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Background Report Reference

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No. 4, February 1994

US EPA

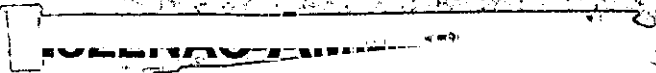
June 1995



NEW ENGLAND AIR QUALITY TESTING

AP-42 Section 11.26
Reference _____
Report Sect. 4
Reference 2

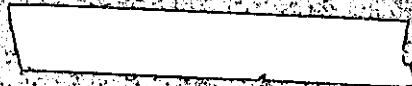
- Source Emissions Testin
- Source Permitting
- Ambient Air Sampling
- Fugitive Emissions Measurement
- Indoor Air Sampling and Analysis
- Consulting/Engineering Services



STACK EMISSIONS MEASUREMENTS

Prepared by:

New England Air Quality Testing



STACK EMISSIONS MEASUREMENTS

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STACK EMISSIONS MEASUREMENTS

1.0 INTRODUCTION

In January of 1994, New England Air Quality Testing (NEAQT) was contracted by [redacted] to quantify Total Suspended Particulate (TSP) and metals (As, Cd, Cr-VI, Ni) emissions from the Gamma Mill baghouse exhaust air at the facility.

Stack emissions sampling was conducted by NEAQT on January 21, 1994. One sample was collected from the baghouse exhaust air and analyzed for the aforementioned constituents at Endyne, Inc., an EPA-certified laboratory. In addition, a sample of the crushed ore product and baghouse fines were collected and analyzed for the same constituents. The following report includes the results of the sampling event with detailed descriptions of the sampling and analytical methods used.

2.0 SOURCE DESCRIPTION

The [redacted] facility in [redacted] contains a Roller Mill which crushes ore into a fine powder. The powder is circulated pneumatically from the Roller Mill through ductwork where it is separated based on particle size. The larger particles are recirculated to be crushed further, while the remainder of the crushed ore passes through additional ductwork where it is either classified as product and removed from the loop or continues on to be recirculated to the roller mill or exhausted through a baghouse.

3.0 SAMPLING AND ANALYTICAL METHODS

Air sampling for TSP and metals (As, Cd, Cr-VI, Ni) was conducted in accordance with the provisions of EPA Method 17 - "Determination of Particulate Emissions from Stationary Sources: (In-Stack Filtration Method)" modified for metals sampling with the substitution of teflon components rather than stainless steel in the sampling train.

The mass of TSP collected during sampling was determined as defined in EPA Method 17, while the mass of metals was determined in accordance with the provisions of NIOSH Method 7300 - "Elements". Stack gas velocity and volumetric flowrates were determined in accordance with EPA Method 1 - "Sample and Velocity Traverses For Stationary Sources" and EPA Method 2 - "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)". The EPA and NIOSH Methods are further described in the sections that follow. Copies of each of the aforementioned standardized methods are included in Appendix 3, pages 1-27.

Testing was conducted by the following New England Air Quality Testing personnel:

David E. Adams	Project Engineer
Curtis J. Puisto	Senior Air Quality Technician

Facility access, production schedule coordination, and operational information were arranged and overseen by Mr. Tim Hicks of Luzenac America, Inc.

3.1 Sample Location

The sampling locations as well as velocity traverse measurement points were selected according to guidelines set forth in Method 1. This method applies to stacks or ducts which are greater than 12 inches in diameter. Air sampling and velocity measurements were conducted at the same ports in the stack.

Samples were collected from a 19" diameter duct with a circular cross-section which exhausts air from the baghouse. The sample ports were located in a horizontal stretch of duct at a location greater than 8 diameters downstream and greater than 2 diameters upstream of the nearest flow disturbance.

3.2 Volumetric Flow

Velocity traverses and sample collection were performed at identical locations. In accordance with EPA Method 1, velocity traverses were performed at a site located greater than two duct diameters downstream and greater than a half diameter upstream from a flow disturbance. The sample port locations met the upstream and downstream distance from flow disturbance criteria. Therefore, sampling ports were accessed to perform velocity traverses. In addition, EPA Method 1 dictates that for the 19" duct sampled, a minimum of 16 velocity traverse points are required (2 transects, 90° opposed x 8 traverse points/transect). A cross-sectional view of the duct and the traverse point layout for the velocity measurements is shown on page 1 of Appendix 1. The volumetric flow of the gas stream was measured in accordance with Method 2. Velocity traverse data is shown on page 3 of Appendix 1.

Gas composition was assumed to be that of ambient air (79.1% N₂, 20.9% O₂, 0% CO₂).

3.3 Sampling Procedures

Sampling for TSP and metals (As, Cd, Cr-VI, Ni) was conducted in accordance with EPA Method 17 modified to allow for metals analysis. EPA Method 17 specifies that the sampling train be fitted with stainless steel equipment while collecting TSP samples. However, the use of stainless steel may bias sample results when analyzing for metals. Therefore, EPA Method 17 was modified through the use of teflon equipment to replace stainless steel in the sampling train. The sampling train set-up was identical to that specified in the Method, as illustrated in Appendix 3, page 5.

Samples were collected isokinetically to determine concentrations of TSP and metals during the test run. The test run was 320 minutes in duration and collected 98.097 dry standard cubic feet.

The Particulate Field Data Form is included on page 2 of Appendix 1. Information derived from a nomograph during sampling is included on page 4 in Appendix 1.

3.3.1 Calibration Procedure

Flowrates for sampling equipment used to collect samples were calibrated according to requirements as indicated in EPA's - "QA Handbook for Air Pollution Measurement Systems: Volume III. Stationary Sources Specific Methods" (EPA 600/4-77/027). The standard pitot tube used to determine the volumetric flow rate met all criteria outlined in EPA Method 2.

3.3.2 Sample Collection

Following modified EPA Method 17, a sample was withdrawn from the stack isokinetically through an in-stack teflon nozzle and teflon filter holder containing a tare weighed 47mm glass fiber filter. Gaseous metals which pass through the filter were collected in a teflon impinger train containing a nitric acid/hydrogen peroxide absorbing solution. The volume of stack gas sampled was metered through an Anderson console containing a rotary vane pump, a calibrated dry gas meter, and an orifice manometer to determine the flowrate required to maintain isokinetic sampling conditions.

3.4 Analytical Procedures

Total Suspended Particulate gravimetric analysis was conducted by NEAQT. Air sample analysis for metals was performed by Endyne, Inc., of Williston, VT. All samples were delivered by NEAQT personnel to Endyne, Inc.

3.4.1 Sample Analysis

Gravimetric analysis was performed by NEAQT following specifications outlined in EPA Method 17 to determine the mass of Total Suspended Particulate collected during each of the two sample runs. A Mettler AJ100 Balance was used for total mass determination. Sample and Tare Weight Data Sheets for filters and beakers used during sampling are included in Appendix 1, pages 5-7.

Sample analysis for metals (As, Cd, Cr-VI, Ni) was performed by Endyne, Inc. In addition, total chromium was reported by Endyne for each sample, as requested by NEAQT. Upon completion of gravimetric analysis for TSP determination at NEAQT's facility, samples were delivered to Endyne, Inc.

All metals analyses were performed in accordance with NIOSH Method 7300. NIOSH Method 7300 involves chemical filter digestion, followed by Inductively Coupled Plasma (ICP) analysis.

3.4.2 Quality Assurance

Analytical method precision and accuracy were monitored by laboratory control standards which included matrix spike, duplicate, and quality control analysis.

Laboratory analytical results and Chain of Custody are included in Appendix 2, pages 1-7.

4.0 RESULTS

Tables 1 - 5 included on the following pages of the text present a summary of the data collected and results obtained. Table 1 contains a summary of analytical results for metals detected in the product and baghouse fines. Table 2 illustrates the velocity and corresponding flowrate measured during the test run at the velocity traverse location. Table 3 lists emissions results. Table 4 provides a comparison between the average emission rates from the baghouse exhaust and the State of Vermont action levels for each constituent.

4.1 Calculations

Sample calculations are included on pages 8-11 of Appendix 1. The sample computations contain mass calculations for the metals and TSP. In addition, sample volume, volume of water vapor, stack moisture content, TSP and metals concentrations, stack gas velocity and volumetric flowrate, isokinetic calculations, and mass emission rates calculations are included in the sample calculations section of Appendix 1.

4.2 Discussion

Samples of crushed ore product as well as baghouse fines were collected and analyzed for As, Cd, Cr, Cr-VI, and Ni to determine concentrations of each of these metals in the process stream, upstream of the baghouse exhaust.

TABLE 1 - PRODUCT/BAGHOUSE FINES METALS

Analyte	Product Metal Concentrations (mg/kg)	Baghouse Fines Concentrations (mg/kg)
As	1.55	3.32
Cd	0.408	0.339
Cr	6.53	12.6
Cr-VI	<0.094 ¹	<0.100 ¹
Ni	207	244

¹ - Value represents the lower analytical detection limit.

Table 1 illustrates the concentrations of metals present in the product and baghouse fines. Chromium-VI was not present at concentrations above the analytical detection limit of 0.094 mg/kg and 0.100 mg/kg for the product and baghouse fines, respectively. These results illustrate that the hexavalent form of Cr (Cr-VI) is less than 1.4% of the total chromium present in the product, and less than 0.8% in the baghouse fines.

TABLE 2 - VELOCITY/FLOWRATE MEASUREMENTS

Test Run	Velocity (ft/sec)	Flowrate (scfm, dry)	% Isokinetic
1-1	54.10	4916.02	100.13

The measured velocity and corresponding stack flowrate for Test Run 1-1 are indicated in Table 2. The %isokinetic results are also included in Table 2.

TABLE 3 - TEST RUN 1-1 RESULTS

Run Time (min)	Analyte	Mass (ug)	Volume (dscf)	Actual Conc. (gr/dscf)	Emission Rate (lb/8-hr)
320	TSP	0.0218 g	98.10	0.00343	0.144 lb/hr
320	As	0 ¹	98.10	0	0
320	Cd	0.477	98.10	7.503×10^{-8}	2.530×10^{-5}
320	Cr	0.94 ¹	98.10	1.479×10^{-7}	4.986×10^{-5}
320	Cr-VI	<1.94 ²	98.10	$<3.051 \times 10^{-7}$	$<1.029 \times 10^{-4}$
320	Ni	13.4 ¹	98.10	2.108×10^{-6}	7.105×10^{-4}

¹ - corrected to account for the presence of the analyte in the blank

² - value represents the lower analytical detection limit

The Test Run 1-1 sample results are summarized in Table 3. A 98.097 dscf volume sample was collected during a 320-minute period. The concentration and mass emission rate for each of the metals and TSP are shown in Table 3. The mass of Chromium-VI collected on the sampling media was not present in quantities above the analytical detection limit of 1.94 ug. Based on the volume sampled and the stack gas flowrate, the Cr-VI analytical detection limit represents an emission rate of 1.029×10^{-4} lb/8-hr.

The mass of As, Cd, and Ni reported in Table 3 have been corrected to account for analytes present in the field blank (see the field blank laboratory report - Appendix 2, page 3).

TABLE 4 - AVERAGE EMISSION RATES AND ACTION LEVELS

Analyte	Emission Rate (lb/8-hr)	Action Level (lb/8-hr)	% of Action Level
TSP	0.144 lb/hr	12.50 lb/hr	1.2
As	0	0.000019	0
Cd	2.530×10^{-5}	0.000047	53.8
Cr	4.986×10^{-5}	0.01	0.5
Cr-VI ¹	$<1.029 \times 10^{-4}$	0.0000071	<1449
Ni	7.105×10^{-4}	0.00026	273.3

¹ Cr-VI was not detected in the sample; see discussion on page 8.

Table 4 contains values for the Gamma Mill emission rates, State of Vermont action levels (VT - Air Pollution Control Regulations: August 13, 1993), and % of the action level measured for each of the analytes in question. Action levels for each of the metals are designated in units of lb/8-hr. However, the regulatory discharge level for TSP must be reported in units of lb/hr, and is determined based on the input process weight (lb/hr). Mr. Tim Hicks indicated that the average input process weight for the Gamma Mill is 7 tons/hour. This corresponds to a maximum discharge weight of 12.50 lb/hr (see Table 1 of the aforementioned Regulations).

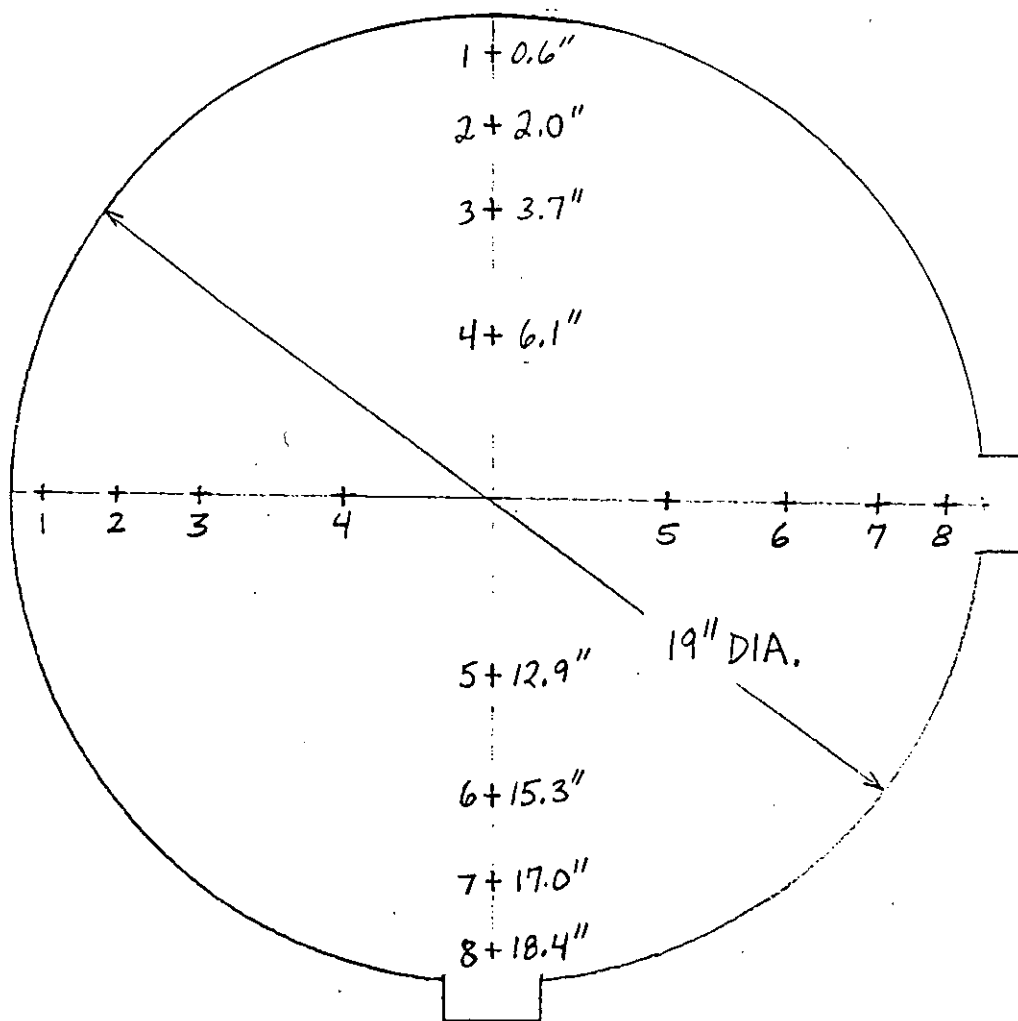
Sampling parameters, including sample volume (98.097 dscf) and run time (320 minutes) were increased during the January 21, 1994 sampling event relative

to prior sampling conducted by NEAQT at the Luzenac-Ludlow facility. In addition to collecting a larger sample volume, NEAQT worked with Endyne to reduce detection limits in an effort to quantify mass emission rates at or below Vermont action levels. As indicated in Table 4, detectable quantities of As, Cd, Cr, and TSP were measured at values below Vermont regulatory levels. Nickel was detected at 273.3 percent of the action level.

Chromium-VI was not detected in the exhaust gas. Total chromium concentrations are reported to provide additional information regarding chromium emissions, due to the fact that the Cr-VI detection limit is above the action level. As indicated in Table 1, less than 0.8% of the total Cr present in the baghouse fines is in the hexavalent form (Cr-VI). A direct correlation between the baghouse fines Cr-VI/Cr ratio and the known mass emission rate of total chromium yields an emission rate for Cr-VI. Total Cr is emitted from the facility at a rate of 4.986×10^{-5} lb/8-hr. With a less than 0.8% Cr-VI/Cr ratio, the Cr-VI emission rate is less than 3.99×10^{-7} lb/8-hr which is 5.6% of the Cr-VI action level (7.1×10^{-6} lb/8-hr).

Based on the results of the January 21, 1994 emissions test at the Luzenac-Ludlow facility, nickel is the only test constituent present in the Gamma Mill exhaust at concentrations above State of Vermont action levels.

[RPT2-LUDLOW/DA 1-1-94]



NEW ENGLAND
AIR QUALITY TESTING
Burlington, Vermont

TRAVERSE POINT LAYOUT

DATE: 1/17/94 SCALE:

NTS DRN: CJP APPD: DA

PARTICULATE FIELD DATA FORM

PLANT

CITY

DATE

SOURCE

TEST/RUN

OPERATOR

STACK

METER BOX ID

METER FACTOR

METER Δ HE

PITOT FACTOR

BAR. PRESS.

