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BENZENE SAMPLING PROGRAM AT COKE BY-PRODUCT RECOVERY PLANTS: UNITED STATES STEEL CORPORATION, FAIRLESS HILLS, PENNSYLVANIA

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1.0 INTRODUCTION

Scott Environmental Services, a division of Scott Environmental Technology, Inc. conducted a sampling program at United States Steel Corporation in Fairless Hills, Pennsylvania to determine benzene emissions from four sources in the coke by-products recovery plant. The work was performed for the United States Environmental Protection Agency, Emission Measurement Branch, under Contract Number 68-02-2813, Work Assignment 48. The Fairless Works was the fourth of seven coke by-product plants visited to collect data for a possible National Emission Standard for Hazardous Air Pollutants for benzene.

Sampling was conducted at U.S. Steel from September 8th through 11th, 1980. Air and liquid samples for benzene analysis were collected from the cooling tower direct water final cooler, wash oil decanter, and from the two tar dehydrators.

2.0 SUMMARY OF RESULTS

Process	Benzene Emission Rate lb/hr	 kg/hr
Cooling tower-direct water final cooler	70.2	31.8
Wash oil decanter	0.87	0.39
Tar dehydrator-west tank	6.95	3.15
Tar dehydrator-east tank	2.45	1.11

3.0 DISCUSSION OF RESULTS

3.1 COOLING TOWER

Hot water from the direct water final cooler is pumped to a hot well, where it is circulated over a 60-foot high atmospheric cooling tower. The tower has a 17 foot diameter fan on top for pulling air countercurrent to the falling water to effect the cooling. The tower also effectively acts as a stripper for benzene contained in the hot well water.

Four EPA Method 110 runs were done on the cooling tower on September 9th and 11th, 1980. Table 3-1 presents the results of the tests. The third run was voided because the concentration in the collected sample was over twice as high as in the first two, indicating a contaminated sampling bag was used. When this was verified by the bag log book entries, the run was declared void and a fourth test was run. The results of the third test are included in Table 3-1 for comparisons of temperature and flow rate.

A 24-point sampling and velocity traverse was made across two diameters of the 17 foot fan shroud to obtain an integrated sample. The average result for the three good tests was 70.2 lb/hr. All stack flowrates were corrected to the average conditions at which the benzene concentrations were measured in the Tedlar bags; assumed to be saturated at 68°F and 29.92 in. Hg. (2½% moisture). Example calculations are shown in Appendix A.

The fourth test yielded higher mass emission rates than the first two tests although the benzene concentration was similar, due to higher measured stack velocities. This could be a result of the high winds on that day, which affected the vane anemometer used to measure velocity, although it was also somewhat windy on the first day, as noted on the field data sheets in Appendix A.

The stack velocity data of all four tests show that one quadrant of the fan shroud had consistently higher velocities than the others. This could be a consequence of localized wind effects, since the wind was predominantly from that side of the tower, or could be the result of a variation in the internal packing structure of the tower which could cause a higher air flow on one side.

Liquid samples were dipped from the hot well and cold well after each of the first three test runs, with temperatures of approximately 95° and 78°F respectively. As shown in Table 3-1, the first hot well sample contained much less benzene than the other two. Replicate analyses verified this result. This apparently indicates a great deal of variability in the feed water benzene concentration, due to stratification or incomplete mixing in the hot well.

3.2 WASH OIL DECANTER

The wash oil decanter separates water from the wash oil before it enters the wash oil cooler by settling out the water due to density differences. The decanter is vented to the atmosphere, and benzene contained in the hot wash oil could be directly emitted from this process.

Triplicate Method 110 tests were run on the decanter. The data shown in Table 3-2 indicate an average emission rate of 0.87 lb/hr benzene. The moisture content of the stream was high; the average for the three runs was 60.5% moisture, as determined from the volume of water collected in the water trap. (See sample calculations in Appendix A). Three moisture determinations were run using silica gel tubes, dry gas meter and pump

TABLE 3-1

COOLING TOWER DATA SUMMARY

Process: Cooling Tower - direct water final cooler

Stack Diameter: 17 ft.

Plant: U. S. Steel, Fairless Hills, Pa.

Stack Area: 227 ft.^{2}

Run No.	Date	Sample Period	Stack Temp. ($^{\circ}$F)	Barometric Pressure (in.Hg)	Stack Velocity (ft/min.)	Flowrate Stack Conditions (ACFM)	Flowrate Standard Conditions (SCFM)	Benzene Concentration (ppm)	Benzene Emission Rate (lb/hr.)
1	9/9/80	1120-1245	82	30.12	1350	306,000	297000	18.77	67.6
2	9/9/80	1417-1530	85	30.12	1170	266,000	255,000	19.80	61.2
3	9/9/80	1547-1700	87	30.12	1230	279,000	265,000	44.4*	--
4	9/11/80	1525-1650	80	29.99	1680	381,000	369,000	18.30	81.9
									Ave. 70.2

Standard conditions: Saturated at 68°F, 29.92 in. Hg

*Contaminated Tedlar Bag

Liquid Sample Data

Sample Location	Date	Time	Sample Temp. $^{\circ}$F	Benzene Concentration (ppm by weight)
Cooling Tower - hot well	9/9/80	1410	93	8.2
		1535	96	48.2
		1715	97	48.8
Cooling Tower - cold well	9/9/80	1410	78	1.0
		1535	78	1.0
		1715	79	0.8

TABLE 3-2

WASH OIL DECANTER DATA SUMMARY

Process: Wash Oil Decanter

Plant: U. S. Steel, Fairless Hills, PA

Stack Diameter: 8"

Stack Area: 0.35 Ft^2

Run No.	Date	Sample Period	Stack Temp. (^oF)	Barometric Pressure (in. Hg)	Stack Velocity (ft/min.)	Flowrate Stack Conditions (ACFM)	Flowrate Standard Conditions (SCFM)	Benzene Concentration (ppm)	Benzene Emission Rate (lb/hr.)
1	9/10/80	1410-1440	193	29.92	795	280	96	706	0.82
2	9/10/80	1508-1538	193	29.92	820	290	67	1052	0.85
3	9/10/80	1555-1625	193	29.92	810	280	110	694	0.95
									Ave. 0.87

Standard conditions: Saturated at 68^oF, 29.92 in. Hg.

Liquid Sample Data

Sample Location	Date	Time	Sample Temp. (^oF)	Benzene Concentration (ppm by weight)
Wash Oil Decanter - dipped from hatchway	9/10/80	1640	205	42.2
	9/10/80	1640	205	40.3
	9/10/80	1640	205	50.3
				Ave. 44.3

as described in Section 6.2, as an additional check to insure the accuracy of the Method 110 data. The average for these three runs was 59.1% moisture, which confirmed the accuracy of our previous findings. These calculations can also be found in Appendix A.

Triplicate liquid samples were dipped from the hatchway next to the sampling vent immediately after the three tests were run. The liquid temperature was 205°F and the average benzene concentration in the samples was 44.3 ppm by weight.

3.3 TAR DEHYDRATORS

Two tanks in series heat the tar from the tar decanter to drive off the entrained water. Benzene contained in the tar is also potentially released with the water.

Method 110 was modified by bubbling the sample stream through propylene carbonate to remove the naphthalene, as described in Section 6.3. Significant amounts of benzene were found in the propylene carbonate solution, and these amounts were added to the total benzene found in the collected bag samples to determine mass emissions from this source.

As shown in Table 3-3, the mass emission rate from the west tank (1st in series) ranged from 1 to 15 lb/hr. with an average of 6.95 lb/hr. and the east tank emission rate varied from 1 to 4 lb/hr. with an average of 2.45 lb/hr. There was great variability between velocity readings, not only between runs but from reading to reading on each single run, as can be seen from the field data sheets in Appendix B. Also the benzene concentration varied from about 700 ppm to over 3000 ppm between the different runs. Generally the benzene concentration increased when stack temperature increased.

TABLE 3-3

TAR DEHYDRATORS DATA SUMMARY

Process: Tar dehydrators - east & west tanks

Plant: U. S. Steel, Fairless Hills, PA

Stack Diameter: 8"

Stack Area: 0.349 $ft.^2$

Run No.	Date	Sample Period	Stack Temp. (°F)	Barometric Pressure (in.Hg)	Stack Velocity (ft/min.)	Flowrate Stack Conditions (ACFM)	Flowrate Standard Conditions (SCFM)	Benzene Concentration (ppm)	Benzene Emission Rate (lb/hr)
WEST TANK (1st in series)									
1	9/11/80	1015-1045	174	29.99	576	200	120	740	1.04
2	9/11/80	1350-1420	182	29.99	1820	640	410	3010	15.01
3	9/11/80	1730-1800	166	29.99	990	350	260	1520	4.79
									Ave. 6.95
EAST TANK (2nd in series)									
1	9/11/80	1100-1130	174	29.99	310	110	76	2410	2.22
2	9/11/80	1345-1415	142	29.99	460	160	120	840	1.17
3	9/11/80	1730-1800	178	29.99	670	230	110	2990	3.89
									Ave. 2.45

Standard conditions: saturated at 68°F, 29.92 in. Hg.

Liquid Sample Data

Sample Location	Date	Time	Sample Temp. (°F)	Benzene Conc. (ppm by weight)	Average
West Tank - inlet	9/11/80	1805	196	285 295 251	277
West Tank - dipped from tank	9/11/80	1820	207	629 599 634	621
East Tank - dipped from tank	9/11/80	1825	219	282 286 261	276

Liquid samples were collected from each of the tanks through the manway and from the inlet to the west tank in the funnel. As shown in Table 3-3, the west tank inlet had an average benzene concentration of 280 ppm, while the west tank process liquid contained 620 ppm benzene. It is possible that the funnel is not the only inlet to the west tank, or that there is great variability in the composition of the inlet liquid because of multiple sources.

4.0 PROCESS DESCRIPTION

The U.S. Steel facility at Fairless Hills, Pennsylvania, operates two coke batteries, each with 87 Wilputte ovens. The unit operations at the coke by-product plant are primary cooling, tar decanting, exhausting, tar electrostatic precipitation, ammonia recovery, naphthalene recovery, final cooling, and light oil recovery.

The gas leaving the ovens is collected in a collecting main where it is sprayed with flushing liquor to reduce the temperature to about 175°F. When the gas and flushing liquor leave the battery area, the flushing liquor separates from the gas and travels to the flushing liquor decanter. The gas is transported from the collecting main through a crossover main, and it proceeds to the primary coolers; gas enters the primary cooler at about 175°F and leaves at 95°F. Water from the primary cooler goes to the tar decanter, and the tar from the tar decanters and flushing liquor decanter is pumped to the tar dehydrator. Following the primary coolers, the gas stream is pressurized by steam turbine-driven exhausters and then it enters the electrostatic precipitator. The flushing liquor decanter separates the dirty liquor into flushing liquor, tar, and sludge. Flushing liquor is returned to the batteries for reuse, and any excess is pumped to the phenolized weak ammonia tanks. After passing through the electrostatic precipitor, the gas is conveyed to the ammonia recovery area. Ammonia is recovered by using a 5 to 6 percent sulfuric acid solution; the final product of this operation is ammonium sulfate crystals.

The coke oven gas proceeds from the ammonia recovery area to a direct water final cooler. The final cooler water flows from the cooler to a separation basin where crude naphthalene is skimmed from the surface, dried to 3 to 4 percent moisture, and pumped to storage. The water is pumped from the separation basin to the hot well of the final cooler cooling tower.

After cooling, the coke oven gas enters the two wash oil scrubbers which are in series with the countercurrent flow of the wash oil and the gas stream. The benzolized wash oil is pumped to the light oil recovery area where light oil is removed by steam stripping. The light oil flows to the crude residue separation column to separate the primary light oil (0° to 150° fraction). The primary light oil vapors are cooled in a condenser, and the primary light oil goes to a light oil decanter, which is not vented to the atmosphere, for water removal. The primary light oil is collected in a tank and pumped to storage before it is shipped. The secondary light oil (150° to 290° F fraction) is separated, recovered, and stored. No further processing of the recovered oils is performed at the Fairless Hills plant.

After being stripped of light oils, the debenzolized wash oil flows to a wash oil decanter. Water is removed, and the wash oil is cooled in an indirect cooler and is returned to the wash oil scrubbers.

The clean coke oven gas exiting the wash oil scrubbers is used for fuel. Approximately 35 percent of this gas is used for battery underfire while the remainder is piped to the mill for operations such as firing the boilers and furnaces.

4.1 PROCESS OPERATING PARAMETERS

The emissions from the final cooler cooling tower, wash oil decanter, and tar dehydrator were tested.

The following process data were collected during the benzene emission testing.

A. Process parameters

- The water flow rate in the final cooler cooling tower was 2,100 gallons/minute.
- The flow rate of benzolized wash oil from the wash oil decanter was 27,500 gallons/hour.
- The residence time of tar in the tar dehydrator was 24 hours.

