, the method met the precision requirement for each surrogate as specified by Method 301. Two surrogates, ethylbenzene-d_{10} and 2-chlorophenol-d_4, required bias correction factors. These correction factors were 0.798 and 0.889, respectively; therefore, the bias requirement was met by all surrogates as specified by the method. Since the spiked surrogates were chosen because they are chemically similar to the analytes of interest and since all surrogates passed validation, all of the analytes passed validation in the field. Table 5-1 presents the field validation parameters for the paired, as well as the single, data. Table 5-2 presents the validation results for both sets of data.

5.3 ALL MEASURED DATA

The same procedures were followed using all the valid data that were followed using paired train data only. The same two surrogates, ethylbenzene-d_{10} and 2-chlorophenol-d_4, were found to have significant biases with corresponding correction factors of 0.82 and 0.91, respectively, both of which fall within the specified range of 0.7 to 1.3. The %RSD's for all surrogates ranged from 6.79% to 33.26%, all less than the 50% requirement. Therefore, all analytes passed the field validation according to proposed test Method 301.

The same conclusions were reached using all the data as were reached using paired data. The precision of only two compounds were lower using the paired data; the precision of the other three compounds were higher. The biases of the compounds were essentially the same between the paired and single data.

TABLE 5-1.
FIELD VALIDATION PARAMETERS

PAIRED DATA

SURROGATE	MEAN	BIAS	SD	N	SDM	T	RSD	T.025	CF
Ethylbenzene-d10	62.68	12.68	3.48	14	0.93	13.62	5.56	2.160	0.798
2-Chlorophenol-d4	56.25	6.25	3.37	12	0.97	6.42	6.00	2.201	0.889
Acenaphthene-d10	54.75	4.75	16.04	14	4.29	1.11	29.30	2.160	na
Benzo-e-pyrene-d12	60.36	10.36	22.25	14	5.95	1.74	36.86	2.160	na

ALL DATA

SURROGATE	MEAN	BIAS	SD	N	SDM	T	RSD	T.025	CF
Ethylbenzene-d10	61.32	11.32	4.16	19	0.95	11.85	6.79	2.10	0.815
2-Chlorophenol-d4	55.21	5.21	4.24	17	1.03	5.06	7.69	2.12	0.906
Acenaphthene-d10	53.47	3.47	15.61	19	3.58	0.97	29.19	2.10	na
Benzo-e-pyrene-d12	59.17	9.17	19.68	18	4.64	1.98	33.26	2.11	na

na - correction factor not necessary

TABLE 5-2.
RESULTS OF FIELD VALIDATION FOR SURROGATE COMPOUNDS

PAIRED DATA	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
SURROGATE				
Ethylbenzene-d10	5.56%	Pass	0.798	Pass
2-Chlorophenol-d4	6.00%	Pass	0.889	Pass
Acenaphthene-d10	29.30%	Pass	na	Pass
Benzo-e-pyrene-d12	36.86%	Pass	na	Pass

ALL DATA	PRECISION		BIAS	
	%RSD	PASS/FAIL	CORRECTION FACTOR	PASS/FAIL
SURROGATE				
Ethylbenzene-d10	6.79%	Pass	0.815	Pass
2-Chlorophenol-d4	7.69%	Pass	0.906	Pass
Acenaphthene-d10	29.19%	Pass	na	Pass
Benzo-e-pyrene-d12	33.26%	Pass	na	Pass

na - correction factor not necessary

6.0 PROCESS OBSERVATIONS

6.1 OBJECTIVES

The objectives of the process observations were to document the non-recovery coking operating conditions at Jewell Coal and Coke Company during EPA's testing period and, to the extent possible, assure that the test results were representative of normal operation. The process observations were made by Emery Kong, Chemical Engineer, Center for Environmental Systems, of Research Triangle Institute (RTI).

6.2 APPROACHES AND DISCUSSIONS

The process description for Jewell's non-recovery coking process can be found in an earlier visible emissions test report[1]. This report addresses normal operation at Jewell Coal and Coke during the stack testing period.

Detailed below are the approaches which were used to achieve the objectives of the process observations.

6.2.1 Observing and Recording Pushing and Charging Operations

Process observations were conducted by process observers on both the pusher-side and coke-side of Battery C during the pushing/charging period. Half the ovens in Battery C are usually pushed and charged between 5:45 pm and 8:15 pm every day. As the daylight ends, halogen lights on the charging machine and in the coke-side shed provide light for observing the operation. The pushing/charging schedule for each oven was recorded for comparison to the emissions measured during that time period. The pushing schedule for Battery C during the test period is shown in Table 6-1.

A check list was designed and used to record work practices during the pushing and charging operations. A blank form is shown in Table 6-2. Using the information recorded, the process observers were able to determine if the work practices were consistent with the work practices observed during a previous plant visit. The actual observation sheets contain some information plant personnel considered Confidential Business Information (CBI), and, therefore, are not included in this report.

The plant operators followed the routine work practices for coke pushing, coal charging, door sill cleaning, and door sill patching. Damper controls and settings appeared normal. Emissions from pusher-side doors occurred during charging and were easily observed under halogen lights. The charging emissions stopped as soon as the oven doors were replaced after charging. The process observers did not see emissions or direct flame from the stacks during the pushing/charging period.

6.2.2 Reviewing Temperature Profile Records

Jewell Coal & Coke personnel monitored the crown temperature and two sole flue temperatures in every oven in Battery C to control the coking process. These temperatures from a representative oven (Oven 66) in Battery C were recorded every two hours in a graphic format (i.e., a temperature profile) by an operator. Temperatures in other ovens were monitored but were not recorded. These temperatures were spot-checked by the process observers and were found to be reasonably close to the target temperatures.

The graphic temperature profiles recorded for Oven 66 during the stack testing period were compared to the temperature profiles recorded for the same oven from the previous coking cycles to determine if the operation was "normal" during the entire 48-hour coking period. The temperature profiles for Oven 66 were within 10 percent of the target temperatures and were considered to be representative of normal operation. The process observer also talked to operators, who controlled dampers and monitored oven temperatures, and concluded that the coking process during non-pushing/charging period was normal.

6.2.3 Quantity of Coal Charged

The amount of coal charged into the oven affects the quantity of gases emitted, the oven temperature, and coking time. Limited by the amount of SO_2 emissions permitted, Jewell Coal & Coke normally charges 40 tons of coal to each oven for a 48-hour coking cycle. If charging is delayed for some reason, the amount of coal charged is reduced to shorten the coking time and maintain the pushing schedule for ovens.

The exact amount of coal charged to each oven in Battery C during the test period was acquired from the plant manager. Jewell considered the coal charge data CBI; therefore, they are not included in this report. Review of the coal charge data showed that ovens were charged with a regular amount of coal in the test period, except for Oven 70 which was charged with less coal on October 29, 1991 because of a delay in pushing.

6.2.4 Coal and Coke Analyses

The coal and coke properties are needed to provide a reference point for the comparison of stack emissions from the non-recovery process with emissions from by-product coke batteries. The coal analyses included fixed carbon, ash, sulfur, stability, hardness, and coke reactivity as determined by the ASTM procedures. These data were acquired from the plant manager and are presented in Table 6-3. Similar coal and coke analyses data provided by Jewell for other occasions are also presented in Table 6-3 for comparison. The comparison shows the coal feed and coke product were consistent with Jewell's normal operations.

6.3 CONCLUSIONS

Based on the process observations made during the stack testing period between October 29 and November 1, 1991, RTI concluded that the non-recovery process was operated under normal conditions except for one unusual incident during the pushing of Oven 70 on October 29, 1991. Oven 70 of Battery C was not completely coked-out at the time it was scheduled to be pushed, probably because the coal charged on October 27 had a moisture content that was higher than normal. Oven 70 was allowed to continue coking for three more hours. When this oven was pushed at

11:55 pm, the pushing emissions appeared to be much greater than the emissions from regular pushes. Oven 70 was charged with less coal on October 29, 1991 to compensate for the delayed pushing (i.e., to allow the oven to be pushed on schedule with other ovens at the end of the coking cycle).

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[1] Determination of Visible Emissions from Coke Oven Batteries, Visible Emissions Test Report (Draft), Jewell Coal and Coke Company, Vansant, Virginia, July 1991. Prepared by Radian Corporation for U. S. Environmental Protection Agency, Emissions Measurement Branch, Research Triangle Park, NC 27709. Section 3: Process Description is under review for CBI clearance.

TABLE 6-1. PUSHING SCHEDULE DURING STACK TESTING PERIOD

Oven No.	Time Pushed	Oven No.	Time Pushed	Oven No.	Time Pushed
Day 1	10/29/91	Day 2	10/30/91	Day 3	10/31/91
64	5:44 pm	63	6:19 pm	64	5:54 pm
66	5:51 pm	65	6:29 pm	66	6:02 pm
68	6:01 pm	67	6:35 pm	68	6:09 pm
70[a]	11:55 pm	69	6:42 pm	70	6:17 pm
72	6:15 pm	71	6:49 pm	72	6:24 pm
74	6:22 pm	73	6:56 pm	74	6:31 pm
76	6:35 pm	75	7:03 pm	76	6:39 pm
78	6:47 pm	77	7:11 pm	78	6:46 pm
80	6:54 pm	79	7:18 pm	80	6:54 pm
82	7:04 pm	81	7:25 pm	82	7:03 pm
84	7:12 pm	83	7:32 pm	84	7:10 pm
86	7:20 pm	85	7:39 pm	86	7:18 pm
88	7:29 pm	87	7:46 pm	88	7:25 pm
90	7:37 pm	89	7:54 pm	90	7:32 pm
92	7:45 pm	91	8:02 pm	92	7:41 pm
94	7:55 pm	93	8:10 pm	94	7:50 pm
96	8:05 pm	95	8:18 pm	96	7:58 pm
98	8:26 pm	97	8:26 pm	98	8:06 pm

[a] Pushing delayed because Oven 70 was not completely coked-out at the scheduled pushing time.

TABLE 6-2.

CHECKLIST FOR PROCESS OBSERVATIONS AT JEWELL COAL AND COKE COMPANY DURING STACK TESTING PERIOD

DATE: ____________________ OBSERVER: ____________________

BATTERY NO.: ____________________ WEATHER CONDITION: ____________________

COKE SIDE/PUSH SIDE OBSERVATION (CIRCLE ONE)
LOCATION OF OBSERVATION ____________________
SOURCE AND ADEQUACY OF LIGHT: ____________________
GENERAL COMMENTS: ____________________

OVEN NO.	STARTING TIME FOR PUSHING/CHARGING	DOOR VE (sec. if any)	STACK VE (sec. if any)	DOOR SILL CLEANING/ PATCHING	DAMPER CONTROL (% Open Init.) D1/D2/D3/D4	DAMPER ADJ. OBSERVED (% OPEN) DATE TIME
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Enter clock time in Column 2.
Enter Y for item observed or performed, N for item not observed, and NP for item not performed in Columns 3-5.

TABLE 6-3.

COAL AND COKE ANALYSIS DATA PROVIDED BY JEWELL

	AISE Paper[a]	VE Test[b]	Stack Test
A. Coal Analysis			
1. Moisture, %	10.0	10.67	10.84[c]
2. Volatile, %	23.0	23.25	23.61[c]
3. Ash, %	5.75	NA	5.49[d]
4. Sulfur, %	0.90	NA	0.85[d]
B. Coke Analysis			
1. Fixed Carbon, %	92.60	NA	92.9[d]
2. Ash, %	7.20	NA	6.86[d]
3. Sulfur, %	0.77	NA	0.73[d]
4. Stability	62.0	(62.3)	62.3[e] (61.3)[f]
5. Hardness	64.3	(66.1)	65.2[e] (64.3)[f]
6. Coke Reactivity Index (CRI)	25.6	(25.3)	(22.9)[f]
7. Coke Strength after Reaction (CSR)	63.2	(63.2)	(66.4)[f]

[a] Data obtained from a paper presented by Jerome Knoerzer of Sun Coal Co. at the AISE annual meeting held in Toronto, Canada on October 1, 1990.

[b] Coal analysis data were provided by Jewell for a visible emission (VE) test conducted by Radian Corporation for U. S. EPA between July 9 and July 13, 1991. Coke reactivity analysis was performed by UEC (USX Engineers & Consultants, Inc.) in Monroeville, PA for a composite coke sample; the results are shown in parentheses.

[c] Percent moisture and volatiles for coal were provided by Jewell for eight daily analyses between 10/26/91 and 10/31/91; their averages are presented in this table.

[d] Sulfur, ash, and fixed carbon contents were analyzed by Jewell for composite coal and coke samples taken during the stack testing period.

[e] Coke stability and hardness data were provided by Jewell for eight daily analyses between 10/28/91 and 11/2/91; their averages are presented in this table.

[f] Coke reactivity and stability data shown in parentheses were analyzed by UEC for a composite coke sample taken by Jewell during the stack test period.

7.0 PARTICULATE MATTER, TOLUENE SOLUBLE ORGANIC COMPOUNDS, AND CONDENSIBLE PARTICULATE MATTER

7.1 TEST MATRIX FOR PARTICULATE MATTER/TOLUENE SOLUBLE ORGANICS/CONDENSIBLE PARTICULATE MATTER

Emissions sampling for filterable and condensible particulate matter was conducted on Stack 1 using EPA Methods 5 and 202, respectively; both methods are considered reference methods. The sampling trains were combined for sample collection and analyses of filterable and condensible particulate matter, plus the analysis of TSO. This sampling procedure is referred to as Method 5/TSO/202. The Method 5/TSO/202 sampling used the sampling train shown in Figure 7-1. The TSO fraction is not a promulgated method. However, the modification did not affect the sample matrix; it only modified the analyses to include TSO. Complete details of the sampling and analysis procedure are contained in Appendix D.

7.2 M5/TSO/202 SAMPLING EQUIPMENT, PREPARATION, AND SAMPLE RECOVERY

7.2.1 Sampling Equipment and Preparation

All sampling glassware were cleaned prior to the testing program according to the procedures prescribed in Method 202, with the exception of the final rinse being toluene instead of methylene chloride. The Method 202 preparation was selected because it had the most stringent preparation requirements. Glass fiber filters were desiccated, tare-weighed, and stored in labeled plastic petri dishes. After assembly of the impinger train, all ambient-exposed openings were covered with Teflon™ tape.

Calibrations and leak checks of the sampling equipment (meter boxes, temperature sensors, nozzles, pitot tubes, and umbilicals) were performed according to the requirements specified in EPA's "Quality Assurance Handbook, Volume III" (600/4-77-027b). The results were documented.

The impinger trains were assembled in the sample recovery area as prescribed in the method. Tare-weighed filters were loaded into the filter holders and the sampling train was leak checked. All openings on the probe assembly and the impinger train were capped for transfer to the sampling locations.

The sampling trains were operated according to Method 202 (based on EPA Method 5 isokinetic sampling procedures). All M5/TSO/202 sampling was conducted simultaneously with CEM, VOST, MM5, and MMTL, and had a total run time of approximately two hours for Condition I, and four hours for Conditions II, III, and IV. The Method 202 optional nitrogen purge was conducted to remove dissolved SO_2 gas from the impinger contents because SO_2 was present.

Following each sampling run, the probes were removed from the sampling trains and brushed out into the appropriate container, the openings were capped, and the impinger trains were returned to the sample recovery area.

During the second run of Condition II, a M5/TSO/202 field blank train was assembled with glassware previously used for M5/TSO/202 sampling. This train was charged exactly as the other Method 5/TSO/202 trains and left at a sampling location during the test run. The field blank train was recovered with the other M5/TSO/202 trains for that run. The purpose of the field blank train was to measure the level of contamination that occurred from handling, charging, recovering, and transporting the sampling train.

7.2.2 Sample Recovery

Once in the recovery area, the M5/TSO/202 impinger trains, including the field blank train, were recovered according to the method specifications. The sample recovery scheme for M5/TSO/202 is shown in Figure 7-2. Liquid samples were stored in pre-cleaned glass sample jars with Teflon™ lid liners. Filters were placed in pre-cleaned glass sample jars.

Reagent blank samples of deionized water, acetone, and toluene used in sample recovery were collected. The liquid level and sample identification number were marked on each sample container and stored in locked boxes for return to Entropy's laboratory as prescribed by Entropy's chain-of-custody procedures.

7.3 M5/TSO/202 ANALYSIS

The particulate and condensible samples were analyzed by Entropy; the TSO samples were analyzed by TLI.

Filter samples and acetone dry down samples were analyzed gravimetrically according to EPA Method 5 (40 CFR 60). For Method 202 analysis, an aliquot of each aqueous back half sample was taken to be analyzed by ion chromatography (IC) for $SO_4^=$ and Cl^-. The remainder of each sample was extracted with the toluene rinse and two additional aliquots of fresh toluene. The toluene organic phase was evaporated, desiccated, and weighed to a constant weight. The aqueous phase was evaporated to dryness at 105°C to remove any hydrogen chloride (HCl) and then redissolved in DI water. Ammonium hydroxide was added to stabilize any sulfuric acid and the excess was evaporated during a second drying step. Following desiccation, the aqueous phase or inorganic condensible fraction was weighed to a constant weight. The weighed residue was redissolved and an aliquot taken for chloride analysis by IC to determine NH_4Cl. The amount of ammonium ion associated with the sulfate ion as determined by the first IC analysis, and the amount of chloride ion associated with ammonium ion, determined by the final IC analysis, were subtracted from the inorganic condensible particulate matter weight.

The TSO analysis consisted of a toluene extraction on the M5 front half residues from the particle determination, evaporating these extracts and weighing the TSO difference.

7.4 M5/TSO/202 QC PROCEDURES

Quality control procedures for Method 5/TSO/202 were followed and documented to ensure high quality flue gas concentrations and emissions data. Flue gas concentrations were determined by dividing the mass of analyte (particulate matter, TSO, condensible particulate matter) collected by the standardized volume of gas sampled. Sampling QC procedures ensured that a representative amount of the analytes were collected by the sampling system. The following procedures were followed during the testing program:

- The sampling rate was within 10 percent of isokinetic (100 percent).
- The probe and filter temperatures were maintained at 248°F ± 25°F.
- Only properly prepared glassware was used.
- All sampling nozzles were manufactured and calibrated according to EPA standards.
- Filters were weighed, handled, and stored in a manner to prevent any contamination.
- Recovery procedures were completed in a clean environment.
- Field reagent blanks were collected.

Accurate standardized sample volume determinations were ensured by following the respective QC procedures.

7.5 QA/QC CHECKS FOR DATA REDUCTION, VALIDATION, AND REPORTING

Data quality audits were conducted using data quality indicators which required the detailed review of: (1) the recording and transfer of raw data, (2) data calculations, (3) the documentation of procedures, and (4) the selection of appropriate data quality indicators.

Some of the data quality indicators used for data validation were:

1. Comparison of the process and control system instrumentation data to the manual methods readings;
2. Comparison of the relative concentrations of the emissions at the different sampling locations; and
3. Comparison of the results to previous field test results (i.e., are there any similarities).

All data and calculations for flow rates, moisture content, and isokinetic rates made by a computer software program were validated by an independent check. All calculations were spot checked for accuracy and completeness.

In general, all measurement data were validated based on the following criteria:

- process conditions during sampling or testing,
- acceptable sample collection procedures,
- consistency with expected other results, and
- adherence to prescribed QC procedures.

Any suspect data were flagged and identified with respect to the nature of the problem and potential effect on the data quality. After the testing was completed, the field coordinator was responsible for the preparation of a data summary including calculation results and raw data sheets. The QA/QC check for data reduction, validation, and reporting were performed for all data.

The outside laboratory reported its QA/QC results in the analytical report. All QA/QC results were reviewed internally by Entropy before the data were released; any data that failed to meet the program's QA requirements were noted.

7.6 M5/TSO/202 SUMMARY OF RESULTS

The filterable and condensible particulate matter results have been calculated and presented using two approaches. The first approach presents data as calculated weighted averages per cycle from all four stacks. These data are presented in Table 7-1. The second approach presents data as measured in Stack 1, per condition, with Condition I as the Charging, Condition II as the beginning of the cycle (immediately after charging was complete), Condition III as the middle of the cycle, and Condition IV as the end of the cycle. These data are presented in Tables 7-2a through 7-2d, respectively.

It was important to present the data using two approaches because of the sampling technique and assumptions made in designing the testing program. The total weighted averages include a summation of data collected from Stacks 1, 2, 3, and 4. These data include concentration data from Stack 1, Δp and stack temperatures from Stacks 1 through 4, O_2 and CO_2 from Stacks 1, 2, and 4, and moisture from Stacks 1 and 2. In order to calculate a total weighted average, data were needed from each condition during a 24-hour cycle. There were incidences that caused either a voided or aborted run, which left a "hole" in the data. Values were calculated based on the averages from the other runs in that condition. For example, inconel nozzles were originally selected to be used for this sampling train. During the testing program, it was noticed that the color of the filters was very dark compared to the MMTL train which was sampled with a quartz nozzle simultaneously with the M5/TSO/202 train; the MMTL filters did not indicate a color "problem." This led to the conclusion that the inconel nozzles could be contributing to the mass of the sample and those results were not used in this report. Thus, Condition I, Run 1, was voided because of possible contamination to the mass loading due to metal findings. As a result, for Condition I, Run 1, Table 7-2a presents the train data which were unaffected by the choice of nozzle but uses the average concentration from Runs 2 and 3 of the same condition. This average is indicated by "d" superscript next to the value. The emission rate was calculated from the average concentration shown on the table divided by the flow rate from that sampling train. These data are indicated by "e" superscript next to that value. Tables 7-2b and 7-2c present similar occurrences. The data were so consistent from run to run and condition to condition that this seems to be a reasonable assumption. The daily concentrations and emission rates were used to calculate the total weighted averages per cycle.

Concentrations from Stack 1 were assumed to be consistent in each of Stacks 2, 3, and 4 and an emission rate was calculated from flow rate data collected from the respective stacks during each run. These data have been weighted to account for the charging cycle that was approximately 3 hours in duration. Example calculations are presented in Appendix A. Tables 7-2a through 7-2d present the filterable and condensible particulate matter as measured in Stack 1, Conditions I through IV, respectively.

Figures 7-3 and 7-4 present the measured filterable particulate matter in units of g/mg coke. Figure 7-3 is a comparison of the three testing days per condition; Figure 7-4 is a comparison of the four conditions per day. Figures 7-5 and 7-6 present the measured condensible particulate matter in units of g/mg coke. Figure 7-5 is a comparison of the three days per condition and Figure 7-6 is a comparison of the four conditions per day.

In reviewing the data, there appears to be no significant increase or decrease in the amount of emissions from each of the Conditions. Hence, it is not significant to weigh the emissions; arithmetic averages would be sufficient.

Table 7-3 presents the TSO data in several different ways. The front-half (FH) catch weight was determined by the front-half rinses and filter. The back-half (BH) or impinger catch weight represents the organic part of the condensible particulate matter; therefore, the front-half and the back-half together represent the total organic catch per run. This total organic catch is presented in two ways: (1) uncorrected (as measured) and (2) corrected for the field blank catch (1.8 mg). The total emission rates (lb/hr) for the entire battery and the total mass emission rates (g/mg coke) are also presented in Table 7-3. These emission rates were calculated using the uncorrected and the corrected catch weights. Figures 7-7 and 7-8 present the toluene soluble organics in units of g/mg coke. Figure 7-7 is a comparison of the three days per condition; Figure 7-8 is a comparison of the four conditions per day. Figure 7-9 is a comparison of total particulate matter, condensible particulate matter, and TSO per condition.

Very little TSO was found in either the front-half or the back-half, except for Run 1, Condition I. For this run, the back-half catch of 13.6 mg appears to be an outlier. Therefore, the average for Condition I was calculated with and without this catch weight included. For the other runs, the TSO catch was comparable to the field blank catch and, when blank corrected, is close to the constant weight criteria of 0.5 mg. It should be noted that only one field blank was recovered and analyzed for this testing program.

These TSO data should not be compared to previous data reported for BSO (benzene soluble organics) for two main reasons.

First, it has not been documented that toluene and benzene extract with the same efficiency. Second, the sampling method for this TSO test is completely different from the sampling method for the previous BSO testing. The BSO analysis involved a gravimetric procedure used on personal exposure monitors; the TSO analysis involved a gravemetric procedure used on samples collected isokinetically from the stack.

7.7 M5/TSO/202 DATA QUALIFIERS

This section provides a list of data qualifiers. As previously noted, some runs were voided or aborted. These included:

1. Day 1, Condition I used an inconel nozzle. All data were considered void.
2. Day 1, Condition II used an inconel nozzle. All data were considered void.
3. Day 1, Condition III used an inconel nozzle. All data were considered void.
4. Emission rates from Day 1, Conditions I, II, and III were based on the calculated values from the valid runs of the same condition.
5. The total weighted average was calculated using all four conditions, including the calculated values of Day 1, Conditions I, II, and III.
6. Orsat data from Stack 1, Day 1, Condition III were invalid; Day 1, Condition III data from Stack 2 were used. If the average orsat data from Stack 1, Days 2 and 3, Condition III had been used, such that all the data were from the same stack, the emission rates for filterable particulate matter, condensible particulate matter, and TSO would have decreased by only 0.20 %.
7. The TSO check weights from Day 1, Condition IV exceeded the criteria of 0.5 for a constant weight of 0.6. However, the final weighing missed the criteria by only 0.1 mg and, therefore, no loss of data quality has occurred.
8. Tables 7-3 through 7-5 present the flow rates, stack temperatures, moisture, and gas constituent data used in the calculations for stack and condition for Days 1, 2, and 3, respectively.

TABLE 7-1.

FILTERABLE AND CONDENSIBLE PARTICULATE MATTER TOTAL WEIGHTED AVERAGES PER CYCLE JEWELL COAL & COKE COMPANY

Day/Repetition	1	2	3	Average
Filterable Particulate Matter:				
Concentration, gr/DSCF[a]	0.0468	0.0460	0.0438	0.0455
Emission Rate, lb/hr	29.22	29.27	29.60	29.36
Emission Rate, lb/ton charged	0.9764	0.9757	0.9908	0.9810
Condensible Particulate Matter:				
Concentration, gr/DSCF[a]	0.0082	0.0079	0.0044	0.0068
Emission Rate, lb/hr	5.27	5.15	3.0086	4.48
Emission Rate, lb/ton charged	0.1764	0.1717	0.1007	0.1496

[a] 68°F (20°C)--29.92 inches of Mercury (Hg)

TABLE 7-2a.

FILTERABLE AND CONDENSIBLE PARTICULATE MATTER AS MEASURED IN STACK 1, CONDITION I - CHARGING JEWELL COAL & COKE COMPANY

Run Identification	I-S1-M5/TSO/202			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	1741	1752	1719	
Run Finish Time[a]	2009	2010	1932	
Total Run Time, min.	120	120	120	
Test Train Parameters:				
Volume of Metered Dry Gas Sample, SCF[b]	65.933	62.981	59.480	
Percent Isokinetic	95.0	96.6	96.5	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[c]	6.0	6.4	6.5	
O_2, Percent by Volume, Dry[c]	11.2	11.2	10.6	
Flue Gas Temperature, Degrees F	1,581	1,561	1,506	
Velocity, ft/sec	32.13	31.21	28.16	
Air Flow Rate, Dry, SCFM[b]	22,137	21,310	20,142	
Air Flow Rate, Wet, ACFM	103,053	100,102	90,320	
Filterable Particulate Matter:				
Concentration, gr/DSCF[b]	0.0435[d]	0.0444	0.0426	0.0435
Emission Rate, lb/hr	8.25[e]	8.11	7.36	7.91
Condensible Particulate Matter:				
Concentration, gr/DSCF[b]	0.00975[d]	0.0107	0.0088	0.00975
Emission Rate, lb/hr	1.85[e]	1.95	1.52	1.77

---

[a] Inclusive of Port Changes

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

[c] Orsat Analyses From Stack 1, M5/TSO/202 Train

[d] Average of Runs 2 and 3

[e] Calculated using the average concentration (d).

TABLE 7-2b.

FILTERABLE AND CONDENSIBLE PARTICULATE MATTER
AS MEASURED IN STACK 1, CONDITION II
JEWELL COAL & COKE COMPANY

Run Identification	II-S1-M5/TSO/202			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	2157	2202	2205	
Run Finish Time[a]	0323	0305	0249	
Total Run Time, min.	270	270	270	
Test Train Parameters:				
Volume of Metered Dry Gas Sample, SCF[b]	130.266	124.663	142.622	
Percent Isokinetic	94.7	95.7	99.9	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[c]	6.3	7.8	7.9	
O_2, Percent by Volume, Dry[c]	10.7	9.0	8.9	
Flue Gas Temperature, Degrees F	1,549	1,473	1,483	
Velocity, ft/sec	26.66	26.21	29.41	
Air Flow Rate, Dry, SCFM[b]	18,835	18,911	20,739	
Air Flow Rate, Wet, ACFM	85,509	84,065	94,329	
Filterable Particulate Matter:				
Concentration, gr/DSCF[b]	0.0377[d]	0.0287	0.0466	0.0377
Emission Rate, lb/hr	6.09[e]	4.66	8.29	6.35
Condensible Particulate Matter:				
Concentration, gr/DSCF[b]	0.00957[d]	0.0140	0.00513	0.00957
Emission Rate, lb/hr	1.545[e]	2.26	0.912	1.57

---

[a] Inclusive of Port Changes

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

[c] Orsat Analyses From Stack 1, M5/TSO/202 Train

[d] Average of Runs 2 and 3

[e] Calculated using the average concentration (d).

TABLE 7-2c.

FILTERABLE AND CONDENSIBLE PARTICULATE MATTER AS MEASURED IN STACK 1, CONDITION III JEWELL COAL & COKE COMPANY

Run Identification	III-S1-M5/TSO/202			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	0555	0505	0421	
Run Finish Time[a]	0948	0923	0919	
Total Run Time, min.	192	240	288	
Test Train Parameters:				
Volume of Metered Dry Gas Sample, SCF[b]	90.849	103.188	112.135	
Percent Isokinetic	92.2	93.5	95.3	
Flue Gas Parameters:				
CO$_2$, Percent by Volume, Dry[c]	5.7	7.4	7.2	
O$_2$, Percent by Volume, Dry[c]	11.8	9.0	9.5	
Flue Gas Temperature, Degrees F	1,503	1,492	1,460	
Velocity, ft/sec	26.13	24.97	21.77	
Air Flow Rate, Dry, SCFM[b]	18,959	18,031	16,019	
Air Flow Rate, Wet, ACFM	83,809	80,088	69,824	
Filterable Particulate Matter:				
Concentration, gr/DSCF[b]	0.0492[d]	0.0541	0.0442	0.0492
Emission Rate, lb/hr	7.995[e]	8.36	6.07	7.41
Condensible Particulate Matter:				
Concentration, gr/DSCF[b]	0.004055[d]	0.00408	0.00403	0.004055
Emission Rate, lb/hr	0.659[e]	0.631	0.554	0.615

---

[a] Inclusive of Port Changes

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

[c] Orsat Analyses From Stack 1, M5/TSO/202 Train

[d] Average of Runs 2 and 3

[e] Calculated using the average concentration (d).

TABLE 7-2d.

FILTERABLE AND CONDENSIBLE PARTICULATE MATTER AS MEASURED IN STACK 1, CONDITION IV JEWELL COAL & COKE COMPANY

Run Identification	IV-S1-M5/TSO/202			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	1200	1100	1030	
Run Finish Time[a]	1456	1428	1440	
Total Run Time, min.	144	192	240	
Test Train Parameters:				
Volume of Metered Dry Gas Sample, SCF[b]	56.173	79.566	103.516	
Percent Isokinetic	91.5	95.2	95.2	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[c]	7.1	7.9	7.5	
O_2, Percent by Volume, Dry[c]	9.5	8.2	8.5	
Flue Gas Temperature, Degrees F	1,520	1,511	1,508	
Velocity, ft/sec	22.79	23.57	24.55	
Air Flow Rate, Dry, SCFM[b]	16,714	17,070	17,766	
Air Flow Rate, Wet, ACFM	73,096	75,598	78,741	
Filterable Particulate Matter:				
Concentration, gr/DSCF[b]	0.0547	0.0556	0.0411	0.0505
Emission Rate, lb/hr	7.84	8.13	6.25	7.41
Condensible Particulate Matter:				
Concentration, gr/DSCF[b]	0.0105	0.00465	0.00262	0.0059
Emission Rate, lb/hr	1.50	0.681	0.400	0.8603

---

[a] Inclusive of Port Changes

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

[c] Orsat Analyses From Stack 1, M5/TSO/202 Train

TABLE 7-3a.

TSO CATCH WEIGHTS
AS MEASURED AND BLANK CORRECTED

Run Number	FH (mg)	FH blank (mg)	FH blank corrected	BH (mg)	BH Blank (mg)	BH blank corrected	FH + BH (mg)	FH + BH blank corrected (mg)
I-1	1.09	0.94	0.15	13.6	1.8	11.8	14.69	11.95
I-2	0.67	0.94	0.00	2.5	1.8	0.70	3.17	0.70
I-3	0.87	0.94	0.00	2.9	1.8	1.10	3.77	1.10
Average	0.88		0.05	6.3		4.53	7.21	4.58
Average I-1 omitted	0.77		0.00	2.7		0.90	3.47	0.90
II-1	0.99	0.94	0.05	7.0	1.8	5.20	7.99	5.25
II-2	0.15	0.94	0.00	3.0	1.8	1.20	3.15	1.20
II-3	0.40	0.94	0.00	3.1	1.8	1.30	3.50	1.30
Average	0.51		0.02	4.4		2.57	4.88	2.58
III-1	2.05	0.94	1.11	3.3	1.8	1.50	5.35	2.61
III-2	0.85	0.94	0.00	1.0	1.8	0.00	1.85	0.00
III-3	3.33	0.94	2.39	2.3	1.8	0.50	5.63	2.89
Average	2.08		1.17	2.2		0.67	4.28	1.83
IV-1	2.61	0.94	1.67	2.4	1.8	0.60	5.01	2.27
IV-2	3.84	0.94	2.90	3.1	1.8	1.30	6.94	4.20
IV-3	1.87	0.94	0.93	1.3	1.8	0.00	3.17	0.93
Average	2.77		1.83	2.3		0.63	5.04	2.78

TABLE 7-3b.

TSO EMISSION RATES UNCORRECTED AND CORRECTED

Run Number	Total Emission Rate uncorrected (lb/hr)	Total Mass Emission uncorrected (g/mg coke)	Total Emission Rate corrected (lb/hr)	Total Mass Emission corrected (g/mg coke)
I-1	2.612	62.1	2.12	50.5
I-2	0.572	13.5	0.13	2.98
I-3	0.680	16.1	0.20	4.69
Average	1.29	30.6	0.82	19.4
Average I-1 omitted	0.628	14.8	0.17	3.84
II-1	0.612	14.5	0.40	9.56
II-2	0.249	6.02	0.10	2.29
II-3	0.269	6.40	0.10	2.38
Average	0.377	8.97	0.20	4.74
III-1	0.596	14.01	0.29	6.86
III-2	0.166	4.07	0.00	0.00
III-3	0.424	10.13	0.22	3.29
Average	0.396	9.40	0.17	3.38
IV-1	0.788	18.70	0.36	8.50
IV-2	0.784	18.74	0.48	11.3
IV-3	0.290	6.85	0.08	2.01
Average	0.620	14.8	0.31	7.27

TABLE 7-4.

SUMMARY DATA USED IN CALCULATIONS
JEWELL COAL AND COKE COMPANY

DAY 1

DAY 1	STACK 1			STACK 2			STACK 3	STACK 4
	MMTL	M5	AVERAGE	MM5-A	MM5-B	AVERAGE	VOST	CEM
CONDITION I								
% O_2	11.2[1]	11.2[1]		11.2	11.2		11.2[3]	11.3
% CO_2	6.0[1]	6.0[1]		6.0	6.0		6.1[3]	6.4
% H_2O	14.9	14.3		14.5	14.3		14.5[4]	14.5[4]
Temperature, °F	1,583	1,581		1,564	1,564		1,519	1,466
Flow Rate, DSCFM	22,494	22,137	22,316	21,421	21,461	21,441	22,623	22,027[5]
CONDITION II								
%O_2	10.7	10.7		7.3	7.3		9.0[3]	9.1
%CO_2	6.3	6.3		8.9	8.9		7.7[3]	7.8
%H_2O	13.6	13.5		17.0	15.1		14.8[4]	14.8[4]
Temperature, °F	1,550	1,549		1,558	1,558		1,507	1,500
Flow Rate, DSCFM	18,879	18,835	18,857	18,954	19,302	19,128	20,394	19,273[5]
CONDITION III								
%O_2	11.8[1]	11.8[1]		11.8	11.8		10.6[3]	8.3
%CO_2	5.7[1]	5.7[1]		5.7	5.7		6.5[3]	8.2
%H_2O	13.0	13.2		14.6	16.0		14.2[4]	14.2[4]
Temperature,°F	1,501	1,503		1,533	1,533		1,504	1,489
Flow Rate, DSCFM	18,529	18,959	18,744	16,752	16,523	16,638	14,474	17,047[5]
CONDITION IV								
%O_2	9.5	9.5		9.5[2]	9.5[2]		9.2[3]	8.6
%CO_2	7.1	7.1		7.1	7.1		7.3[3]	7.8
%H_2O	13.6	11.5		12.6	12.7		12.6[4]	12.6[4]
Temperature, °F	1,525	1,520		1,571	1,570		1,541	1,518
Flow Rate, DSCFM	15,515	16,714	16,115	17,614	17,609	17,612	18,589	17,208[5]

[1] Orsat invalid; used Stack 2 data
[2] Orsat data invalid; used Stack 1 data
[3] Average of Stacks 1, 2, and 4
[4] Average of Stacks 1 and 2
[5] Average of Stacks 1, 2, and 3

TABLE 7-5.

