SET 1957 05 1280

Note: This is a reference cited in *AP 42, Compilation of Air Pollutant Emission Factors, Volume I Stationary Point and Area Sources.* AP42 is located on the EPA web site at www.epa.gov/ttn/chief/ap42/

The file name refers to the reference number, the AP42 chapter and section. The file name "ref02_c01s02.pdf" would mean the reference is from AP42 chapter 1 section 2. The reference may be from a previous version of the section and no longer cited. The primary source should always be checked.

BENZENE SAMPLING PROGRAM AT COKE BY-PRODUCT RECOVERY PLANTS: BETHLEHEM STEEL CORPORATION BETHLEHEM, PENNSYLVANIA

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Work Assignment 48
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1.0 INTRODUCTION

Scott Environmental Services, a division of Scott Environmental Technology, Inc. conducted a testing program at Bethlehem Steel Corporation, Bethlehem, Pennsylvania to determine benzene emissions from six sources in the coke byproduct recovery plant. The work was performed for the United States Environmental Protection Agency, Emissions Measurement Branch, under Contract No. 68-02-2813, Work Assignment 48. Data collected from this plant and six others are being used for the development of a possible National Emission Standard for Hazardous Air Pollutants for benzene.

Sampling was conducted at Bethlehem Steel from July 7th to 24th, 1980. Integrated air samples and liquid samples for benzene analysis were collected from the following processes: Denver flotation unit, naphthalene melt pit, naphthalene drying tank, cooling tower - direct water final cooler, light oil condenser vent, and the tar decanter from #5 battery.

2.0 SUMMARY OF RESULTS

Process	Benzene Emission Rate lb/hr.	Benzene Emission Rate kg/hr.
Cooling Tower	73.4	33.3
Tar Decanter	2.6	1.2
Light Oil Condenser Vent	28.8	13.1
Naphthalene Drying Tank	0.04*	0.02*
Denver Float Units	28.2	12.8
Naphthalene Melt Pit	19.8*	9.0*

*Not a continuous process.

3.0 RESULTS AND DISCUSSIONS

3.1 COOLING TOWER

The cooling tower circulates the hot water from the direct water final coolers after the naphthalene is removed via the Denver float units. The tower is about 30 feet high and has four 13-foot diameter fans on top for pulling air countercurrent to the falling water. Benzene which is contained in the final cooler water is in part released as a vapor as it passes downward through the cooling tower. This benzene is picked up as a contaminant in the final cooler spray towers.

The three tests run on the cooling tower were fairly consistent, ranging from 66 to 79 lb/hr., with an average result of 73.4 lb/hr. The stack velocities for each run reported in Table 3-1 are an average of the velocities measured across the 24-point traverse. The velocities measured were generally lower near the stack wall and in the center over the hub of the fan, as would be expected. Field data (showing the measured velocities) can be found in Appendix B.

All stack flow rates 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 inches Hg (2 1/2 % moisture). Example calculations are shown in Appendix A.

Liquid samples were collected from the hot and cold wells. Average benzene concentrations were 6.8 ppm and 3.5 ppm respectively. The hot and cold well temperatures were 86°F and 82°F, indicating that the cooling tower was not really cooling the water significantly, and as noted on page 6-1,

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TABLE 3-1

COOLING TOWER DATA SUMMARY

Process: Cooling Tower-direct water final cooler

Plant: Bethlehem Steel, Bethlehem, PA

Stack Diameter: 13 feet (1 of 4 stacks)

Stack Area: 133 $ft.^2$

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) 1 Fan	Benzene Emission Rate (lb/hr) 4 Fans
1	7/10/80	1034-1140	84	29.58	870	115,000	109,000	12.56	16.6	66.2
2	7/10/80	1150-1255	84	29.58	905	120,000	114,000	14.38	19.9	79.5
3	7/10/80	1417-1520	83	29.56	860	114,000	108,000	14.27	18.6	74.5
										Ave. 73.4

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

LIQUID SAMPLE DATA SUMMARY

Sample Location	Date	Time	Sample Temp. (°F)	Benzene Concentration (ppm by weight)
Hot Well	7/10/80	15:40	86°F	7.1) 6.4) Average 6.8
Cold Well	7/10/80	15:45	82°F	3.2) 3.8) Average 3.5

was due to the malfunction of a faulty level control. Past plant operating experience shows that an average temperature reduction from 86°F to 76°F is experienced during the summer months and from 62°F to 48°F during the winter months.

3.2 TAR DECANTER

The tar decanter collects tar and flushing liquor from the #5 battery and from the primary coolers. It is allowed to settle and the flushing liquor is decanted off the top while the tar is drained from the bottom. The decanter is vented to the atmosphere, and is a potential benzene emission source.

The average result for the tar decanter emissions is 2.6 lb/hr., with a range of 1.4 to 3.7 lb/hr. Velocities measured were quite consistent over all three runs but the concentration of benzene differed considerably, as shown in Table 3-2. The large differences between sample runs is probably due to fluctuations or changes in the process feed streams, as the samples were not all collected on the same day.

Liquid samples were collected at three locations: The surface liquid in the decanter, the inlet to the decanter from the coke gas cross-over main from the #5 battery, and the inlet to the decanter from the primary cooler. Average benzene concentrations in the liquid samples were: In the surface samples - 1.6 ppm, in the crossover main samples - 4.9 ppm, and in the primary cooler samples - 16.4 ppm in the light fraction (flushing liquor) and 1810 ppm in the heavy fraction (tar).

TABLE 3-2

TAR DECANTER DATA SUMMARY

Process: Tar Decanter - #5 battery

Plant: Bethlehem Steel, Bethlehem, PA

Stack Diameter: 10-1/8"

Stack Area: 0.559 $ft.^2$

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 Concen-tration (ppm)	Benzene Emission Rate (lb/hr)
1	7/8/80	1453-1523	158	29.53	500	280	170	1447.2	2.9
2	7/9/80	1020-1121	161	29.71	490	275	160	717.8	1.4
3	7/9/80	1530-1600	163	29.67	490	280	150	1975.0	3.6
									Ave. 2.6

Standard Conditions: 68°F, 29.92 inches Hg.

LIQUID SAMPLE DATA

Sample Location	Date	Time	Sample Temp. (°F)	Benzene Conc. (ppm by Weight)
Flushing liquor on surface	7/8/80	1525	176	5.6
	7/8/80	1600	180	4.2 Ave. 4.9
Flushing liquor inlet from coke gas crossover main	7/9/80	1415	N.A.	1.6
Inlet to decanter from primary cooler Heavy fraction (tar)	7/9/80	1545	140	1736 1888 Ave. 1810
Light fraction (liquor)	7/9/80	1545	140	16.4 16.3 Ave. 16.4

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3.3 LIGHT OIL CONDENSER VENT

Benzene in the wash oil is removed by heating the wash oil and condensing out the benzene. Noncondensibles in the wash oil, possibly including some benzene, are vented to the atmosphere. For this reason the light oil condenser vent was considered a potential benzene emission source.

The average of the three good runs on the light oil condenser vent (Run 3 was voided) was 28.8 lb/hr., as shown in Table 3-3. Although the flow rate was very low, the benzene concentration was approximately 10% so the mass emission rates were comparable to higher flow sources.

TABLE 3-3

LIGHT OIL CONDENSER VENT DATA SUMMARY

Process: Light Oil Condenser Vent

Plant: Bethlehem Steel, Bethlehem, PA

Stack Diameter: 6"

Stack Area: 0.20 $ft.^2$

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 Concen-tration (ppm)	Benzene Emission Rate (lb/hr.)
1	7/11/80	1015-1045	95	29.51	120	24	23	91,900	25.3
2	7/11/80	1056-1126	96	29.51	130	25	23	109,800	31.1
3	7/11/80	1212-1242	109	29.51	120	23	21	53,300	*
4	7/11/80	1600-1630	104	29.49	120	24	22	110,500	29.9
									Ave. 28.8

*Run 3 not included in calculations due to sampling system leak.

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

NOTE: No liquid samples were taken at this source.

3.4 NAPHTHALENE DRYING TANK

The drying tank collects the melted naphthalene after the melt process, and steam is applied to the tank to drive off any water present in the naphthalene. This is a batch process and runs for approximately 12 to 14 hours, during which time benzene is emitted along with the steam, through the open process vents on the tank.

Testing the naphthalene drying tank involved a special test modification using impingers which is described in detail in Section 6.4. The test method was given a trial run on July 18, and the resulting total emission rate (stack plus vent) was 1.57 lb/hr. A series of 8 tests were run on July 22 over a 15-hour period, and the average for these runs was 0.04 lb/hr. The results of the 8 tests varied widely since the drying cycle is a batch process. As expected, the emissions dropped off as the cycle progressed and the water was driven off the naphthalene, and emissions increased when the tank temperature increased, as seen in Table 3-4. Vent "A" refers to the process vent stack, and "B" is the large opening in the tank for steam lines, which was tested as a vent.

The benzene emissions from the drying tank vary widely from day to day depending on how long the naphthalene was heated in the melt pit prior to transferral to the drying tank. Ideally the melt process and the drying tank should be sampled on the same day to determine benzene emissions from the naphthalene handling processes as a whole.

TABLE 3-4

NAPHTHALENE DRYING TANK DATA SUMMARY

Process: Naphthalene Drying Tank

Plant: Bethlehem Steel, Bethlehem, PA

Stack Diameter: A: 6" B: 22"

Stack Area: A: 0.196 ft^2 B: 1.6 $ft.^2$

Barometric Pressure: 29.5 in. Hg.

Run No.	Date	Test Period	Stack Temp. (°F)	Stack Velocity (fpm) A	Stack Velocity (fpm) B	Stack Flowrate (ACFM) A	Stack Flowrate (ACFM) B	Stack Flowrate (ACFM) Total	Standard Flowrate (SCFM) Total	Benzene Concentration (ppm)	Benzene Emission Rate (lb/hr.)
T	7/18/80	1353-1423	206	630	350	120	560	680	71	1824	1.57
1	7/22/80	1337-1410	209	850	610	170	980	1150	106	168.60	0.217
2	7/22/80	1610-1640	210	730	110	140	170	310	3	428.11	0.016
3	7/22/80	1813-1843	202	690	*	140	*	140	18	32.34	0.007
4	7/22/80	2130-2200	195	590	*	110	*	110	27	16.09	0.005
5	7/23/80	2415-2445	192	540	*	110	*	110	27	40.78	0.013
6	7/23/80	0220-0300	190	590	*	120	*	120	34	45.40	0.019
7	7/23/80	0255-0320	210	780	100	150	250	400	14	218.16	0.037
8	7/23/80	0404-0455	199	660	*	130	*	130	22	117.05	0.032
											Ave. 0.043

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

NOTE: No liquid samples were collected at this source.

* No flow detected with anemometer.

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3.5 DENVER FLOTATION UNITS

The Denver units skim naphthalene from the surface of the hot water collected from the final coolers. The skimming is accomplished by blades rotating on a shaft that spans the length of the flotation tank. The system is comprised of four adjacent units, three of which are in operation at any given time. This is a constant operation and constitutes a potential benzene emission source because the impure naphthalene is contaminated with benzene and the Denver units are agitated and at temperatures above ambient level.

The results of the tests of the Denver float units are presented in Table 3-5. The tracer gas sampling strategy and sampler locations for each test are detailed in Section 6.5. Each test consisted of two runs, with the second run designed to estimate the contribution of the unit adjacent to the test unit (#2) to the total being measured from the test unit (#1). This became irrelevant in tests 2 and 3 because unit 2 was not in operation.

In tests 2 and 3 the data from sampler 3 was rejected because the sampler was inadvertently located adjacent to a "hot spot" benzene emission point in the naphthalene melt pit.

In each test the benzene/isobutane ratio is lower for the center sampler than the outer samplers. This would be expected because the tracer discharge manifold was not long enough to cover the entire tank axis. Thus, the center portion of the tank shows a higher relative isobutane concentration.

TABLE 3-5

DENVER FLOAT UNITS

Test 1, Run 1
Tracer on Tank #1
Isobutane release rate - 1.39 lb/hr

Denver Units Operating: 1, 2, 3
Date: 7/8/80
Test Start: 11:24

Sample Loc.	Conc. of Benzene (ppm)	Conc. of Isobutane (ppm)	Mass to Mass Ratio ϕ/ic_4	F*	lb/hr Benzene	kg/hr Benzene
1	3.51	0.50	9.38	0.739	9.60	4.36
2	8.61	1.72	6.73	0.789	7.35	3.34
3	14.51	2.02	9.66	0.665	8.91	4.05
				Avg.	8.62	Avg. 3.92

Test 1, Run 2
Tracer on Tank #2
Isobutane release rate - 1.25 lb/hr

Date: 7/8/80
Test Start: 12:09

1	3.03	0.15
2	7.69	0.40
3	16.61	1.13

Test 2, Run 1
Tracer on Tank #1
Isobutane release rate - 1.27 lb/hr

Denver Units Operating: 1, 3, 4
Date: 7/15/80
Test Start: 10:30

1	5.16	1.02	6.80	1.00	8.64	3.93
2	5.42	1.09	6.68	1.00	8.48	3.85
3	18.96	1.16	21.94**	1.00	27.86**	12.66**
				Avg.	8.56	Avg. 3.89

Test 2, Run 2
Tracer on Tank #2
Isobutane release rate - 1.25

Date: 7/15/80
Test Start: 11:19

1	4.42	0.20
2	5.68	0.48
3	18.45	2.40

---

* Fraction from Tank #1.

** Data rejected, interference from another source.

Table 3-5
(Continued)

Test 3, Run 1
Tracer on Tank #2
Isobutane release rate - 1.28 lb/hr

Denver Units Operating: 1, 3, 4
Date: 7/15/80
Test Start: 13:30

Sample Loc.	Conc. of Benzene (ppm)	Conc. of Isobutane (ppm)	Mass to Mass Ratio ϕ/ic_4	F*	lb/hr Benzene	kg/hr Benzene
1	7.13	0.22				
2	8.39	0.50				
3	14.00	1.97				

Test 3, Run 2
Tracer on Tank 1
Isobutane release rate 1.28 lb/hr

Date: 7/15/80
Test Start: 14:00

1	6.64	0.95	9.45	1.00	12.10	5.5
2	7.16	1.26	7.66	1.00	9.80	4.45
3	13.67	1.24	14.83*	1.00	18.98**	8.63**
				Avg.	10.95	Avg. 4.98

* Fraction from Tank #1.
** Data rejected, interference from another source.

The benzene emission rates for a typical Denver Float Unit tank were determined to be 8.6, 8.6 and 11.0 pounds per hour. While statistical determination of confidence limits is not possible, the relative good agreement of data points and the small estimated error due to the assumptions made in the calculations lead to the judgment that the emission rates are within one pound per hour of the true rate at each process/ambient condition tested. The total emissions from the Denver unit with three tanks in operation would be 26, 26 and 33 pounds per hour.

Test 3 was performed on the same day as Test 2 and under the same experimental conditions except that the ambient temperature was approximately $5^{o}F$ higher in Test 3. A comparison of corresponding Test 2 and Test 3 data (2-1 to 3-2 and 2-2 to 3-1) shows that the isobutane tracer concentration changed very little from test to test. Yet, the benzene is clearly higher at Sampling Locations 1 and 2 in each case. This indicates that the higher emission rates in Test 3 can be attributable to the higher ambient temperature.

3.6 NAPHTHALENE MELT PIT

The melt pit collects the naphthalene slurry that was skimmed off in the Denver units, and once a day steam is applied to melt the naphthalene to facilitate pumping into a drainage tank. Benzene contained in the naphthalene cake is released when the steam is applied to the melt pit.

The results of the four tracer gas tests on the naphthalene melt pit during melt operations are shown in Table 3-6. For each test the first half-hour run was conducted while the cake was still melting. The second run was made after ammonium sulfate salt had been added to the melt and prior to its being pumped to the drying tank. There are considerable test to test differences in benzene emission rates. It is believed that the differences are real, and that they are the result of variations in the process step timing, the portion of the process cycle sampled and ambient conditions.

A test was performed on 7/17/80 after the melt was completed and the pit was beginning to refill. The results of this test are presented in Table 3-7. This test serves as the basis for estimates of emissions from the pit at times other than when the melt was in progress. This test was planned to assess the contribution of the Denver float unit to the melt pit emissions measured during the melt cycle. However, it became apparent that the melt pit made a substantial contribution to the benzene found in this test. On 7/22/80, three sets of grab air samples were collected over the melt pit at ground level. The results are as follows.

TABLE 3-6

NAPHTHALENE MELT PIT

Date: 7/15/80
Tracer Test #1, Run #1
Test Start - 8:00 a.m.

Isobutane Emission Rate: 1.16 lb/hr
0.53 kg/hr
Weather Conditions: Wind SSW 0-5 mph
Temp 75°F

Sample Loc.	Conc. of Benzene (ppm)	Conc. of Isobutane (ppm)	Mass to Mass Ratio ϕ/ic_4	lb/hr Benzene	kg/hr Benzene
1	11.48	0.713	21.66	25.13	11.42
2	17.54	1.02	23.06	26.75	12.16
3	16.58	0.93	23.97	27.81	12.64
Upwind	0.71	ND	--		
				Avg. 26.56	Avg. 12.07

Date: 7/15/80
Tracer Test #1, Run #2
Test Start - 8:35 a.m.

Isobutane Emission Rate: 1.16 lg/hr
0.53 kg/hr
Weather Conditions: Wind SSW 0-5 mph
Temp 75°F

1	9.45	0.977	13.01	15.10	6.86
2	13.48	1.32	13.75	15.95	7.25
3	14.90	1.49	13.45	15.60	7.09
Upwind	1.03	ND			
				Avg. 15.55	Avg. 7.07

Date: 7/16/80
Tracer Test #2, Run #1
Test Start: 7:30 a.m.

Isobutane Emission Rate: 1.28 lb/hr
0.58 kg/hr
Weather Conditions: Wind SSW
Temp 75°F

1	14.99	1.25	16.16	20.86	9.40
2	15.18	1.52	13.41	17.16	7.80
3	10.22	0.754	18.24	23.35	10.61
Upwind	1.02	ND			
				Avg. 20.46	Avg. 9.30

Date: 7/16/80
* Tracer Test #2, Run #2
Test Start - 8:09 a.m.

Isobutane Emission Rate: 1.31 lb/hr
0.60 kg/hr
Weather Conditions: Wind SSW
Temp 75°F

1	11.35	1.54	9.93	11.22	5.10
2	5.48	2.12	3.47	4.55	2.07
3	8.87	1.39	8.57	11.23	5.10
Upwind	0.77	ND	--		
				Avg. 9.00*	Avg. 4.09*

* Run voided due to leak in flowmeter.

TABLE 3-6
(Continued)

Date: 7/17/80
Tracer Test #3, Run #1
Test Start - 7:15 a.m.

Isobutane Emission Rate: 1.26 lb/hr, 0.57 kg/hr
Weather Conditions: Wind - Variable
Temp. 75°F

Sample Loc.	Conc. of Benzene (ppm)	Conc. of Isobutane (ppm)	Mass to Mass Ratio ϕ/ic_4	lb/hr Benzene	kg/hr Benzene
1	6.56	0.80	11.03	13.90	6.32
2	6.86	1.41	6.54	8.24	3.75
3	6.80	1.78	5.14	6.48	2.95
Upwind	0.49	ND	--		
				Avg. 9.54	Avg. 4.34

Date: 7/17/80
Tracer Test #3, Run #2
Test Start - 8:18 a.m.

Isobutane Emission Rate: 1.24 lb/hr
0.56 kg/hr
Weather Conditions: Wind - Variable
Temp. 75°F

1	5.28	0.261	27.18	33.70	15.32
2	5.61	0.430	17.64	21.87	9.94
3	6.16	0.421	19.71	24.44	11.11
Upwind	0.61	ND			
				Avg. 26.67	Avg. 12.12

Date: 7/18/80
Tracer Test #4, Run #1
Test Start - 7:36 a.m.

Isobutane Emission Rate: 1.29 lb/hr
0.59 kg/hr
Weather Conditions: Wind N, Steady

1	18.60	3.31	7.56	9.75	4.43
2	19.68	6.84	3.87	4.99	2.67
3	19.44	4.79	5.46	7.04	3.20
Upwind	1.83	0.087	--		
				Avg. 7.26	Avg. 3.43

Date: 7/18/80
Tracer Test #4, Run #2
Test Start - 8:30 a.m.

Isobutane Emission Rate: 1.29 lb/hr
0.59 kg/hr
Weather Conditions: Wind N, Steady

1	4.41	2.57	2.31	2.98	1.35
2	5.70	3.99	1.92	2.48	1.13
3	6.18	4.50	1.85	2.39	1.09
Upwind	2.83	ND	--		
				Avg. 2.62	Avg. 1.19

TABLE 3-7

BACKGROUND FOR NAPHTHALENE MELT PIT

Date: 7/17/80
Tracer Test #1, Run #1
Test Start - 11:22 a.m.

Denver Units Operating - 1, 2, 3
Isobutane Emission Rate: 1.28 lb/hr
0.58 kg/hr
Weather Conditions: Wind SSW, 0-5 mph
Temp 80°F

Sample Loc.	Conc. of Benzene (ppm)	Conc. of Isobutane (ppm)	Mass to Mass Ratio ϕ/ic_4	lb/hr Benzene	kg/hr Benzene
1	14.65	3.01	6.55	8.38	3.81
2	15.21	3.89	5.25	6.72	3.05
3	9.71	2.04	6.42	6.94	3.15
Upwind	0.56	ND	--		
				Avg. 7.35	Avg. 3.34

Date: 7/17/80
Tracer Test #1, Run #2
Test Start - 11:55 a.m.

Denver Units Operating - 1, 2, 3
Isobutane Emission Rate: 1.28 lb/hr
0.58 kg/hr
Weather Conditions: Wind SSW, 0-5 mph
Temp 80°F

1	13.49	1.52	11.93	15.27	6.94
2	15.82	3.54	6.01	7.69	3.50
3	13.43	3.75	4.82	6.17	2.80
Upwind	0.25	ND	--		
				Avg. 9.71	Avg. 4.41

BENZENE OVER MELT PIT AT GROUND LEVEL

Grab Samples Collected 7/22/80

Time	Benzene Concentration (ppm)	
	Edge of Pit	Middle of Pit
1330	46	27
1800	67	36
2300	116	42

It can be seen that the benzene concentration was higher at the edge of the pit, which was above the point where incoming slurry splashed into the pit, than at the center of the pit. In addition, the concentrations increased with time as the pit filled.

The contribution of the Denver unit to the samples collected during the melt tests was estimated to be negligible because the plume rise from the heated pit caused the emissions from the Denver unit to rise well above the samplers. Furthermore, the top of the Denver unit from which point the Denver unit's emissions emanated was approximately six feet above ground level (top of melt pit). Thus, it is quite unlikely that the Denver unit emissions could reach the samplers during the tests on the melt pit when the melt was in progress.

The following engineering estimates of overall daily naphthalene melt pit emissions are based on all of the data collected. The benzene emission rate from the melt pit is highest during the time when the naphthalene cake is being melted. The emission rate during this half hour period is from 20 to 30 pounds per hour. During the following half hour the emissions decrease to the 10 to 20 pounds per hour range.

The emissions continue to decrease over the period that the melted naphthalene remains in the pit and the benzene content in the mix becomes depleted. Once the melt has been transferred to the drying tank and filling of the pit with slurry from the Denver units resumes, benzene emissions begin at the rate of three to six pounds per hour. As filling continues and the liquid level in the pit rises, the emission rate increases to the order of 10 pounds per hour or more until the next melt is started. These emission rates can easily vary by a factor of 2 or 3 from day to day. The temperature of the material in the pit is the primary variable which affects the benzene rate at any given time.

4.0 PROCESS DESCRIPTION AND OPERATION

4.1 PROCESS DESCRIPTION

The by-product recovery operations for tar and flushing liquor at Bethlehem Steel Corporation, Bethlehem, Pennsylvania are two separate systems; Batteries 2, 3, and A as well as a separate system for Battery 5. These gas streams combine before entering the ammonia saturator. Batteries 2 and 3 have 102 ovens each and were constructed in 1941-43 using a Koppers-Becker design. Battery A has 80 McKee-Otto ovens that began operation in 1976. Batteries 2 and 3 produce a heavy tar because the hot top of the oven causes cracking of the carbon compounds in the coke oven gas. The specific gravity of the heavy tar is in the range of 1.25. Battery 5 has 80 Koppers ovens with horizontal flues that were constructed in 1953. Battery 5 produces light tar with a specific gravity of approximately 1.19.

The processes used at the Bethlehem plant for coke oven gas recovery are primary cooling, tar decanting, exhausting, tar electrostatic precipitation, ammonia still and saturator, final cooling, light oil scrubbing and rectifying, and Sulfiban desulfurization with Claus recovery. A process flow diagram of the gas and liquid streams is depicted in Figure 4-1.