B. Production data

Item	9/9/80	9/10/80	9/11/80
Coal charged/day (tons)	3,770	3,776	4,027
Coke produced (tons)	2,760	2,713	2,811
Coke oven gas produced (cubic feet)	40,678 x 10^3	40,918 x 10^3	41,672 x 10^3
Light oil produced (gallons)	7,185	9,733	7,571
Naphthalene produced (gallons)	490	450	475
Tar produced (gallons)	31,030	28,960	33,100

The coal mixture was 70 percent high volatile coal and 30 percent low volatile coal.

5.0 FIELD SAMPLING AND ANALYSIS METHODOLOGY

5.1 DETERMINATION OF BENZENE FROM STATIONARY SOURCES: EPA METHOD 110 AND MODIFICATIONS

EPA Method 110 consists of drawing a time-integrated stack gas sample through a probe into a Tedlar* sample bag, which is enclosed in a leak-free drum, by use of a pump hooked to the drum outlet which slowly evacuates the drum, causing the bag to fill. A copy of the method is included in Appendix D.

The method was modified by Scott because as it stands the method doesn't account for moisture in the sample stream, and is only designed to measure benzene concentration, not mass emission rate. The following modifications were made to all tests done using Method 110:

1. To obtain mass emission rates, velocity and temperature readings were taken at the top of the stack at 5 minute intervals during the 30-minute sampling runs. This information was used to calculate flow-rate, which was used in conjunction with the benzene concentration to yield the mass emission rate. Velocity readings were made using a vane anemometer with direct electronic readout.

2. A personnel sampling pump was substituted for the pump, needle valve, and flowmeter of the method. The personnel pumps have built-in flowmeters and rate adjustment screws and have the further advantage of being intrinsically safe, as required in many areas of the coke plant.

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* Mention of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

3. Swagelok fittings were used in place of quick-connects.

4. Rather than discarding Teflon sample lines after each set of samples, they were washed with propylene carbonate and/or acetone and flushed with nitrogen before reuse.

5. An orifice and magnehelic gauge were inserted in the sampling line before the Tedlar bag to indicate that air flow was reaching the bag.

6. A water knockout trap was inserted between the probe and magnehelic gauge to collect any condensate in the sample line.

7. The following cleanup procedures were followed:

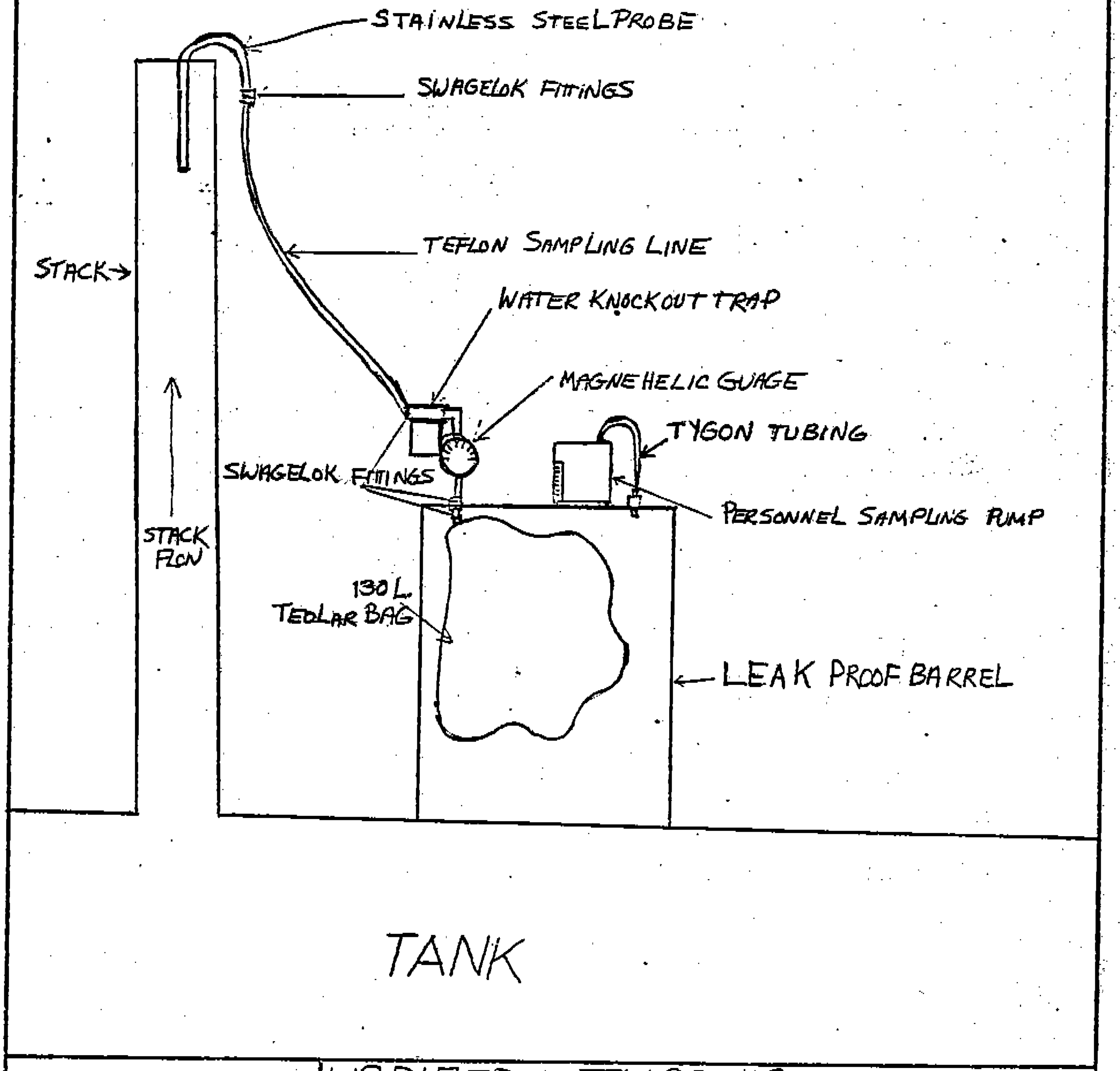
If any condensate was collected in the trap or sample line, it was measured and saved for analysis. The probe, line and trap were then washed with propylene carbonate, which was also saved for analysis. Any benzene found in these washes and water catches was added to the total found in the sample bag to determine mass emission rates.

Bag volumes were measured whenever water was collected in the trap by emptying the bag through a dry gas meter after the sample was analyzed. The volume of water collected in the trap was then converted to an equivalent air volume and was added to the volume in the bag to determine the percent moisture in the sample stream.

After the probe, line and trap washes were completed, the lines were washed with acetone to remove the propylene carbonate film and flushed with nitrogen to dry.

Figure 5-1 shows the modified Method 110 setup.

FIGURE 5-1



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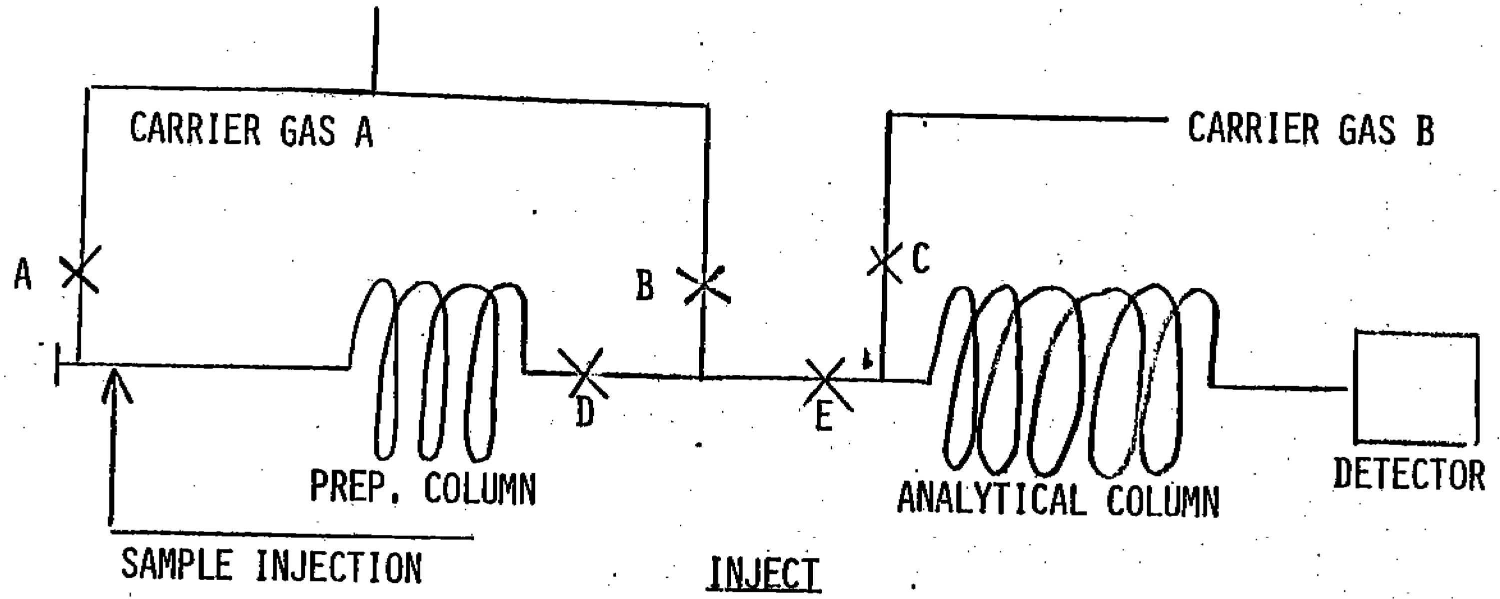
MODIFIED METHOD 110
SAMPLING TRAIN

5.2 SAMPLE HANDLING

After being collected the gas samples were immediately transported to the gas chromatograph and analyzed. The elapsed time between sample collection and analysis never exceeded one hour. To verify that there was no sample degradation in samples of this type some of the samples were retained for 24 hours and reanalyzed. The loss of benzene and isobutane observed was typically less than 5%.

5.3 FIELD ANALYSIS

All gas samples collected were analyzed using a Shimadzu GC Mini 1 gas chromatograph equipped with dual flame ionization detectors, dual electrometers, heated sample loop and a backflush system. Figure 5-2 shows a schematic of the backflush apparatus. The backflush system is composed of a ten port sequence reversal valve and two columns, a scrubber column for retaining high molecular weight compounds and an analytical column. When the system is in the inject mode the scrubber column and the analytical column are connected in series allowing sample components to move from the precolumn to the analytical column. In the backflush mode the columns are disconnected from each other and become two separate systems each with its own carrier gas source. This arrangement allows the separation and measurement of low molecular weight compounds while the scrubber column is being backflushed of heavier sample components. Backflush times for different mixtures of sample components must be predetermined to insure that the compound(s) of interest are transferred to the analytical column before backflushing is started.



INJECT

A, D, E OPEN
B, C CLOSED

BACKFLUSH

A, E CLOSED
B, C, D OPEN

GC COLUMN CONFIGURATION WITH BACKFLUSH

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FIGURE 5-2

Samples for chromatographic analysis were drawn into a 20 cc glass syringe then introduced to the sample loop inlet. The samples once in the sample loop were allowed to come to atmospheric pressure by waiting 15 seconds prior to injection. The following chromatographic conditions were maintained:

Column Temperature (isothermal)	- 100°C
Injector and Detector Temperature	- 200°C
5 ml Sample Loop, Temperature	- 50°C
Carrier Gas Flow Rate	- 32 cc/min.
Hydrogen Flow Rate	- 40 cc/min.
Air Flow Rate	- 240 cc/min.
Analysis Time	- 5 min.
Detector	- Flame Ionization

The columns used for field analysis were:

A - Scrubber Column

10% FFAP on Supelcoport 80/100
1/8" x 1 m Stainless Steel

B - Analytical Column

20% SP-2100, 0.1% Carbowax 1500
100/120 Supelcoport
1/8" x 10' Stainless Steel.

6.0 FIELD SAMPLING

6.1 COOLING TOWER

The cooling tower stands about 60 feet high and has a 17-foot diameter fan on top surrounded by a 15-foot high shroud. Self-contained breathing apparatus was required by the plant for anyone working at the level of the top of the shroud.

Sampling was conducted in accordance with EPA Method 110, modified as described in Section 5.1, and utilizing a 24-point sampling and velocity traverse to collect an integrated sample and an accurate velocity profile. The sampling time at each traverse point was two minutes.

The third run was voided because the concentration of benzene in the collected sample was over twice as high as in the first two, indicating a contaminated sampling bag. When checking the bag history, it was found that the bag had been previously used at a very high concentration source, and cleaning the bag following our standard procedure would have left considerable residual benzene. The bag had not been checked for background benzene prior to use due to an oversight when packing to leave the previous plant. The run was therefore declared void and a fourth test was run.

Liquid samples were extracted from the hot and cold wells after each of the first three test runs using an aluminum can on a rope. Amber glass bottles were then filled from the can and the samples were returned to Scott's laboratory for analysis.