NOZZLE SIZE

LEAK CHECK PRE

POST

PITOT CHECK PRE

POST

392-644

0.999

1.77

0.99

28.72

0.250

0.015 @ 16" Hg

0.000 @ 10" Hg

—

FILTER # 47-0

IMP 1 100 GEL 200

IMP 2 100 212.0

IMP 3 0

— sheer
imping
H₂O,
of fuel

TRAVERSE POINT NUMBER	TIME clk/smp1	VEL. HEAD Δ P "H ₂ O	ORIFICE PRESS DIFF		METER VOL. cu ft	STACK TEMP °F	PROBE TEMP °F	DRY GAS METER TEMPERATURE F		OVEN TEMP °F	IMP TEMP °F	PUMP VAC "Hg
			CALC	REAL				INLET	OUTLET			
1	1107.0	0.33	1.10	0.24	60.676	173	—	82	54	—	—	6.0
2	1127.20	0.44	2.05	0.27	67.138	—	—	84	56	—	—	7.0
3	1147.40	0.47	2.00	0.29	73.32	—	—	80	57	—	—	7
4	1207.60	0.49	2.30	0.30	79.000	—	—	52	43	—	—	9
5	1206.80	0.60	2.00	0.37	90.43	—	—	77	47	—	—	9
6	1246.100	0.62	2.40	0.38	97.53	—	—	80	51	—	—	9
7	1706.120	0.57	2.74	0.35	104.65	—	—	79	52	—	—	9
8	1726.140	0.51	2.44	0.33	116.52	—	—	73	50	—	—	8
END	1746.160	—	—	—	118.175	—	—	—	—	—	—	—
SIDE 1	1755.160	0.50	0.32	0.32	118.175	—	—	84	66	—	—	7.5
2	1815.180	0.54	0.34	0.34	121.5	—	—	88	74	—	—	7
3	1835.200	0.58	0.46	0.36	130.34	—	—	93	77	—	—	7
4	1855.220	0.57	0.36	0.36	137.47	—	—	102	83	—	—	8
5	1915.240	0.58	0.36	0.36	144.70	—	—	107	89	—	—	8
6	1985.260	0.60	0.36	0.36	151.9	—	—	111	93	—	—	8
7	1995.280	0.56	0.35	0.35	159.40	—	—	113	96	—	—	8
8	2015.300	0.45	0.28	0.28	166.70	—	—	112	97	—	—	8
END	2035.320	—	—	—	173.331	—	—	—	—	—	—	—

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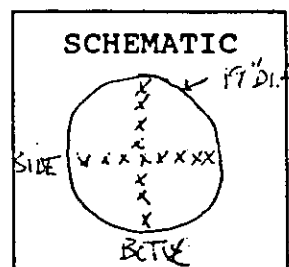
* Restart @ 1600 w/ VOL = 23.800

pump problems 0.3 DH WAY

VELOCITY TRAVERSE DATA SHEET

PLANT _____
 CITY _____
 SOURCE Gamma Mill
 DATE 1/21/94
 RUN 1-1
 PITOT ID _____
 COEFFICIENT 0.99
 BAR PRESSURE 28.72
 OPERATORS CP/DA

% O2 20.9
 % CO2 0
 % N2 79.1
 % H2O ± 2.0



Traverse Point #	Velocity Head "H ₂ O Δp	Stack Temp °F	Cyclonic Flow Angle °	Static Pressure "H ₂ O	$\sqrt{\Delta p}$
BOTT. 1	0.38	178	NA	+0.24	0.616
2	0.44				0.663
3	0.47				0.686
4	0.49				0.700
5	0.60				0.775
6	0.62				0.787
7	0.57				0.755
8	0.51				0.714
SIDE 1	0.50				0.707
2	0.54				0.735
3	0.58				0.762
4	0.57				0.755
5	0.58				0.762
6	0.60				0.775
7	0.56				0.748
8	0.45				0.671

AVG: 0.53

0.726

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CA

NOMOGRAPH DATA FORM

PLANT _____
 CITY _____
 DATE 1/21/94
 SOURCE Gamma

from QA Handbook M5-4.1

Calibrated Orifice Pressure Differential "H ₂ O	ΔH_o	1.77
Meter Temperature Average °F	T_m	100
Moisture in Gas Stream %	B_{wo}	2
Barometric Pressure "Hg	P_b	28.72
Stack Static Pressure "H ₂ O	P_s	+0.24
Static/Barometric Pressure Ratio	P_s/P_b	—
Stack Temperature Average °F	T_s	178
Velocity Head Average "H ₂ O	ΔP_{avg}	0.53
Velocity Head Maximum "H ₂ O	ΔP_{max}	0.62
C Factor		0.99
Calculated Nozzle Diameter "	D_n	0.225
Actual Nozzle Diameter "	D_n	0.15 0.25
Reference Δp "H ₂ O @ 0.53 Δp $\Delta H =$ 2.5 ^{0.33}	—	—

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$$\sqrt{\frac{2.5}{1.77}} \times 0.75 = 0.89 \text{ CFM}$$

TARE WEIGHT LOG SHEET

Project: _____
 Facility: UNKNOWN - TASK 2

Beaker/Filter size: 250ml

FILTER/ BEAKER NUMBER	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME
	mass g	mass g	mass g	mass g	mass g
QC Blanks 31	1/24 0800	1/24 1430	1/25 0800	1/25 1430	1/26 0815
	100.0037g	100.0022g	99.9999 g	100.0020	100.0000g
QC Blanks 28			1/25	1/25 1430	
	104.4393g	104.4419g	104.4403	104.4420	104.4400g
* sample 25/4			1/25 0800	1/25 1430	
		101.3107	101.3080	101.3106	101.3081g
* FID BRK 25/5			1/25 0800	1/25 1430	
		100.2762	100.2737	100.2760	100.2739-g
			VOID A.M.		VOID A.M.

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SAMPLE WEIGHT LOG SHEET

Project:

Facility:

Task 2

Beaker/Filter size:

250 ml

[illegible]

NEW ENGLAND AIR QUALITY TESTING

SAMPLE WEIGHT LOG SHEET

Project:
Facility:

Task 2

Beaker/Filter size:

47 mm Glass Fiber Filter

FILTER/ BEAKER NUMBER	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME	DATE/TIME
	mass g	mass g	mass g	mass g	mass g
47-07 TIR 1-1	tare	1/25/94 0800	1/25/ 1430	NET	
	0.0891	0.1012	0.1012g	0.0121g	

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NEW ENGLAND AIR QUALITY TESTING



PAGE 1 OF 4

PROJECT: _____

DATE: _____

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Page No.

Metal mass calculations

T/R 1-1

- a. As : $1.48 - 1.72 < 0 \mu\text{g}^1$
- b. Cd : $0.477 \mu\text{g}$
- c. Cr : $2.55 - 1.61 = 0.94 \mu\text{g}^1$
- d. Cr VI : $< 1.94 \mu\text{g}$
- e. Ni : $19.7 - 6.30 = 13.4 \mu\text{g}^1$

¹ - Mass corrected to account for blank contamination

Product

- a. As : 1.55 mg/kg
- b. Cd : 0.408 mg/kg
- c. Cr : 6.53 mg/kg
- d. Cr-VI : $< 0.094 \text{ mg/kg}$
- e. Ni : 207 mg/kg

Baghouse Fines

- a. As : 332 mg/kg
- b. Cd : 0.339 mg/kg
- c. Cr : 12.6 mg/kg
- d. Cr-VI : $< 0.100 \text{ mg/kg}$
- e. Ni : 244 mg/kg

NEW ENGLAND AIR QUALITY TESTING



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TSP mass calculations

T/R 1-1

$$\begin{aligned} \text{TSP} &= \text{Filter mass (final - tare)} + \text{beaker mass (final - tare)} \\ &= (0.1012 - 0.0891) + (101.3203 - 101.3106) \end{aligned}$$

$$\text{TSP} = 0.0218 \text{ g}$$

$$\text{BLANK} = 0.0000 \text{ g}$$

T/R 1-1 Additional Calculations

I. Standard meter volume

Eg. 5-10
or Eg. 4-3

$$V_{M(\text{std})} = K \cdot V_M \cdot \frac{P_M}{T_M} = K \cdot V_M \cdot \frac{(P_{\text{bar}} + \Delta H_{\text{AVE}}/13.6)}{T_{M(\text{AVE})}}$$

$$\text{where } K = 17.64^\circ \text{R/in Hg}$$

$$V_{M(\text{std})} = \left(\frac{17.64^\circ \text{R}}{\text{in Hg}} \right) (108.341 \text{ ft}^3) \left(\frac{28.72 + 0.33/13.6}{100 + 460^\circ \text{R}} \right) = 98.097 \text{ scf}$$

II. Volume of H₂O vapor

Eg. 5-2
or Eg. 4-1

$$V_{W(\text{std})} = K (V_t - V_i)$$

$$\text{where } K = 0.04707 \text{ ft}^3/\text{ml}$$

$$V_{W(\text{std})} = \left(0.04707 \frac{\text{ft}^3}{\text{ml}} \right) (69 \text{ ml}) = 3.248 \text{ ft}^3$$

III. % Moisture in stack gas

Eg. 5-3
or Eg. 4-4

$$B_{WS} = \frac{V_{W(\text{std})}}{V_{M(\text{std})} + V_{W(\text{std})}} = \frac{3.248 \text{ ft}^3}{98.097 \text{ ft}^3 + 3.248 \text{ ft}^3} = 0.032$$

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IV Concentration of metals and TSP

$$\text{Eq. 5-6 } C_s = \left(\frac{0.001 \text{ g}}{\text{mg}} \right) \left(\frac{M_n \text{ mg}}{V_m \text{ (STD)}} \right) \left(\frac{15.43 \text{ gr}}{\text{g}} \right) = \text{gr/dscf}$$

$V_m \text{ (STD)} = 98.097 \text{ dscf}$

a. As: $0 \text{ mg} = 0 \text{ grains/dscf}$

b. Cd: $0.000477 \text{ mg} = 7.503 \times 10^{-8} \text{ grains/dscf}$

c. Cr: $0.00094 \text{ mg} = 1.479 \times 10^{-7} \text{ grains/dscf}$

d. Cr-VI: $< 0.00194 \text{ mg} = < 3.051 \times 10^{-7} \text{ grains/dscf}$

e. Ni: $0.0134 \text{ mg} = 2.108 \times 10^{-6} \text{ grains/dscf}$

f. TSP: $21.8 \text{ mg} = 3.429 \times 10^{-3} \text{ grains/dscf}$

V Stack gas velocity

$$\text{Eq. 2-9 } V_s = K_p C_p (\sqrt{\Delta P_{\text{avg}}}) \sqrt{\frac{T_s \text{ (avg)}}{P_s M_s}}$$

where $K_p = 85.49$

$$\text{Eq. 2-5 } M_s = M_d (1 - B_{ws}) + 18.0 B_{ws} = 29(1 - 0.032) + 18(0.032) = 28.65$$

where $M_d = 29.0$

$$\text{Eq. 2-6 } P_s = P_{\text{bar}} + P_g = 28.72 + 0.24/13.6 = 28.73 \text{ in Hg}$$

$$V_s = (85.49)(0.99)(0.726) \sqrt{\frac{178+460}{(28.73)(28.65)}}$$

$$V_s = 54.096 \text{ ft/sec}$$

VI Stack Volumetric Flow

$$\text{Eq. 2-10 } Q_{\text{STD}} = 60(1 - B_{ws}) V_s A \left(\frac{T_{\text{STD}}}{T_s \text{ (avg)}} \right) \left(\frac{P_s}{P_{\text{STD}}} \right)$$

$$Q_{\text{STD}} = 60(1 - 0.032)(54.096 \text{ ft/sec}) \left(\pi \left(\frac{19/2}{12} \right)^2 \right) \left(\frac{528^\circ \text{R}}{178+460^\circ \text{R}} \right) \left(\frac{28.73 \text{ in Hg}}{29.92 \text{ in Hg}} \right) = 49/6.02 \text{ dscfm}$$

NEW ENGLAND AIR QUALITY TESTING



PAGE 4 OF 4

Page No. 11

PROJECT: _____

DATE: _____

VII % Isokinetic

Eg. 5-8
$$I = \frac{K_4 T_s V_{M(STD)}}{P_s V_s A_n \Theta (1 - B_{ws})}$$

where $K_4 = 0.09450$

$$I = \frac{(0.09450)(178 + 460^\circ R)(98.097 \text{ dscf})}{(28.73 \text{ in. Hg})(54.096 \frac{\text{ft}}{\text{sec}})(\pi (\frac{0.15}{12})^2)(320 \text{ mm})(1 - 0.032)}$$

$$I = 100.12 \%$$

NOT A_n for 0.25" nozzle

VIII Emission Rate

i. $C_s \times \frac{\text{lb}}{7000 \text{ grains}} = \text{lb/dscf}$

ii. $\frac{\text{lb}}{\text{dscf}} \times Q_{STD} \times \frac{480 \text{ min}}{8 \text{ hr}} = \text{lb/8-hr}$ $Q_{STD} = 4916.02 \text{ dscfm}$

a. As: 0

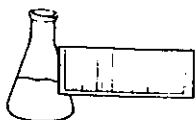
b. Cd: $7.503 \times 10^{-8} \text{ grains/dscf} = 1.072 \times 10^{-11} \frac{\text{lb}}{\text{dscf}} = 2.530 \times 10^{-5} \text{ lb/8-hr}$

c. Cr: $1.479 \times 10^{-7} \text{ grains/dscf} = 2.113 \times 10^{-11} \frac{\text{lb}}{\text{dscf}} = 4.986 \times 10^{-5} \text{ lb/8-hr}$

d. Cr-VI: $< 3.051 \times 10^{-7} \text{ grains/dscf} = < 4.359 \times 10^{-11} \frac{\text{lb}}{\text{dscf}} = < 1.029 \times 10^{-4} \text{ lb/8-hr}$

e. Ni: $2.108 \times 10^{-6} \text{ grains/dscf} = 3.011 \times 10^{-10} \frac{\text{lb}}{\text{dscf}} = 7.105 \times 10^{-4} \text{ lb/8-hr}$

f. TSP: $3.429 \times 10^{-3} \text{ grains/dscf} = 4.899 \times 10^{-7} \frac{\text{lb}}{\text{dscf}} = 0.144 \text{ lb/hr}$



ENDYNE, INC.

Laboratory Services

32 James Brown Drive
Williston, Vermont 05495
(802) 879-4333
FAX 879-7103

REPORT OF LABORATORY ANALYSIS

CLIENT: NEAQT
PROJECT NAME
DATE REPORTED: ~~SEPTEMBER 4, 1994~~
DATE SAMPLED:

PROJECT CODE: NEAQ3755
REF. #: 56,146 - 56,149

Enclosed please find the results of the analyses performed for the samples referenced on the attached chain of custody record.

Chain of custody indicated the samples were not preserved.

All samples were prepared and analyzed by requirements outlined in the referenced methods and within the specified holding times.

All instrumentation was calibrated with the appropriate frequency and verified by the requirements outlined in the referenced methods.

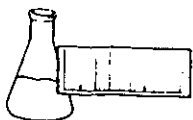
Sample results reflect adjustments made regarding slight blank contamination observed for total chromium only. For other metals, blank contamination was not observed at levels affecting the analytical results.

Analytical method precision and accuracy was monitored by laboratory control standards which included matrix spike, duplicate and quality control analyses. These standards were determined to be within established laboratory method acceptance limits.

Reviewed by,

Harry B. Locker, Ph.D.
Laboratory Director

enclosures



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FAX 879-7103

LABORATORY REPORT

CLIENT: NEAQT
PROJECT NAME:
REPORT DATE: 1 January 7, 1994
DATE SAMPLED: January 7, 1994
DATE RECEIVED: January 7, 1994

PROJECT CODE: NEAQ3755
REF.#: 56,146
STATION: A525 Sample
TIME SAMPLED: Not Indicated
SAMPLER: C. Puisto

Tested parameters are reported in micrograms. Digestion was performed by NIOSH Method 7300. Hexavalent chromium was digested by EPA Method 3060.

<u>Parameter</u>	<u>Concentration</u>	<u>EPA Method</u>	<u>Analysis Date</u>
Total Arsenic	1.48	7060	2/3/94
Total Cadmium	0.477	6010	2/3/94
Total Chromium	2.55	6010	2/2/94
Total Chromium VI	<1.94	7196A	2/2/94
Total Nickel	19.7	6010	2/3/94



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FAX 879-7103

LABORATORY REPORT

CLIENT: NEAQT
PROJECT NAME:
REPORT DATE:
DATE SAMPLED:
DATE RECEIVED

PROJECT CODE: NEAQ3755
REF.#: 56,147
STATION: A526 Field Blank
TIME SAMPLED: Not Indicated
SAMPLER: C. Puisto

Tested parameters are reported in micrograms. Digestion was performed by NIOSH Method 7300. Hexavalent chromium was digested by EPA Method 3060.

<u>Parameter</u>	<u>Concentration</u>	<u>EPA Method</u>	<u>Analysis Date</u>
Total Arsenic	1.72	7060	2/3/94
Total Cadmium	<0.210	6010	2/3/94
Total Chromium	1.61	6010	2/3/94
Total Chromium VI	DU ¹	7196A	2/2/94
Total Nickel	6.30	6010	2/3/94

Notes:

1 Data unavailable because analysis did not meet laboratory QA/QC standards.



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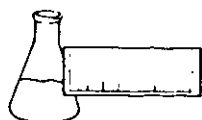
LABORATORY REPORT

CLIENT: NEAQT
PROJECT NAME:
REPORT DATE:
DATE SAMPLED:
DATE RECEIVED: January 20, 1994

PROJECT CODE: NEAQ3755
REF.#: 56,148
STATION: A527 Baghouse Fines
TIME SAMPLED: Not Indicated
SAMPLER: C. Puisto

Tested parameters are reported in milligrams per kilogram, dry weight. Digestion was performed by EPA Method 3050. Hexavalent chromium was digested by EPA Method 3060.

<u>Parameter</u>	<u>Concentration</u>	<u>EPA Method</u>	<u>Analysis Date</u>
Total Arsenic	3.32	7060	2/3/94
Total Cadmium	0.339	6010	2/3/94
Total Chromium	12.6	6010	2/3/94
Total Chromium VI	<0.100	7196A	2/2/94
Total Nickel	244.	6010	2/3/94



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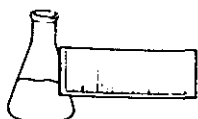
LABORATORY REPORT

CLIENT: NEAQT
PROJECT NAME:
REPORT DATE: 1
DATE SAMPLED:
DATE RECEIVED:

PROJECT CODE: NEAQ3755
REF.#: 56,149
STATION: A528 Product
TIME SAMPLED: Not Indicated
SAMPLER: C. Puisto

Tested parameters are reported in milligrams per kilogram, dry weight. Digestion was performed by EPA Method 3050. Hexavalent chromium was digested by EPA Method 3060.

<u>Parameter</u>	<u>Concentration</u>	<u>EPA Method</u>	<u>Analysis Date</u>
Total Arsenic	1.55	7060	2/3/94
Total Cadmium	0.408	6010	2/3/94
Total Chromium	6.53	6010	2/3/94
Total Chromium VI	<0.094	7196A	2/2/94
Total Nickel	207.	6010	2/3/94



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6.

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32 James Brown Drive
Williston, Vermont 05495
(802) 879-4333
FAX 879-7103

LABORATORY REPORT

MATRIX SPIKE AND DUPLICATE LABORATORY CONTROL DATA

CLIENT: NEAQT
PROJECT NAME:
REPORT DATE:
DATE SAMPLED:

PROJECT CODE: NEAQ3755

SPIKE QA/QC DATA:

<u>Parameter</u>	<u>Ref.#</u>	<u>Sample(mg/L)</u>	<u>Spike(mg/L)</u>	<u>% Recovery</u>
Arsenic	56,149	0.160	0.100	87.8
Cadmium	56,149	0.042	0.200	83.5
Chromium	56,149	0.673	0.400	117.
Chromium VI	56,149	<0.020	0.100	109.
Nickel	56,149	0.213	0.800	88.3

DUPLICATE QA/QC DATA:

<u>Parameter</u>	<u>Ref.#</u>	<u>Dup 1(mg/kg)</u>	<u>Dup 2(mg/kg)</u>	<u>Avg % Deviation</u>
Arsenic	56,148	3.21	3.43	3.
Cadmium	56,148	0.312	0.365	8.
Chromium	56,148	13.5	11.6	8.
Chromium VI	56,148	<0.099	<0.100	ND ¹
Nickel	56,148	236.	252.	3.

Notes:

1 None Detected

008770

Requested Analyses <i>ACG</i>												
1	pH	6	TKN	11	Total Solids	16	Metals (Specify)	21	EPA 624	26	EPA 8270 B/N or Acid	
2	Chloride	7	Total P	12	TSS	17	Coliform (Specify)	22	EPA 625 B/N or A	27	EPA 8010/8020	
3	Ammonia N	8	Total Diss. P	13	TDS	18	COD	23	EPA 418.1	28	EPA 8080 Pes/PCB	
4	Nitrite N	9	BOD ₅	14	Turbidity	19	BTEX	24	EPA 608 Pes/PCB			
5	Nitrate N	10	Alkalinity	15	Conductivity	20	EPA 601/602	25	EPA 8240			
29	TCLP (Specify: volatiles, semi-volatiles, metals, pesticides, herbicides)											
30	Other (Specify): <i>As, Cd, Cr VI, Ni</i>											

METHOD 1—SAMPLE AND VELOCITY TRAVERSES FOR STATIONARY SOURCES

1. Principle and Applicability

1.1 Principle. To aid in the representative measurement of pollutant emissions and/or total volumetric flow rate from a stationary source, a measurement site where the effluent stream is flowing in a known direction is selected, and the cross-section of the stack is divided into a number of equal areas. A traverse point is then located within each of these equal areas.