The gas leaving the ovens is collected in the collecting mains where it is sprayed with flushing liquor. The gas and flushing liquor leave the battery area and are transported from the collecting main through cross-over mains into the suction main and into the by-product recovery area. The gas and liquor initially separate at the downcomer where the flushing liquor falls out and the gas continues to the primary coolers. The flushing liquor from Batteries 2 and 3 enters an interceptor pit before being pumped to the

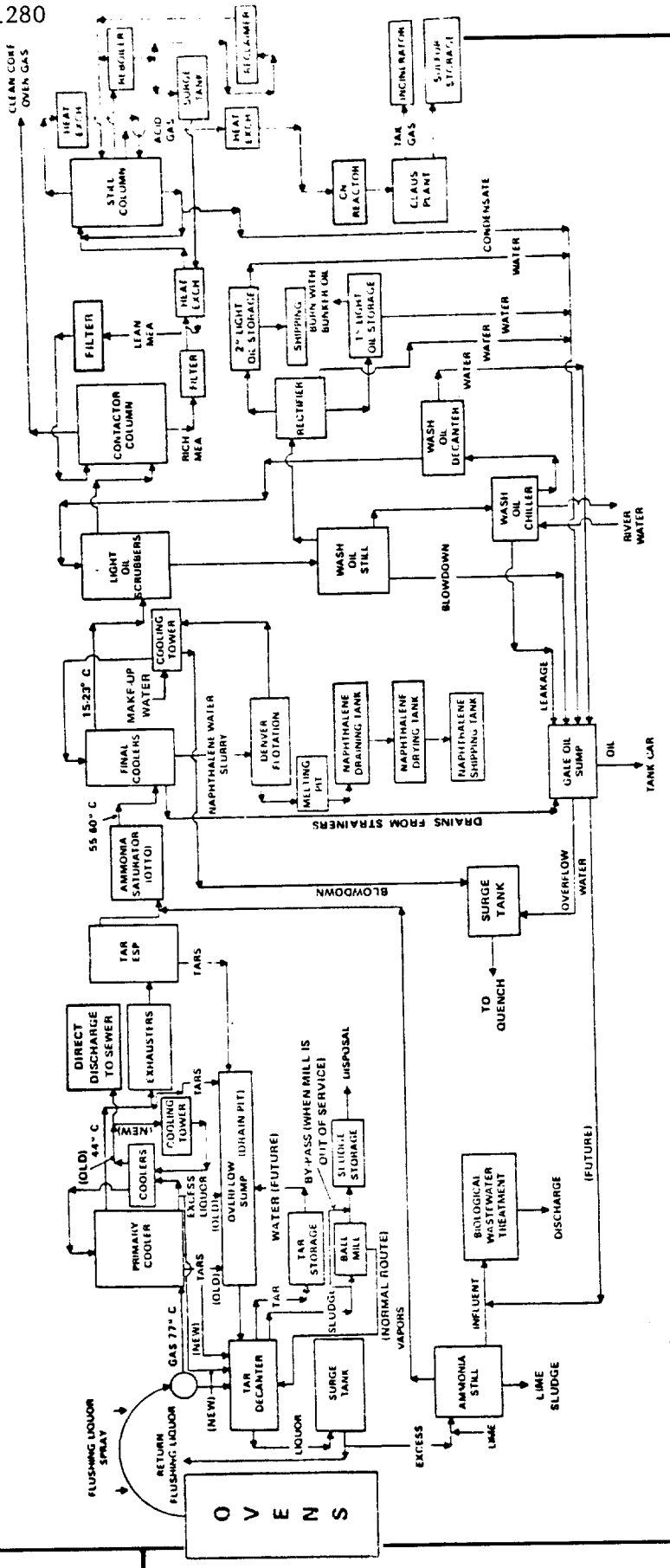

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FIGURE 4-1 BETHLEHEM STEEL CORPORATION, BETHLEHEM, PA

tar decanter because ground elevations will not allow for gravity flow. The interceptor pit removes some sludge which is stored in a dumpster before disposal. The detention time in the interceptor pit is very short with a flow rate of 189 l/s (3000 gpm). The flushing liquor from Battery A does not enter this pit, but flows by gravity to the tar decanter.

As previously stated the tar and flushing liquor operations are two separate, but similar systems. This discussion will address the operations for Batteries 2, 3 and A because the plant tour surveyed this system. The gas stream from Battery 5 joins the gas stream from Batteries 2, 3 and A before the ammonia saturator. Excess flushing liquor from both systems are steam stripped in the same ammonia still.

The dirty flushing liquor enters the two parallel tar decanters where it is separated into liquor, tar, and sludge. Liquor from the overflow pit is also separated in the tar decanters. The flushing liquor flows by gravity to a surge tank before returning to the spray system on the collecting mains. Excess flushing liquor from the surge tank is treated with lime before stripping in the ammonia still. The flushing liquor ammonia concentration is approximately 3000 mg/l before the still. The ammonia rich vapors exit at the top of the ammonia still and combine with the main gas stream before the ammonia saturator. The ammonia concentration in the effluent from the ammonia still is 1.2 mg/l before entering the aeration basins. In the future the plant will increase the ammonia concentration to approximately 40 mg/l to enhance the biological wastewater treatment process.

The tar layer from the tar decanter is pumped to tar storage. The water content of the tar is approximately 10-12% from the tar decanter and 3-4% after tar storage. The water content of the tar from the tar decanter could increase to approximately 35% when charging problems occur. The tar in storage is heated to 94°C for several days before shipping. Heavy tar from Batteries 2, 3, and A is produced at a rate of 181.7 cubic meters (48,000 gallons) per day. Light tar from Battery 5 is produced at a rate of 45.4 cubic meters (12,000 gallons) per day. The sludge layer from the tar decanter is pulverized in a ball mill before storage and disposal.

The gas stream enters four parallel primary coolers at 77°C where it is sprayed with circulating liquor. During the visit two old primary coolers were not operating due to reactivation. The circulating liquor is cooled by indirect coolers before recirculating in the primary coolers. Excess circulating liquor and tars are drained to the overflow sump from the old primary coolers. The excess liquor from the new primary coolers goes directly to the decanters. The gas leaves the primary coolers at approximately 44°C.

The gas stream enters the exhausters where the prime motive power for the system is supplied. The gas then enters four parallel tar electrostatic precipitators where additional tar is removed from the gas and drained to the overflow pit (drain pit).

The gas from the tar electrostatic precipitators is combined with the gas stream from Battery 5 and the vapors from the ammonia still before entering the ammonia saturator. The ammonia saturator is an Otto design that sprays 2% sulfuric acid through the gas as it rises in the saturator

column. The system produces 65.3 metric tons (72 tons) of ammonium sulfate per day. Before the ammonia still was installed the plant produced 54.4 metric tons (60 tons) of ammonium sulfate per day.

The gas leaving the ammonia saturator is approximately 55-60°C before entering the final coolers. The final coolers are arranged in three parallel rows with two rows having two coolers each in series and one row having one cooler. There is normally one cooler in each row in service at any given time. The final coolers circulate water which is indirectly cooled before respray. The naphthalene/water slurry from the bottom of the final coolers is conveyed to a Denver flotation unit via an open trough. In the Denver unit the naphthalene slurry is floated and scraped from the surface and then drained to a melting pit. The naphthalene slurry is heated in the melting pit before pumping to the draining tank. From the draining tank the naphthalene goes to a drying tank and then to a shipping tank. The water from the Denver flotation is pumped to the atmospheric cooling tower for the final coolers. All operations are vented to the atmosphere.

The gas leaves the final coolers and enters the light oil scrubbers at 18°C in the winter but rises as high as 32°C in the summer. The wash oil scrubbers are arranged in three parallel rows with two rows having four scrubbers each in series and one row having two scrubbers in series. In the light oil scrubbers the wash oil flows are countercurrent to the gas stream and remove the light oil from the gas stream. The benzolyzed wash oil is then stripped of the light oil in the wash oil still. The debenzolyzed wash oil from the wash oil still is indirectly cooled in the wash oil chillers

before the wash oil decanter. In the wash oil decanter entrained water is periodically separated from the wash oil and flows by gravity to the Gale oil sump (see Figure 4-2). The wash oil from the wash oil decanter is then returned to the light oil scrubbers for reuse.

The light oil vapors from the wash oil still enter a rectifier which fractionates the light oil into primary and secondary oil. The separation between primary and secondary oil occurs at 140°C (284°F). The crude secondary oil is the BTX fraction of the light oil and is shipped to the Sparrows Point plant operated by Bethlehem Steel Corporation for further refining. The primary oil is the heavy fraction of the light oil and is burned with bunker oil throughout the plant. The plant in the past has refined the secondary crude oil, but in the fall of 1977 the unit was mothballed. The refining operations produced a caustic and acid sludge at 3.8 cubic meters (10,000 gallons) per day each and cost for the ultimate disposal of these sludges made the refining operation economically impractical.

The Gale oil sump receives waste stream inputs from the final cooler, wash oil still, wash oil chiller, wash oil decanter, rectifier, primary light oil storage, secondary light oil storage, desulfurization blowdown or condensate, and miscellaneous runoffs. The Gale oil sump separates the wastewaters into oil and water layers. In the future the water layer will be pumped to the influent to the aeration basins. The oil layer is pumped to a tank car. If the Gale oil sump receives excessive inputs the overflow flows to quench.

The gas stream from the light oil scrubbers then enters the Sulfiban desulfurization process. The gas stream initially enters two

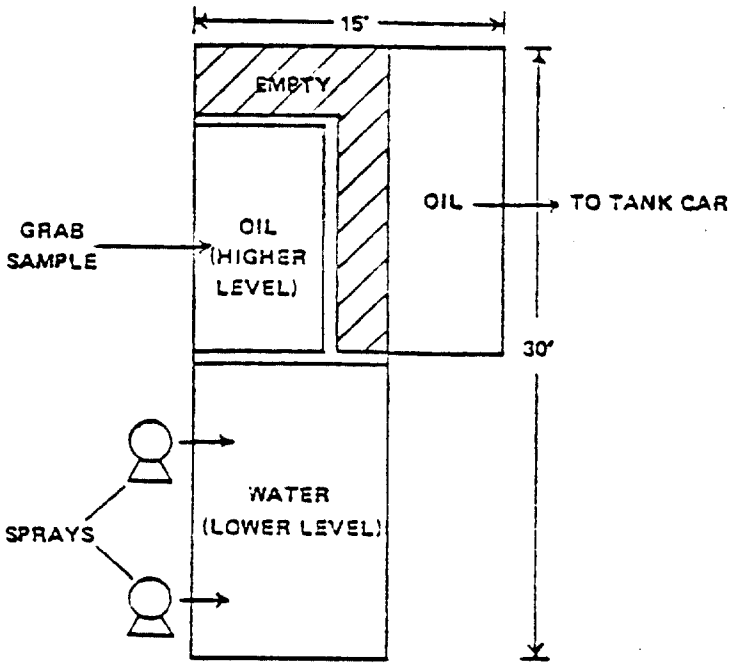

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FIGURE 4-2 GALE OIL SUMP (TOP VIEW)

packed contact columns for absorption of the sulfur and the "sweet" coke oven gas exits the top of the contact columns for reuse. The absorbing solution is 15% monoethanolamine (MEA) in water. The rich MEA is stripped in the still column to lean MEA which is returned to the contactor columns. In the still column the acid gases exit the top and are passed through a heat exchanger before entering the cyanide destructor. Some of the condensate or reflux from the still column condenser and heat exchanger are pumped to the Gale oil sump. The acid gases enter the cyanide reactor at approximately 149°C (300°F) and the cyanide is destroyed by heating to approximately 233°C (450°F) with the aid of bauxite and activated alumina catalyst bed. The acid gases leaving the cyanide reactor are then processed in a Claus sulfur recovery system which produces elemental sulfur and incinerates the tail gas.

The sweet coke oven gas after the contactor columns is held at 25 inches of water by a system that supplies natural gas at 23 inches of water and flares at 27 inches of water. The coke oven gas is used at the coke ovens and at other places within the steel mill. The heat value of the gas is approximately 530 Btu per cubic foot.

4.2 PROCESS OPERATING PARAMETERS

During the two-week test period, the plant average coke production rate was 3,900 tons per day. This resulted in generation, on the average, of 78 x 10^6 cubic feet of raw coke gas per day. Thus, we can state that the plant was operating at about 75% capacity. This capacity factor was discussed with Bethlehem personnel. While it was acknowledged that some

variations result from the longer coking cycles, there is no reason to believe that the benzene emissions, per ton of coke produced, would be significantly different from when the plant is at full capacity.

Other process operating data are presented in Table 4-1.

TABLE 4-1

PROCESS DATA, BETHLEHEM STEEL CORP., BETHLEHEM, PA

Weeks of July 7 and 14, 1980

July Date	Oven Flue Temperatures (Avg., °F) Batt. A	2	3	5	Coke Production (TPD) Batt. 5	Total	Breeze
7	2,409	2,256	2,230	2,262	710	3,995	101
8	2,410	2,260	2,220	2,252	745	4,098	101
9	2,413	2,247	2,238	2,240	725	3,828	101
10	2,415	2,235	2,236	2,240	720	4,074	101
11	2,384	2,234	2,199	2,251	740	3,928	101
14	2,411	2,250	2,272	2,269	704	3,974	182
15	2,390	2,240	2,234	2,256	657	3,718	151
16	2,383	2,233	2,236	2,263	688	3,567	151
17	2,380	2,232	2,221	2,246	707	3,887	151

July Date	Coke-Oven Gas (MSCF)	Tar (GPD)	Light Oil (GPD)	Primary Oil (GPD)	Naphthalene (GPD)
7	80,170	60,093	26,500	1,032	600
8	78,630	66,896	10,500	1,000	500
9	77,070	31,365	17,700	766	700
10	80,840	42,911	12,500	1,400	900
11	77,170	45,338	15,100	2,500	800
14	77,820	37,403	11,000	1,100	1,200
15	78,200	47,900	10,000	1,000	700
16	73,820	45,200	10,000	1,000	500
17	74,600	44,263	9,000	3,500	800

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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:

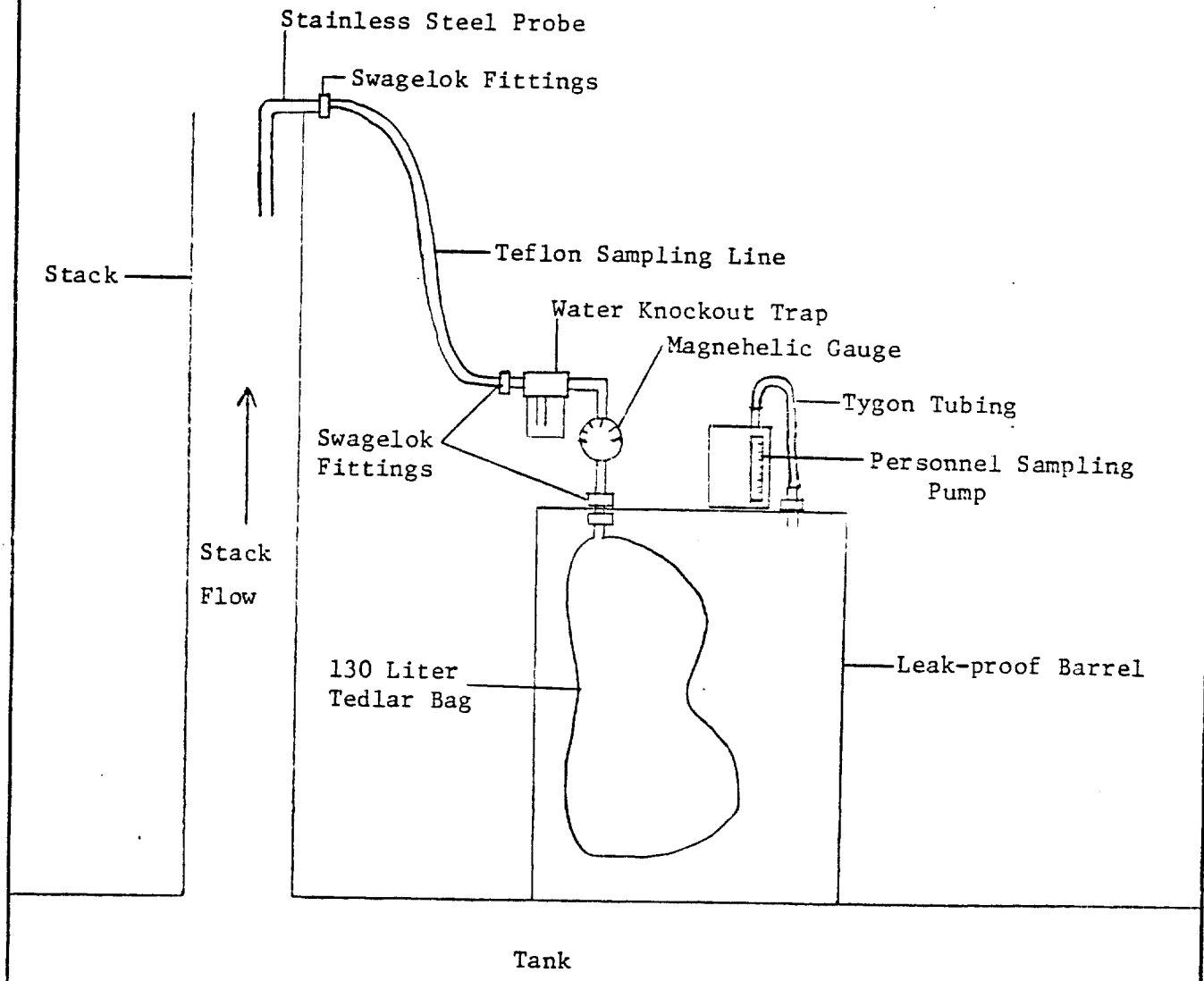
If any condensate was collected in the trap or sample line, it was measured and saved for analysis. The probe, line and trap were then washed with propylene carbonate, which was also saved for analysis. Any benzene found in these washes and water catches was added to the total found in the sample bag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

The tracer gas method is a practical procedure for quantifying mass emissions of volatile organics from sources which are essentially open to the atmosphere without disturbing flow, dispersion patterns or the source operation. This method utilizes the release of a tracer gas directly over the source of interest; the tracer gas will then follow the same dispersion patterns as the emissions from the source. The mass of tracer released over the sampling period is known and the mass to mass ratio of benzene to the tracer gas in the sample is determined by gas chromatography. The emission rate of benzene can be calculated with this information.

This method is based on the principle that the chosen tracer gas will model the dispersion of benzene from the source. The tracer gas chosen for this project was isobutane because it was not present in the sources to be tested and it could readily be separated from other source trace components by the same column used for benzene. In addition, isobutane is a non-toxic gas that can readily be dispensed from a pressurized cylinder at a uniform measured rate.

When this method was used triplicate tests were performed. Each test consisted of two 1/2 hour runs. For each run clean and backgrounded ten-liter Tedlar bags were used. Integrated samples were collected using Emission Measurements, Inc. Air Quality Sampler II systems. The AQS II samplers are self-contained units capable of collecting one or more integrated samples at a preset rate. For tracer tests the sampling rate used was ten liters per hour.

5.3 SAMPLE HANDLING

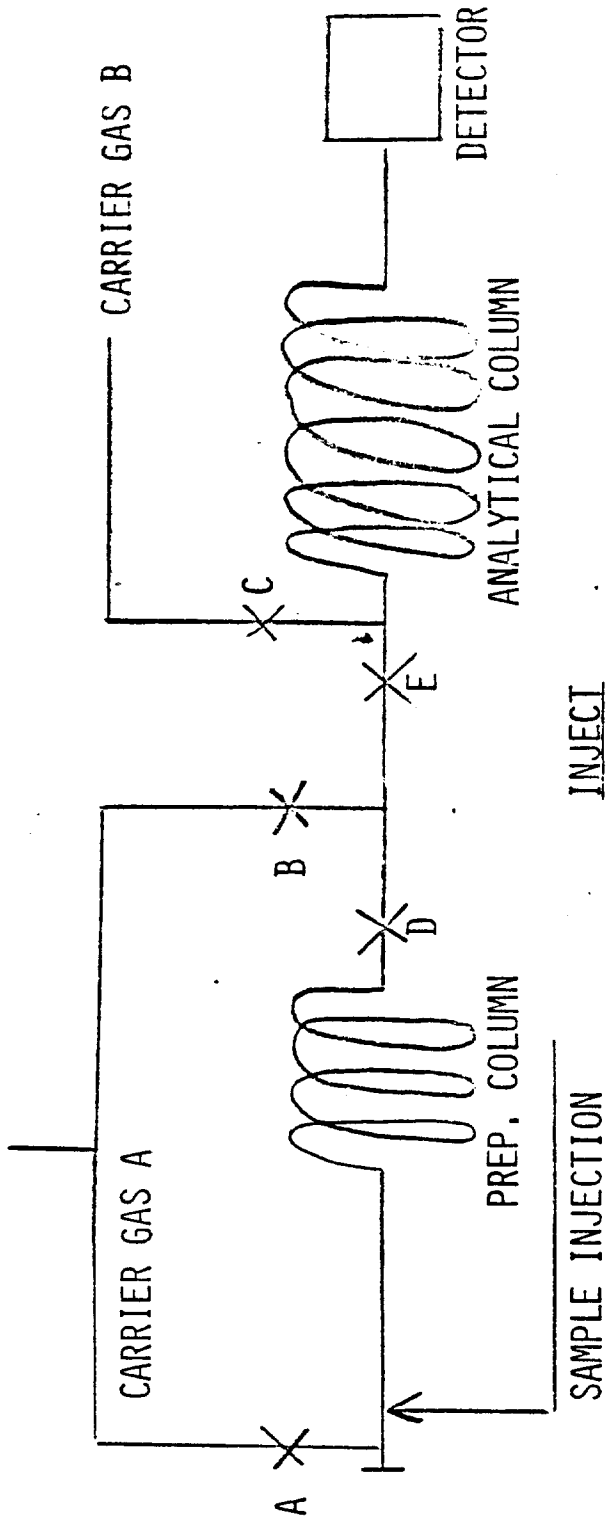
After being collected the gas samples were immediately transported to the gas chromatograph and analyzed. The elapsed time between sample collection and analysis never exceeded one hour. To verify that there was no sample degradation in samples of this type some of the samples were retained for 24 hours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

5.4 FIELD ANALYSIS

All gas samples collected were analyzed using a Shimadzu GC Mini 1 gas chromatograph equipped with dual flame inoization 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 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.

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 the injection. When only benzene was of interest 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



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

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

When samples from tracer tests were analyzed the chromatographic conditions were changed to provide adequate separation of the isobutane tracer from the other light components of the sample. The temperature program used for this analysis was:

1) Start at room temperature with external cooling fan on and oven door open.
2) Inject @ 0.0 min.
3) Turn external cooling fan off @ 1.0 min.
4) Backflush @ 1.8 min.
5) Isobutane elutes @ 2.3 min.
6) Close oven door @ 3.0 min. with oven temperature set at 100°C.
7) Benzene elutes @ 7.0 min.
8) After the elution of benzene, open the oven door and turn on the cooling fan. The next injection can be made after 2 minutes of cooling.
9) When the tracer gas is used analysis time will be approximately 10 minutes.

The columns and flow rates were the same as for isothermal.

6.0 FIELD SAMPLING PROCEDURES

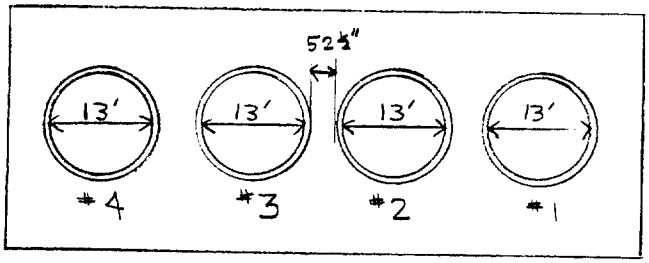
6.1 COOLING TOWER-DIRECT WATER FINAL COOLER

The cooling tower was sampled on July 10, 1980. The tower has four 13-foot diameter fans on top as shown in Figure 6-1. Sampling was conducted at only one fan and the results were multiplied by four to obtain mass emission rates from the whole cooling tower. This approach is expected to yield accurate emissions data without the necessity of testing at all four fans, because the fans were operating under identical conditions.

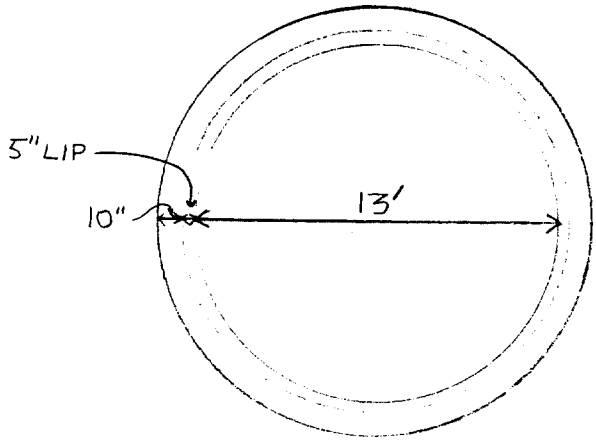
Air sampling was conducted following EPA Method 110 using a 24-point sampling and velocity traverse across two diameters of the fan shroud to obtain an integrated sample. At two minutes per point, each of the three sampling runs lasted 48 minutes.

Triplicate liquid samples were dipped from the hot and cold wells with temperatures of 30°C and 27.8°C respectively (86°F and 82°F). At the time of sampling, the cold well was mixing back into the hot well at one location due to a faulty level control. Liquid samples were dipped from points well clear of the mixing area. The plant indicated that average normal operating temperatures for summer are 86°F and 76°F.