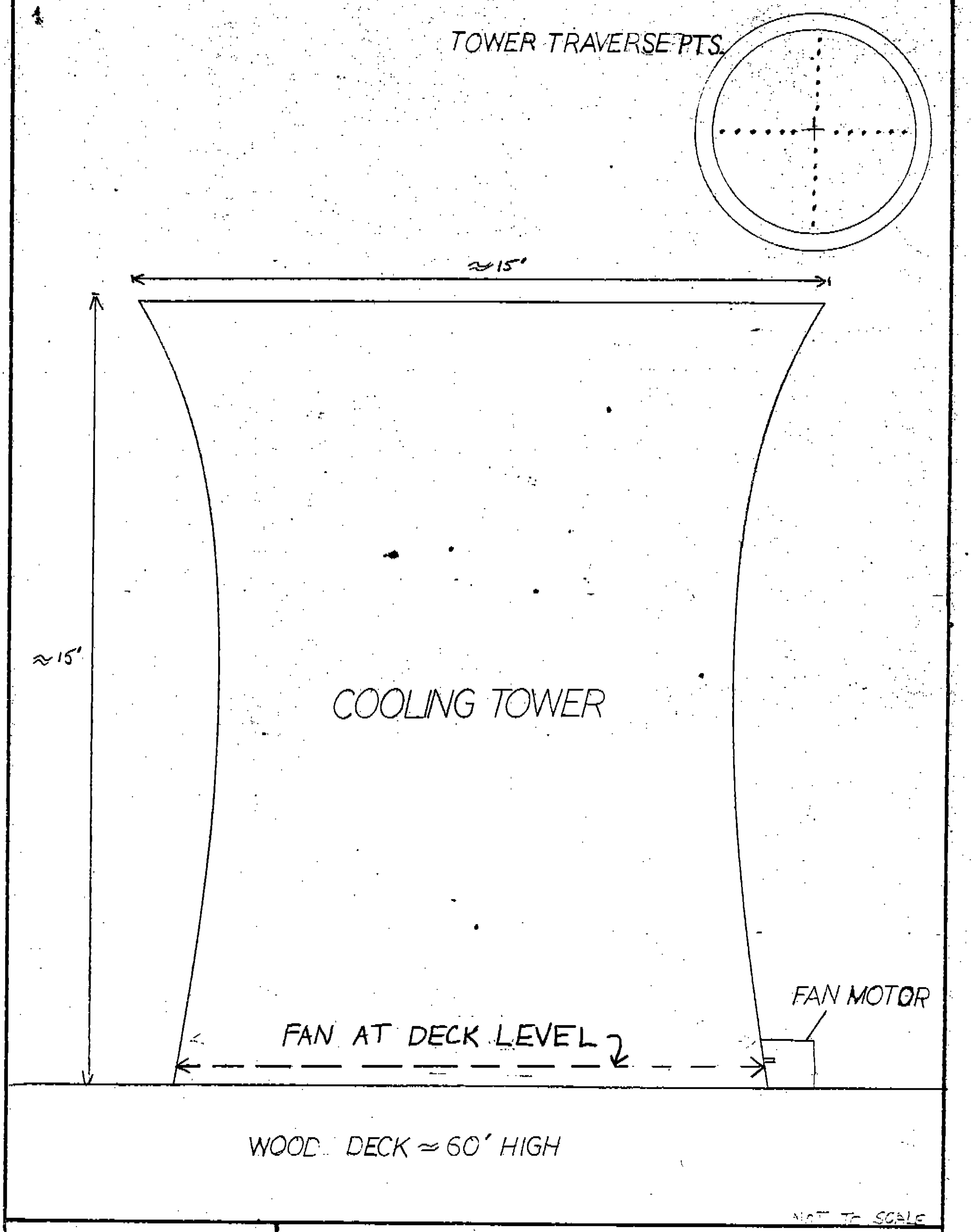

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FIGURE 6-1 COOLING TOWER FAN SHROUD

6.2 WASH OIL DECANTER

Three Method 110 tests were conducted on the wash oil decanter. The moisture content of the stack gas was high, and the water trap catch volume was measured to use in calculating the percent moisture. As a check on the accuracy of the moisture content determined from the water catch, three separate moisture determination tests were run by drawing a source sample through tared silica gel tubes connected to a calibrated dry gas meter using a personnel sampling pump. The average results of the two moisture determination methods were 60.5% for the water catch and 59.1% for the silica gel, indicating that the method using the water catch volume is accurate and can be used with assurance for other process calculations.

The wash oil in the decanter is very hot (205°F) and the hatchways on the tank (see Figure 6-2) are normally open in summer to release heat. During sampling, the hatchways were closed so emissions were coming only from the vent stack.

Liquid samples were dipped from the hatchway on the inlet end of the tank, next to the sampling vent. The liquid level was within 6" of the top of the decanter. Samples were dipped with an aluminum sampling container and allowed to cool slightly before transferring to amber glass bottles.

6.3 TAR DEHYDRATORS

Triplicate Method 110 tests were run simultaneously on the east and west tar dehydrators shown in Figure 6-3. In addition to the method modifications described in Section 5.1, some additional changes were made to deal with the problem of naphthalene constantly plugging the probe and

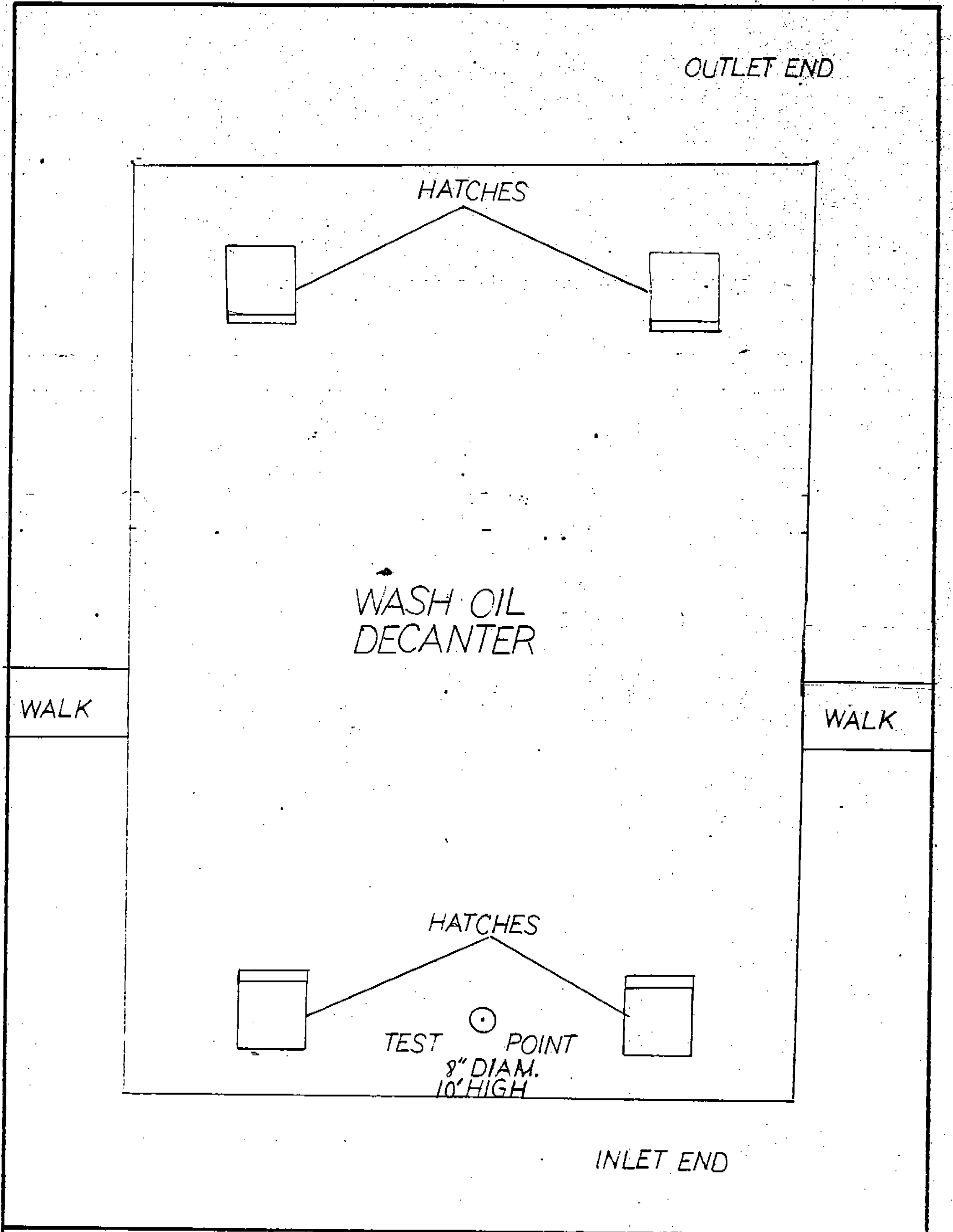

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FIGURE 6-2 WASH OIL DECANTER

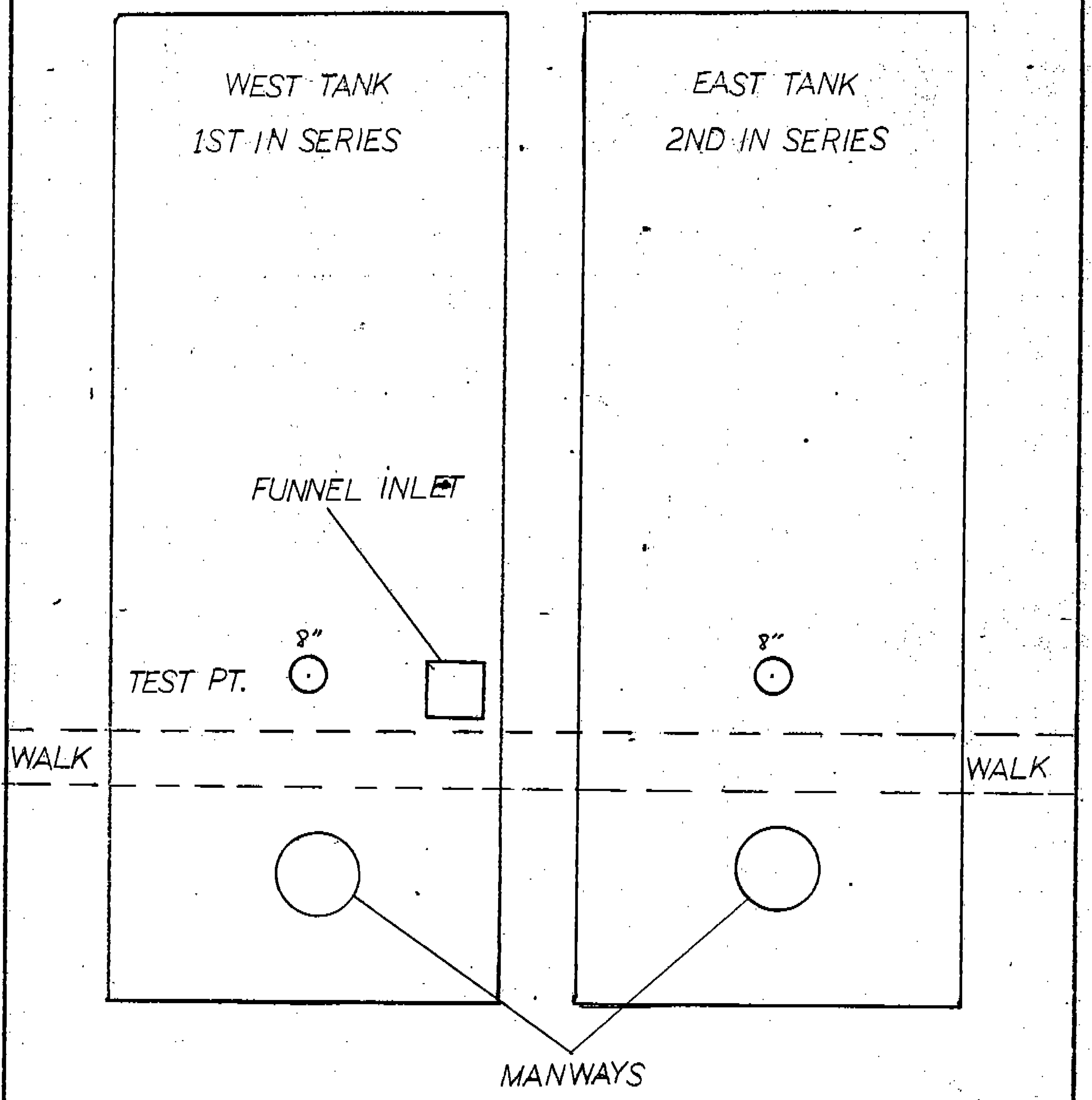

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FIGURE 6-3 TAR DEHYDRATORS - EAST & WEST TANKS sample line. The sample stream was bubbled through propylene carbonate to knock out the naphthalene, using a large diameter glass elbow as a probe. After the naphthalene was scrubbed out, the sample stream passed through Teflon tubing and on into the sampling drum as usual. Figure 6-4 shows the propylene carbonate sampling train. The glass probe was connected directly to two impingers each containing 100 ml of propylene carbonate and a third empty impinger. The impingers were contained in a bucket which was hooked onto the top of the stack.