SUMMARY DATA USED IN CALCULATIONS
JEWELL COAL AND COKE COMPANY

DAY 2

DAY 2	STACK 1			STACK 2			STACK 3	STACK 4
	MMTL	M5	AVERAGE	MM5-A	MM5-B	AVERAGE	VOST	CEM
CONDITION I								
% O_2		11.2		9.5			10.1[2]	9.6
% CO_2		6.4		7.3			7.0[2]	7.4
% H_2O		15.9		17.7			16.8[3]	16.8[3]
Temperature, °F		1,561		1,566			1,555	1,521
Flow Rate, DSCFM		21,310	21,310	22,742		22,742	20,987	21,680[4]
CONDITION II								
%O_2	9.0	9.0		8.9	8.9		8.9[2]	8.9
%CO_2	7.8	7.8		8.1	8.1		7.9[2]	7.8
%H_2O	12.7	15.0		16.7	16.8		15.3[3]	15.3[3]
Temperature, °F	1,461	1,473		1,559	1,559		1,545	1,546
Flow Rate, DSCFM	19,055	18,911	18,983	19,727	19,689	19,708	19,231	19,323[4]
CONDITION III								
%O_2	9.0	9.0		9.0[1]	9.0[1]		9.0[2]	8.9
%CO_2	7.4	7.4		7.4[1]	7.4[1]		7.5[2]	7.7
%H_2O	13.0	13.5		14.8	15.2		14.1[3]	14.1[3]
Temperature, °F	1,491	1,492		1,546	1,545		1,520	1,519
Flow Rate, DSCFM	18,336	18,031	18,184	19,182	19,149	19,166	14,043	17,748[4]
CONDITION IV								
%O_2	8.2	8.2		8.2[1]	8.2[1]		8.6[2]	9.3
%CO_2	7.9	7.9		7.9[1]	7.9[1]		7.6[2]	7.0
%H_2O	13.0	12.4		13.6	13.8		13.2[3]	13.2[3]
Temperature, °F	1,510	1,511		1,551	1,557		1,498	1,503
Flow Rate, DSCFM	16,585	17,070	16,828	18,810	18,779	18,795	20,784	18,406[4]

[1] Orsat data invalid; used Stack 1 data
[2] Average of Stacks 1, 2, and 4
[3] Average of Stacks 1 and 2
[4] Average of Stacks 1, 2, and 3

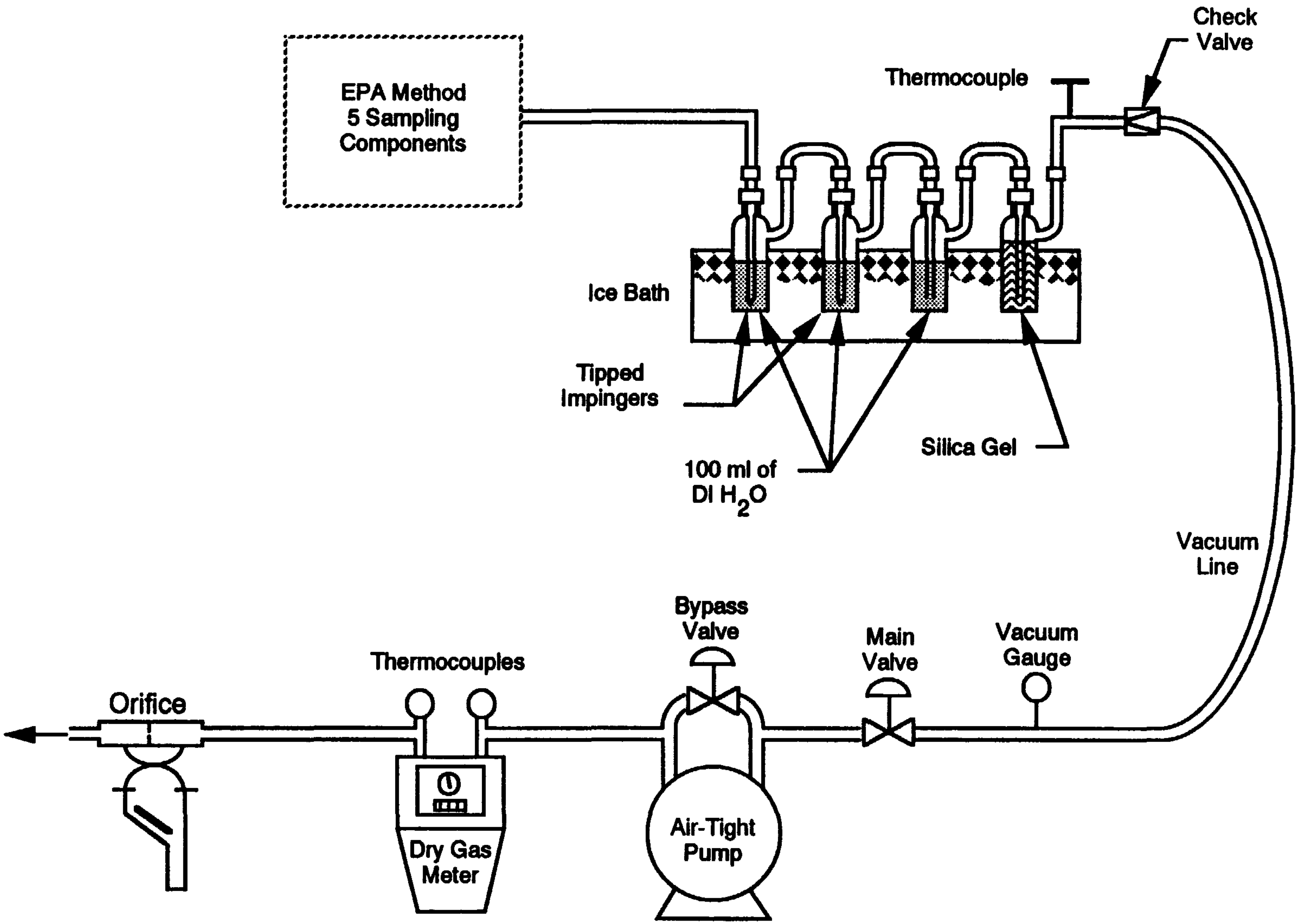
TABLE 7-6.

SUMMARY DATA USED IN CALCULATIONS
JEWELL COAL AND COKE COMPANY

DAY 3

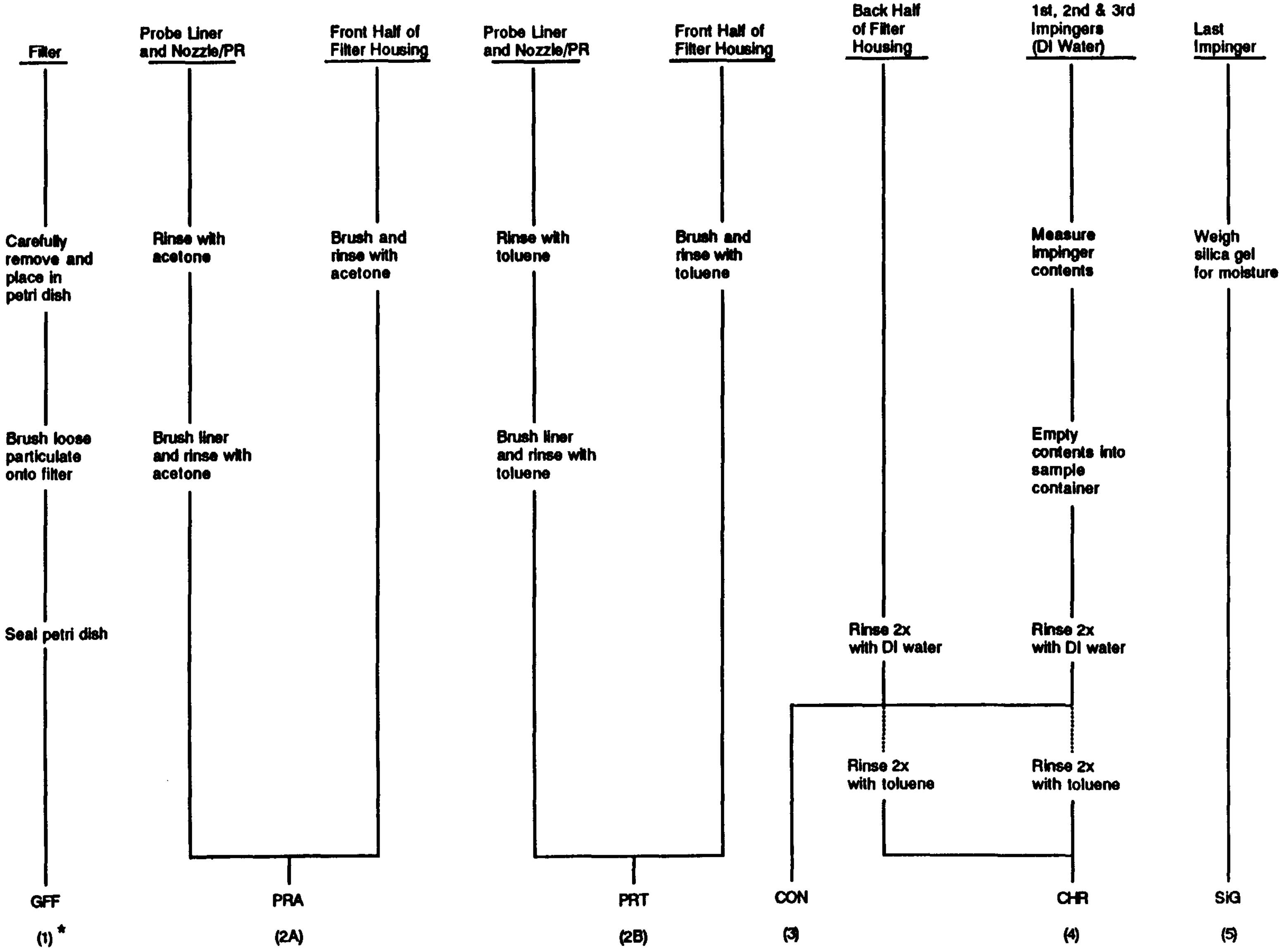
DAY 3	STACK 1			STACK 2			STACK 3	STACK 4
	MMTL	M5	AVERAGE	MM5-A	MM5-B	AVERAGE	VOST	CEM
CONDITION I								
% O_2	10.6	10.6		9.4	9.4		10.3[1]	11.0
% CO_2	6.5	6.5		7.8	7.8		6.9[1]	6.5
% H_2O	15.6	14.3		15.1	15.1		15.0[2]	15.0[2]
Temperature, °F	1,511	1,506		1,621	1,621		1,543	1,533
Flow Rate, DSCFM	20,672	20,142	20,407	21,170	21,187	21,179	25,490	21,732[3]
CONDITION II								
%O_2		8.9			7.4		8.6[1]	9.4
%CO_2		7.9			9.1		8.2[1]	7.5
%H_2O		16.5			17.4		17.0[2]	17.0[2]
Temperature, °F		1,483			1,549		1,522	1,529
Flow Rate, DSCFM		20,739	20,739		16,965	16,965	23,289	20,331[3]
CONDITION III								
%O_2		9.5			7.2		8.5[1]	8.9
%CO_2		7.2			8.6		7.9[1]	7.8
%H_2O		13.0			15.5		14.3[2]	14.3[2]
Temperature,°F		1,460			1,574		1,536	1,508
Flow Rate, DSCFM		16,019	16,019		17,952	17,952	23,774	19,248[3]
CONDITION IV								
%O_2		8.5		8.0			8.3[1]	8.6
%CO_2		7.5		7.9			7.7[1]	7.6
%H_2O		12.3		13.7			13.0[2]	13.0[2]
Temperature, °F		1,508		1,575			1,502	1,516
Flow Rate, DSCFM		17,766	17,766	17,964		17,964	20,279	18,670[3]

[1] Average of Stacks 1, 2, and 4
[2] Average of Stacks 1 and 2
[3] Average of Stacks 1, 2, and 3



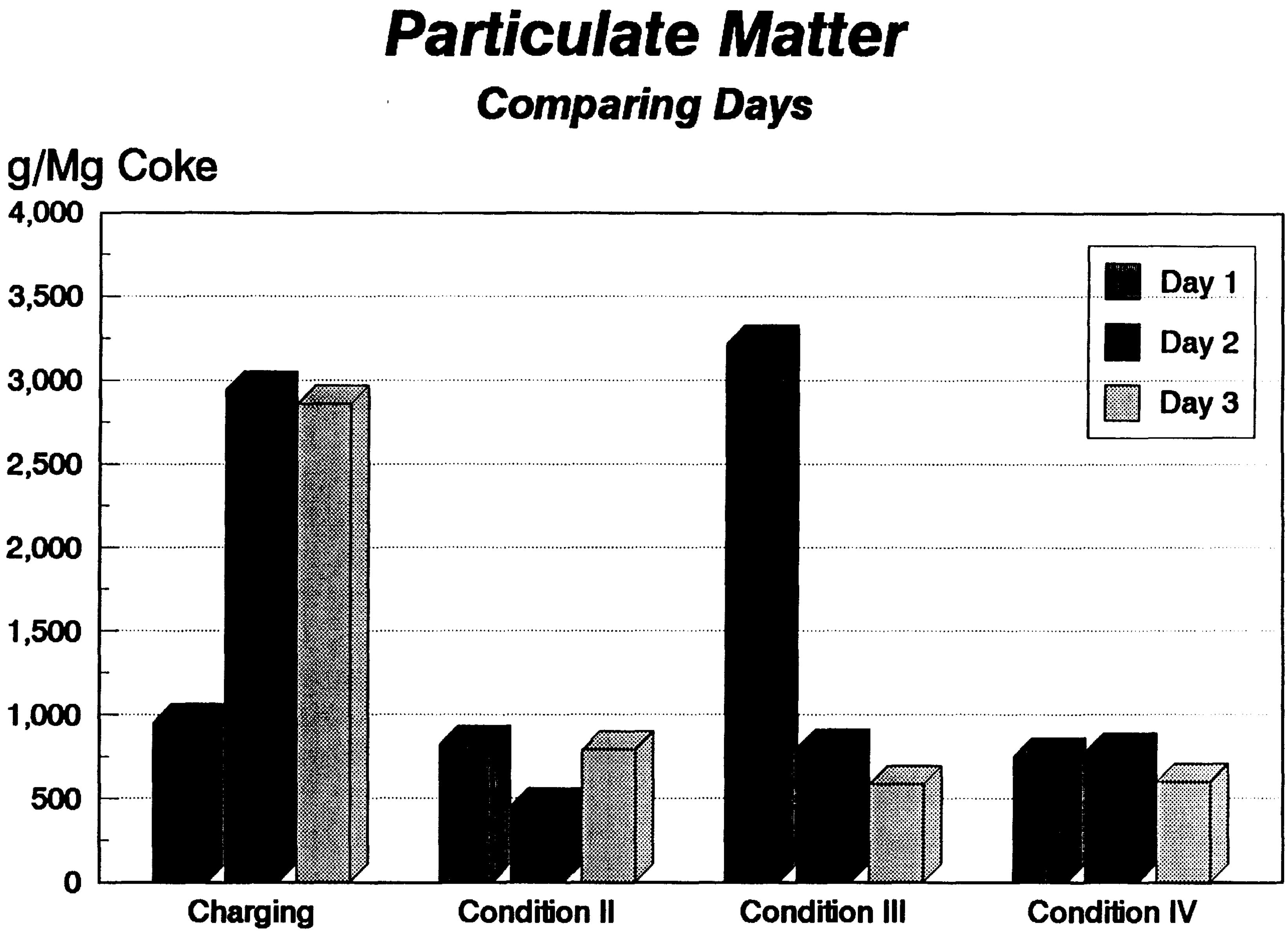
3694 1/92

Figure 7-1. M5/TSO/202 sampling train.



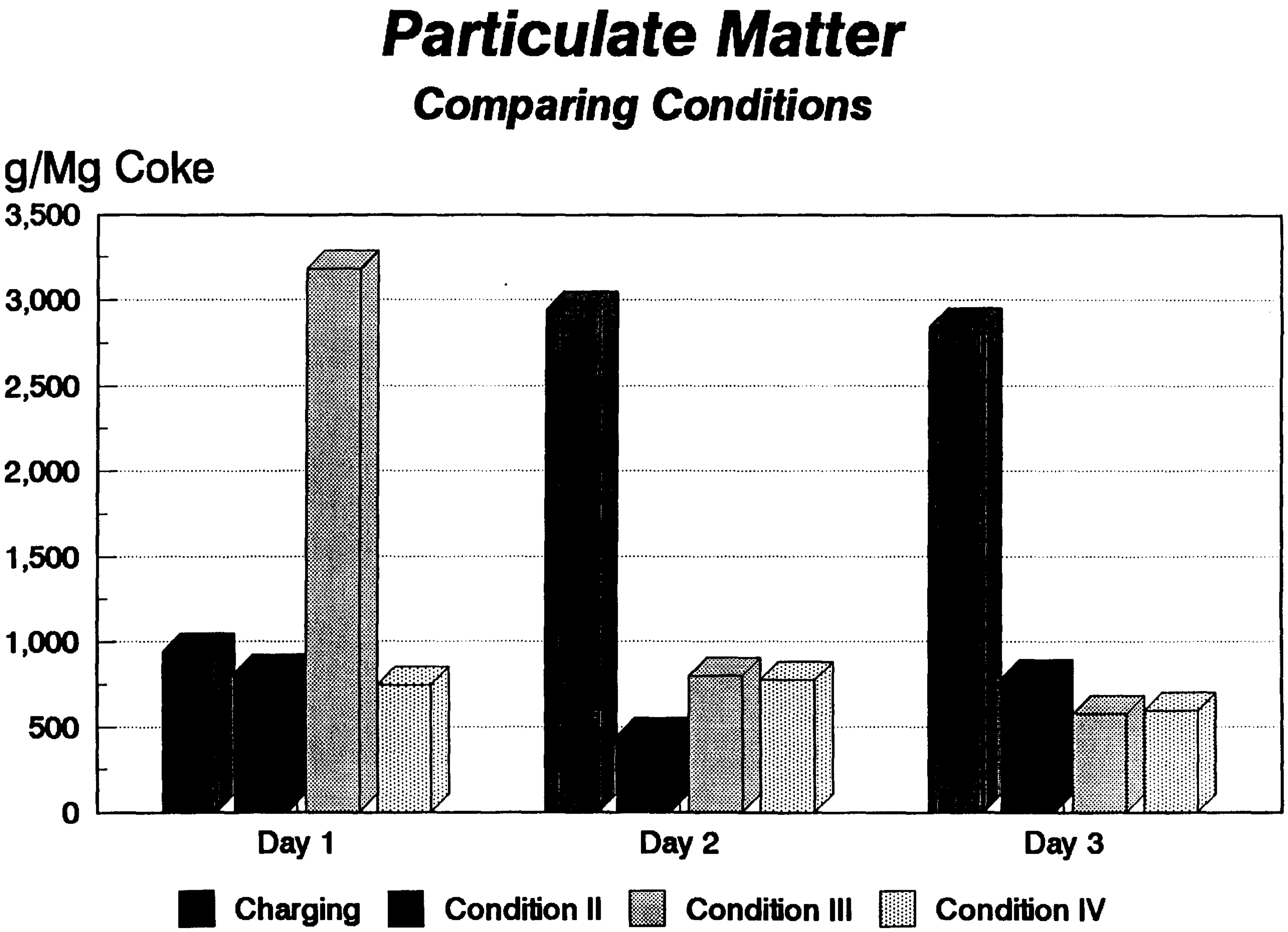
*Sample container number.

Figure 7-2. Sample recovery scheme for M5/TSO/202 samples.



Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-3.



Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-4.

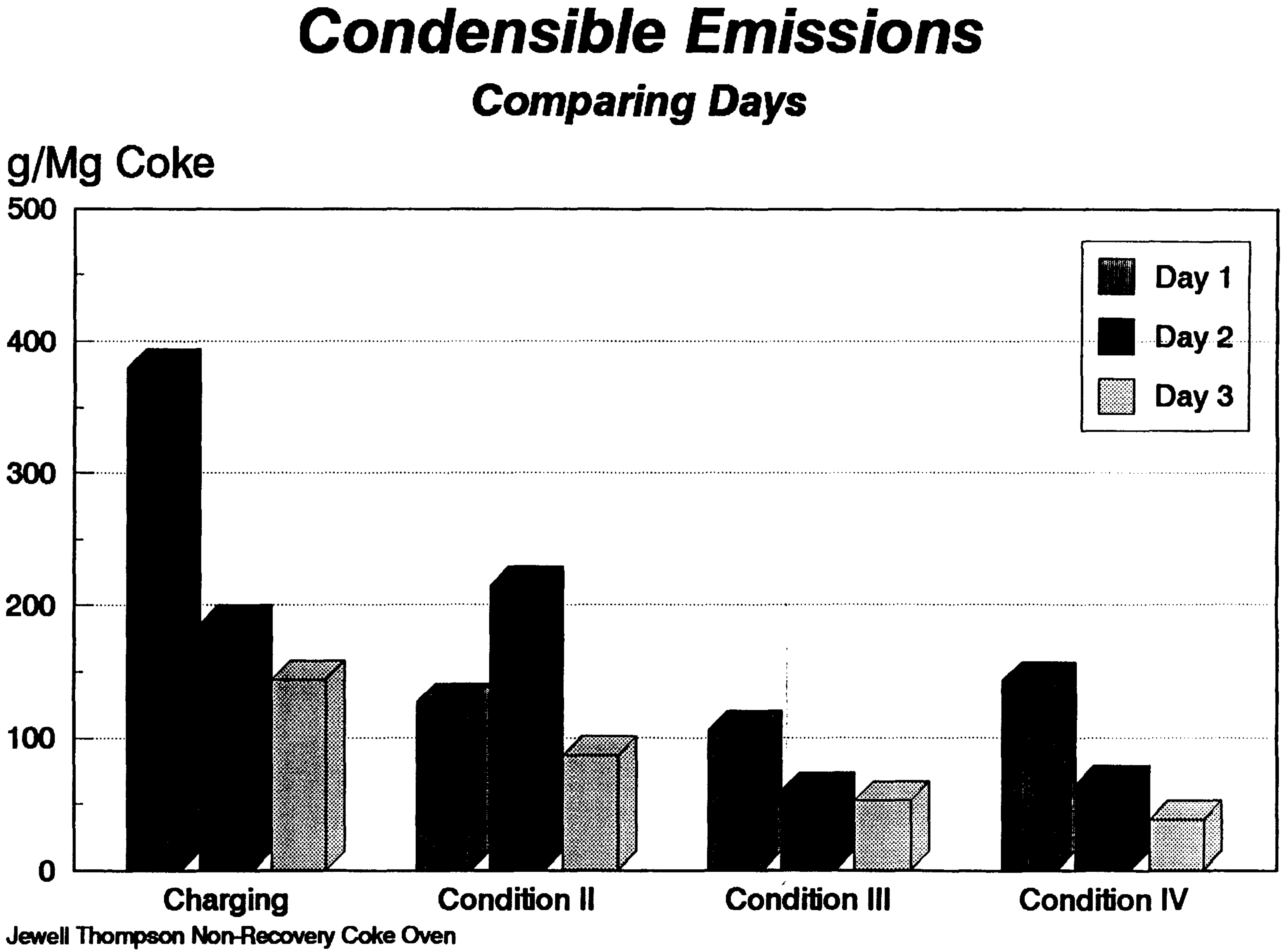

Condensible Emissions
Comparing Days
g/Mg Coke
500
400
300
200
100
0
Day 1
Day 2
Day 3
Charging
Condition II
Condition III
Condition IV
Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-5.

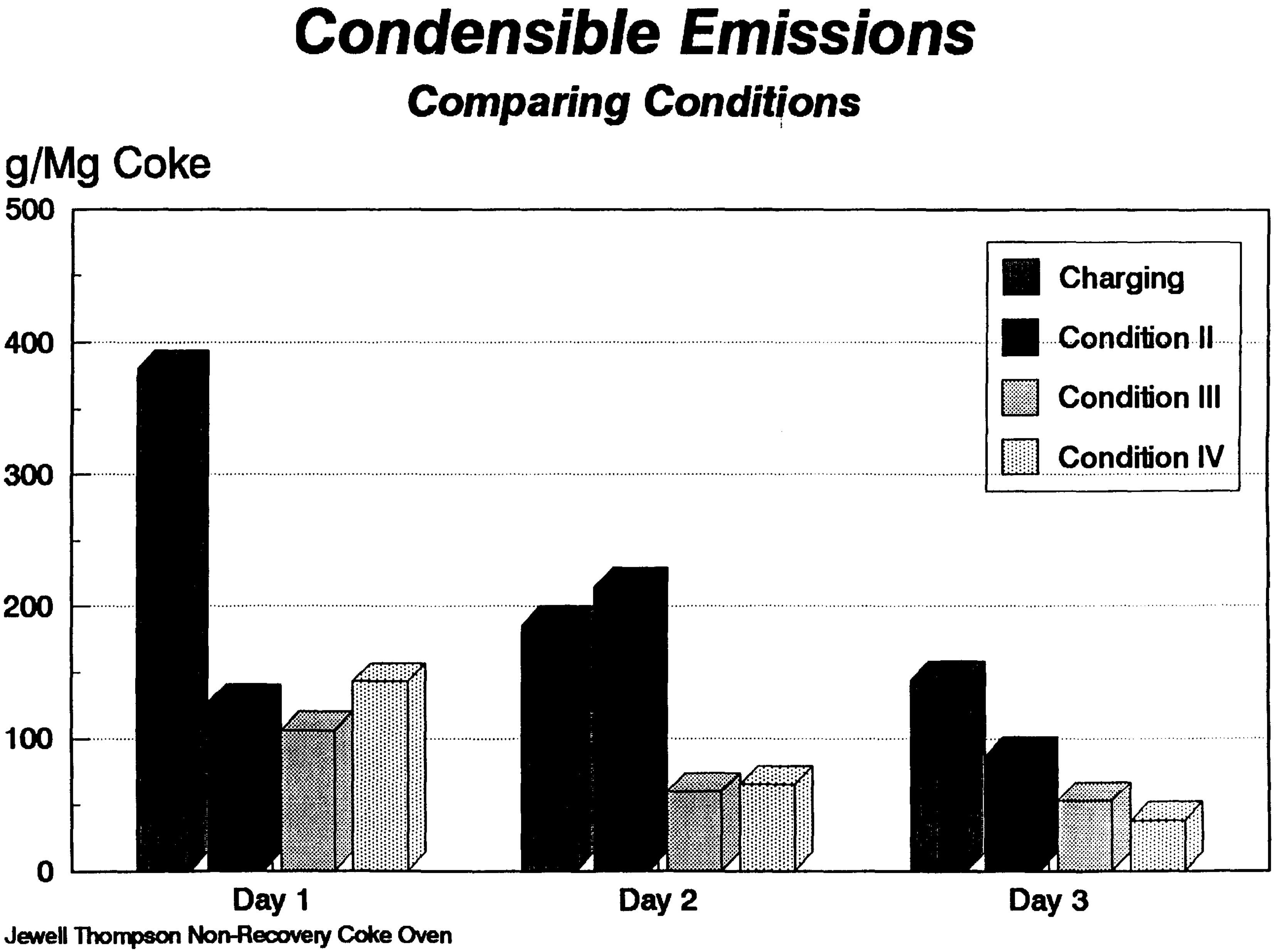
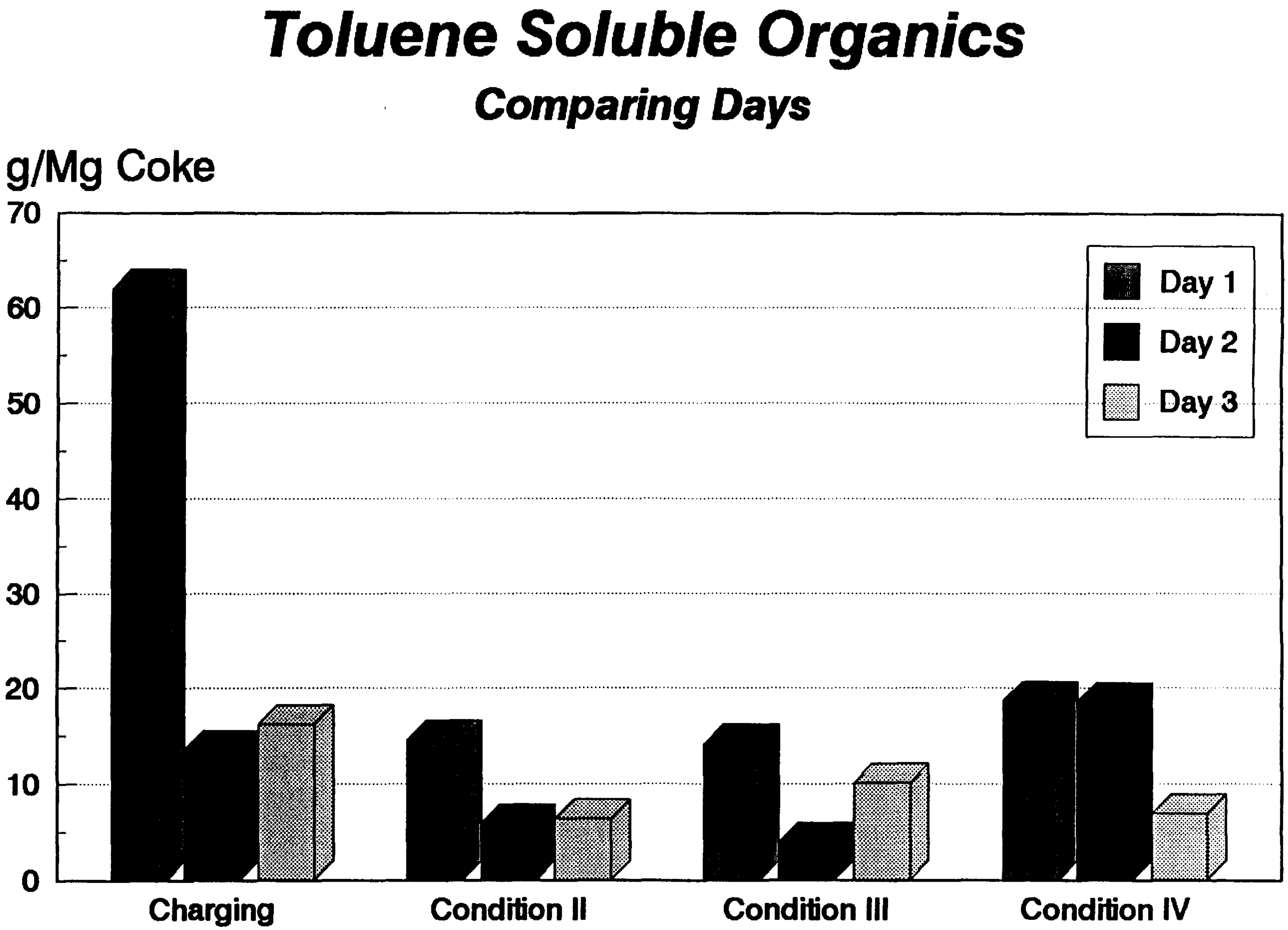
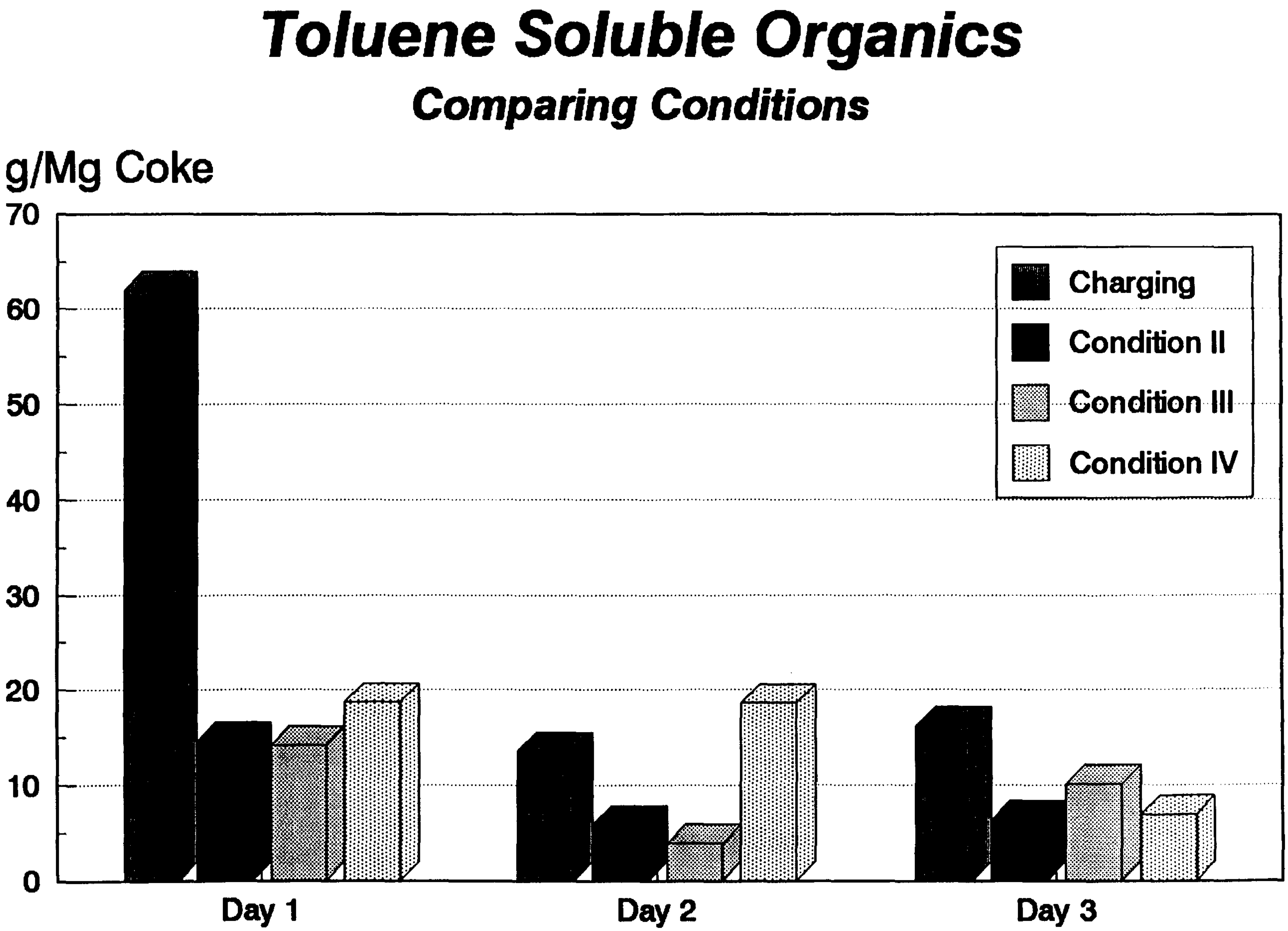

Condensible Emissions
Comparing Conditions
g/Mg Coke
500
400
300
200
100
0
Charging
Condition II
Condition III
Condition IV
Day 1
Day 2
Day 3
Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-6.



Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-7.



Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-8.