1.2 Applicability. This method is applicable to flowing gas streams in ducts, stacks, and flues. The method cannot be used when: (1) flow is cyclonic or swirling (see Section 2.4), (2) a stack is smaller than about 0.30 meter (12 in.) in diameter, or 0.071 m² (113 in.²) cross-sectional area, or (3) the measurement site is less than two stack or duct diameters downstream or less than a half diameter upstream from a flow disturbance.

The requirements of this method must be considered before construction of a new facility from which emissions will be measured; failure to do so may require subsequent alterations to the stack or deviation from the standard procedure. Cases involving variants are subject to approval by the Administrator, U.S. Environmental Protection Agency.

2. Procedure

2.1 Selection of Measurement Site. Sampling or velocity measurement is performed at a site located at least eight stack or duct diameters downstream and two diameters upstream from any flow disturbance such as a bend, expansion, or contraction in the stack, or from a visible flame. If necessary, an alternative location may be selected, at a position at least two stack or duct diameters downstream and a half diameter upstream from any flow disturbance. For a rectangular cross section, an equivalent diameter (D_e) shall be calculated from the following equation, to determine the upstream and downstream distances:

$$D_e = \frac{2LW}{(L+W)}$$

where L=length and W=width.

An alternative procedure is available for determining the acceptability of a measurement location not meeting the criteria above. This procedure, determination of gas flow angles at the sampling points and, comparing the results with acceptability criteria, is described in Section 2.8.

2.2 Determining the Number of Traverse Points

2.2.1 Particulate Traverses. When the eight- and two-diameter criterion can be met, the minimum number of traverse points shall be: (1) twelve, for circular or rectangular stacks with diameters (or equivalent diameters) greater than 0.61 meter (24 in.); (2) eight, for circular stacks with diameters between 0.30 and 0.61 meter (12-24 in.); (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12-24 in.).

When the eight- and two-diameter criterion cannot be met, the minimum number of traverse points is determined from Figure 1-1. Before referring to the figure, however, determine the distance from the chosen

measurement site to the nearest upstream and downstream disturbances, and divide each distance by the stack diameter or equivalent diameter, to determine the distance in terms of the number of duct diameters. Then, determine from Figure 1-1 the minimum number of traverse points that corresponds: (1) to the number of duct diameters upstream; and (2) to the number of diameters downstream. Select the higher of the two minimum numbers of traverse points, or a greater value, so that for circular stacks the number is a multiple of 4, and for rectangular stacks, the number is one of those shown in Table 1-1.

TABLE 1-1. CROSS-SECTION LAYOUT FOR RECTANGULAR STACKS

Number of Traverse Points	Stack Layout
9	3x3
12	4x3
18	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

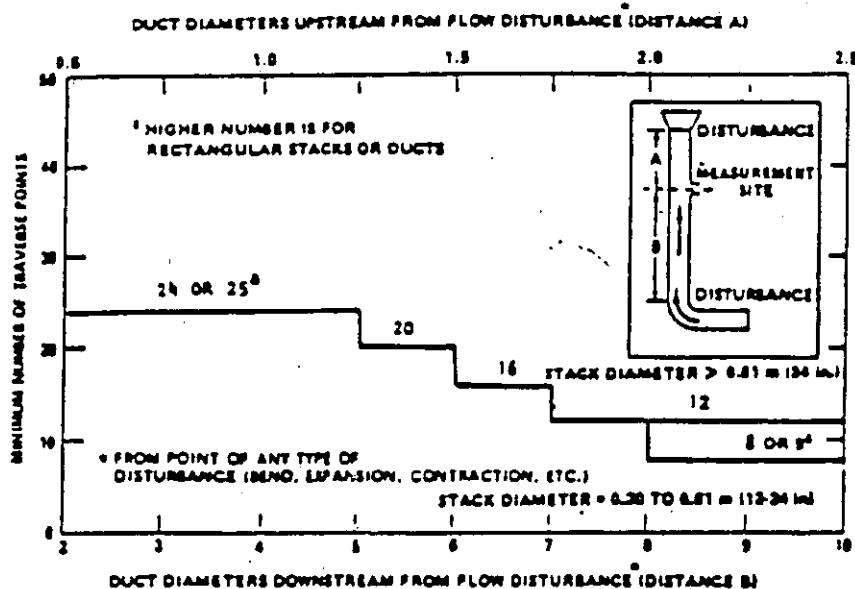


Figure 1-1. Minimum number of traverse points for particulate traverses.

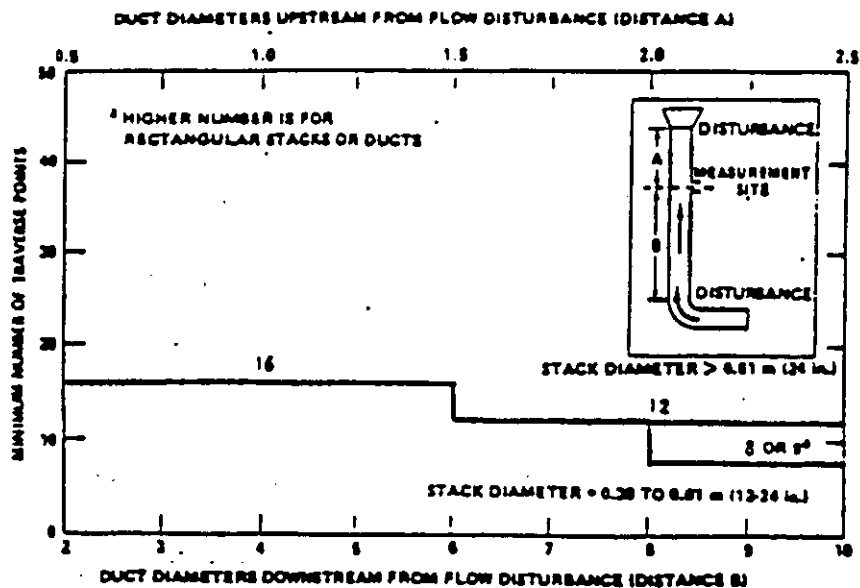


Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

2.2.2 Velocity (Non-Particulate) Traverses. When velocity or volumetric flow rate is to be determined (but not particulate matter), the same procedure as that for particulate traverses (Section 2.2.1) is followed, except that Figure 1-2 may be used instead of Figure 1-1.

2.3 Cross-sectional Layout and Location of Traverse Points.

2.3.1 Circular Stacks. Locate the traverse points on two perpendicular diameters according to Table 1-2 and the example shown in Figure 1-3. Any equation (for example, see Citations 2 and 3 in the Bibliography) that gives the same values as those in Table 1-2 may be used in lieu of Table 1-2.

For particulate traverses, one of the diameters must be in a plane containing the greatest expected concentration variation, e.g., after bends, one diameter shall be in the plane of the bend. This requirement becomes less critical as the distance from the disturbance increases; therefore, other diameter locations may be used, subject to approval of the Administrator.

In addition for stacks having diameters greater than 0.61 m (24 in.) no traverse points shall be located within 2.5 centimeters (1.00 in.) of the stack walls; and for stack diameters equal to or less than 0.61 m (24 in.), no traverse points shall be located within 1.3 cm (0.50 in.) of the stack walls.

To meet these criteria, observe the procedures given below.

2.3.1.1 Stacks With Diameters Greater Than 0.61 m (24 in.). When any of the traverse points as located in Section 2.3.1 fall within 2.5 cm (1.00 in.) of the stack walls, relocate them away from the stack walls to: (1) a distance of 2.5 cm (1.00 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger. These relocated traverse points (on each end of a diameter) shall be the "adjusted" traverse points.

Whenever two successive traverse points are combined to form a single adjusted traverse point, treat the adjusted point as two separate traverse points, both in the sampling (or velocity measurement) procedure, and in recording the data.

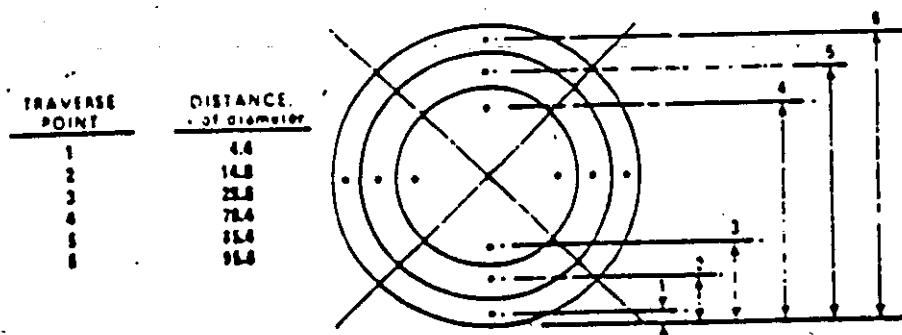


Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

TABLE 1-2. LOCATION OF TRAVERSE POINTS IN CIRCULAR STACKS

(Percent of stack diameter from inside wall to traverse point)

Traverse point number on a diameter	Number of traverse points on a diameter—																						
	2	4	6	8	10	12	14	16	18	20	22	24											
1	14.8	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1											
2	85.4	25.0	14.8	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2											
3		75.0	29.8	19.4	14.0	11.8	9.9	8.5	7.5	6.7	6.0	5.5											
4			65.3	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9											
5				55.4	37.7	24.2	25.0	20.1	16.9	14.8	12.9	11.6											
6					45.8	35.8	26.9	22.0	18.5	16.5	14.6	13.2											
7						35.8	28.0	23.3	20.4	18.0	16.1	14.6											
8							28.0	23.3	20.4	18.0	16.1	14.6											
9								23.3	20.4	18.0	16.1	14.6											
10									20.4	18.0	16.1	14.6											
11										18.0	16.1	14.6											
12											16.1	14.6											
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24																							14.6

2.3.1.2 Stacks With Diameters Equal to or Less Than 0.61 m (24 in.). Follow the procedure in Section 2.3.1.1, noting only that any "adjusted" points should be relocated away from the stack walls to: (1) a distance of 1.3 cm (0.50 in.); or (2) a distance equal to the nozzle inside diameter, whichever is larger.

2.3.3 Rectangular Stacks. Determine the number of traverse points as explained in Sections 2.1 and 2.2 of this method. From Table 1-1, determine the grid configuration. Divide the stack cross-section into as many equal rectangular elemental areas as traverse points, and then locate a traverse point at the centroid of each equal area according to the example in Figure 1-4.

If the tester desires to use more than the minimum number of traverse points, expand the "minimum number of traverse points" matrix (see Table 1-1) by adding the extra traverse points along one or the other or both legs of the matrix; the final matrix need not be balanced. For example, if a 4x3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9x4 or 12x3, and would not necessarily have to be 6x6. After constructing the final matrix, divide the stack cross-section into as many equal rectangular, elemental areas as traverse points, and locate a traverse point at the centroid of each equal area.

The situation of traverse points being too close to the stack walls is not expected to arise with rectangular stacks. If this problem should ever arise, the Administrator must be contacted for resolution of the matter.

2.4 Verification of Absence of Cyclonic Flow. In most stationary sources, the direction of stack gas flow is essentially parallel to the stack walls. However, cyclonic flow may exist (1) after such devices as cyclones and inertial demisters following venturi scrubbers, or (2) in stacks having tangential inlets or other duct configurations which tend to induce swirling; in these instances, the presence or absence of cyclonic flow at the sampling location must be determined. The following techniques are acceptable for this determination.

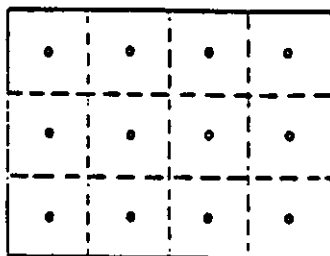


Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

Level and zero the manometer. Connect a Type S pitot tube to the manometer. Position the Type S pitot tube at each traverse point, in succession, so that the planes of the face openings of the pitot tube are perpendicular to the stack cross-sectional plane; when the Type S pitot tube is in this position, it is at "0" reference. Note the differential pressure (Δp) reading at each traverse point. If a null (zero) pitot reading is obtained at 0° reference at a given traverse point, an acceptable flow condition exists at that point. If the pitot reading is not zero at 0° reference, rotate the pitot tube (up to $\pm 90^\circ$ yaw angle), until a null reading is obtained. Carefully determine and record the value of the rotation angle (α) to the nearest degree. After the null technique has been applied at each traverse point, calculate the average of the absolute values of α ; assign a value of 0° to those points for which no rotation was required, and include these in the overall average. If the average value of α is greater than 20°, the overall flow condition in the stack is unacceptable and alternative methodology, subject to the approval of the Administrator, must be used to perform accurate sample and velocity traverses.

The alternative procedure described in Section 2.3 may be used to determine the rotation angles in lieu of the procedure described above. The limit of acceptability for the average value of α would remain 20°.

2.5 Alternative Measurement Site Selection Procedure. This alternative applies to sources where measurement locations are less than 2 equivalent stack or duct diameters downstream or less than $\frac{1}{4}$ duct diameter upstream from a flow disturbance. The alternative should be limited to ducts larger than 24 in. in diameter where blockage and wall effects are minimal. A directional flow-sensing probe is used to measure pitch and yaw angles of the gas flow at 40 or more traverse points; the resultant angle is calculated and compared with acceptable criteria for mean and standard deviation.

Note.—Both the pitch and yaw angles are measured from a line passing through the traverse point and parallel to the stack axis. The pitch angle is the angle of the gas flow component in the plane that INCLUDES the traverse line and is parallel to the stack axis. The yaw angle is the angle of the gas flow component in the plane PERPENDICULAR to the traverse line at the traverse point and is measured from the line passing through the traverse point and parallel to the stack axis.

2.5.1 Apparatus.

2.5.1.1 Directional Probe. Any directional probe, such as United Sensor Type DA Three-Dimensional Directional Probe, capable of measuring both the pitch and yaw angles of gas flows is acceptable. (Note: Mention of trade name or specific products does not constitute endorsement by the U.S. Environmental Protection Agency.) Assign an identification number to the directional

probe, and permanently mark or engrave number on the body of the probe. The pressure holes of directional probes are susceptible to plugging when used in particulate-laden gas streams. Therefore, system for cleaning the pressure holes by "back-purging" with pressurized air is required.

2.5.1.2 Differential Pressure Gauges. Inclined manometers, U-tube manometers, other differential pressure gauges (e.g., magnetic gauges) that meet the specifications described in Method 2, § 2.1

Note.—If the differential pressure gauge produces both negative and positive readings then both negative and positive pressure readings shall be calibrated at a minimum three points as specified in Method 2, § 2.1

2.5.2 Traverse Points. Use a minimum of 40 traverse points for circular ducts and 41 points for rectangular ducts for the gas flow angle determinations. Follow § 2.3 and Table 1-1 or 1-2 for the location and layout of the traverse points. If the measurement location is determined to be acceptable according to the criteria in this alternative procedure, use the same traverse point number and location for sampling and velocity measurements.

2.5.3 Measurement Procedure.

2.5.3.1 Prepare the directional probe as a differential pressure gauge as recommended by the manufacturer. Capillary tubing or surge tanks may be used to dampen pressure fluctuations. It is recommended, but not required, that a pretest leak check be conducted. To perform a leak check, pressurize or use suction on the impact opening until a reading of at least 7.5 cm (in.) H₂O registers on the differential pressure gauge, then plug the impact opening. The pressure of a leak-free system will remain stable for at least 15 seconds.

2.5.3.2 Level and zero the manometer. Since the manometer level and zero may change because of vibrations and temperature changes, periodically check the level and zero during the traverse.

2.5.3.3 Position the probe at the appropriate locations in the gas stream. Rotate until zero deflection is indicated for yaw angle pressure gauge. Determine and record the yaw angle. Record the pressure gauge readings for the pitch angle, and determine the pitch angle from the calibration curve. Repeat this procedure for each traverse point. Complete a "back-purge" the pressure lines and the impact opening prior to measurements of each traverse point.

A post-test check as described in § 2.3 is required. If the criteria for a leak-free system are not met, repair the equipment, repeat the flow angle measurements.

2.5.4 Calculate the resultant angle at traverse point, the average resultant angle and the standard deviation using the following equations. Complete the calculations retaining at least one extra significant figure beyond that of the input data. Round the values after the final calculations.

2.3.4.1 Calculate the resultant angle at each traverse point:

$$L = \arccosine [(\cosine Y) (\cosine P)] \quad \text{Eq. 1-2}$$

Where:

L = Resultant angle at traverse point L , degree.

Y = Yaw angle at traverse point L , degree.

P = Pitch angle at traverse point L , degree.

2.3.4.2 Calculate the average resultant for the measurements:

$$\bar{L} = \frac{\sum R_i}{n} \quad \text{Eq. 1-3}$$

where:

$\bar{L}$ = Average resultant angle, degree.

n = Total number of traverse points.

2.3.4.3 Calculate the standard deviation:

$$S_d = \sqrt{\frac{\sum_{i=1}^n (R_i - \bar{L})^2}{(n-1)}} \quad \text{Eq. 1-4}$$

Where:

S_d = Standard deviation, degree.

2.3.5 The measurement location is acceptable if $\bar{L} < 20^\circ$ and $S_d < 10^\circ$.

2.3.6 Calibration. Use a flow system as described in Sections 4.1.2.1 and 4.1.2.2 of Method 2. In addition, the flow system shall have the capacity to generate two test-section velocities: one between 365 and 730 m/min (1200 and 2400 ft/min) and one between 730 and 1100 m/min (2400 and 3600 ft/min).

2.3.6.1 Cut two entry ports in the test section. The axes through the entry ports shall be perpendicular to each other and intersect in the centroid of the test section. The ports should be elongated slots parallel to the axis of the test section and of sufficient length to allow measurement of pitch angles while maintaining the pitot head position at the test-section centroid. To facilitate alignment of the directional probe during calibration, the test section should be constructed of plexiglass or some other transparent material. All calibration measurements should be made at the same point in the test section, preferably at the centroid of the test-section.

2.3.6.2 To ensure that the gas flow is parallel to the central axis of the test section, follow the procedure in Section 2.4 for cyclonic flow determination to measure the gas flow angles at the centroid of the test section from two test ports located 90° apart. The gas flow angle measured in each port must be $\pm 2^\circ$ or 0° . Straightening vanes should be installed, if necessary, to meet this criterion.

2.3.6.3 Pitch Angle Calibration. Perform a calibration traverse according to the manufacturer's recommended protocol in 5° increments for angles from -60° to $+60^\circ$ at one velocity in each of the two ranges

specified above. Average the pressure ratio values obtained for each angle in the two flow ranges, and plot a calibration curve with the average values of the pressure ratio (or other suitable measurement factor as recommended by the manufacturer) versus the pitch angle. Draw a smooth line through the data points. Plot also the data values for each traverse point. Determine the differences between the measured data values and the angle from the calibration curve at the same pressure ratio. The difference at each comparison must be within 2° for angles between 0° and 40° and within 3° for angles between 40° and 60° .

2.3.6.4 Yaw Angle Calibration. Mark the three-dimensional probe to allow the determination of the yaw position of the probe. This is usually a line extending the length of the probe and aligned with the impact opening. To determine the accuracy of measurements of the yaw angle, only the zero or null position need be calibrated as follows. Place the directional probe in the test section, and rotate the probe until the zero position is found. With a protractor or other angle measuring device, measure the angle indicated by the yaw angle indicator on the three-dimensional probe. This should be within 2° of 0° . Repeat this measurement for any other points along the length of the pitot where yaw angle measurements could be read in order to account for variances in the pitot markings used to indicate pitot head positions.

3. Bibliography

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METHOD 2—DETERMINATION OF STACK GAS VELOCITY AND VOLUMETRIC FLOW RATE (TYPE S PITOT TUBE)

1. Principle and Applicability

1.1 Principle. The average gas velocity in a stack is determined from the gas density and from measurement of the average velocity head with a Type S (Stauscheibe or reverse type) pitot tube.

1.2 Applicability. This method is applicable for measurement of the average velocity of a gas stream and for quantifying gas flow.

