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Emission Test Report Corning Glass Works Martinsburg, West Virginia

EMISSION TEST REPORT

METHOD DEVELOPMENT AND TESTING FOR
ARSENIC FROM GLASS PLANTS
Corning Glass Works
Martinsburg, West Virginia

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SECTION 1

INTRODUCTION

Arsenic is listed as a hazardous air pollutant under Section 112 of the Clean Air Act (National Emission Standards for Hazardous Air Pollutants). To protect public health from unreasonable risks associated with exposure to airborne arsenic, the U.S. Environmental Protection Agency (EPA) has developed standards to decrease inorganic arsenic emissions from the following source categories: high-arsenic primary copper smelters, low-arsenic primary copper smelters, and glass manufacturing plants.

To support the standards review process and provide additional arsenic emissions data from glass manufacturing facilities, PEI Associates, Inc., (under contract to the Emission Standards and Engineering Division - Emission Measurement Branch) performed a series of atmospheric emission tests on a glass melting furnace at the Corning Glass Works facility in Martinsburg, West Virginia, from October 15 through 17, 1984. These tests were conducted to determine if the quantity of particulate arsenic as measured by EPA Reference Method 108 varies with flue gas and sample temperatures. Reference Method 108* provides total arsenic results (particulate plus gaseous fraction).

A total of five quad train runs (see Figure 2-1) were conducted using draft Method 108 procedures except that probe and filter temperatures were elevated to 177° and 260°C (350° and 500°F) in order to evaluate the effects

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*40 CFR 61, Appendix B, Method 108, July 1984.

of sample temperature on arsenic distribution in the sampling train. During the quad runs, a single Method 108 sampling train with probe and filter temperature of 121°C (250°F) was run for reference purposes.

Section 2 summarizes and discusses the test results; Section 3 addresses quality assurance considerations specific to this project; Section 4 describes the sampling locations and test procedures; and Section 5 describes source operation. Appendix A presents sample calculations and computer printouts; Appendices B and C contain the field data sheets and laboratory analytical results, respectively; Appendix D details the sampling and analytical procedures; Appendix E summarizes equipment calibration procedures and results; Appendix F is a quality assurance element finder; and Appendix G is a list of project participants and a sampling log.

SECTION 2

SUMMARY AND DISCUSSION OF TEST RESULTS

2.1 SAMPLING AND ANALYTICAL PROTOCOL

A four-train (quad) sampling system was used to collect samples in the rectangular breeching connecting the furnace to the exit stack. This system allows four trains to sample simultaneously at essentially a single point in the duct (see Section 4). Therefore, this sampling approach reduces the effect of variations in the velocity and particulate profiles on the sampling results. It also permits a statistically significant number of samples to be taken in a short amount of time.

The quad runs conducted were designed to determine if the quantity of filterable arsenic collected varies with sampling train temperature. For comparative purposes, two of the trains were heated to approximately 177°C (350°F) and two trains were heated to approximately 260°C (500°F). At each temperature, one train possessed a backup filter heated to 121°C (250°F) prior to the impinger section.

Figure 2-1 depicts the quad train configuration used in these tests. Individual train components were recovered and separately analyzed for arsenic to evaluate the distribution of arsenic in the sampling train.

During these runs, a single Method 108 sampling system (designated RT) (121°C) was run for reference purposes. The reference train was located on the opposite side of the breeching and as close as possible to the quad probe system.
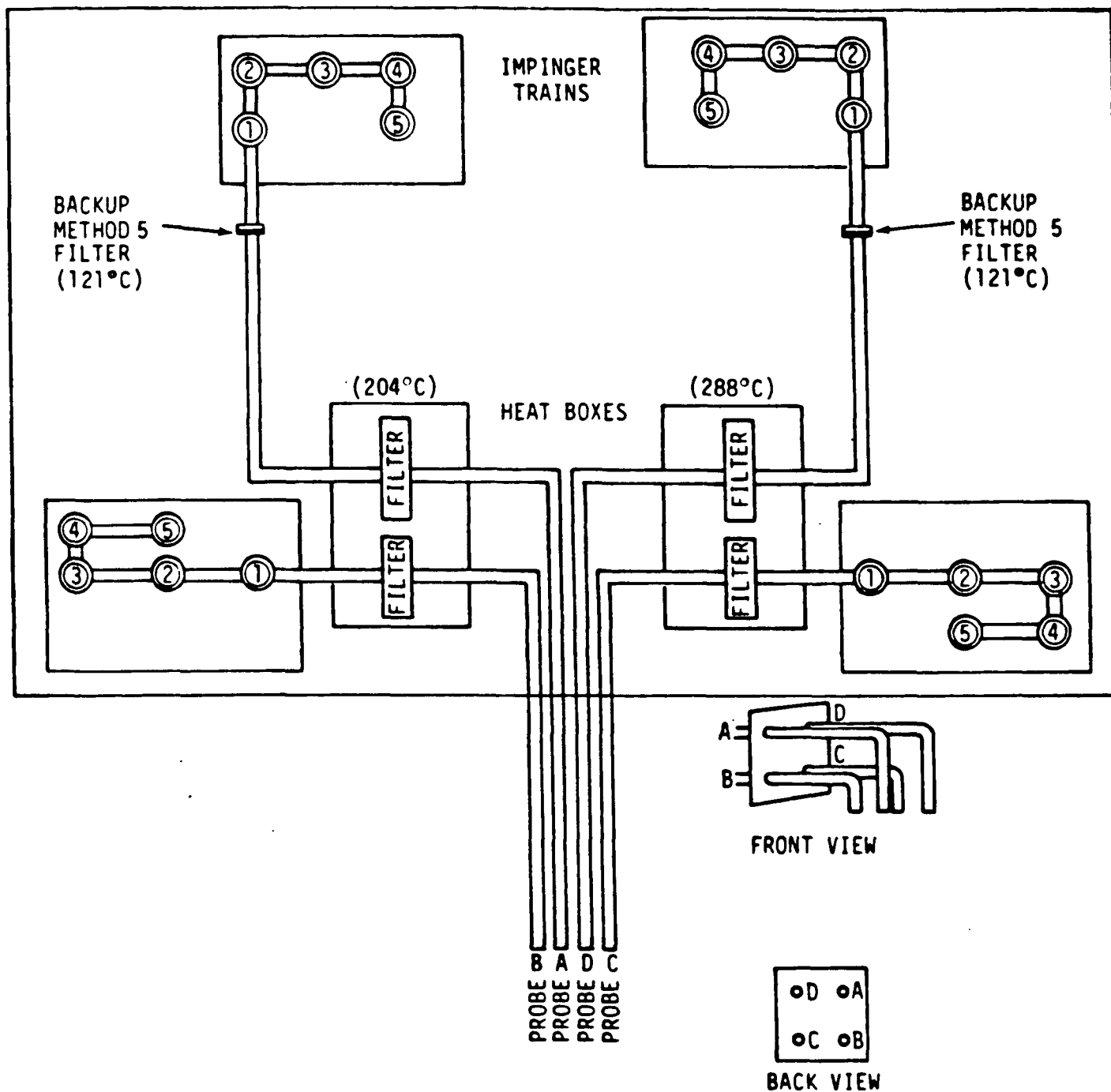
Figure 2-1. Quad train system for elevated temperature tests.

In each train, the probe and filter temperatures were set at a predetermined temperature and monitored using multiterminal digital indicators with thermocouple leads located in each probe and immediately behind the Method 5 filter frits. Table 2-1 presents the sampling matrix followed in this test program.

2.2 TEST RESULTS--QUAD AND REFERENCE TRAIN

Table 2-2 summarizes sampling conditions for the quad and reference train test runs. Table 2-3 summarizes the arsenic analytical results by sample fraction. Sample volumes are expressed in dry standard cubic meters (dsm³), arsenic weights in milligrams (mg), and arsenic concentrations in milligrams per dry standard cubic meter (mg/dsm³).

The filterable or front-half arsenic reported in Table 2-3 represents that material collected on the front filters and in the sampling probes which were maintained at 177° and 260°C for the quad runs and 121°C for the reference train runs. The condensible or back-half arsenic represents that material which passed through the front filter and was collected in either the connecting glassware, backup filter, or impinger section of the sampling train.

Sample volumes were consistent and ranged between 1.19 and 1.58 dsm³ for the quad runs and 1.31. to 1.62 dsm³ for the reference train runs. Isokinetic sampling rates ranged from 93.5 to 103.1 percent, which is within the acceptable range of 90 to 110 percent.

The desired temperature for paired Trains A and B was 177°C (350°F) and for paired Trains C and D, 260°C (500°F). The reported probe and filter temperatures represent average values determined from data recorded on the

TABLE 2-1. SAMPLING MATRIX

Quad Run No.	Sample ID	Method 108 sample temperatures[a]		Reference Method 108 train at 121°C (250°F)
		177°C (350°F)	260°C (500°F)	
1	1A	X (BU)		
	1B	X		
	1C		X	
	1D		X (BU)	
Reference train				X
2	2A	X (BU)		
	2B	X		
	2C		X	
	2D		X (BU)	
Reference train				X
3	3A	X (BU)		
	3B	X		
	3C		X	
	3D		X (BU)	
Reference train				X
4	4A	X (BU)		
	4B	X		
	4C		X	
	4D		X (BU)	
Reference train				X
5	5A	X (BU)		
	5B	X		
	5C		X	
	5D		X (BU)	
Reference train				X

[a]The designation BU indicates a backup filter maintained at 121°C (250°F) was located prior to the impinger section of the sampling train. Sampling train components (i.e., probe, filter(s), impingers) were recovered and analyzed separately.