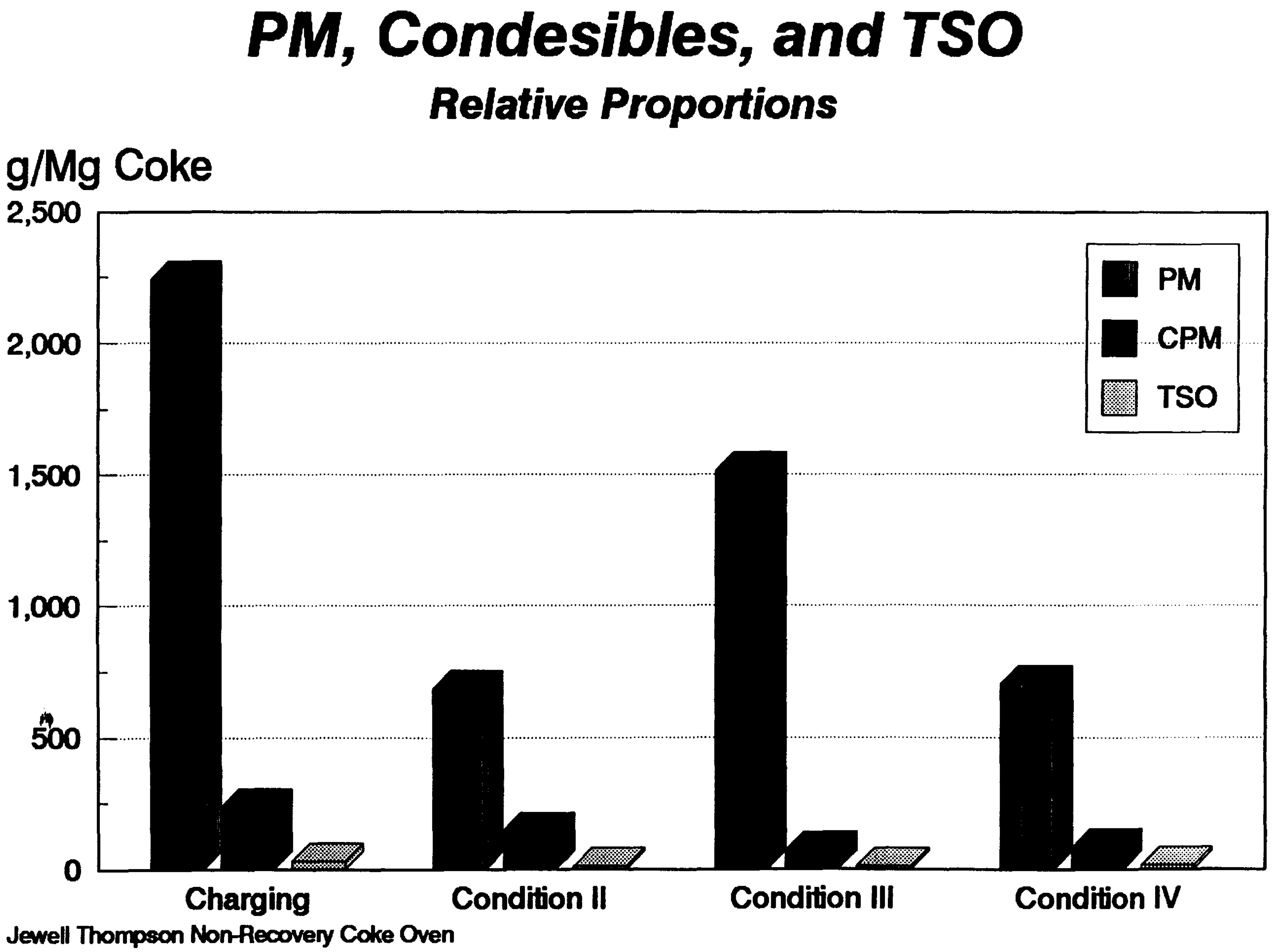

PM, Condesibles, and TSO
Relative Proportions
g/Mg Coke
2,500
2,000
1,500
1,000
500
0
PM
CPM
TSO
Charging
Condition II
Condition III
Condition IV
Jewell Thompson Non-Recovery Coke Oven

FIGURE 7-9.

8.0 MULTIPLE-METALS

8.1 TEXT MATRIX FOR MULTIPLE-METALS (MMTL)

Emissions sampling for 16 trace metals (Sb, As, Ba, Be, Cd, Cr, Cu, Pb, Mn, Hg, Ni, P, Se, Ag, Tl, and Zn) were conducted on Stack 1 using a draft EPA method (a copy is included in Appendix D) entitled "Methodology for the Determination of Metals Emissions in Exhaust Gases from Hazardous Waste Incineration and Similar Combustion Processes." This method is considered to be a reference method.

8.2 MMTL SAMPLING EQUIPMENT, PREPARATION, AND RECOVERY

8.2.1 Sampling Equipment and Preparation

The MMTL sampling used the sampling train shown in Figure 8-1. The equipment was modified Method 5 sampling equipment, differing by the use of low metals background quartz fiber filters (QFF), Teflon™ filter supports, HNO_3/H_2O_2 and $KMnO_4/H_2SO_4$ instead of H_2O in the impingers, and additional impingers.

Glassware and sample containers were cleaned prior to the testing program according to the procedures shown in Table 8-1.

The sampling train, minus the probe, was assembled in the sample recovery area. 5% HNO_3/10% H_2O_2, 8 N HCl, and 0.1 N HNO_3 were prepared daily and stored in vented glass bottles. There were seven impingers in the sampling train; the first impinger was empty and functioned as a moisture knockout. A pre-weighed charge of 200 mL of 5% HNO_3/10% H_2O_2 was divided among the second and third impingers. The fourth impinger was empty. A pre-weighed charge of 300 mL of $KMnO_4$/ H_2SO_4 was divided among the fifth and sixth impingers. The seventh impinger contained ~300 grams of indicating silica gel. The third impinger had a Greenburg-Smith type tip. All other impingers were modified Greenburg-Smith type tips. An untared filter was loaded into the front half filter holder and leak checked. All openings on the probe assembly and the impinger train were capped for transfer to the sampling location.

The sampling train was operated according to Method 5 and the sampling was conducted simultaneously with CEM, M5/TSO/202, VOST, and MM5 testing. The total sampling run times were approximately two hours for Condition I and four hours for Conditions II, III, and IV. Following each sampling run, the probe was removed from the sampling train, the openings were capped, and the sampling train was returned to the sample recovery area.

During the second run of Condition III, a MMTL field blank train was assembled with glassware that had been previously used for MMTL sampling. This train was charged exactly as the other MMTL trains and left at a sampling location during the test run. The field blank train was then recovered with the other MMTL trains for that run.

8.2.2 Sample Recovery

Once in the recovery area, the MMTL sampling trains, including the field blank train, were recovered following the scheme shown in Figure 8-2. Liquid samples were stored in pre-cleaned glass sample jars with Teflon™ lid liners. The sample identification codes for train fractions are listed in Table 8-2.

Reagent blank samples of HNO_3, $KMnO_4$, and HCl used in sample recovery were collected. The liquid level and sample identification number were marked on each sample container and samples were stored in locked boxes for return to Entropy's laboratory.

8.3 MMTL ANALYSES

The three analytical procedures used to determine the metals emissions of interest were: Cold Vapor Atomic Absorption (CVAA), Graphite Furnace Atomic Absorption (GFAA), and Inductively Coupled Plasma Spectroscopy (ICAP). Mercury was determined by CVAA. Antimony, arsenic, lead, selenium, silver, and thallium were determined by GFAA. Barium, manganese, and zinc were determined by ICAP. Beryllium, cadmium, chromium, copper, and nickel were determined by ICAP when concentrations were high enough; otherwise, GFAA was used. High levels of the elements of interest were found in the front half and were determined by ICAP, but the low levels in the back half required GFAA. The detection limit of each metal is shown in Table 8-3.

Analyses of each component recovered as shown in Table 8-2 depended on the metal of interest within each fraction. Because the train was not used for the determination of particulate matter, the filter could be analyzed with the front half HNO_3 rinse. The front half components include QFF and the HNO_3 rinse of the probe and front half (PRN). The component, including the back half impingers 1, 2, and 3 and their HNO_3 rinse (BHN), was analyzed as above by ICAP and CVAAS for all 16 metals. Components containing the empty impinger and HNO_3 rinse (BHNHg), impingers 5 and 6 and their $KMnO_4$ rinse ($BHKMnO_4$), and back half rinse with concentrated HCl (BHHCl) were analyzed for Hg using only CVAAS. All analytical procedures were performed as specified in the method.

8.4 MMTL QC PROCEDURES

Calibration and leak checking of the appropriate sampling equipment, including meter boxes, temperature sensors, nozzles, pitot tubes, and umbilicals, were performed according to the requirements specified in EPA's "Quality Assurance Handbook, Volume III" (600/4-77-027b). The results were documented and retained.

Special attention was given to the following QC checks:

- Prior to and following each run and port change, the sampling train was leak checked; the leak rate should not have exceeded the lesser of 0.02 actual cubic feet per minute (acfm) or 1.5 percent of the actual sampling rate. If a final leak rate did not meet the acceptance criterion, the test run was considered acceptable upon approval of the EPA Work Assignment Manager. In this situation, the measured leak rate was reduced by the allowable leak rate and then multiplied by the period of time over which the leak occurred. This "leak volume" was subtracted from the measured gas volume in calculating the emission results. This corrective action was noted in any reporting.

- The probe and front half filter compartment were maintained at 248°F ± 25°F and the outlet of the silica gel impinger was maintained at less than 68°F during sampling.
- Isokinetic sampling was maintained within 100 ± 10 percent; readings were recorded for each traverse point.

There are several key QC requirements for the various train fractions. These include:

- ICAP Analysis -
  - -- Instrument check samples - blank and midpoint curve
  - -- Interference check sample - known concentration of interfering elements
  - -- QC sample - check calibration
  - -- Duplicate analyses - ± 10 %
- CVAAS Analysis -
  - -- All samples in duplicate
  - -- QC sample - check calibration
  - -- Matrix spike samples - spike field samples

8.5 MMTL SUMMARY OF RESULTS

MMTL data were calculated using two approaches for data presentation. Table 8-4 presents the total weighted averages per cycle. Due to voiding or aborting runs and equipment problems, only two days worth of data are presented.

Tables 8-5a through 8-5d present the 16 targeted metals as measured in Stack 1, Conditions I through IV, respectively. As shown in Tables 8-5b, 8-5c, and 8-5d, only two days of data were calculated. As shown in Table 8-5a, three days of data are presented, however, only two days worth of data were from the sampling runs; Day 2 data were calculated based on the M5/TSO/202 train data which are indicated with a "b" superscript. The concentrations are the averages of the concentrations from Runs 1 and 3 as indicated with a "c" superscript. Emission rates were calculated from the averaged concentrations and flow rate data obtained from the M5/TSO/202 train data and is indicated as a "d" superscript.

No other runs during Conditions II, III, and IV were qualified. It was necessary to qualify Condition I, Run 2 for the total weighted averages only, a third run allowed us to do this.

Reviewing each Condition as measured in Stack 2 illustrates the trends in the data. The data indicate that in all but three cases, the metals results followed a downward trend during Condition II, returning to an upward trend for Conditions III and IV. There were three exceptions including chromium, copper, and nickel. Chromium appears to be an outlier in Condition II, Run 1; it had a higher value than any of the other runs. Additionally, copper appears to be an outlier in Condition II, Run 1; its concentration was lower than during any of the other runs. Nickel during the same condition and run appears to be an outlier; however, it is not as significant. If each of these outliers is removed, the trend is consistent for all compounds.

8.6 MMTL DATA QUALIFIERS

Provided in this section is a list of data qualifiers. As previously mentioned, some runs were voided or aborted or other less significant incidences occurred. These included:

1. Orsat data from Stack 1, Day 1, Condition III was invalid; Day 1, Condition III data from Stack 2 was used.

2. Day 2, Condition I run was aborted because of a probe heat failure caused moisture to condense in the probe and filter.

3. EPA prioritized the importance of data in the case of equipment shortages. Both the MM5 probe sets were destroyed due to the heat and weight of the probe. Semi-volatile organic compounds testing, which included the field validation program, was deemed more important than additional MMTL runs. The MMTL sampling probe was used to complete the sampling of semi-volatile organic compounds. MMTL Day 3, Conditions II, III, and IV were not sampled.

4. Refer to Tables 7-3, 7-4, and 7-5 for flowrate, temperature, moisture, and gas constituent data used in the calculations.

TABLE 8-1.

MULTIPLE-METALS GLASSWARE CLEANING PROCEDURE (TRAIN COMPONENTS, SAMPLE CONTAINERS, AND LABORATORY GLASSWARE)

1. Rinse with hot tap water.
2. Wash in hot soapy water.
3. Rinse 3 times with tap water.
4. Rinse 3 times with DI water.
5. Soak 4 hours in 10% HNO_3.
6. Rinse 3 times with DI water.
7. Rinse with Acetone.
8. Air dry.
9. Cover openings with non-metallic seal.

TABLE 8-2.

MMTL SAMPLE FRACTIONS

Container/ Component	Code	Fraction
1	QFF	Quartz fiber filter
2	PRN	HNO_3 rinse of probe and front half
3	BHN	Back half impingers contents and 1, 2, 3 rinse with HNO_3
4	BHNHg	Empty impinger and HNO_3 rinse
5	$BHKMnO_4$	Back half impingers 5, 6 Contents and rinse with $KMnO_4$, then water
6	BHHCl	Back half rinse with 25 mL 8N HCl
7	SiG	Silica gel

TABLE 8-3.

ANALYTICAL DETECTION LIMITS (ng/mL) FOR METALS FROM THE MMTL TRAIN

Element	Detection Limit
Sb	3
As	1
Ba	2
Be	0.2
Cd	0.1
Cr	1
Cu	6
Pb	1
Mn	2
Hg	0.2
Ni	15
P	75
Se	2
Ag	7
Tl	1
Zn	2

TABLE 8-4.

MULTIPLE METALS EMISSIONS DATA
TOTAL WEIGHTED AVERAGES PER CYCLE
JEWELL COAL & COKE COMPANY

Day/Repetition Number	1	2	Average
Antimony:			
Concentration, μg/DSCM[a]	14.06	12.59	13.33
Emission Rate, (10^{-3}) lb/hr	3.79	3.50	3.65
Emission Rate, (10^{-3}) lb/ton charged	0.13	0.12	0.125
Arsenic:			
Concentration, μg/DSCM[a]	52.50	137.30	94.90
Emission Rate, (10^{-3}) lb/hr	36.32	38.37	37.35
Emission Rate, (10^{-3}) lb/ton charged	1.22	1.28	1.25
Barium:			
Concentration, μg/DSCM[a]	17.53	9.55	13.54
Emission Rate, (10^{-3}) lb/hr	4.75	2.71	3.73
Emission Rate, (10^{-3}) lb/ton charged	0.16	0.09	0.125
Beryllium:			
Concentration, μg/DSCM[a]	2.18	2.43	2.31
Emission Rate, (10^{-3}) lb/hr	0.58	0.67	0.63
Emission Rate, (10^{-3}) lb/ton charged	0.02	0.02	0.02
Cadmium:			
Concentration, μg/DSCM[a]	17.85	19.96	18.91
Emission Rate, (10^{-3}) lb/hr	5.10	5.49	5.30
Emission Rate, (10^{-3}) lb/ton charged	0.17	0.18	0.175
Chromium:			
Concentration, μg/DSCM[a]	93.00	40.15	66.58
Emission Rate, (10^{-3}) lb/hr	26.21	11.16	18.69
Emission Rate, (10^{-3}) lb/ton charged	0.88	0.37	0.63
Copper:			
Concentration, μg/DSCM[a]	278.25	351.68	314.97
Emission Rate, (10^{-3}) lb/hr	72.50	97.11	84.81
Emission Rate, (10^{-3}) lb/ton charged	2.43	3.24	2.84
Lead:			
Concentration, μg/DSCM[a]	348.75	396.47	372.61
Emission Rate, (10^{-3}) lb/hr	93.90	109.32	101.61
Emission Rate, (10^{-3}) lb/ton charged	3.14	3.64	3.39

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-4. (cont.)

Day/Repetition Number	1	2	Average
Manganese:			
Concentration, µg/DSCM•	47.75	19.51	33.63
Emission Rate, (10^{-3}) lb/hr	12.41	5.37	8.89
Emission Rate, (10^{-3}) lb/ton charged	0.42	0.18	0.30
Mercury:			
Concentration, µg/DSCM•	37.75	34.52	36.14
Emission Rate, (10^{-3}) lb/hr	10.11	9.67	9.89
Emission Rate, (10^{-3}) lb/ton charged	0.34	0.32	0.33
Nickel:			
Concentration, µg/DSCM•	71.50	53.63	62.57
Emission Rate, (10^{-3}) lb/hr	19.63	14.84	17.24
Emission Rate, (10^{-3}) lb/ton charged	0.66	0.49	0.58
Phosphorus:			
Concentration, µg/DSCM•	1482.25	1489.23	1485.74
Emission Rate, (10^{-3}) lb/hr	398.51	415.34	406.93
Emission Rate, (10^{-3}) lb/ton charged	13.34	13.84	13.59
Selenium:			
Concentration, µg/DSCM•	32.25	38.73	35.49
Emission Rate, (10^{-3}) lb/hr	8.75	10.64	9.70
Emission Rate, (10^{-3}) lb/ton charged	0.29	0.35	0.32
Silver:			
Concentration, µg/DSCM•	5.075	4.53	4.80
Emission Rate, (10^{-3}) lb/hr	1.39	1.26	1.33
Emission Rate, (10^{-3}) lb/ton charged	0.05	0.04	0.045
Thallium:			
Concentration, µg/DSCM•	18.45	19.16	18.81
Emission Rate, (10^{-3}) lb/hr	4.97	5.31	5.14
Emission Rate, (10^{-3}) lb/ton charged	0.17	0.18	0.175
Zinc:			
Concentration, µg/DSCM•	555.25	562.81	559.03
Emission Rate, (10^{-3}) lb/hr	149.40	157.12	153.26
Emission Rate, (10^{-3}) lb/ton charged	5.00	5.24	5.12

•68°F (20°C) --29.92 Inches of Mercury (Hg)

TABLE 8-5a.

MULTIPLE METALS EMISSIONS DATA AS MEASURED IN STACK 1 CONDITION I - CHARGING JEWELL COAL & COKE COMPANY

Run Identification	I-S1-MMTL			
Day/Repetition Number	1	2[b]	3	Average
Run Date	10/29/91		10/31/91	
Run Start Time	1741		1719	
Run Finish Time	2009		1932	
Total Run Time, min.	120		120	
Test Train Parameters:				
Volume of Metered Dry Gas Sample, SCF[a]	65.753		62.627	
Percent Isokinetic	95.3		98.8	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry	6.0	6.4	6.5	
O_2, Percent by Volume, Dry	11.2	11.2	10.6	
Flue Gas Temperature, Degrees F	1,583	1,561	1,511	
Velocity, ft/sec	33.23	31.21	29.72	
Air Flow Rate, Dry, SCFM[a]	22,494	21,310	20,672	
Air Flow Rate, Wet, ACFM	105,493	100,102	94,348	
Antimony:				
Concentration, µg/DSCM[a]	9.02	14.96[c]	20.9	14.96
Emission Rate, (10^{-3}) lb/hr	0.76	1.19[d]	1.62	1.19
Arsenic:				
Concentration, µg/DSCM[a]	215	216.5[c]	218	216.5
Emission Rate, (10^{-3}) lb/hr	18.1	17.28[d]	16.9	17.43
Barium:				
Concentration, µg/DSCM[a]	23.4	20.3[c]	17.2	20.3
Emission Rate, (10^{-3}) lb/hr	1.97	1.62[d]	1.33	1.64
Beryllium:				
Concentration, µg/DSCM[a]	1.24	1.74[c]	2.23	1.74
Emission Rate, (10^{-3}) lb/hr	0.105	0.14[d]	0.172	0.139

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

[b] Sample void; flue gas parameter values presented are from the M5/TSO/202 train.

[c] Average concentrations from Runs 1 and 3.

[d] Calculated using the M5/TSO/202 train data (b) and the average concentrations (c).

(continued)

TABLE 8-5a. (cont.)

Run Identification		I-S1-MMTL		
Day/Repetition Number	1	2	3	Average
Cadmium:				
Concentration, µg/DSCM[a]	13.5	11.14[c]	8.77	11.14
Emission Rate, (10^{-3}) lb/hr	1.14	0.89[d]	0.679	0.90
Chromium:				
Concentration, µg/DSCM[a]	45.4	46.80[c]	48.2	46.80
Emission Rate, (10^{-3}) lb/hr	3.83	3.74[d]	3.73	3.77
Copper:				
Concentration, µg/DSCM[a]	199	276[c]	353	276
Emission Rate, (10^{-3}) lb/hr	16.8	22.03[d]	27.3	22.04
Lead:				
Concentration, µg/DSCM[a]	294	391[c]	488	391
Emission Rate, (10^{-3}) lb/hr	24.8	31.21[d]	37.8	31.27
Manganese:				
Concentration, µg/DSCM[a]	9.79	11.15[c]	12.5	11.15
Emission Rate, (10^{-3}) lb/hr	0.825	0.89[d]	0.967	0.894
Mercury:				
Concentration, µg/DSCM[a]	37.7	42.3[c]	46.9	42.3
Emission Rate, (10^{-3}) lb/hr	3.18	3.38[d]	3.63	3.40
Nickel:				
Concentration, µg/DSCM[a]	35.4	44.25[c]	53.1	44.25
Emission Rate, (10^{-3}) lb/hr	2.98	3.53[d]	4.12	3.54
Phosphorus:				
Concentration, µg/DSCM[a]	1,358	1,498.5[c]	1,639	1,498.5
Emission Rate, (10^{-3}) lb/hr	114	119.63[d]	127	120.21

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
[c] Average concentrations from Runs 1 and 3.
[d] Calculated using the M5 train data (b) and the average concentration (c).

(continued)

TABLE 8-5a. (cont.)

Run Identification		I-S1-MMTL		
Day/Repetition Number	1	2	3	Average
Selenium:				
Concentration, μg/DSCM[a]	43.0	34.6[c]	26.2	34.6
Emission Rate, (10^{-3}) lb/hr	3.62	2.76[d]	2.03	2.80
Silver:				
Concentration, μg/DSCM[a]	9.05	6.83[c]	4.61	6.83
Emission Rate, (10^{-3}) lb/hr	0.763	0.55[d]	0.357	0.56
Thallium:				
Concentration, μg/DSCM[a]	17.9	20.25[c]	22.6	20.25
Emission Rate, (10^{-3}) lb/hr	1.51	1.62[d]	1.75	1.63
Zinc:				
Concentration, μg/DSCM[a]	518	647[c]	776	647
Emission Rate, (10^{-3}) lb/hr	43.7	51.65[d]	60.1	51.82

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[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
[c] Average concentration from Runs 1 and 3.
[d] Calculated using the M5 train data (b) and the average concentration (c).

TABLE 8-5b.

MULTIPLE METALS EMISSIONS DATA
AS MEASURED IN STACK 1 CONDITION II
JEWELL COAL & COKE COMPANY

Run Identification	II-S1-MMTL		
Day/Repetition Number	1	2	Average
Run Date	10/29/91	10/30/91	
Run Start Time	2157	2202	
Run Finish Time	0323	0305	
Total Run Time, min.	270	270	
<u>Test Train Parameters</u>:			
Volume of Metered Dry Gas Sample, SCF[a]	121.066	122.168	
Percent Isokinetic	92.9	92.9	
<u>Flue Gas Parameters</u>:			
CO_2, Percent by Volume, Dry	6.3	7.8	
O_2, Percent by Volume, Dry	10.7	9.0	
Flue Gas Temperature, Degrees F	1,550	1,461	
Velocity, ft/sec	27.03	25.79	
Air Flow Rate, Dry, SCFM[a]	18,879	19,055	
Air Flow Rate, Wet, ACFM	85,832	81,899	
<u>Antimony</u>:			
Concentration, μg/DSCM[a]	12.5	7.19	9.85
Emission Rate, (10^{-3}) lb/hr	0.882	0.513	0.698
<u>Arsenic</u>:			
Concentration, μg/DSCM[a]	76.8	54.9	65.85
Emission Rate, (10^{-3}) lb/hr	5.43	3.92	4.68
<u>Barium</u>:			
Concentration, μg/DSCM[a]	8.17	4.35	6.26
Emission Rate, (10^{-3}) lb/hr	0.578	0.311	0.445
<u>Beryllium</u>:			
Concentration, μg/DSCM[a]	1.12	1.21	1.17
Emission Rate, (10^{-3}) lb/hr	0.079	0.0867	0.0829

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5b. (cont.)

Run Identification	II-S1-MMTL		
Day/Repetition Number	1	2	Average
<u>Cadmium</u>:			
Concentration, μg/DSCM[a]	8.53	6.74	7.64
Emission Rate, (10^{-3}) lb/hr	0.603	0.481	0.542
<u>Chromium</u>:			
Concentration, μg/DSCM[a]	200	21.1	110.55
Emission Rate, (10^{-3}) lb/hr	14.1	1.52	7.81
<u>Copper</u>:			
Concentration, μg/DSCM[a]	4.66	107	55.83
Emission Rate, (10^{-3}) lb/hr	0.329	7.63	3.98
<u>Lead</u>:			
Concentration, μg/DSCM[a]	253	153	203
Emission Rate, (10^{-3}) lb/hr	17.9	10.9	14.4
<u>Manganese</u>:			
Concentration, μg/DSCM[a]	18.4	7.85	13.13
Emission Rate, (10^{-3}) lb/hr	1.3	0.56	0.93
<u>Mercury</u>:			
Concentration, μg/DSCM[a]	16.9	23.2	20.05
Emission Rate, (10^{-3}) lb/hr	1.2	1.66	1.43
<u>Nickel</u>:			
Concentration, μg/DSCM[a]	104	21.3	62.65
Emission Rate, (10^{-3}) lb/hr	7.35	1.52	4.44
<u>Phosphorus</u>:			
Concentration, μg/DSCM[a]	919	659	789
Emission Rate, (10^{-3}) lb/hr	65	47	56

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[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5b. (cont.)

Run Identification		II-S1-MMTL	
Day/Repetition Number	1	2	Average
Selenium:			
Concentration, μg/DSCM[a]	17.5	12.5	15
Emission Rate, (10^{-3}) lb/hr	1.23	0.889	1.06
Silver:			
Concentration, μg/DSCM[a]	1.32	1.10	1.21
Emission Rate, (10^{-3}) lb/hr	0.0934	0.0788	0.0861
Thallium:			
Concentration, μg/DSCM[a]	10	7.45	8.73
Emission Rate, (10^{-3}) lb/hr	0.709	0.531	0.62
Zinc:			
Concentration, μg/DSCM[a]	355	202	278.5
Emission Rate, (10^{-3}) lb/hr	25.1	14.4	19.75

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

TABLE 8-5c.

MULTIPLE METALS EMISSIONS DATA AS MEASURED IN STACK 1 CONDITION III JEWELL COAL & COKE COMPANY

Run Identification		III-S1-MMTL	
Day/Repetition Number	1	2	Average
Run Date	10/30/91	10/31/91	
Run Start Time	0555	0505	
Run Finish Time	0948	0923	
Total Run Time, min.	192	240	
Test Train Parameters:			
Volume of Metered Dry Gas Sample, SCF[a]	84.188	104.494	
Percent Isokinetic	92.6	92.8	
Flue Gas Parameters:			
CO_2, Percent by Volume, Dry	5.7	7.4	
O_2, Percent by Volume, Dry	11.8	9.0	
Flue Gas Temperature, Degrees F	1,501	1,491	
Velocity, ft/sec	25.71	25.47	
Air Flow Rate, Dry, SCFM[a]	18,529	18,336	
Air Flow Rate, Wet, ACFM	81,641	80,884	
Antimony:			
Concentration, µg/DSCM[a]	15.1	17.2	16.15
Emission Rate, (10^{-3}) lb/hr	1.05	1.18	1.12
Arsenic:			
Concentration, µg/DSCM[a]	164	172	168
Emission Rate, (10^{-3}) lb/hr	11.4	11.8	11.6
Barium:			
Concentration, µg/DSCM[a]	16.7	8.28	12.49
Emission Rate, (10^{-3}) lb/hr	1.16	0.569	0.86
Beryllium:			
Concentration, µg/DSCM[a]	3.10	2.73	2.92
Emission Rate, (10^{-3}) lb/hr	0.215	0.188	0.20

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5c. (cont.)

Run Identification		III-S1-MMTL	
Day/Repetition Number	1	2	Average
Cadmium:			
Concentration, μg/DSCM[*]	26.6	24.7	25.65
Emission Rate, (10^{-3}) lb/hr	1.85	1.70	1.78
Chromium:			
Concentration, μg/DSCM[*]	44.9	52.5	48.7
Emission Rate, (10^{-3}) lb/hr	3.11	3.61	3.36
Copper:			
Concentration, μg/DSCM[*]	439	423	431
Emission Rate, (10^{-3}) lb/hr	30.5	29	29.75
Lead:			
Concentration, μg/DSCM[*]	406	581	493.5
Emission Rate, (10^{-3}) lb/hr	28.2	39.9	34.05
Manganese:			
Concentration, μg/DSCM[*]	74.1	26.4	50.25
Emission Rate, (10^{-3}) lb/hr	5.14	1.82	3.48
Mercury:			
Concentration, μg/DSCM[*]	39.4	36.1	37.75
Emission Rate, (10^{-3}) lb/hr	2.73	2.48	2.61
Nickel:			
Concentration, μg/DSCM[*]	59	62.6	60.8
Emission Rate, (10^{-3}) lb/hr	4.1	4.3	4.2
Phosphorus:			
Concentration, μg/DSCM[*]	1,695	1,439	1,567
Emission Rate, (10^{-3}) lb/hr	118	98.8	108.4

[*] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5c. (cont.)

Run Identification		III-S1-MMTL	
Day/Repetition Number	1	2	Average
Selenium:			
Concentration, μg/DSCM[a]	35.6	59.4	47.5
Emission Rate, (10^{-3}) lb/hr	2.47	4.08	3.28
Silver:			
Concentration, μg/DSCM[a]	4.35	5.63	4.99
Emission Rate, (10^{-3}) lb/hr	0.302	0.387	0.345
Thallium:			
Concentration, μg/DSCM[a]	22.1	24.1	23.1
Emission Rate, (10^{-3}) lb/hr	1.54	1.66	1.6
Zinc:			
Concentration, μg/DSCM[a]	680	549	614.5
Emission Rate, (10^{-3}) lb/hr	47.2	37.7	42.45

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[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

TABLE 8-5d.

MULTIPLE METALS EMISSIONS DATA
AS MEASURED IN STACK 1 CONDITION IV
JEWELL COAL & COKE COMPANY

Run Identification	IV-S1-MMTL		
Day/Repetition Number	1	2	Average
Run Date	10/30/91	10/31/91	
Run Start Time	1200	1100	
Run Finish Time	1456	1428	
Total Run Time, min.	144	192	
<u>Test Train Parameters</u>:			
Volume of Metered Dry Gas Sample, SCF[a]	53.411	78.775	
Percent Isokinetic	93.5	96.8	
<u>Flue Gas Parameters</u>:			
CO_2, Percent by Volume, Dry	7.1	7.9	
O_2, Percent by Volume, Dry	9.5	8.2	
Flue Gas Temperature, Degrees F	1,525	1,510	
Velocity, ft/sec	21.93	23.27	
Air Flow Rate, Dry, SCFM[a]	15,515	16,585	
Air Flow Rate, Wet, ACFM	69,645	73,869	
<u>Antimony</u>:			
Concentration, μg/DSCM[a]	16.5	12.7	14.6
Emission Rate, (10^{-3}) lb/hr	0.962	0.787	0.875
<u>Arsenic</u>:			
Concentration, μg/DSCM[a]	125	162	143.5
Emission Rate, (10^{-3}) lb/hr	7.29	10.1	8.70
<u>Barium</u>:			
Concentration, μg/DSCM[a]	25.5	12.9	19.2
Emission Rate, (10^{-3}) lb/hr	1.48	0.801	1.14
<u>Beryllium</u>:			
Concentration, μg/DSCM[a]	2.68	3.56	3.12
Emission Rate, (10^{-3}) lb/hr	0.156	0.221	0.189

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[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5d. (cont.)

Run Identification	IV-S1-MMTL		
Day/Repetition Number	1	2	Average
<u>Cadmium</u>:			
Concentration, μg/DSCM[a]	24.8	31.0	27.9
Emission Rate, (10^{-3}) lb/hr	1.44	1.92	1.68
<u>Chromium</u>:			
Concentration, μg/DSCM[a]	52.2	44.9	48.55
Emission Rate, (10^{-3}) lb/hr	3.03	2.79	2.91
<u>Copper</u>:			
Concentration, μg/DSCM[a]	421	547	484
Emission Rate, (10^{-3}) lb/hr	24.5	34	29.25
<u>Lead</u>:			
Concentration, μg/DSCM[a]	408	457	432.5
Emission Rate, (10^{-3}) lb/hr	23.7	28.4	26.05
<u>Manganese</u>:			
Concentration, μg/DSCM[a]	65.1	26.7	45.9
Emission Rate, (10^{-3}) lb/hr	3.79	1.66	2.73
<u>Mercury</u>:			
Concentration, μg/DSCM[a]	56.8	42	49.4
Emission Rate, (10^{-3}) lb/hr	3.3	2.61	2.96
<u>Nickel</u>:			
Concentration, μg/DSCM[a]	65.5	79.7	72.6
Emission Rate, (10^{-3}) lb/hr	3.81	4.95	4.38
<u>Phosphorus</u>:			
Concentration, μg/DSCM[a]	1,880	2,367	2,123.5
Emission Rate, (10^{-3}) lb/hr	109	147	128

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 8-5d. (cont.)

Run Identification	IV-S1-MMTL		
Day/Repetition Number	1	2	Average
Selenium:			
Concentration, µg/DSCM[a]	39.3	45.5	42.4
Emission Rate, (10^{-3}) lb/hr	2.28	2.83	2.56
Silver:			
Concentration, µg/DSCM[a]	8.01	6.19	7.10
Emission Rate, (10^{-3}) lb/hr	0.466	0.384	0.425
Thallium:			
Concentration, µg/DSCM[a]	23.5	25.6	24.55
Emission Rate, (10^{-3}) lb/hr	1.36	1.59	1.48
Zinc:			
Concentration, µg/DSCM[a]	645	913	779
Emission Rate, (10^{-3}) lb/hr	37.5	56.7	47.1

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

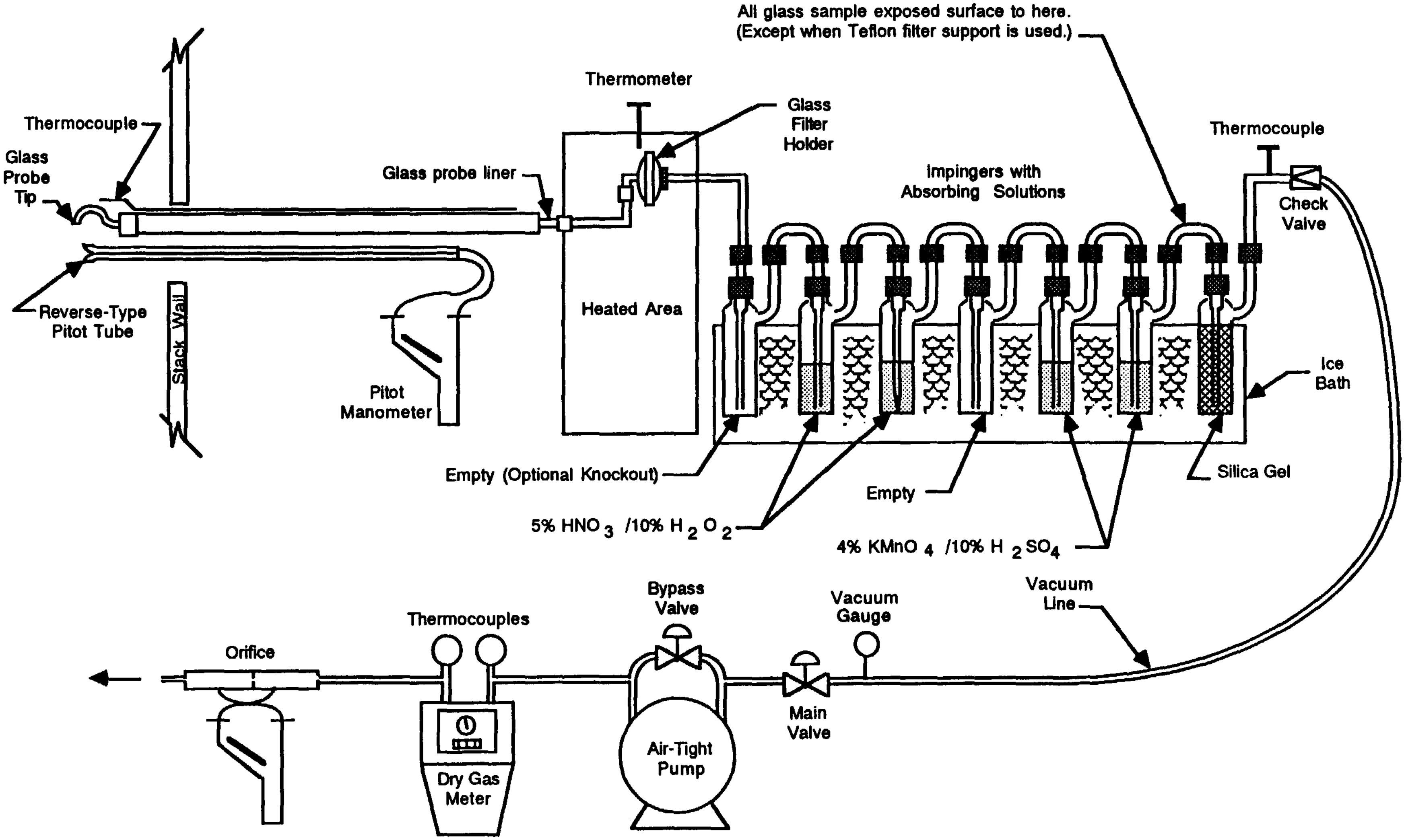

Figure 8-1 MMTL sampling train.