This procedure is not applicable at measurement sites which fail to meet the criteria of Method 1, Section 2.1. Also, the method cannot be used for direct measurement in cyclonic or swirling gas streams. Section 2.4 of Method 1 shows how to determine cyclonic or swirling flow conditions. When unacceptable conditions exist, alternative procedures, subject to the approval of the Administrator, U.S. Environmental Protection Agency, must be employed to make accurate flow rate determinations; examples of such alternative procedures are: (1) to install straightening vanes; (2) to calculate the total volumetric flow rate stoichiometrically, or (3) to move to another measurement site at which the flow is acceptable.

2. Apparatus

Specifications for the apparatus are given below. Any other apparatus that has been demonstrated (subject to approval of the Administrator) to be capable of meeting the specifications will be considered acceptable.

2.1 Type S Pitot Tube. The Type S pitot tube (Figure 2-1) shall be made of metal tubing (e.g. stainless steel). It is recommended that the external tubing diameter (dimension D, Figure 2-2b) be between 0.48 and 0.95 centimeters ($\frac{1}{8}$ and $\frac{1}{4}$ inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P and P, Figure 2-2b); it is recommended that this distance be between 1.05 and 1.50 times the external tubing diameter. The face openings of the pitot tube shall, preferably, be aligned as shown in Figure 2-2; however, slight misalignments of the openings are permissible (see Figure 2-3).

The Type S pitot tube shall have a known coefficient, determined as outlined in Section 4, an identification number shall be assigned to the pitot tube; this number shall be permanently marked or engraved on the body of the tube.

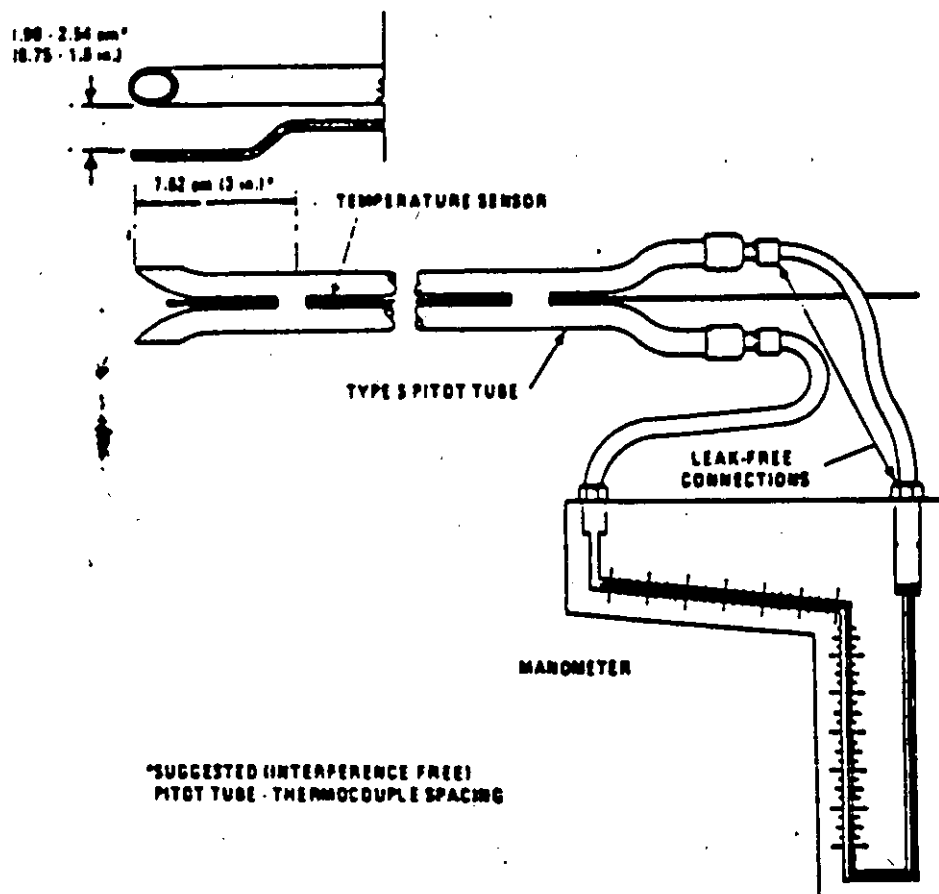


Figure 2-1. Type S pitot tube manometer assembly.

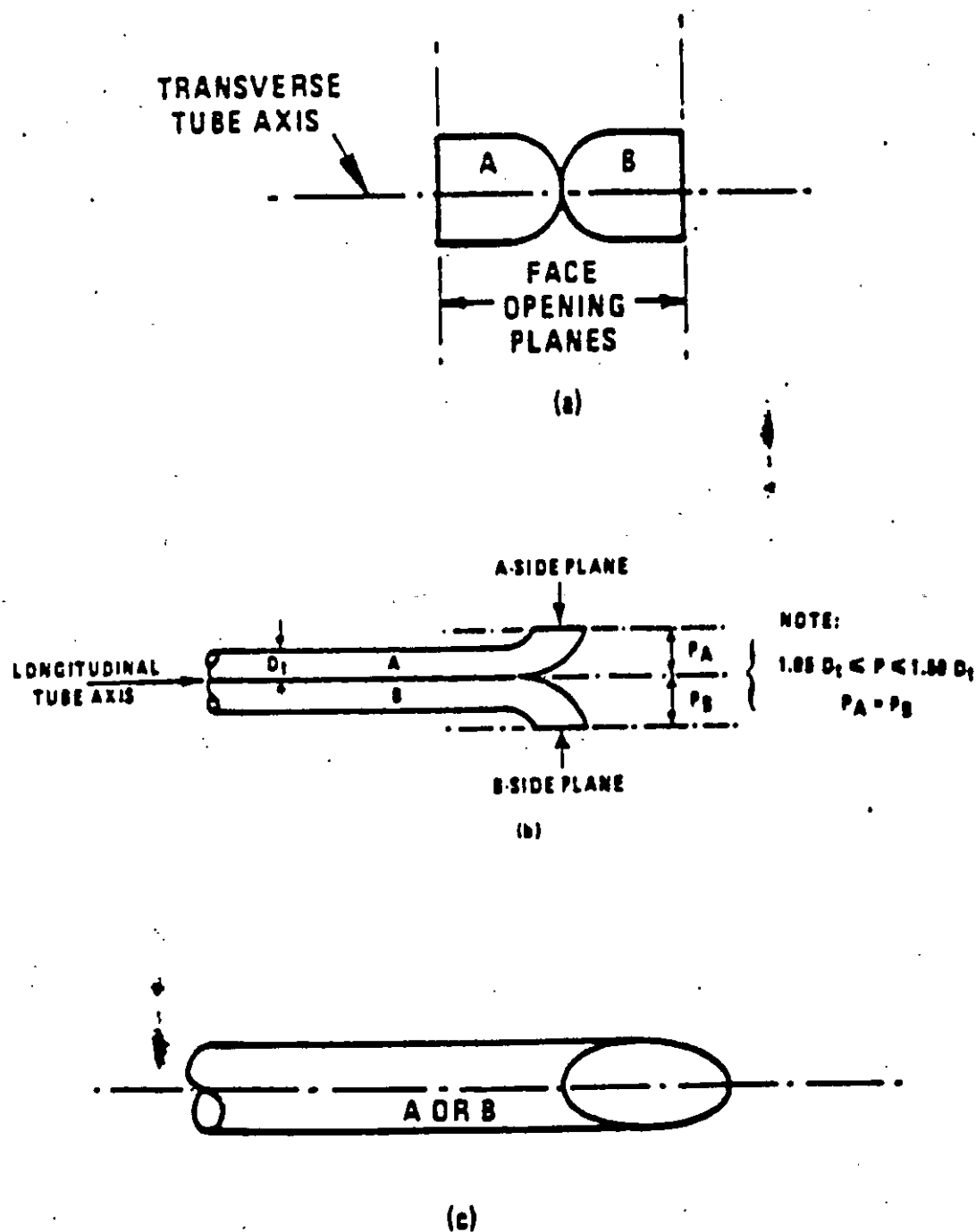


Figure 2-2. Properly constructed Type S pitot tube, shown in: (a) end view; face opening planes perpendicular to transverse axis; (b) top view; face opening planes parallel to longitudinal axis; (c) side view; both legs of equal length and centerlines coincident, when viewed from both sides. Baseline coefficient values of 0.84 may be assigned to pitot tubes constructed this way.

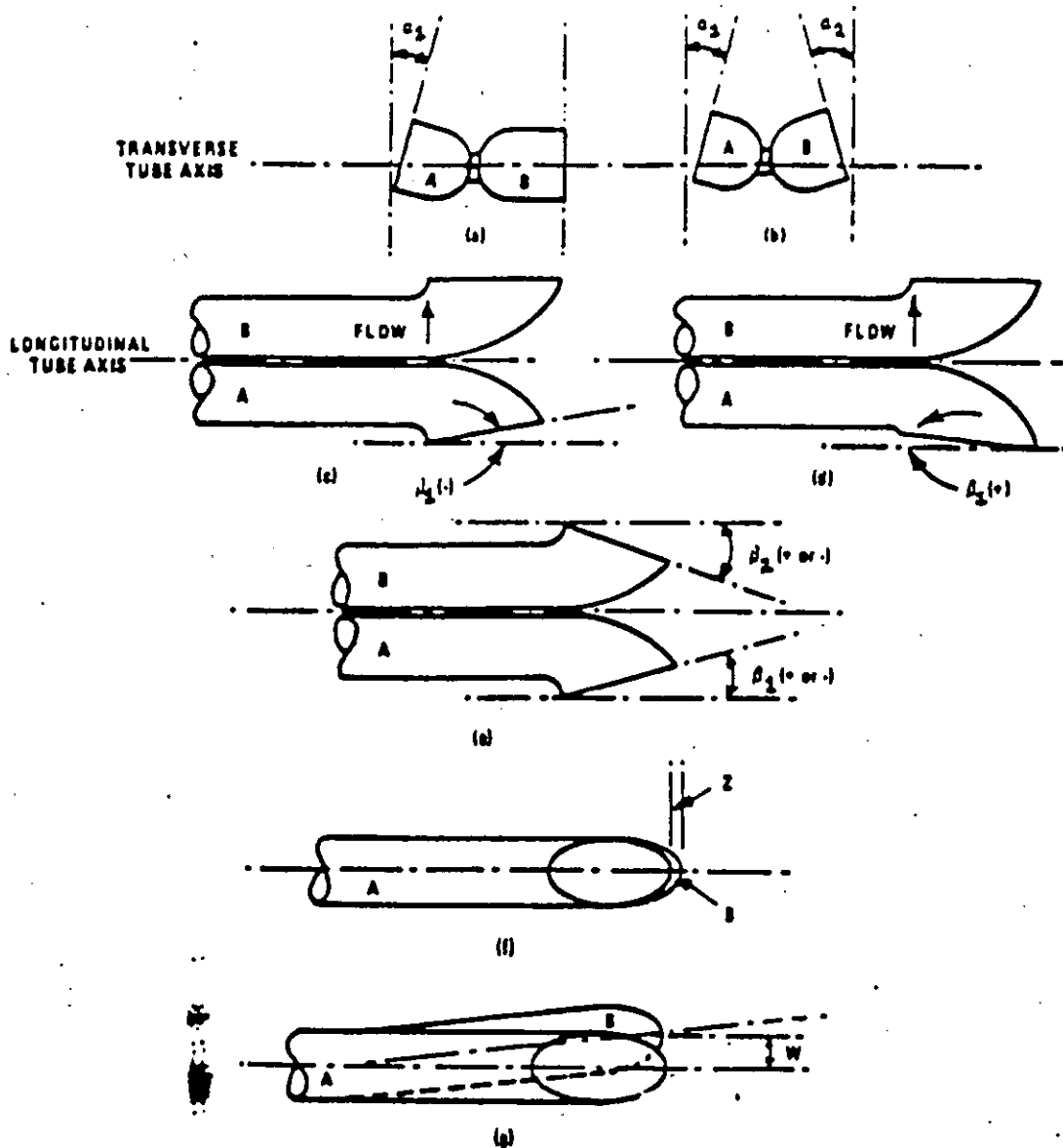


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of $C_p(s)$ so long as α_1 and α_2 10° , β_1 and β_2 5° , z 0.22 cm (1/8 in.) and w 0.08 cm (1/32 in.) (criterion 11 in Bibliography).

A standard pitot tube may be used instead of a Type S, provided that it meets the specifications of Sections 2.7 and 4.2; note, however, that the static and impact pressure holes of standard pitot tubes are susceptible to plugging in particulate-laden gas streams. Therefore, whenever a standard pitot tube is used to perform a traverse, adequate proof must be furnished that the openings of the pitot tube have not plugged up during the traverse period; this can be done by taking a velocity head (Δp) reading at

the final traverse point, cleaning out the impact and static holes of the standard pitot tube by "back-purging" with pressurized air, and then taking another Δp reading. If the Δp readings made before and after the air purge are the same (± 5 percent), the traverse is acceptable. Otherwise, reject the run. Note that if Δp at the final traverse point is unsuitably low, another point may be selected. If "back-purging" at regular intervals is part of the procedure, then comparative Δp readings shall be

taken, as above, for the last two back purges at which suitably high Δp readings are observed.

2.2 Differential Pressure Gauge. An inclined manometer or equivalent device is used. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H₂O divisions on the 0 to 1-in. inclined scale, and 0.1-in. H₂O divisions on the 1- to 10-in. vertical scale. This type of manometer (or other gauge of equivalent sensitivity) is satisfactory for the measurement of Δp values as low as 1.3 mm (0.05 in.) H₂O. However, a differential pressure gauge of greater sensitivity shall be used (subject to the approval of the Administrator), if any of the following is found to be true: (1) the arithmetic average of all Δp readings at the traverse points in the stack is less than 1.3 mm (0.05 in.) H₂O; (2) for traverses of 12 or more points, more than 10 percent of the individual Δp readings are below 1.3 mm (0.05 in.) H₂O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H₂O. Citation 18 in Bibliography describes commercially available instrumentation for the measurement of low-range gas velocities.

As an alternative to criteria (1) through (3) above, the following calculation may be performed to determine the necessity of using a more sensitive differential pressure gauge:

$$T = \frac{\sum_{i=1}^n \sqrt{\Delta p_i} + K}{\sum_{i=1}^n \sqrt{\Delta p_i}}$$

where:

Δp_i = Individual velocity head reading at a traverse point, mm H₂O (in. H₂O).

n = Total number of traverse points.

K = 0.13 mm H₂O when metric units are used and 0.006 in. H₂O when English units are used.

If T is greater than 1.05, the velocity head data are unacceptable and a more sensitive differential pressure gauge must be used.

NOTE: If differential pressure gauges other than inclined manometers are used (e.g., magnetic gauges), their calibration must be checked after each test series. To check

the calibration of a differential pressure gauge, compare Δp readings of the gauge with those of a gauge-oil manometer at a minimum of three points, approximately representing the range of Δp values in the stack. If, at each point, the values of Δp as read by the differential pressure gauge and gauge-oil manometer agree to within 5 percent, the differential pressure gauge shall be considered to be in proper calibration. Otherwise, the test series shall either be voided, or procedures to adjust the measured Δp values and final results shall be used subject to the approval of the Administrator.

2.3 Temperature Gauge. A thermocouple, liquid-filled bulb thermometer, bimetallic thermometer, mercury-in-glass thermometer, or other gauge, capable of measuring temperature to within 1.5 percent of the minimum absolute stack temperature shall be used. The temperature gauge shall be attached to the pitot tube such that the sensor tip does not touch any metal; the gauge shall be in an interference-free arrangement with respect to the pitot tube face openings (see Figure 2-1 and also Figure 2-7 in Section 4). Alternative positions may be used if the pitot tube-temperature gauge system is calibrated according to the procedure of Section 4. Provided that a difference of not more than 1 percent in the average velocity measurement is introduced, the temperature gauge need not be attached to the pitot tube; this alternative is subject to the approval of the Administrator.

2.4 Pressure Probe and Gauge. A piezometer tube and mercury- or water-filled U-tube manometer capable of measuring stack pressure to within 2.5 mm (0.1 in.) Hg is used. The static tap of a standard type pitot tube or one leg of a Type S pitot tube with the face opening planes positioned parallel to the gas flow may also be used as the pressure probe.

2.5 Barometer. A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg) may be used. In many cases, the barometric reading may be obtained from a nearby national weather service station. In which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and the sampling point shall be applied at a rate of minus 2.5 mm (0.1 in.) Hg per 30-meter (100 foot) elevation increase or vice-versa for elevation decrease.

2.6 Gas Density Determination Equipment. Method 3 equipment, if needed (see Section 3.6), to determine the stack gas dry molecular weight, and Reference Method 1 or Method 5 equipment for moisture content determination; other methods may be used subject to approval of the Administrator.

2.7 Calibration Pitot Tube. When calibration of the Type S pitot tube is necessary (see Section 4), a standard pitot tube is used as a reference. The standard pitot tube shall, preferably, have a known coefficient obtained either (1) directly from the National Bureau of Standards, Route 270, Quantico Road, Gaithersburg, Maryland, or (2) by calibration against another standard pitot tube with an NBS-traceable coefficient. Alternatively, a standard pitot tube designed according to the criteria given 2.7.1 through 2.7.5 below and illustrated Figure 2-4 (see also Citations 7, 8, and 1 in Bibliography) may be used. Pitot tubes designed according to these specifications will have baseline coefficients of about 0.99 ± 0.01 .

2.7.1 Hemispherical (shown in Figure 4), ellipsoidal, or conical tip.

2.7.2 A minimum of six diameters straight run (based upon D , the external diameter of the tube) between the tip and static pressure holes.

2.7.3 A minimum of eight diameters straight run between the static pressure holes and the centerline of the exit tube, following the 90 degree bend.

2.7.4 Static pressure holes of equal (approximately 0.1 D), equally spaced piezometer ring configuration.

2.7.5 Ninety degree bend, with smooth mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent is used. If the six velocity calibration technique is employed (see Section 4.1.2.3), the calibration differential pressure gauge shall be readable to the nearest 0.13 mm H₂O (0.006 in. H₂O). For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm H₂O (0.006 in. H₂O) for Δp values between 25 mm H₂O (0.08 in. H₂O) and 1.0 in. H₂O, and to the nearest 1.3 mm H₂O (0.05 in. H₂O) for Δp values above 25 mm H₂O (1.0 in. H₂O). special, more sensitive gauge will be required to read Δp values below 1.3 mm (0.05 in. H₂O) (see Citation 18 in Bibliography).