TABLE 2-2. SUMMARY OF SAMPLING CONDITIONS

Run No.	Sampling type	Date (1984) and time (24-h)	Metered volume, dsm³	Isoki-netic, %	Mois-ture, %	Sampling conditions			
						Flue gas tempera-ture, °C	Probe tempera-ture, °C	Filter tempera-ture, °C	Backup fil-ter tempera-ture, °C
1A	Modified	10/15	1.58	101.4	9.95	393	177	183	128
1B	Method 108	1315-1451	1.55	99.9	9.92	390	177	178	NA
1C[a]			-	-	-	-	-	-	-
1D			1.46	103.1	10.07	388	255	266	124
RT-1	Method 108	10/15 1315-1445	1.62	100.4	8.15	392	111	122	NA
2A	Modified	10/16	1.46	98.8	10.33	395	175	177	125
2B	Method 108	0952-1122	1.44	101.1	11.69	395	186	176	NA
2C			1.22	95.1	10.92	394	266	262	NA
2D			1.30	100.0	10.17	395	259	270	118
RT-2[b]	Method 108	10/16 0952-1122	1.31	81.5	7.64	386	98	129	NA
3A	Modified	10/16	1.53	99.0	10.27	380	177	180	121
3B	Method 108	1344-1514	1.52	98.8	10.48	378	181	178	NA
3C			1.32	93.5	10.69	379	259	268	NA
3D			1.41	99.9	10.40	375	263	275	124
RT-3	Method 108	10/16 1503-1633	1.53	99.1	10.25	369	128	118	NA

(continued)

TABLE 2-2 (continued)

Run No.	Sampling type	Date (1984) and time (24-h)	Metered volume, dsm^3	Isoki- netic, %	Mois- ture, %	Sampling conditions			
						Flue gas tempera- ture, °C	Probe tempera- ture, °C	Filter tempera- ture, °C	Backup fil- ter tempera- ture, °C
4A	Modified	10/17	1.43	98.0	9.83	375	181	177	119
4B	Method 108	0840-1010	1.41	99.2	9.84	380	181	175	NA
4C			1.19	94.2	10.00	376	268	263	NA
4D			1.27	99.9	10.23	379	266	270	124
RT-4	Method 108	10/17 0840-1010	1.61	97.9	9.55	382	146	115	NA
5A	Modified	10/17	1.57	99.3	9.94	365	174	181	121
5B	Method 108	1253-1428	1.55	98.6	10.13	370	179	180	NA
5C			1.36	94.2	10.05	367	259	269	NA
5D			1.43	99.8	9.95	370	261	275	124
RT-5	Method 108	10/17 1243-1428	1.57	98.2	9.57	370	121	119	NA

[a]Run No. 1C is void due to excessive post-test leak rate.

[b]Run No. RT-2 is void due to a nonisokinetic sample condition.

NA = Not applicable.

TABLE 2-3. SUMMARY OF ARSENIC ANALYTICAL RESULTS
(QUAD AND REFERENCE TRAIN RUNS)

Run No.	Sample volume, dsm³	Filterable			Back-half (condensibles)						Concentration, mg/dsm³			Filterable arsenic, % of total
		Probe rinse	Front filter	Total front half	Glass connector	Backup filter	Impinger			Total back half	Front half	Back half	Total train	
							1	2	3 and 4					
1A	1.58	10.9	33.8	44.7	56.6	5.5	34.3	0.89	0.10	97.4	28.3	61.6	89.9	31.5
1B	1.55	21.7	33.4	55.1	NA	NA	131.4	2.5	0.26	134.3	35.5	86.6	122.2	29.1
1C[a]	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1D	1.46	15.5	32.2	47.7	34.1	26.3	17.4	0.42	0.05	78.3	32.7	53.6	86.3	37.9
RT-1	1.62	13.0	28.9	41.9	NA	NA	75.9	0.87	0.41	77.2	25.9	47.6	73.5	35.2
2A	1.46	22.9	30.8	53.7	19.5	19.1	12.5	0.30	0.045	51.4	36.8	35.2	72.0	51.1
2B	1.44	20.4	29.2	49.6	NA	NA	90.4	1.8	0.20	92.4	34.4	64.2	98.6	34.9
2C	1.22	16.9	24.6	41.5	NA	NA	60.1	3.9	0.82	64.8	34.0	53.1	87.1	39.0
2D	1.30	10.3	25.7	36.0	8.6	26.5	7.9	0.32	0.09	43.4	27.7	33.4	61.1	45.3
RT-2	1.31	8.2	17.8	26.0	NA	NA	59.4	1.61	0.09	61.1	19.8	46.6	66.4	29.9
3A	1.53	14.1	35.9	50.0	84.9	30.3	16.6	0.31	0.03	132.1	32.7	86.3	119.0	27.5
3B	1.52	22.0	33.6	55.6	NA	NA	130.3	4.5	0.30	135.1	36.6	88.9	125.5	29.2
3C	1.32	6.1	33.4	39.5	NA	NA	77.9	1.80	1.01	80.7	29.9	61.1	91.0	32.9
3D	1.41	22.5	31.6	54.1	53.0	34.9	12.7	0.60	0.05	101.3	38.4	71.8	110.2	34.8
RT-3	1.53	40.3	35.3	75.6	NA	NA	108.5	2.1	0.35	110.95	49.4	72.2	121.6	40.6
4A	1.43	32.3	31.3	63.6	62.5	21.8	7.8	0.20	0.02	92.3	44.5	64.5	109.0	40.8
4B	1.41	22.0	30.8	52.8	NA	NA	92.7	1.08	0.22	94.0	37.4	66.7	104.1	36.0
4C	1.19	17.7	26.9	44.6	NA	NA	69.9	1.18	1.02	72.1	37.5	60.6	98.1	38.2
4D	1.27	9.3	28.4	37.7	27.5	3.3	24.2	0.86	0.09	55.95	29.7	44.0	73.7	40.3
RT-4	1.61	38.4	37.4	75.8	NA	NA	92.1	1.92	0.10	94.1	47.1	58.4	105.5	44.6
5A	1.57	19.4	34.4	53.8	84.4	35.1	8.5	0.14	0.48	128.6	34.3	81.9	116.2	29.5
5B	1.55	19.4	32.1	51.7	NA	NA	121.0	4.65	0.07	125.7	33.4	81.1	114.5	29.1
5C	1.36	8.4	30.5	38.9	NA	NA	104.5	1.74	0.96	107.2	25.1	69.2	94.3	26.6
5D	1.43	17.1	29.4	46.5	54.4	42.1	10.1	0.40	0.05	107.1	32.5	74.9	107.4	30.3
RT-5	1.57	41.6	33.8	75.4	NA	NA	95.5	2.55	0.17	98.2	48.0	62.5	110.5	43.4

[a] Run 1C is void due to excessive post-test leak rate.

NA = Not applicable.

field data sheets. As shown, filter temperatures for Trains A and B ranged from 175° to 183°C and the probe temperatures ranged between 174° and 186°C. In Trains C and D, the filter temperatures ranged between 262° and 275°C and the probe temperatures ranged between 255° and 268°C. The backup filter temperatures for each quad run, Trains A and D, ranged between 118° and 128°C. The reference train probe temperatures ranged between 98° and 146°C and the filter temperatures ranged between 115° and 129°C.

The moisture content of the flue gas was generally consistent for each run and ranged between 9 and 11 percent. Flue gas temperatures ranged between 367° and 393°C during the test program. As shown in Table 2-2, the flue gas moisture content determined from the reference train for Tests 1 and 2 is at least 20 percent lower than the corresponding quad train moisture data. This was the result of a leakage problem that developed in the reference train during these runs. This problem was not detected during the tests because the sampling train could not be thoroughly leak checked according to the Method 108 procedure. As a result of the geometric configuration of the breeching and the location of scaffolding near the test port, the sampling probe was first inserted into the duct and then connected to the Method 108 sample box containing the heated filter and impingers. Each component (probe and sample box) was leak checked separately before and after each test. Because neither sampling train component experienced a leakage problem during these runs, the leak must have occurred at the probe front-filter connection. Therefore, the reference train arsenic results for Runs 1 and 2 are biased low; the magnitude of which is unknown.

As shown in Table 2-3, arsenic sample weights are reported separately for each sample fraction analyzed. Sample concentrations are also reported on a

filterable, condensible, and total train basis. The front filter weight includes results for both the NaOH extract and the Parr bomb (HF/HNO_3) extract. The Parr bomb extract results constituted less than 1 percent of the total arsenic on the front filter.

Arsenic was found throughout each sample train; the filterable or front-half arsenic constituted between 28 and 51 percent of the total arsenic collected in the 177°C quad trains (A and B) and between 26 and 45 percent of the total arsenic collected in the 260°C quad trains (C and D). In each individual quad run, except Train 2A, more than 50 percent of the total arsenic collected was found in the back half of the quad sampling trains.

This same trend was observed in the reference train tests, although the leakage problems associated with Runs RT-1 and 2 tend to distort comparisons between these data and the corresponding quad train results. The percentage of filterable arsenic found in the reference train ranged between 41 and 45 percent for Runs 3 through 5 compared with a range of 26 to 41 percent and an overall average of 33 percent for the corresponding quad runs. Data from Runs 3 through 5 suggest that a greater percentage of filterable arsenic is collected at 121°C than at 177° or 260°C. More than 50 percent of the total arsenic measured, however, was collected in the back half of the sampling trains regardless of sample temperature.

Tables 2-4 through 2-6 present statistical data for the quad runs on both a total train and filterable/condensible basis. The mean arsenic concentration and standard deviation for each set of runs are presented along with the coefficient of variation (CV), which is the standard deviation expressed as a percent of the group mean.

TABLE 2-4. STATISTICAL DATA FOR GROUPED RUNS
(TOTAL TRAIN)

Quad Run No.	Individual run value, mg/dsm³	Group mean $\overline{X}$,[b] mg/dsm³	σ,[c] mg/dsm³	CV,[d] %
1A	89.9			
1B	122.2	99.5	19.8	19.9
1C[a]	-			
1D	86.3			
2A	72.0			
2B	98.6	79.7	16.5	20.7
2C	87.1			
2D	61.1			
3A	119.0			
3B	125.5	111.4	15.0	13.4
3C	91.0			
3D	110.2			
4A	109.0			
4B	104.1	96.2	15.7	16.3
4C	98.1			
4D	73.7			
5A	116.2			
5B	114.5	108.1	10.0	9.2
5C	94.3			
5D	107.4			
Overall means		99.0[e]	15.7[f]	15.9[g]

[a]Run 1C was voided due to excessive post-test leak rate.

[b]Mean concentration.

[c]Within-run standard deviation with N-1 weighting for sampling data.

[d]Within-run coefficient of variation is the standard deviation expressed as a percent of the mean concentration.

[e]Simple averages of tabulated data.

[f]Pooled standard deviation; $\sqrt{\dfrac{\Sigma\sigma^2}{n}}$.