Cleanup consisted of saving the impinger catches and washes in addition to the sample line and water trap washes for analysis of benzene.

7.0 LABORATORY SAMPLE ANALYSIS

Two types of liquid samples were collected: process liquids, and sample line and water trap catches and washes. All liquid samples were stored in amber glass bottles and returned to Scott's Plumsteadville laboratory for analysis.

7.1 SAMPLE PREPARATION

Depending upon the complexity of the sample, one of the following sample preparation procedures was followed prior to the "purge and trap" procedure and analysis.

Samples Containing Immiscible Liquid Phases

Using a clinical centrifuge (International Equipment Company, Massachusetts) immiscible liquid phases were separated and each phase was analyzed separately for benzene.

Samples Containing Solid and Immiscible Liquid Phases

Samples containing solids of higher density than the liquid phase were separated by centrifuge or by simple decantation of the liquid. The different phases in the liquid fraction were then further separated by centrifuging. Solid and liquid phases were analyzed separately.

Samples Containing Finely Crystalline Solid Suspension

In analyzing these samples the stoppered sample jars were shaken for at least half an hour for homogenizing the solution. The uniform distribution of suspended fine crystalline solid particles was tested by determining the percentage of dry solid in several aliquots of the homogenized mixture. A weighed amount of the mixture was analyzed for benzene.

Sampling System Washings

All washings were clear solutions having only one liquid phase. The total weight of the liquid phase was determined using a balance correct to ±0.1 g. The total weight of each washing was more than 25 grams, so an error of 0.1 g in weighing the mass will contribute an error of only 0.4% to the final analytical data. A weighed aliquot of the washing was analyzed for benzene by following the "purge and trap" and analysis procedures outlined in the following sections, and using this analysis data the weight of benzene present in the total mass of washing was calculated.

7.2 PURGE AND TRAP PROCEDURE FOR EXTRACTION OF BENZENE FROM LIQUID PHASE TO GASEOUS PHASE

An accurately weighed quantity of the sample to be analyzed was diluted with 20-25 ml of propylene carbonate in a specially designed glass purging apparatus which was kept immersed in a thermostatted water bath maintained at 78°C. Benzene free nitrogen gas was bubbled through the propylene carbonate solution in the purging apparatus at the rate of 0.2 - 0.3 liters/minute, and collected in leak free Tedlar bags. Under these experimental conditions, 1 1/2 - 2 hours were sufficient to purge off all the benzene from the liquid phase to the gaseous phase. The total volume of nitrogen gas used to purge the sample was accurately measured by a calibrated dry gas meter. A diagram of the purge and trap set-up is shown in Figure 7-1.

Propylene carbonate was found to be an ideal diluting solvent for the extraction of benzene from all types of liquid samples containing viscous tar, pitch, light and heavy oil and insoluble particulates. It was chosen for its high boiling point, low density, and good solvating capacity.

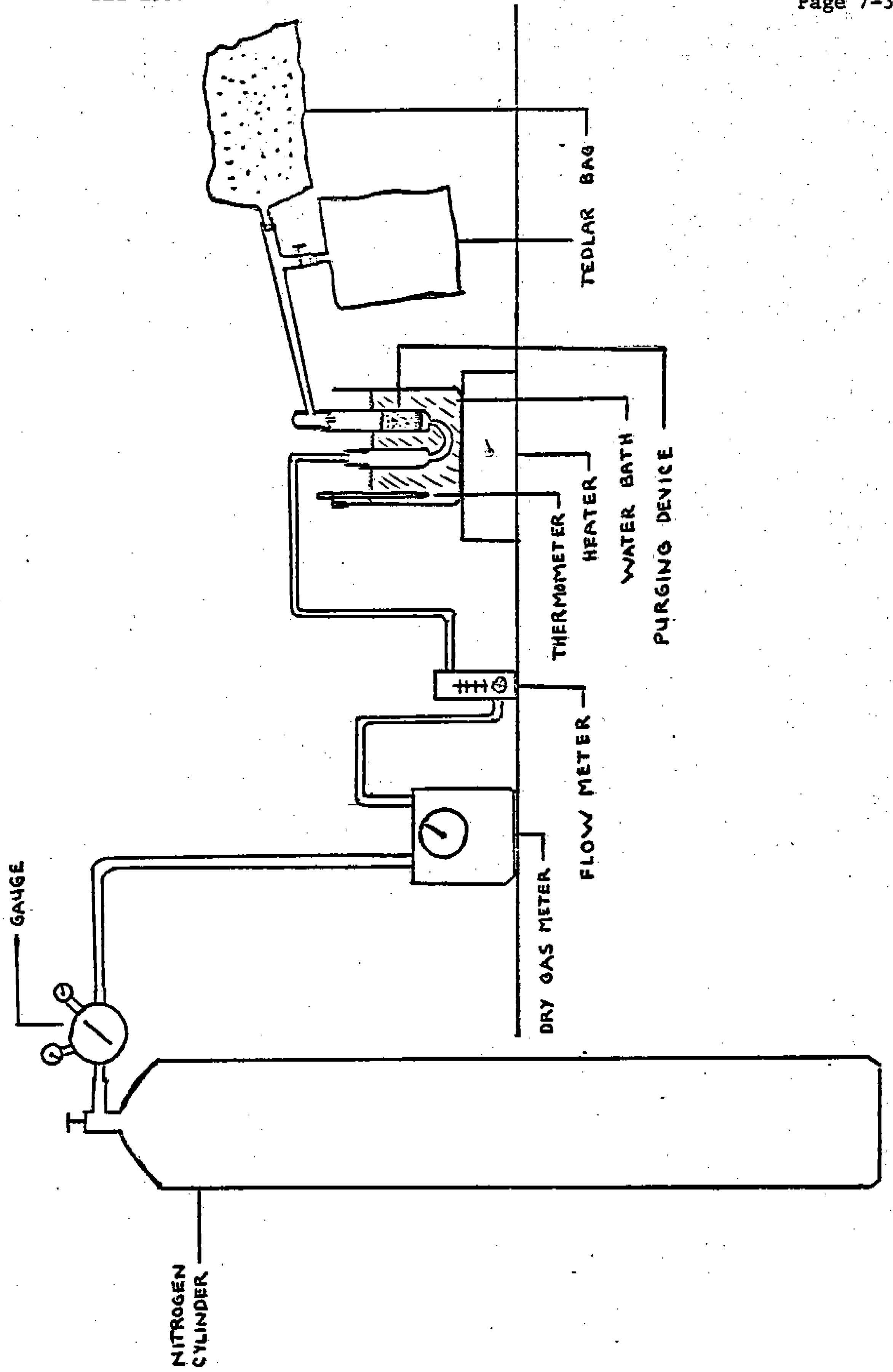


FIGURE 7-1 PURGE AND TRAP METHOD EQUIPMENT SET-UP

7.3 GAS CHROMATOGRAPH

A Perkin-Elmer 900 gas chromatograph was used for the analysis of the purge bags. A 10 ft. by 1/8 inch stainless steel column packed with 20% SP-2100/0.1% Carbowax 1500 on 80/120 mesh Supelcoport was used for the analysis. This column gave complete resolution of the benzene peak from other components present in the purge bags. The 'peak height' method was utilized to calculate the concentration of benzene in the purge bags analyzed. The Perkin-Elmer 900 used for analysis was not equipped with a backflushing unit. Gas chromatograph conditions were as follows:

GC column temperature: 70°C isothermal
Detector temperature: 190°C
5 ml loop at a temperature of 120°C
Carrier gas flow rate: 30 cc/min He
Hydrogen flow rate: 45 cc/min
Oxygen flow rate: 400 cc/min
Detector: Flame Ionization Detector (FID)

In addition to benzene, the purge bags contained other volatile hydrocarbons present in the liquid samples such as toluene and naphthalene. Because this chromatograph was not equipped with a backflush, it was necessary to elute all heavy organics from the column by heating the column to 150°C after every two injections for one hour with the carrier gas on. After cooling the column to 70°C the absence of any organic in the column which might overlap the benzene peak in the next analysis was checked. When the column was found to be satisfactorily clean, the next analysis was continued under the conditions previously described.

8.0 QUALITY CONTROL AND QUALITY ASSURANCE

The following sections will address quality control and quality assurance procedures for the field analysis of benzene in air samples and the laboratory analysis of process liquids and BaP samples.

8.1 FIELD ANALYSIS PROCEDURES

All samples were analyzed in duplicate and as a rule peak heights were reproduced to within 5%. For some very high concentration samples (percent range) it was necessary to make dilutions for analysis. When this was done a fresh dilution was prepared for each injection and peak heights were reproduced to within 10%. To verify that the system was retaining no benzene, frequent injections of the standard and nitrogen were made. In all cases the result was satisfactory.

The Tedlar bags that were reused for sampling were flushed three times with nitrogen and allowed to sit overnight after being filled to approximately three quarters of their capacity. They were analyzed for benzene content the following day. The background concentrations of the bags were recorded and varied from 0 to 10 ppm benzene. Care was taken to use sample bags whose background concentration was very low compared to the expected concentration of the source.

The accuracy and linearity of the gas chromatographic techniques used in this program were tested through the use of EPA Audit Samples. Two standards, a 122.5 ppm and 6.11 ppm benzene were used to analyze the audit cylinders.

8.2 PROCEDURES FOR ANALYSIS OF PROCESS LIQUIDS

Scott's benzene standards, checked against EPA Audit Standards, were used as reference standards throughout this program. The accuracy and linearity of the gas chromatographic technique for benzene analysis was tested through the use of EPA Audit Standards which were available to Scott. Gas chromatographic analysis of the samples and standard were performed under identical conditions to assure the accuracy of the analytical data generated.

Each batch of propylene carbonate which was used as the diluting solvent in the purge and trap technique was analyzed for benzene content by subjecting 25 ml of propylene carbonate to the purge and trap procedure followed by gas chromatographic analysis of the trapped gas under identical conditions as described in Section 5.2. All batches of analytical grade propylene carbonate were found to be free from benzene.

Every day before the analysis of samples the purging apparatus and trapping bags were tested for absence of benzene. Whenever the whole system was found to be free from benzene to the lowest detectable limit of the instrument, the samples were analyzed using the purging apparatus and the trapping gas sampling bags.

Generally an accurately weighed mass of each sample was subjected to purge and trap procedure only once and the trapped gas sample was repeatedly analyzed by GC until the analytical data of consecutive GC analyses varied by ±0.5% or less.

For randomly selected samples, the whole analytical procedure was repeated with a different weighed mass of the source sample to check the validity and accuracy of the analytical methodology. The analytical data for different runs were found not to vary by more than 5%.

By purging the sample with nitrogen under the experimental conditions as utilized by Scott, the recovery of benzene from the sample was quantitative and this has been verified by analyzing a standard benzene solution in propylene carbonate containing tar and pitch.

APPENDIX A

SAMPLE CALCULATIONS

APPENDIX A

SAMPLE CALCULATIONS

1. Calculation of Percent Moisture

A. From water trap catch and bag volume

Example: Wash oil decanter, Run 3

Water trap catch volume: 26.5 cc H_2O

Tedlar bag volume (gas sample): 35.09 liters

Gaseous volume of collected water, standard conditions

$$26.5 \text{ cc} \times \frac{1 \text{ gm}}{\text{cc}} \times \frac{1 \text{ mole}}{18 \text{ g}} \times \frac{24.15 \text{ l}}{\text{mole}} = 35.55 \text{ liters}$$

Percent Moisture

$$\frac{35.55}{35.55 + 35.09} = 50.33\%$$

B. From silica gel moisture determination

Example: Wash oil decanter, silica gel, Run 3

Metered gas volume: 10.28 liters

Mass of water in silica gel: 9.15 g

Gaseous volume of collected water, standard conditions

$$9.15 \text{ g} \times \frac{1 \text{ mole}}{18 \text{ g}} \times \frac{24.15 \text{ l}}{\text{mole}} = 12.20 \text{ l}$$

Percent Moisture

$$\frac{12.20}{12.20 + 10.28} = 54.3\%$$

2. Flow Rate at Standard Conditions (Saturated at 68°F, 29.92 in. Hg)

A. Correction for temperature and pressure:

$$\text{Flow Rate (STP)} = \text{Flow Rate (Source} \times \frac{528°\text{R}}{\text{T(°F)} + 460} \times \frac{\text{P}_{\text{bar}}\text{(in. Hg)}}{29.92}$$

Example: Wash oil decanter, Run 3

$$\text{Flow Rate (STP)} = 300 \text{ cfm} \times \frac{528}{193 + 460} \times \frac{29.92}{29.92} = 240 \text{ cfm}$$