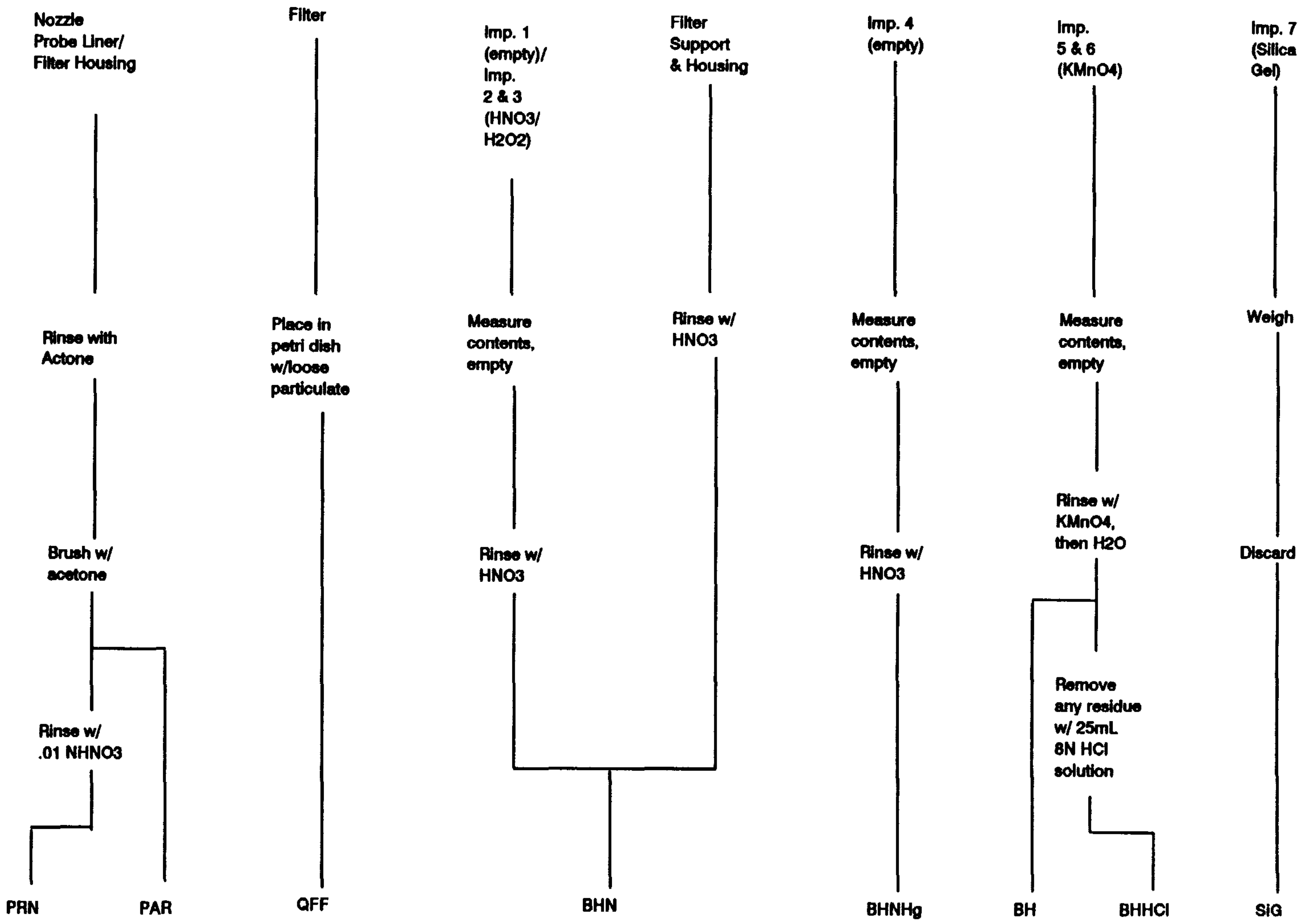

Figure 8-2. MMTL Sample Recovery Scheme.

9.0 SEMI-VOLATILE ORGANIC COMPOUNDS

9.1 SEMI-VOLATILE ORGANIC COMPOUNDS EMISSIONS TESTING (MM5)

The semi-volatile organic compound testing was conducted using SW-846 Method 0010 [Modified Method 5 (MM5)] sampling trains on Stack 2. Paired sampling trains were used to collect isokinetic samples concurrently with the CEM, M5/TSO/202, VOST, and MMTL sampling. The MM5 samples were analyzed quantitatively using the analytical protocol designed by TLI for BaP, cresol, napthalene, phenol, toluene, and xylenes (o-, m-, p-). The analysis is based on isotope dilution methodology. This involves adding a deuterium-labeled analog for the analytes of interest to the sample during extraction. The analyte is then measured relative to its labeled analog, which behaves in a chemically identical manner. Therefore, any loss of the analyte during laboratory handling is corrected during the analytical calculations. Additionally, TLI analyzed the samples semi-quantitatively for the CAA list of compounds which were targeted for identification and qualitatively for tentatively identified compounds (TICs).

Quantitative analysis is designed to identify an unknown compound and to measure how much of it is present in the sample. The precision and accuracy of the quantitation is a function of the level of quality control that is implemented in the design and execution of the analytical method. The quality control procedures for this test were designed for the targeted compounds, BaP, cresol, napthalene, phenol, toluene, and xylenes; therefore, the amounts reported for these compounds are considered quantitative values. The identified compounds on the CAA list fell outside the calibration curve for some fractions; therefore, the amounts reported are estimated values and are considered semi-quantitative. Besides the targeted compounds, there are usually a number of non-target components that are observed in the chromatogram. The mass spectrum of these compounds is searched against two libraries, the NBS library and the CAA library. Any compounds that are observed and identified are referred to as TICs. This type of analysis is considered qualititative analysis which focuses on the identity of the unknown compound, not the amount of the compound. Qualititative analysis may provide a **very approximate** value for the level of a detected compound by comparing the area of the peak to the area of the nearest internal standard, assuming a response factor of 1.

9.2 MM5 SAMPLING EQUIPMENT, PREPARATION, AND RECOVERY

9.2.1 Modified Method 5 Sampling Equipment and Preparation

A paired MM5 sampling train, shown in Figure 9-1, was used to collect the semi-volatile organic compound samples. All sampling equipment specifications are detailed in the copy of SW-846 Method 0010 provided in Appendix D.

The standard MM5 pre-cleanup requirements included several unique preparation steps which ensured that the sampling train components were not contaminated with organic compounds that may have interfered with the analysis. The glassware, glass fiber filters, and XAD adsorbing resin were pre-cleaned using strict protocols outlined in SW-846 Method 0010. Non-glass components were treated in the same manner as glassware with the exception of baking the equipment.

9.2.1.1 Glassware Preparation -- The glassware was cleaned as shown in Table 9-1. Glassware was washed in soapy water, rinsed with distilled water, baked, and rinsed with acetone, methylene chloride and methanol. Once the glassware was dry, the open ends were sealed with Teflon™ tape.

9.2.1.2 XAD Resin and Filters Preparation -- The XAD resin for all samples was pre-spiked prior to field sampling with 50 micrograms (μg) of the surrogates ethyl benzene-d_{10}, 2-Cl phenol-d_4, acenaphthene-d_{10}, and benzo-e-pyrene-d_{12} (solution C2, Table 4-1, Section 4.0) and also the PRE-SPIKE compound terphenyl-d_{14}. These labeled compounds were chosen because they represent compounds chemically similar to the analytes of interest. The C2 solutions relate to the C1 solutions as follows:

Ethyl benzene-d_{10} -----> Toluene and Xylenes
2-Chlorophenol-d_4 -----> Phenol and Cresol
Acenaphthene-d_{10} -----> Naphthalene-d_8
Benzo-e-pyrene-d_{12} -----> Benzo-e-pyrene-d_{12}

Recoveries of these compounds give an indication of the overall sampling and recovery efficiency and are intended to demonstrate the ability of the analytical methodology to achieve the proposed Method 301 requirements of a bias correction factor within the range of 0.7 - 1.3 and a precision of $\leq$50% RSD. The surrogates were directly introduced (methylene chloride solution) into the inlet end of the XAD trap via syringe immediately before sealing and packing the traps for shipping to the field.

To prepare the filters, a batch of 50 filters was placed in a soxhlet extraction apparatus and extracted using methylene chloride for 16 hours. Afterwards, the filters were checked for holes, tears, creases, and discoloration. Acceptable filters were placed in pre-cleaned containers and labeled by date of extraction and sealed with Teflon™ tape.

9.2.1.3 Other Equipment Preparation -- The remaining equipment preparation involved routine checking and calibration of the Method 5-type equipment as specified in Method 0010.

The sampling train minus the probe/nozzle was assembled in the sample recovery area as per method specifications. An optional empty first impinger with a short stem was used as a knockout impinger to collect the majority of the condensate formed in the condenser coil and XAD resin module. The train components were then transferred to the sampling location and completely assembled.

Samples were collected for a total of three hours using a sampling rate between 0.5 and 0.75 scfm to collect approximately 90 scf for Condition I, and a total of four hours to collect approximately 100 scf for Conditions II, III, and IV. Sampling train data were recorded on standard data forms presented in

Appendix B.

During the second run, Condition III, a MM5 field blank train was assembled with glassware that had already been used for MM5 sampling. The field blank results were used to assess any contamination that occurred from handling, charging, recovering, and transporting the sampling train.

9.2.2 Modified Method 5 Sample Recovery

The MM5 sampling trains, including the field blank train, were recovered following the scheme presented in Figure 9-2. Liquid samples were placed in pre-cleaned amber glass sample containers with Teflon™-lined lids. The filters were placed in pre-cleaned glass petri dishes.

Reagent blank samples were collected in pairs for all liquid reagents and rinses used in sample recovery, and singularly for the XAD sorbent module and glass fiber filter.

Duplicate blank samples were retained. The second set of blanks were archived for future examination if contamination was determined. A laboratory method blank was analyzed by TLI using the control XAD blank that was withheld during the XAD module preparation phase.

9.3 MM5 SAMPLE ANALYSIS

The MM5 semi-volatile organic samples were analyzed by TLI. Table 9-2 presents the sample fractions for analyses. As shown in Table 9-3, the sample fractions of Containers 1 and 2 were the front half fraction and Containers 3 and 4 were the back half fractions. Container 5 was the condensate fraction. Each fraction was analyzed using an isotope dilution method for the target compounds for BaP, cresol, napthalene, phenol, toluene, and xylenes (o-, m-, p-) and a non-target compound scan. The MM5 sample fractions presented in Table 9-4 were spiked with the listed internal standards prior to extraction. These compounds are also referred to as C1 compounds in Table 4-1.

9.3.1 Sample Lab Preparation Internal Standards Spiking

Prior to extraction, each fraction was spiked with 50 micrograms (μg) of the internal standards phenol-d_5, o-Cresol-d_7H, naphthalene-d_8, toluene-d_8, benzo-e-pyrene-d_{12}, and o-xylene-d_{10} (solution C1, Table 4-1). The analytes are measured relative to these internal standards, which behave in a chemically identical manner. Therefore, any loss of the analyte during laboratory handling is corrected during the analytical calculations, because the corresponding labeled standard has the same loss. The recovery of the internal standards was measured to give an indication of the laboratory and method efficiency for extraction and recovery of these compounds.

9.3.2 Extraction Description

All samples were extracted in fractions. These fractions, called front half (FH), back half (BH), and condensate (CON), contained the following train components:

Fraction Identification	Sample Components
FH	methanol/methylene chloride rinses, FH filter(s)
BH	XAD methanol/methylene chloride rinses
CON	Impinger water, impinger rinses

The FH and BH fractions were Soxhlet extracted with methylene chloride for 16 hours. Prior to extraction, the rinses were concentrated with a Kuderna-Danish evaporator and the concentrate added to the Soxhlet. After extraction, the concentrated extracts were split 50:50, one half used for the analysis and the other half archived for future use, if required.

The CON fractions were extracted with methylene chloride using a separatory funnel. These extractions were performed in two stages, one at a pH of 12 and the other at a pH of 2. These combined extracts were split 50:50, one half used for the analysis and the other half archived for future use, if required. All extracts for analysis were brought to a final volume of 2.5 mL prior to analysis. This sample was half the extract; therefore, this final volume was equivalent to a final extract volume of 5 mL for the entire extract and was used for the purposes of concentration calculations.

9.3.3 MM5 Quantitative and Semi-Quantitative Analysis

The samples were analyzed for target compounds which included BaP, napthalene, cresol, phenol, and xylenes; these compounds were analyzed quantitatively. Additionally, non-target compounds were analyzed semi-quantitatively.

9.3.3.1 Quantitative Analysis -- Quantitative identification of semi-volatile organic compounds was performed in one of two ways:

(1) For the target compounds listed and for other compounds for which authentic standards were available, the GC/MS system was calibrated and the mass spectrum and retention time for each standard were stored in a user created library. A compound was identified when its retention time and mass spectrum agreed with the library retention time and spectrum.

(2) For chromatographic peaks which were not identified by (1) above, the background corrected spectrum at the peak maximum was compared with spectra in the EPA/NIH Mass Spectral Library. Tentative identification of compounds was established when the spectrum agreed with the analyte of interest.

9.3.3.2 Semi-Quantitative Analysis -- Semi-quantitative analysis was performed in one of four ways by GC/MS using extracted ion current profile (EICP) areas:

(1) For compounds where standards and labeled analogs were available, the GC/MS system was calibrated and the compound concentration determined using an isotope dilution technique.

(2) For compounds where authentic standards but no labeled compounds were available, the GC/MS system was calibrated and the compound concentration determined using an internal standard technique.

(3) For compounds with standards not available, compound concentrations were determined using known response factors.

(4) For compounds where neither standards nor known response factors were available, compound concentrations were determined using the sum of the EICP areas relative to the sum of the EICP areas of the internal standard.

A list of semi-volatile organic compounds analyzed is presented in Table 9-4.

9.4 MM5 SEMI-VOLATILE ORGANIC ANALYSIS QUALITY CONTROL

The major QC procedures contained within the method included GC/MS tuning, calibration system performance checks, and analysis of specific QC check samples. During actual sample measurements, a field blank and a lab method blank were analyzed with the field samples. The criteria for acceptable performance for the QC measurements are listed in Table 9-5. The quality of the analysis was assured through reproducible calibration and testing of the extraction and GC/MS systems.

The SW-846 Method 8270 analytical protocol (see Appendix D) provides the procedures for the analysis of the stack emission samples for semi-volatile organic compounds.

The following QC procedures assist in defining the precision and accuracy for determining the concentration of the target semi-volatile compounds.

- Reference materials and standard solutions were composed of the highest quality chemicals available and when possible, were traceable to EPA primary references or NIST standard reference materials.
- Initial calibration was performed using a set of five initial calibration solutions. Each solution was analyzed once and the analytes' relative response factors (RRF) were determined.
- The instrument tuning performance requirements of Method 8270 were followed. Calibration checks involving analyses of known control samples were performed daily, using DFTPP.
- The analytical performance was monitored through the use of blank samples and surrogate standards.

- A laboratory method blank of an XAD sorbent module spiked with the surrogate standards identical to the XAD field samples was analyzed.
- The detection limit reported for the field samples for a specific analyte was at least 2.5 times lower than the concentration of the same analyte found in the fortified method blank.

The EPA has not established quantitative criteria for the precision and accuracy of Method 8270. However, QC objectives for the calibration and analytical procedures can be established based on previous laboratory experience. Accuracy is the degree of agreement of a measurement, or average of measurements, with an accepted reference value or true value.

9.5 MM5 SUMMARY OF RESULTS

Tables 9-6a through 9-6d present, as measured in Stack 2, the targeted semi-volatile organic compound emissions data, including BaP, cresol, napthalene, phenol, toluene, and xylenes. BaP was found in only one sample: Condition III, Day 2, Run 2, with a concentration of 0.214 ppb_{vd} and an emission rate realizing 0.0202 mg/sec.

Cresol and xylenes were not detected during any of the runs. Napthalene was detected during all but three runs. Xylene was erratically detected. Toluene was detected during all runs.

Tables 9-7a through 9-7d present, as measured in Stack 2, CAA non-targeted semi-volatile organic compound emissions data. Bis (2-ethylhexyl) phthalate and di-n-butylphthalate was detected in Stack 2 at least once during each condition. Phthalic anhydride, biphenyl, aniline, acetophenone, and butyl benzylphthlate were detected sporadically. Because semivolatile organic compounds were not consistently found, the data have not been weighted. Rather, the total emitted from the ovens are presented as an average per cycle. (See Table 9-8.)

9.5.1 Sample Analysis Summary

In an attempt to reduce the amount of analytical work and reduce costs, two samples, I-2A and 1V-3A, were chosen for a preliminary rapid extraction and analysis to determine if any of the sample fractions could be combined. The results indicated that erratic recoveries might be expected, and the decision was made to extract all fractions separately to monitor for any analytical anomalies.

Each fraction from each train was analyzed separately. Details of the separate fractions are presented in Tables 7-13 in Appendix D. This section presents a summary of these results. The results indicated, as expected, that recoveries from each fraction were erratic.

9.5.1.1 Front-half analysis -- The target analytes toluene, total xylenes, phenol, cresol, napthalene, and BaP demonstrated very erratic recoveries for the internal standards spiked onto the filter during the extraction phase. For example, for Condition III-2, Sample A had no recovery for phenol-d_5, cresol-d_7 or benzo-e-pyrene-d_{12}, whereas Sample B had approximately 40% recoveries for all of these spiked compounds. The difference between these samples is further demonstrated by the fact that the sample extracts for these two samples were observed to be brown versus light yellow. The origin of these differences is not known.

Similar differences were observed for Condition I-1 A and B; phenol-d_5 was not recovered in Sample A, but showed a 62% recovery in Sample B. Neither of these samples showed recovery of the benzo-a-pyrene-d_{12} spike. All of the other spiked compounds showed similar recoveries for these two samples. Since all of these spike compounds are prepared in a single spiking solution and added at the same time, these differences in recoveries must relate to a very selective chemical loss, presumably in the extraction process. No selective losses were observed for either the laboratory extraction blanks or the field blank, thus, pointing to something associated with the field sampled filters only, and possibly implicating the presence of particles on these filters.

Three of the four samples taken under Condition I showed no recovery of benzo-a-pyrene-d_{12}, and two of those three had no recovery of phenol-d_5. Condition II had generally good recoveries with one sample out of five (II-1B) having a low (14%) recovery of o-cresol-d_7. Condition IV had one sample out of six with no recovery of phenol-d_5, o-cresol-d_7, or benzo-a-pyrene-d_{12}. The other samples for Condition III were acceptable. Condition IV had one sample out of five with no recovery of benzo-a-pyrene-d_{12}, and two others (IV-1A and IV-1B) with low recoveries of phenol-d_5 or o-cresol-d_7 and benzo-a-pyrene-d_{12}.

The only consistently detected analyte on the front half filters was toluene. Since this is a common solvent in the extraction laboratory and was observed at comparable levels in the laboratory blanks, this must be attributed to the laboratory environment and not the field samples. Phenol was observed in one sample (III-1-B) and naphthalene in another (III-2-B).

Of the CAA analytes, the presence of di-n-butyl phthalate in several of the samples and bis(2-ethylhexyl) phthalate in two samples. All of the observations were at low levels and may well occur from some coincidental contact with plastic tubing in the lab or the field. The occurrence is not consistent or high enough to represent a true presence in the field samples.

Biphenyl was observed in one sample at approximately 8 µg, which is near the detection limit.

The Tentatively Identified Compounds (TICs) observed were acid esters, phthalates, siloxanes, an oxygenated butane and some hydrocarbons. No consistent pattern differences were noted between conditions, although Condition II had the most consistent appearance of the oxygenated butane compound. These data are not reported in this document.

The total mass observed as TICs for Condition I ranged from 51 to 257 µg, for Condition II from 14 to 1712 µg, for Condition III from 153 to 635 µg and for Condition IV from 41 to 565 µg.

9.5.1.2 <u>Back Half Results</u> -- The analytical results for the back-half fractions gave more consistent recoveries of the internal standard spikes, compared with the front half extractions. Sample I-1A showed no recovery for o-cresol-d_7H. No recovery of phenol-d_5 was observed for samples III-1B and IV-3A. Sample IV-3A not only gave no recovery of phenol-d_5, cresol-d_7 and benzo-a-pyrene-d_{12}, but also no peak was observed for perylene-d_{12}, which is added immediately before GC/MS analysis. To confirm this result, the extract was reanalyzed immediately after a successful analysis of the calibration solution and the same failure was observed on the Recovery Standards Solution C3 for perylene-d_{12}. The cause of this loss of perylene-d_{12} is unknown.

For the other field samples, the internal standard recoveries were generally in the 50% range. The average recoveries for the internal standards ranged from 47% for cresol-d_7 to 67% for xylene-d_{10}.

Toluene was again observed at comparable levels in the laboratory blanks and field samples. This must be attributed to the laboratory environment and not the field samples.

Phenol was observed in five of the 19 field samples, ranging from 6 to 108 µg. Because phenol was not consistently observed, it is difficult to consider it as a constant emission from this source for the back half fractions. Its corresponding prespiked surrogate compound, chlorophenol-d_4, was recovered in 17 of the 19 field samples with an average 96% recovery.

Naphthalene was observed in nine of the 19 field samples, ranging from 2 to 33 µg. Inexplicably, the dual trains reported no consistency in the naphthalene results as shown in Table 8 of Appendix D: I-1A had no detect, but I-1B showed 27 µg; II-1A had no detect, but II-1B showed 33 µg; II-2A showed 5 µg, but II-2B showed no detect; III-1A had no detect, but III-1B showed 26 µg; III-2A had no detect, but III-2B showed 17 µg; and IV-2A had no detect, but IV-2B showed 10 µg. Ignoring the results from II-2A which was near the detection limits, the average amount from I-1B, II-1B, III-1B, III-2B and IV-2B was 22.6 µg with a 40% RSD.

Because naphthalene was not consistently observed, it is difficult to consider it as a constant emission from this source for the back half fraction. However, under certain conditions, it was observed in the XAD fractions.

The XAD pre-spiked surrogates measured versus the internal standards all demonstrated recoveries near 100%. The average recovery for ethylbenzene-d_{10} was 118% with a 23% RSD. The average recovery for 2-chlorophenol-d_4 was 96% with a 42% RSD. The average recovery for acenaphthene-d_{10} was 103% with a 36% RSD. The average recovery for benzo-e-pyrene-d_{12} was 107% with a 46% RSD. Terphenyl-d_{14}, which is measured versus a recovery standard and is thus not corrected for lab losses, had an average recovery of 74% with a 32% RSD.

The CAA analytical results demonstrated the presence of di-n-butyl phthalate in six of the field samples and bis (2-ethylhexyl) phthalate in five field samples. Most of the observations were at low levels and may well occur from some coincidental contact with plastic tubing in the lab or the field. Several occurrences were in the range of 50 to 100 µg, and represent a more serious contamination problem. The occurrence is not consistent or high enough to represent a true presence in the field samples.

Acetophenone was observed in three samples. Chloroacetophenone was reported in both lab blanks, as well as the field blank, but not in the field samples. Inspection of the mass spectrum of this contaminant showed a compound whose mass spectrum was very similar, but not identical to that of chloroacetophenone. The origin of this contaminant is unknown. Biphenyl was observed in a single sample, a different sample than the front half observation.

The tentatively identified compounds observed were benzoic acid, benzoic acid esters, other acid esters, phthalates and an aromatic hydrocarbon. The benzoic acid was the dominant species observed, accounting for approximately 75% of the total mass associated with these TICs. No consistent pattern differences were noted between conditions.

The total mass observed as TICs for Condition I ranged from 764 to 1532 µg, for Condition II from 494 to 2351 µg, for Condition III from 1199 to 2240 µg, and for Condition IV from 212 to 1471 µg. These data are presented in Table 13 in Appendix D.

Emission rates have not been calculated.

9.5.1.3 Impinger Results -- There were no examples of complete loss of internal standards in the impinger extractions, although II-2A and III-3A had relatively low recoveries. This implies that the losses observed in the other fractions were associated with particulate matter. The front half fractions, containing the majority of these particles, demonstrated the most loss of standards during extraction. This was observed less frequently in the back half fractions and no example of complete loss of internal standards was seen in the impingers.

Toluene was again observed at comparable levels in the laboratory blanks and field samples. This must be attributed to the laboratory environment and not the field conditions.

Phenol was observed in two of the nineteen field samples, at 177 μg (Sample III-1-B) and 196 μg (Sample II-1B). Because these were not consistently observed, they could not be considered as a constant emission from this source for these fractions. However, the relatively large amount observed may indicate that this analyte occurs sporadically from this source. Sample II-1B back half contained 108 μg of phenol. Unfortunately, sample III-1B back half, had no recovery of the phenol-d_5 internal standard, and comparable results are not available. Sample II-1-B also contained approximately 70 μg of aniline.

The CAA analytical results demonstrated the presence of butyl benzyl phthalate in one field sample. The observation was at low levels and may well occur from some coincidental contact with plastic tubing in the lab or the field. The lab extraction blank had a large contamination with bis(2-ethylhexyl) phthalate, but no field samples showed any of this analyte. Aniline was observed at approximately 70 μg in sample II-1B.

Very few tentatively identified compounds were observed in the impinger fractions. These were primarily phthalates and some hydrocarbons. For sample II-1-B, three TICs were observed whose mass spectra were consistent with pyrazine and methyl pyrazines. This was the same sample found to contain aniline. No consistent pattern differences were noted between conditions.

The total mass observed as TICs for Condition I and Condition III was 0 μg. For Condition II the mass ranged from 0 to 782 μg and for Condition IV the mass ranged from 41 to 78 μg.

9.6 QA/QC Results

9.6.1 Field Blanks and Laboratory Blanks

The analytical results for the laboratory blank, field blanks, and reagent blanks for analytes are displayed in Tables 7 through 9 in Appendix D. Results for these blanks indicate no consistent contamination except for toluene, which is a common lab solvent. Because of this, toluene cannot be analyzed for by this method in this laboratory environment. Naphthalene was not observed in the laboratory blanks for this set of extractions. However, it is known to be a common contaminant of XAD resin, and can occasionally arise as a blank problem from XAD extractions. Benzo-a-pyrene was observed at the detection limit in the impinger lab blank. However, since no sample showed the presence of benzo-a-pyrene, this does not compromise the data set.

9.6.2 Surrogate Recovery Results

Results of the pre-spiked surrogate recoveries are presented in Table 8 in Appendix D. The average recoveries are within the guidelines of Method 301. The only problems encountered were with the loss of the corresponding internal standards, which was discussed earlier.

The XAD pre-spiked surrogates that were measured versus the internal standards all demonstrated recoveries near 100%. The average recovery for ethylbenzene-d_{10} was 118% with a 23% RSD. The average recovery for 2-chlorophenol-d_4 was 96% with a 42% RSD. The average recovery for acenaphthene-d_{10} was 103% with a 36% RSD. The average recovery for benzo-e-pyrene-d_{12} was 107% with a 46% RSD. Terphenyl-d_{14}, which is measured versus a recovery standard, and thus, is not corrected for lab losses, had an average recovery of 74% with a 32% RSD.

9.7 CONCLUSIONS

Tables 9-9 and 9-10 present the emission rates expressed as a range for the targeted semi-volatile compounds and the Clean Air Act semi-volatile compounds. To calculate this range, non-detects were assumed to be from 0 to the minimum detectable limit, which was 5μg for semi-volatiles. Therefore, since each sample cocsisted of three fractions which were analyzed separately, a non-detected compound had catch weights of 0 to 15 μg. Using this range for non-detects, concentration ranges were calculated for each train; these concentration ranges were averaged and combined with an average flow rate to get an average emission rate. The emission rates for the targeted compounds range from (0, 13) lb/yr for cresol and xylene to (91, 101) lb/yr for toluene. Since toluene is a common laboratory solvent and thwas detected in all field blank fractions at relatively high levels, these values for toluene should be used with care. Figure 9-3 illustrates the concentration of phenol per day, grouped by condition. Phenol was chosen to be representative of the targeted semi-volatile compounds. As can be seen in Figure 9-3, the presence of phenol was very erratic. On Day 1, there was a relatively large concentration of phenol during Conditions II and III, with no phenol detected during Condition IV. On Day 2, phenol was detected during Conditions II and III, but not during Conditions I and IV. On Day 3, phenol was only detected during Condition I. Overall, Condition II had the highest concentration of phenol across all three days.

The results from the front half extractions were surprising in exhibiting the extreme variability among duplicate samples from dual trains. The extreme recovery variability demonstrates the need for performing separate extractions and analyses on the individual fractions of the train, if one is interested in monitoring specific problem areas in the analysis, or at least performing separate analysis of the front half and back half combined with the condensate.

The use of isotopic labeled standards, prior to lab extraction, successfully allowed for correction for laboratory losses in the analytical process. This, in turn, enabled the achievement of surrogate recoveries within guidelines called for in Method 301. Without this correction for laboratory handling, the recoveries would have been in the 30 to 60% range.

Also, the use of isotopically labeled standards demonstrated that a recovery problem existed is the laboratory extraction stages. These problems may not be identified without the use of such standards.

The analytical complexity will be compounded by the addition of more analytes. The limits to which this type of methodology can be extended remains to be determined. Achieving Method 301 test criteria via dynamic spiking of the sampling train will require further discussion and evaluation.

9.8 MM5 DATA QUALIFIERS

Provided in this section is a list of data qualifiers. These include:

1. I-2-S2-MM5-2a had a recorded static pressure of -1.15. Because the value was recorded as such, it was used in the calculation of the flow rate. However, to be consistent with all other recorded static pressures, the number was probably -0.15. If -0.15 had been used, there would have been only a 0.08% difference in the emission rates.

2. During Condition I, Day 3, both probes broke at the end of the run. Train 3A was leak checked through the first impinger, and failed at 1 cfm. Train 3B failed at 6 cfm. Train 3A samples were analyzed.

3. During Condition I, Day 2, Run 2B lost probe heat; ran Train 2A only.

4. During Condition II, Day 1, Run 1B, leak check was wide open; there was also water in probe. However, the measured moisture content (~15%) was consistent with the typical moisture content for Stack 2. This indicates that the leak probably occurred at the end of the run. Therefore, the sample was analyzed.

5. Condition IV, Day 3, Run 3B, lost probe heat resulted in water on the filter; the run was unacceptable and not analyzed.

6. Condition II, Day 3, Run 3B not charged with DI water. The moisture was compared with the other runs and moisture data were comparable.

7. Condition II, Day 2, Run 2A did not record final gas volume; the final volume was extrapolated. However, if the last recorded gas volume had been used instead of the extrapolated volume, the emission rates would have changed by only 0.30%.

8. During Condition II, Day 3, Run 3b failed post-test leak check at 0.06 cfm. The filter caps were tightened and the train passed the leak check at 0.005 cfm. The samples were analyzed.

TABLE 9-1.

SEMI-VOLATILE ORGANIC GLASSWARE CLEANING PROCEDURE
(TRAIN COMPONENTS, SAMPLE CONTAINERS, AND LABORATORY GLASSWARE)

1. Soaked all glassware in hot soapy water (Alconox™).
2. Tap water rinsed to remove soap.
3. Rinsed 3 times with deionized water
4. Baked at 450 °F for 2 hours.
5. Rinsed 3 times with methylene chloride (pesticide grade).
6. Rinsed 3 times with methanol (pesticide grade).
7. Capped glassware with Teflon™ tape.
8. Marked cleaned glassware with color-coded identification sticker.

Step (4) has been added to the cleanup procedure to replace the dichromate soak specified in the reference method. Entropy has demonstrated that baking at 450 °F sufficiently removes organic artifacts. It is not used for probe liners and non-glass components of the train that cannot withstand 450 °F (i.e. Teflon™-coated filter screen and seals, tweezers, Teflon™ squeeze bottles, nylon probe and nozzle brushes).

TABLE 9-2. MODIFIED METHOD 5 SAMPLE FRACTIONS

Container	Code	Fraction
1	PRM	Probe, nozzle front half MeOH followed by $MeCl_2$ rinse,
2	GFF	Glass fiber filter,
3	XAD	XAD sorbent module
4	BHM	Back half solvent rinses of front filter housing back half, filter support, condenser coil and connecting glassware, plus impingers and connecting glassware.
5	CON	Impingers contents and HPLC water rinses of impingers and connecting glassware,
6	SiG	Silica gel

TABLE 9-3.
SAMPLE FRACTIONS TO BE COMBINED AND ANALYZED

Container Analysis No.	Sample Fractions	Type Analysis	Spiked Labeled Standards
1,2	Probe, nozzle, FH rinse with front filter (A)	Quantitative	Toluene d_8 o-Xylene d_{10} Phenol-d_5 o-Cresol d_7 Naphthalene d_8 Benz-a-pyene d_{12}
3,4	XAD sorbent combined with BHR (B)	Quantitative	Toluene d_8 o-Xylene d_{10} Phenol-d_5 o-Cresol d_7 Naphthalene d_8 Benz-a-pyene d_{12}
5	Impinger water (CON) (C)	Quantitative	Toluene d_8 o-Xylene d_{10} Phenol-d_5 o-Cresol d_7 Naphthalene d_8 Benz-a-pyene d_{12}

TABLE 9-4.

SEMI-VOLATILES - COKE OVEN
CAA COMPOUNDS

CAS NUMBER	CHEMICAL COMPOUND
62533	Aniline
92875	Benzidine
117817	Bis(2-ethylhexyl)phthalate (DEHP)
95487	o-Cresol (2-Methylphenol)
106445	p-Cresol (4-Methylphenol) co-elutes w/m-Cresol?
132649	**Dibenzofuran**
84742	Dibutylphthalate
106467*	1,4-Dichlorobenzene (p)
91941	3,3-Dichlorobenzidine
111444	**Dichloroethyl ether** (bis(2-chloroethyl)ether)
131113	Dimethyl Phthalate
51285	2,4-Dinitrophenol
121142	2,4-Dinitrotoluene
534521	4,6-Dinitro-o-cresol (2-Methyl-4,6-dinitrophenol)
118741	Hexachlorobenzene
87683	Hexachlorobutadiene
77474	Hexachlorocyclopentadiene
67721	Hexachloroethane
78591	Isophorone
91203	Naphthalene
98953	Nitrobenzene
100027	4-Nitrophenol
62759	N-Nitrosodimethylamine
87865	Pentachlorophenol
108952	Phenol
120821	1,2,4-Trichlorobenzene
95954	2,4,5-Trichlorophenol
88062	2,4,6-Trichlorophenol
98862	DERAcetophenone
53963	B2-Acetylaminofluorine
92671	B4-Aminobiphenyl
90040	Bo-Anisidine (2-Methoxyaniline)
98077	**Benzotrichloride** (a,a,a-Trichlorotoluene)
100447*	Benzyl chloride
92524	Biphenyl
120809	DERCatechol (1,2-Dihydroxybenzene)
532274	DER2-Chloroacetophenone
108394	m-Cresol (3-Methylphenol) co-elutes w/p-cresol?
96128	1,2-Dibromo-3-chloropropane

(Continued)

TABLE 9-4. (continued)

CAS NUMBER	CHEMICAL COMPOUND
91667	N,N-Diethyl aniline
121697	N,N-Dimethylaniline
119904	3,3'-Dimethoxybenzidine
119937	3,3'-Dimethylbenzidine
122667	1,2-Diphenylhydrazine
123319	DERHydroquinane (1,4-Dihydroxybenzene)
101144	B4,4'-Methylene bis(2-chloroaniline)
101779	B4,4'-Methylenedianiline
92933	4-Nitrobiphenyl
59892	N-Nitrosomorpholine
106503	Bp-Phenylenediamine
85449	Phthalic anhydride
106514	Quinone (1,4-Benzoquinone)
1746016	2,3,7,8-Tetrachlorodibenzo-p-dioxin (LR or HR analysis)
95807	B2,4-Toluene diamine
95534	Bo-Toluidine
1582098	Trifluralin (2,6-Dinitro N,N-dipropyl-a,a,a-trifluoro-p-toluidine)

* Compounds on both volatile and semi-volatile list.
B - BASE DER- Derivatization R- Reactive
Bold face type- CAAA first year compounds.