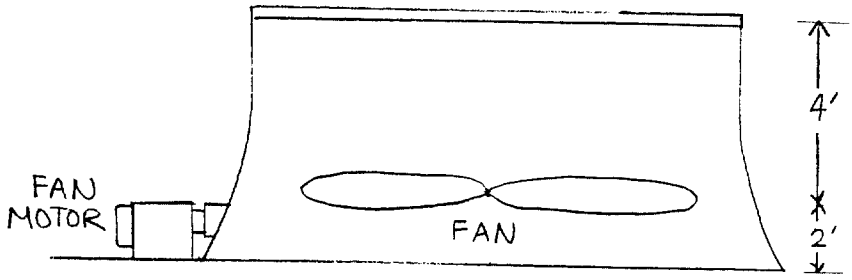
FIGURE 6-1



PLAN VIEW



#2 FAN SHROUD



SIDE VIEW

6.2 TAR DECANTER

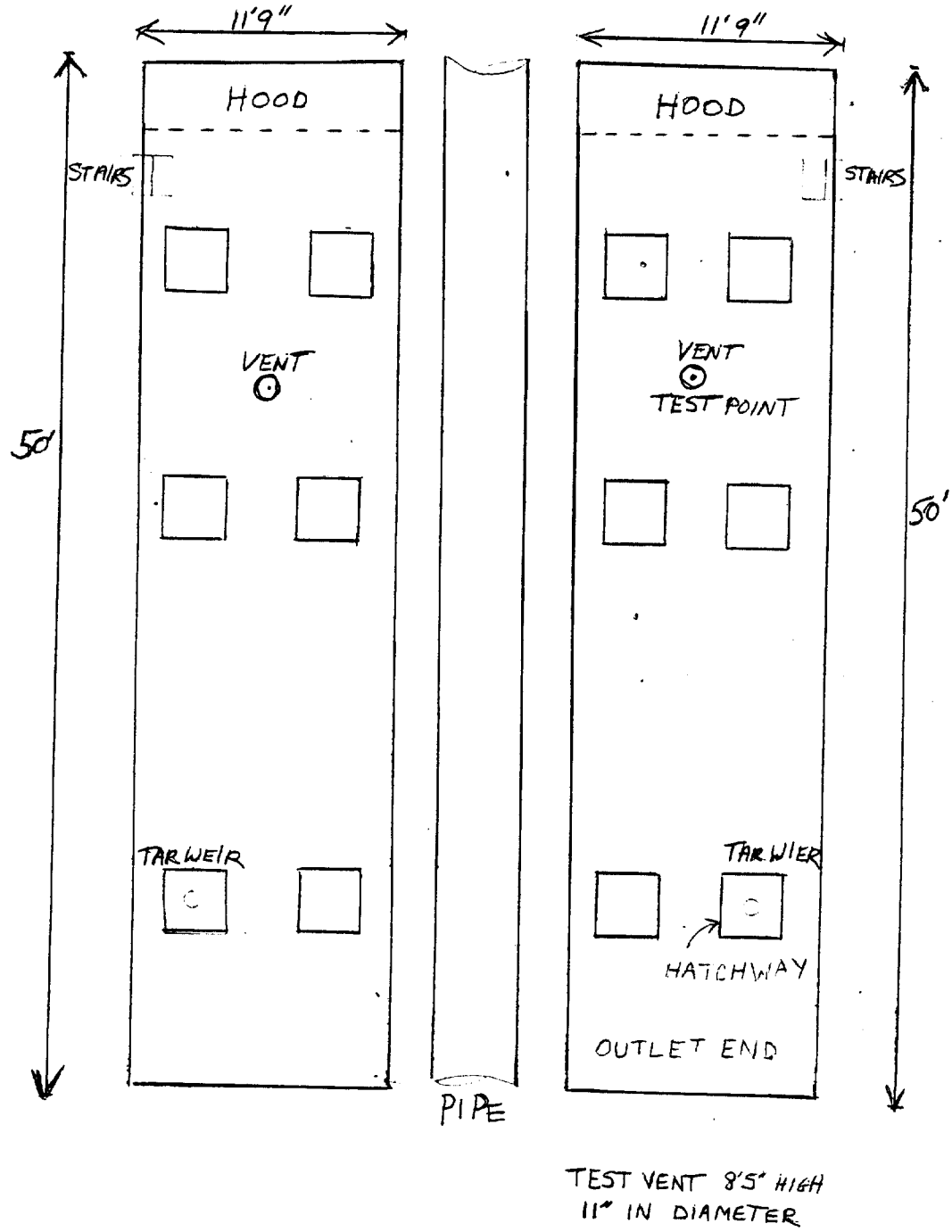
Three half-hour Method 110 tests were conducted on the tar decanter from the #5 battery on July 8th and 9th, 1980. The tar decanter is shown in Figure 6-2. Problems were encountered with naphthalene plugging the sample line. The decanter was the first source we tested using Method 110, and at the beginning several tests were run in which the sample line clogged without our knowledge resulting in no sample collection.

At this point we spent considerable time revising the method for application to this project. The equipment was modified to include an orifice and magnehelic gauge in the sample line to register flow into the bag and a water knockout trap in the line before the orifice to prevent moisture from entering the bag. Clean-up procedures were as described in Section 5.1.

From here on, all tests referred to as Method 110 include these revisions.

The tar decanter receives tar and flushing liquor from the coke gas crossover main from the #5 battery and also from the primary cooler. A total of five liquid samples were collected as follows: Two were dipped from a hatchway on top of the decanter at the outlet end, one was collected from the gas crossover main, and two were taken from the primary cooler outlet.

FIGURE 6-2



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BATTERY #5 TAR DECANTER
EPA METHOD 110

6.3 LIGHT OIL CONDENSER VENT

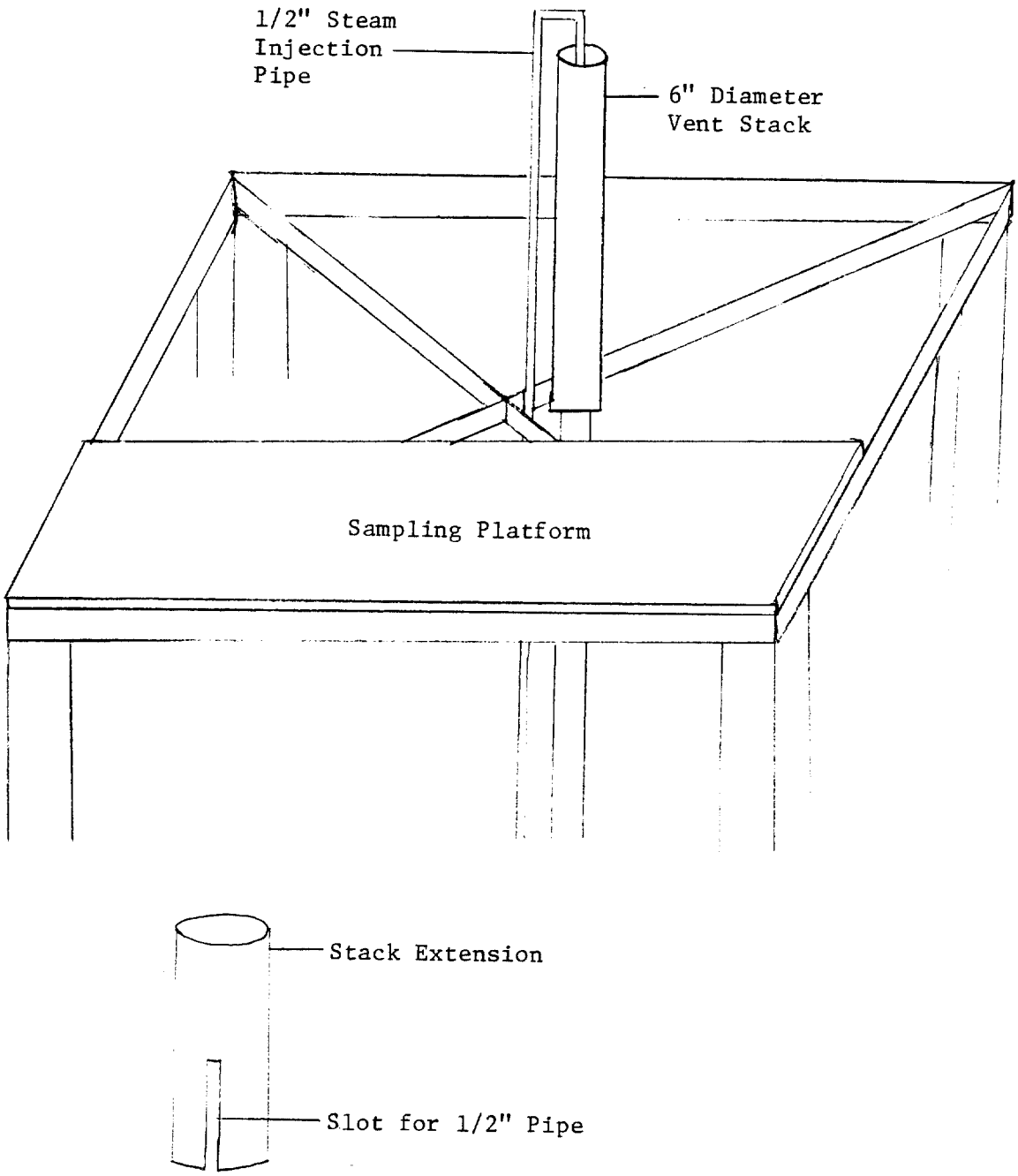
Four half-hour EPA Method 110 tests were conducted on the light oil condenser vent on July 11, 1980. The results of the analysis showed the benzene concentration in the third sample to be about half that found in the first two, indicating a possible leak in the system. Upon inspection of the sample line, the leak was found to be caused by an improperly seated gasket in the water knockout trap, and the third run was voided. A fourth test was run, and the analytical results were consistent with those of the first two runs.

The top of the existing stack had a 1/2 inch steam injection pipe running into the top, as shown in Figure 6-3. A stack extension was constructed from a section of steel stovepipe that extended the top of the stack past the steam pipe so we could accurately measure flow rate with a vane anemometer.

The plant maintenance crew provided scaffolding for access to the testing site.

No liquid samples were collected at this source.

FIGURE 6-3



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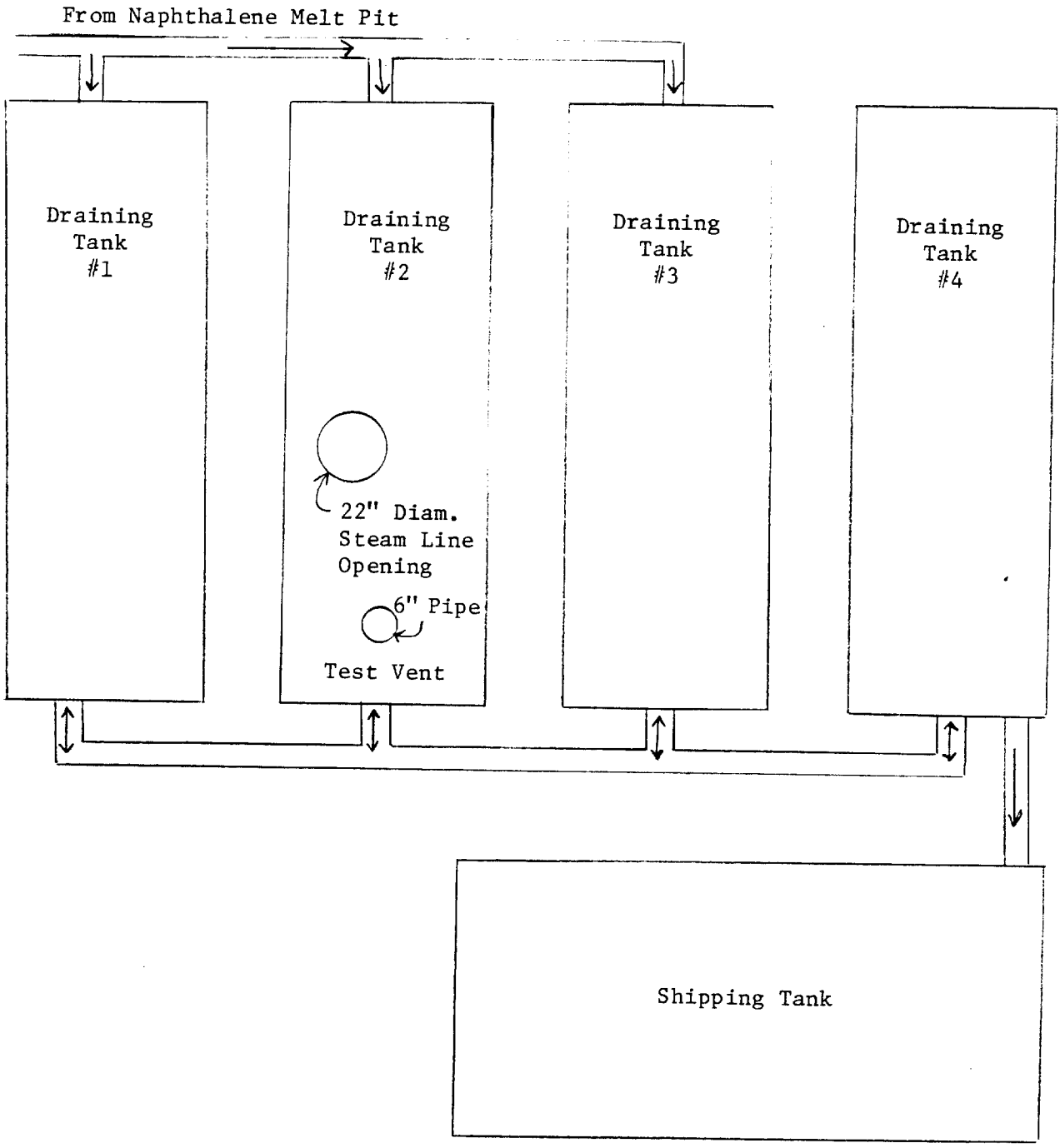
LIGHT OIL CONDENSER VENT

6.4 NAPHTHALENE DRYING TANK

The naphthalene drying tank presented several new problems in sampling strategy. The tank is shown in Figure 6-4 as Draining Tank #2. There is a large opening in the center of the tank with steam lines running in, in addition to a tall 6-inch diameter vent stack located at the end of the tank. More emissions come from the large opening than from the vent, and an attempt was made to cover the opening with plywood and fiberglass packing, but due to the pipes in the opening this was not very successful in stopping leaks. It was decided to construct a sheet metal collar around the opening, with slots to fit around the steam lines, and treat it as a vent stack. Method 110 samples were collected from the tall vent stack and velocity readings were taken at both the stack and the big vent opening. The assumption was made that the concentration of benzene is the same at the big vent opening as it is in the stack. Mass emission rates were therefore determined using the benzene concentration in the stack sample with the flow rates from the stack and the vent opening.

The second major problem encountered was naphthalene plugging the sample line and probe. The line plugged so fast there was no use in cleaning the line periodically. The solution was to bubble the sample stream through propylene carbonate to knock out naphthalene, using a large diameter glass elbow as a probe. A bucket containing three impingers was hooked on top of the stack. The first two

FIGURE 6-4



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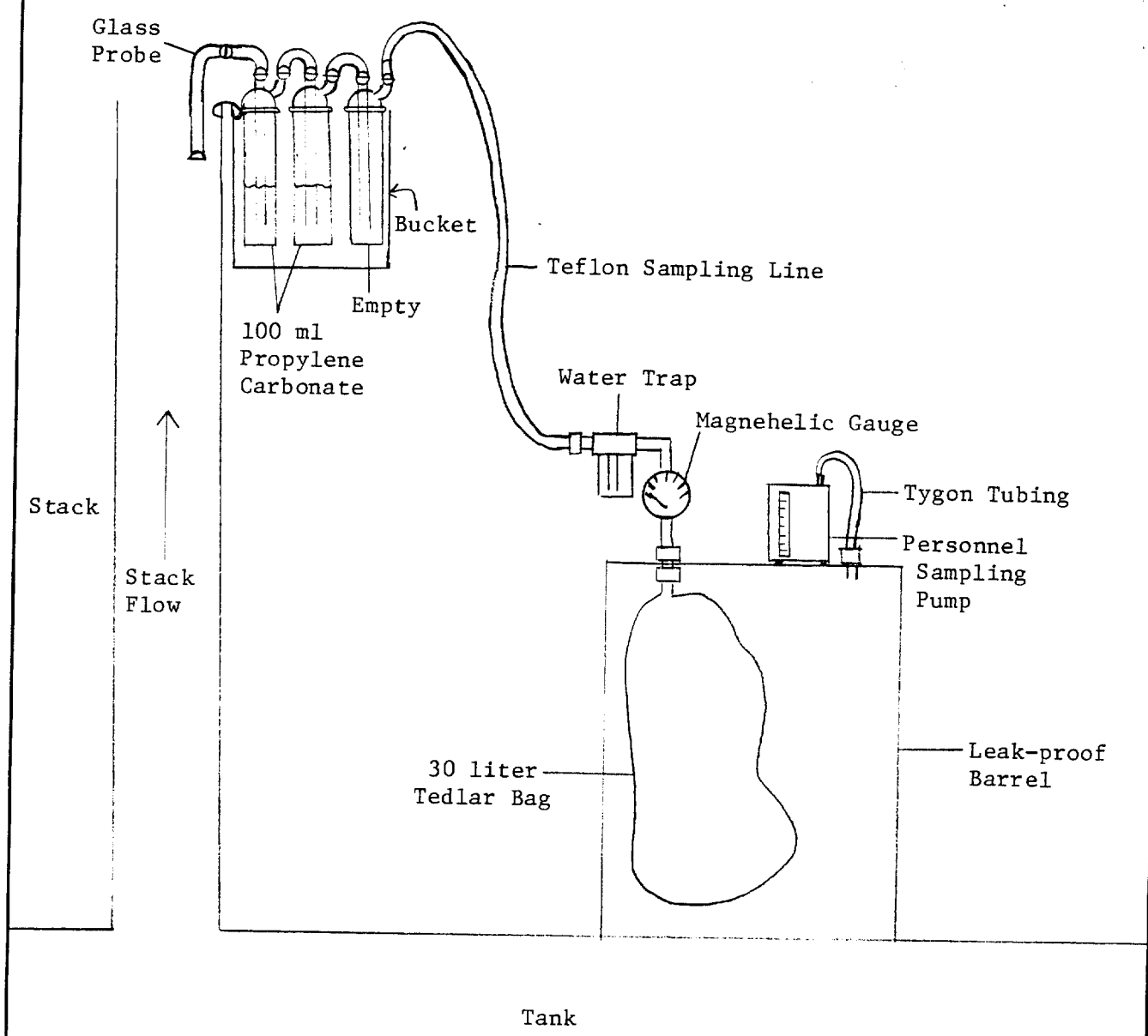
NAPHTHALENE HANDLING TANKS

impingers contained 100 ml of propylene carbonate and the third was empty. A Teflon sample line connected the impinger train to the sampling drum and the glass elbow used for the probe was connected directly to the first impinger (See Figure 6-5).

Cleanup consisted of saving the impinger catches and washes in addition to the sample line and water trap washes. The sample volume contained in the Tedlar bag was measured after the sample was analyzed by emptying the bag through a dry gas meter.

A test run was done on the drying tank on July 18 to verify the success of the new procedures. The bag sample collected was analyzed but the propylene carbonate catch was not, as it was just a trial run. Results of the bag analysis are included with the data in Table 3-4 for purposes of comparison. Naphthalene from the melt pit is pumped into a draining tank after the melt each morning, and the tank is steam heated from about 1:00 p.m. until about 4:00 a.m. when a night shift operator shuts it off. Benzene emissions are not expected to be constant over the heating cycle, so in order to measure accurately the emissions from the tank it must be tested over the entire heating cycle. We collected eight half-hour Method 110 tests modified as described at about two hour intervals during the cycle on the night of July 22, 1980.

FIGURE 6-5



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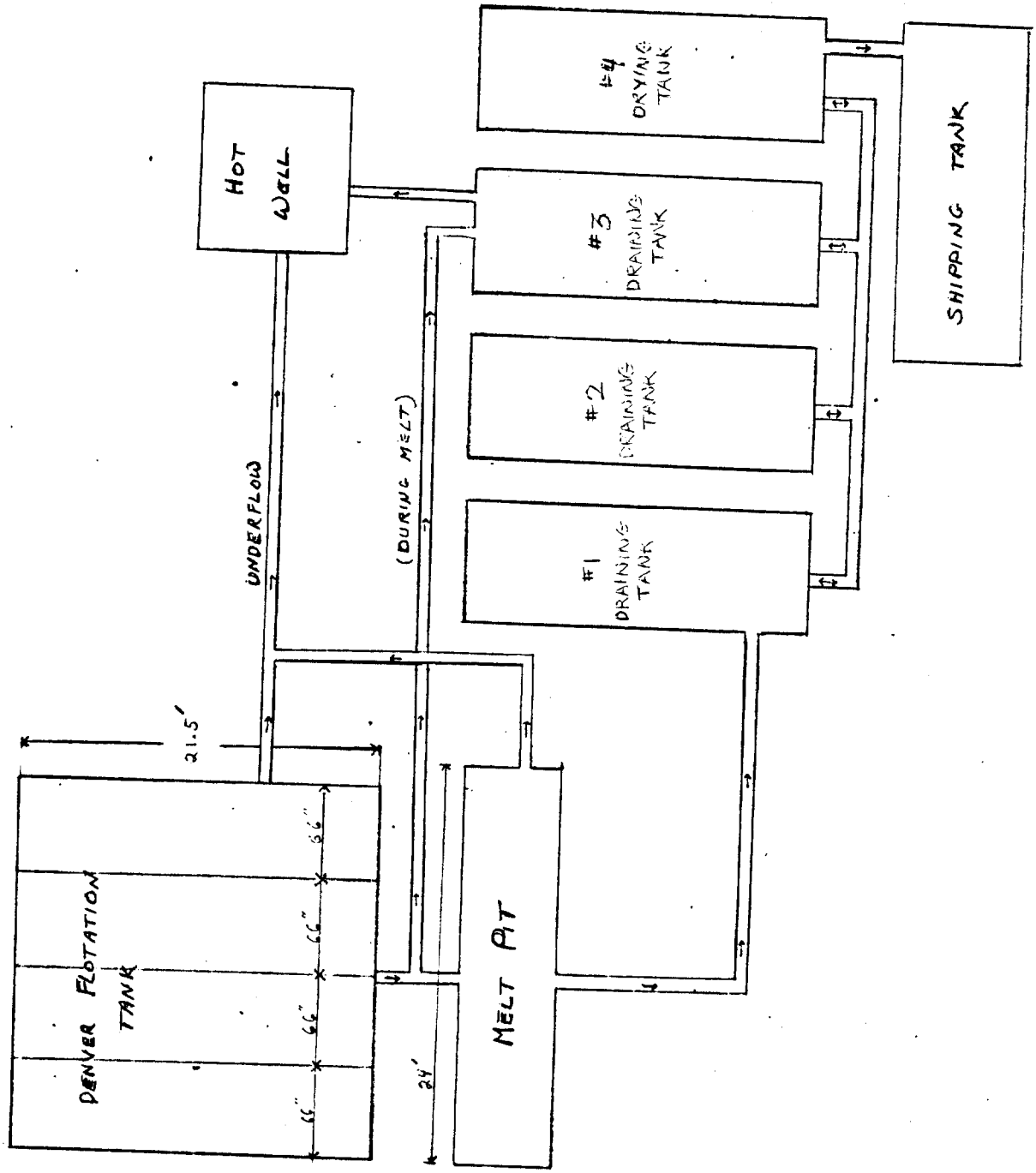
MODIFIED METHOD 110 SAMPLING TRAIN WITH PROPYLENE CARBONATE KNOCKOUT TRAP

6.5 DENVER FLOAT UNITS

The Denver float unit presented a complex problem to the use of a tracer gas for quantifying the unit's benzene mass emissions. First, there are 4 separate Denver float tanks, of which 3 were normally in use during these tests. The particular tank which was out of service varied from day to day. Second, the naphthalene melt pit was immediately adjacent to the Denver float tanks on one of the sides that was physically accessible for downwind sampling. While the emission rate from the melt pit was low, compared to the Denver float tanks, some "hot spot" points contributed to the downwind samples. For example, the point at which the Denver float overflow trough serving Units 1 and 2 empties its contents into the melt pit was shown to be a "hot spot" for benzene in subsequent grab samples. Figure 6-6 shows the processes and flow directions for the entire naphthalene handling operation. Figure 6-7 shows specifically the Denver float units and the positions of the samplers for the Denver unit tests.