B. Correction for moisture:

Percent Moisture: 50.33%

$$\text{Flow Rate (dry)} = \text{Flow Rate (STP)} \times (100 - \%\text{ moisture})/100$$

$$= 240 \text{ cfm} \times (100 - 50.33)/100$$

$$= 120 \text{ cfm}$$

$$\text{Flow Rate (Saturated at 68°F)} = \text{Flow Rate (dry)} \times 1.025$$

$$= 120 \text{ cfm} \times 1.025$$

$$= 123 \text{ cfm}$$

3. Calculation of Mass Emission Rate

Example: Wash oil decanter, Run 3

Flow Rate (standard conditions) = 123 cfm

Benzene concentration: 693.68 ppm

$$123\frac{\text{ft}^3}{\text{min}} \times 28.32\frac{\text{l}}{\text{ft}^3} \times 60\frac{\text{min}}{\text{hr}} \times \frac{693.68}{10^6} \times 78\frac{\text{g}}{\text{mole}} \times \frac{1 \text{ mole}}{24.15 \text{ l}} \times \frac{1 \text{ lb}}{454 \text{ g}}$$

$$= 1.03 \text{ lb/hr}$$

4. Correcting Benzene Concentration for Benzene Found in Water Trap Catch

Example: Tar dehydrator, east tank, Run 1

Mg benzene in catch: 317 mg

Tedlar bag volume (gas sample): 40.78 1

Measured benzene concentration: 4.11 ppm

A. Mg benzene in collected gas sample:

$$\frac{4.11}{10^6} \text{ x } 40.78 \text{ 1 x } \frac{78 \text{ g}}{\text{mole}} \text{ x } \frac{1 \text{ mole}}{24.15 \text{ 1}} = 0.54 \text{ mg}$$

B. Total mass of benzene (air + liquid)

$$0.54 + 317 = 317.54 \text{ mg}$$

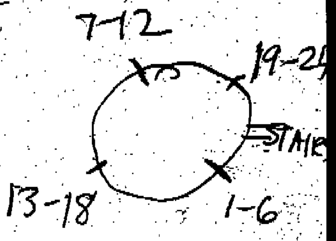
C. Corrected benzene concentration:

$$0.31754 \text{ g x } \frac{1 \text{ mole}}{78 \text{ g}} \text{ x } \frac{24.15 \text{ 1}}{\text{mole}} \text{ x } \frac{10^6}{40.78 \text{ 1}} = 2410.87 \text{ ppm}$$

APPENDIX B

FIELD DATA SHEETS

RUN 1



SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

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PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless Hills

PROCESS: Cooling tower dwfc

PROCESS NOTES:

17' diam

DATE: 9/9/80

AMBIENT TEMPERATURE: 82 °F

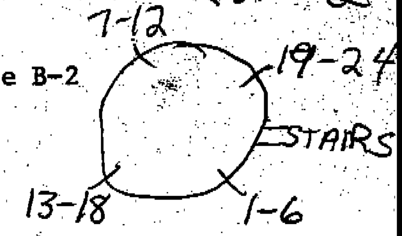
BAROMETRIC PRESSURE: 30.12 "Hg

TEDLAR BAG NUMBER: 7

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
1	0 11:20		250 fpm	
2	2		1250	
3	4		1100	1325
4	6		1800	
5	8		1950	
6	10		1600	
13	12		650 fpm	
14	14		1150	
15	16		1300	1275
16	18		1550	
17	20		1800	
18	22		1200	
12	24		750	
11	26		1050	
10	28		1300	1360
9	30		1850	
8	32		1650	
7	34		1550	
24	36		1200	
23	38		1400	1790
22	40		2100	
21	42		2200	
20	44		1950	
19	46		1900	
	48			

RUN 2

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.



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PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless Hills

PROCESS: Cooling tower dwfc

PROCESS NOTES:

17' diam.

DATE: 9/9/80

AMBIENT TEMPERATURE: 85

BAROMETRIC PRESSURE: 30.12

TEDLAR BAG NUMBER: 11

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
24	0 2:17		1100 fpm	
23	2		1350	
22	4		2200	1790
21	6		2120	
20	8		2020	
19	10		1950	
	~~12~~			
1	12		1400 fpm	
2	14		1350	
3	16		1400	1380
4	18		1550	
5	20		1500	
6	22		1100	
13	24		850	
14	26		1100	
15	28		1250	1120
16	30		1300	
17	32		1350	
18	34		850	
12	36		550	
11	38		600	
10	40		550	670
9	42		650	
8	44		850	
7	46		800	
	48			

RUN 3

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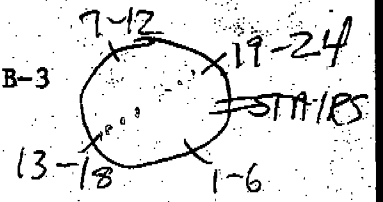


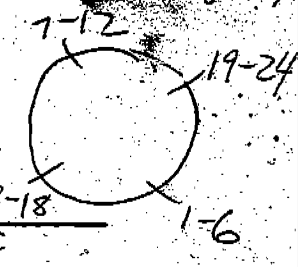
PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless Hills
DATE: 9/9/80
PROCESS: Cooling tower dufc
AMBIENT TEMPERATURE: 87 °F
PROCESS NOTES: 17' diam
very windy - anemometer may be affected
BAROMETRIC PRESSURE: 30.12
TEDLAR BAG NUMBER: 4

	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
12	0 3:47		650 fpm	
11	2		720	
10	4		750	750
9	6		850	
8	8		800	
7	10		750	
24	12		1350 fpm	
23	14		1500	
22	16		2050	1840
21	18		1800	
20	20		2150	
19	22		2200	
1	24		1100	
2	26		1450	
3	28		1600	1550
4	30		1850	
5	32		1550	
6	34		1750	
13	36		850	
14	38		1100	1070
15	40		1100	
16	42		1250	
17	44		1150	
18	46		950	
	48			

RUN 4



SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

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PLANT: US Steel Fairless

PROCESS: Cooling Tower

PROCESS NOTES: 17' diam

DATE: 9/11/80

AMBIENT TEMPERATURE: 80 °F

BAROMETRIC PRESSURE: 29.99

TEDLAR BAG NUMBER: 7

RUN 4

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
24	3:25 0		1250 fpm	
23	2		1850	
22	4		2300	2050
21	6		2350	
20	8		2400	
19	10		2150	
12	12		1200	
11	14		1050	
10	16		1100	1290
9	18		1650	
8	20		1200	
7	22		1550	
13	24		1200	
14	26		1250	
15	28		1600	1700
16	30		2200	
17	32		2100	
18.	34		1850	
1	36		1900	
2	38		2100	
3	40		2050	2170
4	42		2400	
5	44		2300	
6	46		2250	
	48			

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant US Steel Fairless **Process** Cooling tower **Date** 9/9/80

Sample No. Cold well #1 **Time Sampled** 2:10 pm

Sample Type: [✓ Liquid] Air

Sample Temperature 78°F

Ambient Temperature 82°F

Description of Sampling Location:

Sample No. Hot well #1 **Time Sampled** 2:10

Sample Type: [✓ Liquid] Air

Sample Temperature 93°F

Ambient Temperature 82°F

Description of Sampling Location:

Sample No. **Time Sampled**

Sample Type: Liquid Air

Sample Temperature

Ambient Temperature

Description of Sampling Location:

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant US Steel Fairless Process Cooling tower Date 9/9/80

Sample No. cold well #2 Time Sampled 3:35

Sample Type: [✓ Liquid] Air

Sample Temperature 78°F

Ambient Temperature 87 °F

Description of Sampling Location:

Sample No. hotwell #2 Time Sampled 3:35

Sample Type: [✓ Liquid] Air

Sample Temperature 96°F

Ambient Temperature 87°F

Description of Sampling Location:

Sample No. Time Sampled

Sample Type: Liquid Air

Sample Temperature

Ambient Temperature

Description of Sampling Location:

Scott Environmental Technology Inc.

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant US Steel Fairless Hills **Process** Cooling tower **Date** 9/9/80

Sample No. cold well #3 **Time Sampled** 5:15

Sample Type: [✓ Liquid] Air

Sample Temperature 79 °F

Ambient Temperature 82 °F

Description of Sampling Location:

Sample No. hot well #3 **Time Sampled** 5:15

Sample Type: Liquid Air

Sample Temperature 97 °F

Ambient Temperature 82 °F

Description of Sampling Location:

Sample No. **Time Sampled**

Sample Type: Liquid Air

Sample Temperature

Ambient Temperature

Description of Sampling Location:

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922 **METHOD 110 DATA SHEET**

PLANT: USSteel Fairless
PROCESS: Wash oil decanter
PROCESS NOTES:
8" diam.

DATE: 9/10/80
AMBIENT TEMPERATURE: 93 °F - 88 °F
BAROMETRIC PRESSURE: 29.92 TEST 3
TEDLAR BAG NUMBER:

RUN 1 BAG 11

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
[illegible]:10 0	191 °F	820 fpm	
5	194	830 fpm	
10	197	840	
15	192	840	
20	195	870	
25	194	860	
30	188	800	
	RUN 2	BAG 4	
3:08 0	190°F	840 FPM	
5	190	860	
10	194	820	
15	194	920	
20	198	860	
25	194	920	
30	194	840	
	Run 3	BAG	
3:55 0	190°F	860 FPM	
5	190	880	
10	192	870	
15	194	910	
20	195	750	
25	193	890	
30	194	790	

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant USS Fairless Process Wash oil decanter Date 9/16/80

Sample No. Wash oil decanter #1,2,3 Time Sampled 4:40 pm

Sample Type: [✓ Liquid] Air

Sample Temperature 205 °F

Ambient Temperature 87 °F

Description of Sampling Location: dipped from hatchway next to vent.

Sample No. ______ Time Sampled ______

Sample Type: Liquid Air

Sample Temperature ______

Ambient Temperature ______

Description of Sampling Location:

Sample No. ______ Time Sampled ______

Sample Type: Liquid Air

Sample Temperature ______

Ambient Temperature ______

Description of Sampling Location:

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless

PROCESS: Tar dehydrator – ~~east~~ (#1)
west

PROCESS NOTES:

DATE: 9/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 2999

TEDLAR BAG NUMBER:

RUN 1

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOW RATE
0 10:15	174	580 fpm	
15		540 fpm	
20		550 fpm	
25		620	
30		570	

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless

PROCESS: Tar dehydrator A.B.

PROCESS NOTES:
8" ID vent

DATE: 9/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.99

TEDLAR BAG NUMBER:

RUN 2

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
1:50 0			
5	182 °F	2800 fpm	
10		1300	
15		1200	
20		1400	
25		1250	
30		3300	
	RUN 3		
5:30 0	178° F	1250	
5		2040	
10	159 °F	680	
15		900	
20		650	
25		640	
30	160 °F		

[redacted] ENVIRONMENTAL TECHNOLOGY, INC.

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[redacted] 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless

PROCESS: [illegible] dehydrator-west (#2)

PROCESS NOTES: 8" ID

RUN 1

DATE: 9/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.99

TEDLAR BAG NUMBER:

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
0 11:00		290 fpm	
10		300	
15		300	
20		[illegible]	
25			
30			

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: US Steel Fairless

PROCESS: Tardehydrator N.B

PROCESS NOTES: 8" ID vent

DATE: 9/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.99

TEDLAR BAG NUMBER:

RUN 2

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
1:45 0		440 fpm	
15	142 °F	400	
20		430	
25		500	
30		520	
	RUN	3	
0	178	420	
5	[illegible]	840	
10	178	800	
15		800	
20		580	
25			
30	177		

SAMPLE DATA

Plant US Steel Fairless Process Tar dehydrator Date 9/11/80

Sample No. Inlet to 1st tank (1,2,3) Time Sampled 6:05

Sample Type: [✓ Liquid] Air

Sample Temperature 196 °F

Ambient Temperature ______

Description of Sampling Location: from funnel feeding 1st tar dehydr.

Sample No. Tardehyd. #1 (AB) west (1,2,3) Time Sampled 6:20

Sample Type: [✓ Liquid] Air

Sample Temperature 207 °F

Ambient Temperature ______

Description of Sampling Location: dipped from manway

Sample No. Tar dehyd. #2 (NB) East (1,2,3) Time Sampled 6:25

Sample Type: [✓ Liquid] Air

Sample Temperature 219 °F

Ambient Temperature ______

Description of Sampling Location: dipped from manway

APPENDIX C

LABORATORY DATA SHEETS

CHROMATOGRAPHIC ANALYSIS LOG

U.S.S. Fairless Works

Project No. 1922 Date 9-9-80 ? Analyst ____

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
~ 9:30	Col Temp -150°C RT ~ 3 min		ppm/div		Col Temp 150°C all day
	Std 122.5 ppm φ @ 256×10^3	51	2.4/02		Theo. ppm/div @ 16×10^3 from 256×10^3 0.150 ∴ Linear
	Std 6.11 ppm φ @ 16×10^3	40	0.153		
	Cooling Tower Run #1 32×10^3 Bag #7	62.5	0.300	18.77 ppm	
	Std 6.11 ppm φ 16×10^3	39			
	Cooling Tower Run #2 32×10^3 Bag #11	66	0.300	19.80 ppm	
	Std 6.11 ppm φ	38.5			

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CHROMATOGRAPHIC ANALYSIS LOG USS Fairless Bu

Project No. 1422 Date 9-9-80 Analyst

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Std 6.11 ppm ϕ_3 @ 16x10^3	39			
	Cooling Tower Run #3 Bag 4 128x10^3	37	1.20	44.4 ppm	Check Bag #4's history Probably contaminated used at Monessen Stail Stage conc. ≈ 2% ϕ

CHROMATOGRAPHIC ANALYSIS LOG

USS Fairless Ba.