TABLE 9-5.

SUMMARY OF ANALYTICAL ACCEPTANCE CRITERIA ISOTOPE DILUTION SEMI-VOLATILE ORGANICS (MM5)

Stack Gas Parameter	Quality Parameter	Method of Determination	Frequency	Criteria
Semi-volatile Organics	Calibration	Five-level calibration curve; continuing calibration standard	At least once; at beginning of day; continue calibration once every 12 hours and at end	<30% RSD of avg RRF
	Calibration-- Initial Tuning	Hardware tuning using DFTPP*	Daily	Meet criteria of M8270 Sections 7.3 and 7.4
	Calibration-- Continuing	System performance check	Every 12 hours	<30% Deviation from initial calibration
	Accuracy -- Surrogates	Isotopically-labeled compound spiked into XAD prior to sampling	Once per test run series	70-130% recovery
	Precision-- Surrogates	Same as for accuracy	Once per test run series	<50% RsD of surrogate recovery
	Detection limit	Method must be reported in final report	Only if a sample is reported less than MDL	
	Blanks	Method blank for each set of like samples	One per batch of samples	Blank value <2DL; if greater, DL is changed to 1.5 times blank level
		Field blank carried through sample prep and analysis	One per test run series	Evaluated on a case-by-case basis

*Decafluoro Triphenylphosphine

TABLE 9-6a.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, TARGETED COMPOUNDS AS MEASURED IN STACK 2, CONDITION I - CHARGING JEWELL COAL & COKE

Repetition Number	I-1A	I-1B	I-2A	I-3A	Average
Toluene:					
Concentration, ppb dry	38.7	42.68	27.98	32.01	35.34
Emission Rate, mg/sec	1.497	1.658	1.148	1.225	1.382
Xylene:					
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00
Phenol:					
Concentration, ppb dry	0.539	0.00	0.00	0.786	0.3313
Emission Rate, mg/sec	0.0213	0.00	0.00	0.0307	0.013
Cresol:					
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00
Naphthalene:					
Concentration, ppb dry	0.00	1.80	0.00	0.358	0.5395
Emission Rate, mg/sec	0.00	0.0969	0.00	0.019	0.0290
Benzo-a-pyrene:					
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00

Zeroes indicate non-detects

TABLE 9-6b.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, TARGETED COMPOUNDS AS MEASURED IN STACK 2, CONDITION II JEWELL COAL & COKE

Repetition Number	II-1A	II-1B	II-2A	II-2B	II-3B	Average
Toluene:						
Concentration, ppb dry	16.64	23.15	99.36	33.84	36.72	41.94
Emission Rate, mg/sec	0.57	0.808	3.551	1.206	1.127	1.452
Xylene:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Phenol:						
Concentration, ppb dry	0.00	19.05	0.429	0.00	0.00	3.8958
Emission Rate, mg/sec	0.00	0.678	0.0156	0.00	0.00	0.1387
Cresol:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Naphthalene:						
Concentration, ppb dry	0.00	1.53	0.264	0.00	0.67	0.4928
Emission Rate, mg/sec	0.00	0.0743	0.0131	0.00	0.0286	0.0232
Benzo-a-pyrene:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00

Zeroes indicate non-detects

TABLE 9-6c.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, TARGETED COMPOUNDS AS MEASURED IN STACK 2, CONDITION III JEWELL COAL & COKE

Repetition Number	III-1A	III-1B	III-2A	III-2B	III-3B	Average
Toluene:						
Concentration, ppb dry	47.4	69.7	32.37	43.6	52.8	49.17
Emission Rate, mg/sec	1.439	2.079	1.123	1.506	1.714	1.572
Xylene:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Phenol:						
Concentration, ppb dry	0.00	23.8	0.00	0.884	0.00	4.9368
Emission Rate, mg/sec	0.00	0.726	0.00	0.0313	0.00	0.1515
Cresol:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Naphthalene:						
Concentration, ppb dry	0.00	2.02	0.00	0.952	0.00	0.5944
Emission Rate, mg/sec	0.00	0.0841	0.00	0.0458	0.00	0.0260
Benzo-a-pyrene:						
Concentration, ppb dry	0.00	0.00	0.00	0.214	0.00	0.0428
Emission Rate, mg/sec	0.00	0.00	0.00	0.0202	0.00	0.0040

Zeroes indicate non-detects

TABLE 9-6d.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, TARGETED COMPOUNDS AS MEASURED IN STACK 2, CONDITION IV JEWELL COAL & COKE

Repetition Number	IV-1A	IV-1B	IV-2A	IV-2B	IV-3A	Average
Toluene:						
Concentration, ppb dry	48.3	69.9	44.3	68	39.95	54.09
Emission Rate, mg/sec	1.536	2.226	1.508	2.302	1.298	1.774
Xylene:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Phenol:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Cresol:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00
Naphthalene:						
Concentration, ppb dry	0.00	0.00	0.00	0.68	0.0961	0.1552
Emission Rate, mg/sec	0.00	0.00	0.00	0.0321	0.0043	0.0073
Benzo-a-pyrene:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.00	0.00

Zeroes indicate non-detects

TABLE 9-7a.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, CLEAN AIR ACT COMPOUNDS AS MEASURED IN STACK 2, CONDITION I - CHARGING JEWELL COAL & COKE

Repetition Number	I-1A	I-1B	I-2A	I-3A	Average
bis(2-Ethylhexyl)phthalate:					
Concentration, ppb dry	0.32	0.00	0.00	1.09	0.3525
Emission Rate, mg/sec	0.0525	0.00	0.00	0.177	0.0574
Di-n-butylphthalate:					
Concentration, ppb dry	0.00	0.30	0.783	0.575	0.4145
Emission Rate, mg/sec	0.00	0.0351	0.0972	0.0665	0.0497
phthalic Anhydride:					
Concentration, ppb dry	0.00	0.00	11.90	0.00	2.975
Emission Rate, mg/sec	0.00	0.00	0.786	0.00	0.1965
Biphenyl:					
Concentration, ppb dry	0.00	0.00	0.00	0.860	0.2150
Emission Rate, mg/sec	0.00	0.00	0.00	0.0587	0.0147

Zeroes indicate non-detects

TABLE 9-7b.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, CLEAN AIR ACT COMPOUNDS
AS MEASURED IN STACK 2, CONDITION II
JEWELL COAL & COKE

Repetition Number	II-1A	II-1B	II-2A	II-2B	II-3B	Average
bis(2-Ethylhexyl)phthalate:						
Concentration, ppb dry	0.00	1.47	0.242	0.00	0.0887	0.3601
Emission Rate, mg/sec	0.00	0.217	0.0366	0.00	0.0115	0.0530
Di-n-butylphthalate:						
Concentration, ppb dry	0.00	0.00	1.207	0.00	0.00	0.2414
Emission Rate, mg/sec	0.00	0.00	0.13	0.00	0.00	0.0260
Aniline:						
Concentration, ppb dry	0.00	4.28	0.00	0.00	0.00	0.8560
Emission Rate, mg/sec	0.00	0.159	0.00	0.00	0.00	0.0318
Acetophenone:						
Concentration, ppb dry	0.00	0.749	0.723	0.00	0.00	0.2944
Emission Rate, mg/sec	0.00	0.0344	0.0339	0.00	0.00	0.0137
Butylbenzylphthalate:						
Concentration, ppb dry	0.00	0.00	1.39	0.00	0.00	0.2780
Emission Rate, mg/sec	0.00	0.00	0.168	0.00	0.00	0.0336

Zeroes indicate non-detects

TABLE 9-7c.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, CLEAN AIR ACT COMPOUNDS
AS MEASURED IN STACK 2, CONDITION III
JEWELL COAL & COKE

Repetition Number	III-1A	III-1B	III-2A	III-2B	III-3B	Average
<u>bis(2-Ethylhexyl)phthalate</u>:						
Concentration, ppb dry	1.81	1.36	0.00	0.273	0.00	0.6886
Emission Rate, mg/sec	0.24	0.173	0.00	0.04	0.00	0.0906
<u>Di-n-butylphthalate</u>:						
Concentration, ppb dry	0.00	1.792	0.559	0.00	0.00	0.4702
Emission Rate, mg/sec	0.00	0.162	0.0586	0.00	0.00	0.0441
<u>Biphenyl</u>:						
Concentration, ppb dry	0.00	0.469	0.00	0.00	0.00	0.0938
Emission Rate, mg/sec	0.00	0.025	0.00	0.00	0.00	0.0050
<u>Acetophenone</u>:						
Concentration, ppb dry	0.00	0.932	0.00	0.00	0.00	0.1864
Emission Rate, mg/sec	0.00	0.0366	0.00	0.00	0.00	0.0073
<u>Butylbenzylphthalate</u>:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.351	0.0702
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.0387	0.0077

Zeroes indicate non-detects

TABLE 9-7d.

SEMI-VOLATILE ORGANIC EMISSIONS DATA, CLEAN AIR ACT COMPOUNDS AS MEASURED IN STACK 2, CONDITION IV JEWELL COAL & COKE

Repetition Number	IV-1A	IV-1B	IV-2A	IV-2B	IV-3A	Average
bis(2-Ethylhexyl)phthalate:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.659	0.1318
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.0907	0.0181
Di-n-butylphthalate:						
Concentration, ppb dry	0.00	0.00	0.00	0.00	0.187	0.0374
Emission Rate, mg/sec	0.00	0.00	0.00	0.00	0.0184	0.0037

Zeroes indicate non-detects

TABLE 9-8.

SEMI-VOLATILE ORGANICS EMISSIONS DATA, TARGETED COMPOUNDS
TOTAL AVERAGES PER CYCLE
JEWELL COAL & COKE COMPANY

Day/Repetition Number	1	2	3	Average
Benzo-a-pyrene:				
Concentration, ppb$_{vd}$	0.00	0.0306	0.00	0.0102
Emission Rate, mg/sec	0.00	0.0115	0.00	0.00383
Cresol:				
Concentration, ppb$_{vd}$	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00
Naphthalene:				
Concentration, ppb$_{vd}$	0.6688	0.2709	0.2810	0.4069
Emission Rate, mg/sec	0.127	0.0516	0.0572	0.0786
Phenol:				
Concentration, ppb$_{vd}$	5.4236	0.1876	0.1965	1.9359
Emission Rate, mg/sec	0.756	0.0264	0.0294	0.271
Toluene:				
Concentration, ppb$_{vd}$	44.559	49.921	40.37	44.95
Emission Rate, mg/sec	6.08	6.88	5.92	6.29
Xylene:				
Concentration, ppb$_{vd}$	0.00	0.00	0.00	0.00
Emission Rate, mg/sec	0.00	0.00	0.00	0.00

Zeroes indicate non-detects.
Emission rates based on average daily flow rates.

TABLE 9-9.

SEMI-VOLATILE TARGETED COMPOUNDS TOTAL EMISSION RATES JEWELL COAL & COKE

COMPOUND	EMISSION RATE lb/yr
Toluene (uncorrected)	443
Toluene * (blank corrected)	(91, 101)
Xylene	(0, 13)
Phenol	(24, 35)
Cresol	(0, 13)
Naphthalene	(6, 17)
Benzo-a-pyrene	(0.3, 13)

Emission rates based on average of all measured flow rates.

* Toluene was present in the stack gas but, because it is a common laboratory solvent and was detected in all of the blanks at high levels, these calculated emission rates are approximate.

TABLE 9-10.

SEMI-VOLATILE CLEAN AIR ACT COMPOUNDS
TOTAL EMISSION RATES
JEWELL COAL & COKE

COMPOUND	EMISSION RATE lb/yr	COMPOUND	EMISSION RATE lb/yr
bis(2-Ethylhexyl)phthalate	(16, 27)	Di-n-butylphthalate	(8, 19)
Phthalic anhydride	(18, 31)	Biphenyl	(1, 14)
Aniline	(2, 15)	Acetophenone	(2, 14)
Butylbenzylphthalate	(3, 16)	n-Nitrodimethylamine	(0, 13)
Cumene	(0, 13)	a-Pinene	(0, 13)
b-Pinene	(0, 13)	1,2,4-Trimethylbenzene	(0, 13)
Benzyl chloride	(0, 13)	bis-(2-chloroethyl)ether	(0, 13)
n-Nitrosomorpholine	(0, 13)	1,4-Dichlorobenzene	(0, 13)
p-Cymene	(0, 13)	1,2-Dibro-3-chloropropane	(0, 13)
Hexachloroethane	(0, 13)	o-Toluidine	(0, 13)
2-Methylphenol	(0, 13)	Nitrobenzene	(0, 13)
N,N,-Dimethylaniline	(0, 13)	Isophorone	(0, 13)
Catechol	(0, 13)	3/4-Methylphenol	(0, 13)
1,2,4-Trichlorobenzene	(0, 13)	a-Terpineol	(0, 13)
o-Anisidine	(0, 13)	Hexachlorobutadiene	(0, 13)
2-Chloroacetophenone	(0, 13)	a,a,a-Trichlorotoluene	(0, 13)
N,N-Diethylaniline	(0, 13)	1,4-Phenylenediamine	(0, 13)

Emission rates based on average of all measured flow rates.

(continued)

TABLE 9-10. (cont.)

COMPOUND	EMISSION RATE lb/yr	COMPOUND	EMISSION RATE lb/yr
Hydroquinone	(0, 13)	Pentamethylbenzene	(0, 13)
Hexachlorocyclopentadiene	(0, 13)	2,4,6-Trichlorophenol	(0, 13)
2,4,5-Trichlorophenol	(0, 13)	2,4-Toluenediamine	(0, 13)
2,4-Dichlorophenol	(0, 13)	2,3-Dichlorophenol	(0, 13)
2,6-Dichlorophenol	(0, 13)	3,5-Dichlorophenol	(0, 13)
3,4-Dichlorophenol	(0, 13)	Dimethylphthalate	(0, 13)
2,4-Dinitrophenol	(0, 13)	4,6-Dinitro-2-methylphenol	(0, 13)
Dibenzofuran	(0, 13)	4-Nitrophenol	(0, 13)
Trifluralin	(0, 13)	Hexachlorobenzene	(0, 13)
4-Aminobiphenyl	(0, 13)	Pentachlorophenol	(0, 13)
Pentachloronitrobenzene	(0, 13)	4-Nitrobiphenyl	(0, 13)
Pyrene	(0, 13)	Benzidine	(0, 13)
4,4-Methylenedianaline	(0, 13)	Dimethylaminoazobenzene	(0, 13)
3,3-Dimethylbenzidine	(0, 13)	Methylene bis-chloroaniline	(0, 13)
Chrysene	(0, 13)	3,3-Dichlorobenzidine	(0, 13)
3,3-Dimethoxybenzidine	(0, 13)		

Emission rates based on average of all measured flow rates.

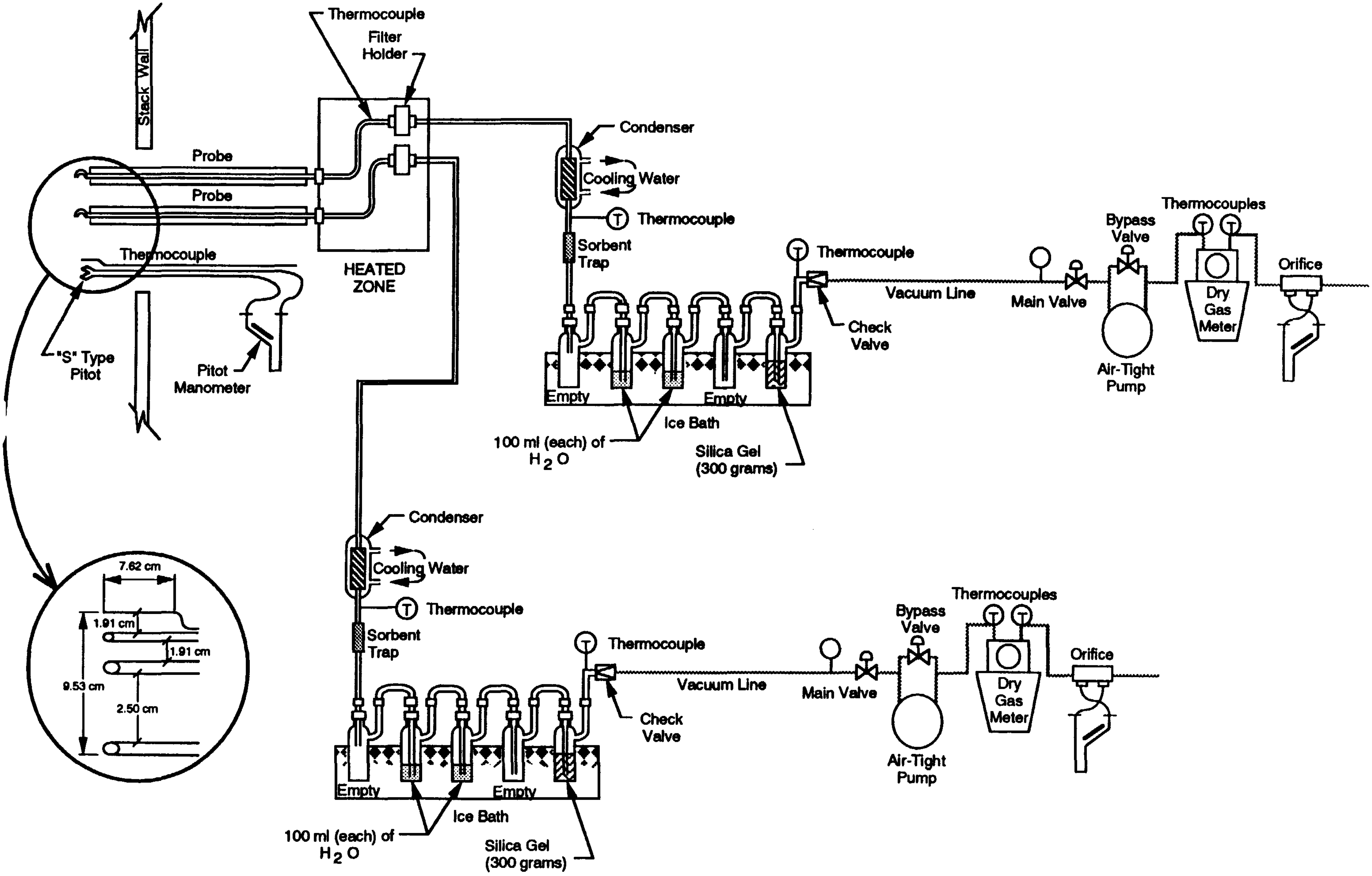

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Figure 9-1. Paired modified Method 5 sampling train.

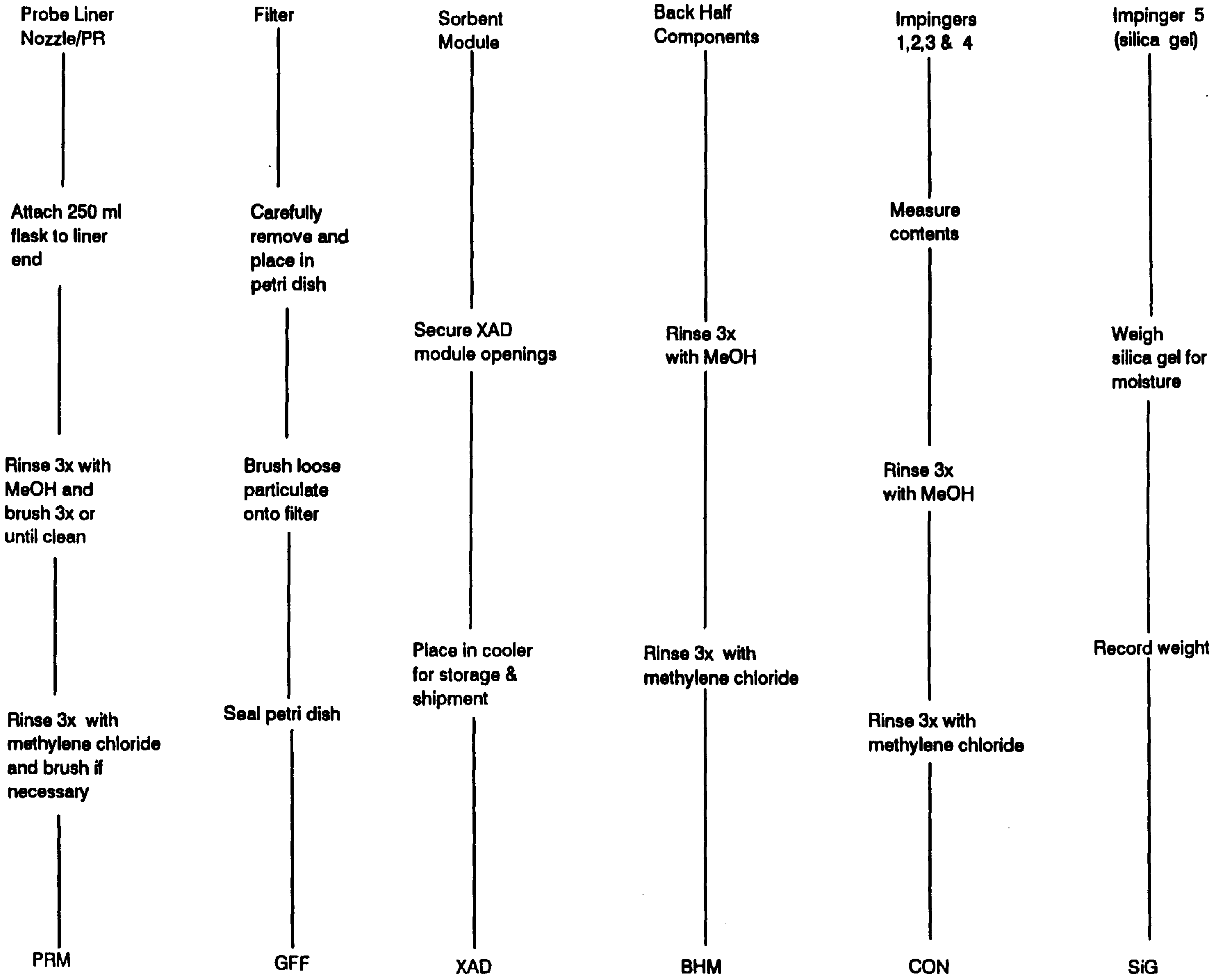
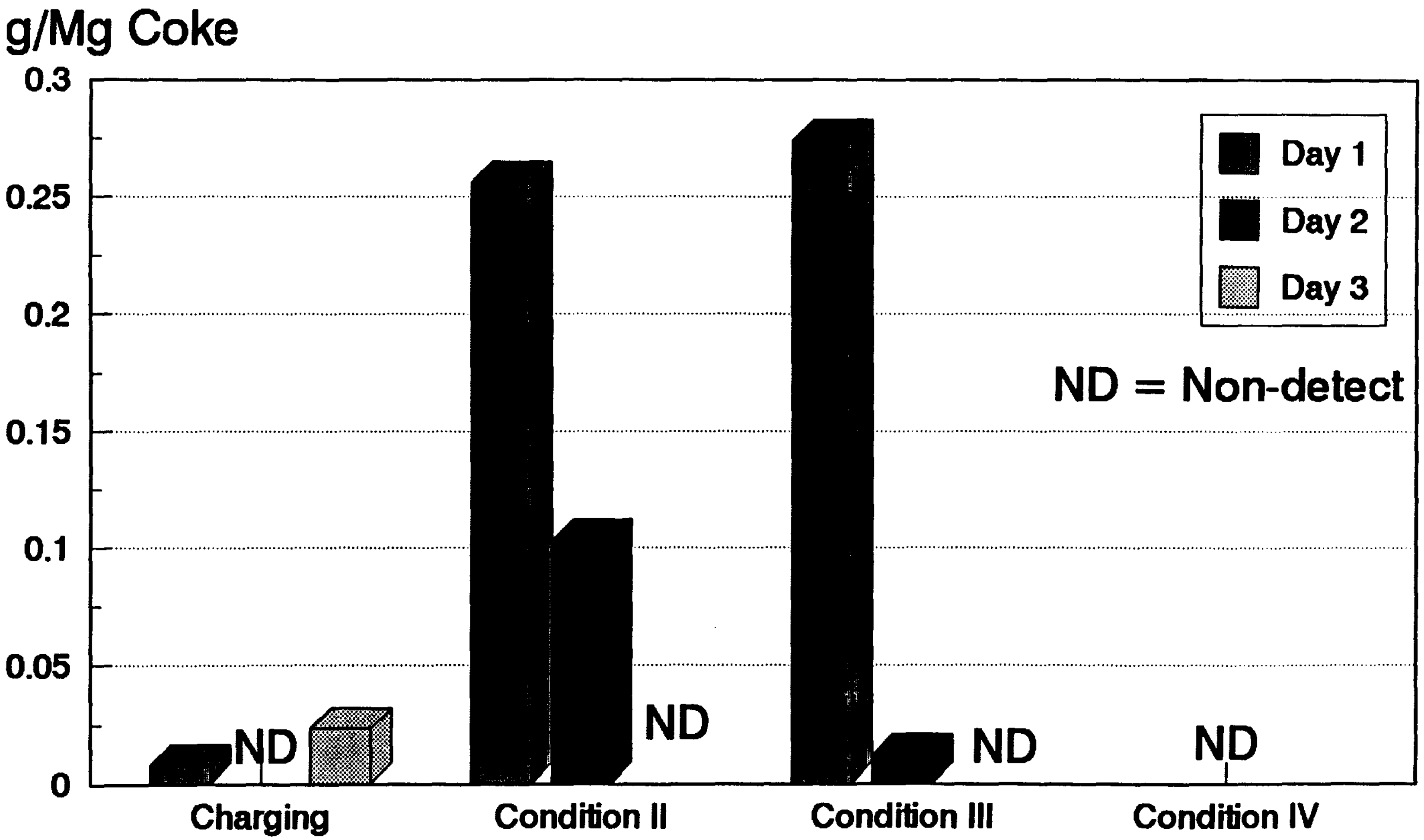


Figure 9-2. Modified Method 5 sample recovery scheme.

Phenol

from SEMI-VOST train



Jewell Thompson Non-Recovery Coke Oven
Minimum Detectable = 0.035g/Mg Coke

Figure 9-3.

10.0 VOLATILE ORGANIC COMPOUNDS

10.1 VOLATILE ORGANIC EMISSIONS TESTING (VOST)

The Volatile Organic Sampling Train (VOST) of SW-846 Method 0030 (reproduced in Appendix D) was used to collect volatile organic compounds. For this test SW-846 Method 0030 was considered validated for benzene and a scan was made for all other volatile organic compounds. The VOST sampling was conducted on Stack 3 and ran concurrently with the four CEM, MM5, M5/TSO/202 and MMTL runs. All samples were recovered; however, not all samples were analyzed. Table 10-1 presents the VOST sample and analysis test matrix.

Archived samples were collected as backups and clearly labeled "ARCHIVE," but were otherwise treated as any other sample. Each run produced a Tenax, a Tenax/Charcoal tube, and a condensate. Condensates collected during each sampling run were recovered separately, but once in the laboratory, TLI personnel consolidated all the condensates for that day's condition into one homogenous sample.

10.2 VOLATILE ORGANIC COMPOUND EQUIPMENT PREPARATION AND RECOVERY

10.2.1 VOST Sampling Equipment and Preparation

The VOST of SW-846 Method 0030 was used to collect the volatile organic samples. Figure 10-1 is a schematic of this train. The sampling equipment specifications are provided in the Method (see Appendix D); the only exception was the addition of an ice bath surrounding the Teflon™ probe to prevent melting.

The VOST equipment was cleaned and calibrated by Entropy; Triangle Laboratories, Inc. (TLI) prepared the sorbent cartridges used for sampling. All clean-ups followed the protocol established in Method 0030.

The volatile organic sampling train was assembled as shown in Figure 10-1. Samples were collected using paired Tenax and Tenax/charcoal cartridges, with each cartridge preceded by a condensing module. All sampling was performed according to the method specifications.

10.2.2 VOST Sample Recovery

The samples collected during each VOST run consisted of one pair of sorbent tubes (Tenax cartridge, Tenax/charcoal cartridge) and the condensate, and were recovered and stored according to the appropriate sections of Method 0030.

During Condition I each day, and Condition IV of Day 3, one pair of Tenax and Tenax/charcoal cartridges, serving as field blanks, were taken to the sampling site and the end caps removed for the period of time required to exchange two pairs of traps on the VOST. After the VOST traps had been exchanged, the end caps were replaced on the field blanks, and they were stored and analyzed with the field samples.

A second pair of blank cartridges was included in the cartridge shipment to the test site. These trip blanks were treated like the other cartridges except that the end caps were not removed during storage at the site. These were not analyzed due to breakage in shipment.

One pair of blank cartridges, a laboratory blank, remained stored in the laboratory. This blank was analyzed only if the field or trip blanks showed high contaminant concentrations.

10.3 VOST SAMPLE ANALYSIS

The major objective was to analyze air samples collected from a coke oven facility, in accordance with the guidelines of Methods 8240 and 5040, for 40 target volatile organic compounds selected from the Clean Air Act (CAA) list, and 21 additional 8240 organic compounds associated with the coke oven industry. These compounds were analyzed quantitatively. In addition, qualitative analyses for other major organic components were compared for agreement and reported as tentatively identified compounds (TICs). The target analytes are listed in Table 10-2. The primary analytes of interest were benzene and toluene, and the QC criteria were designed for those analytes.

Analysis for the targeted volatile organics was performed following the procedures specified in EPA Methods 8240 and 5040. Other non-target compounds were matched against two separate libraries, the National Bureau of Standards (NBS) and the Clean Air Act (CAA) library constructed by Triangle Labs from the mass spectra of the CAA compounds.

The volatile organic contents of each cartridge and condensate sample were purged onto an analytical absorbent column in a purge and trap system. Subsequent thermal desorption onto a GC/MS system completed the analysis. The tenax "front" cartridge was analyzed separately from the Tenax/charcoal cartridge; likewise, the condensate fractions were analyzed separately.

10.3.1 Prespiking of Surrogates

The Tenax-only tube for all samples was pre-spiked prior to field sampling with the labeled compound, o-xylene-d_{10} at 0.25 μg. This labeled analyte was chosen on the basis of availability and because preliminary tests indicated that the potential for interference for this analyte was negligible. The recovery of the surrogate analyte was used to give an indication of the overall sampling and recovery efficiency. The surrogate was directly introduced (in a methanol solution) into the inlet end of the Tenax trap via syringe, immediately before sealing and packing the traps for shipping.

10.3.2 Analytical Methodology

The analysis was based on the guidelines of Methods 8240 and 5040. An initial calibration using the internal standards (bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d_5), the analytes of interest and the labeled surrogates (toluene-d_8, 1-2dichloroethane-d_4, benzene-d_6, 4-bromofluorobenzene, and xylene-d_{10}) was performed as described in Section 1.6 of Appendix D. After GC/MS calibration but immediately prior to analysis by GC/MS, the VOST tube, or an aliquot of the condensate sample, was fortified with the above internal standards and surrogates such that the amount of internal standards and surrogates was 0.25 μg per sample. For the condensate analysis, the same amount of internal standards and surrogates was added to a 5 mL sample aliquot yielding a concentration of 50 μg/L (50 parts per billion, ppb). Specifics concerning instruments used, calibrations, and calculations can be found in the full text in Appendix D.

10.3.3 Data Reporting and Targets, "Find" Databases, and NBS and CAA Library Searches for TICs

10.3.3.1 Targets and "Find" Databases -- Prior to analysis, a library of reference mass spectra and retention time information was assembled into a database, referred to as a "Find" database in LAB-BASE terminology. A quantitation mass, either the molecular ion or the most intense peak in its mass spectrum, was chosen for each of the analytes in the "Find" database. After the GC/MS analysis, the data file retention time window was searched for the occurrence of any mass spectrum containing the quantitation mass for a given analyte. The plot of the intensity of this quantitation mass versus time was the selected ion current profile (SICP) for that mass. The area under that particular peak was used to quantitatively measure the amount of the compound present. If a peak occurs at the proper time and mass for a target analyte, the compound's mass spectrum is compared to the library spectrum stored in the database. The compound was considered detected if its match was above the selected match tolerance. The resulting mass spectra of detected targets are plotted versus their corresponding library spectra. A trained chemist then evaluates whether the match is correct or not. If not, the compound was removed from the list of detected target compounds. For those detected target compounds, the concentration was calculated as described in Section 1.4 of Appendix D.

10.3.3.2 NBS and CAA Library Searches for TICs -- Besides the target compounds, there are generally a number of non-target components that are observed in the chromatogram. The mass spectrum of these compounds is searched against two separate libraries. The National Bureau of Standard's (NBS) library contains the mass spectra of 49, 469 compounds and is typically used to identify an unknown mass spectrum. The CAA library has been constructed by TLI from the mass spectra of the Clean Air Act compounds from the NBS library as well as some spectra generated at TLI. By searching an unknown spectrum against both libraries, one maximizes the possibility of finding CAA compounds while also comparing to the mass spectra of the NBS library. Any compounds that are observed and identified are referred to as TICs, since there is no reference standard analyzed at the same time as the unknown analyte. The quantitation that is given to these TICs is based on the area of the peak compared directly to the area of the nearest internal standard and assumes a response factor of one. The error caused by this approximation is unknown.

10.3.4 Quantitative and Semi-Quantitative Analysis

The analyses of the 40 targeted volatile organic compounds selected from the CAA list and the 21 additional targeted organic compounds were performed quantitatively. The analytical results of benzene and toluene are considered quantitative results. However, the reported data for the other 59 targeted analytes are considered semi-quantitative since calibration criteria were not met for some analytes; the measured results were outside the calibration ranges.

Prior to analysis, a library of reference mass spectra and retention time information was assembled into a database. A quantitation mass, either the molecular ion or the most intense peak in its mass spectrum, was chosen for each of the analytes in the "Find" database. After the GC/MS analysis, the data file retention time window was searched for the occurrence of any mass spectra containing the quantitation mass for a given analyte. The plot of the intensity of this quantitation mass versus time was the selected ion current profile (SICP) for that mass. The area under that particular peak was used to quantitatively measure the amount of the compound present. If a peak occurred at the proper time and mass for a target analyte, the compound's mass spectrum was compared to the library spectrum stored in the database. The compound was considered detected if its match was above the selected match tolerance. The resulting mass spectra of detected targets were plotted versus their corresponding library spectra. A trained chemist then evaluated whether the match was correct or not. If not, the compound was removed from the list of detected target compounds. For those detected target compounds, the concentration was calculated. Acrylonitrile and 1,3-butadiene co-elude. The quantitation mass for acrylonitrile is 53. However, the 1,3-butadiene spectrum contains a mass of 53 which may artificially inflate acrylonitrile values when 1,3 butadiene is present.

10.3.5 Qualitative Analysis

Besides the target compounds, there were generally a number of non-target components that were observed in the chromatogram. The mass spectrum of these compounds was searched against two separate libraries. The National Bureau of Standard's (NBS) library contains the mass spectra of 49,469 compounds and is typically used to identify an unknown mass spectrum. The CAA library has been constructed by TLI from the mass spectra of the Clean Air Act compounds from the NBS library, as well as some spectra generated by TLI. By searching an unknown spectrum against both libraries, one maximizes the possibility of finding CAA compounds while also comparing the mass spectra of the NBS library. Any compounds that are observed and identified are referred to as tentatively identified compounds (TICS), since there is no reference standard analyzed at the same time as the unknown analyte. The quantitation that is given to these TICS is based on the area of the peak compared directly to the area of the nearest internal standard and assumes a response factor of one. The error caused by this approximation is unknown.

10.4 VOLATILE ANALYSIS QUALITY CONTROL

The major QC procedures contained in the method included GC/MS tuning, calibrations system performance checks, and analysis of specific QC check samples. During actual sample measurements, field blanks, a laboratory control spike, and a laboratory method blank were analyzed with the field samples. The criteria for acceptable performance for the QC measurements are listed in Table 10-3. The quality of the analysis was assured through reproducible calibration and testing of the purge and trap and GC/MS system.

10.5 SUMMARY OF RESULTS

Tables 10-4 through 10-7 present the summaries of results for the as measured data in Stack 3 concentration and emission rates for Conditions I, II, III, and IV, respectively. The reported data are the averages per condition of detected compounds. Benzene and toluene are the quantitative results and are presented before other analytes. The only analytes present in the tables were those detected during any of the runs. Benzene and toluene were detected during all four conditions. The full data package reporting the mass for each fraction can be found in Appendix D.

The benzene concentrations were highly variable during Conditions I, III, and IV when viewed against an overall average for all four conditions. Condition I reported 358 μg/dscm; Condition III reported 897 μg/dscm; and Condition IV reported 667 μg/dscf. The average, excluding these values, was 5.5 μg/dscm. The toluene did not track with the benzene. Because the concentration and flow rate data were so consistent from condition to condition, a weighted average was not calculated. Rather, total daily averages were calculated.