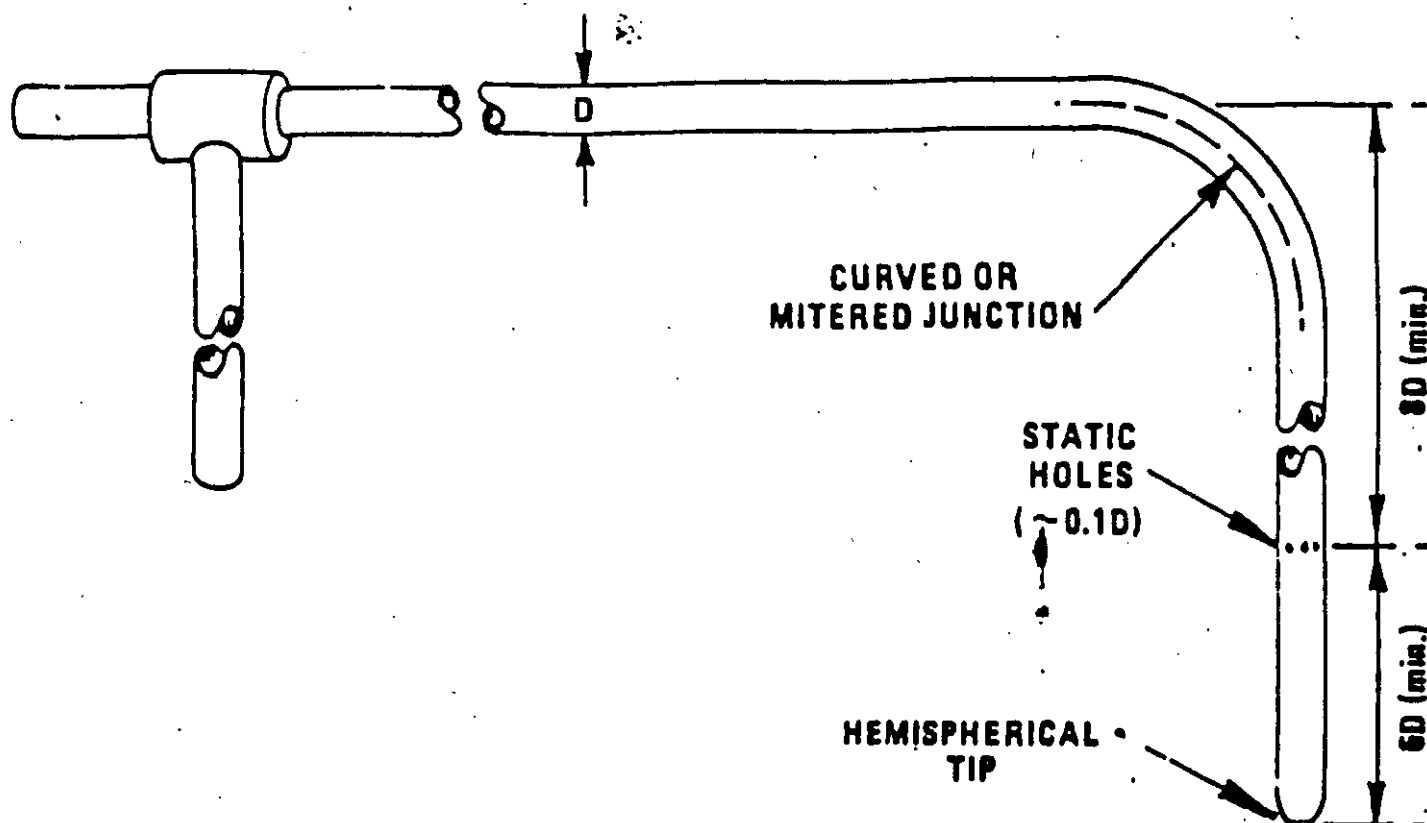


Figure 2-4. Standard pitot tube design specifications.

3. Procedure

3.1 Set up the apparatus as shown in Figure 2-1. Capillary tubing or surge tanks installed between the manometer and pitot tube may be used to dampen Δp fluctuations. It is recommended, but not required, that a pretest leak-check be conducted, as follows: (1) blow through the pitot impact opening until at least 7.6 cm (3 in.) H₂O velocity pressure registers on the manometer; then, close off the impact opening. The pressure shall remain stable for at least 15 seconds; (2) do the same for the static pressure side, except using suction to obtain the minimum of 7.6 cm (3 in.) H₂O. Other leak-check procedures, subject to the approval of the Administrator may be used.

3.2 Level and zero the manometer. Because the manometer level and zero may

drift due to vibrations and temperature changes, make periodic checks during the traverse. Record all necessary data as shown in the example data sheet (Figure 2-3).

3.3 Measure the velocity head and temperature at the traverse points specified by Method 1. Ensure that the proper differential pressure gauge is being used for the range of Δp values encountered (see Section 2.2). If it is necessary to change to a more sensitive gauge, do so, and remeasure the Δp and temperature readings at each traverse point. Conduct a post-test leak-check (mandatory), as described in Section 3.1 above, to validate the traverse run.

3.4 Measure the static pressure in the stack. One reading is usually adequate.

3.5 Determine the atmospheric pressure.

SCHEMATIC OF STACK
CROSS SECTION:

[illegible]

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially CO₂, O₂, CO, and N₂, use Method 3. For processes emitting essentially air, an analysis need not be conducted; use a dry molecular weight of 29.0. For other processes, other methods, subject to the approval of the Administrator, must be used.

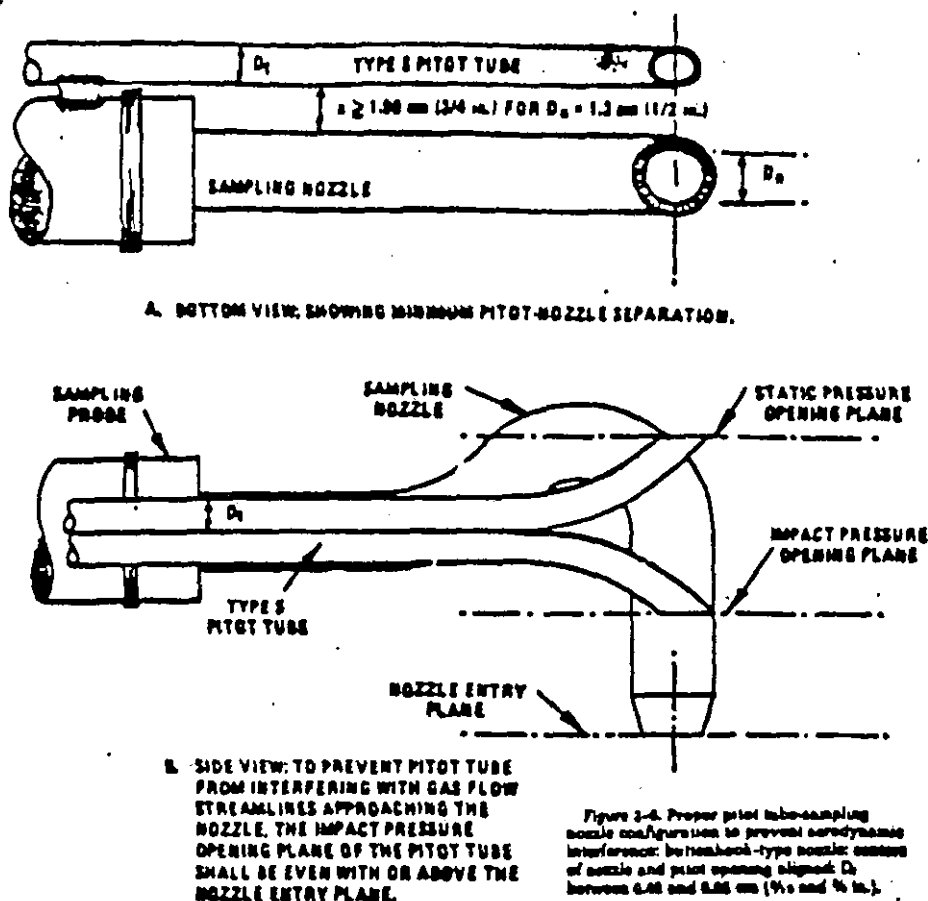
3.8 Determine the cross-sectional area of the stack or duct at the sampling location. Whenever possible, physically measure the stack dimensions rather than using blueprints.

4.1 Type 3 Pitot Tube. Before its initial use, carefully examine the Type 3 pitot tube in top, side, and end views to verify that the face openings of the tube are aligned within the specifications illustrated in Figure 2-2 or 2-3. The pitot tube shall not be used if it fails to meet these alignment specifications.

After verifying the face opening alignment, measure and record the following dimensions of the pitot tube: (a) the external tubing diameter (dimension *D*, Figure 3-7b); and (b) the base-to-opening plane distances (dimensions *P* and *P*₁, Figure 3-7b). If *D* is between 0.48 and 0.96 cm (1/4 and 1/2 in.) and

If P_1 and P_2 are equal and between 1.05 and 1.50 D, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.5 through 4.1.8 below, or (2) a baseline (undisturbed) coefficient value of 0.84 may be assigned to the pitot tube. Note, however, that if the pitot tube is part of an assembly, calibration may still be required, despite the use of the baseline coefficient value (Section 4.1.1).

If D , P_1 , and P_2 are outside the spot limits, the pilot tube must be calibrated outlined in 4.1.2 through 4.1.5 below.



4.1.1 Type S Pitot Tube Assemblies. During sample and velocity traverses, the isolated Type S pitot tube is not always used; in many instances, the pitot tube is used in combination with other source-sampling components (thermocouple, sampling probe, nozzle) as part of an "assembly." The presence of other sampling components can sometimes affect the baseline value of the Type S pitot tube coefficient (Citation 9 in bibliography); therefore an assumed or otherwise known baseline coefficient value may or may not be valid for a given assembly. The baseline and assembly coefficient values will be identical only when the relative placement of the components in the assembly is such that aerodynamic interference effects are eliminated. Figures 2-6 through 2-8 illustrate interference-free component arrangements for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.). Type S pitot tube assemblies that fail to meet any or all of the specifications of Figures 2-6 through 2-8 shall be calibrated according to the procedure outlined in Sections 4.1.3 through 4.1.5 below, and prior to calibration, the values of the intercomponent spacings (pitot-nozzle, pitot-thermocouple, pitot-probe sheath) shall be measured and recorded.

NOTE: Do not use any Type S pitot tube assembly which is constructed such that the impact pressure opening plane of the pitot tube is below the entry plane of the nozzle (see Figure 2-6b).

4.1.3 Calibration Setup. If the Type S pitot tube is to be calibrated, one leg of the tube shall be permanently marked A, and the other, B. Calibration shall be done in a flow system having the following essential design features:

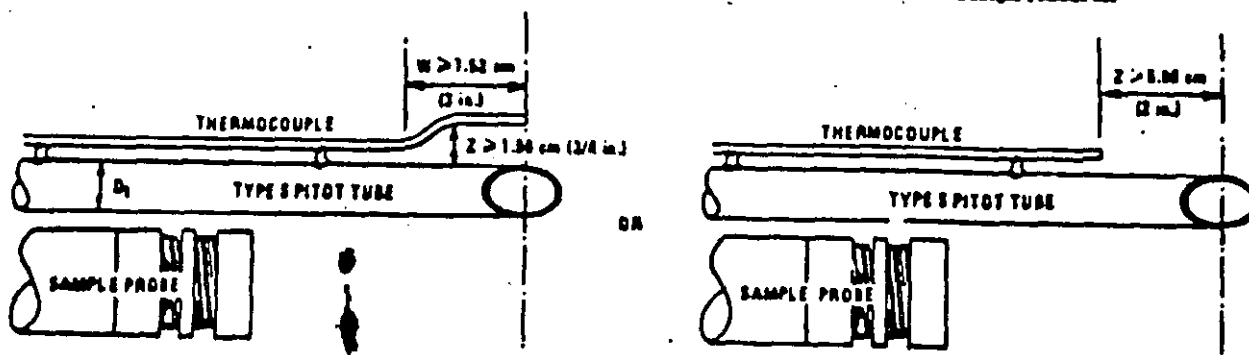


Figure 2-7. Proper thermocouple placement to prevent interference; D_1 between 0.48 and 0.95 cm (3/16 and 3/8 in.).

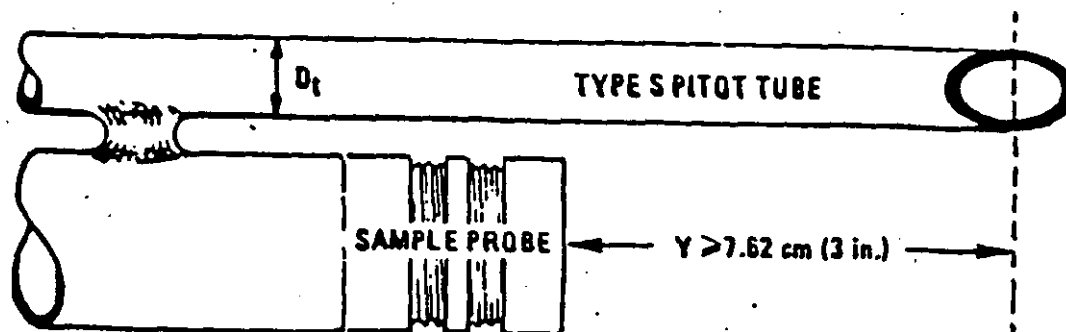


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_1 between 0.48 and 0.95 cm (3/16 and 3/8 in.).

4.1.2.1 The flowing gas stream must be confined to a duct of definite cross-sectional area, either circular or rectangular. For circular cross-sections, the minimum duct diameter shall be 30.5 cm (12 in.); for rectangular cross-sections, the width (shorter side) shall be at least 25.4 cm (10 in.).

4.1.2.2 The cross-sectional area of the calibration duct must be constant over a distance of 10 or more duct diameters. For a rectangular cross-section, use an equivalent diameter, calculated from the following equation, to determine the number of duct diameters:

$$D = \frac{2LW}{L+W}$$

Equation 2-1

where:

D = Equivalent diameter

L = Length

W = Width

To ensure the presence of stable, fully developed flow patterns at the calibration site, or "test section," the site must be located at least eight diameters downstream and two diameters upstream from the nearest disturbances.

Note: The eight- and two-diameter criteria are not absolute; other test section locations may be used (subject to approval of the Administrator), provided that the flow at the test site is stable and demonstrably parallel to the duct axis.

4.1.3 The flow system shall have the capacity to generate a test-section velocity around 918 m/min (3,000 ft/min). This velocity must be constant with time to guarantee steady flow during calibration. Note that Type S pitot tube coefficients obtained by single-velocity calibration at 918 m/min (3,000 ft/min) will generally be valid to within ± 3 percent for the measurement of velocities above 308 m/min (1,000 ft/min) and to within ± 5 to 6 percent for the measurement of velocities between 180 and 308 m/min (600 and 1,000 ft/min). If a more precise correlation between C_p and velocity is desired, the flow system shall have the capacity to generate at least four distinct, time-invariant test-section velocities covering the velocity range from 180 to 1,825 m/min (600 to 6,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in Bibliography for details).

4.1.3.1 Two entry ports, one each for the standard and Type S pitot tubes, shall be cut in the test section; the standard pitot entry port shall be located slightly downstream of the Type S port, so that the standard and Type S impact openings will lie in the same cross-sectional plane during calibration. To facilitate alignment of the pitot tubes during calibration, it is advisable that the test section be constructed of plexiglas or some other transparent material.

4.1.3 Calibration Procedure. Note that this procedure is a general one and must not be used without first referring to the special considerations presented in Section 4.1.5. Note also that this procedure applies only to single-velocity calibration. To obtain calibration data for the A and B sides of the Type S pitot tube, proceed as follows:

4.1.3.1 Make sure that the manometer is properly filled and that the oil is free from contamination and is of the proper density. Inspect and leak-check all pitot lines; repair or replace if necessary.

PITOT TUBE IDENTIFICATION NUMBER: _____ DATE: _____
CALIBRATED BY: _____

"A" SIDE CALIBRATION				
RUN NO.	ΔP_{std} cm H ₂ O (in. H ₂ O)	$\Delta P_{(s)}$ cm H ₂ O (in. H ₂ O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(A)$
1				
2				
3				
			$\bar{C}_p$ (SIDE A)	

"B" SIDE CALIBRATION				
RUN NO.	ΔP_{std} cm H ₂ O (in. H ₂ O)	$\Delta P_{(s)}$ cm H ₂ O (in. H ₂ O)	$C_{p(s)}$	DEVIATION $C_{p(s)} - \bar{C}_p(B)$
1				
2				
3				
			$\bar{C}_p$ (SIDE B)	

$$\text{AVERAGE DEVIATION} = \frac{1}{3} \sum |C_{p(s)} - \bar{C}_p(A \text{ OR } B)| \leftarrow \text{MUST BE } < 1$$

$$|\bar{C}_p(\text{SIDE A}) - \bar{C}_p(\text{SIDE B})| \leftarrow \text{MUST BE } < 0.01$$

Figure 2-9. Pitot tube calibration data.

4.1.3.2 Level and zero the manometer. Turn on the fan and allow the flow to stabilize. Seal the Type S entry port.

4.1.3.3 Ensure that the manometer is level and zeroed. Position the standard pitot tube at the calibration point (determined as outlined in Section 4.1.3.1), and align the tube so that its tip is pointed directly into the flow. Particular care should be taken in aligning the tube to avoid yaw and pitch angles. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.4 Read ΔP_{std} and record its value in a data table similar to the one shown in Figure 2-9. Remove the standard pitot tube from the duct and disconnect it from the manometer. Seal the standard entry port.

4.1.3.5 Connect the Type S pitot tube to the manometer. Open the Type S entry port. Check the manometer level and zero. Insert and align the Type S pitot tube so that its A side impact opening is at the same

point as was the standard pitot tube pointed directly into the flow. Make that the entry port surrounding the properly sealed.

4.1.3.6 Read $\Delta P_{(s)}$ and enter its value data table. Remove the Type S pit from the duct and disconnect it from manometer.

4.1.3.7 Repeat steps 4.1.3.3 through 4.1.3.6 above until three pairs of ΔP are have been obtained.

4.1.3.8 Repeat steps 4.1.3.3 through 4.1.3.7 above for the B side of the pitot tube.

4.1.3.9 Perform calculations, as done in Section 4.1.4 below.

4.1.4 Calculations.

4.1.4.1 For each of the six pairs readings (i.e., three from side A and from side B) obtained in Section 4.1.3, calculate the value of the Type S pit coefficient as follows:

$$C_{MN} = C_{MN(1)} \sqrt{\frac{\Delta p_{(1)}}{\Delta p}}$$

Equation 2-2

where:

C_{MN} = Type S pitot tube coefficient

$C_{MN(1)}$ = Standard pitot tube coefficient; use 0.99 if the coefficient is unknown and the tube is designed according to the criteria of Sections 2.7.1 to 2.7.5 of this method.

Δp_{MN} = Velocity head measured by the standard pitot tube, cm H₂O (in. H₂O)

Δp_s = Velocity head measured by the Type S pitot tube, cm H₂O (in. H₂O)

4.1.4.2 Calculate $\bar{C}_p$ (side A), the mean A-side coefficient, and $\bar{C}_p$ (side B), the mean B-side coefficient; calculate the difference between these two average values.

4.1.4.3 Calculate the deviation of each of the three A-side values of C_p from $\bar{C}_p$ (side A), and the deviation of each B-side value of C_p from $\bar{C}_p$ (side B). Use the following equation:

$$|\text{Deviation}| = C_{MN} - \bar{C}_p (A \text{ or } B)$$

Equation 2-3

4.1.4.4 Calculate σ , the average deviation from the mean, for both the A and B sides of the pitot tube. Use the following equation:

$$\sigma (\text{side A or B}) = \frac{\sum |C_{MN} - \bar{C}_p (A \text{ or } B)|}{3}$$

Equation 2-4

4.1.4.5 Use the Type S pitot tube only if the value of σ (side A) and σ (side B) are less than or equal to 0.01 and if the absolute value of the difference between $\bar{C}_p$ (A) and $\bar{C}_p$ (B) is 0.01 or less.

4.1.5 Special considerations.

4.1.5.1 Selection of calibration point.

4.1.5.1.1 When an isolated Type S pitot tube is calibrated, select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The Type S pitot coefficients so obtained, i.e., $\bar{C}_p$ (side A) and $\bar{C}_p$ (side B), will be valid, so long as either: (1) the isolated pitot tube is used; or (2) the pitot tube is used with other components (nozzle, thermocouple, sample probe) in an arrangement that is free from aerodynamic interference effects (see Figures 2-6 through 2-8).