Project No. 1922

Date 9-10-80

Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	6.11 ppm ϕ @ 16×10^3	39.5	0.153		
	100 l Bag Blk. 1×10^3		~0.01		
	Bag #3	< 100			
	Bag #1	5	0.01	.05 ppm	
	Bag #7	< 100			
	Bag A #70	ND			
	Bag B.	1.5	0.01	.015 ppm	
	Bag #3	ND			
	Bag #11	3	0.01	.03	
	Bag #12	3	0.01	.03	
	Bag #3.	4.5	0.01	.045	

Scott Environmental Technology Inc.

CHROMATOGRAPHIC ANALYSIS LOG

USS Fairless Pa

Project No. 1922 Date 9-10-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Bag #4.	6.5	0.01	0.065 ppm	
	6.11 ppm ϕ @ 16x10^3	42.5			
	1.06 ppm iC_4 @ 16x10^3	6.5			
	w/ No BF Toluene	3			
	Repeat 6.11 ppm @ 16x10^3 w/ BF @ - after iC_4	38.5			
	Std 6.11 ppm ϕ / 1.06 ppm iC_4 } @ 8x10^3				
	after 24 hr 3/4 full	iC_4 - 12.25 ϕ - 81 ϕCH_3 - 5			Dup each
	1/2 "	iC_4 - 12.25 ϕ - 78 ϕCH_3 - 5.5			
	1/3 "	iC_4 - 12.5 ϕ - 79.25 ϕCH_3 - 5.75			

CHROMATOGRAPHIC ANALYSIS LOG USS Fairless Pa

Project No. 1922 Date 9-10-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Wash Oil Decanter 256×10^2 Run #1	~~#~~ 29.5	23.92	705.64 ppm	Moisture Collected 15.5 cc H_2O in Trap SampVol = 0.~~1775~~ .501 cf (corrected) = ~~[illegible]~~ 14.19 l NTP
	Std 122.5 ppm ϕ @ 32×10^2	41	2.99 ppm/div	-	
	Wash Oil Decanter Run #2 256×10^2	44	23.92	1052.48	14.5 cc in Trap SampVol 0.264 (corrected) 7.47 l NTP
	Std 122.5 ppm ϕ @ 32×10^2	43			
	Wash Oil Decanter Run #3 256×10^2	29	23.92	693.68	26.5 cc in Trap 1.24 cf (corrected) 35.09 l NTP
	Std 122.5 ppm ϕ @ 32×10^2	43			

CHROMATOGRAPHIC ANALYSIS LOG

USS - Fairless, Pa

Project No. 1922 Date 9-11-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	122.5 ppm φ Std @ 32×10^2	40.5	3.06		
AB	next ~~First~~ Tox Dehydrator Run #1 32×10^2 Bag #7	22.5	3.06	68.91 ppm	Moisture collected ? - 2.5cc -10l ~~NTP~~ Bag Vol. = 21.14l Total @ NTP - 31.14 l
	122.5 ppm φ Std @ 32×10^2	39			
NB	Tox Dehydrator Run #1 1×10^2 Bag A	43	.097	4.11 ppm	Moisture collected - 6.5cc - 8l Bag Vol = 40.78l Total @ NTP = 4826 l
	Std 122.5 ppm φ @ 32×10^2	40			

G 627

CHROMATOGRAPHIC ANALYSIS LOG

Project No.

Date

Analyst

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	N/2 @ 8x10^3	x10			
	ϕC4 Stability				
	Bag 3/4 full	~~82.5~~			
	ϕ @ 8x10^3	83			
	nC4 @ 8x10^3	13			
	ϕ-CH$_3$ 8x10^3	6			
	Std G11-~~[illegible]~~ϕ @ 8x10^3	83			
	1.06 nC4	13			
	ϕ-CH$_3$	6			

Scott Environmental Technology Inc.

CHROMATOGRAPHIC ANALYSIS LOG

USS-Fairless

Project No. 1922 Date 9/11/80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
A/B [illegible]	Tar Dehydrator Run #2 Bag #12 128×10^3	42.5	1.23	52.06 ppm	Bag Vol - 1.149 x 1.059 = 1.503 cf 42.56 l moisture 10cc catch 13.33 l 23.4% moisture Total Vol 56.89 l @ NTP
	122.5 ppm of Std 256×10^3	50	2.45		
A/B exit	Tar Dehydrator Run #2 Bag #1 64×10^3	26.5	0.61	16.23 ppm	Bag Vol - 0.582 x 1.059 = 0.616 cf 17.45 l moisture 3cc catch 4 l = 18.6% moisture
"	122.5 ppm of Std 256×10^3	51.5			Total Vol 21.45 l
	Cooling Tower Run #4 Bag #7 32×10^3	60	0.305	18.30 ppm	
	122.5 ppm of Std	51.5			

CHROMATOGRAPHIC ANALYSIS LOG

USS Fairless Pa

Project No. 1922 Date 9/11/80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
AB	Tar Dehydrator Rm #3 Bag # A 128x10^3 ? Location	24.5	1.23 ~~30.14~~	30.14 ppm	5cc catch = 6.67 l gas Bag Vol = 43.79 50.46 l Total
NB	Tar Dehydrator Rm #3 Bag E 256x10	33	2.46	81.18 ppm	12cc catch = 16 l gas Bag Vol = 20.39 l 36.39 l Total
	Std 122.5 ppm ϕ 256x10^3	49			

APPENDIX D

EPA METHOD 110

(f) All continuous monitoring systems used in accordance with this section are to complete a minimum of one cycle of operation (sampling, analyzing, and data recording) for each successive 15-minute period.

(g) Owners or operators of all continuous monitoring systems installed in accordance with this subpart shall check the zero and span drift at least once daily in accordance with the method prescribed by the manufacturer of such systems unless the manufacturer of such systems recommends adjustments at shorter intervals, in which case such recommendations shall be followed. The daily span check is to be conducted with reference gas containing a concentration of benzene determined to be equivalent to the emission limit for that source based on the emission tests required by § 61.94.

(h) The calibration is to be done with either—

(1) A calibration mixture prepared from the liquids and gases specified in Section 5.2.1 and 5.2.2 of Test Method 110 and in accordance with Section 7.1 of Test Method 110; or

(2) A calibration gas cylinder standard containing the appropriate concentration of benzene. The gas composition of the calibration gas cylinder standard is to have been certified by the manufacturer. The manufacturer must have recommended a maximum shelf life for each cylinder so gas standards will not be used if their concentration has changed greater than ±5 percent from the certified value. The data of gas cylinder preparation, certified benzene concentration, and recommended maximum shelf life must have been affixed to the cylinder before shipment from the manufacturer to the buyer. If a gas chromatograph is used as the continuous monitoring system, these gas mixtures may be used directly to prepare a chromatograph calibration curve as described in Section 7.2 of Test Method 110 for certification of cylinder standards and for establishment and verification of calibration standards.

(i) After receipt and consideration of written application, the Administrator may approve use of an alternative or equivalent continuous monitoring system, alternative monitoring procedures, or alternative monitoring requirements.

(Sec. 114, Clean Air Act as amended (42 U.S.C. 7414))

§ 61.96 Recordkeeping requirements.

(a) The owner or operator of each source to which this subpart applies shall maintain daily records of the monitoring information specified in § 61.95(a).

(b) Recor are to be retained at the source and ide available for inspection the Administrator for a minimum o. years.

(Sec. 114, Cl Air Act as amended (42 U.S.C. 7414))

Appendix B– est Methods

Method 110. termination of Benzene From Stationary S ces

Performance this method should not be attempted persons unfamiliar with the operation gas chromatograph, nor by those who e unfamilar with source sampling, ause knowledge beyond the scope of th presentation is required. Care must be ex cised to prevent exposure of sampling p onnel to benzene, a carcinogen

1. Applicat y and Prinicple

1.1 Appli bility. This method applies to the measure nt of benzene in stack gases from process as specified in the regulations. e method does not remove benzene con ned in particulate matter.

1.2 Princ . An integrated bag sample of stack gas co ining benzene and other organics is s jected to gas chromatographic (GC) analys using a flame ionization detector (FII

2. Range a Sensitivity

The range this method is 0.1 to 70 ppm. The upper li may be extended by extending th alibration range or by diluting the sample.

3. Interfere es

The chrom ograph columns and the correspondi operating parameters herein described n ally provide an adequate resolution o nzene: however, resolution interference ay be encountered on some sources. The ore, the chromatograph operator sh elect the column and operating p neters best suited to his particular a ysis problem, subject to the approval of Administrator. Approval is automatic p ided that the tester produces confirming through an adequate supplementa nalytical technique, such as analysis wit different column or GC/mass spectroscop nd has the data available for review by th Administrator.

4. Apparat

4.1 Samp g (see Figure 110–1). The sampling tra consists of the following components.

4.1.1 Pro . Stainless steel, Pyrex * glass, or Teflon tu g (as stack temperature permits), eq ped with a glass wool plug to remove part late matter.

4.1.2 Sa e Lines. Teflon, 6.4 mm outside diameter, of fficient length to connect probe to ba se a new unused piece for each series bag samples that constitutes an emission tes nd discard upon completion of the test.

4.1.3 Qu Connects. Stainless steel, male (2) an male (2), with ball checks (one pair without) located as shown in figure 110–1.

4.1.4 Tedlar or aluminized Mylar bags, 100 L capacity, to contain sample.

4.1.5 Bag Containers. Rigid leakproof containers for sample bags with covering to protect contents from sunlight.

4.1.6 Needle Valve. To adjust sample flow rate.

4.1.7 Pump. Leak-free with minimum of 2 L/min capacity.

4.1.8 Charcoal Tube. To prevent admission of benzene and other organics to the atmosphere in the vicinity of samplers.

4.1.9 Flow Meter. For observing sample flow rate; capable of measuring a flow range from 0.10 to L/min.

4.1.10 Connecting Tubing. Teflon, 6.4 mm outside diameter, to assemble sampling train (Figure 110–1.)

4.2 Sample Recovery. Teflon tubing, 6.4 mm outside diameter, is required to connect chromatograph sample loop for sample recovery. Use a new unused piece for each series of bag samples that constitutes an emission test and discard upon conclusion of analysis of those bags.

4.3 Analysis. The following equipment is needed:

4.3.1 Gas Chromatograph. With FID, potentiometric strip chart recorder and 1.0 to 2.0 mL sampling loop in automatic sample valve. The chromatographic system shall be capable of producing a response to 0.1ppm benzene that is at least as great as the average noise level. (Response is measured from the average value of the base line to the maximum of the waveform, while standard operating conditions are in use.)

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* Mention c rade names or specific products does not cons te endorsement by the U.S. Environment rotection Agency.