The sampling strategy used was believed to be the best means of arriving at reasonably accurate emission rates without unduly elaborate and costly sampling procedures. The simultaneous use of a different tracer gas at each tank and tests utilizing different tracer gas release configurations would probably have resulted in better confidence in the emission rates during a particular test period. However, the emission rate varies from day to day due to variations in

FIGURE 6-6



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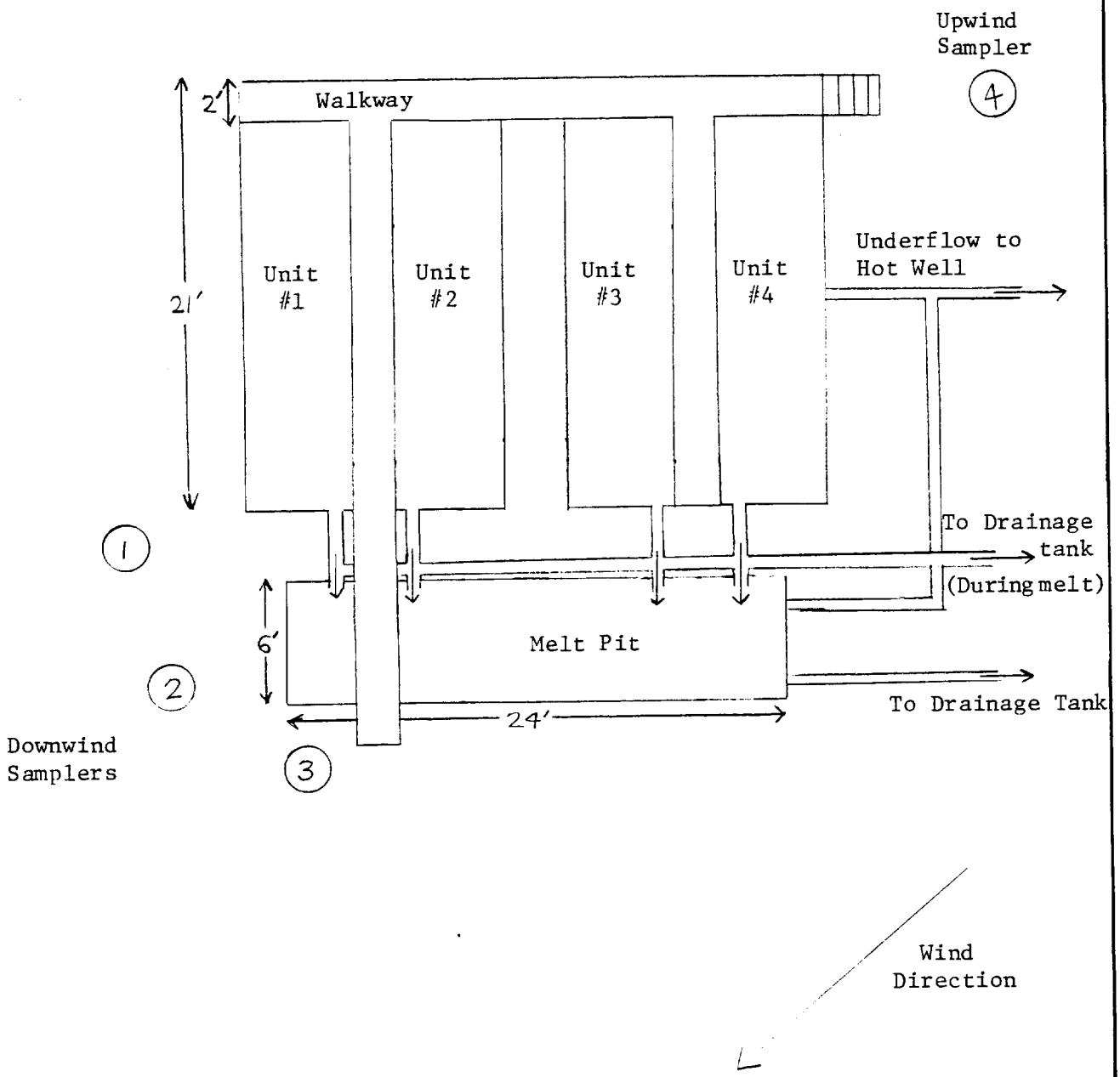
SERIES OF OPERATION: DENVER FLOTATION UNIT-TO-SHIPPING TANK

FIGURE 6-7



Scott Environmental Technology Inc.

DENVER FLOTATION UNITS

both process and ambient conditions. Thus, it was not cost effective to perform very elaborate test procedures.

The approach used in the Denver float unit tests was to measure the emissions from a single tank. The tracer gas was dispersed onto the surface of this tank with the gas discharge probe located along the center longitudinal point; there will be contributions of benzene from the other two Denver tanks then in use. The relative contribution from the second tank was estimated by releasing the tracer onto the surface of the second tank in a test immediately following the first tank test without changing the position of the samplers. The relative contribution of the two tanks to each sampling location is proportional to the relative amounts of tracer found at that location. There are two assumptions inherent to this conclusion. First, the benzene emission rates from the two tanks are equal. This should be true because the temperature, feed material and size were the same for the two tanks. Second, the diffusion patterns were the same in the two tests. This was demonstrated by comparing the benzene concentrations in each sampler for the two tests.

The sampler locations for Test 1 are shown in Figure 6-7. The isobutane tracer concentrations from the two tests were normalized for differences in isobutane release rate and differences in dispersion. The normalized values were then used to calculate the fraction of the benzene due to emissions from Tank 1. The contribution from Tank 3 was not determined. Because of the additional spacing between Tanks 2 and 3, the contribution ratio of Tank 3 to Tank 2 would be less than

that of Tank 2 to Tank 1. It is believed that Tank 3 contributed less than 5% of the total found in the samples. The calculations shown in Appendix A assume a negligible contribution from Tank 3. The upwind background from distant sources was also assumed to be negligible. The trace benzene concentrations found in the upwind sampler were primarily due to the Denver unit tank emissions swirling during wind shifts. No source was immediately upwind of the Denver unit, and grab samples verified the absence of benzene in the background air mass.

In Tests 2 and 3, the test procedure was the same as in Test 1. However, during Tests 2 and 3, Tank 2 was out of service and thus did not contribute to the benzene found. In Tests 2-2 and 3-1 the tracer gas should have been dispersed over Tank 3 rather than Tank 2 which was out of service. Unfortunately, this was not recognized because this newly developed procedure had not been used before under these circumstances. Tests 2-2 and 3-1 serve as replicates for the benzene concentrations found in Tests 2-1 and 3-2, respectively.

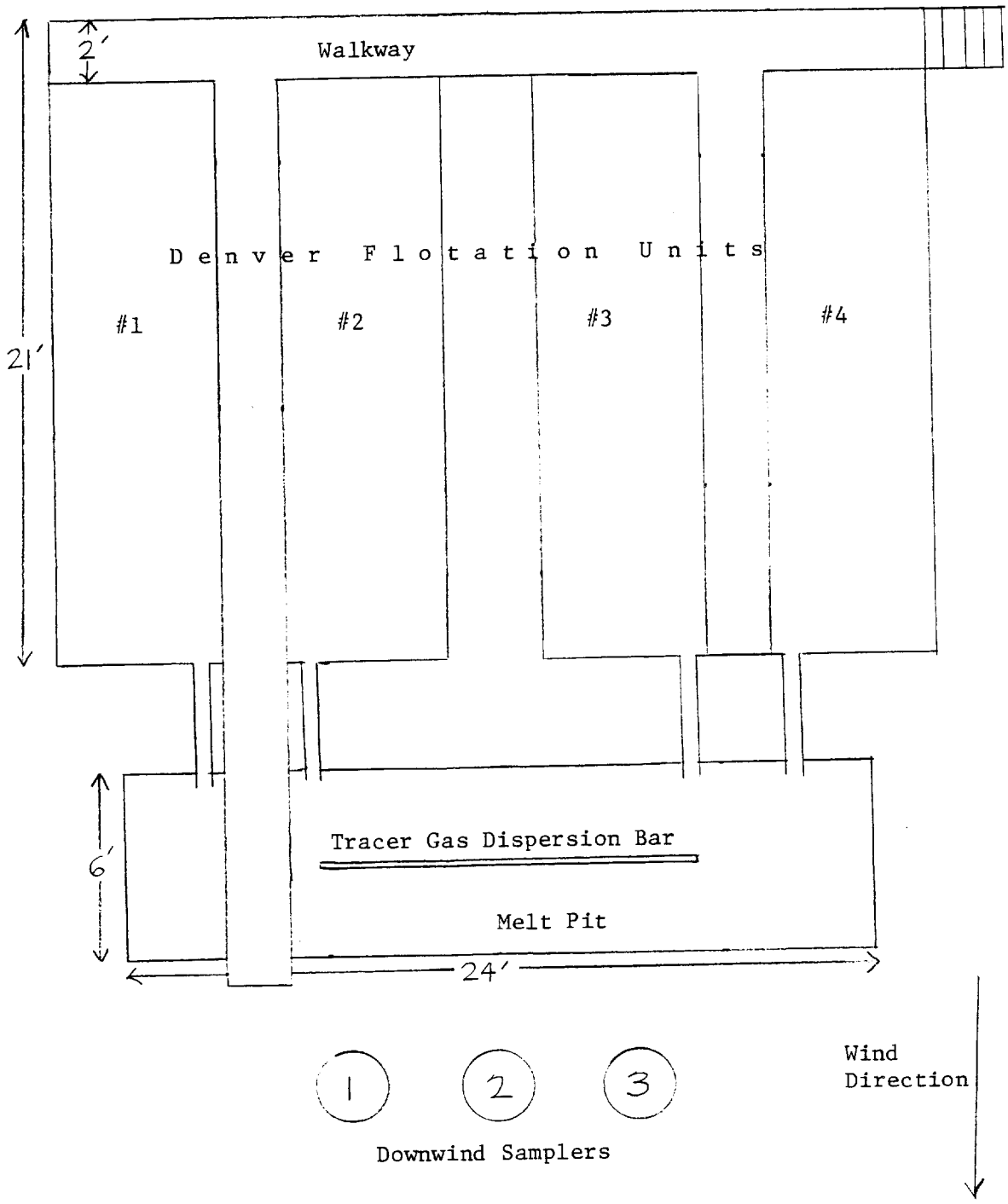
All of the benzene found in Tests 2 and 3 is attributable to Tank 1, since Tank 2 had no emissions, and it is assumed that the Tank 3 contribution to the samplers was negligible as in Test 1.

6.6 NAPHTHALENE MELT PIT

The naphthalene melt pit is diagrammed in Figure 6-8. The melt pit is 24 feet long, 6 feet wide and approximately 8 feet below grade. Generally there is one melt cycle per day at the beginning of the first shift. During the melting operation, which lasts approximately one hour or until all the naphthalene is melted, massive emissions of steam and naphthalene are released from the melt pit. These emissions were sufficiently large that small variations in wind speed and direction would not interfere with plume dispersion and the collection of representative samples.

The sampling strategy was to position samplers downwind from the melting process at a distance that would prevent samplers from becoming clogged with naphthalene. Three samplers were placed approximately 10 feet from the edge of the melt pit and were 5 feet apart, an upwind sampler was also positioned approximately 10 feet from the source. At these sampling locations it was assumed that there was no contribution from the Denver float units because the mass and velocity of the plume rising from the melt pit would essentially block emissions from that source from reaching the sampling locations. The gas dispersion bar was positioned on the grating which covered the melt pit approximately 5 feet above the surface of the naphthalene slurry. It is preferable to disperse the tracer at the liquid level of the source but in this case proper safety procedures precluded that arrangement.

FIGURE 6-8



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NAPHTHALENE MELT PIT

The sampling strategy used was believed to be the best means of arriving at reasonably accurate emission rates without unduly elaborate and costly sampling procedures. The simultaneous use of a different tracer gas at the melt pit and on the Denver float units and tests utilizing different tracer gas release configurations would probably have resulted in better confidence in the emission rates during a particular test period. However, the emission rate varies from day to day due to variations in both process and ambient conditions. This, it was not cost effective to perform very elaborate test procedures.

Between Runs 1 and 2 of Test 2, the dry gas meter was dropped and a leak developed at the rotameter at the exit of the gas meter. This was not detected until after Run 2. As a result, a portion of the tracer gas was released to the air near Sampler 2 instead of through the dispersion probe. Thus, the benzene emission results for Samplers 1 and 3 are somewhat high and that for Sampler 2 is too low. In addition, the leak was after the dry gas meter, so the metered release rate of isobutane was not the rate at which isobutane left the dispersion probe. For these reasons, Test 2, Run 2 was not valid. The results were included in Table 3-6 for comparison of the benzene concentrations measured, which are valid.

Four tests were run on consecutive days. During the first three tests, the wind was from the S to SW and the sampler location was as shown in Figure 6-8. During Test 4, the wind direction was from the north. For this reason sampler positioning for this test was

different than for the first three tests. The samplers were positioned two feet from the melt pit between the melt pit and the Denver units and approximately five feet from the dispersion bar.

After Test 3, a test was performed to measure the emissions present at the sampling locations when a melt cycle was not in progress. The tracer apparatus and samplers were set up as they were for Tests 1, 2 and 3 on the melt pit. During this test the wind was light but steady over the Denver units. The benzene found in these samples could come from the filling melt pit, the feed troughs and from the Denver units. It was believed that the results of this test could be helpful in interpreting the data obtained at the same locations during the melt cycle.

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.

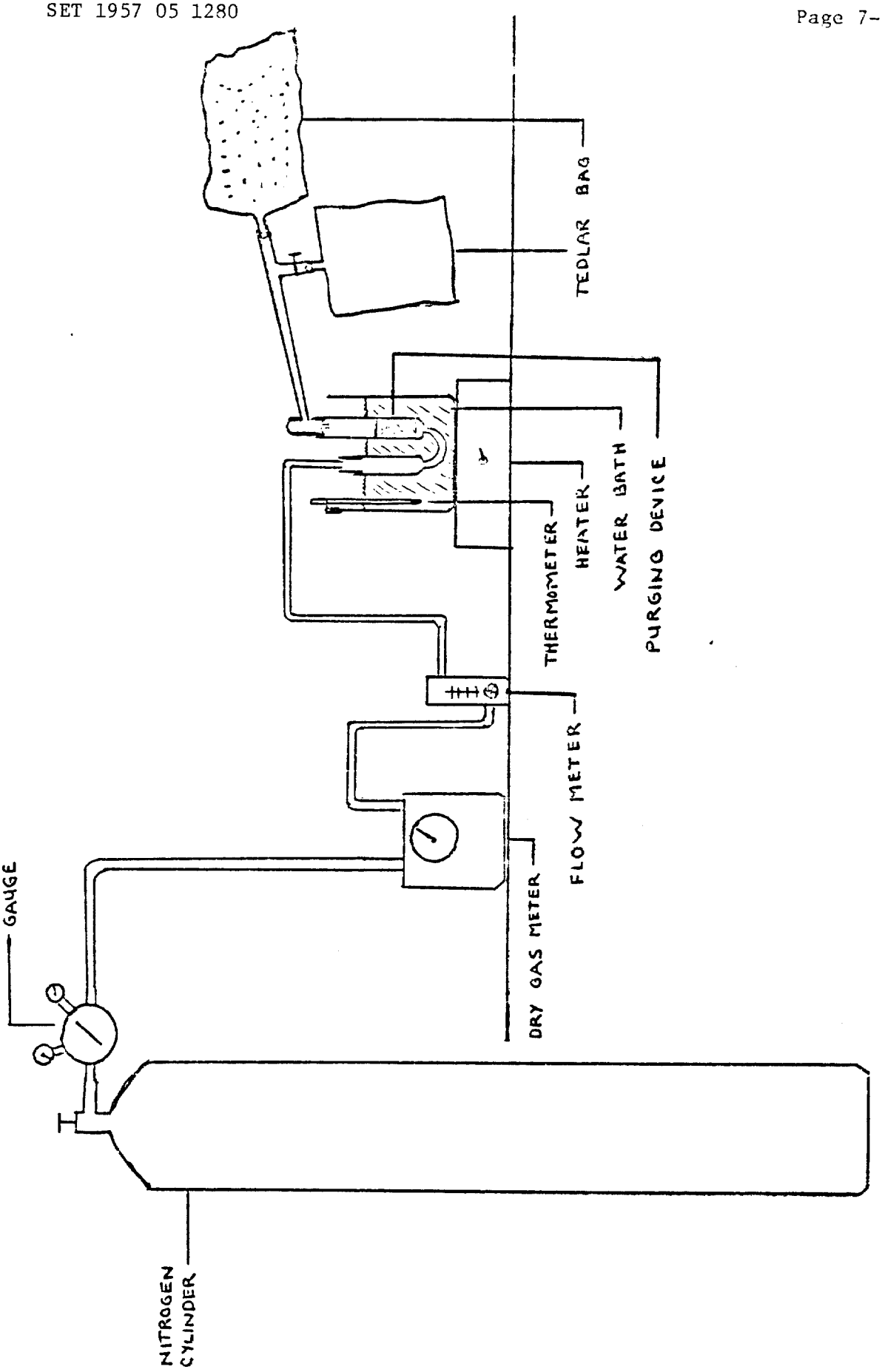

FIGURE 7-1 PURGE AND TRAP METHOD EQUIPMENT SET-UP

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.

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. Tracer Gas Calculations

Example: Naphthalene melt pit, Test 1, Run 1

Concentration of Benzene: 11.48 ppm

Isobutane release rate: 1.16 lb/hr

Calculation of mass to mass ratios:

Benzene 11.48 ppm x 78 g/mole = 895.44

Isobutane 0.713 ppm x 58 g/mole = 41.35

$$\frac{895.44}{41.35} \times 1.16 \text{ lb/hr} = 25.12 \text{ lb/hr benzene}$$

2. Flow Rate at Standard Conditions (saturated at 68°F, 29.92 inches Hg)

Example: Naphthalene drying tank, Run 1

A. Correction for temperature and pressure:

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

$$\text{Flow Rate (STP)} = 1150 \text{ cfm} \times \frac{528}{209 + 460} \times \frac{29.5}{29.92} = 895$$

B. Correction for moisture

Impinger and water trap catch volume: 76cc

Tedlar bag volume(gas sample): 0.474 ft^3 = 13.42 l

Gaseous volume of collected water, standard conditions:

$$76 \text{ cc} \times \frac{1 \text{ gm}}{\text{cc}} \times \frac{1 \text{ mole}}{18 \text{ gm}} \times \frac{24.15 \text{ l}}{\text{mole}} = 101.97 \text{ l}$$

Percent moisture:

$$\frac{101.97}{101.97 + 13.42} = 88.4\ \%$$

Flow Rate corrected for moisture:

Flow Rate (dry) = Flow Rate (STP) x (100 - % Moisture)/100

= 895 x (100 - 88.4)/100

= 104 cfm

Flow Rate (saturated at 68°F) = Flow Rate (dry) x 1.025

= 104 cfm x 1.025

= 106 cfm

3. <u>Correcting Benzene Concentration for Benzene Found in Water Trap Catch</u>

Example: Naphthalene dyring tank, Run 1

Mg benzene in catch: 1.42 mg

Tedlar bag volume (gas sample): 0.474 ft^3 = 13.42 l

Measured benzene concentration: 135.96 ppm

A. Mg benzene in collected gas sample:

$$\frac{135.96}{10^6} \times 13.42\ l \times \frac{78\ g}{mole} \times \frac{1\ mole}{24.15\ l} = 5.89\ mg$$

B. Total mass of benzene (air + liquid)

5.89 + 1.42 = 7.31 mg

C. Corrected benzene concentration:

$$0.00731\ g \times \frac{1\ mole}{78\ g} \times \frac{24.15\ l}{mole} \times \frac{10^6}{13.42\ l} = 168.60\ ppm$$

4. Calculation of Benzene Mass Emission Rate

Example: Naphthalene drying tank, Run 1

Flow Rate (standard conditions) = 106 cfm

Benzene concentration = 168.60 ppm

$$106\frac{ft^3}{min} \times \frac{28.32\ l}{ft^3} \times 60\frac{min}{hr} \times \frac{168.60}{10^6} \times \frac{78\ g}{mole} \times \frac{1\ mole}{24.15\ l} \times \frac{1\ lb}{454\ g} = 0.22\ lg/hr$$

APPENDIX B

FIELD DATA SHEETS

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

RUN 1

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Cooling tower dust c

PROCESS NOTES: Fan #2

DATE: 7/10/80

AMBIENT TEMPERATURE: 80 °F

BAROMETRIC PRESSURE: 29.58

TEDLAR BAG NUMBER: 11

Can #1

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
1	10:34 0	84 °F	~100 ft/min	1.5 lpm	5748.5
2	2	83 °F	300	1.5	5757
3	4	82 °F	500		5766
4	6	83 °F	1000 ft/min		5775
5	8	82	1200	1.5	5784
6	10	83	450 ft/min		5793
12	12	84	600 ft/min	1.6	5802
11	14	85	950 ft/min	1.5	5811
10	16	86	1050	1.5	5820
9	18	85	1200	1.5	5829
8	20	85	1100	1.5	5838
7	22	84 °F	400		5847
13	11:00 24	83	750 ft/min	1.5 lpm	5855
14	26	83 °F	1100	1.5	5865
15	28	84	1100	1.5	5874
16	30	84	1200		5882
17	32	84	1100	1.5	5890
18	34	84 °F	350 ft/min		5899
24	11:15 36	84	600	1.5 lpm	5908
23	38	84	800		5917
22	40	84	1150	1.5	5926
21	42	84	1250		5935
20	44	84	1300 ft/min	1.5	5943
19	46	85	650		5952
	48				5965
		Sample Vol 1.746 cf			

RUN 2

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: cooling tower dwfc

PROCESS NOTES:
diam. 13 ft.

DATE: 7/10/80

AMBIENT TEMPERATURE: 83 °F

BAROMETRIC PRESSURE: 29.58

TEDLAR BAG NUMBER: 3
can #3

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
1	11.50 0	85 °F	~ 650 ft/min	1.5 lpm	5965
2	2	85	400 ft/min	1.5	5974
3	4	85	1000	1.5	
4	6	85	1050		
5	8	84	1200	1.5	
6	10	84	350 ft/min		
12	12	84	600 ft/min	1.5 lpm	
11	14	85	900		
10	16	85	1050	1.5 lpm	
9	18	85	1200		
8	20	85	1200	1.5	
7	22	84	450	1.5	
13	12:27 24	84	700		
14	26	84	1060	1.5	
15	28	84	1100		
16	30	83	1200	1.5 lpm	
17	32	83	1150	1.5	
18	34	83	400		
24	12:30 36	85	500 ft/min	1.5 lpm	
23	38	85	900		
22	40	85	1100		
21	42	85	1250	1.5 lpm	
20	44	85	1250		
19	46	84	500	1.5	
	48	Sample vol.	2.069	ft^3	

RUN 3

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Cooling tower dwfc

PROCESS NOTES:

13 ft. diameter

DATE: 7/10/80

AMBIENT TEMPERATURE: ~85 °F

BAROMETRIC PRESSURE: 29.56

TEDLAR BAG NUMBER: 1

Can #1

POINT	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
1	14:17 0	84 °F	450 ft/min	1.5 lpm
2	2	84 °F	850	1.5
3	4	84 °F	1000	1.5
4	6	82	1100	1.5
5	8	83	1000	1.5
6	10	84	250	1.5
12	12	80 °F	100 ft/min	1.5 lpm
11	14	83	400	
10	16	83	700	1.5
9	18	83	1050	
8	20	84	1200	
7	22	83	650	1.5 lpm
13	14:45 24	84	700 ft/min	1.5
14	26	84	950	1.5
15	28	84	1100	1.5
16	30	83	1250	1.5
17	32	83	1150	1.5
18	34	83	300	1.5
24	36	82 °F	500 ft/min	1.5 lpm
23	38	83	900	1.5
22	40	84	1200	1.5
21	42	84	1250	1.5
20	44	84	1250	1.5
19	46	84	700	1.5
	48			

sample vol. 1.744 ft^3

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant Beth. St. Bethlehem Process Cooling tower duct Date 7/10/80

Sample No. CT HotWell 1 Time Sampled 15:40

Sample Type: (Liquid) Air

Sample Temperature 86 °F

Ambient Temperature 74 °F

hot well & cold well flowing into each other

Description of Sampling Location: from surface near inlet from Denver unit

Sample No. CT HotWell 2 Time Sampled 15:40

Sample Type: Liquid Air

Sample Temperature 86 °F

Ambient Temperature 74°F

Description of Sampling Location: same.