[g]$CV = \sqrt{\dfrac{\Sigma\sigma^2}{n}} / \overline{X}$.

TABLE 2-5. WITHIN-RUN STATISTICAL DATA FOR PAIRED QUAD RUNS
(TOTAL TRAIN BASIS)

Run No.	Desired sampling temperature, °C	Individual run value, mg/dsm³	Mean, $\bar{X}$	σ, mg/dsm³	CV, %	Reference train value, mg/dsm³
1A	177	89.9	106.1	22.8	22	
1B	177	122.2				
1C	260	-	-	-	-	73.5
1D	260	86.3				
2A	177	72.0	85.3	18.8	22	
2B	177	98.6				
2C	260	87.1	74.1	18.4	25	66.4
2D	260	61.1				
3A	177	119.0	122.3	4.6	4	
3B	177	125.5				
3C	260	91.0	100.6	13.6	13	121.6
3D	260	110.2				
4A	177	109.0	106.6	3.5	3	
4B	177	104.1				
4C	260	98.1	86.4	16.5	19	105.5
4D	260	74.7				
5A	177	116.2	115.4	1.2	1	
5B	177	114.5				
5C	260	94.3	100.9	9.3	9	110.5
5D	260	107.4				

TABLE 2-6. STATISTICAL DATA OF FILTERABLE AND CONDENSIBLE
ARSENIC FOR GROUPED QUAD RUNS

Quad Run No.	Filterable arsenic				Condensible arsenic			
	Individual front-half value, mg/dsm³	Group mean, $\overline{X}$	σ, mg/dsm³	CV, %	Individual back-half value, mg/dsm³	Group mean, $\overline{X}$	σ, mg/dsm³	CV, %
1A	28.3				61.7			
1B	35.5	32.2	3.6	11.3	86.6	67.3	17.2	25.6
1C	-				-			
1D	32.7				53.6			
2A	36.8				35.2			
2B	34.4	33.2	3.9	11.7	64.2	46.6	14.6	31.3
2C	34.0				53.1			
2D	27.7				33.9			
3A	32.7				86.3			
3B	36.6	34.4	3.8	11.1	88.9	77.0	13.0	16.9
3C	29.9				61.1			
3D	38.4				71.8			
4A	44.5				64.5			
4B	37.4	37.3	6.0	16.2	66.7	59.0	10.3	17.4
4C	37.5				60.6			
4D	29.7				44.0			
5A	34.3				81.9			
5B	33.4	31.3	4.2	13.4	81.1	76.8	5.9	7.7
5C	25.1				69.2			
5D	32.6				74.9			
Overall means		33.7[a]	4.4[b]	13.1[c]		65.3[a]	12.8[b]	19.6[c]

[a]Simple average of tabulated data.

[b]Pooled standard deviation; $\sqrt{\frac{\Sigma\sigma^2}{n}}$.

[c]$CV = \sqrt{\frac{\Sigma\sigma^2}{n}}/\overline{X}$.

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As presented in Table 2-4, the statistical data on a total train basis showed an overall mean of 99.0 mg/dsm³ with mean arsenic concentrations of individual quad groups ranging from 79.7 to 111.4 mg/dsm³. The standard deviations of the quad groups ranged from 10.0 to 19.8 mg/dsm³ with a pooled mean value of 15.7 mg/dsm³. The mean coefficient of variation for the five runs was 15.9 percent.

Table 2-5 summarizes the within-run statistical data for paired quad runs (either 177° or 260°C) on a total train basis. Comparison of results between the two sample temperatures are difficult because both temperatures showed large variations. This is evidenced by the standard deviations of paired runs 2A and B (σ = 18.8 mg/dsm³) and 2C and D (σ = 18.4 mg/dsm³).

In Runs 2 through 5, however, the paired means for the 177°C trains were consistently higher than the paired means of the 260°C trains. In each quad run, the mean arsenic concentrations determined for the 177°C trains were between 13 and 19 percent higher than the mean concentrations for the 260°C trains.

The statistical data for filterable and condensible arsenic presented in Table 2-6 show a relatively consistent pattern for the filterable arsenic as evidenced by a mean filterable arsenic concentration of 33.7 mg/dsm³ and a pooled standard deviation of 4.4 mg/dsm³. The pooled coefficient of variation for the filterable fraction was 13.1 percent. The individual group mean values ranged from 32.2 to 37.4 mg/dsm³, suggesting a small difference in filterable arsenic concentration as measured by the 177° and 260°C trains.

The condensible or back-half quad train arsenic data were characterized by a mean concentration of 65.3 mg/dsm³ with individual group means ranging between 46.6 and 77.0 mg/dsm³. The standard deviation of the quad groups

ranged between 5.9 and 17.2 mg/dsm³ with a pooled mean standard deviation of 12.8 mg/dsm³ and a mean CV of 19.6 percent.

As presented in Tables 2-3 and 2-5, the test results for the Method 108 reference train compare to within 10 percent of the quad group means on a total train basis. As discussed previously, leak problems with Tests RT-1 and 2 resulted in a low bias of arsenic results for these runs; thus, valid comparisons between the two sampling systems are limited to Runs 3 through 5.

In Run 3, the quad group mean was 111.4 mg/dsm³ compared with a reference train value of 121.6 mg/dsm³. In Run 4, the quad group mean was 96.2 mg/dsm³ compared with a reference value of 105.5 mg/dsm³. In Run 5, the quad group mean was 108.1 mg/dsm³ compared with a reference value of 110.5 mg/dsm³. The reference train results averaged 2 percent lower than the 177°C results and 15 percent higher than the 260°C quad results.

In each run, the amount of filterable arsenic collected in the reference train was greater than the corresponding quad train results. The mean filterable arsenic concentration in Quad Group 3 was 34.4 mg/dsm³ compared with a reference train value of 49.4 mg/dsm³. In Quad Group 4, the mean filterable arsenic concentration was 37.3 mg/dsm³ compared with a reference train value of 47.1 mg/dsm³. In Quad Group 5, the mean filterable arsenic concentration was 31.3 mg/dsm³ compared with a reference train value of 48.0 mg/dsm³.

In summary, the Method 108 reference train run at 121°C consistently collected 20 to 30 percent more arsenic in the front half of the train than the Method 108 trains heated to 177° and 260°C. The total train results are comparable for the reference and 177°C trains; whereas, the 260°C results average 15 percent lower than the reference train results.

Several factors that could have affected test results are addressed as follows. The leak problems associated with Reference Train Tests 1 and 2

resulted in a low bias of arsenic results for these runs; thus, valid comparisons with the corresponding quad runs are not possible.

As indicated in Tables 2-3 and 2-4, Quad Run 1C was void because of an excessive post-test leakage rate. The calculated moisture content for this train was approximately 45 percent lower than the within-run moisture data for Trains 1A, B, and D; thus, this sample was discarded and not analyzed. No leakage problems were detected in any of the reported quad train tests.

A heavy deposition of white condensate was observed in all of the back-half glassware in the two sampling systems. This observation is consistent with the reported arsenic results in the back half of each sampling train. All back-half glassware were rinsed with 0.1 N NaOH, and visible material was removed with the aid of a nylon brush. It is possible that some of the material was not or could not be recovered, which could contribute to the reported deviations in back-half arsenic results.

SECTION 3

PROJECT QUALITY ASSURANCE

Because the desired end product of testing is to achieve representative emission results, quality assurance is one of the main facets of stack sampling. Quality assurance guidelines provide the detailed procedures and actions necessary for defining and producing acceptable data. Four such documents were used in this test program to ensure the collection of acceptable data and to provide a definition of unacceptable data. The following documents comprise the detailed site test plan prepared by PEI and reviewed by the Emission Measurement Branch: the EPA Quality Assurance Handbook Volume III, EPA-600/4-77-027; the PEI Emission Test Quality Assurance Plan; and the PEI Laboratory Quality Assurance Plan. The last two, which are PEI's general guideline manuals, define the company's standard operating procedures and are followed by the emission testing and laboratory groups.

In this specific test program, the following steps were taken to ensure that the testing and analytical procedures produced quality data:

° Calibration of all field sampling equipment.

° Checks on train configuration and calculations.

° Onsite quality assurance checks (i.e., leak checks of the sampling train, pitot tube, and Orsat line) and quality assurance checks of all test equipment prior to use.

° Use of designated analytical equipment and sampling reagents.

° Internal and external audits to ensure accuracy in sampling and analysis.

Table 3-1 lists the sampling equipment used to perform the arsenic tests and the calibration guidelines and limits. In addition to the pre- and post-test calibrations, a field audit was performed on the metering and temperature measurement systems used in the test runs. Critical orifices constructed by PEI were used in the dry gas meter audits. The onsite audits were made at the beginning of the test program. Figures 3-1 through 3-8 present the results of the onsite audits. These data were used to assess the operational status of the sampling equipment relative to guidelines established by the U.S. EPA. The results of the field audits indicate that the sampling equipment was functioning properly throughout this test series.

PEI personnel calculated the sampling rates on site. The data were rechecked and validated at the end of the test program by computer programming. Some minor discrepancies between the hand calculations and computer printouts resulted primarily because of round-off error. Overall, the data compared favorably. Figure 3-9 presents an example calculation form PEI used during this test program. Computerized example calculations are presented in Appendix A.