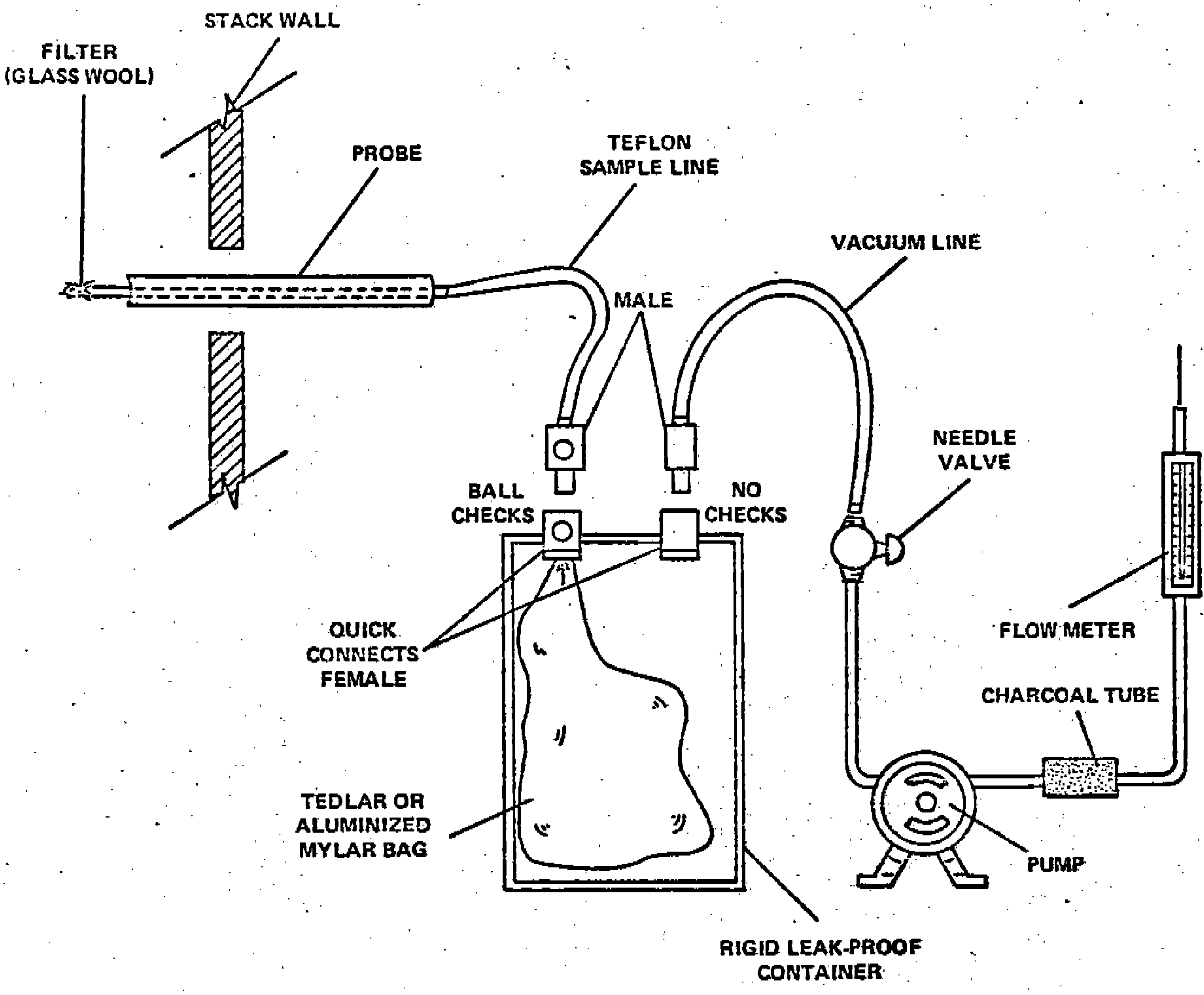

Figure 110-1. Integrated-bag sampling train. (Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.)

BILLING CODE 6560-01-C

4.3.2 Chromatographic Columns. Columns as listed below. The analyst may use other columns provided that the precision and accuracy of the analysis of benzene standards are not impaired and he has available for review information confirming that there is adequate resolution of the benzene peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the benzene peak by an interferent peak. Calculation of area overlap is explained in Appendix E, Supplement A: "Determination of Adequate Chromatographic Peak Resolution.")

4.3.2.1 Column A: Benzene in the Presence of Aliphatics. Stainless steel, 2.44 m by 3.2 mm, containing 10 percent 1,2,3-tris (2-cyanoethoxy) propane (TCEP) on 80/100 Chromosorb P AW.

4.3.2.2 Column B: Benzene With Separation of the Isomers of Xylene. Stainless steel, 1.83 m by 3.2 mm, containing 5 percent SP 1,200/1.75 percent Bentone 34 on 100/120 Suplecoport.

4.3.3 Flow Meters (2). Rotameter type, 100 mL/min capacity.

4.3.4 Gas Regulators. For required gas cylinders.

4.3.5 Thermometer. Accurate to 1° C, to measure temperature of heated sample loop at time of sample injection.

4.3.6 Barometer. Accurate to 5 mmHg, to measure atmospheric pressure around gas chromatograph during sample analysis.

4.3.7 Pump. Leak-free, with minimum of 100 mL/min capacity.

4.3.8 Recorder. Strip chart type, optionally equipped with either disc or electronic integrator.

4.3.9 Planimeter. Optional, in place of disc or electronic integrator, on recorder, to measure chromatograph peak areas.

4.4 Calibration. Sections 4.4.2 through 4.4.5 are for the optional procedure in Section 7.1.

4.4.1 Tubing. Teflon, 6.4 mm outside diameter, separate pieces marked for each calibration consentration.

4.4.2 Tedlar or Aluminized Mylar Bags. 50 L capacity, with valve; separate bag marked for each calibration concentration.

4.4.3 Syringes. 1.0 µL and 10 µL, gas tight, individually calibrated to dispense liquid benzene.

4.4.4 Dry Gas Meter. With Temperature and Pressure Gauges. Accurate to ±2 percent, to meter nitrogen in preparation of standard gas mixtures, calibrated at the flow rate used to prepare standards.

4.4.5 Midget Impinger/Hot Plate Assembly. To vaporize benzene.

5. Reagents

Use only reagents that are of chromatographic grade.

5.1 Analysis. The following are needed for analysis:

5.1.1 Helium or Nitrogen. Zero grade, for chromatograph carrier gas.

5.1.2 Hydrogen. Zero grade.

5.1.3 Oxygen or Air. Zero grade, as required by the detector.

5.2 Calibration. Use one of the following options: either 1.2.1 and 5.2.2, or 5.2.3.

5.2.1 Benzene, 99 Mol Percent Pure. Certified by the manufacturer to contain a minimum of 99 Mol percent benzene; for use in the preparation of standard gas mixtures as described in Section 7.1.

5.2.2 Nitrogen. Zero grade, for preparation of standard gas mixtures as described in Section 7.1.

5.2.3 Cylinder Standards (3). Gas mixture standards (50, 10, and 5 ppm benzene in nitrogen cylinders). The tester may use cylinder standards to directly prepare a chromatograph calibration curve as described in Section 7.2.2, if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ±3 percent or better (see Section 5.2.3.1). (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ±5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified benzene concentration, and recommended maximum shelf life to the cylinder before shipment to the buyer.

5.2.3.1 Cylinder Standards Certification. The manufacturer shall certify the concentration of benzene in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating his analytical procedure on the day of cylinder analysis. To calibrate his analytical procedure, the manufacturer shall use, as a minimum, a three-point calibration curve. It is recommended that the manufacturer maintain (1) a high-concentration calibration standard (between 50 and 100 ppm) to prepare his calibration curve by an appropriate dilution technique; and (2) a low-concentration calibration standard (between 5 and 10 ppm) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration standard exceeds 5 percent of the true concentration, the manufacturer shall determine the source of error and correct it, then repeat the three-point calibration.

5.2.3.2 Verification of Manufacturer's Calibration Standards. Before using, the manufacturer shall verify each calibration standard by (a) comparing it to gas mixtures prepared (with 99 Mol percent benzene) in accordance with the procedure described in Section 7.1 or by (b) having it analyzed by the National Bureau of Standards. The agreement between the initially determined concentration value and the verification concentration value must be within ±5 percent. The manufacturer must reverify all calibration standards on a time interval consistent with the shelf life of the cylinder standards sold.

5.2.4 Audit Cylinder Standards (2). Gas mixture standards with concentrations known only to the person supervising the analysis of samples. The audit cylinder standards shall be identically prepared as those in Section 5.2.3 (benzene in nitrogen cylinders). The concentrations of the audit cylinder should be: one low-concentration cylinder in the range of 5 to 20 ppm benzene and one high-concentration cylinder in the range of 100 to 300 ppm benzene. When available, the tester may obtain audit cylinders by contacting: U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Quality Assurance Branch (MD -77), research Triangle Park, North Carolina 27711. If audit cylinders are not available at the Environmental Protection Agency, the tester must secure an alternative source.

6. Procedure

6.1 Sampling. Assemble the sample train as shown in Figure 110-1. Perform a bag leak check according to Section 7.3.2. Join the quick connects as illustrated, and determine that all connections between the bag and the probe are tight. Place the end of the probe at the centroid of the stack, and start the pump with the needle valve adjusted to yield a flow that will more than half fill the bag in the specified sample period. After allowing sufficient time to purge the line several times, connect the vacuum line to the bag and evacuate the bag until the rotameter indicates no flow. At all times, direct the gas exiting the rotameter away from sampling personnel. At the end of the sample period, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container. Protect the bag container from sunlight.

6.2 Sample Storage. Keep the sample bags out of direct sunlight. Perform the analysis within 4 days of sample collection.

6.3 Sample Recovery. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the gas chromatograph sample valve. Switch the valve to receive gas from the bag through the sample loop. Arrange the equipment so the sample gas passes from the sample valve to a 100-mL/min rotameter with flow control valve followed by a charcoal tube and a 1-in. pressure gauge. The tester may maintain the sample flow either by a vacuum pump or container pressurization if the collection bag remains in the rigid container. After sample loop purging is ceased, always allow the pressure gauge to return to zero before activating the gas sampling valve.

6.4 Analysis. Set the column temperature to 80° C (176° F) for column A or 75° C (167° F) for column B, and the detector temperature to 225° C (437° F). When optimum hydrogen and oxygen flow rates have been determined, verify and maintain these flow rates during all chromatograph operations. Using zero helium or nitrogen as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of approximately 20 mL/min should produce adequate separations. Observe the base line periodically and determine that the noise level has stabilized and that base-line drift has ceased. Purge the sample loop for 30 sec at the rate of 100 mL/min, then activate the sample valve. Record the injection time (the position of the pen on the chart at the time of sample injection), the sample number, the sample loop temperature, the column temperature, carrier gas flow rate, chart speed, and the attenuator setting. From the chart, note the peak having the retention time corresponding to benzene, as determined in Section 7.2.1. Measure the benzene peak area, A_m, by use of a disc integrator, electronic integrator, or a planimeter. Record A_m and the retention time. Repeat the injection at least two times or until two consecutive values for the total area of the benzene peak do not vary more than 5 percent. Use the average value of these two total areas to compute the bag concentration.

6.5 Determination of Bag Water Vapor Content. Measure the ambient temperature and barometric pressure near the bag. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure. (Assume the relative humidity to be 100 percent unless a lesser value is known.)

7. *Preparation of Standard Gas Mixtures, Calibration, and Quality Assurance*

7.1 Preparation of Benzene Standard Gas Mixtures. (Optional procedure—delete if cylinder standards are used.) Assemble the apparatus shown in Figure 110-2. Evacuate a 50-L Tedlar or aluminized Mylar bag that has passed a leak check (described in Section 7.3.2) and meter in about 50 L of nitrogen. Measure the barometric pressure, the relative pressure at the dry gas meter, and the temperature at the dry gas meter. While the bag is filling, use the 10μL syringe to inject 10μL of 99+ percent benzene through the septum on top of the impinger. This gives a concentration of approximately 50 ppm of benzene. In a like manner, use the other syringe to prepare dilutions having approximately 10 ppm and 5 ppm benzene concentrations. To calculate the specific concentrations, refer to Section 8.1. These gas mixture standards may be used for 7 days from the date of preparation, after which time preparation of new gas mixtures is required. (Caution: If the new gas mixture standard is a lower concentration than the previous gas mixture standard, contamination may be a problem when a bag is reused.)

7.2 Calibration.

7.2.1 Determination of Benzene Retention Time. (This section can be performed simultaneously with Section 7.2.2.) Establish chromatograph conditions identical with those in Section 6.4, above. Determine proper attenuator position. Flush the sampling loop with zero helium or nitrogen and activate the sample valve. Record the injection time, the sample loop temperature, the column temperature, the carrier gas flow rate, the chart speed, and the attenuator setting. Record peaks and detector responses that occur in the absence of benzene. Maintain conditions, with the equipment plumbing arranged identically to Section 6.3, and flush the sample loop for 30 sec at the rate of 100 mL/min with one of the benzene calibration mixtures. Then activate the sample valve. Record the injection time. Select the peak that corresponds to benzene. Measure the distance on the chart from the injection time to the time at which the peak maximum occurs. This distance divided by the chart speed is defined as the benzene peak retention time. Since it is quite likely that there will be other organics present in the sample, it is very important that positive identification of the benzene peak be made.

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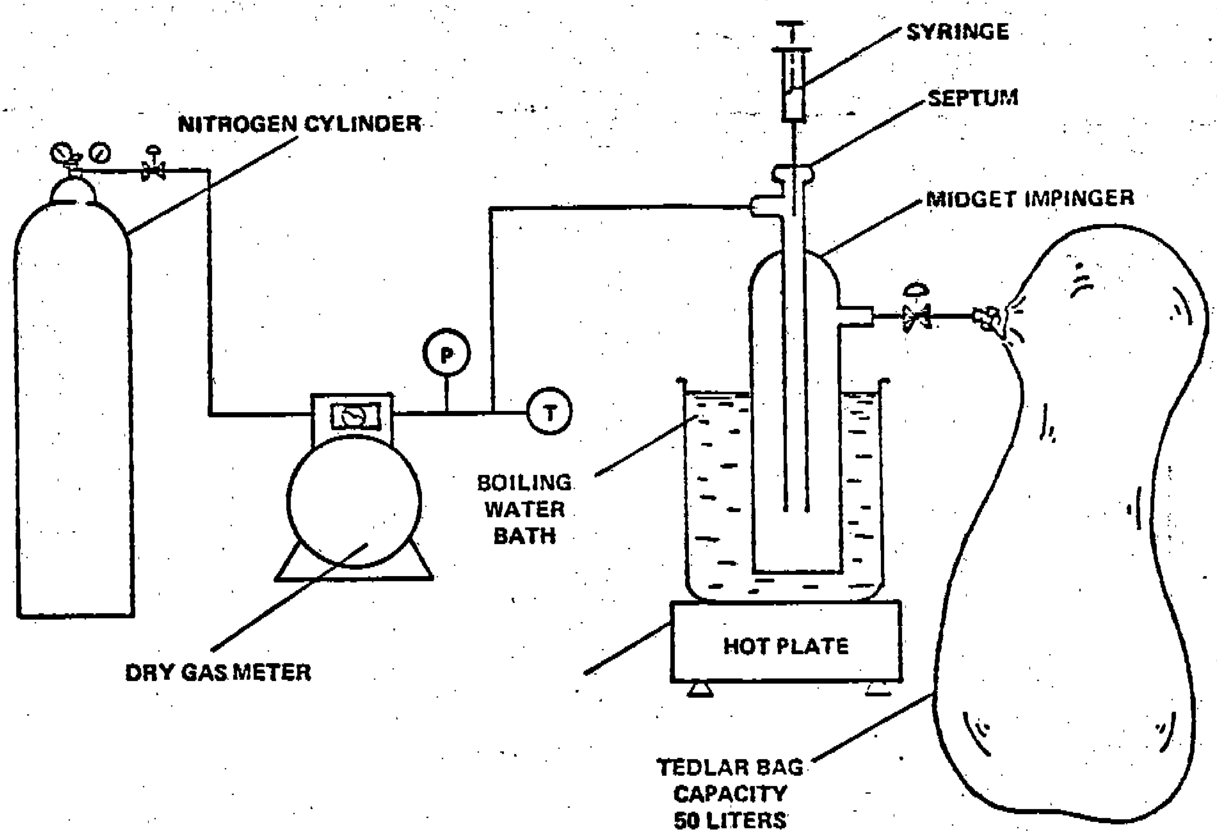

Figure 110-2. Preparation of benzene standards (optional).

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Question.

7.2.2 Preparation of Chromatograph Calibration Curve. Make a gas chromatographic measurement of each standard gas mixture (described in Section 5.2.3 or 7.1.1) using conditions identical with those listed in Sections 6.3 and 6.4. Flush the sampling loop for 30 sec at the rate of mL/min with one of the standard gas mixtures and activate the sampe valve. Record C_c, the concentration of benzene injected, the attenuator setting, chart speed, peak area, sample loop temperature, column temperature, carrier gas flow rate, and retention time. Record the laboratory pressure. Calculate A_c, the peak area multiplied by the attenuator setting. Repeat until two consecutive injection areas are within 5 percent, then plot the average of those two values versus C_c. When the other standard gas mixtures have been similarly analyzed and plotted, draw a straight line through the points derived by the least squares method. Perform calibration daily, or before and after each set of bag samples, whichever is more frequent.

7.3 Quality Assurance.

7.3.1 Analysis Audit. Immediately after the preparation of the calibration curve and before the sample analyses, perform the analysis audit described in Appendix E, Supplement B: "Procedure for Field Auditing GC Analysis."

7.3.2 Bag Leak Checks. While performance of this section is required after bag use, it is also advised that it be performed before bag use. After each use, make sure a bag did not develop leaks by connecting a water manometer and pressurizing the bag to 5 to 10 cm H_2O (2 to 4 in. H_2O). Allow to stand for 10 min. Any displacement in the water manometer indicates a leak. Also, check the rigid container for leaks in this manner. (Note: an alternative leak check method is to pressurize the bag to 5 to 10 cm H_2O or 2 to 4 in. H_2O and allow to stand overnight. A deflated bag indicates a leak.) For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. Failure of the rotameter to register zero flow when the bag appears to be empty indicates a leak.

8. Calculations

8.1 Optional Benzene Standards Concentrations. Calculate each benzene standard concentration (C_c in ppm) prepared in accordance with Section 7.1 as follows:

$$C_c = \frac{B(0.2706)(10^3)}{V_m \; Y \; \frac{293}{T_m} \; \frac{P_m}{760}}$$

$$C_c = 701.9 \frac{B T_m}{V_m \, Y \, P_m} \qquad (110\text{-}1)$$

where:

B = Volume of benzene injected, microliters.

V_m = Gas volume measured by dry gas meter, liters.

Y = Dry gas meter calibration factor, dimensionless.

P_m = Absolute pressure of the dry gas meter, mmHg.

T_m = Absolute temperature of the dry gas meter, °K.

0.2706 = Ideal gas volume of benzene at 293° K and 760 mmHg L/mL.

10^3 = Conversion factor [(ppm)(mL)/μL].

8.2 Benzene Sample Concentrations. From the calibration curve described in Section 7.2.2 above, select the value of C_c that corresponds to A_c. Calculate the concentration of benzene in the sample (C_s in ppm) as follows:

$$C_s = \frac{C_c P_r T_i}{P_i T_r (1-S_{wb})}, \qquad (110\text{-}2)$$

where:

C_s = Concentration of benzene in the sample, ppm.

C_c = Concentration of benzene indicated by the gas chromatograph, ppm.

P_r = Reference pressure, the barometric pressure recorded during calibration, mmHg.

T_i = Sample loop temperature at the time of analysis, °K.

P_i = Barometric pressure at time of analysis, mmHg.

Tr = Reference temperature, the sample loop temperature recorded during calibration, °K.

S_{wb} = Water vapor content of the bag sample, volume fraction.

9. References

1. Feairheller, W. R., A. M. Kemmer, B. J. Warner, and D. Q. Douglas. Measurement of Gaseous Organic Compound Emissions by Gas Chromatography. U.S. Environemental Protection Agency. EPA Contract Number 68-02-1404. January 1978. Revised by EPA August 1978.
2. Knoll, Joseph E., Wade H. Penny, and Rodney M. Midgett. The Use of Tedlar Bags to Contain Gaseous Benzene Samples at Source Level. U.S. Environmental Protection Agency. Research Triangle Park, N.C. Monitoring Series. EPA-600/4-78-057. October 1978.
3. Supelco, Inc. Separation of Hydrocarbons. Bellefonte, Pa. Bulletins 743A, 740C, and 740D. 1974.
4. Current Peaks. *10*:1. Carle Instruments, Inc. Fullerton, Calif. 1977.
5. Knoll, Joseph E. Communications Concerning Chromatographic Columns for Benzene Analysis. October 18, 1977.
6. Knoll, Joseph E. Communications Concerning Gas Chromatographic Columns for Separating Benzene From Other Organics in Cumene and Maleic Anhydride Process Effluents. November 10, 1977.

Appendix C

Supplement A—Determination of Adequate Chromatographic Peak Resolution

In this method of dealing with resoultion, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from -2σ to $+2\sigma$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two peaks are separated by a known distance, b, one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compound overlaps the curve of a compound that is under analysis is found by integrating the contaminant curve over limits $b-2\sigma_s$ to $b+2\sigma_s$, where σ_s is the standard deviation of the sample curve.

There are several ways this calculation can be simplified. Overlap can be determined for curves of unit area; then actual areas can be introduced. The desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calcualtion programs and tables. An example would be Program 15 in Texas Instruments Program Manual ST1, 1975, Texas Instruments, Inc., Dallas, Texas 75222.

$$\frac{1}{\sqrt{2\pi}\,\sigma c}\int_{b-2\sigma_s}^{b+2\sigma_s} e^{-\frac{t_c^2}{2\sigma_c}}\,dt = \frac{1}{\sqrt{2\pi}}\int_{\frac{b-2\sigma_s}{\sigma_c}}^{\infty} e^{-\frac{x^2}{2}}\,dx - \frac{1}{\sqrt{2\pi}}\int_{\frac{b+2\sigma_s}{\sigma_c}}^{\infty} e^{-\frac{x^2}{2}}\,dx.$$

The following calculation steps are required:*

1. $2\sigma_s = t_s/\sqrt{2 \ln 2}$
2. $\sigma_c = t_c/2\sqrt{2 \ln 2}$
3. $x_1 = (b-2\sigma_s)/\sigma_c$
4. $x_2 = (b+2\sigma_s)/\sigma_c$
5. $Q(x_1) = \frac{1}{\sqrt{2\pi}}\int_{x_1}^{\infty} e^{-\frac{x^2}{2}}\,dx$
6. $Q(x_2) = \frac{1}{\sqrt{2\pi}}\int_{x_2}^{\infty} e^{-\frac{x^2}{2}}\,dx$
7. $I_o = Q(x_1) - Q(x_2)$
8. $A_o = I_oA_c/A_s$
9. Percentage overlap = $A_o \times 100$.

where

A_s = The area of the sample peak of interest determined by electronic integration, or by the formula $A_s = h_st_s$.

A_c = The area of the contaminant peak, determined in the same manner as A_s.

b = The distance on the chromatographic chart that separates the maxima of the two peaks.

H_s = The peak height of the sample compound of interest, measured from the average value of the baseline to the maximum of the curve.

t_s = The width of the sample peak of interest at 1/2 of peak height.

t_c = The width of the contaminant peak at 1/2 of peak height.

σ_s = The standard deviation of the sample compound of interest elution curve.

σ_c = The standard deviation of the contaminant elution curve.

$Q(x_1)$ = The integral of the normal distribution function from x_1 to infinity.

$Q(x_2)$ = The integral of the normal distribution function from x_2 to infinity.

I_o = The overlap integral.

A_o = The area overlap fraction.

In judging the suitability of alternate gas chromatographic columns, or the effects of altering chromatographic conditions, one can employ the area overlap as the resolution parameter with a specific maximum permissible value.

The use of Gaussian functions to describe chromatographic elution curves is widespread. However, some elution curves are highly asymmetric. In those cases where the sample peak is followed by a contaiminant that has a leading edge that rises sharply but the curve then tails off, it may be possible to define an effective width for t_c as "twice the distance from the leading edge to a perpendicular line through the maxim of the contaminant curve, measured along a perpendicular bisection of that line."

Supplement B—Procedure for Field Auditing GC Analysis

Responsibilities of audit supervisor and analyst at the source sampling site include the following:

A. Check that audit cylinders are stored in a safe location both before and after the audit to prevent vandalism of same.

B. At the beginning and conclusion of the audit, record each cylinder number and cylinder pressure. Never analyze an audit cylinder when the pressure drops below 200 psi.

C. During the audit, the analyst is to perform a minimum of two consecutive analyses of each audit cylinder gas. The audit must be conducted to coincide with the analysis of source test samples. Normally, it will be conducted immediately after the GC calibration and prior to the sample analyses.

D. At the end of audit analyses, the audit supervisor requests the calculated concentrations from the analyst and then compares the results with the actual audit concentrations. If each measured concentration agrees with the respective actual concentration within ±10 percent, he then directs the analyst to begin the analysis of source samples. Audit supervisor judgment and/or supervisory policy determine course of action with agreement is not within ±10 percent. Where a consistent bias in excess of 10 percent is found, it may be possible to proceed with the sample analyses, with a corrective factor to be applied to the results at a later time. However, every attempt should be made to locate the cause of the discrepancy, as it may be misleading. The audit supervisor is to record each cylinder number, cylinder pressure (at the end of the audit), and all calculated concentrations. The individual being audited must not under any circumstance be told the actual audit concentrations until the calculated concentrations have been submitted to the audit supervisor.

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*In most instances, $Q(x_2)$ is very small and may be neglected.

APPENDIX E

PROJECT PARTICIPANTS

APPENDIX E

PROJECT PARTICIPANTS

The following people participated in some phase of the sampling program at United States Steel - Fairless Works:

From Scott Environmental Technology, Inc.:

Tom Bernstiel, Chemist

Jack Carney, Chemist

P. K. Chattopadhyay, Chemist

Dan FitzGerald, Manager of Eastern Operations

Kevin Gordon, Technician

Carolyn Graham, Chemical Engineer

Joe Marino, Technician

Lou Reckner, Vice President & General Manager

From Research Triangle Institute:

Peter Mehta

From U. S. Environmental Protection Agency

Dan Bivins

Gail Lacy

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
Office of Air Quality Planning and Standards
Research Triangle Park, North Carolina 27711

DATE: 4/21/81

SUBJECT: Source Test Report

FROM: J. E. McCarley, Chief, Field Testing Section,
Emission Measurement Branch, ESED (MD-13)

TO: See Below

The enclosed final source test report is submitted for your information. Any questions regarding the test should be directed to the Project Officer (telephone: 8/629-5543). Additional copies of this report are available from the ERC Library, Research Triangle Park, North Carolina 27711.

Industry: Benzene

Process: Coke Oven By-Product Plants

Company: U. S. Steel

Location: Fairless Hills, PA

Project Report Number: 80-BYC-8

Project Officer: Dan Bivins

Enclosure

Addressees:
Ken Knapp, ESRL (MD-46)
Arch MacQueen, MDAD (MD-14)
Rodney Midgett, EMSL (MD-77)
Mark S. Siegler, DSSE (MD-EN-341)
Director, Air & Hazardous Materials Division, Region III
(copy enclosed for State agency)
Bob Kilgore, EPA Library Services (MD-35)