Sample No. cool well #1, #2 Time Sampled 15:45

Sample Type: (Liquid) Air

Sample Temperature 82 °F

Ambient Temperature 76 °F

Description of Sampling Location: from cold well, flow from bottom of cooling tower into cold well

PROJECT NUMBER 1922 TEST NUMBER 01

PLANT Bethlehem Steel, Bethlehem PA

DATE 7/10/80

SAMPLING LOCATION Cooling tower. duct C

SAMPLE TYPE Method 110

RUN NUMBER 1

OPERATORS CG, JW, FG

AMBIENT TEMPERATURE

BAROMETER

DRY MOLECULAR WEIGHT DETERMINATION BY

SAMPLING TIME (24-hr CLOCK)

SAMPLING LOCATION

SAMPLE TYPE (BAG, INTEGRATED, CONTINUOUS)

ANALYTICAL METHOD

AMBIENT TEMPERATURE

GAS \ RUN	1		2		3		AVERAGE NET VOLUME
	ACTUAL READING	NET	ACTUAL READING	NET	ACTUAL READING	NET	
CO_2							
O_2 (NET IS ACTUAL O_2 READING MINUS ACTUAL CO_2 READING)							
CO (NET IS ACTUAL CO READING MINUS ACTUAL O_2 READING)							
N_2 (NET IS 100 MINUS ACTUAL CO READING)							

PYRITE ANALYSIS

CO_2	O_2	

FIELD DATA

MOISTURE

Volume

Initial Meter Reading

Final Meter Reading

Barometric Pressure

Meter Temp. In

Out

Rotameter Setting

Water Volume Final

Water Volume Initial

Net Volume

Operator

COMMENTS

TRAVERSE POINT LOCATION & VELOCITY DATA BY

TRAVERSE POINT NUMBER	A=FRACTION OF I.D.	B=AxI.D. ID= 14 ft	C=B+NIPPLE NIPPLE=	VELOCITY HEAD (Δp_s), in. H_2O	STACK TEMPERATURE (T_s), °F
1	2.1	3.5"			
2	6.7	11.25"			
3	11.8	1'8"			
4	17.7	2' 6"			
5	25.0	3' 6"			
6	35.6	5'			
7	64.4				
8	75.0				
9	82.3				
10	88.2				
11	93.3				
12	97.9				
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AVERAGE					

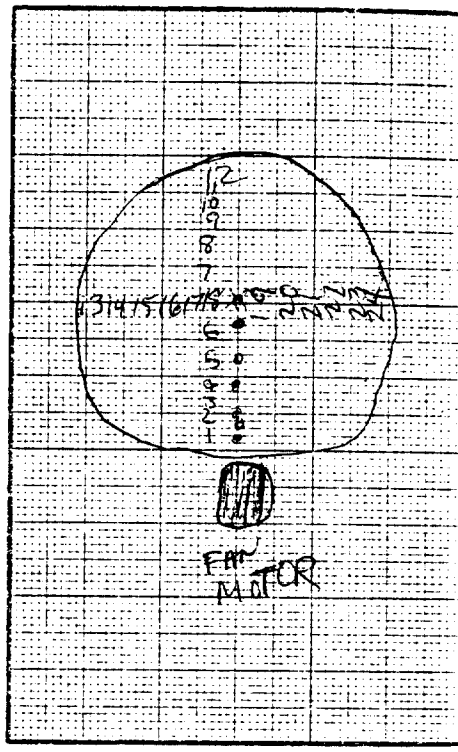


DIAGRAM OF STACK, PORTS, & TRAVERSE POINTS (indicate direction of flow)

INSIDE DIMENSIONS OF SAMPLE PLANE 13 ft

STACK GAUGE PRESSURE in. H_2O

NEAREST UPSTREAM DISTURBANCE 0

NEAREST DOWNSTREAM DISTURBANCE 3 ft

PROCESS & CONTROL EQUIPMENT DESCRIPTION

RUN 1

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Tar Decanter #5 battery

PROCESS NOTES:

10 1/8" ID stack

DATE: 7/8/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.53

TEDLAR BAG NUMBER: 4

can #4

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE		
14:53	70 °C	480 ft/min	init ~~5577.7~~	5580.1	0
7	70.5 °C	540 ft/min	1.8 lpm	5600.4	5
10	70 °C	540 ft/min		56245	11
15	70 °C	440 ft/min			
20	70 °C	490 ft/min	1.8 lpm		
25	69 °C	460 ft/min		56814	25
30	69 °C	~~480~~ ft/min	1.8 lpm	5701.6	30

RUN 2
VOID

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem
PROCESS: tar decanter, #5 battery
PROCESS NOTES:

DATE: 7/8/80
AMBIENT TEMPERATURE: ~80°F
BAROMETRIC PRESSURE: 29.53
TEDLAR BAG NUMBER: 1
Can # 1

	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
15:30	0	69°C	480 ft/min	initial	5701.6
	5	70.5°C	490 ft/min	1.8 lpm	5723.3
		~9min	pump replaced	—	
				initial	3617.9
	10	70.4°C	480 ft/min	1.3 lpm	
	15	70.5°C	480 ft/min		
	20	71.2°C	470 ft/min	1.4 lpm	3657.3
	25	71.0°C	490 ft/min	1.6 lpm	3684.0
	30	70.4°C	500 ft/min		3697.1
			VOID		
			sample line clogged - no sample collected		

RUN 3
VOID

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem
PROCESS: Tar decanter, #5 battery
PROCESS NOTES:

DATE: 7/8/80
AMBIENT TEMPERATURE:
BAROMETRIC PRESSURE: 29.53
TEDLAR BAG NUMBER: 3
Can # 3

	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
16:08	0	70.9°C	460 ft/min	1.3 lpm	3697.1
	5	70.7°C	480	1.2	3716.5
	10	70.1	480	1.2	3735.7
	15	70.2	500	2 lpm	~~3764.0~~
	20	70.0	480	1.8 lpm	3764.0
	25	70.0	480		3773.2
16:38	30	70.0	500		3781.4

VOID
sample line clogged - no sample collected

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant Bethlehem St. Bethlehem Process Tar decanter #5 battery Date 7/8/80

Sample No. TD 1 Time Sampled 15:25

Sample Type: (Liquid) Air

Liquid ~ 2ft below top of decanter

Sample Temperature 80° C

Ambient Temperature

Description of Sampling Location: Dipped from hatchway on top of tar decanter at outlet end

---

Sample No. TD 2 Time Sampled 16:00

Sample Type: (Liquid) Air

Skimmed from surface

Sample Temperature 82 °C

Ambient Temperature

flushing liquor

Description of Sampling Location: dipped from hatchway at outlet end of decanter

---

Sample No. Time Sampled

Sample Type: Liquid Air

Sample Temperature

Ambient Temperature

Description of Sampling Location:

Scott Environmental Technology Inc.

RUN #2

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Tar decanter - #5 battery

PROCESS NOTES:

1.044 ft^3 sample volume

DATE: 7/9/80

AMBIENT TEMPERATURE: ~ 90°F

BAROMETRIC PRESSURE: 29.71

TEDLAR BAG NUMBER: 1

Can # 1

	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
10:20	0	159°F	480 ft/min	1.8 lpm	3799.2
	5	162°F	400 ft/min		3814.8
		shut off	at 8 min due	to line blockage	
10:35	10	160°F	480 ft/min	.2 lpm	
		off at	12 min to install	water catch trap, new lin	
11:05	15	163°F	510 ft/min	2 lpm	3856
	20		480 ft/min		3865
	25		480 ft/min	2 lpm	3879
11:21	30	(163.4°F) 73°C	510 ft/min	2 lpm	3894

RUN 3

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Tar Decanter - #5 battery

PROCESS NOTES:

10 1/8" ID stack

DATE: 7/9/80

AMBIENT TEMPERATURE: ~95 °F

BAROMETRIC PRESSURE: 29.67

TEDLAR BAG NUMBER: 4

can# 4

	TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
15:30	0	72.8 °C	500 ft/min	2 lpm 3894
	5	72.5 °C	510 ft/min	2 lpm 3908
	10	73 °C	460 ft/min	2 lpm 3922
	15	73 °C	460 ft/min	2 lpm 3936
	20	73 °C	480 ft/min	2 lpm 3949
	25	73.1 °C	470 ft/min	2 lpm 3962
0:00	30	73.4	480 ft/min	2 lpm 3975
		water condensate 13 ml		
		Sample Vol. 44,832 L		

1240

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant Beth. Steel, Bethlehem Process Tar decanter, #5 battery Date 7/9/86

Sample No. TD 5 Time Sampled 15:45

Sample Type: (Liquid) Air

Flushing liquor outlet from primary coolers

Sample Temperature 140 °C

Ambient Temperature ~95 °F

Description of Sampling Location:

Sample No. TD 6 Time Sampled 15:45

Sample Type: (Liquid) Air

same

Sample Temperature 140 °C

Ambient Temperature ~95 °F

Description of Sampling Location:

Sample No. _____ Time Sampled _____

Sample Type: Liquid Air

Sample Temperature _____

Ambient Temperature _____

Description of Sampling Location:

RUN 1

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Light oil Condenser vent

PROCESS NOTES:

6" ID vent

DATE: 7/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.51

TEDLAR BAG NUMBER: 8

can # 3

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
10:15 0	96 °F	110	2 lpm	6449
5	95	130 ft/min	2	6422
10	94	130	1.8 lpm	6442
15	94	120	1.8 lpm	6461
20	94	120	2.0 lpm	6480
25	94	130	2.0	6498
30	94	120	2.0	6515

1.45 ft^3 sample vol.

RUN 2

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Light oil condenser vent

PROCESS NOTES:

DATE: 7/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.51

TEDLAR BAG NUMBER: 12

can # 1

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
10:56 0	95 °F	130 ft/min	2 lpm	6516
5	95	120	2 lpm	6534
10	96	140	2	6551
15	96	140	2	6570
20	96	100	2	6587
25	97	150	2	6605
30	98	110		6622

1.22 ft^3

sample vol.

RUN 3

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Light oil condenser vent

PROCESS NOTES:

DATE: 7/11/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE: 29.51

TEDLAR BAG NUMBER: 7

can #4

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
12:12 0	104°F	110 ft/min	2 lpm	6625
5	105°	100	2	6643
10	107	130	2	6661
15	108	130	2 lpm	6678
20	110	115	2	6996
25	113	130	2	6713
30	116	100	2	6731

Sample vol 1.91 ft³

RUN 4

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Light oil condenser vent

PROCESS NOTES:

very windy - anemometer readings are affected by wind

DATE: 7/11/80

AMBIENT TEMPERATURE: ~85 °F

BAROMETRIC PRESSURE: 29.49

TEDLAR BAG NUMBER: 8

can # 4

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE	
16:00 0	104 °F	150 ft/min	2 lpm	6740
5	105	130 ft/min	2 lpm	6758
10	105	120	2 lpm	67789
15	105	130	2 lpm	6796
20	105	110	2	6814
25	105	100	2	6832
30	102 °F	120 ft/min	2 lpm	

Sample vol 1.298 ft^3

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Beth Steel Bethlehem

PROCESS: Naphthalene drying tank

PROCESS NOTES:

DATE: 7/8/80

AMBIENT TEMPERATURE: hot

BAROMETRIC PRESSURE:

TEDLAR BAG NUMBER: 8, can #3

TIME	STACK TEMP	Stack GAS VELOCITY	vent flowrate ~~PUMP FLOWRATE~~
1:53 p 0	190°F	540 fpm	
5 2	204°F		~300
10 4	209		~~260, 180,~~
15 6	209		~350
20 8	209		350 fpm
25 10	209		
30	209	720 fpm	
	127 ml water collected		in 200 ml propylene carbonate

Bruce 694-5921

METHOD 1[illegible] DATA SHEET

PLANT: Bethlehem Steel, Bethlehem
PROCESS: Naphthalene Drying Tank
PROCESS NOTES: Steam started at 1:15 pm

DATE: 7/22/[illegible]
AMBIENT TEMPERATURE: 90°F
BAROMETRIC PRESSURE:
TEDLAR BAG NUMBER: 1

TIME	TEST 1 STACK TEMP	Stack GAS VELOCITY	Cav #4 ~~FUME FLOW~~
1:37 pm 0	208°F	850 fpm	660, ~~[illegible]~~, 480, 700 fpm
5	97°C		660, 450, 650
10	99°C	"	680, 470, 780
15	99	"	670, 450, 760
20	probe	clogged with condensate briefly	
25	99		670, 420, 710
30	99		
	TEST 2	Bag #3	
4:10 ~~3:45~~ 0	99°C	900	150 fpm
5			
10	99		60 fpm
15	99		
20			anemometer too clogged
25	99	248.318 } 1 min	
30		246.000 }	
		2.318 m/min =	760 fpm

3.281 ft/meter

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922 METHOD 11C DATA SHEET

PLANT: Bethlehem Steel, Bethlehem
PROCESS: Naphthalene drying
PROCESS NOTES:

DATE: [illegible]
AMBIENT TEMPERATURE:
BAROMETRIC PRESSURE:
TEDLAR BAG NUMBER:

TEST #3 Bag 7

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
6:13 0	95°C	720 fpm	
			~~248.500~~
12	94		
20	94 °C		no flow can be
25	95°C		measured –
30		660	less than 12 fpm
	run started ~ 7:00 pm		
	RUN 4 BAG 12		
9:30 0	91 °C	580 fpm	no detectable flow
10	91 °C		looks like light
20	91 °C		steam
30	91 °C	249.40 230	little to no flow
		248.50 sec	
		0.90 / 30 sec	= 1.80 m/min
			= 590 fpm

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT:

PROCESS:

PROCESS NOTES:

DATE:

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

TEDLAR BAG NUMBER:

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
12:15 am 0	89 °C	250.149	no detectable flow
		249.40	
		0.749 m/30 sec = 1.498 m/min = 491 fpm	
10	89 °C	250.900	
30	89 °C	250.000	
		0.90 m 30 sec = 1.8 m/min = 590.6 fpm	
	RUN 6		
		251.890	
2:20 am 0	89 °C	251.000	
		0.890/30 sec = 1.78 m/min = 584 fpm	
5	88 °C		no detectable flow
~15	test stopped briefly due to blockage in 1st impinger		
20	87 °C		
3:00 am 30	87 °C	252.900	
		252.000	
		0.900/30 sec = 1.80 m/min = 591 fpm	
	ambient temp		

RUN 7

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Naphthalene drying tank

PROCESS NOTES: steam on at ~1:30

DATE: 7/24/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

TEDLAR BAG NUMBER:

VOID

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
1:50 0	98°C	660 fpm	
		254.432-253.000	= 1.432 m/30 sec (2.864 m/min) 944
5		940	255.148-254.400 = ~~1.475 m/30 sec~~
			1.496 m/min 0.748/30 sec ~~2.244 m/min~~
10	99°C		256.218-255.300 = 0.918 m/30 sec
			602 1.836 m/min
20	99°C		256.850-256.100 = 0.750 m/30 sec
			492 1.50 m/min
25	99°C	leak	somewhere
	RUN 7	Bag	#8
		~~256.500~~	
2:55 0	99°C	258.348-257.100	
		1.248/30 sec =	258.716 - 258.400 ← 1/min
7		2.496 m/min (819 fpm)	0.316 m/min ~~255.340~~
			= 104 fpm
	~~temp~~	line clogged	
		test stopped after ~15 min	
		30 sec ~~28~~	
		259.830-258.700	= 1.13 m/30 min = 2.26 m/min
		742 fpm	

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

RUN 8

PROJECT 1922

METHOD 110 DATA SHEET

PLANT: Bethlehem

PROCESS: Naphthalene drying

PROCESS NOTES:

DATE: 7/24/80

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

TEDLAR BAG NUMBER: 3

TIME	STACK TEMP	GAS VELOCITY	PUMP FLOWRATE
4:04		261.191 - 260.100 =	1.090/30sec = 2.18 m/min
		715 fpm	
4:25 0	94°C		no detectable flow from
15	93°C		vent
20	92°C		
30		262.112 - 261.200	
		= 0.912/30min	= 1.824 m/min
		598 fpm	

Bethlehem Steel, Bethlehem
Naphthalene drying tank

RUN 1 168 + 108 ml = 76 ml collected
Bag 1 volume = 0.474 ft³

RUN 2
230 + 173 + 70 = 273 ml collected
Bag 3 vol = 0.160 ft³

Run 3
215 + 128 ml = 143 ml collected
Bag 7 [illegible]

RUN 4
182 + 100 ml = ~~[illegible]~~ 82 ml collected
Bag 12 Vol. = 1.590

[illegible] = 60 [illegible]
[illegible] vol = 1.295

[illegible] + [illegible] =
vol = .970

SCOTT ENVIRONMENTAL TECHNOLOGY, INC. TEST 1 RUN 1

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel - Bethlehem PA

PROCESS: Denver Flotation Tank

PROCESS NOTES:
Three units working. Unit nearest Cooling tower is down (#4)

DATE: July 8, 1980

WIND SPEED: Light & variable

WIND DIRECTION:

AMBIENT TEMPERATURE: 75°F

BAROMETRIC PRESSURE: 29.55

Weather: mostly cloudy

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	10 ft		12 ft		12 ft		10 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers	6		3		3		8	
Tedlar bag numbers	13		04		3		9	
Start Time	11:24		11:24		11:24		11:25	
Stop Time	11:54		11:54		11:54		11:55	

ISOBUTANE RELEASE: Gas Temperature

	TIME	METERED VOLUME
11:24	0	73.530
	3	73.973
	4	74.123
	6	74.412
	8	74.704
	10	74.998
	12	75.285
	14	75.577
	16	75.870
	18	76.156
	20	76.449
	22	76.740
	24	77.027
	26	77.321
	28	77.607
11:54	30	77.890

Gas Pressure

TIME	METERED VOLUME

4.360 ft³/30min = 0.1453 cfm

x 1.059 = 0.1539 cfm

SCOTT ENVIRONMENTAL TECHNOLOGY, INC. TEST 1 RUN 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver Float Unit

PROCESS NOTES:

DATE: 7/8/80

WIND SPEED:

WIND DIRECTION:

AMBIENT TEMPERATURE: ~ 80 °F

BAROMETRIC PRESSURE: 29.55

partly cloudy

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	10 ft		12 ft		12 ft		10 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers	6		3		3		8	
Tedlar bag numbers	14		23		34		22	
Start Time	12:09		12:09		12:09		12:10	
Stop Time	12:39		12:39		12:39		12:40	

ISOBUTANE RELEASE: Gas Temperature

TIME	METERED VOLUME
2:09 0	80.025
2	80.311
4	80.592
6	80.880
8	81.158
10	81.447
12	81.725
14	82.012
16	82.294
18	82.575
20	82.856
22	83.138
25	83.557
26	83.700
28	83.972
30	84.258

Gas Pressure

TIME	METERED VOLUME

4.233 ft³/30 min = 0.1411 cfm

× 1.059 = 0.1494 cfm

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 2
RUN 1

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver Float Unit

PROCESS NOTES:
Units 1,3,4 operating
Tracer line on unit 1

DATE: 7/15/80

WIND SPEED:

WIND DIRECTION:

AMBIENT TEMPERATURE: ~80 °F

BAROMETRIC PRESSURE: 29.75

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~10 ft →							
Sampling Rate	10 lph →							
Pump Numbers	3		3		3			
Tedlar bag numbers	6		7		8		9	
Start Time	10:30		10:30		10:30		10:30	
Stop Time								

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME
10:30	0	107.850 ft³
	2	108.125
	4	
	6	108.654
	8	108.920
	10	109.180 .133
	12	109.445
	14	109.710
	16	109.978
	18	110.248
	20	110.514 .133
	22	
	24	111.041
	26	
	28	111.569
	30	111.830 .132

Gas Pressure

TIME	METERED VOLUME

y = 1.059 @ .5 l/min

1 ≠ 6
3 ≠ 3

3.98 ft³/30min = 0.1326 overall
x 1.059 = 0.1405 ft³/min

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 2
RUN 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver Float Units

PROCESS NOTES: Units 1, 3, 4 operating

Tracer line on unit 2

DATE: 7/15/80

WIND SPEED: ~~mostly S~~ variable, breezy

WIND DIRECTION: mostly South

AMBIENT TEMPERATURE: ~80 °F

BAROMETRIC PRESSURE: 29.75

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~10 ft →							
Sampling Rate	10 lph →							
Pump Numbers	6		3		3			
Tedlar bag numbers	10		11		12		13	
Start Time	11:19		11:19		11:19		11:19	
Stop Time	11:49		11:49		11:49		11:49	

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME	
11:19	0	114.400	
	2	114.657	
	4	114.925	
	6	115.185	
	8	115.447	
	10	115.704	.130
	12	115.959	
	14	116.227	
	16	116.496	
	18	116.760	
	20	117.025	.132
	22	117.287	
	24	117.554	
	26	117.816	
	28	118.073	
	30	118.330	.130

Gas Pressure

TIME	METERED VOLUME

3.93 ft³/30 min = 0.131 ft³/min overall

x 1.059 = 0.1387 ft³/min

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 3
RUN 1

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver Float Units

PROCESS NOTES: tracer on unit 2
units 1, 3, 4 operating

DATE: 7/15/80

WIND SPEED:

WIND DIRECTION: roughly South

AMBIENT TEMPERATURE: ~85 °F

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~10 ft →							
Sampling Rate	10 lph →						—	
Pump Numbers	6		3		3			
Tedlar bag numbers	14		15		16		17	
Start Time	1.30						—	
Stop Time								

ISOBUTANE RELEASE: Gas Temperature

TIME	METERED VOLUME
13:00 0	119.060
2	119.326
4	119.583
6	119.844
8	120.121
10	120.392
12	120.667
14	120.940
16	121.205
18	121.468
20	121.735
22	121.997
24	122.262
26	122.532
28	122.805
30	123.071

Gas Pressure

TIME	METERED VOLUME

4.011 ft³/30min = .1337 ft³/min

× 1.059 = 0.1416 ft³/min

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 3
RUN 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver Float Unit

PROCESS NOTES: Tracer on Unit 1
Units 1, 3, 4 operating

DATE: 7/15/80

WIND SPEED:

WIND DIRECTION: mostly south

AMBIENT TEMPERATURE: ~85°F

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~10 ft →							
Sampling Rate	10 lph →							
Pump Numbers								
Tedlar bag numbers	18		19		20		21	
Start Time	14:00							
Stop Time	14:30							

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME
14:00	0	126.700
	2	126.948
	4	127.218
	6	127.492
	8	127.762
	10	.133 128.030
	12	
	14	128.565
	16	128.834
	18	129.100
	20	.1338 129.368
	22	129.640
	24	129.912
	26	130.188
	28	130.460
	30	130.742

Gas Pressure

TIME	METERED VOLUME

4.042 ft³/30min = 0.1347 ft³/min

×1.059 = 0.1427 ft³/min

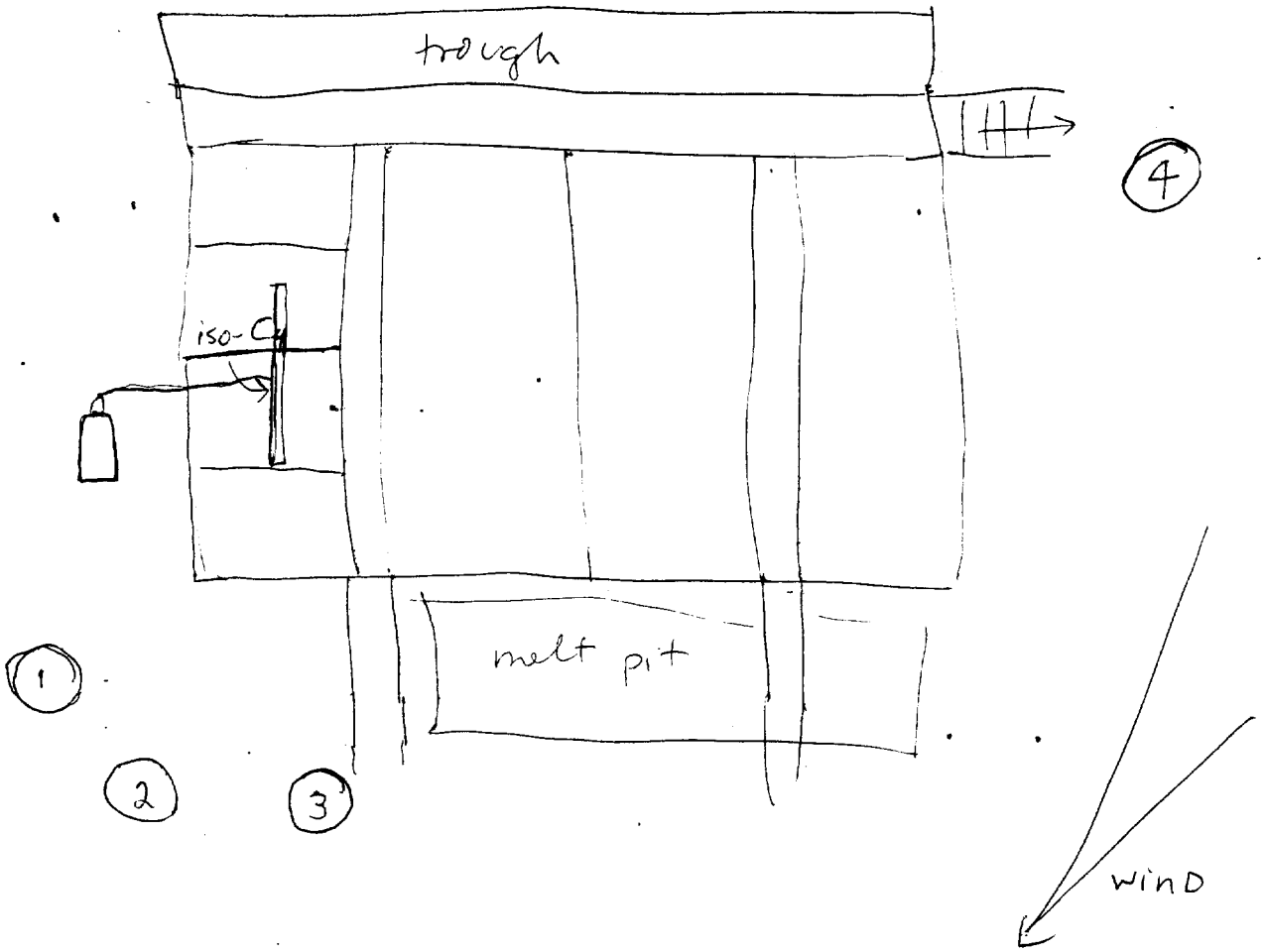
PROJECT 1906 BENZENE/BaP PRESURVEY

RUN 1

Plant Bethlehem Steel, Bethlehem Process Denver Float Unit Date July 8, 1980

Sketch of Process:

Include dimensions and flow directions.