As an additional check of the reliability of the method used to analyze the samples, a blank train was assembled in the recovery area, capped off, and set aside for about 2 hours. The blank train was assembled at the beginning of the test series using clean glassware. The blank train was recovered in the same manner as the test samples. These samples were shipped to the laboratory and analyzed by the same procedures as those used for the actual emission samples. In addition to the blank sampling train, aliquots of the field reagents used in the collection and recovery of the samples were obtained daily and analyzed by the same procedures as those used for the actual

3-2

TABLE 3-1. FIELD EQUIPMENT CALIBRATION

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Meter box	FB-8 Train A	Wet test meter	ΔH @ ±0.15 (Y ±0.05 Y post-test)	-0.08 0.034	X X	
	FB-3 Train B			-0.05 0.01	X X	
	FB-5 Train C			0.01 0.025	X X	
	FB-1 Train D			-0.02 0.007	X X	
	FB-11 (Reference train)			0.0 0.0075	X X	
Pitot tube	511 517 509	Standard pitot tube	Cp ±0.01	- - -	OK OK	Visually inspected on-site
Digital indicator	124 125 221	Millivolt signals	0.5%	0.41% 0.14% 0.41%	X X X	Maximum deviation
Thermocouple	134 - (stack) 128 - (stack)	ASTM-3F	1.5% (±2% saturated)	+0.41% +0.47%	X X	Maximum deviation

(continued)

3-3

TABLE 3-1 (continued)

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Thermocouple (cont'd)	612 - Probe 632 - Filter 429 - Backup filter			+0.57% -0.22% -0.33	X X X	Maximum deviation
	604 - Probe 634 - Filter			+0.57% -0.20%	X X	
	619 - Probe 635 - Filter			-0.63% 0.0%	X X	
	618 - Probe 631 - Filter 427 - Backup filter			0.57% 1.0%	X X	
	608 - Probe 615 - Probe 602 - Probe 607 - Probe			-0.41% +0.57% +0.75% -0.61%	X X X X	
Orsat analyzer	145	Standard gas	±0.5%	0.2% 0.2% 0.2%	X X X	CO_2 O_2 CO
Impinger thermometer	I-3 I-2 434 435 433 446	ASTM-3F	±2°F	+1.0°F +1.0°F +0.5°F +1.0°F +1.5°F +1.0°F	X X X X X X	

(continued)

3-4

TABLE 3-1 (continued)

Equipment	ID No.	Calibrated against	Allowable error	Actual error	Within allowable limits	Comments
Mettler balance	M-1	Type S weights	±0.5 g	+0.1 g	X	
Barometer	229	NBS traceable barometer	+0.10 in.Hg. (0.20 post-test)	0.01 in.Hg.	X	
Dry gas thermometer	FB-8	ASTM-3F	±5°F	+4°F +3°F	X X	Inlet Outlet
	FB-3			+2°F +2°F	X X	Inlet Outlet
	FB-5			+1°F +3°F	X X	Inlet Outlet
	FB-1			-3°F +2°F	X X	Inlet Outlet
	FB-11			+2°F +2°F	X X	Inlet Outlet
Probe nozzle	1A 1B 1C 1D	Caliper	Dn ±0.004 in.	0.001 in. 0.001 in. 0.001 in. 0.000 in.	X X X X	
	2A 2B 2C 2D			0.001 in. 0.001 in. 0.000 in. 0.001 in.	X X X X	
	RT tests			0.001 in.	X	

FIELD AUDIT REPORT: DRY GAS METER
BY CRITICAL ORIFICE

DATE: __10·14·84__   CLIENT: __US EPA__

BAROMETRIC PRESSURE (P_{bar}): __29.4__ in.Hg   METER BOX NO. __FB·8__

ORIFICE NO. __7__   PRETEST Y: __0.990__ ΔH@ __1.91__ in.H_2O

ORIFICE K FACTOR: __4.964 ×10⁻⁴__   AUDITOR: __D. Schffel__

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft³	Temperatures						Duration of run ∅ min.
		Ambient		Dry gas meter				
		T_{ai}/T_{af}, °F	Average T_a, °F	Inlet T_{ii}/T_{if}, °F	Outlet T_{oi}/T_{of}, °F	Average T_m, °F		
2.05	817.100	82	82	82	78	83	19.3 Hg. 1625-1645
	833.035	82	542	88	80		20

Dry gas meter V_m, ft³	$V_{m_{std}}$, ft³	$V_{m_{act}}$, ft³	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O
15.935	15.303	15.082	.986	-.4	1.91	0.0

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar}^{29.55} + \Delta H/13.6)}{(T_m + 460)_{543}} = 15.303 \text{ ft}^3$$

$$V_{m_{act}} = \frac{1203(\emptyset)(K)(P_{bar})}{(T_a + 460)^{1/2}_{23.81}} = 15.082 \text{ ft}^3$$

$$\text{Audit Y} = \frac{V_{m_{act}}}{V_{m_{std}}} = .986 \qquad \text{Y deviation} = \frac{\text{Audit Y} - \text{Pre-test Y}}{\text{Audit Y}} \times 100 = -.4$$

$$\text{Audit } \Delta H@ = (0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{\emptyset}{Y(V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = 1.91 \text{ in.}H_2O$$

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

Figure 3-1. Field audit report: dry gas meter by critical orifice (Meter Box FB-8, A Train).

FIELD AUDIT REPORT: DRY GAS METER
BY CRITICAL ORIFICE

DATE: __10·14·84__   CLIENT: __USEPA__

BAROMETRIC PRESSURE (P_{bar}): __29.40__ in.Hg   METER BOX NO. __FB-3__

ORIFICE NO. __3__   PRETEST Y: __1.016__   ΔH@ __1.88__ in.H_2O

ORIFICE K FACTOR: __5.377×10^{-4}__   AUDITOR: __D. Scheffel__

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft³	Temperatures					Duration of run ∅ min.
		Ambient		Dry gas meter			
		T_{ai}/T_{af}, °F	Average T_a, °F	Inlet T_{ii}/T_{if}, °F	Outlet T_{oi}/T_{of}, °F	Average T_m, °F	1630 1650
2.3	896.103 / 912.700	82 / 82	82	86 / 86	80 / 82	83.5	20

Dry gas meter V_m, ft³	$V_{m_{std}}$, ft³	$V_{m_{act}}$, ft³	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O
16.597	15.935	16.337	1.025	+.9	1.87	0.01

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar}\,^{29.57} + \Delta H/13.6)}{(T_m + 460)_{543.5}} = 15.935 \text{ ft}^3$$

$$V_{m_{act}} = \frac{1203(\,∅\,)(\,K\,)(P_{bar})}{(T_a + 460)_{13.741}^{1/2}} = 16.337 \text{ ft}^3$$

$$\text{Audit Y} = \frac{V_{m_{act}}}{V_{m_{std}}} = 1.025 \qquad \text{Y deviation} = \frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}} \times 100 = +.9$$

$$\text{Audit } \Delta H@ = (0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{∅}{Y\,(V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = 1.87 \text{ in.}H_2O$$

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

Figure 3-2. Field audit report: dry gas meter by critical orifice (Meter Box FB-3, B Train).

FIELD AUDIT REPORT: DRY GAS METER
BY CRITICAL ORIFICE

DATE: _10·14·84_ CLIENT: _US EPA_

BAROMETRIC PRESSURE (P_{bar}): _29.4_ in.Hg METER BOX NO. _FB-5_

ORIFICE NO. _13_ PRETEST Y: _0.985_ ΔH@ _1.75_ in.H_2O

ORIFICE K FACTOR: _4.555×10⁴_ AUDITOR: _D. Schiff_

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft³	Temperatures					Duration of run ∅ min.
		Ambient		Dry gas meter			
		T_{ai}/T_{af}, °F	Average T_a, °F	Inlet T_{ii}/T_{if}, °F	Outlet T_{oi}/T_{of}, °F	Average T_m, °F	_1635_
_1.65_	_866.330_ / _880.560_	_82_ / _82_	_82_	_82_ / _82_	_74_ / _74_	_78_	_20_

Dry gas meter V_m, ft³	$V_{m_{std}}$, ft³	$V_{m_{act}}$, ft³	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O
_14.230_	_13.779_	_17.840_	_1.004_	_1.93_	_1.93_	_.18_

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar} + \Delta H/13.6)}{(T_m + 460)} = 13.779 \text{ ft}^3$$

(29.52, 538 written above)

$$V_{m_{act}} = \frac{1203(\emptyset)(k)(P_{bar})}{(T_a + 460)^{1/2}} = 13.840 \text{ ft}^3$$

(23.281 written below)

Audit Y = $\frac{V_{m_{act}}}{V_{m_{std}}}$ = _1.004_   Y deviation = $\frac{\text{Audit Y - Pre-test Y}}{\text{Audit Y}}$ × 100 = _1.93_

Audit ΔH@ = $(0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{\emptyset}{Y(V_m)(P_{bar} + \Delta H/13.6)}\right]^2$ = _1.93_ in.H_2O

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

Figure 3-3. Field audit report: dry gas meter by critical orifice (Meter Box FB-5, C Train).

FIELD AUDIT REPORT: DRY GAS METER
BY CRITICAL ORIFICE

DATE: __10·14·84__   CLIENT: __USEPA__

BAROMETRIC PRESSURE (P_{bar}): __29.40__ in.Hg METER BOX NO. __FB-1__

ORIFICE NO. __7__   PRETEST Y: __0.958__ ΔH@ __1.69__ in.H_2O

ORIFICE K FACTOR: __4.964X10⁴__   AUDITOR: __D. Schull__

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft³	Temperatures						Duration of run θ min.
		Ambient		Dry gas meter				
		T_{ai}/T_{af}, °F	Average T_a, °F	Inlet T_{ii}/T_{if}, °F	Outlet T_{oi}/T_{of}, °F	Average T_m, °F		
1.85	324.720	92	82	92	75	79		1709 -
	341.145	82		83	76			

Dry gas meter V_m, ft³	$V_{m_{std}}$, ft³	$V_{m_{act}}$, ft³	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O
16.425	15.885	16.082	.949	-.9	1.72	.03

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar} + \Delta H/13.6)}{(T_m + 460)\ 539} = 15.885 \text{ ft}^3$$

(29.54 written above P_{bar})

$$V_{m_{act}} = \frac{1203(\theta)(K)(P_{bar}^-)}{(T_a + 460)\ 33.481}^{1/2} = 15.082 \text{ ft}^3$$

$$\text{Audit Y} = \frac{V_{m_{act}}}{V_{m_{std}}} = .949 \qquad \text{Y deviation} = \frac{\text{Audit Y} - \text{Pre-test Y}}{\text{Audit Y}} \times 100 = -.9$$

$$\text{Audit ΔH@} = (0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{\theta}{Y\ (V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = 1.72 \text{ in.}H_2O$$

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

Figure 3-4. Field audit report: dry gas meter by critical orifice (Meter Box FB-1, D Train).

FIELD AUDIT REPORT: DRY GAS METER
BY CRITICAL ORIFICE

DATE: _10.14.84_ CLIENT: _US EPA_

BAROMETRIC PRESSURE (P_{bar}): _29.4_ in.Hg METER BOX NO. _FB-11_

ORIFICE NO. _3_ PRETEST Y: _1.052_ ΔH@ _115_ in.H_2O

ORIFICE K FACTOR: _5.397 × 10^{-4}_ AUDITOR: _D. Scheffel_

Orifice manometer reading ΔH, in.H_2O	Dry gas meter reading V_i/V_f, ft³	Temperatures						Duration of run θ min.
		Ambient		Dry gas meter				
		T_{ai}/T_{af}, °F	Average T_a, °F	Inlet T_{ii}/T_{if}, °F	Outlet T_{oi}/T_{of}, °F	Average T_m, °F		1721—
1.35	206.935	82	82	86	77	80.25		25
	227.050	82		87	77			

Dry gas meter V_m, ft³	$V_{m_{std}}$, ft³	$V_{m_{act}}$, ft³	Audit, Y	Y deviation, %	Audit ΔH@, in.H_2O	ΔH@ Deviation, in.H_2O
20.115	19.382	20.4V	1.054	−0.2	1.09	−.06

$$V_{m_{std}} = \frac{17.647(V_m)(P_{bar} + \Delta H/13.6)}{(T_m + 460)\,546.25} = 19.382 \text{ ft}^3$$

(29.56 above P_{bar})

$$V_{m_{act}} = \frac{1203(\theta)(K)(P_{bar})}{(T_a + 460)^{1/2}\,23.281} = 20.42 \text{ ft}^3$$

$$\text{Audit } Y = \frac{V_{m_{act}}}{V_{m_{std}}} = 1.054 \qquad Y \text{ deviation} = \frac{\text{Audit Y} - \text{Pre-test Y}}{\text{Audit Y}} \times 100 = -0.2$$

$$\text{Audit } \Delta H@ = (0.0317)(\Delta H)(P_{bar})(T_m + 460)\left[\frac{\theta}{Y(V_m)(P_{bar} + \Delta H/13.6)}\right]^2 = 1.09 \text{ in.}H_2O$$

Audit Y must be in the range, pre-test Y ±0.05 Y.
Audit ΔH@ must be in the range pre-test ΔH@ ±0.15 inches H_2O.

Figure 3-5. Field audit report: dry gas meter by critical orifice (Meter Box FB-11, Reference Train).

ON-SITE AUDIT DATA SHEET

Audit Name: __USEPA__   Date: _10·15·84_ Auditor: _D. Scheffel_

Equipment	Reference	Reference Value	Value Determined		Deviation		Max. Allowable Deviation
Meter box inlet thermo.	ASTM-3F at ambient temp.	45	#B-8 48	#B-3 46	#B-8 +3	#B-3 -1	5°F
Meter box outlet thermo.	ASTM-3F at ambient temp.	45	43	48	-2	+3	5°F
Impinger thermometer	ASTM-3F at ambient temp.	45	#446 48	#443 46	#446 +3	#443 +1	2°F
Stack Imp. thermometer or	ASTM-3F at ambient temp.	45	#434 46	#434 45	#434 +1	#435 0	7°F
Thermocouple	ASTM-3F at stack temp.						See table
Orsat analyzer	% O$_2$ in ambient air	20.8%	20.7		0.1		0.7%
Trip balance	IOLM std. weight	NA					0.5 grams
Barometer	Corrected* NWS value	NA					0.20 in. Hg

Reference temp. °F	32-140	141-273	274-406	407-540	541-673	674-760
Max. deviation °F	7	9	11	13	15	17

* Correction factor:

   NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

** 0.74 in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-6. Onsite audit data sheet.

ON-SITE AUDIT DATA SHEET

Audit Name: _____ Date: _10·15·84_ Auditor: _N. Schiffel_

Equipment	Reference	Reference Value	Value Determined	Deviation	Max. Allowable Deviation
Meter box inlet thermo.	ASTM-3F at ambient temp.	45	FB-11 42	-3	5°F
Meter box outlet thermo.	ASTM-3F at ambient temp.	45	48	+3	5°F
Impinger thermometer	ASTM-3F at ambient temp.				2°F
Stack thermometer or	ASTM-3F at ambient temp.				7°F
Thermocouple	ASTM-3F at stack temp.	48	#134 50	+2	See table
Orsat analyzer	% O₂ in ambient air	20.8%			0.7%
Trip balance	IOLM std. weight				0.5 grams
Barometer	Corrected* NWS value				0.20 in. Hg

Reference temp. °F	32-140	141-273	274-406	407-540	541-673	674-760
Max. deviation °F	7	9	11	13	15	17

* Correction factor:

   NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

** 0.74 in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-7. Onsite audit data sheet.

ON-SITE AUDIT DATA SHEET

Audit Name: _____ Date: _/0·/5·84_ Auditor: _A. Schifful_

Equipment	Reference	Reference Value	Value Determined		Deviation		Max. Allowable Deviation
Meter box inlet thermo.	ASTM-3F at ambient temp.	45	FB5 46	FB-1 44	FB5 +1	FB-1 -1	5°F
Meter box outlet thermo.	ASTM-3F at ambient temp.	45	44	40	-1	-5	5°F
Impinger thermometer	ASTM-3F at ambient temp.						2°F
Stack thermometer or	ASTM-3F at ambient temp.						7°F
Thermocouple	ASTM-3F at stack temp.						See table
Orsat analyzer	% O$_2$ in ambient air	20.8%					0.7%
Trip balance	IOLM std. weight						0.5 grams
Barometer	Corrected* NWS value						0.20 in. Hg

Reference temp. °F	32-140	141-273	274-406	407-540	541-673	674-760
Max. deviation °F	7	9	11	13	15	17

* Correction factor:

   NWS value (in. Hg) - [Altitude (ft)/1000(ft/in. Hg)] + 0.74 in. Hg**

** 0.74 in. Hg is the nominal correction factor for the reference barometer against which the field barometer was calibrated.

If it is not feasible to perform the audit on any piece of equipment, record "N/A" in the space provided for the data.

Figure 3-8. Onsite audit data sheet.

ISOKINETIC CALCULATION

SITE __Corning - Martinsburg, W. Va.__   TEST NO. __1__ (10/15/84)

		RUN 1	RUN 2	RUN 3	RUN 4	
1. Volume of dry gas sampled corrected to standard conditions. Note: V_m must be corrected for leakage if any leakage rates exceed L_a). $$V_{m_{std}} = 17.65 \times V_m \times Y \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{T_m} \right]$$	V_m, ft^3	57.389	64.79	49.754 *44.354	54.36	55.178
	Y	0.99	1.016	.985	.958	1.052
	P_{bar}, in.Hg	29.55	29.55	29.55	29.55	29.55
	$\overline{\Delta H}$, in.H_2O	1.35	1.36	1.02	1.03	.857
	$\overline{T_m}$, °R	532	531	529	527	533
	$V_{m_{std}}$, dscf	55.878	54.85		51.66	51.22
2. Volume of water vapor at standard conditions, ft^3. $$V_{w_{std}} = 0.04707 V_{lc} =$$	V_{lc}, g	131.2	128.3		122.8	107.8
	$V_{w_{std}}$, ft^3	6.176	6.039		5.78	5.07
3. Moisture content in stack gas. $$B_{ws} = \frac{V_{w_{std}}}{V_{w_{std}} + V_{m_{std}}} =$$	B_{ws}	.099	.099		.101	.081
	$1-B_{ws}$	0.90	.901		.899	.919
4. Dry molecular weight of stack gas, lb/lb-mole. $$M_d = 0.440 (\% CO_2) + 0.320 (\% O_2)$$ $$+ 0.280 (\% N_2 + \% CO) =$$	% CO_2	4.5				
	% O_2	14				
	% N_2 + % CO	81.5				
	M_d, lb/lb-mole	29.3				
5. Molecular weight of stack gas. $$M_s = M_d (1-B_{ws}) + 18 B_{ws} =$$	M_s, lb/lb-mole	28.16	28.18		28.16	28.39
6. Stack velocity at stack conditions, fps. $$V_s = 85.49 \, Cp \left(avg. \sqrt{\Delta P} \right) \sqrt{\frac{T_s}{P_s M_s}} =$$	P_{static}, in.H_2O	-1.0				-1.0
	P_s, in.Hg	29.48				29.48
	$\overline{T_s}$, °R	1199	1195	1200	1193	1198
	$\sqrt{\Delta P}$	.621	.621	—	.619	.720
	Cp	.84				
	V_s, fps	55.6	53.5		53.3	61.9
7. Isokinetic variation $$\% I = \frac{V_{m_{std}} \times \frac{T_s}{V_m} \times 17.32}{V_s \times B_m \times \theta \times P_s \times (1-B_{ws})}$$	Dn, in.	.299	.298	.286	.285	.280
	θ, min.	90				
	% I	101.4 ✓	99.9 ✓	—	103.4 ✓	103 ✓

*-corrected for .06 cfm leakage

Figure 3-9. Example of onsite calibration data sheet.

samples. Table 3-2 presents the results of the blank sampling train and field blank analyses. The results are very low and indicate that background arsenic contamination was not a problem in the sample recovery area.

Laboratory reagent blank analyses were performed during the analysis of the field samples. The results of these analyses are presented in Table 3-3. The average value for four filter blanks was 0.026 mg out of a range of 0.021 to 0.031; because this value is insignificant compared with the measured values, no blank correction was made. All of the blank values for the rinse and impinger samples were below the analytical detection limit of 0.002 to 0.006 mg.

Each sample was first analyzed by the flame technique. Sample concentrations below 30 mg/liter were also analyzed using the graphite furnace. The 30-mg/liter limit was based on previous experience with Method 108, which indicated good agreement above this level. As the analyses were completed and the data were reduced by the laboratory, the results were reviewed by the Quality Assurance Officer (QAO). The QAO reviewed instrument calibration, the analysis of the standard reference solution (SRS), agreement between flame and furnace results, and general consistency of the data. He then prepared a list of samples for reanalysis.