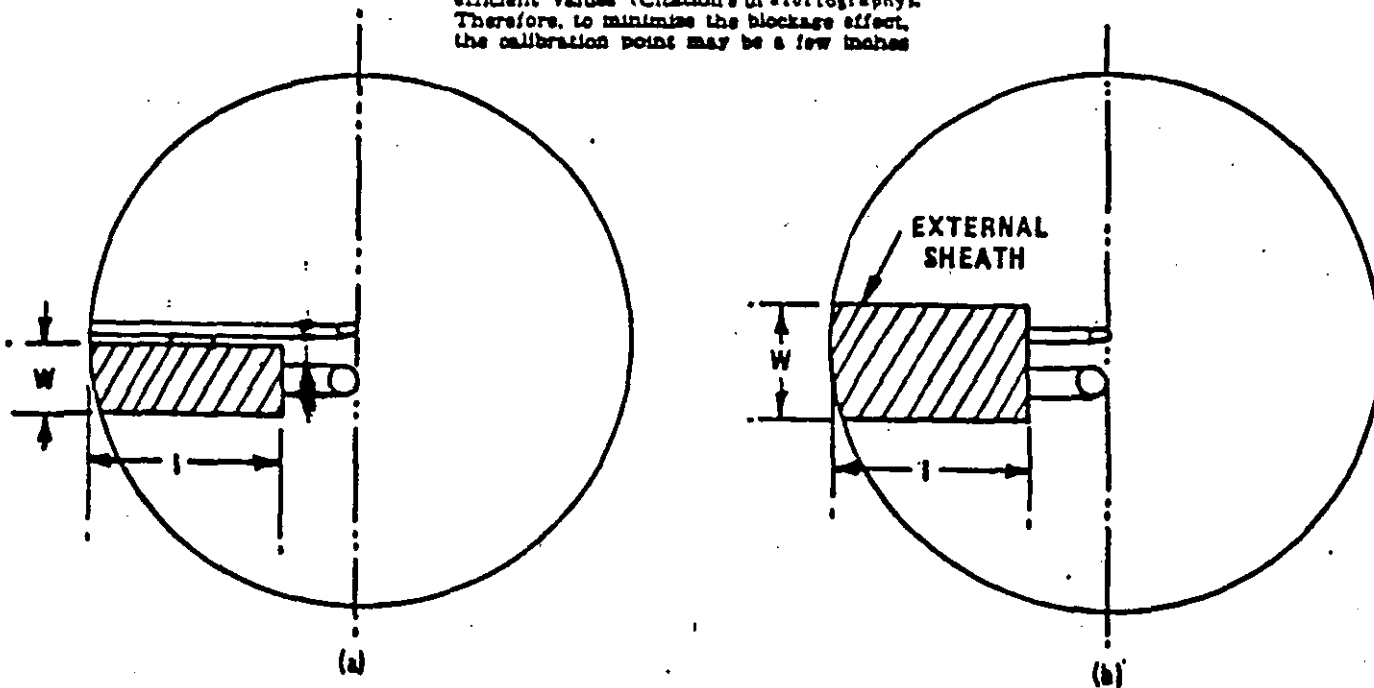
4.1.5.1.2 For Type S pitot tube-thermocouple combinations (without sample probe), select a calibration point at or near the center of the duct, and follow the procedures outlined in Sections 4.1.3 and 4.1.4 above. The coefficients so obtained will be valid so long as the pitot tube-thermocouple combination is used by itself or with other components in an interference-free arrangement (Figures 2-6, and 2-8).

4.1.5.1.3 For assemblies with sample probes, the calibration point should be located at or near the center of the duct; however, insertion of a probe sheath into a small duct may cause significant cross-sectional area blockage and yield incorrect coefficient values (Citation 8 in Bibliography). Therefore, to minimize the blockage effect, the calibration point may be a few inches

off-center if necessary. The actual blockage effect will be negligible when the theoretical blockage, as determined by a projected-area model of the probe sheath, is 2 percent or less of the duct cross-sectional area for assemblies without external sheaths (Figure 2-10a), and 3 percent or less for assemblies with external sheaths (Figure 2-10b).

4.1.5.2 For those probe assemblies in which pitot tube-nozzle interference is a factor (i.e., those in which the pitot-nozzle separation distance fails to meet the specification illustrated in Figure 2-4a), the value of C_p depends upon the amount of free-space between the tube and nozzle, and therefore is a function of nozzle size. In these instances, separate calibrations shall be performed with each of the commonly used nozzle sizes in place. Note that the single-velocity calibration technique is acceptable for this purpose, even though the larger nozzle sizes (>0.635 cm or 1/4 in.) are not ordinarily used for isokinetic sampling at velocities around 915 m/min (3,000 ft/min), which is the calibration velocity; note also that it is not necessary to draw an isokinetic sample during calibration (see Citation 19 in Section 6).

4.1.5.3 For a probe assembly constructed such that its pitot tube is always used in the same orientation, only one side of the pitot tube need be calibrated (the side which will face the flow). The pitot tube must still meet the alignment specifications of Figure 2-2 or 2-3, however, and must have an average deviation (σ) value of 0.01 or less (see Section 4.1.4.4).



$$\text{ESTIMATED SHEATH BLOCKAGE (\%)} = \left[\frac{1 \times W}{\text{DUCT AREA}} \right] \times 100$$

Figure 2-10. Projected-area models for typical pitot tube assemblies.

Figure 2-10. Projected-area models for typical pitot tube assemblies.

4.1.6 Field Use and Recalibration

4.1.6.1 Field Use

4.1.6.1.1 When a Type S pitot tube (isolated tube or assembly) is used in the field, the appropriate coefficient value (whether assigned or obtained by calibration) shall be used to perform velocity calculations. For calibrated Type S pitot tubes, the A side coefficient shall be used when the A side of the tube faces the flow, and the B side coefficient shall be used when the B side faces the flow; alternatively, the arithmetic average of the A and B side coefficient values may be used, irrespective of which side faces the flow.

4.1.6.1.2 When a probe assembly is used to sample a small duct (12 to 36 in. in diameter), the probe sheath sometimes blocks a significant part of the duct cross-section, causing a reduction in the effective value of C_p . Consultation of the bibliography for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 18 inches (Citation 16 in Section 6).

4.1.6.2 Recalibration

4.1.6.2.1 Isolated Pitot Tubes. After each field use, the pitot tube shall be carefully reexamined in top, side, and end views. If the pitot face openings are still aligned within the specifications illustrated in Figure 2-3 or 2-3, it can be assumed that the baseline coefficient of the pitot tube has not changed. If, however, the tube has been damaged to the extent that it no longer meets the specifications of Figure 2-3 or 2-3, the damage shall either be repaired to restore proper alignment of the face openings or the tube shall be discarded.

4.1.6.2.2 Pitot Tube Assemblies. After each field use, check the face opening alignment of the pitot tube, as in Section 4.1.6.2.1; also, remeasure the intercomponent spacings of the assembly. If the intercomponent spacings have not changed and the face opening alignment is acceptable, it can be assumed that the coefficient of the assembly has not changed. If the face opening alignment is no longer within the specifications of Figures 2-2 or 2-3, either repair the damage or replace the pitot tube (calibrating the new assembly, if necessary). If the intercomponent spacings have changed, restore the original spacings or recalibrate the assembly.

4.2 Standard pitot tube (if applicable). If a standard pitot tube is used for the velocity traverse, the tube shall be constructed according to the criteria of Section 2.7 and shall be assigned a baseline coefficient value of 0.99. If the standard pitot tube is used as part of an assembly, the tube shall be in an interference-free arrangement (subject to the approval of the Administrator).

4.3 Temperature Gauges. After each field use, calibrate dual thermometers, liquid-filled bulb thermometers, thermocouple-potentiometer systems, and other gauges at a temperature within 10 percent of the average absolute stack temperature. For temperatures up to 405° C (761° F), use an ASTM mercury-in-glass reference thermometer, or equivalent, as a reference; alternatively, either a reference thermocouple and potentiometer (calibrated by NBS) or thermometric fixed points, e.g., ice bath and boiling water (corrected for barometric pressure) may be used. For temperatures above 405° C (761° F), use an NBS-calibrated reference thermocouple-potentiometer system or an alternate reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperatures measured with the gauge being calibrated and the reference gauge agree within 1.5 percent, the temperature data taken in the field shall be considered valid. Otherwise, the pollutant emission test shall either be considered invalid or adjustments (if appropriate) of the test results shall be made, subject to the approval of the Administrator.

4.4 Barometer. Calibrate the barometer used against a mercury barometer.

5. Calculations

Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after final calculation.

5.1 Nomenclature

A = Cross-sectional area of stack, m² (ft²).
 E_w = Water vapor in the gas stream (from Method 8 or Reference Method 4), proportion by volume.
 C_p = Pitot tube coefficient, dimensionless.
 K_p = Pitot tube constant.

$$34.97 \frac{\text{mm}}{\text{sec}} \left[\frac{(\text{g/g-mole})(\text{mm Hg})}{(^{\circ}\text{K})(\text{mm H}_2\text{O})} \right]^{1/2}$$

for the metric system and

$$K(4.9) \frac{\text{ft}}{\text{sec}} \left[\frac{(\text{lb/lb-mole})(\text{in. Hg})}{(^{\circ}\text{R})(\text{in. H}_2\text{O})} \right]^{1/2}$$

for the English system.

M_d = Molecular weight of stack gas, dry basis (see Section 3.6) g/g-mole (lb/lb-mole).
 M_w = Molecular weight of stack gas, wet basis g/g-mole (lb/lb-mole).
 $= M_d(1 - E_w) + 18.0 E_w$

Equation 2-5

P_m = Barometric pressure at measurement site, mm Hg (in. Hg).

P_s = Stack static pressure, mm Hg (in. Hg).

P_a = Absolute stack gas pressure, mm Hg (in. Hg).

$= P_m + P_s$

Equation 2-6

P_{ms} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_w = Dry volumetric stack gas flow rate corrected to standard conditions, dscm/hr (dscf/hr).

t = Stack temperature, °C (°F).

T_s = Absolute stack temperature, °K (°R).

$= 273 + t$ for metric

Equation 2-7

$= 460 + t$ for English

Equation 2-8

T_{ms} = Standard absolute temperature, 293 °K (528° R)

v = Average stack gas velocity, m/sec (ft/sec).

h_v = Velocity head of stack gas, mm H₂O (in. H₂O).

3,600 = Conversion factor, sec/hr.

18.0 = Molecular weight of water, g/g-mole (lb/lb-mole).

5.2 Average stack gas velocity.

$$v = K_p C_p (\Delta p)^{1/2} \sqrt{\frac{T_{ms}}{P_m M_d}}$$

Equation 2-9

5.3 Average stack gas dry volumetric flow rate.

$$Q_w = 3,600(1 - E_w) v A \left(\frac{T_{ms}}{T_s} \right) \left(\frac{P_s}{P_m} \right)$$

Equation 2-10

"To convert Q_w from dscm/hr (dscf/hr) to dscm/min (dscf/min), divide Q_w by 60."

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METHOD 17—DETERMINATION OF PARTICULATE EMISSIONS FROM STATIONARY SOURCES (IN-STACK FILTRATION METHOD)

Introduction

Particulate matter is not an absolute quantity; rather, it is a function of temperature and pressure. Therefore, to prevent variability in particulate matter emission regulations and/or associated test methods, the temperature and pressure at which particulate matter is to be measured must be carefully defined. Of the two variables (i.e., temperature and pressure), temperature has the greater effect upon the amount of particulate matter in an effluent gas stream; in most stationary source categories, the effect of pressure appears to be negligible.

In method 5, 250° F is established as a nominal reference temperature. Thus, where Method 5 is specified in an applicable subpart of the standards, particulate matter is defined with respect to temperature. In order to maintain a collection temperature of 250° F, Method 5 employs a heated glass sample probe and a heated filter holder. This equipment is somewhat cumbersome and requires care in its operation. Therefore, where particulate matter concentrations (over the normal range of temperature associated with a specified source category) are known to be independent of temperature, it is desirable to eliminate the glass probe and heating systems, and sample at stack temperature.

This method describes an in-stack sampling system and sampling procedures for

use in such cases. It is intended to be used only when specified by an applicable subpart of the standards, and only within the applicable temperature limits (if specified), or when otherwise approved by the Administrator.

1. Principle and Applicability.

1.1 Principle. Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at stack temperature. The particulate mass is determined gravimetrically after removal of uncombined water.

1.2 Applicability. This method applies to the determination of particulate emissions from stationary sources for determining compliance with new source performance standards, only when specifically provided for in an applicable subpart of the standards. This method is not applicable to stacks that contain liquid droplets or are saturated with water vapor. In addition, this method shall not be used as written if the projected cross-sectional area of the probe extension-filter holder assembly covers more than 5 percent of the stack cross-sectional area (see Section 4.1.2).

2. Apparatus.

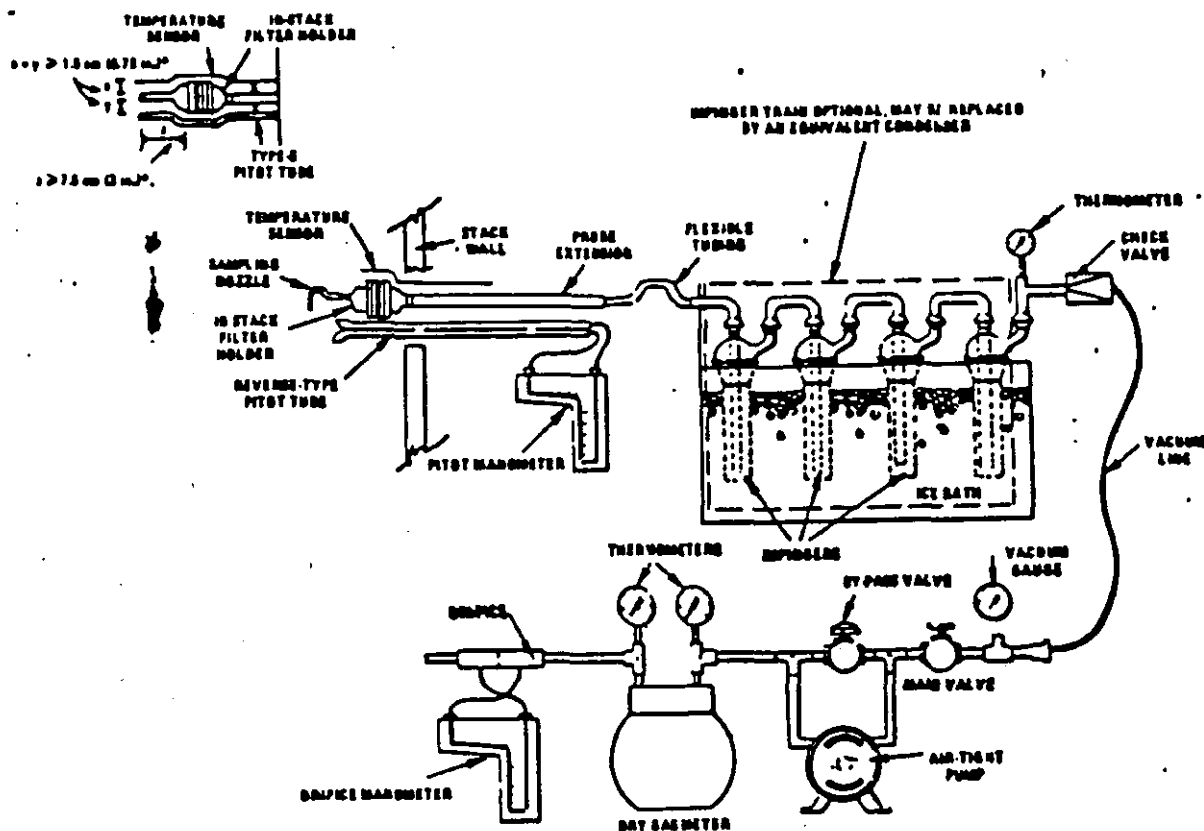
2.1 Sampling Train. A schematic of the sampling train used in this method is shown in Figure 17-1. Construction details for many, but not all, of the train components are given in APTD-0581 (Citation 2 in Bibliography); for changes from the APTD-0581 document and for allowable modifications to Figure 17-1, consult with the Administrator.

The operating and maintenance procedures for many of the sampling train components are described in APTD-0576 (Citation 3 in Bibliography). Since correct usage is important in obtaining valid results, all users should read the APTD-0576 document and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The sampling train consists of the following components:

2.1.1 Probe Nozzle. Stainless steel (316) or glass, with sharp, tapered leading edge. The angle of taper shall be 30° and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used subject to the approval of the Administrator.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm ($\frac{1}{8}$ to $\frac{1}{2}$ in)—or larger if higher volume sampling trains are used—inside diameter (ID) nozzles in increments of 0.16 cm ($\frac{1}{16}$ in). Each nozzle shall be calibrated according to the procedures outlined in Section 5.1.

2.1.2 Filter Holder. The in-stack filter holder shall be constructed of borosilicate or quartz glass, or stainless steel; if a gasket is used, it shall be made of silicone rubber, Teflon, or stainless steel. Other holder and gasket materials may be used subject to the approval of the Administrator. The filter holder shall be designed to provide a positive seal against leakage from the outside or around the filter.



* 20052770 UNTERFERENCE-FREE SPACERS

Figure 17-1. Particulate Sampling Train, Equipped with In-Stack Filter.

2.1.3 Probe Extension. Any suitable rigid probe extension may be used after the filter holder.

2.1.4 Pitot Tube. Type S, as described in Section 2.1 of Method 2, or other device approved by the Administrator, the pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity (see Figure 17-1). The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane during sampling (see Method 2, Figure 2-6b). It is recommended: (1) that the pitot tube have a known baseline coefficient, determined as outlined in Section 4 of Method 2 and (2) that this known coefficient be preserved by placing the pitot tube in an interference-free arrangement with respect to the sampling nozzle, filter holder, and temperature sensor (see Figure 17-1). Note that the 1.9 cm (0.75 in) free-space between the nozzle and pitot tube shown in Figure 17-1, is based on a 1.3 cm (0.5 in) ID nozzle. If the sampling train is designed for sampling at higher flow rates than that described in APTD-0581, thus necessitating the use of larger sized nozzles, the free-space shall be 1.9 cm (0.75 in) with the largest sized nozzle in place.

Source-sampling assemblies that do not meet the minimum spacing requirements of Figure 17-1 (or the equivalent of these requirements, e.g., Figure 2-7 of Method 2) may be used; however, the pitot tube coefficients of such assemblies shall be determined by calibration, using methods subject to the approval of the Administrator.

2.1.5 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in Section 2.3 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

2.1.6 Condenser. It is recommended that the impinger system described in Method 3 be used to determine the moisture content of the stack gas. Alternatively, any system that allows measurement of both the water condensed and the moisture leaving the condenser, each to within 1 ml or 1 g, may be used. The moisture leaving the condenser can be measured either by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a silica gel trap with exit gases kept below 20° C (68° F) and determining the weight gain.

Flexible tubing may be used between the probe extension and condenser. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

2.1.7 Metering System. Vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 3° C (5.4° F), dry gas meter capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 17-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall enable checks of isokinetic rates.

Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0578 may be used provided that the specifications of this method are met.

2.1.8 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in. Hg). In many cases, the barometric reading may be obtained from a nearby national weather service station, in which case the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be applied at a rate of minus 2.5 mm Hg (0.1 in. Hg) per 30 m (100 ft) elevation increase or vice versa for elevation decrease.

2.1.9 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in Sections 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3.

The temperature sensor shall be attached to either the pitot tube or to the probe extension in a fixed configuration. If the temperature sensor is attached in the field, the sensor shall be placed in an interference-free arrangement with respect to the Type S pitot tube openings (as shown in Figure 17-1 or in Figure 2-7 of Method 2). Alternatively, the temperature sensor need not be attached to either the probe extension or pitot tube during sampling, provided that a difference of not more than 1 percent in the average velocity measurement is introduced. This alternative is subject to the approval of the Administrator.