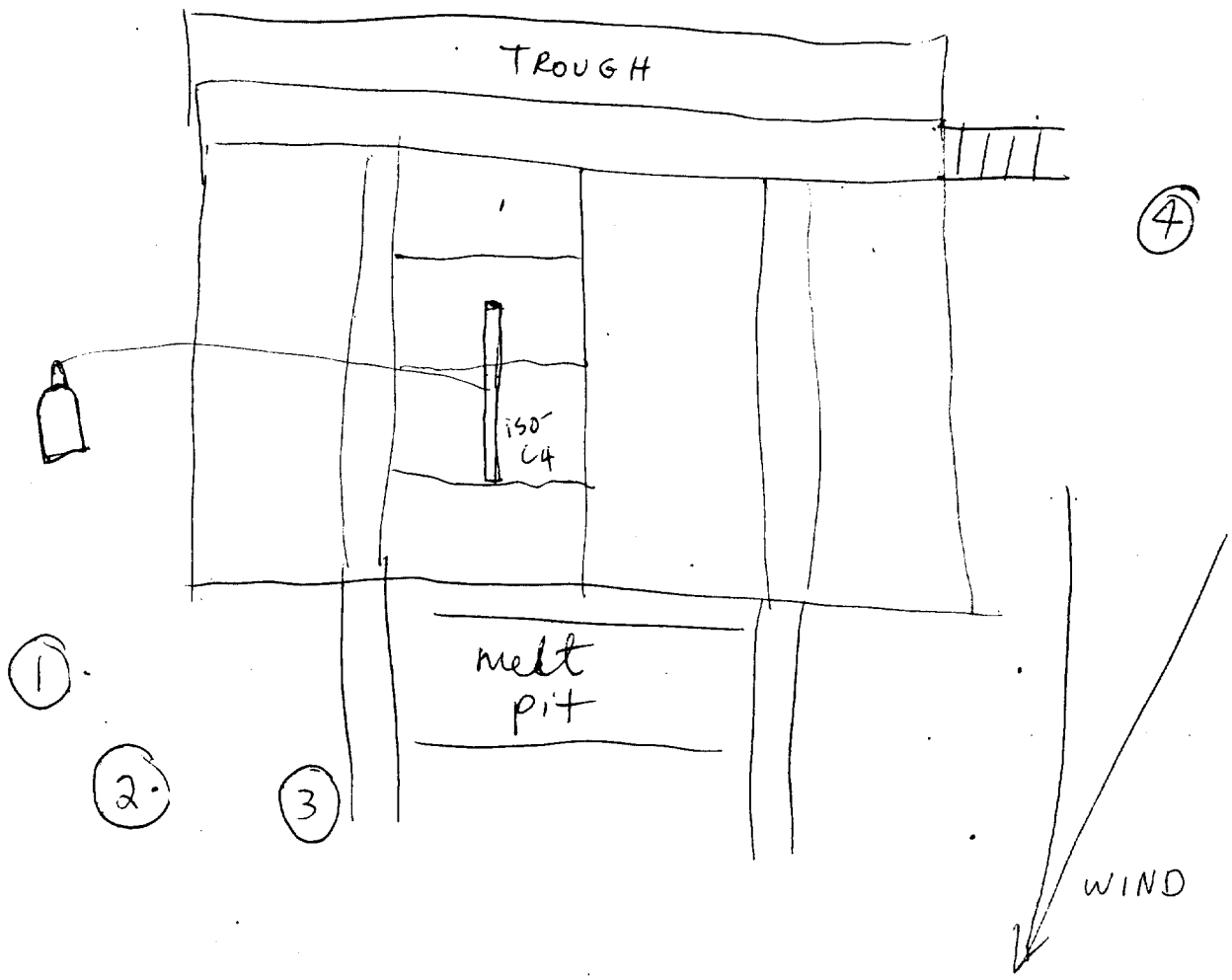
Process Description:

RUN 2

Plant Bethlehem St. Bethlehem Process Denver Float Unit Date 7/8/80

Sketch of Process:

Include dimensions and flow directions.



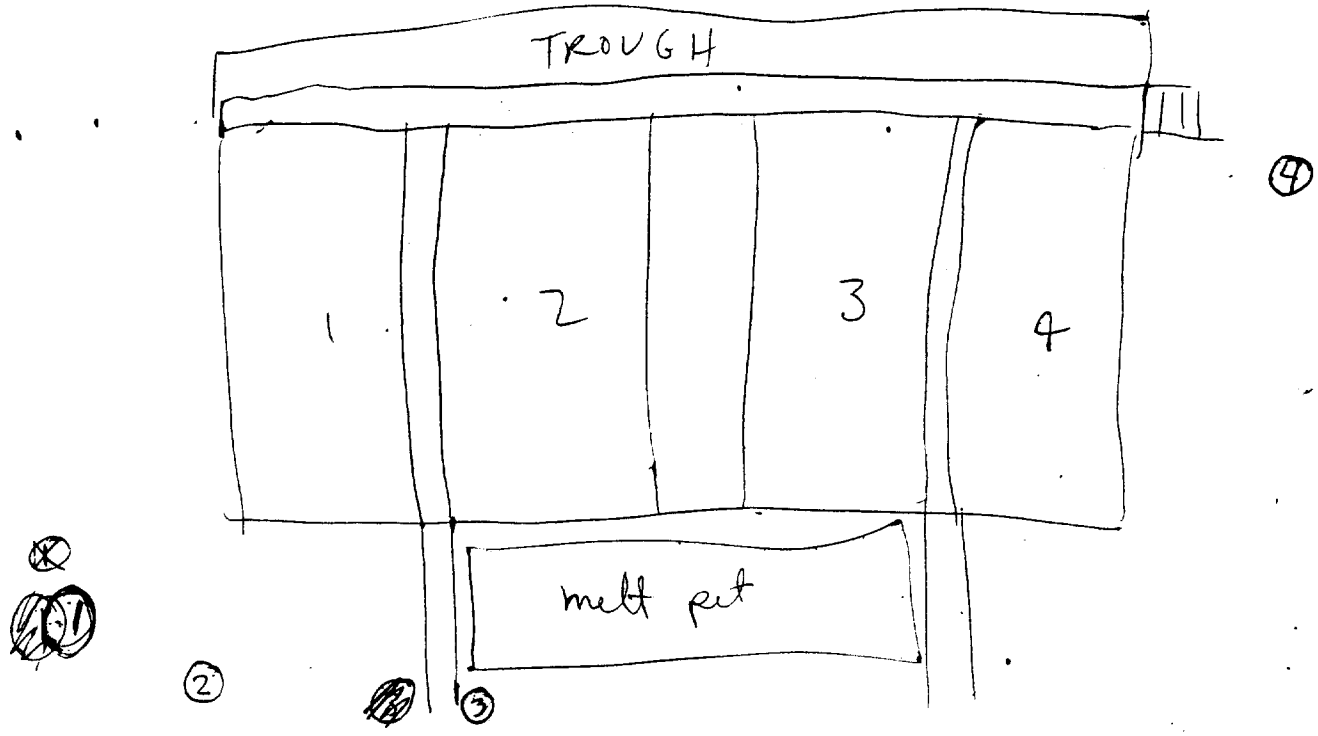
Process Description:

Rm #3

Plant Beth Steel - Bethlehem Process Denver Float Unit Date 7/15/80

Sketch of Process:

Include dimensions and flow directions.



Process Description:

Units 1, 3, and 4 working

PROJECT 1906 BENZENE/BaP PRESURVEY

SAMPLE DATA

Plant Beth Steel Beth. Process Denver Unit Date 7/15/80

Sample No. Trough next to Denver Unit Time Sampled 10:50

Sample Type: (Liquid) Air

During tracer test #1

Sample Temperature 38 °C

Ambient Temperature 30 °C

Description of Sampling Location: open Trough in ground next to Denver Unit

Sample No. Denver Unit Section #1 Time Sampled 10:52

Sample Type: (Liquid) Air

Sample Temperature 36 °C

Ambient Temperature 30 °C

Description of Sampling Location:

Sample No. Denver Unit Section #2 Time Sampled 10:53

Sample Type: (Liquid) Air

Sample Temperature 35 °C

Sec. 2 not operating at time of sample

Ambient Temperature ~30 °C

Description of Sampling Location:

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TEST 1 RUN 1

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Naphthalene melt

PROCESS NOTES:

melt started 7:50 am (steam started)

DATE: 7/15/80

WIND SPEED: SSW

WIND DIRECTION:

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	10 ft		10 ft		10 ft		10 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers	~~6~~ 4		3		3		8	
Tedlar bag numbers	5		2		3		4	
Start Time	8:00		8:08		8:00			
Stop Time								

ISOBUTANE RELEASE: Gas Temperature

TIME	METERED VOLUME	
8:00 0	89.900	
2	90.095	
4	90.362	
6	90.629	
8	90.890	
10	91.154	.125
12	91.418	
14		
16	91.935	
18	92.190	
20	92.445	.129
22	92.698	
24	92.949	
26	93.200	
28	93.463	
30	93.746	.129

Gas Pressure

TIME	METERED VOLUME
cake begins to melt	
cake ~1/3 melted	
really starting to steam	
cake still 1/2 melted	
looks fully melted	
changed drum 1 over to pump 4	
drum 1 weak	
too much steam to see pit	

3.84 ft³ / 30 min = .128 ft³/min overall

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TEST 1 RUN 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Naphthalene melt

PROCESS NOTES:
melt on 8:00
Salt in 8:30

DATE: 7/15/80

WIND SPEED:

WIND DIRECTION:

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	6 ft		6 ft		6 ft		6 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers	4		3		~~3~~ 1			
Tedlar bag numbers	13		34		64		3	
Start Time								
Stop Time								

ISOBUTANE RELEASE: Gas Temperature

TIME	METERED VOLUME
8:37 0	95.000 ft^3
2	95.265
4	95.528
6	95.790
8	96.050 .131
10	96.308
12	96.560
14	96.811
16	97.062
18	97.315 .126
20	97.569
22	97.827
24	98.095
26	98.365
28	98.630 .131
30	98.890

3.890 ft^3/30 min

0.129 ft^3/min overall

Gas Pressure

TIME	METERED VOLUME
too much steam to see melt pit	
plume straight up	
plume on ground	
plume on ground	
can #3 switched to pump #1	
can #3 not doing too well - hour 10	

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TEST 2

RUNS 1 & 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel - Bethlehem

PROCESS: [illegible]

PROCESS NOTES:

Unit started ~7.00

Salt dumped ~8:04

NOTE: Barrels 1 and 2 are more in the plume than #3

DATE: 7-6-80

WIND SPEED:

WIND DIRECTION: S-SW

AMBIENT TEMPERATURE: ~75°F

BAROMETRIC PRESSURE: 29.57

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~10 ft							
Sampling Rate	1/10 lpm							
Pump Numbers	6		3		5		8	
Tedlar bag numbers	9	13	22	17	23	21	4	10
Start Time	7.30	8:09	7.30	8:09	7.30	8:09	7.30	8:09
Stop Time								

meter dropped between Runs 1 & 2

Meter Y=1.059

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME ft³
7.30	0	131.83
	2	132.113
	4	132.394
	6	132.659
	8	132.934
	10	.137 133.201
	12	133.467
	14	133.735
	16	
	18	134.263
	20	.132 134.521
	22	134.789
	24	135.060
	26	135.327
	28	135.591
	30	.133 135.855

4.025 ft³/30 min = 0.1342

x 1.059 = 0.142 ft³/min

Gas Pressure

TIME		ft³	METERED VOLUME
8:09	0		137.050
	2		137.329
	4		137.608
	6		137.885
	8		138.157
	10	.138	138.435
	12		138.705
	14		138.975
	16		139.249
	18		139.518
	20	.135	139.788
	22		140.065
	24		140.330
	26		140.610
	28		140.880
	30		141.145

4.095 ft³/30 min = 0.1365 ft³/min

x 1.059 = 0.1445 ft³/min

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 3

RUNS 1 & 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Naphthalene melt pit

PROCESS NOTES:

Denver units 2,3,4 operating

NOTE: raining, wind not steady

DATE: 7/17/80

WIND SPEED: variable

WIND DIRECTION: variable

AMBIENT TEMPERATURE: ~75 °F

BAROMETRIC PRESSURE: 29.51

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~8 ft		8 ft		8 ft		10 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers	6	6	3	3	3	3	8	8
Tedlar bag numbers	2	8	3	10	5	34	6	18
Start Time								
Stop Time								

WIND WRONG

Meter Y = 1.059

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME ft³
7:15	0	155.30
	2	155.567
	4	155.828
	6	156.09
	8	156.352
	10	156.875
	12	157.139
	14	157.407 – steam starting to show
	16	157.680
	18	157.948
	20	158.210
	22	158.470
	24	
	26	
	28	158.998
	30	159.258

Gas Pressure

TIME		ft³ METERED VOLUME	
8:18	0	160.05	wind good
	2	160.320	"
	4	160.578	"
	6	160.842	"
	8	161.106	"
	10	161.372	"
	12	161.634	"
	14	161.890	bad
	16	162.149	OK
	18	162.41	bad
	20	162.675	"
	22	162.938	"
	24	163.200	good
	26	163.459	bad
	28	163.727	OK
	30	163.950	good

3.958 ft³/30min = .1319

× 1.059 = 0.1397 ft³/min

3.90 ft³/30min = 0.130

× 1.059 = 0.1376 ft³/min

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TEST 4
RUNS 1 & 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel

PROCESS: Naphthalene melt

PROCESS NOTES:
melt started ~ 7:25
Denver units 1,2,3 up
salt dumped ~8:30

DATE: 7/18/80

WIND SPEED: ~~8~~ 8 & steady

WIND DIRECTION: N

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	2 ft		2 ft		2 ft		25 ft	
Sampling Rate	10 lph		10 lph		10 lph		10 lph	
Pump Numbers								
Tedlar bag numbers	9	14	16	19	17	34	23	22
Start Time	7:36	8:30	7:36	8:30	7:36	8:30	7:36	8:30
Stop Time								

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME	
7:36	0		175.800
	2		
	4		176.332
	6	steam visible	176.607
	8		176.876
	10	.134	177.141
	12		177.403
	14		177.668
	16		177.950
	18		178.231
	20	.137	178.513
	22	wind shifts S	
	24		179.062
	26		179.329
	28	wind N	179.592
	30		179.855

Gas Pressure

TIME		METERED VOLUME	
8:30	0		182.100
	2		182.373
wind	4		182.639
good	6		182.904
whole	8		183.175
time	10	.134	183.447
	12		183.702
	14		183.988
	16		184.264
	18		184.540
	20	.137	184.817
	22		185.084
	24		185.356
	26		185.627
	28		185.890
	30		186.153

Samplers are between melt pit & Denver units

4.055 ft³/30min = 0.135 ft³/min
x 1.059 = 0.143 ft³/min

4.053 ft³/30min = 0.135 ft³/min
x 1.059 = 0.143 ft³/min

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 1
RUNS 1 & 2

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Background for melt pit

PROCESS NOTES:
tracer manifold on melt pit
Denver units 1, 2, 3 operating
melt pit just starting to fill

DATE: 7/17/80

WIND SPEED: light

WIND DIRECTION: steady SSW - occasional swing

AMBIENT TEMPERATURE:

BAROMETRIC PRESSURE:

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	8 ft		8 ft		8 ft		10 ft	
Sampling Rate								
Pump Numbers	6		3		3		8	
Tedlar bag numbers	12	4	13	15	14	21	23	22
Start Time	11:22		11:19		11:19		11:20	
Stop Time								

Barrels are ~6 ft from melt and ~15 ft from Denver unit

ISOBUTANE RELEASE: Gas Temperature

TIME			METERED VOLUME
11:19	0		166.300
	2		
	4		
	6		167.092
	8		167.352
	10	.132	167.623 - Steam on
	12		167.887
	14		
	16		168.411
	18		168.670
	20	.131	168.937
	22		~~169.075~~
	24		169.494
	26		169.763
	28		170.030
	30		170.300

Gas Pressure

TIME			METERED VOLUME
13:07	0		170.700
	2		170.971
	4		Steam on to clean lines
	6		171.514
	8		171.777
	10	.135	172.050
	12		172.317
	14		172.575
	16		172.829
	18		173.103
	20		
	22		173.638
	24		173.898
	26		174.177
	28		174.447
	30		174.709

Steam on to clean lines for ~4 min

$4.000\ ft^3/30\ min = 0.133$

$\times 1.059 = 0.1412\ ft^3/min$

$4.009\ ft^3/30\ min = 0.1336$

$\times 1.059 = 0.1415\ ft^3/min$

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.

TEST 1
RUNS 1 &
VOID

PROJECT 1922

TRACER GAS DATA SHEET

PLANT: Bethlehem Steel, Bethlehem

PROCESS: Denver unit / naphth. melt

PROCESS NOTES:
Background for melt pit data
Drums & trace manifold in positions same as for melt

DATE: 7/16/80

WIND SPEED:

WIND DIRECTION: S - SW

AMBIENT TEMPERATURE: ~80 °F

BAROMETRIC PRESSURE: 29.57

Units 2, 3, 4 operating

	DOWNWIND						UPWIND	
Sampler Number	1		2		3		4	
Distance from Source	~8 ft		8 ft		8 ft		10 ft	
Sampling Rate	10 lph →							
Pump Numbers	6						8	
Tedlar bag numbers	12	7	14	11	15	19	16	20
Start Time	10:28	11.08	10:28	11.08	10:28	11.08	10:29	11:08
Stop Time								

Melt pit is empty at 0

Meter Y = 1.059

ISOBUTANE RELEASE: Gas Temperature

TIME		METERED VOLUME LL3
10:28	0	141.850
	2	
	4	142.370
	6	142.657
	8	142.947
	10	.137 143.219
	12	143.495
	14	143.762
	16	144.028
	18	144.292
	20	.134 144.557
	22	144.840
	24	145.134
	26	145.425
	28	145.719
	30	146.014

Gas Pressure melt pit empty at 2cm

TIME		METERED VOLUME
11.08	0	147.450
	2	147.735
	4	148.021
	6	148.310
	8	148.698
	10	
	12	149.167
	14	149.447
	16	149.714
	18	149.992
	20	150.270
	22	
	24	150.875
	26	
	28	151.490
	30	151.763

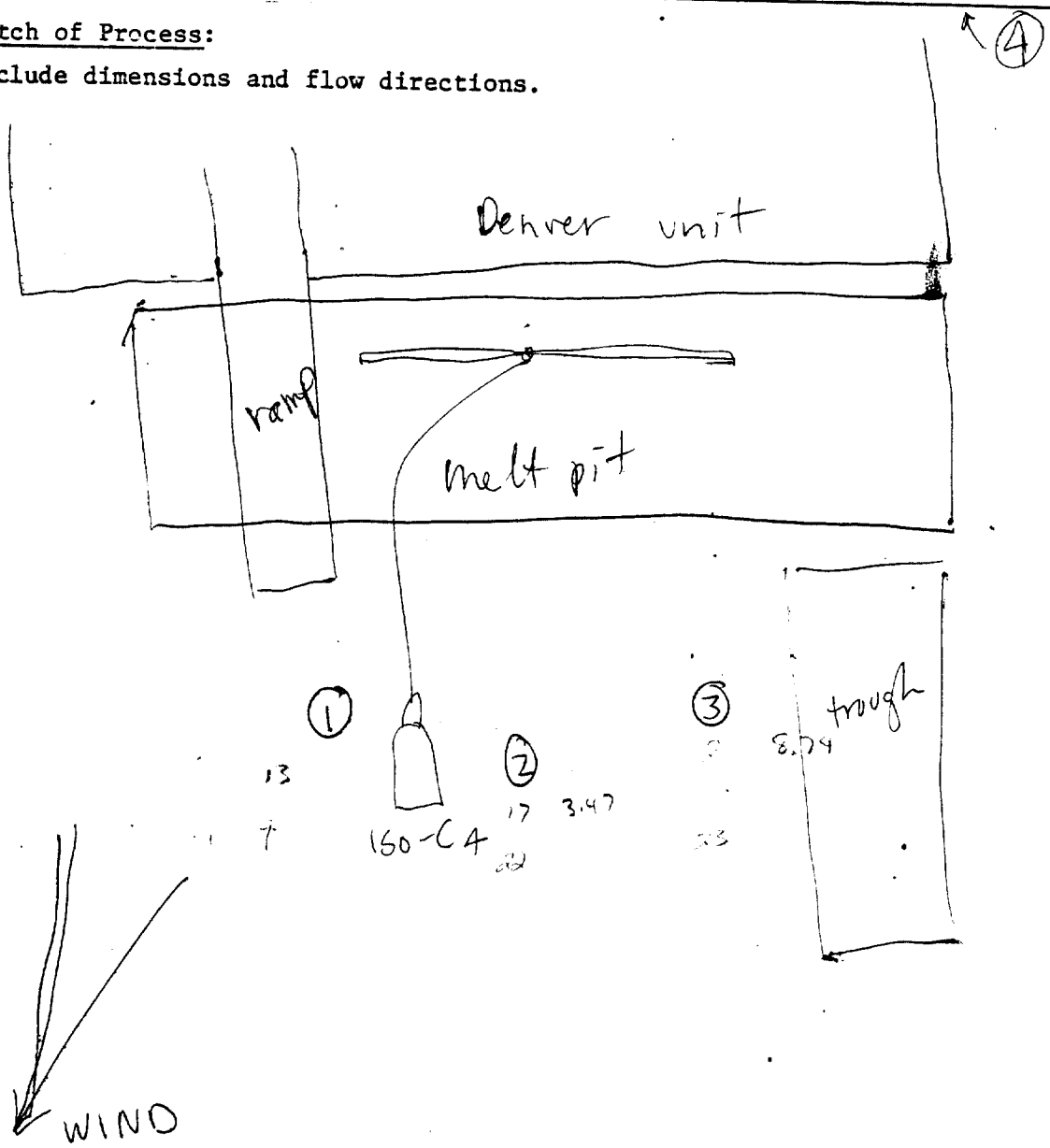
Rotameter is leaking isobutane
melt pit is filling at end

RUNS 1 & 2

Plant Bethlehem Process melt pit Date

Sketch of Process:

Include dimensions and flow directions.



Process Description:

APPENDIX C

LABORATORY DATA SHEETS

.092 - .64x
.023 - 1x

CHROMA[illegible]C ANALYSIS LOG

Bethlehem Steel [illegible]

Project No. [illegible] Date [illegible] Analyst [illegible] (1)

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Tracer Gas Dense Float Unit Run #				1/[illegible]
	(2) Bag # 04	ø - 55 @ 128x [illegible] - 55.75 @ 32x	.246 .048	8.61 ppm ø 1.72 ppm	671.6
	(1) Bag # 13	ø - 28.5 @ 64x [illegible] 10.5 @ 32x	.123 .048	3.51 ppm ø .504 ppm	273.8 9.38
	(3) Bag # 3	ø - 59 @ 128 [illegible] - 42 @ 32x	.246 .048	14.51 ppm ø 2.02 ppm	1132.1 9.[illegible] 117.2
	(Approx) Bag # 9	3 - 8 @ 8x [illegible] ND	.015	0.10 ppm ø	
	Bag # 2				
	(1) Bag # 14	ø - 47.25 - 32x [illegible] - 12.25 - 8x	.062 .012	3.03 ppm ø 0.15 ppm	[illegible]3 = 27.2 8.7
	(2) Bag # 23	ø - 1[illegible].25 - 64x [illegible] - 16.75 - 16x	.123 .024	7.69 ppm ø 0.40 ppm	599.8 = 25.9 23.2
	(3) Bag # 34	ø - 67.5 - 128x [illegible] - 23.5 - 32x	.246 .048	.61 ø 1.13 ppm	1295.6 65.4
	[illegible] Bag # 22	ø - 17 @ 8x [illegible] - ND	.015	.72 ppm ø	

ø - .123 @ .64x

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CHROMATOGRAPHIC ANALYSIS LOG Bethlehem Steel

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Iso C_4 - 4. Std 6.11 ϕ @ 64x10⁴	66	.0125 ppm/div		
	ϕ Std 1225 ppm 1024x10⁴	83.5	1.47 ppm/div ([illegible])		
	Amlet Cyl #1 4 @ 64x10⁴	~~[illegible]~~ 84.3	.0821 ppm/div	7.76 ppm	Collective 1.66 ppm
	Amlet Cyl #2 4 1024x10⁴	91.3	1.47 ppm/div	134.21 ppm	Collective [illegible] ppm

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	[illegible]	77.5	18.625	1447.2 [illegible]	
	[illegible]	57.5	[illegible]		
	[illegible]	51	[illegible]	950.2 [illegible]	
	[illegible]	[illegible]	[illegible]	742.2 [illegible]	

CHROMATOGRAPHIC ANALYSIS LOG Bethlehem Steel Co.