The flame analysis was performed on six days. Twenty-eight sets of standards (0, 10, 30, 50, 80, 100 ppm) were analyzed with the samples. Table 3-4 presents linear regression data on all the standards analyzed for the 11 analysis runs. The average correlation coefficient is 0.9988, out of a range of 0.9994 to 0.9980. The average detection limit is 2.3 ppm. A value of twice the range of the 0-ppm standard above the Y-intercept was used to calculate the detection limit. A standard reference solution independently

TABLE 3-2. ARSENIC BLANK DATA

Blank sampling train arsenic values[a]

Train No.	Filter, mg	NaOH probe rinse, mg	Impinger section, mg	Total train blank, mg
1	0.021	0.030	<0.010	0.051

Field blank arsenic values

Date samples taken	Corresponding Run No.	Filter total, mg	NaOH,[b] mg/liter	H_2O,[c] mg/liter
10/15	1	0.027	<0.013	<0.013
10/16	2 + 3	0.031	<0.013	<0.013
10/17	4 + 5	0.024	<0.013	<0.013
Average blank values		0.027	<0.013	<0.013

[a]Sampling train was fully assembled in recovery area and then recovered and analyzed as a sample.

[b]Between 150 and 493 ml of NaOH was used to rinse the probe. Between 36 and 167 ml of the NaOH was used to rinse Impingers 1 and 2. Between 53 and 126 ml of the NaOH was used to rinse Impingers 3 and 4. Between 206 and 302 ml of the NaOH was used to rinse the connector. The maximum blank for the NaOH corresponds to 0.006 mg for the probe rinse, 0.003 mg for the impinger samples, and 0.004 mg for the connector samples.

[c]On all days, 150 ml of water was added to arsenic Impingers 1, 2, and 3. The maximum blank for the water corresponds to 0.002 mg for Impingers 1, 2, and 3.

TABLE 3-3. ARSENIC LABORATORY REAGENT BLANK DATA

Date (1984)	Filter total, mg	Rinse,[a] mg/liter	Impingers,[b] mg/liter	Connector,[c] mg/liter
11/15	0.001	<0.013	<0.013	<0.013

[a]Between 150 and 493 ml of samples were received as the rinse fraction. The maximum laboratory reagent blank corresponds to 0.006 mg for this fraction.

[b]Between 188 and 400 ml of samples were received as the Impingers 1 and 2 fractions and between 205 and 280 ml as the Impingers 3 and 4 fractions. These correspond to maximum laboratory reagent blanks of 0.005 mg and 0.004 mg, respectively.

[c]Between 206 and 302 ml of samples were received as the connector fraction. The maximum laboratory reagent blank corresponds to 0.004 mg for this fraction.

TABLE 3-4. LINEAR REGRESSION DATA (FLAME)

Date (1984)	No. of standard curves	Y-intercept	Slope	Correlation coefficient	Detection limit, ppm
10/31	3	+0.0026	0.00498	0.9987	1.2
10/31	2	-0.0003	0.00485	0.9990	1.2
11/1	3	+0.0077	0.00470	0.9980	1.7
11/1	2	+0.0035	0.00484	0.9991	2.5
11/5	3	+0.0075	0.00464	0.9986	2.2
11/5	2	+0.0076	0.00447	0.9992	1.8
11/7	3	+0.0052	0.00446	0.9982	3.6
11/7	2	+0.0032	0.00441	0.9994	2.7
11/8	4	+0.0067	0.00462	0.9989	3.5
11/8	2	+0.0075	0.00468	0.9989	0.8
11/16	2	+0.0005	0.003656	0.9988	4.4

prepared from As_2O_3 with a nominal value of 150 ppm was analyzed (1-2 dilution) with each set of standards. (Standards were prepared from a commercially available 1000-ppm standard solution.) The average value obtained in the 28 analyses of this standard reference solution (SRS) was 157.9 ppm, with a standard deviation (SD) of 10.6 ppm [6.7 percent relative standard deviation (RSD)]. Only 1 of the 28 determinations made fell outside the range of the mean ±2 SD (one was 136 ppm).

These data indicate that the precision and accuracy of the flame atomic absorption analyses are well within acceptable limits. The percent difference of the average measured value of the SRS and its predicted value is 5.3 percent; the RSD of the measured value is 6.7 percent.

Table 3-5 presents the results of four samples checked by the standard addition method. The slopes of all the standard addition analyses are between 0.9 and 1.1. The results of standard addition show no consistent bias attributable to the sample matrices.

All samples below 30 ppm were also analyzed by furnace techniques. Values obtained from flame and furnace techniques cannot be accurately compared below 10 ppm because this value is too close to the flame detection limit. Nine sets of standards (0, 0.01, 0.05, 0.10, and 0.15 mg/liter) were analyzed with the furnace samples on a single analysis day. All the data were reduced by linear regression analysis. The correlation coefficient for the linear regression analysis was 0.9930. The detection limit for the graphite furnace was 0.0064 ppm. A value of twice the range of the 0-ppm standard above the Y-intercept was used to calculate the detection limit.

A standard reference solution independently prepared from As_2O_3 with a nominal value of 0.0750 ppm was analyzed with each set of standards. (Standards were prepared from a commercially available 1000-ppm standard solution.)

TABLE 3-5. ARSENIC STANDARD ADDITION RESULTS

Lab No.	Spike, ppm	Previously determined flame, ppm	Measured, ppm	Linear regression analysis
DW185 filter (1-10 dilution)	0 9.09 18.18 27.27	37.3	34.88 41.44 52.66 59.77	Slope = 0.945 Y intercept = 34.30 Corr. = 0.9947 X intercept = 36.30
DW216 probe	0 9.09 18.18 27.27	40.8	38.98 47.46 54.85 63.87	Slope = 0.903 Y intercept = 38.98 Corr. = 0.9993 X intercept = 43.18
DW240 impinger	0 9.09 18.18 27.27	35.6	34.06 44.73 55.12 62.50	Slope = 1.053 Y intercept = 34.75 Corr. = 0.9967 X intercept = 33.00
DW182 bomb	0 9.09 18.18 27.27	63.1	62.23 72.62 80.56 Lost	Slope = 1.008 Y intercept = 62.64 Corr. = 0.9970 X intercept = 62.13

The average value obtained for the nine analyses of this SRS was 0.0774 ppm with a standard deviation of 0.0047 (6.0 percent relative standard deviation). Historically, the mean value for this SRS is 0.0762, with a standard deviation of 0.0027. The values obtained for the SRS solution during this project are in good agreement with our historical data. These data indicate that the precision and accuracy of the furnace atomic absorption analyses are well within acceptable limits. The difference in the average measured value of the SRS and its predicted value is 3.2 percent; the SRD of the measured value is 6.0 percent.

The results of duplicate analyses are presented in Tables 3-6 and 3-7. The absolute value of the percent difference was calculated according to the following equation.

$$\% \text{ Difference} = \frac{X_1 - X_2}{\bar{X}} \times 100$$

where X_1 and X_2 are the individual values

$\bar{X}$ is the average of X_1 and X_2

Duplicate analyses by flame atomic absorption above 15 ppm yields very good results. The maximum percent difference is 6.3 percent. Duplicate analyses by furnace atomic absorption yield generally good results (less than 10 percent difference) except for Samples DW258 and DW325. Sample DW325, although a 23 percent difference, contains less than 0.2 mg of arsenic. Sample DW258 gives a larger percent difference; one of the aliquots may have been slightly contaminated. At less than 2 mg of arsenic, this is not a significant problem considering 100 mg of arsenic was measured in each train.

TABLE 3-6. DUPLICATE ANALYSIS DATA (FLAME)

Sample fraction (Lab No.)	Arsenic, mg	% Difference
Filter[a] (DW177)	33.6, 33.9	0.8
(DW188)	25.6, 26.4	2.9
(DW201)	26.8, 27.5	2.7
Backup filter[a] (DW192)	29.9, 29.9	0.2
Bomb (DW182B)	3.16, 3.15	0.1
(DW187B)	0.45^b, 0.49^b	8.5^b
(DW196B)	8.97, 8.46	5.8
(DW200B)	0.40^b, 0.31^b	26.8^b
Probe rinse[c] (DW221)	21.7, 20.4	6.3
(DW273)	22.5, 21.3	5.5
Impinger[c] (DW231)	17.4, 17.7	1.8
(DW248)	60.1, 61.4	2.2
(DW274)	53.0, 54.3	2.5
(DW296)	27.5, 27.3	0.8
(DW223)	2.73^b, 2.84^b	3.8^b
(DW258)	2.38^b, 1.40^b	52.1^b
(DW281)	$<0.5^b$, $<0.8^b$	b
Probe and connector rinse[d] (DW313)	7.73, 8.08	4.4
(DW305)	84.4, 83.0	1.6
Impinger[d] (DW314)	104.5, 98.5	5.9
(DW325)	$<0.8^b$, $<0.8^b$	b
Connector[e] (DW283)	62.5, 62.0	0.9
Impinger[e] (DW288)	92.7, 93.9	1.3
(DW301)	92.1, 94.1	2.2

[a]Same aliquot analyzed on different days.

[b]Flame analysis below 12 ppm; which is 5 times the average flame detection limit.

[c]Sample aliquots prepared and analyzed on different days.

[d]Sample aliquots prepared and analyzed on the same day.

[e]Different dilutions of same aliquot analyzed the same day.

TABLE 3-7. DUPLICATE ANALYSIS DATA (FURNACE)[a]

Sample fraction (Lab No.)	Arsenic, mg	% Difference
Filter bomb (DW187B)[b]	0.31, 0.33	8.8
(DW200B)[b]	0.17, 0.18	8.3
Probe rinse (DW313)[c]	8.39, 7.70	8.6
Impinger (DW223)[d]	2.61, 2.67	2.0
(DW258)[d]	1.61, 0.78	70
(DW281)[d]	0.35, 0.36	3.6
(DW325)[c]	0.17, 0.14	23

[a]All furnace analyses performed on the same day.

[b]Different aliquots of same subsample diluted for furnace analysis.

[c]Sample aliquots prepared on same day, a week prior to analysis.

[d]Sample aliquots prepared on different days, 7 to 14 days prior to analysis. Sample DW258 exhibited a laboratory contamination problem as evidenced by the large percentage difference.

SECTION 4

SAMPLING LOCATION AND TEST METHODS

A four-train (quad) sampling system was used to collect samples in the breeching connecting the glass melting furnace to the exit stack. This system allows four trains to sample simultaneously at essentially a single point in the stack (see Figures 4-1 and 4-2). Therefore, this system reduces the effect of variations in the velocity and particulate profiles on the sampling results. It also permits a statistically significant number of samples to be taken in a short amount of time. Further, since all five trains are identical for every run, the within-train precision can be determined at the same time as the relationship of the different trains is being compared. This methodology for determining method precision was developed and validated in a previous EPA study.* A total of five quad-train runs representing 20 individual samples were collected. During these runs, a single Method 108 train was run with the sample nozzle positioned as close as possible to the quad nozzle unit.

4.1 SAMPLING LOCATION

All samples were extracted from a rectangular brick breeching connecting the furnace and stack. Figures 4-3 and 4-4 depict the sampling location.

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*Mitchell, W. J., and M. R. Midgett. A Means to Evaluate the Performance of Stationary Source Test Methods. ES and T, 10:85-88, 1976.
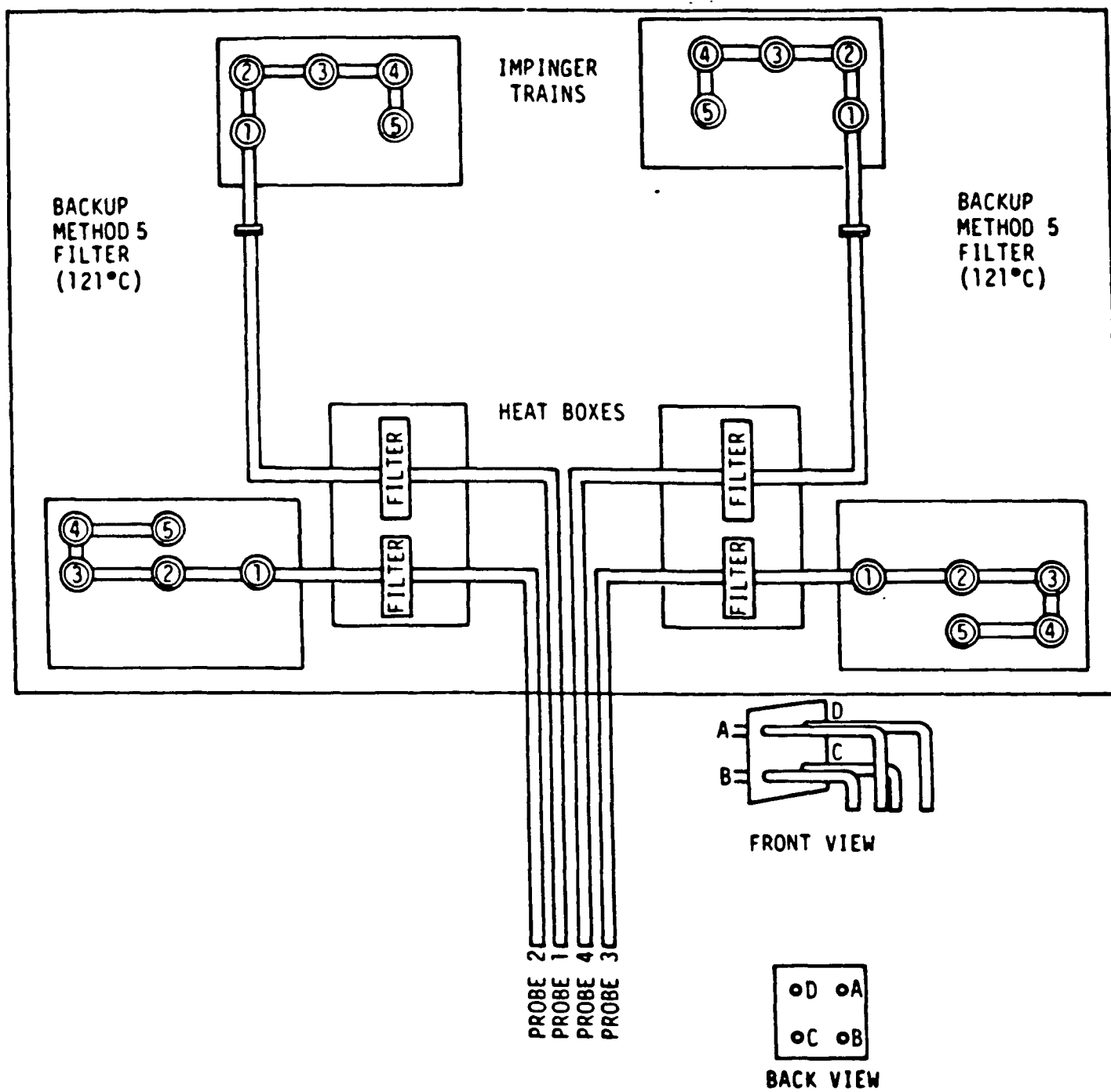
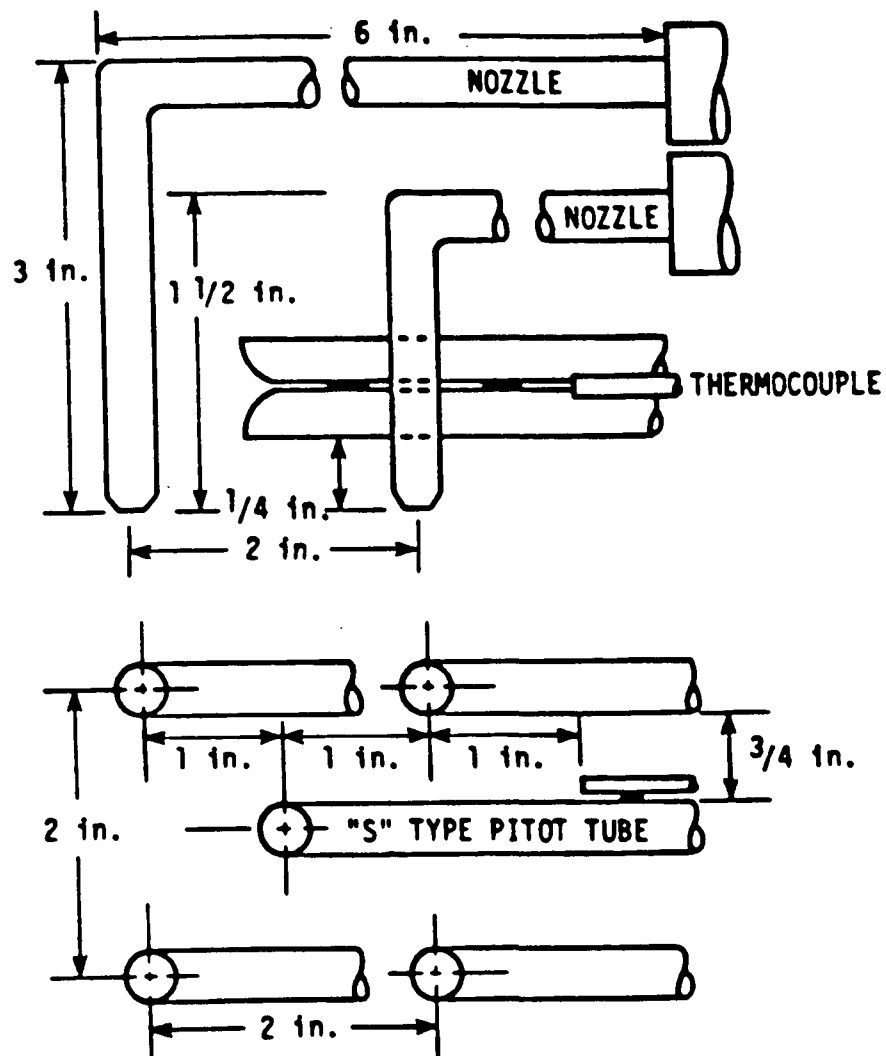Figure 4-1. Quad train system for elevated temperature tests.

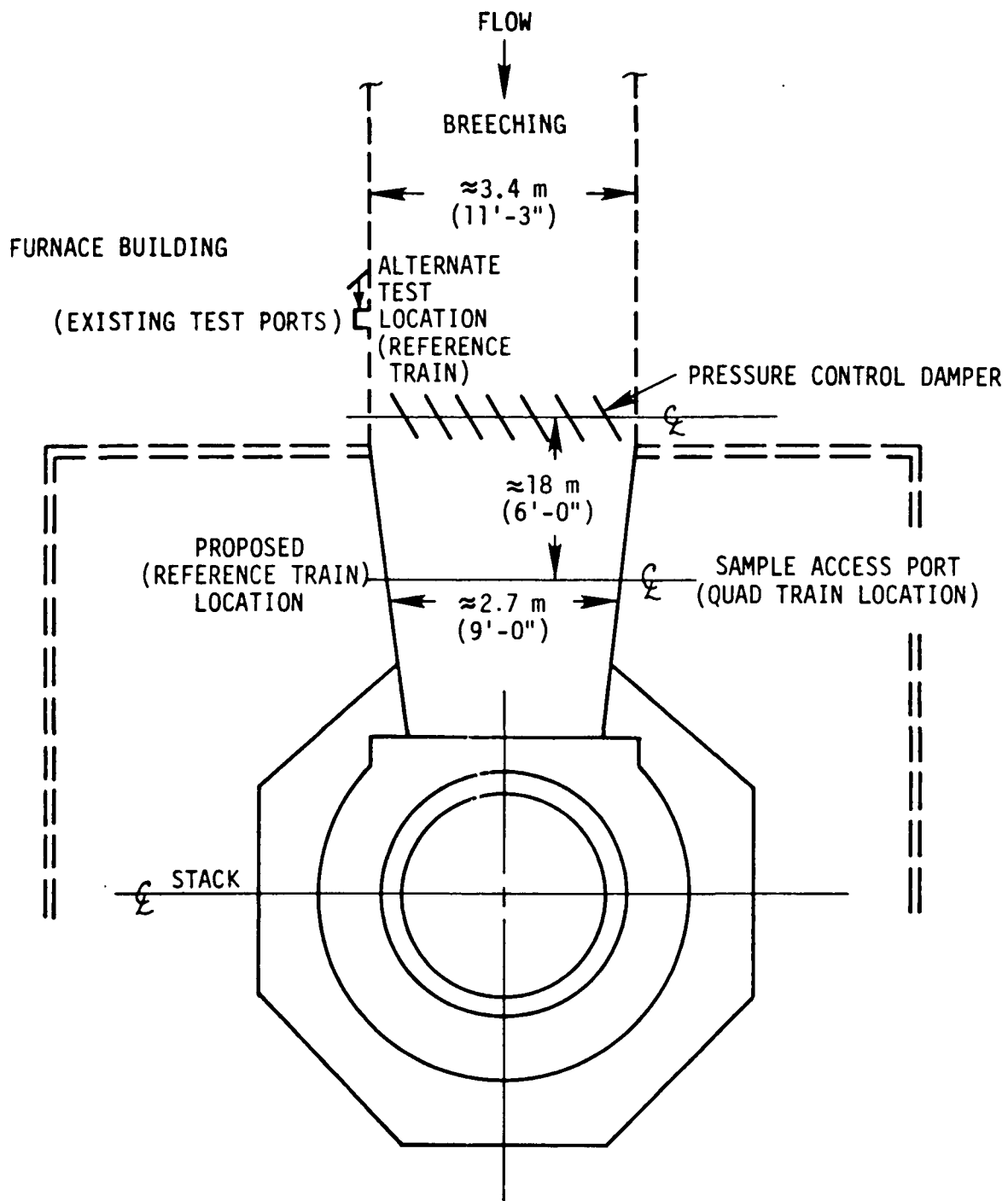Figure 4-2. Four-train sampling system showing nozzle, pitot tube, and thermocouple position.

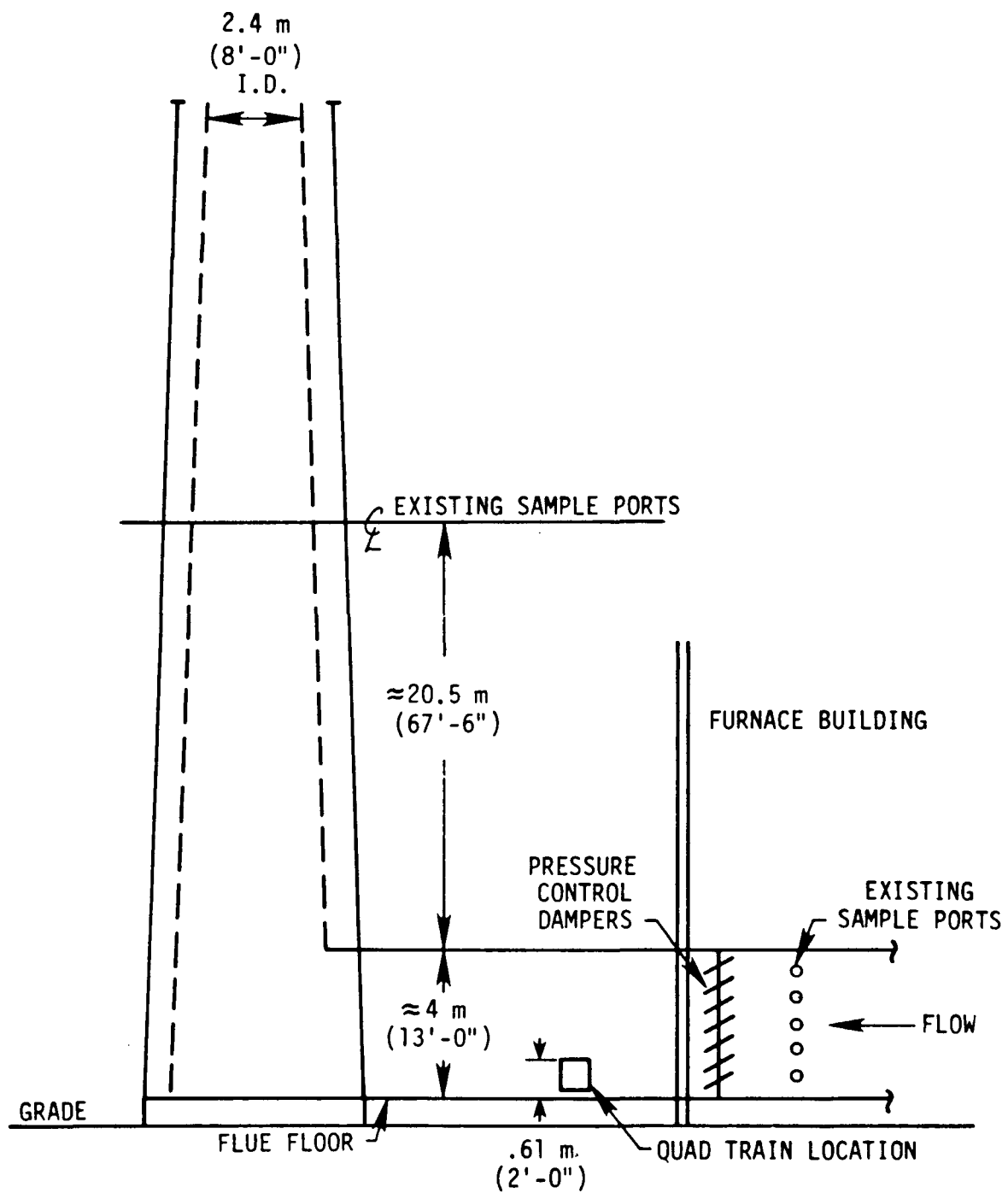Figure 4-3. Sampling location (plan view).

Figure 4-4. Sampling location (elevation).

Two sampling ports are located approximately 23.8 meters (78 feet) above grade in the tapered brick-lined stack. Based on the pre-test site survey, the sampling platform was determined to be too small to accommodate the quad train sampling system to be used in these tests. As a result of the short lead time needed to conduct the tests and the expense involved in modifying the stack platform, an alternate location was selected for sample collection.

As depicted in Figures 4-3 and 4-4, a 35 x 46 cm (14 x 18 in.) access port was available on the south side of the breeching for the quad system. The opening was approximately 1.8 m (6 feet) downstream from a pressure control damper, and the distance from the top of the access port to the floor of the breeching was 61 m (24 in.). A visual inspection of the duct cross section showed no significant deposition of material on the floor of the breeching. The quad train probe system was inserted near the top of the access port so that the minimum distance between the quad probes and the duct floor was approximately 51 cm (20 in.). The quad nozzles were positioned at least 76 cm (30 in.) inside the duct for each test. The single Method 108 train was inserted on the opposite side of the breeching at approximately the same level as the quad probes. By locating the reference train as close as possible to the quad probe system, a direct comparison can be made between arsenic distribution and sample temperature. In Quad Runs 1 and 2, a 2.4-m (8-ft) glass-lined probe was used in the reference train tests so that the reference train sample nozzle was positioned approximately 30.5 cm (12 in.) from the quad nozzles. In Quad Runs 3 through 5, a 1.5-m (5-ft) glass-lined probe was used in the reference train so that the distance between the reference and quad train nozzles was approximately 122 m (48 in).

Single-point, isokinetic sampling techniques were employed in each quad and reference train test. The sampling time for all tests was 90 minutes, and readings of stack flue gas and sampling train data were recorded at 5-minute intervals for each quad train and at 10-minute intervals for the reference train. A pitot tube and thermocouple attached to the quad and reference train probes were used to set isokinetic sampling rates for each train. Sampling rates were determined using programmable calculators. Prior to sampling, velocity and temperature measurements were made to define sampling rates and nozzle sizes.

In each train, the probe and filter temperatures were set at the pre-determined temperature and monitored using multiterminal digital indicators with thermocouple leads located in each probe and immediately behind the Method 5 filter frits.

4.2 SAMPLING AND ANALYTICAL PROCEDURES

The sampling and analytical procedures used in this test program followed those described in EPA Reference Methods 1 through 4* and proposed Method 108 as detailed in the site test plan prepared by PEI and reviewed and approved by EMB. The procedures, which are described briefly here, are detailed in Appendix D.

4.2.1 Velocity and Gas Temperature

A Type-S pitot tube and an inclined draft gauge manometer were used to measure gas velocity pressures at the test site. Temperature was measured with a thermocouple and digital readout.

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*40 CFR 60, Appendix A, Reference Methods 1 through 4, July 1984.

4.2.2 Molecular Weight

Flue gas composition was determined in accordance with the basic procedures described in Reference Method 3.* Grab samples were collected before any sampling began in order to establish baseline contents of oxygen, carbon dioxide, and carbon monoxide. Bag samples were collected periodically during sampling and analyzed with an Orsat gas analyzer.

Method 108* was used to measure arsenic concentration except that the impingers containing hydrogen peroxide (H_2O_2) for SO_2 determination were replaced with distilled H_2O because of low (less than 30 ppm) concentrations of SO_2. All tests were conducted isokinetically by regulating the sampling flow rate relative to the gas velocity in the stack as measured by the pitot tube and thermocouple attached to the quad probe arrangement (see Figure 4-2). Each individual sampling train consisted of a heated glass-lined probe, a heated 7.6-cm (3-in.) diameter glass fiber filter (Whatman Reeve Angel 934AH), and a series of five Greenburg-Smith impingers followed by a vacuum line, vacuum gauge, leak-free vacuum pump, dry gas meter, thermometers, and a calibrated orifice. In each train, probe and filter temperatures were monitored using digital indicators and thermocouple leads located in each probe and immediately behind the Method 108 filter frit. In the quad runs, a 53-cm (21-in.) glass connector was used to attach the front filter to a backup filter maintained at approximately 121°C. The impingers followed the backup filter for these trains.

The amount of water collected in the impinger section of the sampling train was measured gravimetrically at the end of each sample run to determine

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*Method 108 is proposed. 40 CFR 61, Appendix B, Method 108, July 1983.

the moisture content of the flue gas. The contents of the first three imping-
ers, each of which had been charged initially with 150 ml of distilled water,
were transferred to separate polyethylene containers. These impingers and all
associated connecting glassware were rinsed with 0.1 N NaOH; the rinses were
then added to the appropriate container(s). The contents of the fourth
impinger and 0.1 N NaOH rinse were placed in the container for the third
impinger.

All sample fractions were prepared using procedures described in EPA
Method 108 and analyzed by atomic absorption (AA) spectroscopy.

SECTION 5

PROCESS DESCRIPTION

The off-gases from a glass melting furnace (designated Tank No. 161) were tested. All samples were collected in the rectangular breeching connecting the furnace to the exit stack.

Personnel from Radian Corporation (an EPA Contractor) monitored the process operation during the test series. A description of the process and the operating parameters monitored during the test period is considered confidential by Corning Glass Works and will be treated as such, pending determination by the EPA.