2.3 Sample Recovery.

2.2.1 Probe Nozzle Brush. Nylon bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.

2.2.2 Wash Bottles—Two. Glass wash bottles are recommended; polyethylene wash bottles may be used at the option of the tester. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

2.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

2.2.4 Petri Dish. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

2.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 1 g. Graduated cylinders shall have subdivisions no greater than 2 ml. Most laboratory balances are capable of weighing to the nearest 0.5 g or less. Any of these balances is suitable for use here and in Section 2.3.4.

2.2.6 Plastic Storage Containers. Air tight containers to store silica gel.

2.2.7 Funnel and Rubber Pollooman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

2.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

2.3 Analysis.

2.3.1 Glass Weighing Dish.

2.3.2 Desiccator.

2.3.3 Analytical Balance. To measure to within 0.1 mg.

2.3.4 Balance. To measure to within 0.5 mg.

2.3.5 Beakers. 250 ml.

2.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

2.3.7 Temperature Gauge. To measure the temperature of the laboratory environment.

3. Reagents.

3.1 Sampling.

3.1.1 Filters. The in-stack filters shall be glass mats or thimble fiber filters, without organic binders, and shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency tests shall be conducted in accordance with ASTM Standard Method D2984-71 (Reapproved 1978) (incorporated by reference—see 100.17). Test data from the supplier's quality control program are sufficient for this purpose.

3.1.2 Silica Gel. Indicating type, 6- to 16-mesh. If previously used, dry at 175° C (350° F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

3.1.3 Crushed Ice.

3.1.4 Stopcock Grease. Acetone-insoluble, heat-stable silicone grease. This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used. Alternatively, other types of stopcock grease may be used, subject to the approval of the Administrator.

3.1.5 Water. Same as in Method 3, Section 3.1.3.

3.2 Sample Recovery. Acetone, reagent grade, 0.001 percent residue, in glass bottles. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers. Thus, acetone blanks shall be run prior to field use and only acetone with low blank values (0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

3.3 Analysis.

3.3.1 Acetone. Same as 3.2.

3.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

4. Procedure.

4.1 Sampling. The complexity of this method is such that, in order to obtain reliable results, testers should be trained and experienced with the test procedures.

4.1.1 Pretest Preparation. All components shall be maintained and calibrated according to the procedure described in APTD-0678, unless otherwise specified herein.

Weigh several 200 to 300 g portions of silica gel in air-tight containers to the nearest 0.5 g. Record the total weight of the silica gel plus container, on each container. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

Check filters visually against light for irregularities and flaws or pinhole leaks. Label filters of the proper size on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or plastic petri dishes) and keep the filters in these containers at all times except during sampling and weighing.

Dedicate the filters at $20 \pm 5.6^\circ \text{C}$ ($68 \pm 10^\circ \text{F}$) and ambient pressure for at least 24 hours and weigh at intervals of at least 4 hours to a constant weight, i.e., 0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing the filter must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105°C (220°F) for 2 to 3 hours, dedicated for 2 hours, and weighed. Procedures other than those described, which account for relative

humidity effects, may be used, subject to the approval of the Administrator.

4.1.2 Preliminary Determinations. Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Make a projected-area model of the probe extension-filter holder assembly, with the pitot tube face openings positioned along the centerline of the stack, as shown in Figure 17-2. Calculate the estimated cross-section blockage, as shown in Figure 17-2. If the blockage exceeds 5 percent of the duct cross sectional area, the tester has the following options: (1) a suitable out-of-stack filtration method may be used instead of in-stack filtration; or (2) a special in-stack arrangement, in which the sampling and velocity measurement sites are separate, may be used; for details concerning this approach, consult with the Administrator (see also Citation 10 in Bibliography). Determine the stack pressure, temperature, and the range of velocity heads using Method 2. It is recommended that a leak-check of the pitot lines (see Method 2, Section 3.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, Section 3.8; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particular sample run.

Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.3 of Method 2).

Select a probe extension length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the length of probes.

Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that: (1) the sampling time per point is not less than 2 minutes (or some greater time interval if specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

In some circumstances, e.g., batch cycles, it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

4.1.3 Preparation of Collection Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

If impingers are used to condense stack gas moisture, prepare them as follows: place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of

preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

If some means other than impingers is used to condense moisture, prepare the condenser (and, if appropriate, silica gel for condenser outlet) for use.

Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as not to allow the sample gas stream to circumvent the filter. Check filter for tears after assembly is completed. Mark the probe extension with heat

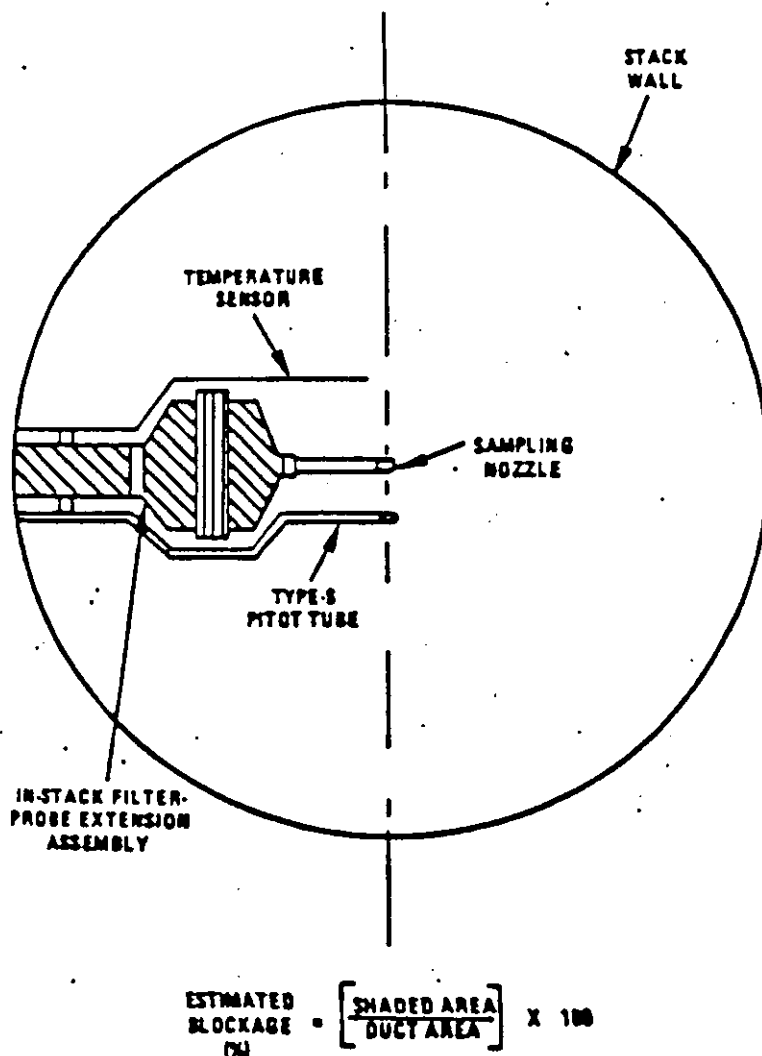


Figure 17-2. Projected-area model of cross-section blockage (approximate average for a sample traverse) caused by an in-stack filter holder-probe extension assembly.

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be conducted at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either record the leakage rate and correct the sample volume as shown in Section 6.3 of this method, or shall void the sampling run.

4.1.5 Particulate Train Operation. During the sampling run, maintain a sampling rate such that sampling is within 10 percent of true isokinetic, unless otherwise specified by the Administrator.

For each run, record the data required on the example data sheet shown in Figure 17-3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak check, and when sampling is halted. Take other readings required by Figure 17-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

Clean the portholes prior to the test run to minimize the chance of sampling the deposited material. To begin sampling, remove the nozzle cap and verify that the pitot tube and probe extension are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump and adjust the flow to isokinetic conditions. Nomographs are available, which aid in the rapid adjustment to the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type 8 pitot tube coefficient is 0.85 ± 0.02 , and the stack gas equivalent density (dry molecular weight) is equal to 29 ± 4 . APTD-0576 details the procedure for using the nomographs. If C, and M₁ are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Citation 7 in Bibliography) are taken to compensate for the deviations.

When the stack is under significant negative pressure (height of impinger stem), take care to close the coarse adjust valve before inserting the probe extension assembly into the stack to prevent water from being forced backward. If necessary, the pump may be turned on with the coarse adjust valve closed.

When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

Traverse the stack cross section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe extension through the portholes, to minimize chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain a temperature of less than 20° C (68° F) at the condenser outlet; this will prevent excessive moisture losses. Also, periodically check the level and zero of the manometer.

If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of a sample run. It is recommended that another complete filter holder assembly be used rather than attempting to change the filter itself. Before a new filter holder is installed, conduct a leak check, as outlined in Section 4.1.4.3. The total particulate weight shall include the summation of all filter assembly catches.

A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or, in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator. Note that when two or more trains are used, a separate analysis of the collected particulate from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the particulate catches from the individual trains may be combined and a single analysis performed.

At the end of the sample run, turn off the pump, remove the probe extension assembly from the stack, and record the final dry gas meter reading. Perform a leak check, as outlined in Section 4.1.4.3. Also, leak-check the pitot lines as described in Section 3.1 of Method 2; the lines must pass this leak-check, in order to validate the velocity head data.

4.1.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Section 6.11) to determine whether another test run should be made. If there is difficulty in maintaining isokinetic rates due to source conditions, consult with the Administrator for possible variance on the isokinetic rates.

4.2 Sample Recovery. Proper cleanup procedure begins as soon as the probe extension assembly is removed from the stack at the end of the sampling period. Allow the assembly to cool.

When the assembly can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle and place a cap over it to prevent losing or gaining particulate matter. Do not cap off the probe tip tightly while the sampling train is cooling down as this would create a vacuum in the filter holder, forcing condenser water backward.

Before moving the sample train to the cleanup site, disconnect the filter holder-probe nozzle assembly from the probe extension; cap the open inlet of the probe extension. Be careful not to lose any condensate, if present. Remove the umbilical cord from the condenser outlet and cap the outlet. If a flexible line is used between the first impinger (or condenser) and the probe extension, disconnect the line at the probe extension and let any condensed water or liquid drain into the impingers or condenser. Disconnect the probe extension from the condenser; cap the probe extension outlet. After wiping off the silicone grease, cap off the condenser inlet. Ground glass stoppers, plastic caps, or serum caps (whichever are appropriate) may be used to close these openings.

Transfer both the filter holder-probe nozzle assembly and the condenser to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

Save a portion of the acetone used for cleanup as a blank. Take 200 ml of this ace-

tone directly from the wash bottle being used and place it in a glass sample container labeled "acetone blank."

Inspect the train prior to and during disassembly and note any abnormal conditions. Treat the samples as follows:

Container No. 1. Carefully remove the filter from the filter holder and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the particulate cake is inside the fold. Carefully transfer to the petri dish any particulate matter and/or filter fibers which adhere to the filter holder gasket, by using a dry Nylon bristle brush and/or a sharp-edged blade. Seal the container.

Container No. 2. Taking care to see that dust on the outside of the probe nozzle or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe nozzle, fitting, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator; in these cases, save a water blank and follow Administrator's directions on analysis. Perform the acetone rinses as follows:

Carefully remove the probe nozzle and clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until acetone rinses shows no visible particles, after which make a final rinse of the inside surface with acetone.

Brush and rinse with acetone the inside parts of the fitting in a similar way until no visible particles remain. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Rinse the brush with acetone and quantitatively collect these washings in the sample container. Between sampling runs, keep brushes clean and protected from contamination.

After ensuring that all joints are wiped clean of silicone grease (if applicable), clean the inside of the front half of the filter holder by rubbing the surfaces with a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make final rinses of the brush and filter holder. After all acetone washings and particulate matter are collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether or not leakage occurred during transport. Label the container to clearly identify its contents.

Container No. 3. If silica gel is used in the condenser system for moisture content determination, note the color of the gel to determine if it has been completely spent; make a notation of its condition. Transfer the silica gel back to its original container and seal. A funnel may make it easier to pour the silica gel without spilling, and a rubber policeman may be used as an aid in removing the silica gel. It is not necessary to remove the small amount of dust particles that may adhere to the walls and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 under "Analysis."

Condenser Water. Treat the condenser or impinger water as follows: make a notation of any color or film in the liquid catch. Measure the liquid volume to within ± 1 ml by using a graduated cylinder or, if a balance is available, determine the liquid weight to within ± 0.5 g. Record the total volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight.

4.3 Analysis. Record the data required on the example sheet shown in Figure 17-4. Handle each sample container as follows:

Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight and report the results to the nearest 0.1 mg. For purposes of this Section, 4.3, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

Alternatively, the sample may be oven dried at the average stack temperature or 105°C (220°F), whichever is less, for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The tester may also opt to oven dry the sample at the average stack temperature or 108°C (226°F), whichever is less, for 2 to 3 hours, weigh the sample, and use this weight as a final weight.

FIGURE 17-4—ANALYTICAL DATA

Plant _____
Date _____
Run No. _____
Filter No. _____
Amount liquid lost during transport _____
Acetone blank volume, ml _____
Acetone wash volume, ml _____
Acetone blank concentration, mg/mg (equation 17-4) _____
Acetone wash blank, mg (equation 17-5) _____

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1			
2			
Total			
Less acetone blank			
Weight of particulate matter			

	Volume of liquid water collected	
	Impinger volume, ml	Shim gas weight, g
Final		
Initial		
Liquid collected		
Total volume collected		

*Convert weight of water to volume by dividing total weight reported by density of water (1 g/ml).

$$\frac{\text{Impinger, g}}{(1 \text{ g/ml})} = \text{Volume water, ml}$$

Container No. 2. Note the level of liquid in the container and confirm on the analysis sheet whether or not leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 ml or gravimetrically to ± 0.5 g. Transfer the contents to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

Container No. 3. This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

"Acetone Blank" Container. Measure acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250-ml beaker and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE: At the option of the tester, the contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be stirred occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

8. Calibration. Maintain a laboratory log of all calibrations.

8.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When nozzles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

8.2 Pitot Tube. If the pitot tube is placed in an interference-free arrangement with respect to the other probe assembly components, its baseline (isolated tube) coefficient shall be determined as outlined in Section 4 of Method 2. If the probe assembly is not interference-free, the pitot tube assembly coefficient shall be determined by calibration, using methods subject to the approval of the Administrator.

8.3 Metering System. Before its initial use in the field, the metering system shall be calibrated according to the procedure outlined in APTD-0876. Instead of physically adjusting the dry gas meter dial readings to correspond to the wet test meter readings, calibration factors may be used to mathematically correct the gas meter dial readings to the proper values.

Before calibrating the metering system, it is suggested that a leak-check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at $0.00057 \text{ m}^3/\text{min}$ (0.02 cfm).

At the end of the run, take the difference of the measured wet test meter and dry gas meter volumes; divide the difference by 10, to get the leak rate. The leak rate should not exceed $0.00057 \text{ m}^3/\text{min}$ (0.02 cfm).

After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the calibration factor. If the calibration has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as outlined in APTD-0876.

Alternative procedures, e.g., using the orifice meter coefficients, may be used, subject to the approval of the Administrator.

NOTE: If the dry gas meter coefficient values obtained before and after a test series differ by more than 5 percent, the test series shall either be voided, or calculations for the test series shall be performed using whichever meter coefficient value (i.e., before or after) gives the lower value of total sample volume.

8.4 Temperature Gauge. Use the procedure in Section 4.3 of Method 2 to calibrate in-stack temperature gauges. Dial thermometers, such as are used for the dry gas meter and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

8.5 Leak Check of Metering System. Shown in Figure 17-1. That portion of the sampling train from the pump to the orifice meter should be leak checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 17-5). Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

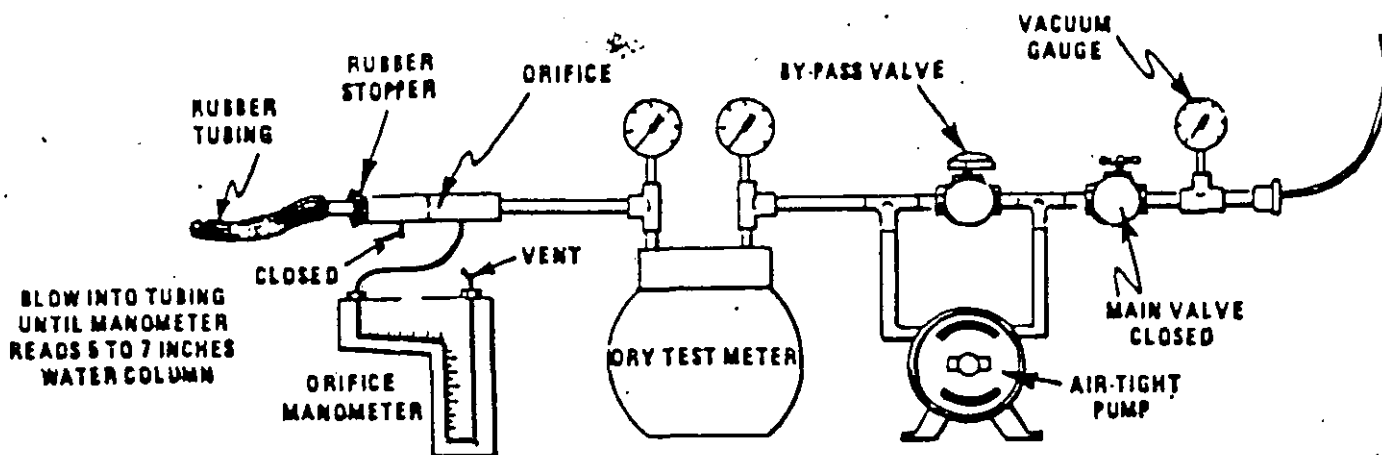


Figure 17-5. Leak check of meter box.

5.6 Barometer. Calibrate against a mercury barometer.

6. Calculations. Carry out calculations, retaining at least one extra decimal figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

6.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).
 E_w = Water vapor in the gas stream, proportion by volume.
 C_a = Acetone blank residue concentration, mg/mg .
 c_p = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, $g/dscm$ ($g/dscf$).
 I = Percent of isokinetic sampling.
 L_1 = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to $0.00087 m^3/min$ ($0.02 cfm$) or 4 percent of the average sampling rate, whichever is less.
 L_2 = Individual leakage rate observed during the leak check conducted prior to the "i" component change ($i = 1, 2, 3 \dots n$), m^3/min (cfm).
 L_3 = Leakage rate observed during the post-test leak check, m^3/min (cfm).
 m_p = Total amount of particulate matter collected, mg .
 M_w = Molecular weight of water, $18.0 g/g-mole$ ($18.0 lb/lb-mole$).
 m_a = Mass of residue of acetone after evaporation, mg .
 P_w = Barometric pressure at the sampling site, $mm Hg$ ($in. Hg$).
 P_s = Absolute stack gas pressure, $mm Hg$ ($in. Hg$).
 P_{std} = Standard absolute pressure, $760 mm Hg$ ($29.92 in. Hg$).
 R = Ideal gas constant, $0.08206 mm Hg \cdot m^3 / K \cdot g-mole$ ($21.85 in. Hg \cdot ft^3 / R \cdot lb-mole$).
 T_a = Absolute average dry gas meter temperature (see Figure 17-3), $^{\circ}K$ ($^{\circ}R$).
 T_s = Absolute average stack gas temperature (see Figure 17-3), $^{\circ}K$ ($^{\circ}R$).
 T_{std} = Standard absolute temperature, $293^{\circ}K$ ($52^{\circ}F$).
 V_a = Volume of acetone blank, ml .
 V_w = Volume of acetone used in wash, ml .
 V_b = Total volume of liquid collected in impingers and silica gel (see Figure 17-4), ml .

V_n = Volume of gas sample as measured by dry gas meter, $dscm$ ($dscf$).