Project No. 1922 Date 7-9-[illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
8:15 PM	System Background	5 @ 16x10[illegible]	.031	0.15 ppm	
	Bag #4 Background	66.5 @ 32x	.062	4.13 ppm	
	Bag #7 "	33.5 @ 8x	.015	0.52 ppm	
	Bag #8 "	61.5 @ 32x	.062	3.81 ppm	
10:00	Std 122.5 ppm ϕ	8[illegible]5 @ 10x4			
	Indet Cyl #2	88.5 @ 10x4	1.52	134.67 ppm	
	Tar Decanter [illegible] #2 Dil 10x	37.5 @ 128x10[illegible]	1.91	7178 ppm x10	[illegible]
				7178 ppm	
	Std 122.5 ppm ϕ 128x10[illegible]	64 @ 128x10[illegible]	1.91		

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CHROMATOGRAPHIC ANALYSIS LOG [illegible]

Project No. 1922 Date 7-16 [illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	[illegible] 512	77.5	1.58		[illegible]
[illegible]	[illegible] Dil 10x @ [illegible]	62.5 ([illegible])	~~[illegible]~~ 3.16	1975 ~~[illegible]~~ ppm	~~[illegible]~~
[illegible]	Bag # [illegible]	10	~~.098~~ .098	.098 ~~[illegible]~~ ppm	→ System [illegible]
12 30	Bag # 11 [illegible]	63.15 ([illegible])	~~.098~~ .197	12.50 ~~[illegible]~~ ppm	[illegible]
12 42	System [illegible] @ [illegible]	2	~~.098~~ .197	.394 0 ~~.196~~ ppm	[illegible]
	10 l Bag # 3 [illegible]	2	~~.098~~ .197	.394 ~~[illegible]~~ ppm	[illegible]
	10 l Bag # 22 "	3.5	~~.098~~ .197	.393 0 ~~.393~~ ppm	[illegible]
	10 l Bag # 9 "	1.5	~~.098~~ .197	.296 0 ~~.197~~ ppm	
	10 l Bag # 34 "	1.5	~~.098~~ .197	.296 0 ~~.197~~	

CHROMATOGRAPHIC ANALYSIS LOG [illegible]

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	[illegible] ppm [illegible]	[illegible]	1.65		
	[illegible] #2 [illegible]	700 [illegible]	~~.105~~ .206	[illegible] ppm ~~[illegible]~~ ppm	
	[illegible]	1	~~.105~~ .206	~~.105~~ ppm	
	[illegible] #14 [illegible]	1	~~.105~~ .206	.105 ppm	
	[illegible] #[illegible] "	1	~~.105~~ .206	.105 ppm	
	[illegible]	1	~~.105~~ .206	.105 ppm	
	[illegible]	1	~~.105~~ .206	.105 ppm	
[illegible]	[illegible] ppm [illegible]	745			
	[illegible] #3 [illegible]	67.75 [illegible]	~~.105~~ ~~.105~~ .206	14.27 ppm ~~[illegible]~~ ppm	
	[illegible] 10x [illegible] #1	46 [illegible]	~~1.65~~ 3.30	151.8 ppm ~~[illegible]~~ x10	~~[illegible]~~ ppm [illegible]
	[illegible] #2	57.5	~~1.65~~ 3.30	18.75 ppm ~~[illegible]~~ x10	~~[illegible]~~ ppm 18.975 ppm

C [illegible] x10⁴

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]
[illegible]	[illegible]	69 [illegible]	[illegible]	14.26 [illegible]	
	[illegible]	73.25 [illegible]	[illegible]	15.16 [illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	
	[illegible]	59.25 [illegible]	[illegible]		

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-11-80 Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
12.00	[illegible]				[illegible]
	1000 x - Dil #1	11 @ 236 [illegible] 22 @ 125	8.26 4.13	90.86 90.86 x1000	90860 ppm
	100 x Dil #2 @ 125 x 10³	225 225 225	4.13	92.93 x1000	92930 ppm
					91895 ppm
1.15	[illegible] Vent [illegible] 126 x 10³				
	1000 x - Dil #4	25.25 25.5 25	4.13	104.28 x1000	104280 ppm
	1000 x Dil #2	28	4.13	115.64 x1000	115640 ppm
	[illegible] Dil #3	2.05	4.13	109.45 x1000	109450 ppm
	[illegible] Vent [illegible] @ 64 x 10³				
	1000 x Dil #1	27 27 27	2.07	55.84 x1000	55870 ppm
	1000 x Dil #2	24.5	2.07	50.72 x1000	50720 ppm

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-11-86 Analyst TB

PM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Septa [illegible]	5.5	.207	1.14	
5:36	Big #8 [illegible]	54	1.47	79.38	
	Room #4 [illegible]				
	Room Background				
	[illegible]	48	.099	4.73 ppm	
	" " "	—	—		
	Front Seat [illegible]	48	.394	18.91	
	" "	45	.394	[illegible]	
	AC Inlet [illegible]	34.5	.394	13.59	
	" " "	29	"	1.43	
	[illegible]	1	"	.399	
	AC Inlet "	20	"	7.88	
	[illegible] "	23.5	"	9.[illegible]	
	Front Seat "	18	"	7.09	

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-11-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Sit Oil Condensed Vent Run #4 [illegible]				
	Dil #1 1000x	26.5	4/13	109.45	109450 ppm
	Dil #2 1000x	27	4/13	111.51	111510 ppm
	122.5 ppm Std 64x10³	58	2/11		

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. M22 Date 7-14-80 Analyst TB Bethlehem Pa

AM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	122.5 ppm ø Std 512 x 10^4	76.2 15 11 [illegible] [illegible] 70	1.61 (.201)		
	6.11 ppm ø Std 64 x 10^4	27.5 [illegible] 32	.222 (.212 ave)		
	Ball Mill Gab Sample 128 x 10^4 Vent Flask # 19	8 85 75	.424	3.40 ppm	
	Flask # 17	13.5	.424	5.72 ppm	
PM	100 l Bag # 7 Bk 128 x 10^4	3	.424	1.27 ppm	
	100 l Bag # 8 Bk 128 x 10^4	33	.424	13.99 ppm	
	100 l Bag # 12 Bk 128 x 10^4	40	.424	16.96 ppm	

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7/1/90 Analyst TP

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
PM	~~100 l Bag #12 Bk 128x10⁴~~	~~[illegible]~~ 40	~~.424~~	~~5.512~~	
	100 l Bag #4 Bk 128x10⁴	13	.424	5.512	
	Gib @ Denver Float Tan Tan 128x10⁴	9	.424	3.82	
	Bag #11 - North Storage Tank - 128x10⁴	72	.424	30.53	
	Oil 10x 128x10⁴	75	.424	3.18x10 31.8	
	Bag #3 - North Storage ~~[illegible]~~ 512x10⁴	565	161	0.77	

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-15-80 Analyst TB

AM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
9:00	6.11 ppm ø Std 32x10⁴	47.3	.129			
	1.06 ppm iso C4. 16x10⁴	16.9	.062			
	~~Bag # 01 Test #~~					
10:34	Melt Pit Tracer Run # 2 - Test # 1	~~[illegible]~~				ø/iso4
	Bag # 04 - isobutane - 6x	15.75	.062	0.977 ppm	x 58 = 56.61	13.01
	- ø - 32x	73.35	.129	9.45 ppm	x 78 = 737.10	
	Bag # 13 - iso C4 - 16x	21.25	.062	1.32 ppm	x58 = 76.45	13.75
	ø - 64x	52.25	.258	13.48 ppm	x78 = 1054.50	
	Bag # 34 - iso C4 - 16x	24	.062	1.49 ppm	x58 = 86.42	13.45
	ø - 64x	57.75	.258	14.90 ppm	x78 = 1162.16	
	Bag # 5 Upwind C.					
	iso C4 - 16x	ND	—	—		
	ø - 16x	16	.065	1.03 ppm		

CHROMATOGRAPHIC ANALYSIS LOG

[illegible]

Project No. [illegible] Date [illegible] Analyst [illegible]

PM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	[illegible] Test #				4/[illegible]
	Run # 1				
	Bag # 5 - iso C4 - 16x	11.5	.062	0.713 ppm	x58 = 41.35 21.66
	∅ - 32x	89	.129	11.48 ppm	x78 = 895.52
	Std. 6.11 ppm ∅ 32x	46			
	1.06 ppm iC4 16x	16.5			
	Bag # 4 - Upwind				
	iso C4 - 16x	ND	—	—	
	∅ - 32x	55	.129	0.71 ppm	
	Bag # 2. iso C4 - 16x	16.5	.062	~~17.54~~ .102	x58 ~~[illegible]~~ 59.33 23.66
	∅ - 64x	68	~~.129~~ .258	17.54 ppm	x78 = ~~57.33~~ 1368.43
	Bag # 3 iso C4 16x	15	.062	0.93 ppm	x58 = 53.94 23.97
	∅ 64x	64.25	~~.129~~ .258	16.58 ppm	x78 = 1292.97

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date [illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Dense Cloud Test #2 Run #1				¢/iC4
	Bag #6				
	IsoC4 - 16x	16.5	.062	1.02 ppm	x58 = 59.16
	¢ - 32x	40	.129	5.16 ppm	x78 = 402.48
					6.80
	Bag #7				
	IsoC4 - 16x	17.5	.062	1.09 ppm	x58 = 63.22
	¢ - 32x	42	.129	5.42 ppm	x78 = 422.60
					6.68
	Bag #8				32G
	IsoC4 - 16x	18.75	.062	1.16 ppm	x58 = 67.43
	¢ - 64x	73.5	.258	18.96 ppm	x78 = 1479.11
					21.94
					higher like hydrocarbons than others from melt pit
	Bag #9				
	IsoC4 - 16x	ND	–	–	
	¢ - 16x	115	.065	0.75 ppm	

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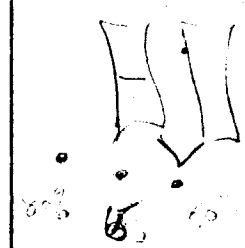
CHROMATOGRAPHIC ANALYSIS LOG

Project No. 922 Date 7-15-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments		
PM ~3:00	Denver Float Test #2 Run #2					ϕ/CO_2	#
	Bag #10 Iso-C_4 - 8x	6.5	.031	0.202 ppm	x58 = 11.72	29.42	1
	ϕ - 16x	68	.005	442 ppm	x78 = 344.16		
	Bag #11 Iso-C_4 - 8x	15.75	.031	0.488 ppm	x58 = 28.32	15.63	2
	ϕ - 32x	44	.124	5.68 ppm	x78 = 442.73		
~4:15	Bag #12 Iso-C_4 8x	77.5	.031	2.40 ppm	x58 = 139.35	5.33	3
	ϕ - 64x	71.5	.258	18.45 ppm	x78 = 1438.87		
	Std 6.11 ppm ϕ 32x	49					
	1.06 ppm CO_2 16x	175					
	Bag #13 Ground 100° Iso	6.5	.10	0.65			



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CHROMATOGRAPHIC ANALYSIS LOG

Bethlehem Pa

Project No. 1922 Date 7-15-80 Analyst TB

PM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments		X_{10}#
~~[illegible]~~	Denver Float Unit Test #3 Run #1					6/ic4	
4:36	Bag #14						
	$SaCl_4$ - 8x	7	.031	0.22 ppm	x58 = 12.76	43.57	1
	∅ - 32x	55.25	.129	7.13 ppm	x78 = 555.93	52%	
	Bag #15 $SaCl_4$ - 8x	16	.031	0.496 ppm	x58 = 28.77	22.75	2
	∅ - 32x	65	.129	8.39 ppm	x78 = 654.42	42%	
	Bag #16 $SaCl_4$ - 8x	64.0	.031	1.97 ppm	x58 = 114.26	9.55	3
	∅ - 64x	54.25	.258	14.0 ppm	x78 = 1091.73		
	Std 1.06 ppm ic_4 - 16x	17					
	6.11 ppm ∅ - 32x	49					
	Bag #17 Upward 100° Dao	6	0.1	0.60 ppm			

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CHROMATO... ...IC ANALYSIS LOG

Project No. [illegible] Date 7-5-80 Analyst TB

BM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
6:00	Denver Float Unit					1
	Test #3 Run #2					
	Bag #18					
	AroC4 - 8x	~~40.5~~ 30.5	.031	0.45 ppm	x58 = 54.84	9.45
	5 - ~~64x~~ 32x	51.5	~~[illegible]~~ .129	6.64 ppm	x78 = 518.19	
	Bag #19					2
	AroC4 - 8x	45.5	.031	1.20 ppm	x58 = 72.82	7.60
	4 - 32x	55.5	.129	7.16 ppm	x78 = 558.44	
7:22	Bag #20					3
	AroC4 - 8x	40	.031	1.24	x58 = 71.92	14.83
	4 - 64x	53	.058	13.67	x78 = 1060.57	
	Std					
	100 ppm AC4	165				
	6.11 ppm 4	48				
	Bag #21 Ground	4.5	0.1	0.45 ppm		
	100° Aro					

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date 7-16-[illegible] Analyst [illegible]

AM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
	Std 6.11 ppm ∅ 32x10⁴	48	0.127			
	1.06 ppm iC_4 16x10⁴	18.2	0.058			
	Naph Melt Pot Test #2 Run #1					
	Bag #9 iC_4 - 8x	~~59~~ 43	.029	1.25 ppm	x58 = 72.33	16.16 1
	∅ - 64x	59	.254	14.99 ppm	x78 = 1168.91	
	Bag #22 iC_4 8x	57.5	.029	1.52 ppm	x58 = 88.31	13.41 2
	∅ - 64x	59.75	.254	15.18 ppm	x78 = 1184.04	
	Bag #23 iC_4 - 8x	26	.029	.754 ppm	x58 = 43.73	18.24 3
	∅ - 64x	40.25	.254	10.22 ppm	x78 = 797.43	
	Bag #4 iC_4	ND	—	—		
	∅ - 64x	4	.254	1.02 ppm		
	Std 1.06 ppm iC_4	18				
	6.11 ppm ∅	45.5				

CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	10 l Bag Background 100° Dia				
	Bag # 19 @ 52 x 10^4	ND	0.10		
2 24	Bag # 20	ND			
	Bag # 7	ND			
	Bag # 11	ND			
	Bag # 16	ND			
	Bag # 14	ND			
	Bag # 12	ND			
	Bag # 15	3	.1	.3 ppm	
	6.1 ppm Std 100° Dia 33 vol	61	.1		

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-16-80 Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments		
12:45	Std 6.11 ppm Ø	51					
	1.06 ppm CC_4	18					
	North Mult Pot. Test #2 Run #2						
	Bag #10 Upwind						
	CC_4 - 4x	ND	—	—			
	Ø - 32x	6	.129	0.774 ppm			
	Bag #7 CC_4 - 8x	73.25	.029	2.12 ppm	x58 = 123.21	3.47	2
	Ø - 32x	42.5	.129	5.48 ppm	x78 = 427.69		
	Bag #21 CC_4 - 8x	48	.029	1.39 ppm	x58 = 80.74	8.57	3
	Ø - 32x	68.75	.129	8.87 ppm	x78 = 691.76		
3:49	Bag #13 - CC_4 - 8x	53	.029	1.54 ppm	x58 = 89.45	9.93	1
	Ø - 64x	44	.258	11.35 ppm	x78 = 885.46		
	Std 6.11 ppm Ø - 32x	50					
	1.06 ppm CC_4 - 16x	175					

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CHROMATOGRAPHIC ANALYSIS LOG

Bethlehem

Project No. 1922 Date 7-17 [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
[illegible]	Std 6.1 ppm + 3R [illegible]	60.5	0.100		
	Bag # 17 Background	2.5	.25 ppm →		
	Bag # 16 "	ND	—		
	Bag # 9 "	2	.20 ppm →		
	Bag # 21	1	.10 ppm		
	Bag # 22 "	1.5	.15 ppm		
	Bag # 15	2.5	.25 ppm		
	Bag # 12	3.0	.30 ppm		
	Bag # 14	3.0	0.10	.3 ppm	
8:17	Bag # 23	1.0		.1 ppm	
	Bag # 13	1.0		.1 ppm	

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-17-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Std 6.11 ppm ∅ 32x10⁴	50	0.122		Raining
	106 ppm K_4 - 8x10⁴	8.67	0.57		
	Melt Pit Test #3 Run #1				Dro Cyanate are 0.1397 ft³/min
	Bag #2 K_4 - 8x	27.5	0.29	0.80 ppm	x55 - 46.40 1/03
	∅ - 32x	53.75	.122	6.56 ppm	x78 - 511.68
	Bag #3 K_4 - 8x	48.75	0.029	1.41 ppm	x55 - 81.78 6.54
	∅ - 32x	56.35	.122	6.80 ppm	x78 - 535.08
	Bag #5 K_4 - 8x	61.5	0.029	1.78 ppm	x55 - 103.24 5.14
	∅ - 32x	55.75	.122	6.80 ppm	x78 - 530.4
	Bag #6 K_4 - 8x	No.	—		
	∅ - 32x	4	.122	0.49 ppm	
	Std 6.11 ppm ∅ 32x10⁴	51			
	106 ppm K_4 16x	18.5			

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-17-[illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments		
[illegible]	Naph Mill Pit Test #3 Run #2				[illegible]		
	Bag #8 wy-8x	9	.029	0.261 ppm	x58 = 15.14	27.18	1
	∅ - 32x	43.25	.122	5.28 ppm	x78 = 411.57		
	Bag #10 wy-8x	14.75	.029	0.430 ppm	~~[illegible]~~ = 24.[illegible]	17.64	2
	∅ - 32x	46	.122	~~[illegible]~~ 5.61 ppm	x78 = 437.74		
	Bag #34 wy-8x	14.5	.029	0.421 ppm	x58 = 24.39	19.71	3
	∅ - 32x	50.5	.122	6.16 ppm	x78 = 480.56		
	Bag #18 Upward						
	wy-8x	NO	~~NO~~				
	∅ - 32x	5	.122	0.61 ppm			

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1322 Date 7-17-80 Analyst TB

PM Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
	Std 6.11 ppm ϕ - 32x	55.5	0.111			
	1.06 ppm IC_4 - 8x	37.5	0.028			
	Tracer Background @ Melt Pit for Denver Unit Test # Run #					
	Bag #12 IC_4 - 16x	51	.059	3.01 ppm	x58 174.52	6.55 1
	ϕ - 64x	66	.222	14.65 ppm	x78 1142.87	
	Bag #13 IC_4 - 16x	66	.059	3.89 ppm	x58 225.85	5.25 2
3:10	ϕ - 64x	68.5	.222	15.21 ppm	x78 1186.5	
	Bag #14 IC_4 - 16x	34.5	.059	2.04 ppm	x58 118.06	6.42 3
	ϕ - 64x	43.75	.222	9.71 ppm	x78 757.38	
	Bag #23 Ground IC_4 - 5x	No				
	ϕ - 32x	5	.111	0.56 ppm	x78 43.68	

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-17-80 Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
	Std 6.11 ppm ϕ 32x	54.5				
	1.06 ppm K_4 16x	19				
	Tracer Background for Melt Pit @ Denver Float Unit Test #1, Run #2					
	Bag #4 K_4 - 16x	25.75	0.059	1.52 ppm	x58 = 88.16	4.93
	ϕ - 64x	60.75	0.222	13.49 ppm	x78 = 105.95	
	Bag #15 K_4 - 32x	~~.118~~ 30	.118	3.54 ppm	x58 = 205.32	6.01
	ϕ - 64x	~~.222~~ 71.25	.222	15.82 ppm	x78 = 1233.77	
	Bag #21 K_4 - 32x	31.75	.118	3.75 ppm	x58 = 217.50	4.82
	ϕ - 64x	60.5	.222	13.43 ppm	x78 = 1047.62	
	Bag #22 K_4 - 8x	[illegible]				
	ϕ - 32x	2.5	.111	.25 ppm	x78 = 19.50	
	Std 6.11 ppm ϕ - 32x	50				
	1.06 ppm K_4 - 16x	18				

1

2

3

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CHROMATOGRAPHIC ANALYSIS LOG

[illegible]

Project No. 1922 Date 7-18-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Std 6.11 ppm ø 32x	52			
	1.06 ppm ice 16x				
	100° Iso				
	Bag Backgrounds				
	Bag # 19	2.5			
	Bag # 14	3			
	Bag # 6	6.5			
	Bag # 34	4			
	Std 6.11 ppm ø 32x	52			
	1.06 ppm ice 16x	19			
	Temp Prog.				

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-18-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments	
	Zapata Malt Pit Run #1 Test #4					
	Bag #9 IC4 - 16x	58	0.057	3.31 ppm (3.22)	x58 = 186.76	7.00 1
	Ø - 64x	77.5	0.240	18.60 (16.77)	x78 = 1308.00	
	Bag #16 IC4 - 64x	30	0.228	6.84 (6.75)	x58 391.5	3.57 2
	Ø - 128x	41	0.480	19.68 (17.85)	x78 1542.50	
	Bag #17 IC4 - 32x	42	0.114	4.79 (4.70)	x58 = 277.82	4.94 3
	Ø - ~~64x~~ 128x	40.5	0.~~240~~ .480	19.44 (17.61)	x78 ~~615.92~~ 1373.58	
	Bag #23 [illegible]					
	IC4 - 8x	3	.029	.087 ppm	~~[illegible] these values from other samples~~	
	Ø - 16x	30.5	.06	1.83 ppm		
	Std 6.11 ppm 32x	50	.120			
	1.06 ppm 16x	18	.057			
	[illegible]					

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CHROMATOGRAPHIC ANALYSIS LOG

Bethlehem Pa

Project No. 1920 Date 7-18-80 Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Napth Myell Pit Test #4 Run #2				
	Bag #14 ICy 16x	45	0.057	2.57 ppm	x58 = 149.06
	Ø 32x	36.75	.120	4.41 ppm	x78 = 343.98 2.31
	Bag #19 ICy 32x	35	0.114	3.99 ppm	x58 = 231.42
	Ø - 32x	47.5	.120	5.70 ppm	x78 = 444.6 1.92
	Bag #34 ICy - 16x	79	0.057	4.50 ppm	x58 = 261.17
	Ø - 64x	25.75	.240	6.18 ppm	x78 = 482.01 1.85
	Std 6.11 ppm Ø	48			
	1.06 ppm ICy	18.5			
	Bag #22 ICy - 8x	ND			
	Ø - 64x	28.5	~~2.83~~ .10	2.83 ppm	
	Std 6.11 ppm 32x10⁴	61.5	0.10		

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-18-80 Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
Call @ 1:53	North Dryer Tank Run #1 Method 110 Dil 10x	47.5	3.84	182.4 ppm	182.4 ppm

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CHROMATOGRAPHIC ANALYSIS LOG

[illegible]

Project No. 1922 Date 7-22-[illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
12:00	122.5 ppm of Sdd 64×10^3	59.5	2.06		
	cool Bag #3 32×10^3	2	1.03	2.06 ppm	
	Naph Melt Pot [illegible] Collected @ 1:30				Naph Melt Tank was full ~~[illegible]~~ with [illegible] ≈ 3/4 of the tank except where water was going in.
1)	Edge of pot near inlet of DU #1+2 32×10^3	44.5	1.03	45.84 ppm	
2)	Middle of Pot [illegible] 32×10^3	26	1.03	26.78 ppm	Ambient Temp 90°+
3)	Near Waste Trough (vicinity of [illegible]) 4×10^3	7	0.129	9.16 ppm	
4)	Base of Walkway 32×10^3	15	1.03	15.45 ppm	
5)	Sampler #1 Location 4×10^4	8.5	0.129	1.10 ppm	good charge

All collected at ground level

CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Bag #12 100 l 4x10³	1.5	.129		
	Bag #8 100 l 4x10³	30	.129	38.7 ppm	
	Std 122.5 ppm ϕ 64x10³	55			
Cell C 1:37	Test #1 Naph Dry Tank 128x10³ No Dil	33	4.12	135.96	.474 ft³ - [illegible]
Cell C 4:10	Test #2 Naph Dry Tank 32x10³ Dil 10x	32.5	1.03	33.45 x10 334.5 ppm	.100 ft³
	Std 122.5 ppm ϕ 64x10³	54	~~[illegible]~~		

Test #3 6.13

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. [illegible] Date [illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
-7:00	Naph Melt Pit Gas Samples Collected ~6:00 PM				
	Std 1225 ppm 64x10⁴	66	1.86		
1)	Edge of [illegible] of DC [illegible] 64x10³	36	1.86	66.96 ppm	Pit still filling [illegible]
2)	Middle of Nap Melt pit (cabinet [illegible]) 32x10³	39	0.93	36.27 ppm	[illegible]
3)	Near Waste Trough 32x10³	12.5	0.93	11.63 ppm	
4)	[illegible] 32x10³	29.5	0.93	27.44 ppm	
5)	Sampler #1 Loc. 4x10⁸	32	0.116	3.72 ppm	[illegible]
Call @ 8:15 PM	Nap Dry Tank Test #3 No Dil @ 3.2x10³	30	.93	27.90 ppm	1.354 ft³ [illegible]

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-23-80 Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Std 122.5 ppm ϕ 64x10³	6.8	1.83		
Call @ 9:30 PM	Nap Dry Tank Test #4 16x10³	32	0.45	[illegible] ppm	[illegible]
	Bag #1 Background 100l 4x10³	6.5	.116	0.754 ppm	
	Bag #3 Background 100l 4x10³	7.5	.116	0.870 ppm	
	Std 122.5 ppm ϕ 64x10³	63	1.94		
Call @ ~1100 PM 1)	Edge of Melt Pit near DO 1 inlets 64x10³	60	1.94	116.40 ppm	[illegible]
2)	Middle of Nap Pit (Schedule) 32x10³	43.5	0.97	42.20 ppm	Raining lightly Ambient [illegible]
3)	Near Water Tank [illegible] 32x10³	11	0.97	10.67 ppm	

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date [illegible] Analyst [illegible]

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
4)	Base of Wellhead 32x10³	16	0.97	15.52 ppm	
3)	Sample #1 [illegible] 8x10³	8.5	.243	2.07 ppm	[illegible]
	Nap Dry Tank Test #5	41	0.97	39.77 ppm	[illegible]
	Blank #1 BK @ 4x10³	NO	—	—	
	Std 1.275 ppm @ 10x10³	64			
	Blank #1 BK 4x10³	7	.121	0.85 ppm	
	Blank #2 BK 4x10³	5	.121	0.605 ppm	
	Blank #3 BK 4x10³	10	.121	1.21 ppm	
	Int with Cooling Tower Blank #1 2x10³	1	.06	0.06 ppm	
	Int Cooling Tower Blank #2 1x10³	1			
	Blank #3 / 4x10⁴	5			

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-22-8[illegible] Analyst TB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Flask # 4 Rt side of Cooling Tower 1x10³	1			
	Std 122.5 ppm p. @ 64x10³	60			
	Hyperbolic Dry Tanks 32x10³ Test # 6	30.25	0.97	29.34 ppm	

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CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-24-80 Analyst YB

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	Std 122.5 ppm ϕ 6cc/x10³	59.5	2.06		
	Bag Background 10l @ 1x10³		.033		
	Bag # 12	9			
	Bag # 21	12			
	Bag # 2	2			
	Bag # 11	1			
	Bag # 8	2			
	Bag # 7 @ 2x10³ (House)	2			
	Bag # 10 "	NO			
	Bag # 20 "	2			
	Bag # 13 - Old (1x10³)	2.5			
	Bag # 17.	15			

Scott Environmental Technology Inc.

CHROMATOGRAPHIC ANALYSIS LOG

Project No. 1922 Date 7-24-80 Analyst

Time	Sample Identification	Peak Height/Area	Concentration Factor	Concentration	Comments
	100l Bag #12 2×10^3	72	~~.022~~ .005		
	100l Bag #7 "	45		0.34 ppm	
	100l Bag #11 "				
	100l Bag #1 "	11			
	Std 122.5 ppm 64×10^3	60			
	Test # NOT Voided 16×10^3	15			
	Test # 7 NOT 64×10^3 No Dil	76	2.06	156.56 ppm	.420 ft^3 = sampval.
	Test #8 32×10^3	11.5	~~1.12~~ 1.03	~~47.38 ppm~~ 11.86	1.101 ft^3 = sampval

APPENDIX D

TRACER GAS METHOD DEVELOPMENT

APPENDIX D

TRACER GAS METHOD DEVELOPMENT

D.1 Tracer Gas Selection

The initial consideration when using the tracer gas method is the choice of a suitable gas. There are several criteria used in the selection: First, the tracer gas must not be present in the atmosphere at the sampling location. Second, the tracer gas must be separable from other components in the background at the sampling location and quantifiable on the same GC column without interfering with the elution of the compound(s) of primary interest. The tracer gas should also be readily available, transportable, economically feasible, and safe for the given usage situation.

For the determination of benzene emissions at secondary by-products plants, isobutane is the recommended tracer gas. The second choice for a tracer gas is a halogenated hydrocarbon. At secondary by-products plants the hydrocarbons in the background atmosphere are almost exclusively emissions from the coking operation and neither isobutane nor halogenated hydrocarbons are present to any significant degree. Isobutane was chosen over a halogenated hydrocarbon on the basis of chromatographic elution characteristics. Isobutane elutes well before the benzene peak thus eliminating any interference when using a temperature program for the chromatographic analysis.

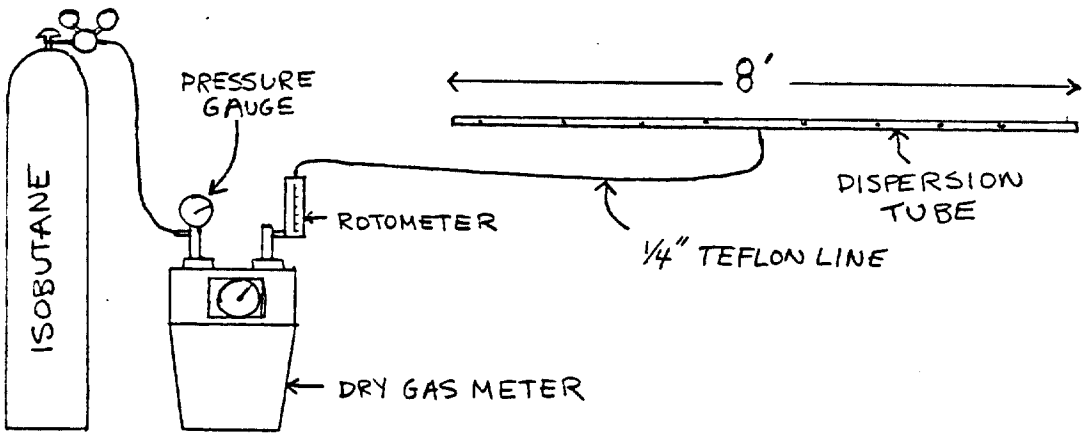
The separation of isobutane from mixtures containing concentrations of hydrocarbons typical of secondary by-products plants was verified by spiking samples collected at different sources in a secondary by-products plant with various concentrations of isobutane and performing

a temperature program of chromatographic analysis to achieve the desired degree of separation. In all cases the desired separation was achieved.

D-2 Dispersion Apparatus

The apparatus for the dispersion of tracer consists of a cylinder of the tracer gas connected to a dry gas meter, a rotameter and a dispersion tube. All necessary connecting lines are Teflon.

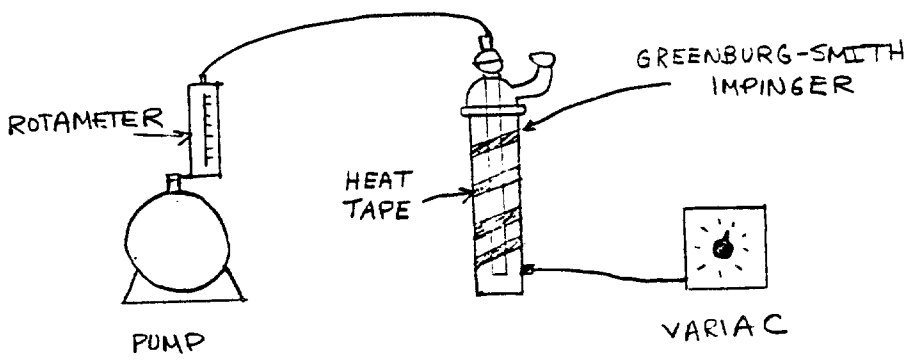
Two different dispersion tube configurations were tested, both were constructed from 1/4" O.D. stainless steel tubing. The first tube tested was 8' long with the tracer source connected to one end of the tube. The tube contained holes every 19" which were progressively larger moving away from the gas source. The hole size ranged from 0.062" to 0.031". The second tube was 8' long in two 4' sections which are connected via a T-joint to each other and to the tracer gas source. This dispersion tube has 0.041" holes every 19" and the ends are capped.



Of the two types of dispersion tubes tested the latter described was more efficient for the dispersion of the tracer. This judgement was made by visual inspection of the holes in each tube while isobutane was flowing at

0.1 CFM. At this rate isobutane can be seen as it leaves the dispersion tube and differences in the relative volume leaving each hole are visually discernible. The first configuration had all gas coming out of the first 2 holes, whereas the second configuration had uniform emissions from each orifice.

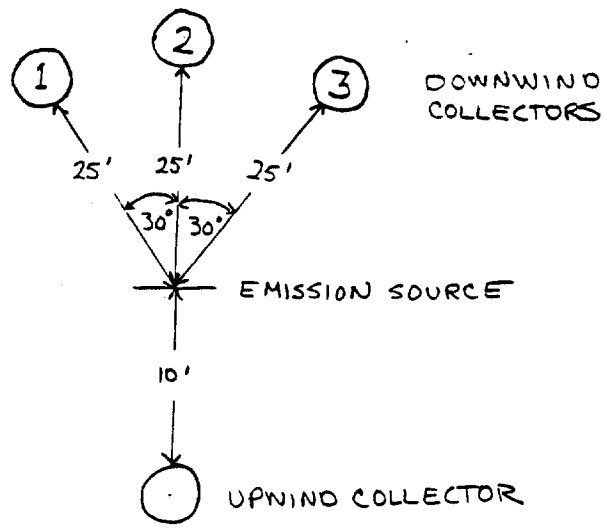
Benzene was also released in two ways; by evaporation and a heated bubbler. Both methods proved adequate for experimental determinations. When evaporation was used to release benzene, a stainless steel pan 16" x 24" x 1/2" was employed to contain the benzene. During an experimental determination benzene was added to the pan in 50 cc aliquots at intervals frequent enough to maintain a constant surface area of benzene. This was done in order to keep the emission of benzene at a constant rate. However, this evaporation method proved unsatisfactory on days when the wind speed exceeded 15-20 MPH due to the changing evaporation rate resulting from gusting wind. A more steady emission of benzene was achieved by using a heated bubbler. The bubbler system consisted of a 500 cc impinger of the Greenburg-Smith design wrapped with a heat tape. The impinger was kept at a constant temperature below the boiling point of benzene. A rubber diaphragm pump was used to push atmospheric air through a bubbler. Flow was regulated with a rotameter.



It was necessary to add more benzene during an experimental run, because the emission rate drops substantially if the benzene level drops too low in the impinger. The frequency of addition and the quantity of benzene per addition are dependent on the emission rate being used. For our determinations it was necessary to add 50 cc of benzene at intervals of approximately 10 minutes.

D-3 Experimental Determinations

An experiment consists of the release of a known amount of isobutane and benzene simultaneously. Samples are collected along a 30° arc, 25 feet downwind from the source of the emissions.



Initially samples were grab samples collected in clean one liter glass gas flasks. Later samples were integrated over a 1/2 hour period and collected in clean 10-liter Tedlar bags via Emission Measurements Air Quality Sampler with a flow rate of 10 LPH.

In initial determinations, portions of actual presurvey samples containing 62% benzene were released in an effort to simulate

the type of sample which would be encountered in the field. Various amounts of the sample mixture from 0.20 to 10 cc were released and samples were collected downwind in 1-liter gas flasks. When these samples were analyzed the amount of benzene detected was very small, approximately 20 ppb. From this it was apparent that it would be necessary to release significantly more benzene in order to produce the necessary concentration at the sampling location so that quantative mass to mass ratios could be calculated.

Because of the necessity of releasing more benzene and avoiding the foul odor which the high concentration benzene field samples possessed, it was decided that pure benzene be used for all subsequent determinations.

For the next series of experiments evaporation as previously described was used to release benzene. This series of experiments produced results accurate to within 10% of the theoretical mass to mass ratios with a minimum benzene emission of 0.54 lb/hr for the series. These experiments were performed on days when the wind speed was light (5 - 10 MPH) and the wind direction was steady (See Table D-1).

The next experiment was designed to test the variations which might be introduced when the wind speed and direction were less than favorable. On the day selected the wind speed was 20-25 MPH and the direction was 180° variable due to a changing weather system. The rate of evaporation of the benzene was noticeably affected by the conditions as were the dispersion patterns of the emissions. Erratic results were produced by the meteorological stress on key experimental variables. Calculated mass to mass ratios differed from the theoretical value from 15% to as much as 56%, demonstrating the effect of high and variable winds on the technique. In order to reduce stress on the experiment the benzene bubbler as described was used to provide

a steady source of benzene emission at a rate that would be independent of meteorological conditions. On the day chosen to use the bubbler system the wind speed was 15-20 MPH and the direction was steady. Favorable results were obtained despite the relatively strong wind demonstrating that the tracer technique is valid in winds up to 20 MPH depending on the sampling location (see Table D-1).

D-4 Summary

When using the tracer gas method it is necessary to verify that the tracer gas is detectable at the sampling location of choice as the method is somewhat dependent upon meteorological conditions. The method works best when the wind speed is light to moderate, 5-15 MPH, and the wind direction is steady. When the wind speed exceeds approximately 20 MPH or if there is no wind and/or the wind direction is too variable, dispersion patterns condusive to accurate sampling are disturbed and quantitative mass to mass relationships are difficult to establish. The upper limit of stress with respect to meteorological conditions can be examined by the spread of mass to mass ratios for each individual sample for a given sampling run. If the calculated ratios are inconsistent or the deviation between each calculated ratio and their mean is greater than 20%, it would be necessary to seek an explanation based on process variations or meteorological conditions or to void the sampling run and possibly suspend sampling until conditons are more favorable.

D-5 Field Sampling Strategy

The program for a sampling run will generally involve the collection of triplicate downwind samples and a single point upwind sample. Actual sampler locations will be determined by the gas chromatograph on

site. Grab samples will be collected in glass flasks and analyzed to determine the benzene concentration in the vicinity of the source to be tested. This information will be correlated with wind speed and direction to choose the exact sampler locations. In the ideal case downwind samplers will be equidistant from the source and along approximately a 30° arc.

Two sets of samples will be integrated over separate one-half hour periods and together constitute a single test. Samples will be collected by Environmental Measurements AQS II sampling system into clean 10-liter Tedlar bags. Tedlar bags to be reused for sampling will be flushed three times with nitrogen and allowed to sit overnight three quarters full. Prior to their next use each will be analyzed for benzene content.

The tracer gas dispersion apparatus will be positioned over the source to be tested as near as possible to the actual emissions. Ideally the dispersion tube or support member will span the source of the emissions at its center.

TABLE D-1

EXPERIMENTAL DATA

Release Rate g/min	Benzene Release Method	Sample Type	Wind Speed	Wind Direction	Theoretical $T\phi/ic_4$	ϕ/ic_4 #1	ϕ/ic_4 #2	ϕ/ic_4 #3	Average
ϕ 0.027 ic_4 5.26	Evaporation	Grab	0-5 MPH	Steady	0.005	*NO	NO	NO	---
ϕ 0.993 ic_4 8.27	Evaporation	Grab	0-5 MPH	Steady	0.120	*NO	NO	NO	---
ϕ 4.05 ic_4 7.16	Evaporation	Grab	0-5 MPH	Steady	0.59	0.64	0.64	0.65	0.645
ϕ 9.40 ic_4 6.38	Evaporation	Integrated	5-10 MPH	Steady	1.47	1.57	1.43	---	1.50
ϕ 10.85 ic_4 13.59	Evaporation	Integrated	20-25 MPH	Variable	0.80	1.29	1.82	0.94	1.35
ϕ 9.40 ic_4 8.25	Bubbler	Integrated	15-20 MPH	Steady	1.14	1.40	1.93	1.02	1.18
ϕ 6.33 ic_4 6.48	Bubbler	Integrated	0-5 MPH	Steady	0.91	0.97	0.96	0.96	0.96
ϕ 6.48 ic_4 6.48	Bubbler	Integrated	0-5 MPH	Steady	1.00	0.91	0.86	0.89	0.89

ϕ - Benzene

ic_4 - Isobutane

* No benzene, only isobutane detected.

APPENDIX E

FIELD AUDIT REPORT

FIELD AUDIT REPORT

PART A - To be filled out by organization supply unit cylinders (RTI)

1. Organization supplying audit sample(s) and shipping address

   Research Triangle Institute, Post Office Box 12194, Research Triangle Park, NC 27709

2. Audit supervisor, organization, and phone number (EMB Technical Manager)

   Dan Bivens, EPA

3. Shipping instructions - Name, Address, Attention

   Scott Environmental Technology

   Post Office Box D-11

   Plumsteadville, PA 18949 ATTN: Bob Denyszyn

4. Guaranteed arrival date for cylinders 6/10/80

5. Planned shipping date for cylinders 6/10/80

6. Details on audit cylinders for last analysis

		Low Conc.	High Conc.
a.	Date of last analysis	5/30/80	5/30/80
b.	Cylinder number	B-1372	B-921
c.	Cylinder pressure, PSI	1750	1500
d.	Audit gas(es)/balance gas	Benzene/N_2	Benzene/N_2
e.	Audit gas(es) ppm	7.93	154.4
f.	Cylinder construction	Steel	Steel

PART B - To be filled out by audit supervisor

1. Organic chemical manufacturing process Coke Oven By-Product Recovery Plant

2. Location of audit Bethlehem, Pa

3. Name of individual audit and organization Tom Bernstein, Scott Environmental Tech.

4. Audit results

	Low Conc.	High Conc.
a. Cylinder number	B-1372	B-921
b. Cylinder pressure before audit, psi	1750	1500
c. Cylinder pressure after audit, psi	?	1400
d. Audit date and measured concentration, ppm		
Date		
Analysis #1 7/8/80	7.66	137.36
Analysis #2 7/8/80	7.67	134.67
Analysis #3 ?	7.76	134.21
e. RTI concentration, ppm (Part A, 6d)	7.03	154.4

f. Audit accuracy*

Analysis #1	-3.4	-14.92
Analysis #2	-3.3	-12.78
Analysis #3	-2.1	-13.08

*Percent accuracy = $\frac{\text{Measured Conc. - RTI Conc.}}{\text{RTI Conc.}} \times 100$

g. Problems detected (if any): Scott is the company who prepared cylinder # R-921 several years ago. Then it's concentration was 134.8 ppm. Scott's gas dept. analyzed cylinder 10 times on 7/18/90. The results are attached.

SCOTT ENVIRONMENTAL TECHNOLOGY, INC.
Plumsteadville, Pennsylvania 18949

INTERDEPARTMENTAL MEMORANDUM

TO: D.F

FROM: P.K.C.

DATE: July 18/1980

SUBJECT: Analysis of Audit Cylinder #B-92

PROJECT #1922

The Primary Standard is taken from Gas Dept. & its Conc. is 110.9 ppm.

The audit cylinder Conc. is 134.8 ppm
Standard deviation in analysis ±1.1 ppm

Individual analysis results are

(1) → 133.8 ppm
(2) → 135.2 ppm
(3) → 135.5 ppm
(4) → 133.8 ppm
(5) → 136.4 ppm
(6) → 135.9 ppm
(7) → 133.1 ppm
(8) → 133.8 ppm
(9) → 135.2 ppm
(10) → 135.7 ppm

Mean 134.8 ppm
STD. Deviation ±1.1 ppm.

7/18/80

APPENDIX F

PROJECT PARTICIPANTS

APPENDIX F

PROJECT PARTICIPANTS

The following people participated in some phase of the sampling program at Bethlehem Steel.

From Scott Environmental Technology, Inc.:

Tom Bernstiel, Chemist

Jack Carney, Chemist

P. K. Chattopadhyay, Chemist

Dan FitzGerald, Manager, Eastern Operations

Kevin Gordon, Technician

Carolyn Graham, Chemical Engineer

Scott Henderson, Environmental Scientist

Lou Reckner, Vice President & General Manager

Joe Wilson, Senior Technician

From Research Triangle Institute:

Ralph Roberson

Dave Marsland

From U. S. Environmental Protection Agency

Lee Beck

Dan Bivins

APPENDIX G

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) Records are to be retained at the source and made available for inspection by the Administrator for a minimum of 2 years.

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

Appendix B—Test Methods

Method 110. Determination of Benzene From Stationary Sources

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph, nor by those who are unfamilar with source sampling, because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to benzene, a carcinogen.

1. Applicability and Prinicple

1.1 Applicability. This method applies to the measurement of benzene in stack gases from processes as specified in the regulations. The method does not remove benzene contained in particulate matter.

1.2 Principle. An integrated bag sample of stack gas containing benzene and other organics is subjected to gas chromatographic (GC) analysis, using a flame ionization detector (FID).

2. Range and Sensitivity

The range of this method is 0.1 to 70 ppm. The upper limit may be extended by extending the calibration range or by diluting the sample.

3. Interferences

The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of benzene; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis problem, subject to the approval of the Administrator. Approval is automatic provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. Apparatus

4.1 Sampling (see Figure 110–1). The sampling train consists of the following components:

4.1.1 Probe. Stainless steel, Pyrex * glass, or Teflon tubing (as stack temperature permits), equipped with a glass wool plug to remove particulate matter.

4.1.2 Sample Lines. Teflon, 6.4 mm outside diameter, of sufficient length to connect probe to bag. Use a new unused piece for each series of bag samples that constitutes an emission test and discard upon completion of the test.

4.1.3 Quick Connects. Stainless steel, male (2) and female (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 of trade names or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.

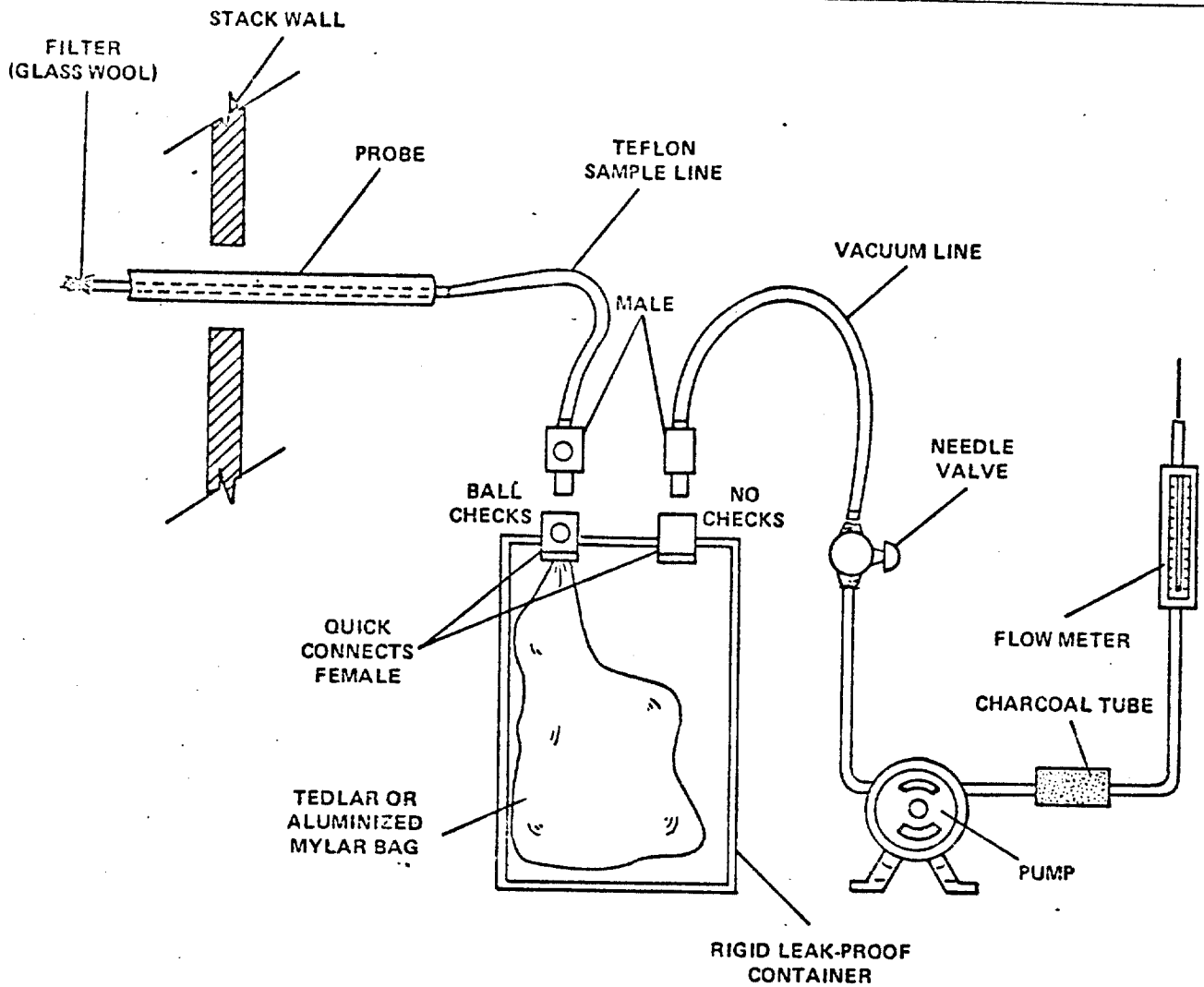

Figure 110-1. Integrated-bag sampling train. (Mention of trade names or specific products does not constitute endorsement by the Environmental Protection Agency.)

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

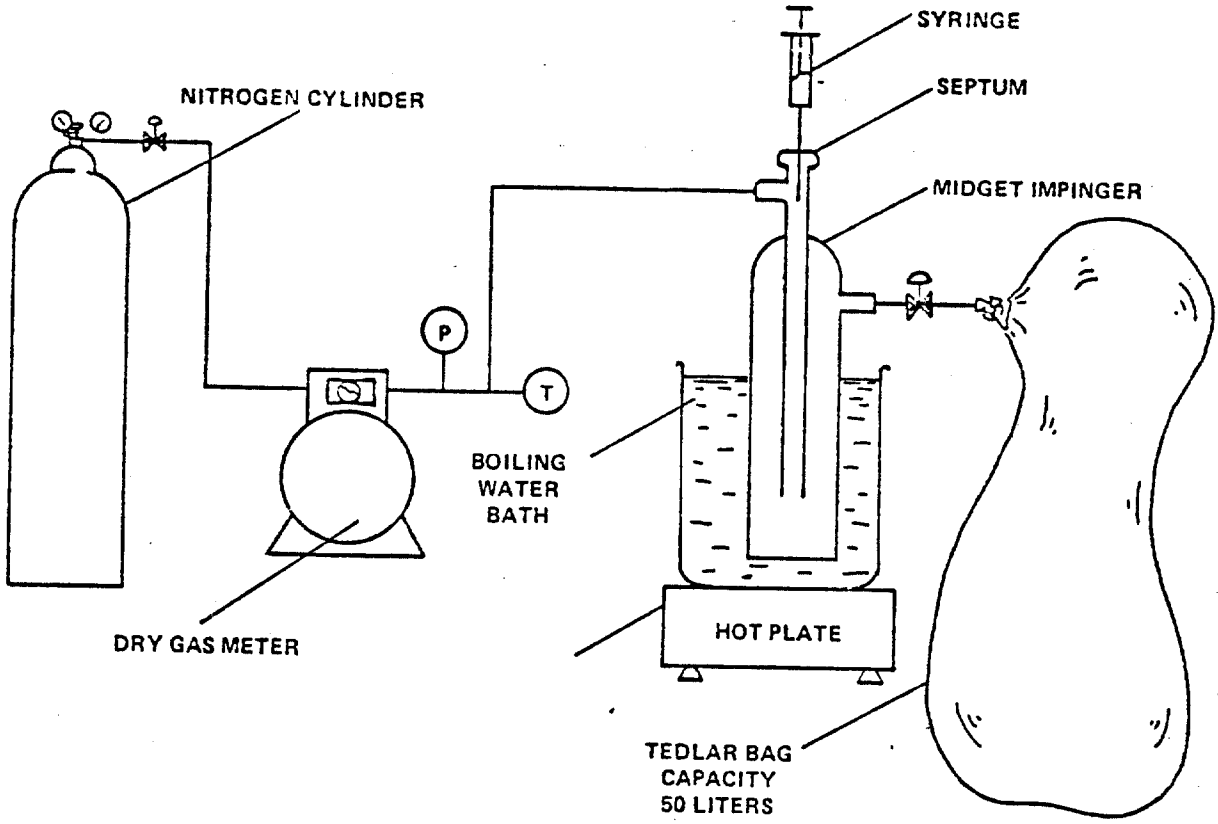

Figure 110-2. Preparation of benzene standards (optional).

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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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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 sample 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:

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

$$C_c = 701.9 \frac{BT_m}{V_m \; Y \; P_m} \qquad (110\text{-}1)$$

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

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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.

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

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. $A_o = I_o A_c/A_s$
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 $A_s = h_s t_s$.

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

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 contaminant 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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