Figures 10-2 and 10-3 illustrate the concentrations of benzene and toluene, respectively. The graphs are grouped by condition and compare the three days within each condition. The concentrations are reported as grams per miligram of coke charged. As can be seen on Figure 10-2, for Condition I, charging, benzene concentrations for Days 1 and 3 were relatively the same at less than 0.25 g/mg coke; however, Day 2 showed an increase in concentration at approximately 0.75 g/mg coke. For Condition II, the concentrations of benzene for all three days was less than 0.25 g/mg coke. For

Conditions III and IV, the concentrations of benzene were much higher for Day 1 than the other two days; during both conditions, the concentrations for Day 1 were approximately 2.25 g/mg coke. From Figure 10-3 it can be seen that the concentrations of toluene remained relatively stable across days and conditions with two extreme exceptions. During Conditions I and II for Day 3, the concentrations of toluene were approximately 0.7 g/mg coke; the concentrations for Days 1 and 2 were approximately 0.3 g/mg coke during Conditions I and II.

Other analytes detected follow the benzene and toluene results. Although actual weighted averages have not been calculated, total data have been estimated and are presented in Table 10-8. These data have been calculated assuming the concentration was the same in each of the stacks with an emission rate calculated from each stack and added together. TICs have not been calculated. Table 10-9 presents the total emission rates for the volatile compounds expressed as a range in pounds per year. The emission ranges were calculated following the same procedures outlined in Section 9.7 for semi-volatile compounds except the minimum detectable limit for volatile compounds was assumed to be 50 ng. The emission rates ranged from (0, 16) lb/yr for o-xylene to (2849, 2865) lb/yr for acrolein. This high number for acrolein is unexplained. However, acrolein is common in woodsmoke and since acrolein appeared in only one sample, it is possible that contamination occurred from the forest fires in the area.

10.5.1 <u>Tenax Results</u>

The non-target compounds found in the Tenax tubes were primarily sulfur dioxide, substituted hydrocarbons, siloxanes, and silanols. Sulfur dioxide accounted for the largest quantity of the TICs in most of the analyses. In several runs (Condition IV, days 1 and 2), the siloxanes were observed in large amounts, and sulfur dioxide was not identified. The total mass observed as TICs for Condition I ranged from 12 to 50 μg, for Condition II from 24 to 80 μg, for Condition III from 40 to 140 μg, and for Condition IV from 5 to 210 μg.

Most samples showed the presence of a large peak around scan number 200 that had a mass spectrum that matched that of sulfur dioxide; for those samples the peak was identified as sulfur dioxide. However, a few of the samples showed the presence of another peak near scan number 500 that had a spectrum that also matched that of sulfur dioxide; for those samples, the peak was labeled as Unknown.

10.5.2 Tenax-Charcoal Results

Benzene and toluene were both consistently observed on the Tenax-Charcoal tubes. Benzene levels were less (10-30% of the values) than on the corresponding Tenax tubes and probably represent a small amount of breakthrough. The toluene values were generally higher than those observed on the corresponding Tenax tubes. IF this is the result of breakthrough, it is unclear why benzene did not exhibit the same phenomenon. The corresponding field and laboratory blanks did NOT show evidence of contamination.

Bromomethane and chloromethane were detected at concentrations ranging from 0.23 to 2.02 μg and 0.38 to 3.39 μg, respectively. These must represent breakthrough since the two analytes were only observed in two of the Tenax tubes.

Methylene chloride was observed in 19 of the 21 Tenax-Charcoal tubes, at higher amounts (from 0.029 to 0.345 μg) than the corresponding Tenax tubes. Two of the four field blanks contained methylene chloride at 0.026 and 0.095 μg levels.

Hexane, isooctane, carbon disulfide, trichlorofluoromethane and acetone were sporadically observed; these levels were generally low and would not be considered constant emissions in these samples.

The non-target compounds found in the samples were primarily sulfur dioxide, substituted hydrocarbons, siloxanes, silanes and silanols. Sulfur dioxide accounted for the largest quantity of the TICs in most of the analyses. In several runs, siloxanes were observed in large amounts, and sulfur dioxide was not identified or was a lesser component. The total mass observed as TICs for Condition I ranged from 0.4 to 7 ug, for Condition II from 1.5 to

2 ug, for Condition III from 2 to 36 ug, and Condition IV from 0.8 to 28 ug. The amount of material observed on the Tenax-Charcoal tubes was usually less than 10% of that observed on the corresponding Tenax tubes, and probably represents a small amount of breakthrough.

Most samples showed the presence of a large peak around the scan number 200 that had a mass spectrum that matched that of sulfur dioxide; for those samples the peak was identified as sulfur dioxide. However, a few of the samples showed the presence of another peak around the scan number 500 that has a spectrum that also matches that of sulfur dioxide; for those samples, the peak was labeled as Unknown.

Sample I-1-S3-V-1B TC was cracked and was therefore repacked after it was spiked with internal and surrogate standards. This affected the internal standard areas. It accounts for the low 1,2-dichloroethane-d_4 and the unusually high toluene-d_8 and 4-bromofluorobenzene recoveries. The surrogate standard recoveries are unusually high and the sample results are suspect.

10.5.3 Condensate Results

Benzene was not observed in any of the condensate analyses. Toluene was consistently observed in the condensates. However, toluene was also observed in the field blanks at levels similar to those observed in the field samples. The levels of toluene observed in the laboratory blanks were much lower than those observed in the field blanks and samples. Therefore, the toluene observed in the field samples can be attributed to contamination found in the field.

Benzene and toluene concentrations did not exceed the calibration range for any of the samples. Although concentrations above 200 µg/L were reported for certain analytes, these are qualitative values only.

Major components observed in the condensates were dibromomethane, 2-butanone, methylene chloride, toluene, acetone, and 2-hexanone. Methylene chloride was detected at concentrations ranging from 10.1 to 96.2 µg/L and acetone was detected at concentrations ranging from 70.4 to 246.9 µg/L. The absence of high levels of methylene chloride and acetone in the Tenax and Tenax-Charcoal tube analyses and the presence of them in the condensate field blanks implies a field or laboratory contamination problem. Acetone was observed in the laboratory blanks at 20 or less µg/L but up to 262 µg/L in the field blanks. Methylene chloride was observed at less than 1 µg/L in the laboratory blanks but up to 102 µg/L in the field blanks. This leads one to believe that a field contamination problem existed.

The analyte 2-butanone was detected at concentrations ranging from 0.7 to 30.7 µg/L, but it was also detected in the laboratory blanks (up to 2.7 µg/L) and in the field blanks (up to 8.8 µg/L). This compound is a known contaminant of methanol used in the analysis. The levels observed were not significantly higher than those in the lab blanks. Conceivably, 2-butanone could be a result of contamination from the analytical effort.

2-Hexanone was detected in many of the field samples at concentrations ranging from 0.6 to 19 µg/L, but it was also detected in the laboratory blanks (up to 2.5 µg/L) and in the field blanks (up to 3.9 µg/L).

Ethylbenzene, m/p-xylene, dibromomethane and o-xylene were all observed at blank levels similar to those seen in the field blanks. Thus, none of these compounds can be attributed to the source in this analytical fraction. A compound identified as acrolein was observed in three samples. A similar compound was detected in one of the field blanks. The mass spectrum contained additional peaks NOT consistent with the spectrum of acrolein.

There were not many TICs observed in the condensate samples. The identified compounds included siloxanes, hydrocarbons, methyl ethyl benzene and some aldehydes. The total amounts observed as TICs for Condition I ranged from 1.2 to 1.8 µg, for Condition II from 0.2 to 1.3 µg, for Condition III from 0.2 to 4.8 µg, and Condition IV from 0.1 to 6.9 µg. These values represent total amounts detected in the combined condensate fractions and calculated by multiplying the volume of the combined condensate (in L) by the concentration (in µg/L).

10.6 DATA QUALIFIERS

(1). Sample IV-3-S3-V-3A TC was inadvertently spiked with 0.167 μg of internal standards and o-xylene-d_{10} and with 0.334 μg of other surrogate standards. All surrogate recoveries are within the acceptable limits of the method and the results appear to be unaffected.

(2). Sample I-1-S3-V-1B TC was cracked and was therefore repacked after it was spiked with internal and surrogate standards. This affected the internal standards area. It accounts for the low 1,2-dichloroethane-d_4 and the unusually high toluene-d_8 and 4-bromofluorobenzene recoveries. The surrogate standard recoveries are unusually high and the sample results are suspect.

TABLE 10-1.

VOST SAMPLING AND ANALYSIS TEST MATRIX

Run No.	Fraction	Analytical Treatment
I-1-53-V-1A	TNX, T/C	Analyzed
I-1-53-V-1B	TNX, T/C	Analyzed
I-1-53-V-1C	TNX, T/C	Analyzed
I-1-53-C-1D	TNX, T/C	Analyzed
I-1-53-V-1A,1B,1C,1D	Condensate, Consolidated	Analyzed
II-1-53-V-1A	TNX, T/C	Analyzed
II-1-53-V-1B	TNX, T/C	Analyzed
II-1-53-V-1C	TNX, T/C	Archived
II-1-53-V-1A,1B,1C	Condensate, Consolidated	Analyzed
III-1-53-V-1A	TNX, T/C	Archived
III-1-53-V-1B	TNX, T/C	Analyzed
III-1-53-V-1C	TNX, T/C	Analyzed
III-1-53-V-1A,1B,1C	Condensate, Consolidated	Analyzed
IV-1-53-V-1A	TNX, T/C	Analyzed
IV-1-53-V-1B	TNX, T/C	Archived
IV-1-53-V-1C	TNX, T/C	Analyzed
IV-1-53-V-1A,1B,1C	Condensate, Consolidated	Analyzed
I-2-53-V-2A	TNX, T/C	Analyzed
I-2-53-V-2B	TNX, T/C	Analyzed
I-2-53-V-2C	TNX, T/C	Analyzed
I-2-53-V-2D	TNX, T/C	Analyzed
I-2-53-V-1A,1B,1C,1D	TNX, T/C	Analyzed
II-2-53-V-1A	TNX, T/C	Analyzed
II-2-53-V-1B	TNX, T/C	Archived
II-2-53-V-1A,1B	Condensate, Consolidated	Analyzed
III-2-53-V-2A	TNX, T/C	Archived
III-2-53-V-2B	TNX, T/C	Analyzed
III-2-53-V-2A,2B	Condensate, Consolidated	Analyzed
IV-2-53-V-2A	TNX, T/C	Analyzed
IV-2-53-V-2B	TNX, T/C	Archived
IV-2-53-V-2A,2B	Condensate, Consolidated	Analyzed

(continued)

TABLE 10-1. (continued)

Run No.	Fraction	Analytical Treatment
I-3-53-V-3A	TNX, T/C	Analyzed
I-3-53-V-3A	Condensate	Analyzed
II-3-53-V-1A	TNX, T/C	Lost Sample
II-3-53-V-1B	TNX, T/C	Analyzed
II-3-53-V-1C	TNX, T/C	Archived
II-3-53-V-1A,1B,1C	Condensate, Consolidatd	Analyzed
III-3-53-V-3A	TNX, T/C	Lost Sample
III-3-53-V-3B	TNX, T/C	Archived
III-3-53-V,3A,3B	Condensate, Colsolidated	Analyzed
IV-3-53-V-1A	TNX, T/C	Analyzed
IV-3-53-V-1A	Condensate	Analyzed

TABLE 10-2.
VOLATILE COMPOUNDS ON THE CAA LIST
OCTOBER 1, 1991
(air/water matrices; approximate boiling points 30°C to 130°C)

CAS NUMBER	CHEMICAL COMPOUND
4107028	WS Acrolein (bp 53°C)
107131	WS **Acrylonitrile** (bp 77°C)
107051	Allyl chloride (3-Chloropropene) (bp 45°C)
71432	**Benzene** (bp 80°C)
100447	SV Benzyl chloride (Alpha-chlorotoluene) (bp 179°C)
75252*	Bromoform (bp 150°C)
106990	**1,3-Butadiene** (bp -4.5°C)
75150	Carbon Disulfide (bp 46°C)
56235	Carbon Tetrachloride (bp 77°C)
108907*	Chlorobenzene (bp 132°C)
67663	Chloroform (bp 61°C)
126998	**Chloroprene** (2-Chloro 1,3-butadiene) (bp 59°C)
98828	SV Cumene (bp 153°C)
106467	SV 1,4-Dichlorobenzene(p) (bp 174°C)
	cis-1,3-Dichloropropene (bp 108°C)
542756	**trans-1,3-Dichloropropene** (bp 108°C)
100414*	Ethyl Benzene (bp 136°C)
106934	**Ethylene dibromide** (Dibromethane) (bp 131°C)
75343	Ethylidene dichloride (1,1-Dichloroethane) (bp 57°C)
107062	Ethylene dichloride (1,2-Dichloroethane) (bp 83°C)
110543	Hexane (bp 69°C)
74839*	Methyl Bromide (Bromomethane) (bp 4°C)
74873*	Methyl Chloride (Chloromethane) (bp -24°C)
71556	Methyl Chloroform (1,1,1-Trichloroethane) (bp 75°C)
78933	WS Methyl Ethyl Ketone (2-Butanone) (bp 80°C)
74884	Methyl Iodide (Iodomethane) (bp 43°C)
108101	WS Methyl isobutyl ketone (4-methyl-2-pentanone) (bp 118°C)
75092	Methylene Chloride (Dichloromethane) (bp 40°C)
1634044	MS Methyl tert butyl ether (bp 55°C)
78875	Propylene dichloride (1,2-Dichloropropane) (bp 96°C)
100425*	Styrene (bp 145°C)
79345*	**1,1,2,2-Tetrachloroethane** (bp 147°C)
127184	Tetrachloroethylene (perchloroethylene) (bp 121°C)
108883	Toluene (bp 110°C)
79005	1,1,2-Trichloroethane (bp 115°C)
79016	Trichloroethylene (Trichlorothene) (bp 87°C)
540841	2,2,4-Trimethylpentane (Isooctane) (bp 98°C)
108054	WS Vinyl Acetate (bp 73°C)
593602	Vinyl bromide (bp 16°C)
75014*	**Vinyl chloride** (bp -13°C)

(Continued)

TABLE 10-2. (continued)

CAS NUMBER	CHEMICAL COMPOUND
75354	**Vinylidene chloride** (1,1-Dichloroethylene) (bp 32°C)
95476*	o-Xylenes (bp 144°C)
108383*	m-Xylenes (reported combined w/ p-Xylenes) (bp 139°C)
106423*	p-Xylenes (reported combined w/ m-Xylenes) (bp 139°C)

* Analysis can be performed; however, results may not be quantitative.
Bold Face Type- CAA first year compounds.
WS - Water soluble compounds.
SV - Can be analyzed as semi-volatile compound also.

TABLE 10-3.

SUMMARY OF ANALYTICAL ACCEPTANCE CRITERIA METHOD 8240 VOLATILE ORGANICS (VOST)

Stack Gas Parameter	Quality Parameter	Method of Determination	Frequency	Criteria
Volatile Organics	Field Blanks--sample integrity and field contamination	Field blanks-1 pair of traps	One pair per day	Less than lowest standard
	Trip blanks--verify no cross-contamination in storage and shipment	Lab blanks - 1 pair of traps	One pair of traps were analysed in tandem	Less than lowest standard
	Lab blanks--verify no lab contamination and system control	Lab blanks - 1 pair of traps	Daily, before sample analysis every 12 hours	Less than lowest standard
	Calibration--initial	5-point calibration	Prior to sample analysis	Variability of avg RRF <30% RSD
	Calibration--mass	Hardware tuning using BFB	Every 12 hours before every continuing calibration	Meet criteria of M8240 Section 7.2
	Calibration--continuing	System performance check compounds	Every 12 hours before every continuing calibration	Meet criteria of M8240 Section 7.3
	Chromatography--consistency	Monitor internal std; retention time and area	Every sample, standard, and blank	Relative retention time (RRT) within 0.06 RRT units of daily standard
	Precision and accuracy	Analysis of each fraction spiked with a standard independent of calibration standards	Demonstrated prior to sample analysis	50-150% recovery
	Accuracy--continuing check	Spike each sample with surrogate compounds	Every sample	Within 50-150% recovery; within 25% RSD
	Detection limit	Method must be reported in final report	At least once for principle constituents	NA

TABLE 10-4.

VOLATILE ORGANIC EMISSIONS DATA
AS MEASURED IN STACK 3, CONDITION I - CHARGING
JEWELL COAL & COKE COMPANY

Run Identification	I-S3-VOST			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	1715	1744	1905	
Run Finish Time	1955	2030	1945	
Total Run Time, min.	160	166	40	
<u>Test Train Parameter</u>:				
Volume of Metered Dry Gas Sample, SL[a]	72.986	80.545	21.357	
<u>Flue Gas Parameters</u>:				
CO_2, Percent by Volume, Dry	6.1	7.0	6.9	
O_2, Percent by Volume, Dry	11.2	10.1	10.3	
Flue Gas Temperature, Degrees F	1,519	1,555	1,543	
Velocity, ft/sec	32.56	31.62	37.56	
Air Flow Rate, Dry, SCFM[a]	22,623	20,987	25,490	
Air Flow Rate, Wet, ACFM	102,331	99,366	118,038	
<u>Benzene Emission</u>:				
Concentration, μg/DSCM[a]	3.93	89.30	1.80	31.68
Emission Rate, lb/hr	3.33E-4	7.05E-3	1.72E-4	2.52E-3
<u>Toluene</u>:				
Concentration, μg/DSCM[a]	35.43	18.85	77.1	43.79
Emission Rate, lb/hr	3.00E-3	1.48E-3	7.36E-3	3.95E-3
<u>Chloromethane</u>:				
Concentration, μg/DSCM[a]	62.38	56.25	109	75.88
Emission Rate, lb/hr	5.30E-3	4.43E-3	1.04E-2	6.71E-3
<u>Bromomethane</u>:				
Concentration, μg/DSCM[a]	41.33	42	94.70	59.34
Emission Rate, lb/hr	3.50E-3	3.30E-3	9.04E-3	5.28E-3
<u>Methylene Chloride</u>:				
Concentration, μg/DSCM[a]	81.50	20.20	92.30	64.67
Emission Rate, lb/hr	6.93E-3	1.59E-3	8.81E-3	5.78E-3

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 10-4. (continued)

Run Identification		I-S3-VOST		
Day/Repetition Number	1	2	3	Average
<u>Acetone</u>:				
Concentration, µg/DSCM[a]	150.50	87.75	315	184.42
Emission Rate, lb/hr	1.28E-2	6.88E-3	3.01E-2	1.66E-2
<u>Carbon Disulfide</u>:				
Concentration, µg/DSCM[a]	2.18	1.32	6.03	3.18
Emission Rate, lb/hr	1.84E-4	1.04E-4	5.76E-4	2.88E-4
<u>2-Butanone</u>:				
Concentration, µg/DSCM[a]	0.46	20	5.32	8.59
Emission Rate, lb/hr	3.85E-5	1.57E-3	5.08E-4	7.06E-4
<u>1,1,1-Trichloroethane</u>:				
Concentration, µg/DSCM[a]	1.04	0.00	0.00	0.648
Emission Rate, lb/hr	8.80E-5	0.00	0.00	2.93E-5
<u>Trichloroethene</u>:				
Concentration, µg/DSCM[a]	1.30	2.01	0.00	1.10
Emission Rate, lb/hr	1.10E-4	1.58E-4	0.00	8.93E-5
<u>Ethylbenzene</u>:				
Concentration, µg/DSCM[a]	0.25	0.33	0.24	0.270
Emission Rate, lb/hr	2.10E-5	2.58E-5	2.29E-5	2.32E-5
<u>m-/p-Xylene</u>:				
Concentration, µg/DSCM[a]	1.09	1.17	1.60	1.29
Emission Rate, lb/hr	9.20E-5	9.18E-5	1.53E-4	1.12E-4
<u>o-Xylene</u>:				
Concentration, µg/DSCM[a]	0.21	0.30	0.39	0.300
Emission Rate, lb/hr	1.78E-5	2.34E-5	3.74E-5	2.62E-5
<u>Iodomethane</u>:				
Concentration, µg/DSCM[a]	1.90	1.73	0.00	1.21
Emission Rate, lb/hr	1.61E-4	1.36E-4	0.00	9.90E-5
<u>Dibromomethane</u>:				
Concentration, µg/DSCM[a]	0.38	0.94	1.69	1.00
Emission Rate, lb/hr	3.25E-5	7.35E-5	1.61E-4	8.90E-5

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-4. (continued)

Run Identification	I-S3-VOST			
Day/Repetition Number	1	2	3	Average
Trichlorofluoromethane:				
Concentration, μg/DSCM*	2.59	0.00	0.00	0.863
Emission Rate, lb/hr	2.20E-4	0.00	0.00	7.33E-5
n-Hexane:				
Concentration, μg/DSCM*	0.73	0.70	1.57	1.00
Emission Rate, lb/hr	6.13E-5	5.53E-5	1.50E-4	8.89E-5
Isooctane:				
Concentration, μg/DSCM*	1.25	1.86	3.48	2.20
Emission Rate, lb/hr	1.06E-4	1.46E-4	3.32E-4	1.95E-4
P-Cymene:				
Concentration, μg/DSCM*	0.23	0.48	0.49	0.40
Emission Rate, lb/hr	1.92E-5	3.79E-5	4.71E-5	3.47E-5
Cumene:				
Concentration, μg/DSCM*	0.40	0.50	0.00	0.30
Emission Rate, lb/hr	3.40E-5	3.93E-5	0.00	2.44E-5
2-Hexanone:				
Concentration, μg/DSCM*	0.00	11.80	1.36	4.39
Emission Rate, lb/hr	0.00	9.28E-4	1.30E-4	3.53E-4
Ethyl Methacrylate:				
Concentration, μg/DSCM*	0.00	2.73	0.00	0.91
Emission Rate, lb/hr	0.00	2.15E-4	0.00	7.17E-5
Acrolein:				
Concentration, μg/DSCM*	0.00	40.25	0.00	13.42
Emission Rate, lb/hr	0.00	3.18E-3	0.00	1.06E-3
Styrene:				
Concentration, μg/DSCM*	0.00	0.25	0.00	0.083
Emission Rate, lb/hr	0.00	1.96E-5	0.00	6.53E-6
Vinyl Acetate:				
Concentration, μg/DSCM*	0.00	1.20	0.00	0.40
Emission Rate, lb/hr	0.00	9.45E-4	0.00	3.15E-4

* 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-4. (continued)

Run Identification		I-S3-VOST		
Day/Repetition Number	1	2	3	Average
1,2,3-Trichloropropane:				
Concentration, μg/DSCM[a]	0.00	4.25	0.00	1.42
Emission Rate, lb/hr	0.00	3.35E-4	0.00	1.12E-4
Chloroform:				
Concentration, μg/DSCM[a]	0.00	9.00	0.00	3.00
Emission Rate, lb/hr	0.00	7.05E-4	0.00	2.35E-4
Dibromochloromethane:				
Concentration, μg/DSCM[a]	0.00	0.27	0.00	0.09
Emission Rate, lb/hr	0.00	2.14E-5	0.00	7.13E-6
1,1,2-Trichloroethane:				
Concentration, μg/DSCM[a]	0.00	0.64	0.00	0.21
Emission Rate, lb/hr	0.00	5.03E-5	0.00	1.68E-5
Bromoform:				
Concentration, μg/DSCM[a]	0.00	1.27	0.00	0.42
Emission Rate, lb/hr	0.00	9.98E-5	0.00	3.33E-5
4-Methyl-2-Pentanone:				
Concentration, μg/DSCM[a]	0.00	9.88	0.00	3.29
Emission Rate, lb/hr	0.00	7.75E-4	0.00	2.58E-4
1,1,2,2-Tetrachloroethane:				
Concentration, μg/DSCM[a]	0.00	2.23	0.00	0.74
Emission Rate, lb/hr	0.00	4.40E-5	0.00	1.47E-5
1,4-Dichloro-2-butene:				
Concentration, μg/DSCM[a]	0.00	1.52	0.00	0.51
Emission Rate, lb/hr	0.00	2.98E-5	0.00	9.93E-6
Tetrachloroethane:				
Concentration, μg/DSCM[a]	0.44	0.00	0.00	0.15
Emission Rate, lb/hr	3.75E-5	0.00	0.00	1.25E-5
Tert-Butyl methyl ether:				
Concentration, μg/DSCM[a]	0.05	0.00	0.00	0.02
Emission Rate, lb/hr	4.40E-6	0.00	0.00	1.47E-6

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[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

TABLE 10-5.

VOLATILE ORGANIC EMISSIONS DATA
AS MEASURED IN STACK 3, CONDITION II
JEWELL COAL & COKE COMPANY

Run Identification	II-S3-VOST			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	2145	2310	2240	
Run Finish Time	0240	0032	0245	
Total Run Time, min.	295	142	245	
Test Train Parameter:				
Volume of Metered Dry Gas Sample, SL[a]	39.392	19.695	22.157	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry	7.7	7.9	8.2	
O_2, Percent by Volume, Dry	9.0	8.9	8.6	
Flue Gas Temperature, Degrees F	1,507	1,545	1,522	
Velocity, ft/sec	29.28	28.32	34.74	
Air Flow Rate, Dry, SCFM[a]	20,394	19,231	23,289	
Air Flow Rate, Wet, ACFM	92,007	88,991	109,183	
Benzene Emission:				
Concentration, µg/DSCM[a]	3.85	2.65	1.74	2.75
Emission Rate, lb/hr	2.94E-4	1.91E-4	1.52E-4	2.12E-4
Toluene:				
Concentration, µg/DSCM[a]	47.70	48.00	84.80	60.17
Emission Rate, lb/hr	3.60E-3	3.46E-3	7.39E-3	4.82E-3
Chloromethane:				
Concentration, µg/DSCM[a]	90.0	73.10	132	98.37
Emission Rate, lb/hr	6.90E-3	5.26E-3	1.15E-2	7.89E-3
Bromomethane:				
Concentration, µg/DSCM[a]	67	45.50	61.90	58.13
Emission Rate, lb/hr	5.10E-3	3.28E-3	5.40E-3	4.59E-3
Methylene Chloride:				
Concentration, µg/DSCM[a]	125	50.40	105	93.47
Emission Rate, lb/hr	9.60E-3	3.63E-3	9.16E-3	7.46E-3

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 10-5. (continued)

Run Identification	II-S3-VOST			
Day/Repetition Number	1	2	3	Average
Acetone:				
Concentration, µg/DSCM*	313.50	179	248	246.83
Emission Rate, lb/hr	2.40E-2	1.29E-2	2.17E-2	1.95E-2
Carbon Disulfide:				
Concentration, µg/DSCM*	1.28	0.00	1.31	0.86
Emission Rate, lb/hr	9.80E-5	0.00	1.14E-4	7.07E-5
2-Butanone:				
Concentration, µg/DSCM*	4.58	4.54	4.99	4.70
Emission Rate, lb/hr	3.50E-4	3.27E-4	4.35E-4	3.71E-4
Trichloroethene:				
Concentration, µg/DSCM*	0.88	0.00	0.00	0.29
Emission Rate, lb/hr	6.69E-5	0.00	0.00	2.23E-5
Ethylbenzene:				
Concentration, µg/DSCM*	0.64	0.30	0.20	0.38
Emission Rate, lb/hr	4.86E-5	2.13E-5	1.78E-5	2.92E-5
m-/p-Xylene:				
Concentration, µg/DSCM*	0.84	1.96	1.53	1.44
Emission Rate, lb/hr	6.40E-5	1.41E-4	1.33E-4	1.13E-4
o-Xylene:				
Concentration, µg/DSCM*	0.00	0.55	0.36	0.30
Emission Rate, lb/hr	0.00	3.98E-5	3.17E-5	2.38E-5
Dibromomethane:				
Concentration, µg/DSCM*	0.80	1.69	1.48	1.32
Emission Rate, lb/hr	6.10E-5	1.22E-4	1.29E-4	1.04E-4
Trichlorofluoromethane:				
Concentration, µg/DSCM*	2.41	0.00	0.00	0.80
Emission Rate, lb/hr	1.84E-4	0.00	0.00	6.13E-5
n-Hexane:				
Concentration, µg/DSCM*	0.76	0.00	0.00	0.25
Emission Rate, lb/hr	5.80E-5	0.00	0.00	1.93E-5

* 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-5. (continued)

Run Identification	II-S3-VOST			
Day/Repetition Number	1	2	3	Average
2-Hexanone:				
Concentration, μg/DSCM[a]	2.74	2.39	1.70	2.28
Emission Rate, lb/hr	2.10E-4	1.72E-4	1.48E-4	1.77E-4
Ethyl Methacrylate:				
Concentration, μg/DSCM[a]	0.53	0.00	0.00	0.18
Emission Rate, lb/hr	4.04E-5	0.00	0.00	1.35E-5

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

TABLE 10-6.

VOLATILE EMISSIONS DATA
AS MEASURED IN STACK 3, CONDITION III
JEWELL COAL & COKE COMPANY

Run Identification	III-S3-VOST			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	0410	0500	0500	
Run Finish Time	1010	0740	0740	
Total Run Time, min.	360	160	160	
<u>Test Train Parameter</u>:				
Volume of Metered Dry Gas Sample, SL*	41.806	21.195	22.176	
<u>Flue Gas Parameters</u>:				
CO_2, Percent by Volume, Dry	6.5	7.5	7.9	
O_2, Percent by Volume, Dry	10.6	9.0	8.5	
Flue Gas Temperature, Degrees F	1,504	1,520	1,536	
Velocity, ft/sec	20.84	20.27	34.71	
Air Flow Rate, Dry, SCFM*	14,474	14,043	23,774	
Air Flow Rate, Wet, ACFM	65,512	63,719	109,093	
<u>Benzene Emission</u>:				
Concentration, μg/DSCM*	448.50	13.90	1.65	154.68
Emission Rate, lb/hr	2.43E-2	7.34E-4	1.47E-4	8.39E-3
<u>Toluene</u>:				
Concentration, μg/DSCM*	67.50	49.10	61.20	59.27
Emission Rate, lb/hr	3.66E-3	2.58E-3	5.45E-3	3.90E-3
<u>Chloromethane</u>:				
Concentration, μg/DSCM*	64.60	86.70	153	101.43
Emission Rate, lb/hr	3.50E-3	4.56E-3	1.36E-2	7.22E-3
<u>Bromomethane</u>:				
Concentration, μg/DSCM*	44.0	57.3	75.3	58.87
Emission Rate, lb/hr	2.39E-3	3.01E-3	6.71E-3	4.04E-3
<u>Methylene Chloride</u>:				
Concentration, μg/DSCM*	71.50	56.30	64.60	64.13
Emission Rate, lb/hr	3.88E-3	2.96E-3	5.75E-3	4.20E-3

* 68°F (20°C) --29.92 Inches of Mercury (Hg)

(continued)

TABLE 10-6. (continued)

Run Identification	III-S3-VOST			
Day/Repetition Number	1	2	3	Average
<u>Acetone</u>:				
Concentration, μg/DSCM[a]	289.50	254	178	240.5
Emission Rate, lb/hr	1.57E-2	1.34E-2	1.59E-2	1.50E-2
<u>Carbon Disulfide</u>:				
Concentration, μg/DSCM[a]	0.43	0.58	3.64	1.55
Emission Rate, lb/hr	2.35E-5	3.02E-5	3.24E-4	1.26E-4
<u>2-Butanone</u>:				
Concentration, μg/DSCM[a]	2.76	3.73	7.31	4.60
Emission Rate, lb/hr	1.50E-4	1.96E-4	6.51E-4	3.32E-4
<u>1,1,1-Trichloroethane</u>:				
Concentration, μg/DSCM[a]	0.68	0.00	0.00	0.23
Emission Rate, lb/hr	3.66E-5	0.00	0.00	1.22E-5
<u>Trichloroethene</u>:				
Concentration, μg/DSCM[a]	2.08	1.49	0.00	1.19
Emission Rate, lb/hr	1.13E-4	7.84E-5	0.00	6.38E-5
<u>Ethylbenzene</u>:				
Concentration, μg/DSCM[a]	0.47	0.29	0.22	0.33
Emission Rate, lb/hr	1.47E-5	1.55E-5	2.00E-5	1.67E-5
<u>m-/p-Xylene</u>:				
Concentration, μg/DSCM[a]	0.70	1.01	0.82	0.84
Emission Rate, lb/hr	3.79E-5	5.30E-5	7.31E-5	5.47E-5
<u>o-Xylene</u>:				
Concentration, μg/DSCM[a]	0.00	0.41	0.39	0.27
Emission Rate, lb/hr	0.00	2.18E-5	3.51E-5	1.90E-5
<u>Iodomethane</u>:				
Concentration, μg/DSCM[a]	0.00	4.16	0.00	1.39
Emission Rate, lb/hr	0.00	2.19E-4	0.00	7.30E-5
<u>Dibromomethane</u>:				
Concentration, μg/DSCM[a]	0.69	1.60	1.51	1.27
Emission Rate, lb/hr	3.72E-5	8.42E-5	1.34E-4	8.38E-5

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-6. (continued)

Run Identification	III-S3-VOST			
Day/Repetition Number	1	2	3	Average
Trichlorofluoromethane:				
Concentration, µg/DSCM*	0.00	0.00	4.90	1.63
Emission Rate, lb/hr	0.00	0.00	4.37E-4	1.46E-4
n-Hexane:				
Concentration, µg/DSCM*	8.15	0.00	2.84	3.66
Emission Rate, lb/hr	4.42E-4	0.00	2.53E-4	2.32E-4
Isooctane:				
Concentration, µg/DSCM*	14.30	0.00	0.00	4.77
Emission Rate, lb/hr	7.73E-4	0.00	0.00	2.58E-4
2-Hexanone:				
Concentration, µg/DSCM*	0.00	2.01	2.77	1.59
Emission Rate, lb/hr	0.00	1.06E-4	2.47E-4	1.18E-4
Acrolein:				
Concentration, µg/DSCM*	11,677	0.00	0.00	3892.33
Emission Rate, lb/hr	0.63	0.00	0.00	0.21
Dimethyl Sulfide:				
Concentration, µg/DSCM*	1.77	0.00	0.00	0.59
Emission Rate, lb/hr	9.60E-5	0.00	0.00	3.20E-5
Styrene:				
Concentration, µg/DSCM*	7.52	0.00	0.00	2.51
Emission Rate, lb/hr	4.08E-4	0.00	0.00	1.36E-4
Chloroform:				
Concentration, µg/DSCM*	0.00	4.51	0.00	1.50
Emission Rate, lb/hr	0.00	2.37E-4	0.00	7.90E-5

* 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

TABLE 10-7.

VOLATILE EMISSIONS DATA
AS MEASURED IN STACK 3, CONDITION IV
JEWELL COAL & COKE COMPANY

Run Identification	IV-S3-VOST			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	1130	1200	1200	
Run Finish Time	1531	1500	1240	
Total Run Time, min.	241	180	40	
<u>Test Train Parameter</u>:				
Volume of Metered Dry Gas Sample, SL*	37.001	20.842	22.226	
<u>Flue Gas Parameters</u>:				
CO_2, Percent by Volume, Dry	7.3	7.6	7.7	
O_2, Percent by Volume, Dry	9.2	8.6	8.3	
Flue Gas Temperature, Degrees F	1,541	1,498	1,502	
Velocity, ft/sec	26.46	29.36	28.74	
Air Flow Rate, Dry, SCFM*	18,589	20,784	20,279	
Air Flow Rate, Wet, ACFM	83,161	92,281	90,335	
<u>Benzene Emission</u>:				
Concentration, μg/DSCM*	333.5	2.70	1.46	112.55
Emission Rate, lb/hr	2.33E-2	2.10E-4	1.11E-4	7.87E-3
<u>Toluene</u>:				
Concentration, μg/DSCM*	38.30	59.20	46.90	48.13
Emission Rate, lb/hr	2.67E-3	4.61E-3	3.57E-3	3.62E-3
<u>Chloromethane</u>:				
Concentration, μg/DSCM*	44.30	0.00	60.80	35.03
Emission Rate, lb/hr	3.09E-3	0.00	4.62E-3	2.57E-3
<u>Bromomethane</u>:				
Concentration, μg/DSCM*	37.85	73.60	42.20	51.22
Emission Rate, lb/hr	2.64E-3	5.73E-3	3.21E-3	3.86E-3
<u>Methylene Chloride</u>:				
Concentration, μg/DSCM*	23.35	55.30	58.50	45.72
Emission Rate, lb/hr	1.63E-3	4.30E-3	4.45E-3	3.46E-3

* 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-7. (continued)

Run Identification	IV-S3-VOST			
Day/Repetition Number	1	2	3	Average
<u>Acetone</u>:				
Concentration, µg/DSCM[a]	333	258	273	288
Emission Rate, lb/hr	2.32E-2	2.01E-2	2.07E-2	2.13E-2
<u>Carbon Disulfide</u>:				
Concentration, µg/DSCM[a]	0.92	0.00	1.23	0.72
Emission Rate, lb/hr	6.43E-5	0.00	9.35E-5	5.26E-5
<u>2-Butanone</u>:				
Concentration, µg/DSCM[a]	7.20	6.34	10.90	8.15
Emission Rate, lb/hr	5.00E-4	4.94E-4	8.26E-4	6.07E-4
<u>1,1,1-Trichloroethane</u>:				
Concentration, µg/DSCM[a]	0.94	0.00	0.00	0.31
Emission Rate, lb/hr	6.56E-5	0.00	0.00	2.19E-5
<u>Ethylbenzene</u>:				
Concentration, µg/DSCM[a]	0.54	0.32	0.26	0.37
Emission Rate, lb/hr	3.77E-5	2.50E-5	1.96E-5	2.74E-5
<u>m-/p-Xylene</u>:				
Concentration, µg/DSCM[a]	1.76	1.80	1.64	1.73
Emission Rate, lb/hr	1.23E-4	1.40E-4	1.24E-4	1.29E-4
<u>o-Xylene</u>:				
Concentration, µg/DSCM[a]	0.46	0.48	0.37	0.44
Emission Rate, lb/hr	3.17E-5	3.74E-5	2.78E-5	3.23E-5
<u>Dibromomethane</u>:				
Concentration, µg/DSCM[a]	0.97	1.53	1.48	1.33
Emission Rate, lb/hr	6.75E-5	1.19E-4	1.12E-4	9.95E-5
<u>n-Hexane</u>:				
Concentration, µg/DSCM[a]	4.45	0.00	0.00	1.48
Emission Rate, lb/hr	3.10E-4	0.00	0.00	1.03E-4

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)
Zeroes indicate non-detects

(continued)

TABLE 10-7. (cont.)

Run Identification	IV-S3-VOST			
Day/Repetition Number	1	2	3	Average
Cumene:				
Concentration, µg/DSCM[a]	0.83	0.00	0.00	0.28
Emission Rate, lb/hr	5.74E-5	0.00	0.00	1.91E-5
2-Hexanone:				
Concentration, µg/DSCM[a]	3.29	1.50	4.36	3.05
Emission Rate, lb/hr	2.28E-4	1.17E-4	3.31E-4	2.225E-4
Ethyl Methacrylate:				
Concentration, µg/DSCM[a]	0.76	0.00	0.00	0.25
Emission Rate, lb/hr	5.25E-5	0.00	0.00	1.75E-5
Acrolein:				
Concentration, µg/DSCM[a]	53.60	0.00	0.00	17.87
Emission Rate, lb/hr	3.74E-3	0.00	0.00	1.25E-3
Styrene:				
Concentration, µg/DSCM[a]	2.03	0.00	0.00	0.68
Emission Rate, lb/hr	1.41E-4	0.00	0.00	4.70E-5
Vinyl Acetate:				
Concentration, µg/DSCM[a]	0.67	0.00	0.00	0.22
Emission Rate, lb/hr	4.65E-5	0.00	0.00	1.55E-5
Chlorobenzene:				
Concentration, µg/DSCM[a]	1.68	0.00	0.00	0.56
Emission Rate, lb/hr	1.17E-4	0.00	0.00	3.90E-5
Dimethyl Sulfide:				
Concentration, µg/DSCM[a]	0.00	2.50	0.00	0.83
Emission Rate, lb/hr	0.00	1.95E-4	0.00	6.50E-5
1,2,3-Trichloropropane:				
Concentration, µg/DSCM[a]	0.74	0.00	0.65	0.46
Emission Rate, lb/hr	5.15E-5	0.00	4.91E-5	3.35E-5

[a] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

TABLE 10-8.

VOLATILE ORGANICS EMISSIONS DATA
TOTAL DAILY AVERAGE ESTIMATES FROM BATTERY C[a]
JEWELL COAL & COKE COMPANY

Day/Repetition	1	2	3	Average
Benzene Emission:				
Concentration, μg/DSCM[b]	197.45	27.14	1.66	75.42
Emission Rate, lb/hr	5.04E-2	8.5E-3	5.0E-4	1.98E-2
Chloromethane:				
Concentration, μg/DSCM[b]	65.32	54.02	113.70	77.68
Emission Rate, lb/hr	1.86E-2	1.55E-2	3.44E-2	2.28E-2
Bromomethane:				
Concentration, μg/DSCM[b]	47.55	54.60	68.53	56.89
Emission Rate, lb/hr	1.35E-2	1.56E-2	2.10E-2	1.67E-2
Methylene Chloride:				
Concentration, μg/DSCM[b]	73.34	45.55	80.10	66.33
Emission Rate, lb/hr	2.18E-2	1.28E-2	2.44E-2	1.97E-2
Acetone:				
Concentration, μg/DSCM[b]	271.63	194.69	253.50	239.94
Emission Rate, lb/hr	7.51E-2	5.46E-2	7.70E-2	6.89E-2
Carbon Disulfide:				
Concentration, μg/DSCM[b]	1.20	0.475	3.05	1.58
Emission Rate, lb/hr	4.0E-4	1.5E-4	9.0E-4	4.83E-4
2-Butanone:				
Concentration, μg/DSCM[b]	3.75	8.65	7.13	6.51
Emission Rate, lb/hr	1.0E-3	2.6E-3	2.1E-3	1.90E-3
1,1,1-Trichloroethane:				
Concentration, μg/DSCM[b]	0.6675	0.00	0.00	0.22
Emission Rate, lb/hr	2.25E-4	0.00	0.00	7.50E-5
Trichloroethene:				
Concentration, μg/DSCM[b]	1.065	0.875	0.00	0.65
Emission Rate, lb/hr	5.33E-4	2.5E-4	0.00	2.61E-4
Toluene:				
Concentration, μg/DSCM[b]	47.23	43.79	67.50	52.84
Emission Rate, lb/hr	1.31E-2	1.23E-2	2.06E-2	1.53E-2

[a] Based on average concentration data obtained from Stack 3

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

(continued)

TABLE 10-8. (cont.)

Day/Repetition Number	1	2	3	Average
Ethylbenzene:				
Concentration, µg/DSCM[b]	0.48	0.31	0.23	0.34
Emission Rate, lb/hr	1.32E-4	9.0E-5	6.9E-5	9.70E-5
m-/p-Xylene:				
Concentration, µg/DSCM[b]	1.10	1.49	1.40	1.33
Emission Rate, lb/hr	3.10E-4	4.28E-4	4.23E-4	3.87E-4
o-Xylene:				
Concentration, µg/DSCM[b]	0.17	0.44	0.38	0.33
Emission Rate, lb/hr	4.75E-5	1.24E-4	1.14E-4	9.52E-5
Iodomethane:				
Concentration, µg/DSCM[b]	0.475	1.475	0.00	0.65
Emission Rate, lb/hr	1.57E-4	4.10E-4	0.00	1.89E-4
Dibromomethane:				
Concentration, µg/DSCM[b]	0.71	1.44	1.54	1.23
Emission Rate, lb/hr	1.96E-4	4.09E-4	4.66E-4	3.57E-4
Trichlorofluoromethane:				
Concentration, µg/DSCM[b]	1.25	0.00	1.23	0.825
Emission Rate, lb/hr	3.90E-4	0.00	3.53E-4	2.47E-4
n-Hexane:				
Concentration, µg/DSCM[b]	3.52	0.175	1.11	1.60
Emission Rate, lb/hr	9.16E-4	5.68E-5	3.36E-4	4.36E-4
Isooctane:				
Concentration, µg/DSCM[b]	3.89	0.47	0.87	1.74
Emission Rate, lb/hr	9.99E-4	5.68E-5	1.16E-3	4.80E-4
P-Cymene:				
Concentration, µg/DSCM[b]	0.08	0.12	0.12	0.10
Emission Rate, lb/hr	1.90E-5	3.90E-4	4.08E-5	3.29E-5
Cumene:				
Concentration, µg/DSCM[b]	0.31	0.13	0.00	0.145
Emission Rate, lb/hr	8.70E-5	4.05E-5	0.00	4.25E-5

[a] Based on average concentration data obtained from Stack 3

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

(continued)

TABLE 10-8. (cont.)

Day/Repetition Number	1	2	3	Average
2-Hexanone:				
Concentration, µg/DSCM[b]	1.51	4.43	2.55	2.83
Emission Rate, lb/hr	4.14E-4	1.37E-3	7.47E-4	8.44E-4
Ethyl Methacrylate:				
Concentration, µg/DSCM[b]	0.33	0.68	0.00	0.34
Emission Rate, lb/hr	8.80E-5	2.22E-4	0.00	1.03E-4
Acrolein:				
Concentration, µg/DSCM[b]	2932.65	10.06	0.00	980.90
Emission Rate, lb/hr	0.74	3.28E-3	0.00	2.46E-1
Styrene:				
Concentration, µg/DSCM[b]	2.39	0.06	0.00	0.82
Emission Rate, lb/hr	6.05E-4	2.03E-5	0.00	2.08E-4
Vinyl Acetate:				
Concentration, µg/DSCM[b]	0.17	0.30	0.00	0.82
Emission Rate, lb/hr	4.38E-5	9.75E-5	0.00	2.08E-4
1,2,3-Trichloropropane:				
Concentration, µg/DSCM[b]	0.19	1.06	0.00	0.42
Emission Rate, lb/hr	4.83E-5	3.45E-4	0.00	1.31E-4
Chloroform:				
Concentration, µg/DSCM[b]	0.00	3.38	0.00	1.13
Emission Rate, lb/hr	0.00	1.03E-3	0.00	3.42E-4
Dibromochloromethane:				
Concentration, µg/DSCM[b]	0.00	0.07	0.00	0.02
Emission Rate, lb/hr	0.00	2.20E-5	0.00	7.33E-6
1,1,2-Trichloroethane:				
Concentration, µg/DSCM[b]	0.00	0.16	0.00	0.05
Emission Rate, lb/hr	0.00	5.20E-5	0.00	1.73E-5
Bromoform:				
Concentration, µg/DSCM[b]	0.00	0.32	0.00	0.11
Emission Rate, lb/hr	0.00	1.03E-4	0.00	3.44E-5

[a] Based on average concentration data obtained from Stack 3

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

(continued)

TABLE 10-8. (cont.)

Day/Repetition Number	1	2	3	Average
<u>4-Methyl-2-Pentanone</u>:				
Concentration, μg/DSCM[b]	0.00	2.47	0.00	0.82
Emission Rate, lb/hr	0.00	8.03E-4	0.00	2.68E-4
<u>1,1,2,2-Tetrachloroethane</u>:				
Concentration, μg/DSCM[b]	0.00	0.56	0.00	0.19
Emission Rate, lb/hr	0.00	1.81E-4	0.00	6.03E-5
<u>1,4-Dichloro-2-butene</u>:				
Concentration, μg/DSCM[b]	0.00	0.38	0.00	0.13
Emission Rate, lb/hr	0.00	1.24E-4	0.00	4.12E-5
<u>Tetrachloroethane</u>:				
Concentration, μg/DSCM[b]	0.11	0.00	0.00	0.04
Emission Rate, lb/hr	3.65E-5	0.00	0.00	1.22E-5
<u>Tert-Butyl methyl ether</u>:				
Concentration, μg/DSCM[b]	0.01	0.00	0.00	4.17E-3
Emission Rate, lb/hr	4.25E-6	0.00	0.00	1.42E-6
<u>Chlorobenzene</u>:				
Concentration, μg/DSCM[b]	0.42	0.00	0.00	0.14
Emission Rate, lb/hr	1.10E-4	0.00	0.00	3.65E-5
<u>Dimethyl Sulfide</u>:				
Concentration, μg/DSCM[b]	0.44	0.63	0.00	0.36
Emission Rate, lb/hr	1.11E-4	1.75E-4	0.00	9.54E-5

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[a] Based on average concentration data obtained from Stack 3

[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

Zeroes indicate non-detects

TABLE 10-9.

VOLATILE CLEAN AIR ACT COMPOUNDS
TOTAL EMISSION RATES
JEWELL COAL & COKE

COMPOUND	EMISSION RATE lb/yr	COMPOUND	EMISSION RATE lb/yr
Benzene	(238, 246)	Toluene (uncorrected) (blank corrected)	(114, 115) (33, 40)
Chloromethane	(172, 184)	Bromomethane	(125, 135)
Vinyl Chloride	(0, 19)	Chloroethane	(0, 19)
Methylene Chloride (uncorrected) (blank corrected)	(160, 163) (41, 48)	Acetone (uncorrected) (blank corrected)	(546, 557) (140, 148)
Carbon Disulfide	(3, 17)	1,1-Dichloroethene	(0, 19)
1,1-Dichloroethane	(0, 19)	trans-1,2-Dichloroethene	(0, 19)
Chloroform	(3, 21)	1,2-Dichloroethane	(0, 19)
2-Butanone	(19, 31)	1,1,1-Trichloroethane	(1, 18)
Carbon Tetrachloride	(0, 19)	Vinyl Acetate	(1, 18)
Bromodichloromethane	(0, 19)	1,2-Dichloropropane	(0, 19)
trans-1,3-Dichloropropene	(0, 19)	Trichloroethene	(2, 17)
Dibromochloromethane	(0, 18)	1,1,2-Trichloroethane	(0, 18)
cis-1,3-Dichloropropene	(0, 19)	2-Chloroethylvinylether	(0, 19)
Bromoform	(0, 18)	2-Hexanone	(9, 24)
4-Methyl-2-Pentanone	(5, 23)	Tetrachloroethene	(0, 19)
1,1,2,2-Tetrachloroethane	(1, 19)	Chlorobenzene	(0, 19)
Ethylbenzene	(1, 12)	Styrene	(1, 19)
m-/p-Xylene	(3, 11)	o-Xylene	(0, 16)
Acrolein *	(2849, 2865)	Acrylonitrile	(0, 19)
Dichlorodifluoromethane	(0, 19)	Iodomethane	(1, 18)
Dibromomethane	(3, 15)	1,4-Dichloro-2-butene	(1, 18)
Ethyl methacrylate	(2, 17)	1,2,3-Trichloropropane	(2, 18)
Trichlorofluoromethane	(2, 19)	1,3 Dichlorobenzene	(0, 19)

* Acrolein is common in woodsmoke and, since acrolein appeared in only one sample, it is possible that contamination occurred from the forest fires in the area.

(continued)

TABLE 10-9. (cont.)

COMPOUND	EMISSION RATE lb/yr	COMPOUND	EMISSION RATE lb/yr
1,4 Dichlorobenzene	(0, 19)	1,2 Dichlorobenzene	(0, 19)
cis-1,2-Dichloroethene	(0, 19)	n-Hexane	(3, 17)
Tert-butyl methyl ether	(0, 18)	1,3 Butadiene	(0, 19)
Vinyl bromide	(0, 19)	Isooctane	(3, 19)
Allyl chloride	(0, 19)	Cumene	(0, 18)
Dimethyl sulfide	(1, 18)	Dimethyl disulfide	(0, 19)
a-Pinene	(0, 19)	b-Pinene	(0, 19)
p-Cymene	(0, 18)	▨	▨

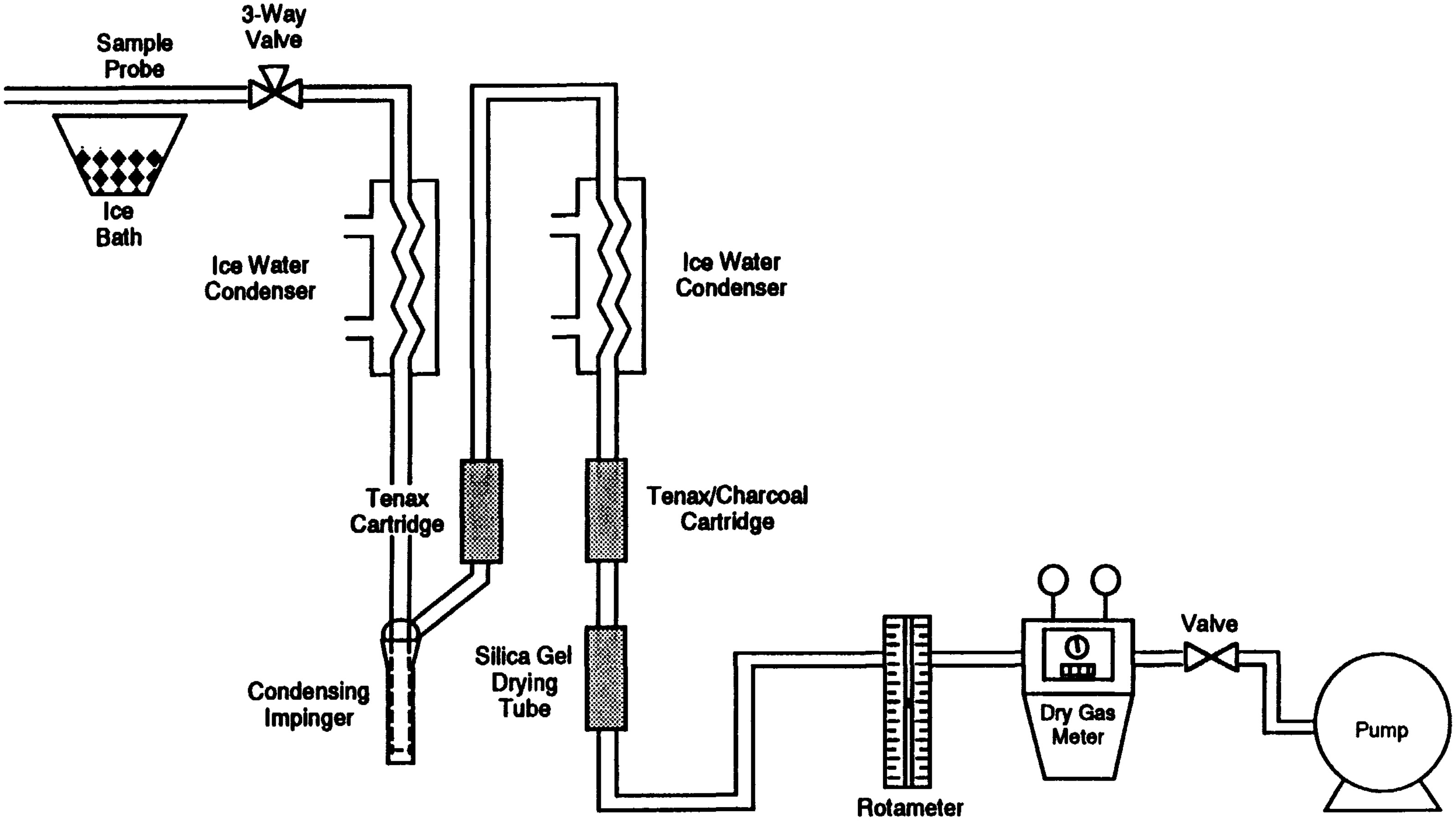
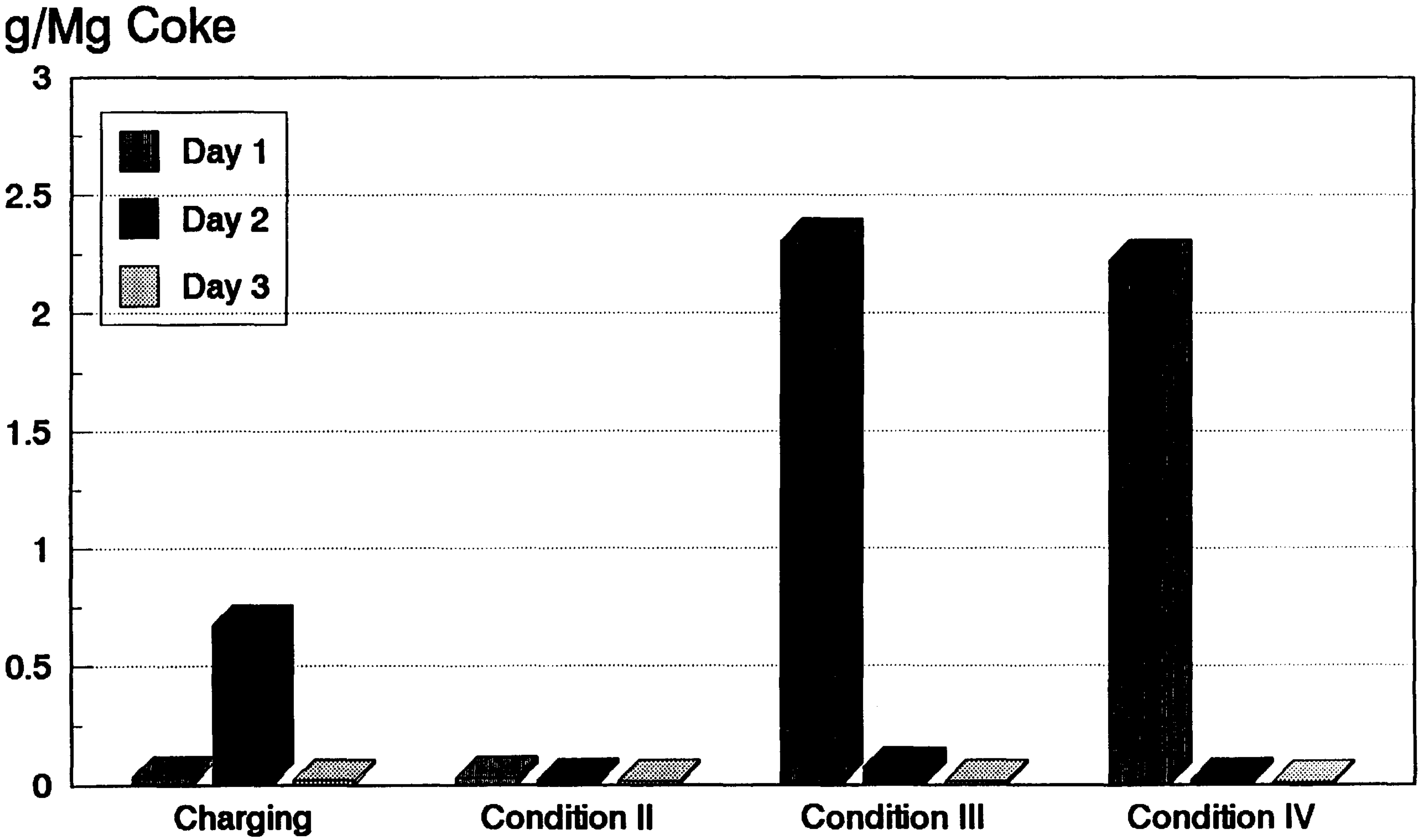


Figure 10-1. Schematic of volatile organic sampling train.

3694 1/92

Benzene

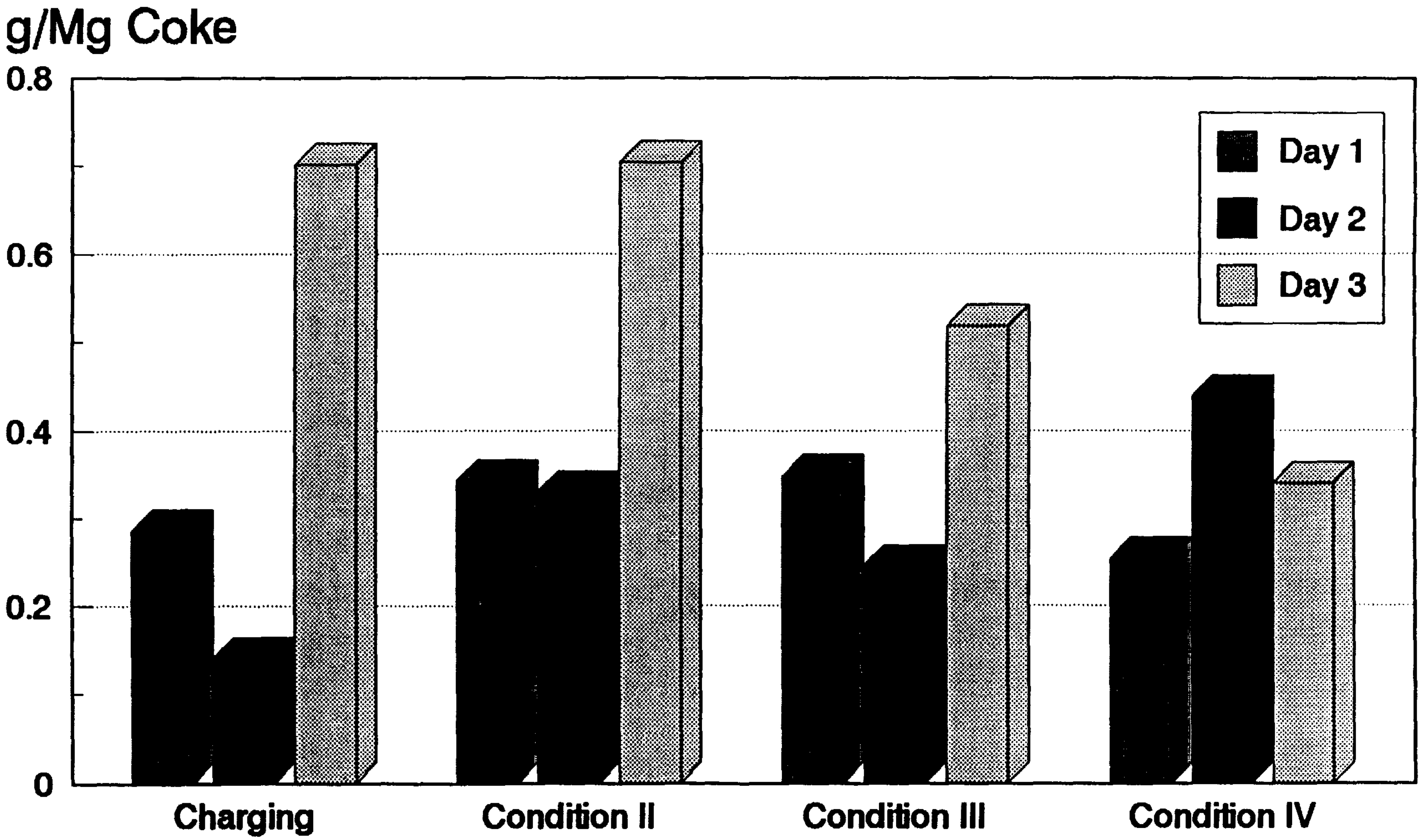
from VOST train



Jewell Thompson Non-Recovery Coke Oven
Minimum Detectable = .035 g/Mg Coke

Toluene

from VOST train



Jewell Thompson Non-Recovery Coke Oven
Minimum Detectable = 0.035g/Mg Coke

11.0 INSTRUMENTAL TEST METHODS, CONTINUOUS EMISSION MONITORING (CEM)

11.1 INSTRUMENTAL METHOD TESTING

EPA instrumental Test Methods 3A, 6C, 7E, and 10 were used to continuously measure emissions of oxygen (O_2) and carbon dioxide (CO_2), sulfur dioxide (SO_2), nitrogen oxides (NO_x), and carbon monoxide (CO), respectively. These methods are considered refernce methods. CEM testing was conducted at Stack 4. Each method has been reproduced in Appendix D.

11.2 INSTRUMENTAL MEASUREMENT SYSTEM EQUIPMENT

An extractive sample system was used at Stack 4 to obtain flue gas samples for the appropriate analyzers. Samples were withdrawn continuously at a single point and conveyed to Entropy's CEM mobile laboratory through a Teflon™ sample line. The flue gas was conditioned (moisture and particles removed) and sent to a manifold where it was distributed to the O_2 and CO_2, SO_2, NO_x, and CO analyzers. Figure 11-1 is a schematic of the instrumental measurement system. The sample extraction and conditioning system and the analyzers used during the testing program met each of the method specifications. Instrumentation and calibration gas ranges used for the CEM testing are provided in Table 11-1. The CEMS's were operated according to the methods and calibrated according to EPA Method 6C at approximately three- to five-hour intervals.

11.2.1 <u>Data Acquisition System</u>

The data acquisition system (DAS) employed for this testing program consisted of a portable computer with a 20 MB hard disk and an internal 12-bit analog-to-digital converter with a 16-channel multiplexer. In addition to providing an instantaneous display of analyzer responses, the DAS compiled and averaged the analyzer data collected once each second, calculated emission rates, and documented analyzer calibrations. The test data and calibrations were stored on hard disk and printed on an Epson dot matrix printer.

11.2.2 Instrumental Testing Procedures

At the beginning of each condition, the instrumental measurement systems were first leak-checked. The analyzers were calibrated using a minimum of three certified calibration gases (zero and two upscale). This was followed by a calibration error check utilizing two or three certified gases. The acceptance criterion for the calibration error check was less than 2 percent of span for the O_2, CO_2, SO_2, NO_x, and CO analyzers.

System bias checks were conducted for the O_2, CO_2, SO_2, NO_x, and CO analyzers where zero and upscale calibration gases were introduced at the back end of the sample probe to assess sample losses in the system. The acceptance criterion for the bias checks was less than 5 percent of span.

Methods 3A, 6C, 7E, and 10 sampling runs were conducted concurrently with the M5/TSO/202, volatile and semi-volatile organic compounds, and MMTL runs. Run duration varied with condition; Condition I runs were three hours in duration; Conditions II, III, and IV runs were six hours in duration.

In accordance with method specifications, zero and span gases were introduced into the sampling system and directed to the appropriate analyzers to check for drift. An exception to the method protocol was the greater than usual time lapses between calibrations. Although the checks are usually done before, after, and at a maximum of one-hour intervals during runs, this criterion was dropped due to the length of individual tests. Instead, calibrations were performed at the beginning and end of a run and at a convenient stopping point during the run. The drift was required to be within 3 percent of span. All instrumental results were corrected for drift.

11.3 CEM SUMMARY OF RESULTS

The SO_2, NO_x, and CO_2 and O_2 uncorrected concentration data, in ppm_{vd}, have been calculated and graphically displayed by day, as shown in Figures 11-2 through 11-10. Figures 11-2 through 11-4 represent Day 1; Figures 11-5 through 11-7 represent Day 2; and Figures 11-8 through 11-10 represent Day 3. CO was not graphed because it was not detected. The breaks in the data represent analyzer downtime for calibrations.

As shown in Table 11-2, the total weighted average emission rate was 271 lb/hr. For NO_x, the average emission rate was 27 lb/hr. CO emission concentrations were measured, and none were reported. These data represent SO_2, NO_x, and CO emitted from all 4 stacks in a 24-hour period on a lb/hr basis.

Tables 11-13a through 11-13d present the as-measured data from Stack 1, Conditions I, II, III, and IV, respectively.

11.4 CEM DATA QUALIFIERS

Provided in this section is a list of data qualifiers. These included:

(1) Moisture data from Stacks 3 and 4 were assumed to be the average of all % water from Stacks 1 and 2, per condition, per day.

(2) The flow rates for Stack 4 were calculated by averaging all flow rate data for Stacks 1 through 3, per condition.

(3) The ppm_{vd} graphic data were uncorrected for drift, the ppm_{vd} for run averages and the lb/hr presented in Tables 11-2 through 11-3d were corrected for drift. The drift corrections were less than 3%.

(4) Data were corrected by using the pre-run and post-run calibration checks as specified in the method with the exception of longer time lapse between calibrations. All drift were within method specifications.

(5) Refer to Tables 7-3, 7-4, and 7-5 in Section 7.0 for flowrate, temperature, moisture, and gas constituent data used in the calculations.

TABLE 11-1.

INSTRUMENT RANGES AND CALIBRATION GASES

Analyte	Gas Instrument/Concentration
O_2	
Instrument	Teledyne Model 320P
Ranges	0 - 25% O_2
Zero Gas	Zero air
Mid-Range Gas Value	40 - 60%
High-Range Gas Value	80 - 100%
CO_2	
Instrument	ACS Fuji 3300
Ranges	0 - 20% CO_2
Zero Gas	Zero air
Mid-Range Gas Value	40 - 60% of span
High-Range Gas Value	80 - 100% of span
SO_2	
Instrument	Western Research Model 721AT2
Ranges	0 -500 ppm
Zero Gas	Zero air
Mid-Range Gas Value	40 - 60% of span
High-Range Gas Value	80 - 100% of span
NO_x	
Instrument	TECO Model 10
Ranges	0 -250 ppm
Zero Gas	Zero air
Midrange Gas Value	40 to 60% of span
High Range Gas Value	80 to 100% of span
CO	
Instrument	Fuji Model 3300
Ranges	0-100 ppm
Zero Gas	UPC Grade Air
Midrange Gas Value	60% of span
Low Range Gas Value	30% of span

TABLE 11-2.

CONTINUOUS EMISSIONS MONITORING DATA
TOTAL AVERAGES PER CYCLE
JEWELL COAL & COKE COMPANY

Day/Repetition	1	2	3	Average
SO_2:				
Concentration, ppm_{vd}	372.15	366.20	345.51	361.29
Emission Rate, lb/hr	269.95	272.73	269.83	270.84
Emission Rate, lb/ton charged	9.036	9.091	9.032	9.053
NO_x:				
Concentration, ppm_{vd}	47.03	46.80	52.35	48.73
Emission Rate, lb/hr	24.87	25.21	29.83	26.64
Emission Rate, lb/ton charged	0.8325	0.8403	0.9985	0.8904
CO:				
Concentration, ppm_{vd}	0.00	0.00	0.00	0.00
Emission Rate, lb/hr	0.00	0.00	0.00	0.00
Emission Rate, lb/ton charged	0.00	0.00	0.00	0.00

TABLE 11-3a.

CONTINUOUS EMISSIONS MONITORING DATA
AS MEASURED IN STACK 4, CONDITION I - CHARGING
JEWELL COAL & COKE COMPANY

Run Identification	I-S4-CEM			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	1716	1701	1756	
Run Finish Time	2034	2100	2100	
Total Run Time, min.	198	239	184	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[a]	6.39	7.35	6.54	
O_2, Percent by Volume, Dry[a]	11.25	9.62	10.99	
Flue Gas Temperature, Degrees F	1,466	1,521	1,533	
Velocity, ft/sec	32.72	33.07	32.69	
Air Flow Rate, Dry, SCFM[b]	22,027	21,680	21,732	
Air Flow Rate, Wet, ACFM	101,794	103,051	101,551	
SO_2 Results:				
Concentration, ppm_{vd}	324.9	347.9	286.5	319.77
Emission Rate, lb/hr	71.28	75.12	62.01	69.47
NO_x Results:				
Concentration, ppm_{vd}	44.1	49.2	68.0	57.77
Emission Rate, lb/hr	6.96	7.64	10.59	8.40
CO Results:				
Concentration, ppm_{vd}	0.00	0.00	0.00	0.00
Emission Rate, lb/hr	0.00	0.00	0.00	0.00

[a] Analyzers
[b] 68°F (20°C) --29.92 Inches of Mercury (Hg)

TABLE 11-3b.

CONTINUOUS EMISSIONS MONITORING DATA AS MEASURED IN STACK 4, CONDITION II JEWELL COAL & COKE COMPANY

Run Identification	II-S4-CEM			
Day/Repetition Number	1	2	3	Average
Run Date	10/29/91	10/30/91	10/31/91	
Run Start Time	2123	2116	2101	
Run Finish Time	0330	0315	0255	
Total Run Time, min.	367	359	354	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[a]	7.77	7.76	7.54	
O_2, Percent by Volume, Dry[a]	9.14	8.85	9.39	
Flue Gas Temperature, Degrees F	1,500	1,546	1,529	
Velocity, ft/sec	28.52	28.45	30.39	
Air Flow Rate, Dry, SCFM[b]	19,273	19,323	20,331	
Air Flow Rate, Wet, ACFM	88,633	88,355	94,992	
SO_2 Results:				
Concentration, ppm_{vd}	350.7	364.0	330.3	348.33
Emission Rate, lb/hr	67.32	70.05	66.88	68.08
NO_x Results:				
Concentration, ppm_{vd}	58.8	57.2	65.7	60.57
Emission Rate, lb/hr	8.12	7.92	9.57	8.54
CO Results:				
Concentration, ppm_{vd}	0.00	0.00	0.00	0.00
Emission Rate, lb/hr	0.00	0.00	0.00	0.00

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[a] Analyzers

[b] 68°F (20°C)--29.92 Inches of Mercury (Hg)

TABLE 11-3c.

CONTINUOUS EMISSIONS MONITORING DATA
AS MEASURED IN STACK 4, CONDITION III
JEWELL COAL & COKE COMPANY

Run Identification	III-S4-CEM			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	0401	0333	0331	
Run Finish Time	1020	1000	1007	
Total Run Time, min.	379	387	396	
<u>Flue Gas Parameters</u>:				
CO_2, Percent by Volume, Dry[a]	8.23	7.65	7.80	
O_2, Percent by Volume, Dry[a]	8.29	8.87	8.86	
Flue Gas Temperature, Degrees F	1,489	1,519	1,508	
Velocity, ft/sec	24.68	25.94	28.26	
Air Flow Rate, Dry, SCFM[b]	17,047	17,748	19,248	
Air Flow Rate, Wet, ACFM	76,799	80,530	88,084	
<u>SO_2 Results</u>:				
Concentration, ppm_{vd}	389.5	376.3	362.1	375.97
Emission Rate, lb/hr	66.13	66.52	69.42	67.36
<u>NO_x Results</u>:				
Concentration, ppm_{vd}	43.9	43.8	48.0	45.23
Emission Rate, lb/hr	5.36	5.57	6.62	5.85
<u>CO Results</u>:				
Concentration, ppm_{vd}	0.00	0.00	0.00	0.00
Emission Rate, lb/hr	0.00	0.00	0.00	0.00

[a] Analyzers

[b] 68°F (20°C)--29.92 Inches of Mercury (Hg)

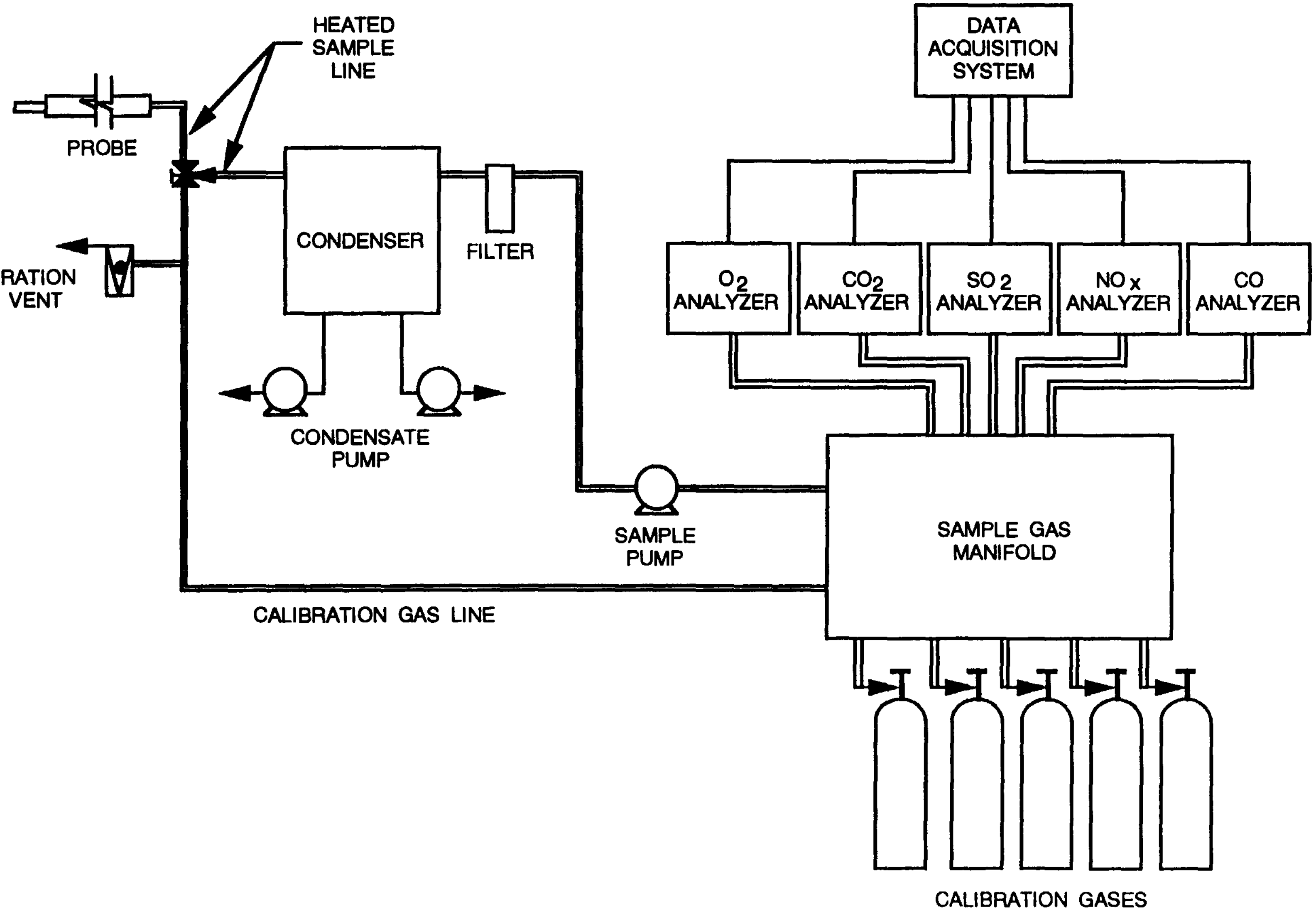
TABLE 11-3d.

CONTINUOUS EMISSIONS MONITORING DATA
AS MEASURED IN STACK 4, CONDITION IV
JEWELL COAL & COKE COMPANY

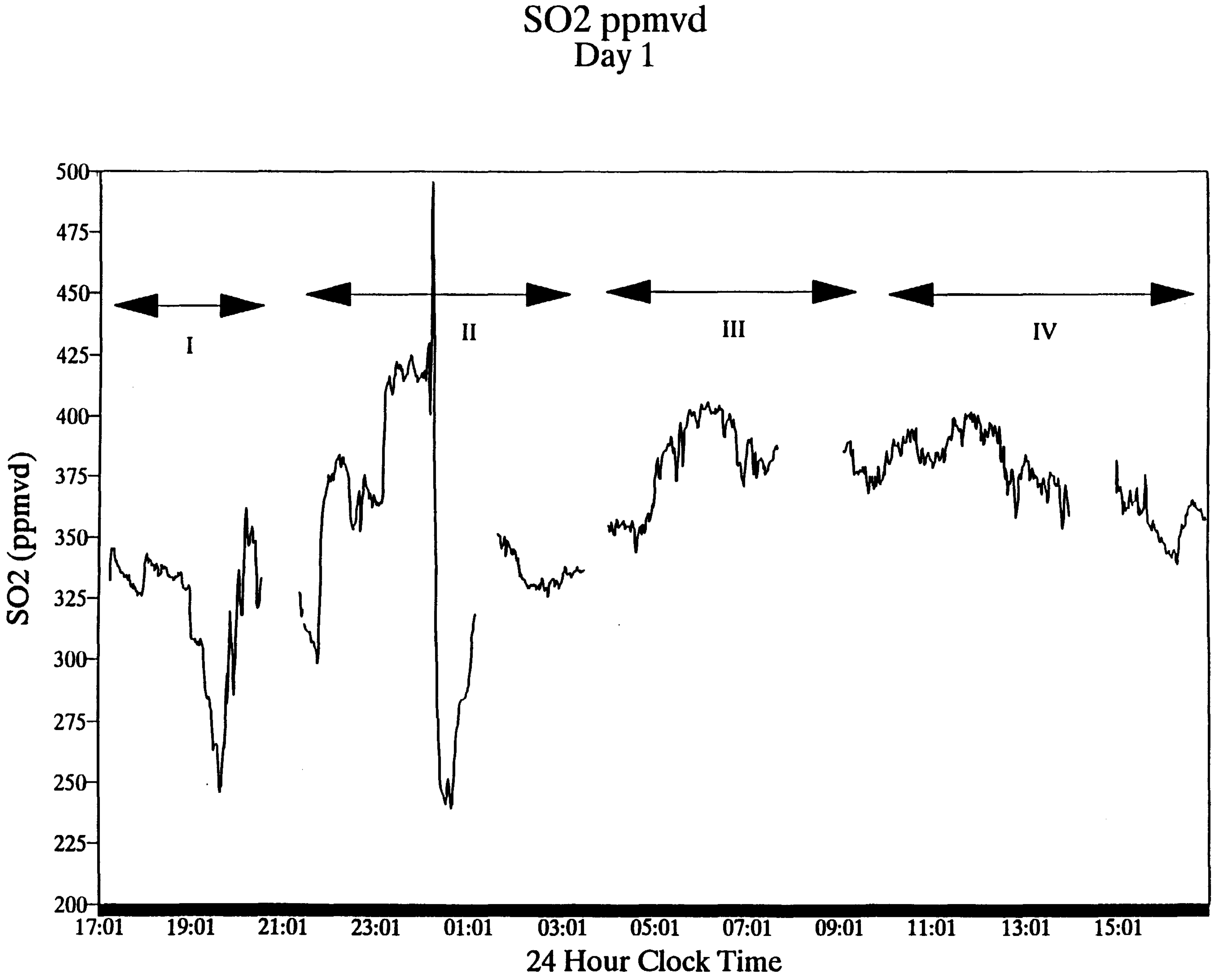
Run Identification	IV-S4-CEM			
Day/Repetition Number	1	2	3	Average
Run Date	10/30/91	10/31/91	11/01/91	
Run Start Time	1021	1001	1049	
Run Finish Time	1700	1700	1516	
Total Run Time, min.	399	419	267	
Flue Gas Parameters:				
CO_2, Percent by Volume, Dry[a]	7.81	6.98	7.63	
O_2, Percent by Volume, Dry[a]	8.55	9.33	8.50	
Flue Gas Temperature, Degrees F	1,518	1,503	1,516	
Velocity, ft/sec	24.85	26.71	27.01	
Air Flow Rate, Dry, SCFM[b]	17,208	18,406	18,670	
Air Flow Rate, Wet, ACFM	77,191	82,934	84,246	
SO_2 Results:				
Concentration, ppm_{vd}	394.2	363.6	362.0	373.27
Emission Rate, lb/hr	67.56	66.66	67.32	67.18
NO_x Results:				
Concentration, ppm_{vd}	39.5	38.7	38.6	38.93
Emission Rate, lb/hr	4.87	5.10	5.16	5.04
CO Results:				
Concentration, ppm_{vd}	0.00	0.00	0.00	0.00
Emission Rate, lb/hr	0.00	0.00	0.00	0.00

[a] Analyzers

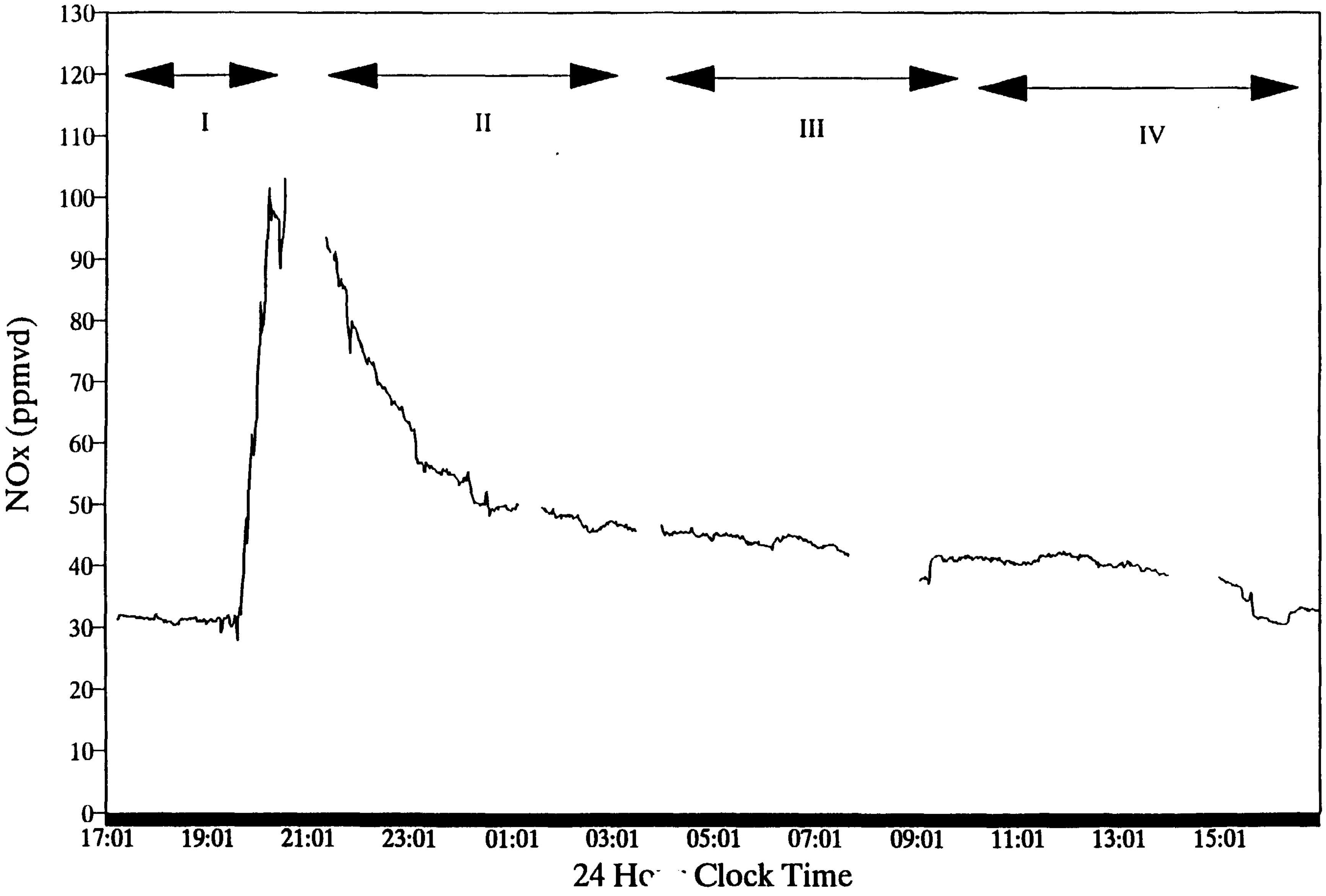
[b] 68°F (20°C)--29.92 Inches of Mercury (Hg)

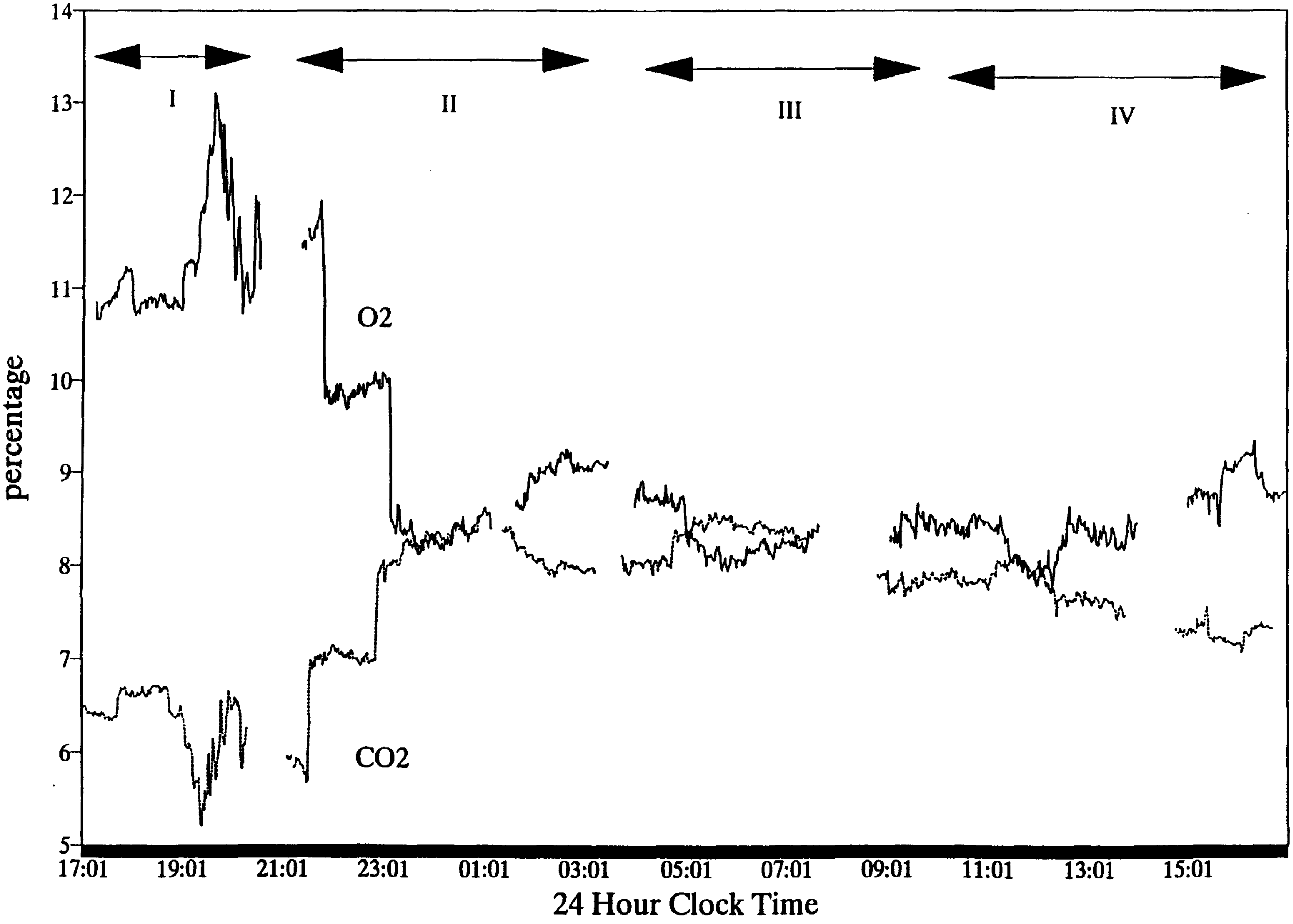


Fiqure 11-1. Figure 11-1 nstrumental measurement system.


SO2 ppmvd
Day 1
SO2 (ppmvd)
500
475
450
425
400
375
350
325
300
275
250
225
200
I
II
III
IV
17:01
19:01
21:01
23:01
01:01
03:01
05:01
07:01
09:01
11:01
13:01
15:01
24 Hour Clock Time

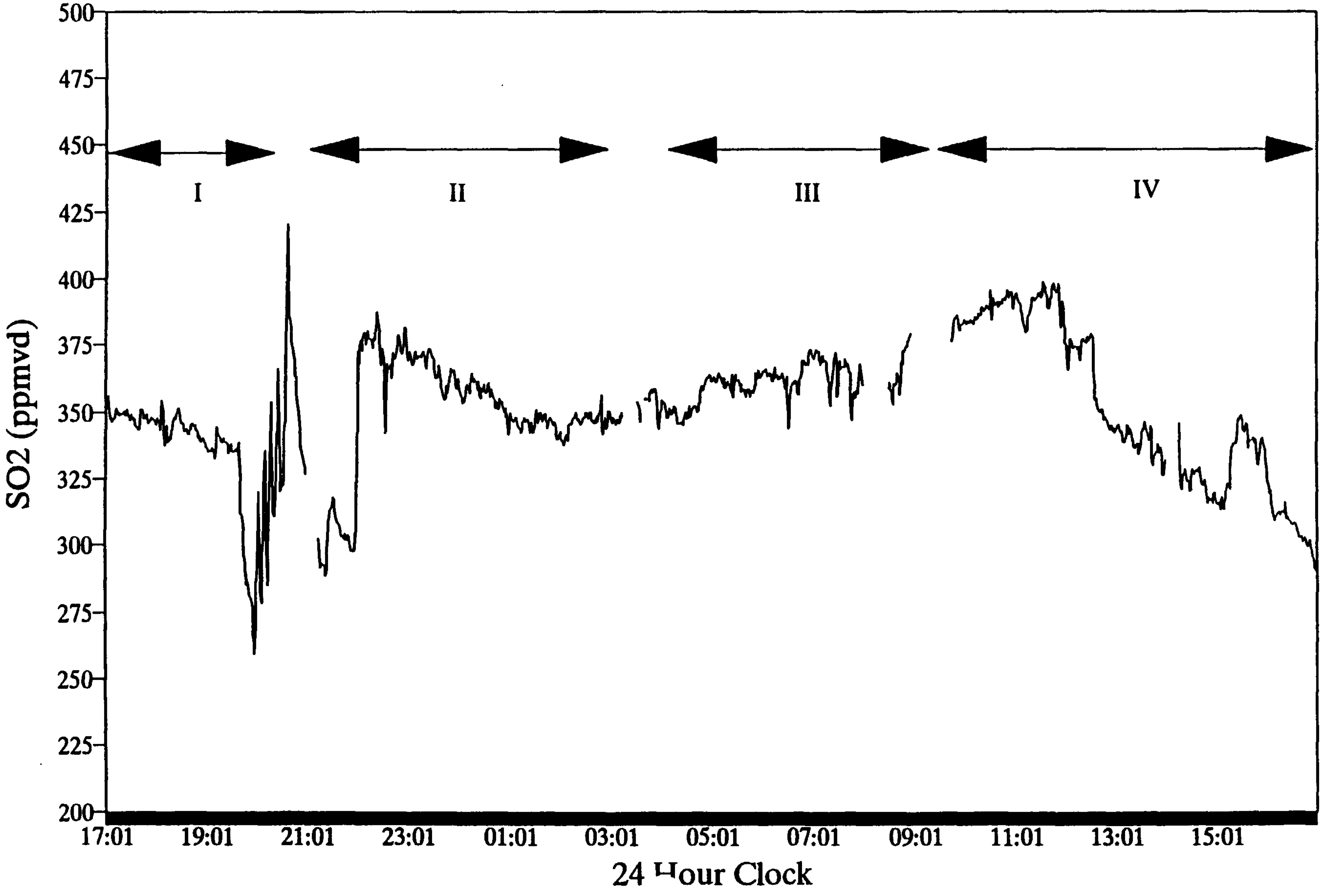
NOx ppmvd
Day 1

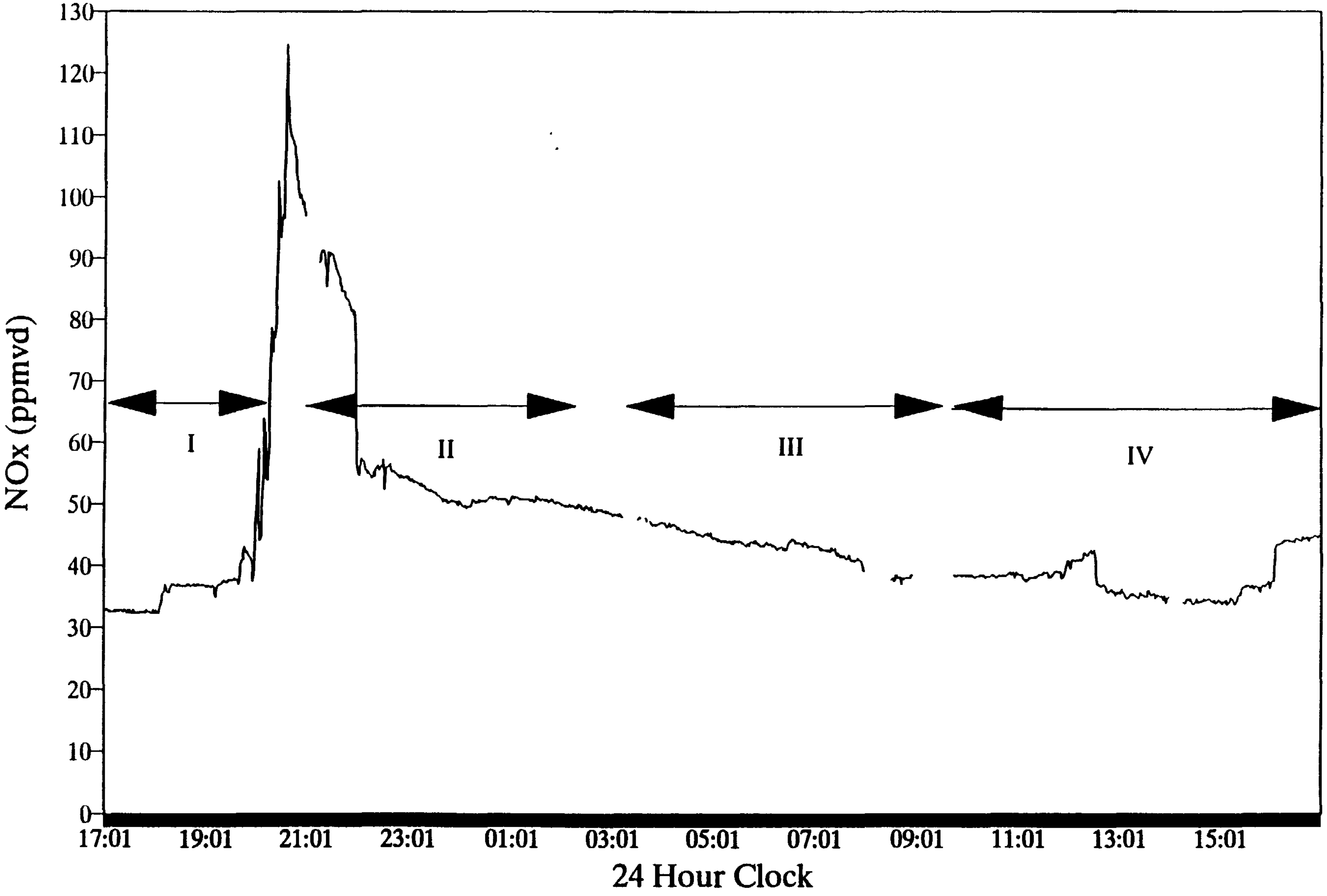




Percent O2 and CO2
Day 1

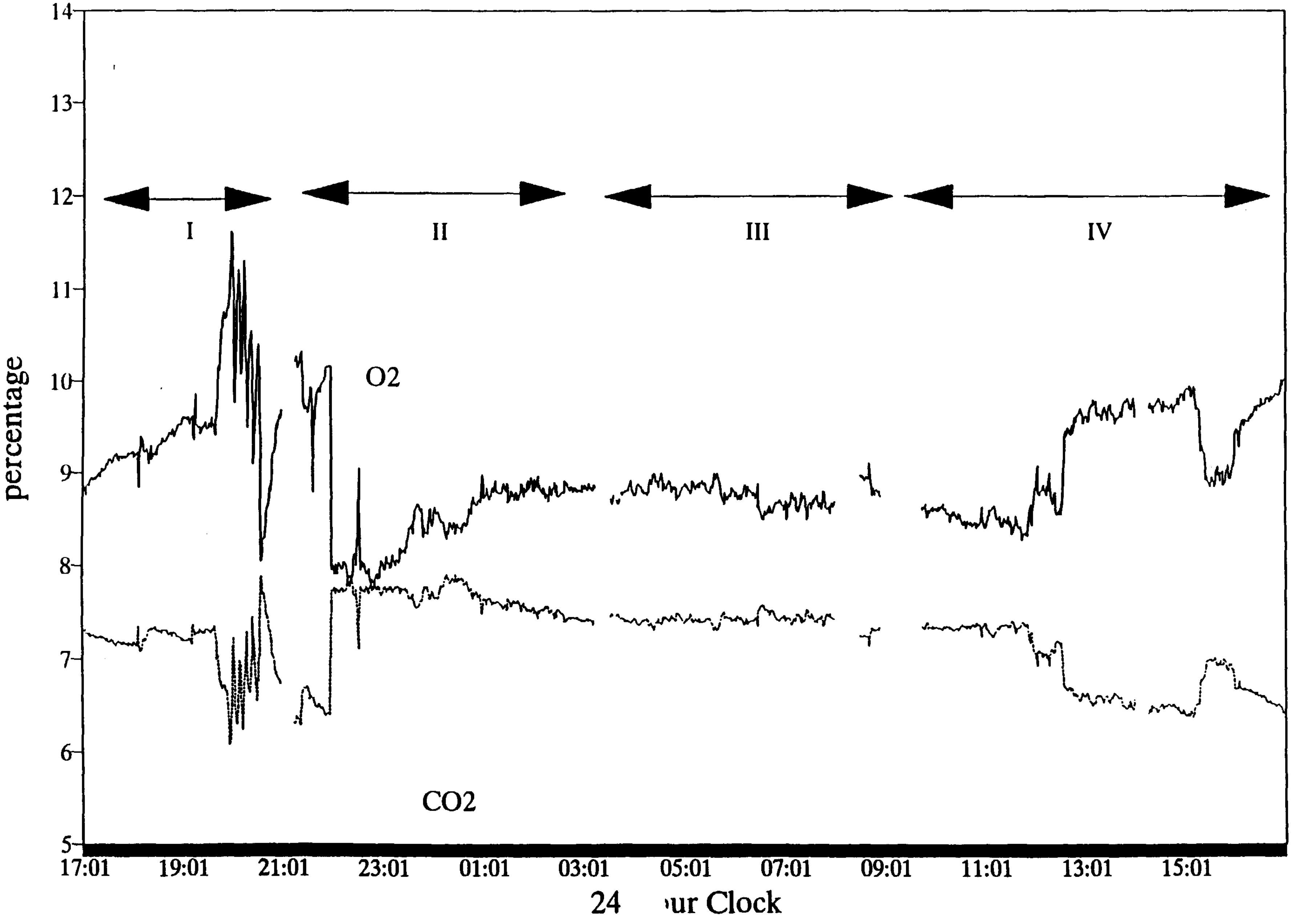
SO2 ppmvd
Day 2



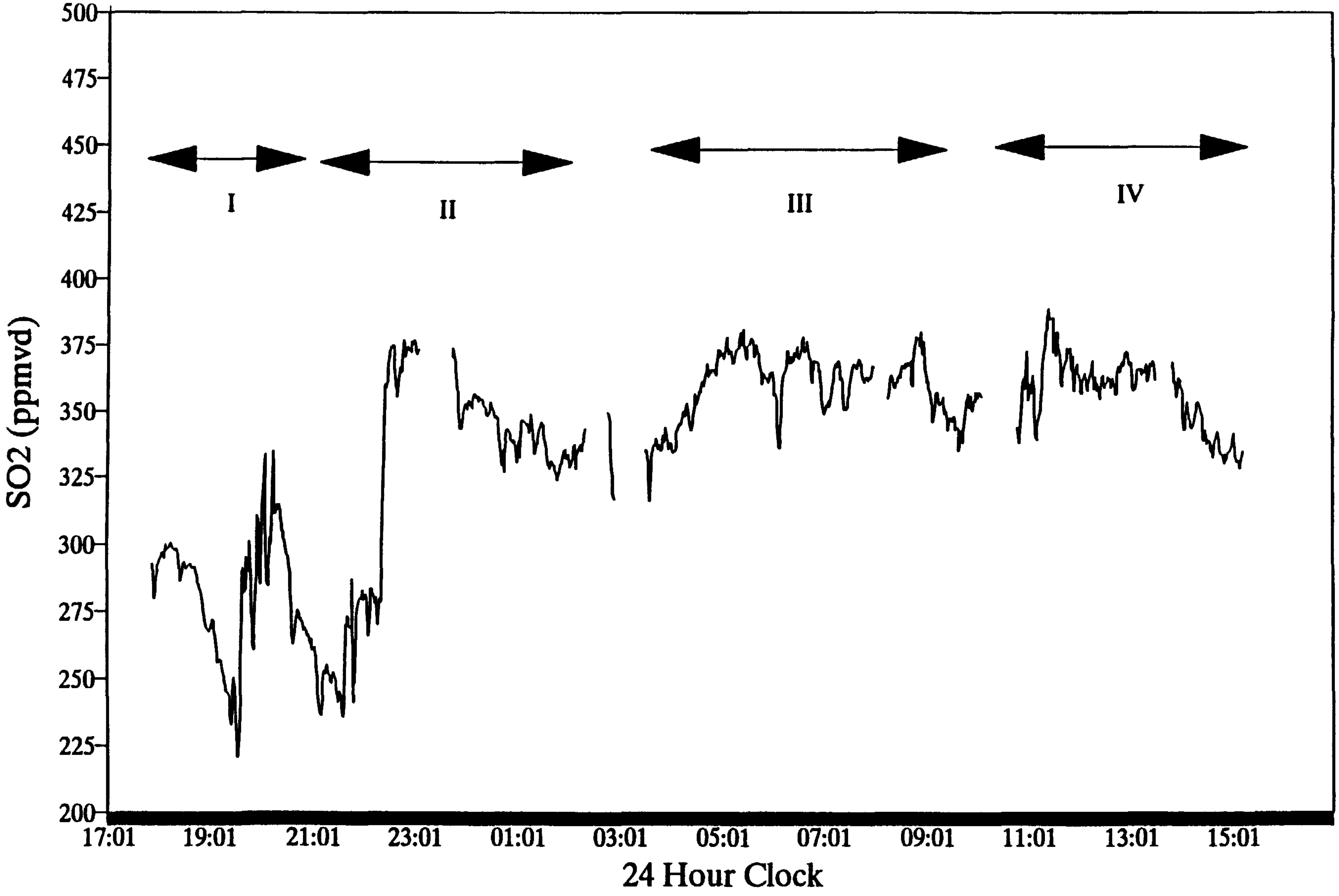


NOx ppmvd
Day 2

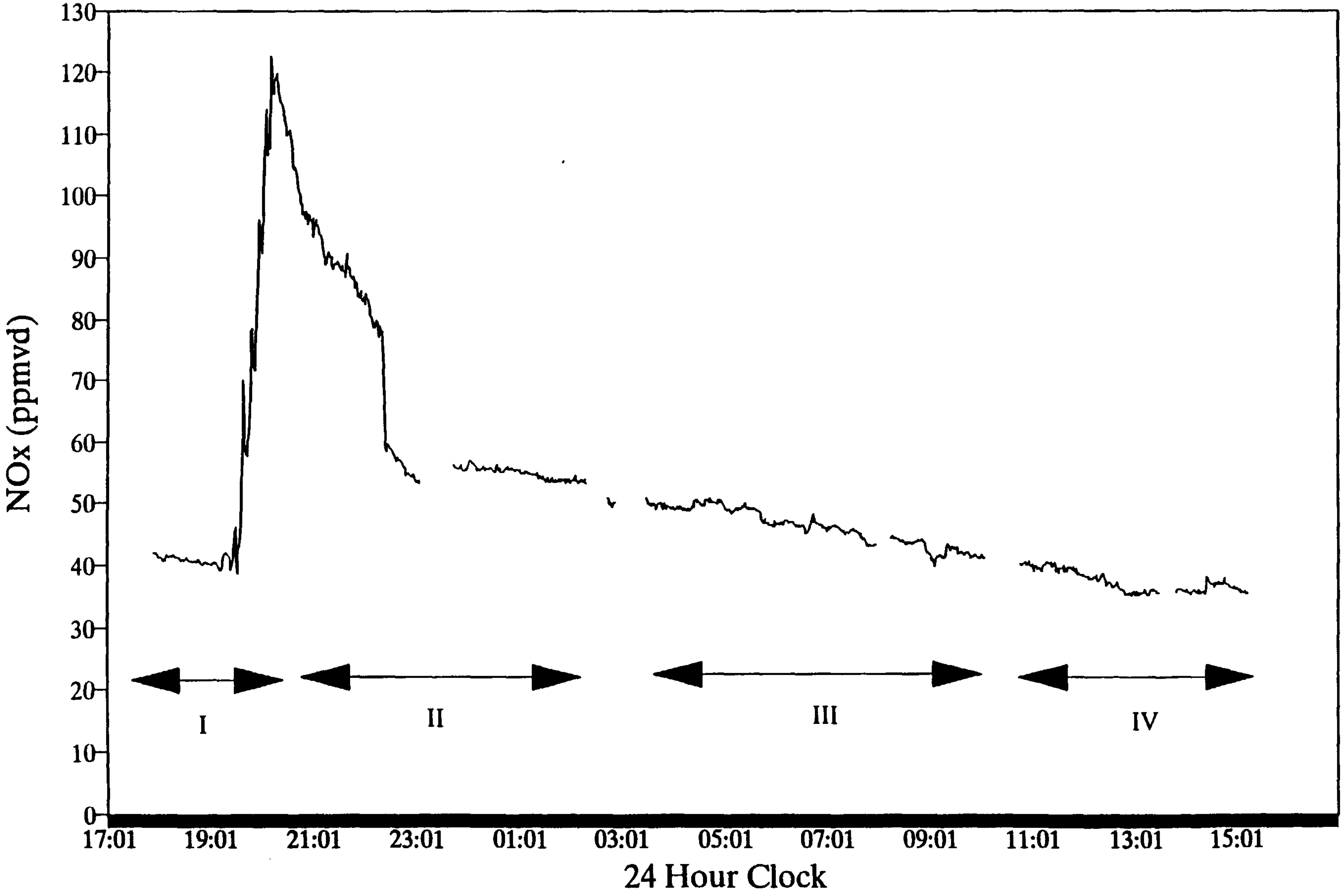
Percentage O2 and CO2
Day 2



SO2 ppmvd
Day 3



NOx ppmvd
Day 3



Percentage O2 and CO2
Day 3