V_{std} = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, $dscm$ ($dscf$).

V_{wv} = Volume of water vapor in the gas sample, corrected to standard conditions, $dscm$ ($dscf$).

v_s = Stack gas velocity, calculated by Method 2, Equation 3-9, using data obtained from Method 17, m/sec (ft/sec).

W_r = Weight of residue in acetone wash, mg .

Y = Dry gas meter calibration coefficient.

ΔH = Average pressure differential across the orifice meter (see Figure 17-3), $mm H_2O$ ($in. H_2O$).

ρ_a = Density of acetone, mg/ml (see label on bottle).

ρ_w = Density of water, $0.9982 g/ml$ ($0.002201 lb/ml$).

t = Total sampling time, min .

t_1 = Sampling time interval from the beginning of a run until the first component change, min .

t_2 = Sampling time interval between two successive component changes, beginning with the interval between the first and second changes, min .

t_n = Sampling time interval from the final (n) component change until the end of the sampling run, min .

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

6.2 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 17-3).

6.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions ($20^{\circ}C$, $760 mm Hg$ or $68^{\circ}F$, $29.92 in. Hg$) by using Equation 17-1.

Equation 17-1

$$V_{n(std)} = V_n Y \left(\frac{T_{std}}{T_n} \right) \left[\frac{P_{bar} + \frac{\Delta H}{13.6}}{P_{std}} \right] \\ = K_1 V_n Y \frac{P_{bar} + (\Delta H/13.6)}{P_{std}}$$

where:

$K_1 = 0.3858^{\circ}K/mm Hg$ for metric units;
 $17.64^{\circ}R/in. Hg$ for English units.

NOTE: Equation 17-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (i.e., the post-test leak check or leak checks conducted prior to component changes) exceeds L_1 . If L_2 or L_3 exceeds L_1 , Equation 17-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_n in Equation 17-1 with the expression:

$$[V_n - (L_2 - L_1)t]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_n in Equation 17-1 by the expression:

$$[V_n - (L_1 - L_2)t_1 - \sum_{i=2}^n (L_1 - L_i)t_i \\ - (L_p - L_2)t_p]$$

and substitute only for those leakage rate (L_2 or L_p) which exceed L_1 .

6.4 Volume of water vapor.

$$V_{w(std)} = V_{1c} \left(\frac{\rho_w}{\rho_a} \right) \left(\frac{RT_{std}}{P_{std}} \right) = K_2 V_{1c}$$

where:

$K_2 = 0.001333 m^3/ml$ for metric unit
 $0.04707 ft^3/ml$ for English units.

Equation 17-2

6.8 Moisture Content.

$$E_m = \frac{V_{\text{moist}}}{V_{\text{moist}} + V_{\text{solid}}}$$

Equation 17-3

6.9 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_{\text{a}}}$$

Equation 17-4

6.7 Acetone Wash Blank.

$$W_a = C_a V_{\text{a}}$$

Equation 17-5

6.8 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1 and 2 less the acetone blank (see Figure 17-4).

NOTE: Refer to Section 4.1.5 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

6.9 Particulate Concentration.

$$C_p = (0.001 \text{ g/mg}) (m_p / V_{\text{moist}})$$

Equation 17-6

6.10 Conversion Factors:

From	To	Multiply by
mg	m ³	0.02832
g/m ³	g/m ³	1.43
g/m ³	g/m ³	2.706 x 10 ⁻³
g/m ³	g/m ³	35.31

6.11 Isokinetic Variation.

6.11.1 Calculation from Raw Data.

$$I = \frac{100 T_2 (L_2 V_{1c} + (V_2 V_{1c}) (P_{1c} + 0.013.6))}{50 V_2 P_2 A_2 \frac{1}{T_2}}$$

Equation 17-7

where:

$K_1 = 0.003484 \text{ mm Hg-m}^3/\text{ml}^\circ\text{K}$ for metric units; $0.002869 \text{ in. Hg-ft}^3/\text{ml}^\circ\text{K}$ for English units.

6.11.2 Calculation from Intermediate Values.

$$I = \frac{T_2 V_2 (std) P_{std} 100}{T_{std} V_{std} A_n P_s 50 (1 - S_{ws})}$$

$$= K_2 \frac{T_2 V_2 (std)}{P_s V_s A_n (1 - S_{ws})}$$

Equation 17-8

where:

$K_2 = 4.320$ for metric units; 0.09450 for English units.

6.12 Acceptable Results. If 90 percent < I < 110 percent, the results are acceptable. If the results are low in comparison to the standard and I is beyond the acceptable range, or, if I is less than 90 percent, the Administrator may opt to accept the results. Use Citation 4 in Bibliography to make judgments. Otherwise, reject the results and repeat the test.

7. Bibliography.

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8. Vollaro, R. P. A Survey of Commercially Available Instrumentation for the Measurement of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, N.C. November, 1976 (unpublished paper).

9. Annual Book of ASTM Standards, Part 28, Gaseous Fuels: Coal and Coke: Atmospheric Analysis. American Society for Testing and Materials, Philadelphia, Pa. 1974, pp. 617-622.

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M.W.: Table 1

ELEMENTS (ICP)

METHOD: 7300
ISSUED: 2/15/84

OSHA/NIOSH/ACGIH: Table 1

PROPERTIES: Table 1

ELEMENTS: aluminum	cobalt	manganese	silver	tungsten
arsenic	copper	molybdenum	sodium	vanadium
beryllium	iron	nickel	tellurium	yttrium
cadmium	lead	phosphorus	thallium	zinc
calcium	lithium	platinum	tin	zirconium
chromium	magnesium	selenium	titanium	

SYNONYMS: vary depending upon the compound.

SAMPLING	MEASUREMENT
SAMPLER: FILTER (0.8- μ m, cellulose ester membrane)	!TECHNIQUE: INDUCTIVELY COUPLED ARGON PLASMA, ! ATOMIC EMISSION SPECTROSCOPY
FLOW RATE: 1 to 4 L/min	!ANALYTE: elements above
VOL-MIN: Table 1 -MAX: Table 1	!ASHING REAGENTS: conc. HNO ₃ , 4 mL; ! and conc. HClO ₄ , 1 mL
SHIPMENT: routine	! CONDITIONS: room temperature, 30 min; ! 150 °C to near dryness
SAMPLE STABILITY: stable	!FINAL SOLUTION: 4% HNO ₃ , 1% HClO ₄ , 10 mL
BLANKS: 2 to 10 field blanks per set	!WAVELENGTH: depends upon element; Table 2
	!BACKGROUND CORRECTION: spectral wavelength shift
	!CALIBRATION: elements in 4% HNO ₃ , 1% HClO ₄
	!RANGE: 2.5 to 1000 μ g per sample [1]
	!ESTIMATED LOD: 1 μ g per sample [1]
	!PRECISION (s _r): Table 2

APPLICABILITY: The working range of this method is 0.005 to 2.0 mg/m³ for each element in a 500-L air sample. This is simultaneous elemental analysis, not compound specific. Verify that the types of compounds in the samples are soluble with this ashing procedure.

INTERFERENCES: Spectral interferences are the primary interferences encountered in ICP-AES analysis. These are minimized by judicious wavelength selection, interelement correction factors and background correction [1,2].

OTHER METHODS: This method replaces P&CAM 351 [2] for trace elements. Atomic absorption spectroscopy (e.g., Methods 70XX) is an alternate analytical technique for many of these elements.

REAGENTS:

1. Nitric acid, conc.
2. Perchloric acid, conc.*
3. Ashing acid: 4:1 (v/v) HNO_3 : HClO_4 .
Mix 4 volumes conc. HNO_3 with
1 volume conc. HClO_4 .
4. Calibration stock solutions,
1000 $\mu\text{g/mL}$. Commercially available,
or prepared per instrument
manufacturer's recommendation (see
step 12).
5. Dilution acid, 4% HNO_3 , 1% HClO_4 .
Add 50 mL ashing acid to 600 mL
water; dilute to 1 L.
6. Argon.
7. Distilled, deionized water.

*See Special Precautions.

EQUIPMENT:

1. Sampler: cellulose ester membrane filter,
0.8- μm pore size, 37-mm diameter; in cassette
filter holder.
2. Personal sampling pump, 1 to 4 L/min, with
flexible connecting tubing.
3. Inductively coupled plasma-atomic emission
spectrometer, equipped as specified by the
manufacturer for analysis of elements of interest.
4. Regulator, two-stage, for argon.
5. Beakers, Phillips, 125-mL, or Griffin, 50-mL, with
watchglass covers.*
6. Volumetric flasks, 10- and 100- mL.*
7. Assorted volumetric pipets as needed.*
8. Hotplate, surface temperature 150 $^{\circ}\text{C}$.

*Clean all glassware with conc. nitric acid and
rinse thoroughly in distilled water before use.

SPECIAL PRECAUTIONS: Perform all perchloric acid digestions in a perchloric acid hood.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Sample at an accurately known flow rate between 1 and 4 L/min for a total sample size of
200 to 2000 L (see Table 1) for TWA measurements. Do not exceed a filter loading of
approximately 2 mg total dust.

SAMPLE PREPARATION:

3. Open the cassette filter holders and transfer the samples and blanks to clean beakers.
4. Add 5 mL ashing acid. Cover with a watchglass. Let stand 30 min at room temperature.
NOTE: Start a reagent blank at this step.
5. Heat on hotplate (120 $^{\circ}\text{C}$) until ca. 0.5 mL remains.
NOTE: Some species of Li, Mn, Mo, Sn, W, and Zr will not be completely solubilized by this
procedure. Alternative solubilization techniques for most of these elements can be
found elsewhere [2,3,4,5,6,7].
6. Add 2 mL ashing acid and repeat step 5. Repeat this step until the solution is clear.
7. Remove watchglass and rinse into the beaker with distilled water.
8. Increase the temperature to 150 $^{\circ}\text{C}$ and take the sample to dryness.
9. Dissolve the residue in 2 to 3 mL dilution acid.
10. Transfer the solutions quantitatively to 10-mL volumetric flasks.
11. Dilute to volume with dilution acid.

CALIBRATION AND QUALITY CONTROL:

12. Calibrate the spectrometer according to the manufacturers recommendations.

NOTE: Typically, an acid blank and 10 $\mu\text{g/mL}$ multi-element working standards are used. The
following multi-element combinations are chemically compatible in 4% HNO_3 /1% HClO_4 :

- a. Ag, Ca, Co, Mn, Pb, V, Zn;
- b. Al, Be, Cd, La, Li, Ni, Ti;
- c. As, B, Ba, Mg, Mo, P, Sn;

- d. Cu, Fe, Na, Pt, Sr, Te, Y;
- e. Cr, K, Sb, Se, Ti, Zr; and
- f. Si, W (distilled water only)

- 13. Analyze a standard for every ten samples.
- 14. Check recoveries with at least two spiked media blanks per ten samples.

MEASUREMENT:

- 15. Set spectrometer to conditions specified by manufacturer.
- 16. Analyze standards and samples.

NOTE: If the values for the samples are above the range of the standards, dilute the solutions with dilution acid, reanalyze and apply the appropriate dilution factor in the calculations.

CALCULATIONS:

- 17. Obtain the solution concentrations for the sample, C_s ($\mu\text{g/mL}$), and the average media blank, C_b ($\mu\text{g/mL}$), from the instrument.
- 18. Using the solution volumes of sample, V_s (mL), and media blank, V_b (mL), calculate the concentration, C (mg/m^3), of each element in the air volume sampled, V (L):

$$C = \frac{C_s V_s - C_b V_b}{V}, \text{ mg/m}^3.$$

EVALUATION OF METHOD:

Method P&CAM 351 was evaluated in 1981 [1,2]. The precision and recovery data were determined at 2.5 and 1000 μg of each element per sample on spiked filters. The precision and recovery data, instrumental detection limits, sensitivity, and analytical wavelengths are listed in Table 2. The values in Table 2 were determined with a Jarrell-Ash Model 1160 ICP operated according to manufacturer's instructions.

REFERENCES:

- [1] Hull, R.D. "Multielement Analysis of Industrial Hygiene Samples," NIOSH Internal Report, presented at the American Industrial Hygiene Conference, Portland, Oregon (May 1981).
- [2] NIOSH Manual of Analytical Methods, 2nd ed., V. 7, P&CAM 351, U.S. Department of Health and Human Services, Publ. (NIOSH) 82-100 (1981).
- [3] Ibid, S341 (Lead).
- [4] Ibid, V. 2, S5 (Manganese), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-B (1977).
- [5] Ibid, V. 4, P&CAM 271 (Tungsten), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 78-175 (1978).
- [6] Ibid, V. 5, P&CAM 173 (Metals by Atomic Absorption), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 79-141 (1979).
- [7] Ibid, V. 3, S183 (Tin), S185 (Zirconium), and S376 (Molybdenum), U.S. Department of Health, Education, and Welfare, Publ. (NIOSH) 77-157-C (1977).

METHOD REVISED BY: R. DeLon Hull and Mark Millson, NIOSH/DPSE.

Table 1. Properties and sampling volumes.

Element (Symbol)	Properties		Permissible Exposure Limits, mg/m ³ TWA OSHA/NIOSH/ACGIH	Air Volume @ OSHA, L	
	Atomic Weight	MP, °C		MIN	MAX
Silver (Ag)	107.87	961	0.01/ — / 0.1	250	2000
Aluminum (Al)	26.98	660	— / — / 10.	5 (g)	100 (g)
Arsenic (As)	74.92	817*	0.5/C 0.002/ 0.2	5	2000
Beryllium (Be)	9.01	1278	0.002/ 0.0005/ 0.002	1250	2000
Calcium (Ca)	40.08	842	5 (b)/ — / 2 (b)	5	200
Cadmium (Cd)	112.40	321	0.2/ 0.04/ 0.05	13	2000
Cobalt (Co)	58.93	1495	0.1/ — / 0.1	25	2000
Chromium (Cr)	52.00	1890	1.0 (c)/ 0.025/ 0.5 (c)	5	1000
Copper (Cu)	63.54	1083	1.0/ — / 1.0	5	1000
Iron (Fe)	55.85	1535	10 (b)/ — / 5 (b)	5	100
Lithium (Li)	6.94	179	0.025 (d)/ — / 0.025 (d)	100	2000
Magnesium (Mg)	24.31	651	15 (b)/ — / 10 (b)	5	67
Manganese (Mn)	54.94	1244	C 5/ — / C 5	5	200
Molybdenum (Mo)	95.94	651	15 (e)/ — / 10 (e)	5	67
Sodium (Na)	22.99	98	2 (f)/ C 2 (f)/ C 2 (f)	13	2000
Nickel (Ni)	58.71	1453	1/ 0.015/ 1 (c)	5	1000
Phosphorus (P)	30.97	44	— / — / 0.1	25 (g)	2000 (g)
Lead (Pb)	207.19	328	0.05/ 0.1/ 0.15	50	2000
Platinum (Pt)	195.09	1769	0.002 (a)/ — / 1 (c)	1250	2000
Selenium (Se)	78.96	217	0.2/ — / —	13	2000
Tin (Sn)	118.69	232	2/ — / 2 (c)	5	500
Tellurium (Te)	127.60	450	0.1/ — / 0.1	25	2000
Titanium (Ti)	47.90	1675	— / — / 10 (b)	5	100
Thallium (Tl)	204.37	304	0.1 (a)/ — / 0.1 (a)	25	2000
Vanadium (V)	50.94	1890	C 0.5/ 1 (c)/ 0.05 (V ₂ O ₅)	5	2000
Tungsten (W)	183.85	3410	— / 5 (e)/ 5 (e)	5 (g)	200 (g)
Yttrium (Y)	88.91	1495	1/ — / 1	5	1000
Zinc (Zn)	65.37	419	5 (b)/ 5 (b)/ 5 (b)	5	200
Zirconium (Zr)	91.22	1852	5/ — / 5	5	200

(a) soluble

(b) oxide

(c) metal

(d) hydride

(e) insoluble

(f) hydroxide

(g) at the ACGIH TLV

METHOD: 7300

ELEMENTS (ICP)

Table 2. Measurement procedures and data (a).

Element	Wavelength (nm)	Instrumental LOD (ng/mL)	Sensitivity (Intensity/ µg/mL)	Recovery (%)		Precision (s _r) (N = 3)	
				@ 2.5 µg/ filter (b)	@ 1000 µg/ filter	@ 2.5 µg/ filter	@ 1000 µg/ filter
Ag	328.3	26	0.65	111	91	0.02	0.075
Al	308.2	14	0.23	93	100	0.092	0.023
As	193.7	13	0.57	103	99	0.062	0.026
Be	313.0	1.5	1.29	107	90	0.040	0.034
Ca	315.9	10	0.49	99	95	0.036	0.014
Cd	226.5	1.6	0.83	107	99	0.032	0.020
Co	231.2	7.4	0.38	101	95	0.040	0.005
Cr	205.6	1.3	0.50	98	106	0.053	0.016
Cu	324.8	2.1	0.72	98	99	0.036	0.022
Fe	259.9	3.9	0.13	94	97	0.068	0.016
Li	670.8	2.8	0.48	89	95	0.171	0.043
Mg	279.6	24	0.22	105	106	0.084	0.027
Mn	257.6	0.4	0.74	84	93	0.062	0.035
Mo	281.6	7.0	0.18	94	88	0.023	0.049
Na	589.0	10	0.76	(c)	101	(c)	0.045
Ni	231.6	3.4	0.41	105	97	0.027	0.020
P	214.9	22	0.17	(c)	91	(c)	0.056
Pb	220.4	17	0.42	105	95	0.060	0.011
Pt	203.7	15	0.69	106	91	0.041	0.075
Se	190.6	21	0.28	105	97	0.068	0.049
Sn	190.0	64	0.49	74	67	0.33	0.16
Te	214.3	29	0.41	102	94	0.050	0.063
Ti	334.9	1.2	0.55	96	108	0.051	0.029
Tl	190.9	17	0.22	103	99	0.043	0.017
V	310.2	3.2	0.88	99	94	0.043	0.014
W	207.9	13	2.58	35	23	0.053	0.60
Y	371.0	0.8	2.35	99	100	0.015	0.013
Zn	213.9	0.6	0.60	101	94	0.013	0.013
Zr	339.2	1.9	0.88	75	98	0.049	0.008

(a) Values reported were obtained with a Jarrell-Ash Model 1160 ICP; performance may vary with instrument and should be independently verified.

(b) 2.5 µg/filter corresponds to 5 µg/m³ for a 500-L air sample.

(c) Blank levels too high to make accurate determinations

Source category: Talc Processing
 Plant name :
 Process :
 Filename: TALC_R16.WQ1
 Location:
 Test date:
 Date: 11/16/94
 Ref. No.: 16/
 Process rate basis: feed

Source	Type of control	Pollutant	Run No.	Test Method	Isokinetic, %	Gas volume, DSCF	Volum. flow rate, DSCFM	Mass, g	Concen., gr/DSCF	Emission rate, lb/hr	Process rate, ton/hr	Emission factor	
												kg/Mg	lb/ton
Roller mill	fabric filter	filterable PM	1	EPA 17	100.1	98.10	4,916	0.0218	0.00343	0.145	7	0.010	0.021
		cadmium	1	EPA 29	100.1	98.10	4,916	4.77E-07	7.50E-08	3.16E-06	7	2.3E-07	4.5E-07
		chromium	1	EPA 29	100.1	98.10	4,916	9.40E-07	1.48E-07	6.23E-06	7	4.5E-07	8.9E-07
		nickel	1	EPA 29	100.1	98.10	4,916	1.34E-05	2.11E-06	8.88E-05	7	6.3E-06	1.3E-05

Data not rated because only one run was conducted and average process rates provided; run-by-run rates not specified.

Basis for rating:

Problems noted:

Other notes: