FINAL REPORT
EMISSIONS TESTING OF COMBUSTION STACK AND PUSHING OPERATIONS AT COKE BATTERY NO. 2 AT BETHLEHEM STEEL CORPORATION'S BURNS HARBOR DIVISION IN CHESTERTON, INDIANA

Volume III
Appendices D through G

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CALCULATIONS

Nomenclature and Dimensions

An = Cross-sectional area of sampling nozzle, ft^2

As = Cross-sectional area of stack, ft^2

Bws = Proportion by volume of water vapor in the gas stream, dimensionless

Cp = Pitot tube coefficient, dimensionless

Cs = Concentration of pollutant matter in stack gas-dry basis, grains per dry standard cubic foot (gr/dscf)

%CO = Percent of carbon monoxide by volume, dry basis

$\%CO_2$ = Percent of carbon dioxide by volume, dry basis

ΔH = Average pressure drop across the sampling meter flow orifice, inches of water (in. H_2O)

GCV = Gross calorific value, Btu/lb

I = Percent of isokinetic sampling

La = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change/ equal to 0.020 cubic foot per minute or 4% of the average sampling rate, whichever is less

Md = Dry molecular weight, lb/lb-mole

Mn = Total amount of pollutant matter collected, milligrams (mg)

Ms = Molecular weight of stack gas (wet basis), lb/lb-mole

$\%N_2$ = Percent of nitrogen by volume, dry basis

$\%O_2$ = Percent of oxygen by volume, dry basis

ΔP = Velocity head of stack gas, inches of water (in. H_2O)

Pbar = Barometric pressure, inches of mercury (in. Hg)

Ps = Absolute stack gas pressure, inches of mercury (in. Hg)

Pstd = Gas pressure at standard conditions, inches of mercury (29.92 in. Hg)

pmr = Pollutant matter emission rate, pounds per hour (lb/hr)

Qs = Volumetric flow rate - wet basis at stack conditions, actual cubic feet per minute (acfm)

Qsstd = Volumetric flow rate - dry basis at stack conditions, actual cubic feet per minute (dscfm)

Tm = Average temperature of dry gas meter, °R

Ts = Average temperature of stack gas, °R

Tstd = Temperature at standard conditions, 528°R

Vlc = Total volume of liquid collected in impingers, ml

Vsg = Volume of moisture collected in silica gel, grams

Vm = Volume of dry gas sampled at meter conditions, ft^3

Vmstd= Volume of dry gas sampled at standard conditions, ft^3

Vs = Average stack gas velocity at stack conditions, ft/s

Vwstd = Volume of water vapor at standard conditions, scf

γ = Dry gas meter calibration factor, dimensionless

Θ = Total sampling time, minutes

NOTE: Standard conditions = 68°F and 29.92 in. Hg

Example Calculations for Pollutant Emissions

1. Volume of dry gas sampled corrected to standard conditions, ft3.
Note: Vm must be corrected for leakage if any leakage rates exceed La.

$$Vmstd = 17.647 * Vm * \gamma * \frac{Pbar + \frac{\Delta H}{13.6}}{Tm, {}^{\circ}R}$$

2. Volume of water vapor at standard conditions, ft^3.

$$Vmstd = 0.04707 * Vlc + 0.04715 * Vsg$$

3. Moisture content in stack gas, dimension less.

$$Bws = \frac{Vwstd}{Vwstd + Vmstd}$$

4. Dry molecular weight of stack gas, lb/lb -mole.

$$Md = 0.44 * \%CO_2 + 0.32 * \%O_2 + 0.28 * (\%N_2 + \%CO)$$

5. Molecular weight of stack gas, lb/lb-mole.

$$Ms = Md\,(1\text{-}Bws) + 18 * BWS$$

6. Stack velocity at stack conditions, f/s.

$$Vs = 85.49 * Cp * ave\sqrt{\Delta P} * \sqrt{\frac{Ts, {}^{\circ}R}{Ps * Ms}}$$

7. Stack gas volumetric flow rate at stack conditions, cfm.

$$Qs = 60 * Vs * As$$

8. Dry stack gas volumetric flow rate at standard conditions, cfm.

$$Qsstd = 17.647 * Qs * \frac{Ps}{Ts, {}^{\circ}R} * (1 - Bws)$$

9. Concentration in gr/dscf.

$$Cs = 0.01543 * \frac{Mn}{Vmstd}$$

10. Concentration in lb/dscf.

$$Cs,\ lb/dscf = \frac{gr/dscf}{7000}$$

11. Pollutant mass emission rate, lb/hr.

$$Pmr,\ lb/hr = lb/dscf * Qsstd * 60$$

12. Pollutant mass emission rate, lb/MMBtu.

$$pmr,\ lb/MMBtu = \frac{pmr,\ lb/hr}{MMBtu/hr}$$

13. F-factor, Fd.

$$Fd = \frac{10^6*(3.64*\%H) + (1.53*\%C) + (0.57*\%S) + (0.14*\%N) - (0.46*\%O_2)}{GCV\ (Btu/lb)}$$

14. F-factor, pollutant mass emission rate, lb/MMBtu.

$$= \frac{lb/dscf * F * 20.9}{(20.9 - \%O_2)}$$

15. Heat imput, MMBtu/hr fuel.

$$= \frac{GVC\ (Btu/lb) * Feed\ Rate\ (lb/hr)}{10^6}$$

16. Heat input, MMBtu/hr, F-factor.

$$= \frac{Qsstd}{Fd} * ((20.9 - \%O_2) + 20.9) * 60$$

Summary of Stack Gas Parameters and Test Results
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Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
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	RUN NUMBER	*B-I-315-1*	*B-I-315-2*	*B-I-315-3*	*Average*
	RUN DATE	*8/11/98*	*8/12/98*	*8/13/98*	
	RUN TIME	*1035-2020*	*0922-1941*	*0842-1736*	
	MEASURED DATA				
P_{static}	Stack Static Pressure, inches H_2O	-2.20	-2.30	-2.20	-2.23
y	Meter Box Correction Factor	1.012	1.012	1.012	1.012
P_{bar}	Barometric Pressure, inches Hg	29.70	29.80	29.80	29.77
V_m	Sample Volume, ft^3	126.092	123.572	121.955	123.873
$Dp^{1/2}$	Average Square Root Dp, (in. H_2O)$^{1/2}$	0.2980	0.2964	0.2926	0.2957
DH	Avg Meter Orifice Pressure, in. H_2O	0.2870	0.2905	0.2844	0.2873
T_m	Average Meter Temperature, °F	80	80	85	82
T_s	Average Stack Temperature, °F	153	158	170	160
V_{lc}	Condensate Collected, ml	66.6	60.9	50.9	59.5
CO_2	Carbon Dioxide content, % by volume	0.5	0.5	0.5	0.5
O_2	Oxygen content, % by volume	20.05	20.0	20.0	20.0
N_2	Nitrogen content, % by volume	79.5	79.5	79.5	79.5
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	114	114	114	114.00
Q	Sample Run Duration, minutes	459	510	483	484
D_n	Nozzle Diameter, inches	0.215	0.215	0.215	0.215
Push	Tons of Coke pushed	1,115	1,128	998	1,080
	Total Test Time, hours	9.75	10.32	8.90	9.66
	Tons of Coke per Hour	114.4	109.3	112.1	111.9
	CALCULATED DATA	**All Calculations are on Time Weighted Average Basis**			
A_n	Nozzle Area, ft^2	0.000252	0.000252	0.000252	0.000252
$V_{m(std)}$	Standard Meter Volume, ft^3	123.890	121.824	119.125	121.613
$V_{m(std)}$	Standard Meter Volume, m^3	3.508	3.450	3.373	3.444
Q_m	Average Sampling Rate, dscfm	0.270	0.239	0.247	0.252
P_s	Stack Pressure, inches Hg	29.54	29.63	29.64	29.60
B_{ws}	Moisture, % by volume	2.5	2.3	2.0	2.2
$B_{ws(sat)}$	Moisture (at saturation), % by volume	27.5	31.0	41.1	33.2
V_{wstd}	Standard Water Vapor Volume, ft^3	3.135	2.867	2.396	2.799
$1-B_{ws}$	Dry Mole Fraction	0.975	0.977	0.980	0.978
M_d	Molecular Weight (d.b.), lb/lb•mole	28.88	28.88	28.88	28.88
M_s	Molecular Weight (w.b.), lb/lb•mole	28.61	28.63	28.67	28.64
V_s	Stack Gas Velocity, ft/s	18.2	18.2	18.1	18.2
A	Stack Area, ft^2	70.9	70.9	70.9	70.88
Q_a	Stack Gas Volumetric flow, acfm	77,509	77,263	76,952	77,242
Q_s	Stack Gas Volumetric flow, dscfm	64,257	63,845	62,601	63,568
Q_s	Stack Gas Volumetric flow, dscmm	1,820	1,808	1,773	1,800
I	Isokinetic Sampling Ratio, %	118.1	105.2	110.8	111.4

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	EMISSIONS DATA				
	EMISSIONS DATA				
	Particulate Matter				
PM	Total Catch, g	2.7253	3.7194	2.7629	3.0692
$_{PM}$ @ 7% O	Concentration, gr/dscf @ 7% O2	5.5513	7.2767	5.5279	6.1187
C_{PM}	Concentration, g/dscm	0.777	1.078	0.819	0.891
$_{PM}$ @ 7% O	Concentration, g/dscm @ 7% O2	1.27E+01	1.67E+01	1.26E+01	1.40E+01
E_{PM}	Emission Rate, µg/hr	8.48E+10	1.17E+11	8.71E+10	9.63E+10
C_{PM}	Concentration, gr/dscf	0.339	0.471	0.358	0.390
C_{PM}	Concentration, lb/dscf	4.85E-05	6.73E-05	5.11E-05	5.56E-05
E_{PM}	Emission Rate, lb/hr	187.0	257.8	192.1	212.3
Push	Pounds per Ton of Coke Pushed	1.63	2.36	1.71	1.90
	Extractable Organic Matter				
EOM	Total Catch, g	0.0082	0.0090	0.0065	0.0079
$_{EOM}$ @ 7%	Concentration, gr/dscf @ 7% O2	0.0167	0.0176	0.0130	0.0158
C_{EOM}	Concentration, g/dscm	2.34E-03	2.61E-03	1.93E-03	2.29E-03
$_{EOM}$ @ 7%	Concentration, g/dscm @ 7% O2	3.82E-02	4.03E-02	2.98E-02	3.61E-02
E_{EOM}	Emission Rate, µg/hr	2.55E+08	2.83E+08	2.05E+08	2.48E+08
C_{EOM}	Concentration, gr/dscf	1.02E-03	1.14E-03	8.42E-04	1.00E-03
C_{EOM}	Concentration, lb/dscf	1.46E-07	1.63E-07	1.20E-07	1.43E-07
E_{EOM}	Emission Rate, lb/hr	0.563	0.624	0.452	0.546
Push	Pounds per Ton of Coke Pushed	4.92E-03	5.71E-03	4.03E-03	4.89E-03

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	<u>Antimony</u>				
Sb	Target Catch, µg	4.3	3.7	3.4	3.8
C_{Sb}	Concentration, µg/dscm	1.23	1.07	1.01	1.10
C_{Sb} @ 7% O_2	Concentration, µg/dscm @ 7%	20.0	16.6	15.6	17.4
E_{Sb}	Emission Rate, µg/hr	133,815	116,345	107,204	119,121
E_{Sb}	Emission Rate, lb/hr	2.94E-04	2.56E-04	2.36E-04	2.62E-04
Push	Pounds per Ton of Coke Pushed	2.57E-06	2.34E-06	2.10E-06	2.34E-06
	<u>Arsenic</u>				
As	Target Catch, µg	62.9	81.8	92.3	79.00
C_{As}	Concentration, µg/dscm	17.9	23.7	27.4	23.0
C_{Sb} @ 7% O_2	Concentration, µg/dscm @ 7%	293	366	423	361
E_{As}	Emission Rate, µg/hr	1,957,431	2,572,166	2,910,274	2,479,957
E_{As}	Emission Rate, lb/hr	4.31E-03	5.66E-03	6.40E-03	5.46E-03
Push	Pounds per Ton of Coke Pushed	3.77E-05	5.18E-05	5.71E-05	4.88E-05
	<u>Barium</u>				
Ba	Target Catch, µg	26.6	34.9	27.6	29.70
C_{Ba}	Concentration, µg/dscm	7.58	10.12	8.18	8.63
C_{Ba} @ 7% O_2	Concentration, µg/dscm @ 7%	124	156	126	136
E_{Ba}	Emission Rate, µg/hr	827,785	1,097,416	870,245	931,815
E_{Ba}	Emission Rate, lb/hr	1.82E-03	2.41E-03	1.91E-03	2.05E-03
Push	Pounds per Ton of Coke Pushed	1.59E-05	2.21E-05	1.71E-05	1.84E-05

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	EMISSIONS DATA - Continued				
	<u>Beryllium</u>				
Be	Target Catch, µg	1.0	1.1	1.0	1.02
C_{Be}	Concentration, µg/dscm	0.274	0.310	0.302	0.295
C_{Be} @ 7% O_2	Concentration, µg/dscm @ 7%	4.47E+00	4.79E+00	4.67E+00	4.65E+00
E_{Be}	Emission Rate, µg/hr	29,875	33,646	32,161	31,894
E_{Be}	Emission Rate, lb/hr	6.57E-05	7.40E-05	7.08E-05	7.02E-05
Push	Pounds per Ton of Coke Pushed	5.75E-07	6.77E-07	6.31E-07	6.28E-07
	<u>Cadmium</u>				
Cd	Target Catch, µg	1.3	2.1	2.6	2.0
C_{Cd}	Concentration, µg/dscm	0.371	0.609	0.771	0.583
C_{Cd} @ 7% O_2	Concentration, µg/dscm @ 7%	6.06E+00	9.40E+00	1.19E+01	9.12E+00
E_{Cd}	Emission Rate, µg/hr	40,456	66,034	81,980	62,823
E_{Cd}	Emission Rate, lb/hr	8.90E-05	1.45E-04	1.80E-04	1.38E-04
Push	Pounds per Ton of Coke Pushed	7.78E-07	1.33E-06	1.61E-06	1.24E-06
	<u>Chromium</u>				
Cr	Target Catch, µg	6.0	9.7	6.8	7.50
C_{Cr}	Concentration, µg/dscm	1.71	2.81	2.02	2.18
C_{Cr} @ 7% O_2	Concentration, µg/dscm @ 7%	2.80E+01	4.34E+01	3.11E+01	3.42E+01
E_{Cr}	Emission Rate, µg/hr	186,718	305,012	214,408	235,380
E_{Cr}	Emission Rate, lb/hr	4.11E-04	6.71E-04	4.72E-04	5.18E-04
Push	Pounds per Ton of Coke Pushed	3.59E-06	6.14E-06	4.21E-06	4.65E-06

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	EMISSIONS DATA - Continued				
	<u>Colbalt</u>				
Co	Target Catch, µg	3.0	5.0	3.0	3.67
C_{Co}	Concentration, µg/dscm	0.855	1.449	0.889	1.065
C_{Co} @ 7% O_2	Concentration, µg/dscm @ 7%	1.40E+01	2.24E+01	1.37E+01	1.67E+01
E_{Co}	Emission Rate, µg/hr	93,359	157,223	94,592	115,058
E_{Co}	Emission Rate, lb/hr	2.05E-04	3.46E-04	2.08E-04	2.53E-04
Push	Pounds per Ton of Coke Pushed	1.80E-06	3.16E-06	1.86E-06	2.27E-06
	<u>Copper</u>				
Cu	Target Catch, µg	14.0	21.0	20.0	18.33
C_{Cu}	Concentration, µg/dscm	3.99	6.09	5.93	5.34
C_{Cu} @ 7% O_2	Concentration, µg/dscm @ 7%	6.53E+01	9.40E+01	9.16E+01	8.36E+01
E_{Cu}	Emission Rate, µg/hr	435,676	660,336	630,612	575,541
E_{Cu}	Emission Rate, lb/hr	9.58E-04	1.45E-03	1.39E-03	1.27E-03
Push	Pounds per Ton of Coke Pushed	8.38E-06	1.33E-05	1.24E-05	1.13E-05
	<u>Lead</u>				
Pb	Target Catch, µg	102.3	152.3	127.3	127.3
C_{Pb}	Concentration, µg/dscm	29.2	44.1	37.7	37.0
C_{Pb} @ 7% O_2	Concentration, µg/dscm @ 7%	4.77E+02	6.82E+02	5.83E+02	5.81E+02
E_{Pb}	Emission Rate, µg/hr	3,183,548	4,789,009	4,013,845	3,995,467
E_{Pb}	Emission Rate, lb/hr	7.00E-03	1.05E-02	8.83E-03	8.79E-03
Push	Pounds per Ton of Coke Pushed	6.12E-05	9.64E-05	7.87E-05	7.88E-05

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	EMISSIONS DATA - Continued				
	<u>Manganese</u>				
Mn	Target Catch, µg	39.0	43.0	41.0	41.00
C_{Mn}	Concentration, µg/dscm	11.1	12.5	12.2	11.9
C_{Mn} @ 7% O_2	Concentration, µg/dscm @ 7%	1.82E+02	1.93E+02	1.88E+02	1.87E+02
E_{Mn}	Emission Rate, µg/hr	1,213,669	1,352,117	1,292,755	1,286,180
E_{Mn}	Emission Rate, lb/hr	2.67E-03	2.97E-03	2.84E-03	2.83E-03
Push	Pounds per Ton of Coke Pushed	2.33E-05	2.72E-05	2.54E-05	2.53E-05
	<u>Mercury</u>				
Hg	Target Catch, µg	0.5	0.6	0.5	0.53
C_{Hg}	Concentration, µg/dscm	0.14	0.17	0.15	0.15
C_{Hg} @ 7% O_2	Concentration, µg/dscm @ 7%	2.33E+00	2.69E+00	2.29E+00	2.44E+00
E_{Hg}	Emission Rate, µg/hr	15,560	18,867	15,765	16,731
E_{Hg}	Emission Rate, lb/hr	3.42E-05	4.15E-05	3.47E-05	3.68E-05
Push	Pounds per Ton of Coke Pushed	2.99E-07	3.80E-07	3.09E-07	3.29E-07
	<u>Nickel</u>				
Ni	Target Catch, µg	87.0	86.5	61.4	78.30
C_{Ni}	Concentration, µg/dscm	24.8	25.1	18.2	22.7
C_{Ni} @ 7% O_2	Concentration, µg/dscm @ 7%	4.06E+02	3.87E+02	2.81E+02	3.58E+02
E_{Ni}	Emission Rate, µg/hr	2,707,416	2,719,956	1,935,979	2,454,450
E_{Ni}	Emission Rate, lb/hr	5.96E-03	5.98E-03	4.26E-03	5.40E-03
Push	Pounds per Ton of Coke Pushed	5.21E-05	5.47E-05	3.80E-05	4.83E-05

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EMISSIONS DATA - Continued

	<u>Phosphorus</u>				
P	Target Catch, µg	40.0	45.0	43.0	42.67
C_P	Concentration, µg/dscm	11.4	13.0	12.7	12.4
C_P @ 7% O_2	Concentration, µg/dscm @ 7%	1.86E+02	2.01E+02	1.97E+02	1.95E+02
E_P	Emission Rate, µg/hr	1,244,789	1,415,006	1,355,816	1,338,537
E_P	Emission Rate, lb/hr	2.74E-03	3.11E-03	2.98E-03	2.94E-03
Push	Pounds per Ton of Coke Pushed	2.39E-05	2.85E-05	2.66E-05	2.63E-05
	<u>Selenium</u>				
Se	Target Catch, µg	14.4	21.1	19.1	18.20
C_{Se}	Concentration, µg/dscm	4.10	6.12	5.66	5.29
C_{Se} @ 7% O_2	Concentration, µg/dscm @ 7%	6.71E+01	9.45E+01	8.74E+01	8.30E+01
E_{Se}	Emission Rate, µg/hr	448,124	663,481	602,234	571,280
E_{Se}	Emission Rate, lb/hr	9.86E-04	1.46E-03	1.32E-03	1.26E-03
Push	Pounds per Ton of Coke Pushed	8.62E-06	1.34E-05	1.18E-05	1.13E-05
	<u>Silver</u>				
Ag	Target Catch, µg	0.3	0.6	0.4	0.43
C_{ag}	Concentration, µg/dscm	0.0855	0.1739	0.1186	0.1260
C_{Ag} @ 7% O_2	Concentration, µg/dscm @ 7%	1.40E+00	2.69E+00	1.83E+00	1.97E+00
E_{Ag}	Emission Rate, µg/hr	9,336	18,867	12,612	13,605
E_{Ag}	Emission Rate, lb/hr	2.05E-05	4.15E-05	2.77E-05	2.99E-05
Push	Pounds per Ton of Coke Pushed	1.80E-07	3.80E-07	2.47E-07	2.69E-07

Summary of Stack Gas Parameters and Test Results
S511.000
Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
Baghouse Inlet
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	RUN NUMBER	***B-I-315-1***	***B-I-315-2***	***B-I-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1035-2020***	***0922-1941***	***0842-1736***	
	EMISSIONS DATA - Continued				
	<u>Thallium</u>				
Tl	Target Catch, µg	3.7	4.9	4.6	4.40
C_{Tl}	Concentration, µg/dscm	1.05	1.42	1.36	1.28
C_{Tl} @ 7% O_2	Concentration, µg/dscm @ 7%	1.72E+01	2.19E+01	2.11E+01	2.01E+01
E_{Tl}	Emission Rate, µg/hr	115,143	154,078	145,041	138,087
E_{Tl}	Emission Rate, lb/hr	2.53E-04	3.39E-04	3.19E-04	3.04E-04
Push	Pounds per Ton of Coke Pushed	2.22E-06	3.10E-06	2.85E-06	2.72E-06
	<u>Zinc</u>				
Zn	Target Catch, µg	167.1	231.1	217.1	205.10
C_{Zn}	Concentration, µg/dscm	47.6	67.0	64.4	59.7
C_{Zn} @ 7% O_2	Concentration, µg/dscm @ 7%	7.79E+02	1.03E+03	9.94E+02	9.36E+02
E_{Zn}	Emission Rate, µg/hr	5,200,107	7,266,841	6,845,293	6,437,414
E_{Zn}	Emission Rate, lb/hr	1.14E-02	1.60E-02	1.51E-02	1.42E-02
Push	Pounds per Ton of Coke Pushed	1.00E-04	1.46E-04	1.34E-04	1.27E-04

Method 315/29 Analytical Results (ug) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Baghouse Inlet

Target Catch, ug/sample							
Metal	B-I-315-1	B-I-315-1	B-I-315-2	B-I-315-2	B-I-315-3	B-I-315-3	Filter Blank
		Corrected		Corrected		Corrected	
Antimony	4.3	4.3	3.7	3.7	3.4	3.4	0.0
Arsenic	62.9	62.9	81.8	81.8	92.3	92.3	0.0
Barium	27.4	26.6	35.7	34.9	28.4	27.6	0.8
Beryllium	1.0	1.0	1.1	1.1	1.0	1.0	0.0
Cadmium	3.0	1.3	3.8	2.1	4.3	2.6	1.7
Chromium	6.0	6.0	9.7	9.7	6.8	6.8	0.0
Colbalt	3.0	3.0	5.0	5.0	3.0	3.0	0.0
Copper	14.0	14.0	21.0	21.0	20.0	20.0	0.0
Lead	103.0	102.3	153.0	152.3	128.0	127.3	0.7
Manganese	42.0	39.0	46.0	43.0	44.0	41.0	3.0
Mercury	0.5	0.5	0.6	0.6	0.5	0.5	0.0
Nickel	87.0	#VALUE!	86.5	#VALUE!	61.4	#VALUE!	
Phosphorus	40.0	40.0	45.0	45.0	43.0	43.0	0.0
Selenium	14.4	14.4	21.1	21.1	19.1	19.1	0.0
Silver	0.3	0.3	0.6	0.6	0.4	0.4	0.0
Thallium	3.7	3.7	4.9	4.9	4.6	4.6	0.0
Zinc	178.0	167.1	242.0	231.1	228.0	217.1	10.9

Method 315 Analytical Results (g) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Baghouse Inlet

All Weights are listed in grams

Run No.	Filters		Acetone-FHR	MeCl2 -FHR	Acetone-BHR	MeCl2 -BHR	H2O Imp. Rinse	Corrected Total	
	PM	**MCEM**	**PM**	**MCEM**	**MCEM**	**MCEM**	**MCEM**	**PM**	**MCEM**
B-I-315-1	2.5592	0.0008	0.17	0.0022	0.0024	0.0019	0.003	**2.7253**	**0.0082**
B-I-315-2	3.3744	0.0003	0.3489	0.0012	0.0016	0.0044	0.0036	**3.7194**	**0.009**
B-I-315-3	2.6308	0.002	0.1360	0.0017	0.0013	0.0006	0.0030	**2.7629**	**0.0065**
Blank Values	0.0017	0.0003	0.0022	0.0001	0.00	0.0001	0.0016	**0.0039**	**0.0021**

Summary of Stack Gas Parameters and Test Results
Bethlehem Steel-Chesterton, Indiana
CARB Method 429 - PAH's
Baghouse Inlet

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	RUN NUMBER	B-I-429-1	B-I-429-2	B-I-429-3	
	RUN DATE	8/11/98	8/12/98	8/13/98	***Average***
	RUN TIME	1030-2016	0924-1935	1332-1934	
	MEASURED DATA				
γ	Meter Box Correction Factor	1.005	1.005	1.005	1.005
ΔH	Avg. Meter Orifice Pressure, in. H_2O	0.2642	0.2385	0.2488	0.2505
P_{bar}	Barometric Pressure, inches Hg	29.70	29.80	29.80	29.77
V_m	Sample Volume, ft^3	88.661	87.370	69.186	81.739
T_m	Average Meter Temperature, °F	76	79	80	78
P_{static}	Stack Static Pressure, inches H_2O	-2.20	-2.30	-2.30	-2.27
T_s	Average Stack Temperature, °F	142	147	145	145
V_{lc}	Condensate Collected, ml	63.3	64.7	59.0	62.333
CO_2	Carbon Dioxide content, % by volume	0.5	0.5	0.5	0.50
O_2	Oxygen content, % by volume	20.05	20.00	20.00	20.02
N_2	Nitrogen content, % by volume	79.45	79.50	79.50	79.48
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
$\Delta p^{1/2}$	Average Square Root Dp, (in. $H_2O)^{1/2}$	0.2716	0.2467	0.2668	0.2617
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	114	114	114	114
Θ	Sample Run Duration, minutes	453	482	362	432
D_n	Nozzle Diameter, inches	0.217	0.215	0.215	0.216
	Tons of Coke pushed	1,115	1,128	660	968
	Total Test Time, hours	9.77	10.18	6.03	8.66
	Tons of Coke per Hour	114.1	110.8	109.5	111.5
	CALCULATED DATA				
A_n	Nozzle Area, ft^2	0.000257	0.000252	0.000252	0.000254
$V_{m(std)}$	Standard Meter Volume, dscf	87.151	85.686	67.729	80.189
$V_{m(std)}$	Standard Meter Volume, dscm	2.468	2.426	1.918	2.271
B_{ws}	Moisture, % by volume	3.3	3.4	3.9	3.6
$B_{ws(sat)}$	Moisture (at saturation), % by volume	20.9	23.7	22.5	22.352
V_{wstd}	Standard Water Vapor Volume, ft^3	2.980	3.045	2.777	2.934
$1\text{-}B_{ws}$	Dry Mole Fraction	0.967	0.966	0.961	0.964
M_d	Molecular Weight (d.b.), lb/lb•mole	28.88	28.88	28.88	28.88
M_s	Molecular Weight (w.b.), lb/lb•mole	28.52	28.51	28.45	28.49
V_s	Stack Gas Velocity, ft/s	16.5	15.0	16.2	15.9
A	Stack Area, ft^2	70.9	70.9	70.9	70.88
Q_a	Stack Gas Volumetric flow, acfm	70,118	63,871	69,027	67,672
Q_s	Stack Gas Volumetric flow, dscfm	58,683	53,111	57,287	56,361
$Q_{s(cmm)}$	Stack Gas Volumetric flow, dscmm	1,662	1,504	1,622	1,596
I	Isokinetic Sampling Ratio, %	90.5	94.1	91.8	92.2

Baghouse Inlet

	B-I-429-1	B-I-429-2	B-I-429-3	Average
Naphthalene				
Molecular Weight, g/g-mole	128.16	128.16	128.16	
Target Catch, ng	26,480	34,480	28,480	29,813
ng to µg	26.48	34.48	28.48	29.81
Concentration, µg/dscm [a]	10.7	14.2	14.8	13.3
Emission Rate, µg/hr [b]	1,069,819	1,282,322	1,445,362	1,265,834
Emission Rate, lb/hr [c]	2.36E-03	2.83E-03	3.19E-03	2.79E-03
Pounds per Ton of Coke Pushed	2.07E-05	2.55E-05	2.91E-05	2.51E-05
2-Methylnaphthalene				
Molecular Weight, g/g-mole	142.20	142.20	142.20	
Target Catch, ng	7,886	6,786	2,986	5,886
ng to µg	7.89	6.79	2.99	5.89
Concentration, µg/dscm [a]	3.20	2.80	1.56	2.52
Emission Rate, µg/hr [b]	318,602	252,373	151,540	240,838
Emission Rate, lb/hr [c]	7.02E-04	5.56E-04	3.34E-04	5.31E-04
Pounds per Ton of Coke Pushed	6.15E-06	5.02E-06	3.05E-06	4.74E-06
Acenaphthylene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	5,500	5,900	2,600	4,667
ng to µg	5.50	5.90	2.60	4.67
Concentration, µg/dscm [a]	2.23	2.43	1.36	2.01
Emission Rate, µg/hr [b]	222,206	219,423	131,950	191,193
Emission Rate, lb/hr [c]	4.90E-04	4.84E-04	2.91E-04	4.22E-04
Pounds per Ton of Coke Pushed	4.29E-06	4.37E-06	2.66E-06	3.77E-06
Acenaphthene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	690.00	470.00	530.00	563.33
ng to µg	0.69	0.47	0.53	0.56
Concentration, µg/dscm [a]	0.280	0.194	0.276	0.250
Emission Rate, µg/hr [b]	27,877	17,479	26,898	24,085
Emission Rate, lb/hr [c]	6.15E-05	3.85E-05	5.93E-05	5.31E-05
Pounds per Ton of Coke Pushed	5.39E-07	3.48E-07	5.42E-07	4.76E-07
Fluorene				
Molecular Weight, g/g-mole	166.21	166.21	166.21	
Target Catch, ng	2,200	1,800	1,300	1,767
ng to µg	2.20	1.80	1.30	1.77
Concentration, µg/dscm [a]	0.891	0.742	0.678	0.770
Emission Rate, µg/hr [b]	88,882	66,943	65,975	73,933
Emission Rate, lb/hr [c]	1.96E-04	1.48E-04	1.45E-04	1.63E-04
Pounds per Ton of Coke Pushed	1.72E-06	1.33E-06	1.33E-06	1.46E-06

	B-I-429-1	B-I-429-2	B-I-429-3	Average
Phenanthrene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	19,976	24,976	19,976	21,643
ng to µg	20.0	25.0	20.0	21.6
Concentration, µg/dscm [a]	8.09	10.29	10.42	9.60
Emission Rate, µg/hr [b]	807,050	928,865	1,013,784	916,566
Emission Rate, lb/hr [c]	1.78E-03	2.05E-03	2.24E-03	2.02E-03
Pounds per Ton of Coke Pushed	1.56E-05	1.85E-05	2.04E-05	1.82E-05
Anthrancene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	730	690	640	687
ng to µg	0.73	0.69	0.64	0.69
Concentration, µg/dscm [a]	0.296	0.284	0.334	0.305
Emission Rate, µg/hr [b]	29,493	25,661	32,480	29,211
Emission Rate, lb/hr [c]	6.50E-05	5.66E-05	7.16E-05	6.44E-05
Pounds per Ton of Coke Pushed	5.70E-07	5.11E-07	6.54E-07	5.78E-07
Fluoranthene				
Molecular Weight, g/g-mole	202.26	202.26	202.26	
Target Catch, ng	14,000	18,000	6,600	12,867
ng to µg	14.00	18.00	6.60	12.87
Concentration, µg/dscm [a]	5.67	7.42	3.44	5.51
Emission Rate, µg/hr [b]	565,614	669,426	334,951	523,330
Emission Rate, lb/hr [c]	1.25E-03	1.48E-03	7.38E-04	1.15E-03
Pounds per Ton of Coke Pushed	1.09E-05	1.33E-05	6.75E-06	1.03E-05
Pyrene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	2,200	2,500	380	1,693
ng to µg	2.20	2.50	0.38	1.69
Concentration, µg/dscm [a]	0.891	1.030	0.198	0.707
Emission Rate, µg/hr [b]	88,882	92,976	19,285	67,048
Emission Rate, lb/hr [c]	1.96E-04	2.05E-04	4.25E-05	1.48E-04
Pounds per Ton of Coke Pushed	1.72E-06	1.85E-06	3.88E-07	1.32E-06
Benzo (a) anthrancene				
Molecular Weight, g/g-mole	228.29	228.29	228.29	
Target Catch, ng	910	770	130	603
ng to µg	0.91	0.77	0.13	0.60
Concentration, µg/dscm [a]	0.3687	0.3173	0.0678	0.2513
Emission Rate, µg/hr [b]	36,765	28,637	6,598	24,000
Emission Rate, lb/hr [c]	8.11E-05	6.31E-05	1.45E-05	5.29E-05
Pounds per Ton of Coke Pushed	7.10E-07	5.70E-07	1.33E-07	4.71E-07

	B-I-429-1	B-I-429-2	B-I-429-3	Average
Chrysene				
Molecular Weight, g/g-mole	228.28	228.28	228.28	
Target Catch, ng	5,500	6,000	1,900	4,467
ng to µg	5.50	6.00	1.90	4.47
Concentration, µg/dscm [a]	2.23	2.47	0.99	1.90
Emission Rate, µg/hr [b]	222,206	223,142	96,425	180,591
Emission Rate, lb/hr [c]	4.90E-04	4.92E-04	2.13E-04	3.98E-04
Pounds per Ton of Coke Pushed	4.29E-06	4.44E-06	1.94E-06	3.56E-06
Benzo (b) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	7,500	8,200	2,900	6,200
ng to µg	7.50	8.20	2.90	6.20
Concentration, µg/dscm [a]	3.04	3.38	1.51	2.64
Emission Rate, µg/hr [b]	303,008	304,961	147,175	251,714
Emission Rate, lb/hr [c]	6.68E-04	6.72E-04	3.24E-04	5.55E-04
Pounds per Ton of Coke Pushed	5.85E-06	6.07E-06	2.96E-06	4.96E-06
Benzo (k) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	3,000	3,100	830	2,310
ng to µg	3.00	3.10	0.83	2.31
Concentration, µg/dscm [a]	1.22	1.28	0.433	0.975
Emission Rate, µg/hr [b]	121,203	115,290	42,123	92,872
Emission Rate, lb/hr [c]	2.67E-04	2.54E-04	9.29E-05	2.05E-04
Pounds per Ton of Coke Pushed	2.34E-06	2.29E-06	8.48E-07	1.83E-06
Benzo (e) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	2,000	1,900	390	1,430
ng to µg	2.00	1.90	0.39	1.43
Concentration, µg/dscm [a]	0.810	0.783	0.203	0.599
Emission Rate, µg/hr [b]	80,802	70,662	19,793	57,085
Emission Rate, lb/hr [c]	1.78E-04	1.56E-04	4.36E-05	1.26E-04
Pounds per Ton of Coke Pushed	1.56E-06	1.41E-06	3.99E-07	1.12E-06
Benzo (a) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	620	430	130	393
ng to µg	0.62	0.43	0.13	0.39
Concentration, µg/dscm [a]	0.251	0.177	0.068	0.165
Emission Rate, µg/hr [b]	25,049	15,992	6,598	15,879
Emission Rate, lb/hr [c]	5.52E-05	3.53E-05	1.45E-05	3.50E-05
Pounds per Ton of Coke Pushed	4.84E-07	3.18E-07	1.33E-07	3.12E-07

	B-I-429-1	B-I-429-2	B-I-429-3	Average
Perylene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	100.00	N D	ND	33.33
ng to µg	0.10	0.00	0.00	0.03
Concentration, µg/dscm [a]	0.0405	0.00	0.00	0.0135
Emission Rate, µg/hr [b]	4,040	0.00	0.00	1346.7
Emission Rate, lb/hr [c]	8.91E-06	0.00E+00	0.00E+00	2.97E-06
Pounds per Ton of Coke Pushed	7.80E-08	0.00E+00	0.00E+00	2.60E-08
Indeno (1,2,3-cd) pyrene				
Molecular Weight, g/g-mole	290.34	290.34	290.34	
Target Catch, ng	2,700	2,700	450	1,950
ng to µg	2.70	2.70	0.45	1.95
Concentration, µg/dscm [a]	1.09	1.11	0.235	0.814
Emission Rate, µg/hr [b]	109,083	100,414	22,838	77,445
Emission Rate, lb/hr [c]	2.40E-04	2.21E-04	5.03E-05	1.71E-04
Pounds per Ton of Coke Pushed	2.11E-06	2.00E-06	4.60E-07	1.52E-06
Dibenz (a,h) anthracene				
Molecular Weight, g/g-mole	278.33	278.33	278.33	
Target Catch, ng	1,100	1,100	240	813
ng to µg	1.10	1.10	0.240	0.813
Concentration, µg/dscm [a]	0.446	0.453	0.125	0.341
Emission Rate, µg/hr [b]	44,441	40,909	12,180	32,510
Emission Rate, lb/hr [c]	9.80E-05	9.02E-05	2.69E-05	7.17E-05
Pounds per Ton of Coke Pushed	8.58E-07	8.14E-07	2.45E-07	6.39E-07
Benzo (g,h,i) perylene				
Molecular Weight, g/g-mole	276.34	276.34	276.34	
Target Catch, ng	2,200	2,200	320	1,573
ng to µg	2.20	2.20	0.32	1.57
Concentration, µg/dscm [a]	0.891	0.907	0.167	0.655
Emission Rate, µg/hr [b]	88,882	81,819	16,240	62,314
Emission Rate, lb/hr [c]	1.96E-04	1.80E-04	3.58E-05	1.37E-04
Pounds per Ton of Coke Pushed	1.72E-06	1.63E-06	3.27E-07	1.22E-06

[a] Milligrams per dry standard cubic meter at 68° F (20° C) and 1 atm.

[b] Micrograms per hour.

[c] Pounds per hour.

ND Not Detectable - Results are below target analyte detection limit. Values are counted as zero (0) in averages.

{ } Estimate - Analyte results are below the quantitation limit and above the detection limit.

Baghouse Inlet

Trunc'd	CMPD	Quanterra Compound Name		Mol. Wt.	Method Blank	B-I-429-1	Blank Adjusted	B-I-429-2	Blank Adjusted	B-I-429-3	Blank Adjusted
Naphthal	cmpd1	Naphthalene	1	128.16	520	27,000	26,480	35,000	34,480	29,000	28,480
2-Methyl	cmpd2	2-Methylnaphthalene	2	142.2	14	7,900	7,886	6,800	6,786	3,000	2,986
Acenaph	cmpd3	Acenaphthylene	3	154.21	0	5,500	5,500	5,900	5,900	2,600	2,600
Acenaph	cmpd4	Acenaphthene	4	154.21	0	690	690	470	470	530	530
Fluorene	cmpd5	Fluorene	5	166.21	0	2,200	2,200	1,800	1,800	1,300	1,300
Phenant	cmpd6	Phenanthrene	6	178.22	24	20,000	19,976	25,000	24,976	20,000	19,976
Anthranc	cmpd7	Anthrancene	7	178.22	0	730	730	690	690	640	640
Fluorant	cmpd8	Fluoranthene	8	202.26	0	14,000	14,000	18,000	18,000	6,600	6,600
Pyrene	cmpd9	Pyrene	9	202.24	0	2,200	2,200	2,500	2,500	380	380
Benzo (a	cmpd10	Benzo (a) anthrancene	10	228.29	0	910	910	770	770	130	130
Chrysen	cmpd11	Chrysene	11	228.28	0	5,500	5,500	6,000	6,000	1,900	1,900
Benzo (b	cmpd12	Benzo (b) fluoranthene	12	252.32	0	7,500	7,500	8,200	8,200	2,900	2,900
Benzo (k	cmpd13	Benzo (k) fluoranthene	13	252.32	0	3,000	3,000	3,100	3,100	830	830
Benzo (e	cmpd14	Benzo (e) pyrene	14	252.3	0	2,000	2,000	1,900	1,900	390	390
Benzo (a	cmpd15	Benzo (a) pyrene	15	252.3	0	620	620	430	430	130	130
Perylene	cmpd16	Perylene	16	202.24	0	100	100	ND	N D	ND	ND
Indeno (	cmp17	Indeno (1,2,3-cd) pyrene	17	290.34	0	2,700	2,700	2,700	2,700	450	450
Dibenz (	cmpd18	Dibenz (a,h) anthracene	18	278.33	0	1,100	1,100	1,100	1,100	240	240
Benzo (g	cmpd19	Benzo (g,h,i) perylene	19	276.34	0	2,200	2,200	2,200	2,200	320	320

Summary of Stack Gas Parameters and Test Results
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Baghouse Outlet
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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	
	RUN DATE	***8/11/98***	***8/12/98***	***8/13/98***	***Average***
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	MEASURED DATA				
P_{static}	Stack Static Pressure, inches H_2O	-0.43	-0.43	-0.50	-0.45
y	Meter Box Correction Factor	0.979	0.979	0.979	0.979
P_{bar}	Barometric Pressure, inches Hg	29.70	29.80	29.80	29.77
V_m	Sample Volume, ft^3	132.109	188.898	190.238	170.415
$Dp^{1/2}$	Average Square Root Dp, (in. $H_2O)^{1/2}$	0.4220	0.4106	0.4171	0.4166
DH	Avg Meter Orifice Pressure, in. H_2O	0.5003	0.5859	0.6154	0.57
T_m	Average Meter Temperature, °F	77	77	80	78
T_s	Average Stack Temperature, °F	134	135	135	135
V_{lc}	Condensate Collected, ml	69.8	87.0	83.1	80.0
CO_2	Carbon Dioxide content, % by volume	0.45	0.5	0.5	0.5
O_2	Oxygen content, % by volume	20.50	20.0	20.0	20.2
N_2	Nitrogen content, % by volume	79.1	79.5	79.5	79.4
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	108	108	108	108.00
Q	Sample Run Duration, minutes	368	480	481	443
D_n	Nozzle Diameter, inches	0.220	0.235	0.235	0.230
Push	Tons of Coke Pushed	1,115	1,128	1,047	1,097
	Total Test Time, hours	9.35	10.10	9.15	9.53
	Tons of Coke per Hour	119.3	111.7	114.4	115.1
	CALCULATED DATA	**All Calculations are on Time Weighted Average Basis**			
A_n	Nozzle Area, ft^2	0.000264	0.000301	0.000301	0.000289
$V_{m(std)}$	Standard Meter Volume, ft^3	126.338	181.292	181.577	163.069
$V_{m(std)}$	Standard Meter Volume, m^3	3.577	5.134	5.142	4.618
Q_m	Average Sampling Rate, dscfm	0.343	0.378	0.377	0.366
P_s	Stack Pressure, inches Hg	29.67	29.77	29.76	29.73
B_{ws}	Moisture, % by volume	2.5	2.2	2.1	2.3
$B_{ws(sat)}$	Moisture (at saturation), % by volume	16.9	17.3	17.3	17.2
V_{wstd}	Standard Water Vapor Volume, ft^3	3.285	4.095	3.912	3.764
$1-B_{ws}$	Dry Mole Fraction	0.975	0.978	0.979	0.977
M_d	Molecular Weight (d.b.), lb/lb•mole	28.89	28.88	28.88	28.88
M_s	Molecular Weight (w.b.), lb/lb•mole	28.62	28.64	28.65	28.64
V_s	Stack Gas Velocity, ft/s	25.3	24.6	25.0	25.0
A	Stack Area, ft^2	63.6	63.6	63.6	63.62
Q_a	Stack Gas Volumetric flow, acfm	96,755	94,024	95,502	95,427
Q_s	Stack Gas Volumetric flow, dscfm	83,087	81,147	82,493	82,242
Q_s	Stack Gas Volumetric flow, dscmm	2,353	2,298	2,336	2,329
I	Isokinetic Sampling Ratio, %	99.6	98.3	96.7	98.2

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	
	RUN DATE	***8/11/98***	***8/12/98***	***8/13/98***	***Average***
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA				
	EMISSIONS DATA				
	Particulate Matter				
PM	Total Catch, g	0.0047	0.0044	0.0053	0.0048
$_{PM}$ @ 7% O	Concentration, gr/dscf @ 7% O2	0.0200	0.0058	0.0070	0.0109
C_{PM}	Concentration, g/dscm	1.31E-03	8.57E-04	1.03E-03	1.07E-03
$_{PM}$ @ 7% O	Concentration, g/dscm @ 7% O2	4.57E-02	1.32E-02	1.59E-02	2.49E-02
E_{PM}	Emission Rate, µg/hr	1.85E+08	1.18E+08	1.44E+08	1.49E+08
C_{PM}	Concentration, gr/dscf	5.74E-04	3.75E-04	4.50E-04	4.66E-04
C_{PM}	Concentration, lb/dscf	8.20E-08	5.35E-08	6.43E-08	6.66E-08
E_{PM}	Emission Rate, lb/hr	0.409	0.261	0.319	0.329
Push	Pounds per Ton of Coke Pushed	3.43E-03	2.33E-03	2.78E-03	2.85E-03
	Extractable Organic Matter				
EOM	Total Catch, g	0.0042	0.0046	0.0052	0.0047
$_{EOM}$ @ 7%	Concentration, gr/dscf @ 7% O2	0.0178	0.0060	0.0068	0.0102
C_{EOM}	Concentration, g/dscm	1.17E-03	8.96E-04	1.01E-03	1.03E-03
$_{EOM}$ @ 7%	Concentration, g/dscm @ 7% O2	4.08E-02	1.38E-02	1.56E-02	2.34E-02
E_{EOM}	Emission Rate, µg/hr	1.66E+08	1.24E+08	1.42E+08	1.44E+08
C_{EOM}	Concentration, gr/dscf	5.13E-04	3.92E-04	4.42E-04	4.49E-04
C_{EOM}	Concentration, lb/dscf	7.33E-08	5.59E-08	6.31E-08	6.41E-08
E_{EOM}	Emission Rate, lb/hr	0.365	0.272	0.312	0.317
Push	Pounds per Ton of Coke Pushed	3.06E-03	2.44E-03	2.73E-03	2.74E-03

Summary of Stack Gas Parameters and Test Results
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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***8/11/98***	***8/12/98***	***8/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	<u>Antimony</u>				
Sb	Target Catch, µg	0.0	0.0	0.0	0.0
CSb	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Sb @ 7% O	Concentration, µg/dscm @ 7% O2	0.0000	0.0000	0.0000	0.0000
ESb	Emission Rate, µg/hr	0.000	0.000	0.000	0.000
ESb	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Arsenic</u>				
As	Target Catch, µg	0.0	0.0	0.0	0.00
CAs	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Sb @ 7% O	Concentration, µg/dscm @ 7% O2	0.000	0.000	0.000	0.000
EAs	Emission Rate, µg/hr	0.000	0.000	0.000	0.000
EAs	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Barium</u>				
Ba	Target Catch, µg	0.4	0.7	0.0	0.37
CBa	Concentration, µg/dscm	1.12E-01	1.36E-01	0.00E+00	8.27E-02
Ba @ 7% O	Concentration, µg/dscm @ 7% O2	3.89E+00	2.11E+00	0.00E+00	2.00E+00
EBa	Emission Rate, µg/hr	15,784	18,799	0	11,528
EBa	Emission Rate, lb/hr	3.47E-05	4.14E-05	0.00E+00	2.54E-05
Push	Pounds per Ton of Coke Pushed	2.91E-07	3.70E-07	0.00E+00	2.21E-07

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA - Continued				
	<u>Beryllium</u>				
Be	Target Catch, µg	0.0	0.0	0.0	0.00
CBe	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Be @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
EBe	Emission Rate, µg/hr	0	0	0	0
EBe	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Cadmium</u>				
Cd	Target Catch, µg	0.1	0.0	0.0	0.0
CCd	Concentration, µg/dscm	2.80E-02	0.00E+00	0.00E+00	9.32E-03
Cd @ 7% O	Concentration, µg/dscm @ 7% O2	9.71E-01	0.00E+00	0.00E+00	3.24E-01
ECd	Emission Rate, µg/hr	3,946	0	0	1,315
ECd	Emission Rate, lb/hr	8.68E-06	0.00E+00	0.00E+00	2.89E-06
Push	Pounds per Ton of Coke Pushed	7.28E-08	0.00E+00	0.00E+00	2.43E-08
	<u>Chromium</u>				
Cr	Target Catch, µg	0.0	0.0	0.0	0.00
CCr	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
CCr @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
ECr	Emission Rate, µg/hr	0	0	0	0
ECr	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA - Continued				
	<u>Colbalt</u>				
Co	Target Catch, µg	0.0	0.0	0.0	0.00
CCo	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Co @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
ECo	Emission Rate, µg/hr	0	0	0	0
ECo	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Copper</u>				
Cu	Target Catch, µg	0.0	2.0	0.0	0.67
Ccu	Concentration, µg/dscm	0.00E+00	3.90E-01	0.00E+00	1.30E-01
Cu @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	6.02E+00	0.00E+00	2.01E+00
ECu	Emission Rate, µg/hr	0	53,713	0	17,904
ECu	Emission Rate, lb/hr	0.00E+00	1.18E-04	0.00E+00	3.94E-05
Push	Pounds per Ton of Coke Pushed	0.00E+00	1.06E-06	0.00E+00	3.53E-07
	<u>Lead</u>				
Pb	Target Catch, µg	0.0	0.0	0.0	0.0
CPb	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Pb @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
EPb	Emission Rate, µg/hr	0	0	0	0
EPb	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA - Continued				
	<u>Manganese</u>				
Mn	Target Catch, µg	0.0	4.0	0.0	1.33
CMn	Concentration, µg/dscm	0.00E+00	7.79E-01	0.00E+00	2.60E-01
Mn @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	1.20E+01	0.00E+00	4.01E+00
EMn	Emission Rate, µg/hr	0	107,425	0	35,808
EMn	Emission Rate, lb/hr	0.00E+00	2.36E-04	0.00E+00	7.88E-05
Push	Pounds per Ton of Coke Pushed	0.00E+00	2.12E-06	0.00E+00	7.05E-07
	<u>Mercury</u>				
Hg	Target Catch, µg	0.0	0.0	0.0	0.00
CHg	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Hg @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
EHg	Emission Rate, µg/hr	0	0	0	0
EHg	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Nickel</u>				
Ni	Target Catch, µg	0.0	0.9	0.0	0.30
CNi	Concentration, µg/dscm	0.00E+00	1.75E-01	0.00E+00	5.84E-02
CNi @ 7% O2	Concentration, µg/dscm @ 7% O2	0.00E+00	2.71E+00	0.00E+00	9.03E-01
ENi	Emission Rate, µg/hr	0	24,171	0	8,057
ENi	Emission Rate, lb/hr	0.00E+00	5.32E-05	0.00E+00	1.77E-05
Push	Pounds per Ton of Coke Pushed	0.00E+00	4.76E-07	0.00E+00	1.59E-07

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA - Continued				
	Phosphorus				
P	Target Catch, µg	0.0	0.0	0.0	0.00
CP	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
CP @ 7% O2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
EP	Emission Rate, µg/hr	0	0	0	0
EP	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	Selenium				
Se	Target Catch, µg	0.0	0.5	0.0	0.17
CSe	Concentration, µg/dscm	0.00E+00	9.74E-02	0.00E+00	3.25E-02
Se @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	1.50E+00	0.00E+00	5.01E-01
ESe	Emission Rate, µg/hr	0	13,428	0	4,476
ESe	Emission Rate, lb/hr	0.00E+00	2.95E-05	0.00E+00	9.85E-06
Push	Pounds per Ton of Coke Pushed	0.00E+00	2.65E-07	0.00E+00	8.82E-08
	Silver				
Ag	Target Catch, µg	0.0	2.1	0.0	0.70
Cag	Concentration, µg/dscm	0.00E+00	4.09E-01	0.00E+00	1.36E-01
Ag @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	6.32E+00	0.00E+00	2.11E+00
EAg	Emission Rate, µg/hr	0	56,398	0	18,799
EAg	Emission Rate, lb/hr	0.00E+00	1.24E-04	0.00E+00	4.14E-05
Push	Pounds per Ton of Coke Pushed	0.00E+00	1.11E-06	0.00E+00	3.70E-07

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	RUN NUMBER	***B-O-315-1***	***B-O-315-2***	***B-O-315-3***	***Average***
	RUN DATE	***08/11/98***	***08/12/98***	***08/13/98***	
	RUN TIME	***1037-1958***	***0929-1935***	***0840-1749***	
	EMISSIONS DATA - Continued				
	Thallium				
Tl	Target Catch, µg	0.0	0.0	0.0	0.00
CTl	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
CTl @ 7% O2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
ETl	Emission Rate, µg/hr	0	0	0	0
ETl	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coke Pushed	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	Zinc				
Zn	Target Catch, µg	0.0	0.8	0.0	0.27
CZn	Concentration, µg/dscm	0.00E+00	1.56E-01	0.00E+00	5.19E-02
Zn @ 7% O	Concentration, µg/dscm @ 7% O2	0.00E+00	2.41E+00	0.00E+00	8.02E-01
EZn	Emission Rate, µg/hr	0	21,485	0	7,162
EZn	Emission Rate, lb/hr	0.00E+00	4.73E-05	0.00E+00	1.58E-05
Push	Pounds per Ton of Coke Pushed	0.00E+00	4.23E-07	0.00E+00	1.41E-07

Method 315 Analytical Results (ug) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Baghouse Outlet

Metal	Target Catch, ug/sample						
	B-O-315-1	B-O-315-1	B-O-315-2	B-O-315-2	B-O-315-3	B-O-315-3	Filter Blank
		Corrected		Corrected		Corrected	
Antimony	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Arsenic	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Barium	1.2	0.4	1.5	0.7	0.8	0.0	0.8
Beryllium	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Cadmium	1.8	0.1	1.4	0.0	1.7	0.0	1.7
Chromium	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Colbalt	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Copper	0.0	0.0	2.0	2.0	0.0	0.0	0.0
Lead	0.5	0.0	0.5	0.0	0.5	0.0	0.7
Manganese	3.0	0.0	7.0	4.0	3.0	0.0	3.0
Mercury	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Nickel	0.0	0.0	0.9	0.9	0.0	0.0	0.0
Phosphorus	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Selenium	0.0	0.0	0.5	0.5	0.0	0.0	0.0
Silver	0.0	0.0	2.1	2.1	0.0	0.0	0.0
Thallium	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Zinc	8.3	0.0	11.7	0.8	7.8	0.0	10.9

Method 315 Analytical Results (g) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Baghouse Outlet

All Weights are listed in grams

Run No.	Filters		Acetone-FHR	MeCl2 -FHR	Acetone-BHR	MeCl2 -BHR	H2O Imp. Rinse	Corrected Total	
	PM	MCEM	PM	MCEM	MCEM	MCEM	MCEM	PM	MCEM
B-O-315-1	0.0011	0.0003	0.0061	0.0019	0.0006	0.0000	0.0026	**0.0047**	**0.0042**
B-O-315-2	0.0028	0.0006	0.0041	0.0009	0.0013	0.0002	0.0028	**0.0044**	**0.0046**
B-O-315-3	0.0004	0.000	0.0074	0.0026	0.0008	0.0026	0.0004	**0.0053**	**0.0052**
Blank Values	0.0011	0.0002	0.0014	0.0009	0.0000	0.0000	0.0001	**0.0025**	**0.0012**

Summary of Stack Gas Parameters and Test Results
Bethlehem Steel-Chesterton, Indiana
CARB Method 429 - PAH's
Baghouse Outlet

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	RUN NUMBER	**B-O-429-1**	**B-O-429-2**	**B-O-429-3**	
	RUN DATE	**8/11/98**	**8/12/98**	**8/13/98**	***Average***
	RUN TIME	**1037-1958**	**0928-1934**	**0840-1748**	
	MEASURED DATA				
γ	Meter Box Correction Factor	1.002	1.002	1.002	1.002
ΔH	Avg. Meter Orifice Pressure, in. H_2O	0.5292	0.6925	0.6581	0.6266
P_{bar}	Barometric Pressure, inches Hg	29.70	29.80	29.80	29.77
V_m	Sample Volume, ft^3	150.008	200.895	201.093	183.999
T_m	Average Meter Temperature, °F	79	80	83	81
P_{static}	Stack Static Pressure, inches H_2O	-0.65	-0.49	-0.50	-0.55
T_s	Average Stack Temperature, °F	132	137	136	135
V_{lc}	Condensate Collected, ml	93.8	118.0	95.8	102.533
CO_2	Carbon Dioxide content, % by volume	0.45	0.5	0.5	0.48
O_2	Oxygen content, % by volume	20.50	20.00	20.00	20.17
N_2	Nitrogen content, % by volume	79.05	79.50	79.50	79.35
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
$\Delta p^{1/2}$	Average Square Root Dp, (in. $H_2O)^{1/2}$	0.4351	0.4607	0.4357	0.4438
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	108	108	108	108
Θ	Sample Run Duration, minutes	367	478	480	442
D_n	Nozzle Diameter, inches	0.220	0.235	0.235	0.230
	Tons of Coke pushed	1,115	1,128	1,047	1,097
	Total Test Time, hours	9.35	10.10	9.13	9.53
	Tons of Coke per Hour	119.3	111.7	114.7	115.2
	CALCULATED DATA				
A_n	Nozzle Area, ft^2	0.000264	0.000301	0.000301	0.000289
$V_{m(std)}$	Standard Meter Volume, dscf	146.291	196.291	195.382	179.321
$V_{m(std)}$	Standard Meter Volume, dscm	4.142	5.558	5.533	5.078
B_{ws}	Moisture, % by volume	2.9	2.8	2.3	2.6
$B_{ws(sat)}$	Moisture (at saturation), % by volume	16.1	18.2	17.8	17.363
V_{wstd}	Standard Water Vapor Volume, ft^3	4.415	5.554	4.509	4.826
$1-B_{ws}$	Dry Mole Fraction	0.971	0.972	0.977	0.974
M_d	Molecular Weight (d.b.), lb/lb•mole	28.89	28.88	28.88	28.88
M_s	Molecular Weight (w.b.), lb/lb•mole	28.57	28.58	28.63	28.60
V_s	Stack Gas Velocity, ft/s	26.1	27.7	26.2	26.7
A	Stack Area, ft^2	63.6	63.6	63.6	63.62
Q_a	Stack Gas Volumetric flow, acfm	99,693	105,790	99,873	101,785
Q_s	Stack Gas Volumetric flow, dscfm	85,504	90,478	85,994	87,325
$Q_{s(cmm)}$	Stack Gas Volumetric flow, dscmm	2,421	2,562	2,435	2,473
I	Isokinetic Sampling Ratio, %	112.4	95.9	100.0	102.7

Baghouse Outlet

	B-O-429-1	B-O-429-2	B-O-429-3	Average
Naphthalene				
Molecular Weight, g/g-mole	128.16	128.16	128.16	
Target Catch, ng	7280.00	8780.00	9180.00	8413.33
ng to µg	7.28	8.78	9.18	8.41
Concentration, µg/dscm[a]	1.76	1.58	1.66	1.67
Emission Rate, µg/hr[b]	255,300	242,822	242,426	246,849
Emission Rate, lb/hr[c]	5.63E-04	5.35E-04	5.34E-04	5.44E-04
Pounds per Ton of Coke Pushed	4.72E-06	4.79E-06	4.66E-06	4.72E-06
2-Methylnaphthalene				
Molecular Weight, g/g-mole	142.20	142.20	142.20	
Target Catch, ng	1586.00	2486.00	1686.00	1919.33
ng to µg	1.59	2.49	1.69	1.92
Concentration, µg/dscm[a]	0.383	0.447	0.305	0.378
Emission Rate, µg/hr[b]	55,619	68,754	44,524	56,299
Emission Rate, lb/hr[c]	1.23E-04	1.52E-04	9.82E-05	1.24E-04
Pounds per Ton of Coke Pushed	1.03E-06	1.36E-06	8.56E-07	1.08E-06
Acenaphthylene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	300.00	280.00	340.00	306.67
ng to µg	0.300	0.280	0.340	0.307
Concentration, µg/dscm[a]	0.0724	0.0504	0.0615	0.0614
Emission Rate, µg/hr[b]	10520.61	7743.76	8978.73	9081.03
Emission Rate, lb/hr[c]	2.32E-05	1.71E-05	1.98E-05	2.00E-05
Pounds per Ton of Coke Pushed	1.94E-07	1.53E-07	1.73E-07	1.73E-07
Acenaphthene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	140.00	120.00	97.00	119.00
ng to µg	0.140	0.120	0.097	0.119
Concentration, µg/dscm[a]	0.0338	0.0216	0.0175	0.0243
Emission Rate, µg/hr[b]	4,910	3,319	2,562	3,597
Emission Rate, lb/hr[c]	1.08E-05	7.32E-06	5.65E-06	7.93E-06
Pounds per Ton of Coke Pushed	9.08E-08	6.55E-08	4.92E-08	6.85E-08
Fluorene				
Molecular Weight, g/g-mole	166.21	166.21	166.21	
Target Catch, ng	300.00	270.00	200.00	256.67
ng to µg	0.30	0.27	0.20	0.26
Concentration, µg/dscm[a]	0.0724	0.0486	0.0361	0.0524
Emission Rate, µg/hr[b]	10520.61	7467.20	5281.61	7756.47
Emission Rate, lb/hr[c]	2.32E-05	1.65E-05	1.16E-05	1.71E-05
Pounds per Ton of Coke Pushed	1.94E-07	1.47E-07	1.02E-07	1.48E-07

	B-O-429-1	B-O-429-2	B-O-429-3	Average
Phenanthrene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	1,176.00	946.00	606.00	909.33
ng to µg	1.18	0.95	0.61	0.91
Concentration, µg/dscm [a]	0.284	0.170	0.110	0.188
Emission Rate, µg/hr [b]	41240.79	26162.84	16003.27	27802.30
Emission Rate, lb/hr [c]	9.09E-05	5.77E-05	3.53E-05	6.13E-05
Pounds per Ton of Coke Pushed	7.62E-07	5.16E-07	3.08E-07	5.29E-07
Anthrancene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	84.00	38.00	20.00	47.33
ng to µg	0.08	0.04	0.02	0.05
Concentration, µg/dscm [a]	0.02028	0.00684	0.00361	0.01024
Emission Rate, µg/hr [b]	2,946	1,051	528	1,508
Emission Rate, lb/hr [c]	6.49E-06	2.32E-06	1.16E-06	3.33E-06
Pounds per Ton of Coke Pushed	5.45E-08	2.07E-08	1.02E-08	2.85E-08
Fluoranthene				
Molecular Weight, g/g-mole	202.26	202.26	202.26	
Target Catch, ng	480.00	240.00	150.00	290.00
ng to µg	0.48	0.24	0.15	0.29
Concentration, µg/dscm [a]	0.1159	0.0432	0.0271	0.0621
Emission Rate, µg/hr [b]	16,833	6,638	3,961	9,144
Emission Rate, lb/hr [c]	3.71E-05	1.46E-05	8.73E-06	2.02E-05
Pounds per Ton of Coke Pushed	3.11E-07	1.31E-07	7.62E-08	1.73E-07
Pyrene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	280.00	180.00	96.00	185.33
ng to µg	0.28	0.18	0.10	0.19
Concentration, µg/dscm [a]	0.0676	0.0324	0.0174	0.0391
Emission Rate, µg/hr [b]	9819.24	4978.13	2535.17	5777.51
Emission Rate, lb/hr [c]	2.16E-05	1.10E-05	5.59E-06	1.27E-05
Pounds per Ton of Coke Pushed	1.82E-07	9.83E-08	4.87E-08	1.10E-07
Benzo (a) anthrancene				
Molecular Weight, g/g-mole	228.29	228.29	228.29	
Target Catch, ng	55.00	43.00	23.00	40.33
ng to µg	0.06	0.04	0.02	0.04
Concentration, µg/dscm [a]	0.01328	0.00774	0.00416	0.00839
Emission Rate, µg/hr [b]	1,929	1,189	607	1,242
Emission Rate, lb/hr [c]	4.25E-06	2.62E-06	1.34E-06	2.74E-06
Pounds per Ton of Coke Pushed	3.57E-08	2.35E-08	1.17E-08	2.36E-08

	B-O-429-1	B-O-429-2	B-O-429-3	Average
Chrysene				
Molecular Weight, g/g-mole	228.28	228.28	228.28	
Target Catch, ng	110.00	140.00	63.00	104.33
ng to µg	0.11	0.14	0.06	0.10
Concentration, µg/dscm [a]	0.0266	0.0252	0.0114	0.0210
Emission Rate, µg/hr [b]	3,858	3,872	1,664	3,131
Emission Rate, lb/hr [c]	8.50E-06	8.54E-06	3.67E-06	6.90E-06
Pounds per Ton of Coke Pushed	7.13E-08	7.64E-08	3.20E-08	5.99E-08
Benzo (b) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	73.00	63.00	38.00	58.00
ng to µg	0.07	0.06	0.04	0.06
Concentration, µg/dscm [a]	0.01762	0.01133	0.00687	0.01194
Emission Rate, µg/hr [b]	2,560	1,742	1,004	1,769
Emission Rate, lb/hr [c]	5.64E-06	3.84E-06	2.21E-06	3.90E-06
Pounds per Ton of Coke Pushed	4.73E-08	3.44E-08	1.93E-08	3.37E-08
Benzo (k) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	68.00	32.00	25.00	41.67
ng to µg	0.07	0.03	0.03	0.04
Concentration, µg/dscm [a]	0.01642	0.00576	0.00452	0.00890
Emission Rate, µg/hr [b]	2,385	885	660	1,310
Emission Rate, lb/hr [c]	5.26E-06	1.95E-06	1.46E-06	2.89E-06
Pounds per Ton of Coke Pushed	4.41E-08	1.75E-08	1.27E-08	2.47E-08
Benzo (e) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	48.00	33.00	24.00	35.00
ng to µg	0.05	0.03	0.02	0.04
Concentration, µg/dscm [a]	0.01159	0.00594	0.00434	0.00729
Emission Rate, µg/hr [b]	1,683	913	634	1,077
Emission Rate, lb/hr [c]	3.71E-06	2.01E-06	1.40E-06	2.37E-06
Pounds per Ton of Coke Pushed	3.11E-08	1.80E-08	1.22E-08	2.04E-08
Benzo (a) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	48.00	27.00	20.00	31.67
ng to µg	0.05	0.03	0.02	0.03
Concentration, µg/dscm [a]	0.01159	0.00486	0.00361	0.00669
Emission Rate, µg/hr [b]	1,683	747	528	986
Emission Rate, lb/hr [c]	3.71E-06	1.65E-06	1.16E-06	2.17E-06
Pounds per Ton of Coke Pushed	3.11E-08	1.47E-08	1.02E-08	1.87E-08

	B-O-429-1	B-O-429-2	B-O-429-3	Average
Perylene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	11.00	13.00	ND	8.00
ng to µg	0.01	0.01	0.00	0.01
Concentration, µg/dscm [a]	0.00266	0.00234	0.00000	0.00166
Emission Rate, µg/hr [b]	386	360	0.00	248
Emission Rate, lb/hr [c]	8.50E-07	7.93E-07	0.00E+00	5.48E-07
Pounds per Ton of Coke Pushed	7.13E-09	7.10E-09	0.00E+00	4.74E-09
Indeno (1,2,3-cd) pyrene				
Molecular Weight, g/g-mole	290.34	290.34	290.34	
Target Catch, ng	63.00	30.00	25.00	39.33
ng to µg	0.06	0.03	0.03	0.04
Concentration, µg/dscm [a]	0.01521	0.00540	0.00452	0.00837
Emission Rate, µg/hr [b]	2,209	830	660	1,233
Emission Rate, lb/hr [c]	4.87E-06	1.83E-06	1.46E-06	2.72E-06
Pounds per Ton of Coke Pushed	4.08E-08	1.64E-08	1.27E-08	2.33E-08
Dibenz (a,h) anthracene				
Molecular Weight, g/g-mole	278.33	278.33	278.33	
Target Catch, ng	14.00	ND	ND	4.67
ng to µg	0.01	0.00	0.00	0.00
Concentration, µg/dscm [a]	0.00338	0.00000	0.00000	0.00113
Emission Rate, µg/hr [b]	491.0	0.00	0.00	163.65
Emission Rate, lb/hr [c]	1.08E-06	0.00E+00	0.00E+00	3.61E-07
Pounds per Ton of Coke Pushed	9.08E-09	0.00E+00	0.00E+00	3.03E-09
Benzo (g,h,i) perylene				
Molecular Weight, g/g-mole	276.34	276.34	276.34	
Target Catch, ng	140.00	45.00	64.00	83.00
ng to µg	0.14	0.05	0.06	0.08
Concentration, µg/dscm [a]	0.03380	0.00810	0.01157	0.01782
Emission Rate, µg/hr [b]	4,910	1,245	1,690	2,615
Emission Rate, lb/hr [c]	1.08E-05	2.74E-06	3.73E-06	5.76E-06
Pounds per Ton of Coke Pushed	9.08E-08	2.46E-08	3.25E-08	4.93E-08

[a] Milligrams per dry standard cubic meter at 68° F (20° C) and 1 atm.

[b] Micrograms per hour.

[c] Pounds per hour.

ND Not Detectable - Results are below target analyte detection limit. Values are counted as zero (0) in averages.

{ } Estimate - Analyte results are below the quantitation limit and above the detection limit.

Baghouse Outlet

Trunc'd Name	CMPD	Quanterra Compound Name		Mol. Wt.	Method Blank	B-O-429-1	Blank Adjusted	B-O-429-2	Blank Adjusted	B-O-429-3	Blank Adjusted
Naphthalene	cmpd1	Naphthalene	1	128.16	520	7,800	7,280	9,300	8,780	9,700	9,180
2-Methylnaphthalene	cmpd2	2-Methylnaphthalene	2	142.2	14	1,600	1,586	2,500	2,486	1,700	1,686
Acenaphthylene	cmpd3	Acenaphthylene	3	154.21	0	300	300	280	280	340	340
Acenaphthene	cmpd4	Acenaphthene	4	154.21	0	140	140	120	120	97	97
Fluorene	cmpd5	Fluorene	5	166.21	0	300	300	270	270	200	200
Phenanthrene	cmpd6	Phenanthrene	6	178.22	24	1,200	1,176	970	946	630	606
Anthrancene	cmpd7	Anthrancene	7	178.22	0	84	84	38	38	20	20
Fluoranthene	cmpd8	Fluoranthene	8	202.26	0	480	480	240	240	150	150
Pyrene	cmpd9	Pyrene	9	202.24	0	280	280	180	180	96	96
Benzo (a) anthrancene	cmpd10	Benzo (a) anthrancene	10	228.29	0	55	55	43	43	23	23
Chrysene	cmpd11	Chrysene	11	228.28	0	110	110	140	140	63	63
Benzo (b) fluoranthene	cmpd12	Benzo (b) fluoranthene	12	252.32	0	73	73	63	63	38	38
Benzo (k) fluoranthene	cmpd13	Benzo (k) fluoranthene	13	252.32	0	68	68	32	32	25	25
Benzo (e) pyrene	cmpd14	Benzo (e) pyrene	14	252.3	0	48	48	33	33	24	24
Benzo (a) pyrene	cmpd15	Benzo (a) pyrene	15	252.3	0	48	48	27	27	20	20
Perylene	cmpd16	Perylene	16	202.24	0	11	11	13	13	ND	ND
Indeno (1,2,3-cd) pyren	cmp17	Indeno (1,2,3-cd) pyrene	17	290.34	0	63	63	30	30	25	25
Dibenz (a,h) anthracen	cmpd18	Dibenz (a,h) anthracene	18	278.33	0	14	14	ND	ND	ND	ND
Benzo (g,h,i) perylene	cmpd19	Benzo (g,h,i) perylene	19	276.34	0	140	140	45	45	64	64

Summary of Stack Gas Parameters and Test Results
S511.000
Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
Underfire Stack
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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	
	RUN DATE	***8/14/98***	***8/14/98***	***8/15/98***	***Average***
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	0				
	MEASURED DATA				
P_{static}	Stack Static Pressure, inches H_2O	-0.80	-0.81	-0.87	-0.83
y	Meter Box Correction Factor	0.981	0.981	0.981	0.981
P_{bar}	Barometric Pressure, inches Hg	29.80	29.80	29.65	29.75
V_m	Sample Volume, ft^3	99.017	81.886	135.910	105.604
$Dp^{1/2}$	Average Square Root Dp, $(in.\ H_2O)^{1/2}$	0.2067	0.1728	0.2276	0.2024
DH	Avg Meter Orifice Pressure, in. H_2O	0.8592	0.5650	1.6520	1.03
T_m	Average Meter Temperature, °F	95	103	95	98
T_s	Average Stack Temperature, °F	444	451	438	444
V_{lc}	Condensate Collected, ml	402.8	334.2	534.2	423.7
CO_2	Carbon Dioxide content, % by volume	4.40	5.5	5.0	5.0
O_2	Oxygen content, % by volume	11.30	9.55	10.45	10.4
N_2	Nitrogen content, % by volume	84.3	85.0	84.6	84.6
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	177	177	177	177.00
Q	Sample Run Duration, minutes	180	180	185	182
D_n	Nozzle Diameter, inches	0.442	0.435	0.498	0.458
Charge	Tons of Coal Charged	647	491	550	563
	Total Test Time, hours	4.07	3.47	3.42	3.65
	Tons of Coal per Hour	159.0	141.5	160.8	153.8
	CALCULATED DATA				
A_n	Nozzle Area, ft^2	0.001065	0.001032	0.001353	0.001150
$V_{m(std)}$	Standard Meter Volume, ft^3	92.198	75.109	126.161	97.823
$V_{m(std)}$	Standard Meter Volume, m^3	2.611	2.127	3.572	2.770
Q_m	Average Sampling Rate, dscfm	0.512	0.417	0.682	0.537
P_s	Stack Pressure, inches Hg	29.74	29.74	29.59	29.69
B_{ws}	Moisture, % by volume	17.1	17.3	16.6	17.0
$B_{ws(sat)}$	Moisture (at saturation), % by volume	2829.7	3041.3	2671.5	2847.5
V_{wstd}	Standard Water Vapor Volume, ft^3	18.960	15.731	25.145	19.945
$1-B_{ws}$	Dry Mole Fraction	0.829	0.827	0.834	0.830
M_d	Molecular Weight (d.b.), lb/lb•mole	29.16	29.26	29.22	29.21
M_s	Molecular Weight (w.b.), lb/lb•mole	27.25	27.31	27.35	27.31
V_s	Stack Gas Velocity, ft/s	15.7	13.1	17.2	15.3
A	Stack Area, ft^2	170.9	170.9	170.9	170.87
Q_a	Stack Gas Volumetric flow, acfm	160,715	134,733	176,514	157,321
Q_s	Stack Gas Volumetric flow, dscfm	77,362	64,153	85,538	75,684
Q_s	Stack Gas Volumetric flow, dscmm	2,191	1,817	2,422	2,143
I	Isokinetic Sampling Ratio, %	106.2	107.7	100.7	104.9

Summary of Stack Gas Parameters and Test Results
S511.000
Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
Underfire Stack
Page 2 of 2

	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	
	RUN DATE	***8/14/98***	***8/14/98***	***8/15/98***	***Average***
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA				
	EMISSIONS DATA				
	Particulate Matter				
PM	Total Catch, g	0.2036	0.2176	0.3258	0.2490
$_{PM}$ @ 7% O	Concentration, gr/dscf @ 7% O2	0.0493	0.0548	0.0530	0.0524
C_{PM}	Concentration, g/dscm	0.0780	0.1023	0.0912	0.0905
$_{PM}$ @ 7% O	Concentration, g/dscm @ 7% O2	1.13E-01	1.25E-01	1.21E-01	1.20E-01
E_{PM}	Emission Rate, µg/hr	1.03E+10	1.12E+10	1.33E+10	1.16E+10
C_{PM}	Concentration, gr/dscf	3.41E-02	4.47E-02	3.99E-02	3.95E-02
C_{PM}	Concentration, lb/dscf	4.87E-06	6.39E-06	5.69E-06	5.65E-06
E_{PM}	Emission Rate, lb/hr	22.6	24.6	29.2	25.5
Push	Pounds per Ton of Coal Charged	1.42E-01	1.74E-01	1.82E-01	1.66E-01
	Extractable Organic Matter				
EOM	Total Catch, g	0.0408	0.0323	0.1379	0.0703
$_{EOM}$ @ 7%	Concentration, gr/dscf @ 7% O2	0.0099	0.0081	0.0224	0.0135
C_{EOM}	Concentration, g/dscm	0.0156	0.0152	0.0386	0.0231
$_{EOM}$ @ 7%	Concentration, g/dscm @ 7% O2	2.26E-02	1.86E-02	5.13E-02	3.09E-02
E_{EOM}	Emission Rate, µg/hr	2.05E+09	1.66E+09	5.61E+09	3.11E+09
C_{EOM}	Concentration, gr/dscf	6.83E-03	6.64E-03	1.69E-02	1.01E-02
C_{EOM}	Concentration, lb/dscf	9.76E-07	9.48E-07	2.41E-06	1.44E-06
E_{EOM}	Emission Rate, lb/hr	4.53	3.65	12.37	6.85
Push	Pounds per Ton of Coal Charged	2.85E-02	2.58E-02	7.69E-02	4.37E-02

Summary of Stack Gas Parameters and Test Results
S511.000
Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
Underfire Stack
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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***8/14/98***	***8/14/98***	***8/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	<u>Antimony</u>				
Sb	Target Catch, µg	0.0	0.0	0.0	0.0
C_{Sb}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Sb} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.0000	0.0000	0.0000	0.0000
E_{Sb}	Emission Rate, µg/hr	0.000	0.000	0.000	0.000
E_{Sb}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Arsenic</u>				
As	Target Catch, µg	4.8	4.0	5.8	4.87
C_{As}	Concentration, µg/dscm	2.19E-03	2.20E-03	2.39E-03	2.26E-03
C_{Sb} @ 7% O_2	Concentration, µg/dscm @ 7% O2	3.17E-03	2.70E-03	3.19E-03	3.02E-03
E_{As}	Emission Rate, µg/hr	630,905	435,987	842,910	636,601
E_{As}	Emission Rate, lb/hr	6.34E-07	5.28E-07	7.66E-07	6.42E-07
Push	Pounds per Ton of Coal Charged	3.99E-09	3.73E-09	4.76E-09	4.16E-09
	<u>Barium</u>				
Ba	Target Catch, µg	0.4	1.8	0.5	0.90
C_{Ba}	Concentration, µg/dscm	1.83E-04	9.91E-04	2.06E-04	4.60E-04
C_{Ba} @ 7% O_2	Concentration, µg/dscm @ 7% O2	2.64E-04	1.21E-03	2.75E-04	5.84E-04
E_{Ba}	Emission Rate, µg/hr	52,575	196,194	72,665	107,145
E_{Ba}	Emission Rate, lb/hr	5.28E-08	2.38E-07	6.60E-08	1.19E-07
Push	Pounds per Ton of Coal Charged	3.32E-10	1.68E-09	4.10E-10	8.07E-10

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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***08/14/98***	***08/14/98***	***08/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA - Continued				
	Beryllium				
Be	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Be}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Be} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Be}	Emission Rate, µg/hr	0	0	0	0
E_{Be}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	Cadmium				
Cd	Target Catch, µg	0.2	0.0	0.0	0.1
C_{Cd}	Concentration, µg/dscm	9.13E-05	0.00E+00	0.00E+00	3.04E-05
C_{Cd} @ 7% O_2	Concentration, µg/dscm @ 7% O2	1.32E-04	0.00E+00	0.00E+00	4.41E-05
E_{Cd}	Emission Rate, µg/hr	26,288	0	0	8,763
E_{Cd}	Emission Rate, lb/hr	2.64E-08	0.00E+00	0.00E+00	8.80E-09
Push	Pounds per Ton of Coal Charged	1.66E-10	0.00E+00	0.00E+00	5.54E-11
	Chromium				
Cr	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Cr}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Cr} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Cr}	Emission Rate, µg/hr	0	0	0	0
E_{Cr}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00

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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***08/14/98***	***08/14/98***	***08/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA - Continued				
	<u>Colbalt</u>				
Co	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Co}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Co} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Co}	Emission Rate, µg/hr	0	0	0	0
E_{Co}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Copper</u>				
Cu	Target Catch, µg	0.0	3.0	2.0	1.67
C_{Cu}	Concentration, µg/dscm	0.00E+00	1.65E-03	8.26E-04	8.26E-04
C_{Cu} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	2.02E-03	1.10E-03	1.04E-03
E_{Cu}	Emission Rate, µg/hr	0	326,990	290,659	205,883
E_{Cu}	Emission Rate, lb/hr	0.00E+00	3.96E-07	2.64E-07	2.20E-07
Push	Pounds per Ton of Coal Charged	0.00E+00	2.80E-09	1.64E-09	1.48E-09
	<u>Lead</u>				
Pb	Target Catch, µg	3.4	2.9	2.0	2.8
C_{Pb}	Concentration, µg/dscm	1.55E-03	1.60E-03	8.26E-04	1.32E-03
C_{Pb} @ 7% O_2	Concentration, µg/dscm @ 7% O2	2.25E-03	1.96E-03	1.10E-03	1.77E-03
E_{Pb}	Emission Rate, µg/hr	446,891	316,091	290,659	351,213
E_{Pb}	Emission Rate, lb/hr	4.49E-07	3.83E-07	2.64E-07	3.65E-07
Push	Pounds per Ton of Coal Charged	2.82E-09	2.71E-09	1.64E-09	2.39E-09

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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***08/14/98***	***08/14/98***	***08/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA - Continued				
	<u>Manganese</u>				
Mn	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Mn}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Mn} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Mn}	Emission Rate, µg/hr	0	0	0	0
E_{Mn}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Mercury</u>				
Hg	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Hg}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Hg} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Hg}	Emission Rate, µg/hr	0	0	0	0
E_{Hg}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Nickel</u>				
Ni	Target Catch, µg	0.0	0.0	0.0	0.00
C_{Ni}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Ni} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Ni}	Emission Rate, µg/hr	0	0	0	0
E_{Ni}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00

Summary of Stack Gas Parameters and Test Results
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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***08/14/98***	***08/14/98***	***08/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA - Continued				
	<u>Phosphorus</u>				
P	Target Catch, µg	0.0	0.0	0.0	0.00
C_P	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_P @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_P	Emission Rate, µg/hr	0	0	0	0
E_P	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00
	<u>Selenium</u>				
Se	Target Catch, µg	2.3	2.0	1.0	1.77
C_{Se}	Concentration, µg/dscm	1.05E-03	1.10E-03	4.13E-04	8.55E-04
C_{Se} @ 7% O_2	Concentration, µg/dscm @ 7% O2	1.52E-03	1.35E-03	5.49E-04	1.14E-03
E_{Se}	Emission Rate, µg/hr	302,309	217,994	145,329	221,877
E_{Se}	Emission Rate, lb/hr	3.04E-07	2.64E-07	1.32E-07	2.33E-07
Push	Pounds per Ton of Coal Charged	1.91E-09	1.87E-09	8.21E-10	1.53E-09
	<u>Silver</u>				
Ag	Target Catch, µg	0.0	0.0	0.0	0.00
C_{ag}	Concentration, µg/dscm	0.00E+00	0.00E+00	0.00E+00	0.00E+00
C_{Ag} @ 7% O_2	Concentration, µg/dscm @ 7% O2	0.00E+00	0.00E+00	0.00E+00	0.00E+00
E_{Ag}	Emission Rate, µg/hr	0	0	0	0
E_{Ag}	Emission Rate, lb/hr	0.00E+00	0.00E+00	0.00E+00	0.00E+00
Push	Pounds per Ton of Coal Charged	0.00E+00	0.00E+00	0.00E+00	0.00E+00

Summary of Stack Gas Parameters and Test Results
S511.000
Bethlehem Steel - Chesterton, Indiana
US EPA Test Method 315 - EOM/Particulate Matter
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	RUN NUMBER	***B-U-315-1***	***B-U-315-2***	***B-U-315-3***	***Average***
	RUN DATE	***08/14/98***	***08/14/98***	***08/15/98***	
	RUN TIME	***0921-1325***	***1441-1809***	***0838-1203***	
	EMISSIONS DATA - Continued				
	<u>Thallium</u>				
Tl	Target Catch, µg	0.9	0.0	1.0	0.63
C_{Tl}	Concentration, µg/dscm	4.11E-04	0.00E+00	4.13E-04	2.75E-04
C_{Tl} @ 7% O_2	Concentration, µg/dscm @ 7% O2	5.95E-04	0.00E+00	5.49E-04	3.81E-04
E_{Tl}	Emission Rate, µg/hr	118,295	0	145,329	87,875
E_{Tl}	Emission Rate, lb/hr	1.19E-07	0.00E+00	1.32E-07	8.36E-08
Push	Pounds per Ton of Coal Charged	7.47E-10	0.00E+00	8.21E-10	5.23E-10
	<u>Zinc</u>				
Zn	Target Catch, µg	1.8	0.9	0.0	0.90
C_{Zn}	Concentration, µg/dscm	8.22E-04	4.95E-04	0.00E+00	4.39E-04
C_{Zn} @ 7% O_2	Concentration, µg/dscm @ 7% O2	1.19E-03	6.07E-04	0.00E+00	5.99E-04
E_{Zn}	Emission Rate, µg/hr	236,589	98,097	0	111,562
E_{Zn}	Emission Rate, lb/hr	2.38E-07	1.19E-07	0.00E+00	1.19E-07
Push	Pounds per Ton of Coal Charged	1.49E-09	8.40E-10	0.00E+00	7.78E-10

Method 315 Analytical Results (ug) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Underfire Stack

Metal	Target Catch, ug/sample						
	B-U-315-1	B-U-315-1	B-U-315-2	B-U-315-2	B-U-315-3	B-U-315-3	Filter Blank
		Corrected		Corrected		Corrected	
Antimony	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Arsenic	4.8	4.8	4.0	4.0	5.8	5.8	0.0
Barium	1.2	0.4	2.6	1.8	1.3	0.5	0.8
Beryllium	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Cadmium	1.9	0.2	1.6	0.0	1.1	0.0	1.7
Chromium	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Colbalt	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Copper	0.0	0.0	3.0	3.0	2.0	2.0	0.0
Lead	4.1	3.4	3.6	2.9	2.7	2.0	0.7
Manganese	3.0	0.0	3.0	0.0	1.0	0.0	3.0
Mercury	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Nickel	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Phosphorus	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Selenium	2.3	2.3	2.0	2.0	1.0	1.0	0.0
Silver	0.0	0.0	0.0	0.0	0.0	0.0	0.0
Thallium	0.9	0.9	0.0	0.0	1.0	1.0	0.0
Zinc	12.7	1.8	11.8	0.9	10.1	0.0	10.9

Method 315 Analytical Results (g) and Blank Corrections
Bethlehem Steel - Chesterton, Indiana
Underfire Stack

All Weights are listed in grams

Run No.	Filters		Acetone-FHR	MeCl2 -FHR	Acetone-BHR	MeCl2 -BHR	H2O Imp. Rinse	Corrected Total	
	PM	MCEM	PM	MCEM	MCEM	MCEM	MCEM	PM	MCEM
B-U-315-1	0.0792	0.0003	0.1324	0.0404	0.0001	0.0000	0.0003	**0.2036**	**0.0408**
B-U-315-2	0.0989	0.0001	0.1267	0.0314	0.0005	0.0004	0.0002	**0.2176**	**0.0323**
B-U-315-3	0.0722	0.000	0.2616	0.1373	0.0005	0.0001	0.0003	**0.3258**	**0.1379**
Blank Values	0.0058	0.0000	0.0022	0.0003	0.0000	0.0000	0.0000	**0.0080**	**0.0003**

Summary of Stack Gas Parameters and Test Results
Bethlehem Steel-Chesterton, Indiana
CARB Method 429 - PAH's
Underfire Stack

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	RUN NUMBER	**B-U-429-1**	**B-U-429-2**	**B-U-429-3**	
	RUN DATE	**8/14/98**	**8/14/98**	**8/15/98**	***Average***
	RUN TIME	**0921-1325**	**1442-1809**	**0838-1208**	
	MEASURED DATA				
γ	Meter Box Correction Factor	1.004	1.004	1.004	1.004
ΔH	Avg. Meter Orifice Pressure, in. H_2O	0.86	1.125	1.25	1.08
P_{bar}	Barometric Pressure, inches Hg	29.80	29.80	29.65	29.75
V_m	Sample Volume, ft^3	96.703	107.900	119.731	108.111
T_m	Average Meter Temperature, °F	115	109	115	113
P_{static}	Stack Static Pressure, inches H_2O	-0.83	-0.83	-0.85	-0.84
T_s	Average Stack Temperature, °F	453	448	440	447
V_{lc}	Condensate Collected, ml	396.2	568.5	382.8	449.167
CO_2	Carbon Dioxide content, % by volume	4.4	5.5	5.0	4.97
O_2	Oxygen content, % by volume	11.30	9.55	10.45	10.43
N_2	Nitrogen content, % by volume	84.30	84.95	84.55	84.60
C_p	Pitot Tube Coefficient	0.84	0.84	0.84	0.84
$\Delta p^{1/2}$	Average Square Root Dp, (in. $H_2O)^{1/2}$	0.2224	0.2572	0.2220	0.2339
	Circular Stack? 1=Y,0=N:	1	1	1	
As	Diameter or Dimensions, inches:	177	177	177	177
Θ	Sample Run Duration, minutes	180	180	180	180
D_n	Nozzle Diameter, inches	0.442	0.434	0.475	0.450
Charge	Tons of Coal Charged	647	491	550	563
	Total Test Time, hours	4.07	3.45	3.50	3.67
	Tons of Coal per Hour	159.0	142.3	157.1	152.8
	CALCULATED DATA				
A_n	Nozzle Area, ft^2	0.001066	0.001027	0.001231	0.001108
$V_{m(std)}$	Standard Meter Volume, dscf	88.949	100.360	109.683	99.664
$V_{m(std)}$	Standard Meter Volume, dscm	2.519	2.842	3.106	2.822
B_{ws}	Moisture, % by volume	17.3	21.1	14.1	17.5
$B_{ws(sat)}$	Moisture (at saturation), % by volume	3104.1	2949.4	2728.1	2927.198
V_{wstd}	Standard Water Vapor Volume, ft^3	18.649	26.759	18.018	21.142
$1\text{-}B_{ws}$	Dry Mole Fraction	0.827	0.789	0.859	0.825
M_d	Molecular Weight (d.b.), lb/lb•mole	29.16	29.26	29.22	29.21
M_s	Molecular Weight (w.b.), lb/lb•mole	27.22	26.89	27.64	27.25
V_s	Stack Gas Velocity, ft/s	17.0	19.7	16.7	17.8
A	Stack Area, ft^2	170.9	170.9	170.9	170.87
Q_a	Stack Gas Volumetric flow, acfm	173,886	201,774	171,478	182,379
Q_s	Stack Gas Volumetric flow, dscfm	82,595	92,035	85,412	86,681
$Q_{s(cmm)}$	Stack Gas Volumetric flow, dscmm	2,339	2,606	2,419	2,455
I	Isokinetic Sampling Ratio, %	96.0	100.8	99.1	98.6

Underfire Stack

	B-U-429-1	B-U-429-2	B-U-429-3	Average
Naphthalene				
Molecular Weight, g/g-mole	128.16	128.16	128.16	
Target Catch, ng	10,480	30,480	179,480	73,480
ng to µg	10.48	30.48	179.48	73.48
Concentration, µg/dscm [a]	4.16	10.73	57.79	24.22
Emission Rate, µg/hr [b]	583,882	1,677,094	8,385,796	3,548,924
Emission Rate, lb/hr [c]	1.29E-03	3.70E-03	1.85E-02	7.82E-03
Pounds per Ton of Coal Charged	8.10E-06	2.60E-05	1.18E-04	5.06E-05
2-Methylnaphthalene				
Molecular Weight, g/g-mole	142.20	142.20	142.20	
Target Catch, ng	5,786	1,986	8,186	5,319
ng to µg	5.79	1.99	8.19	5.32
Concentration, µg/dscm [a]	2.30	0.699	2.64	1.88
Emission Rate, µg/hr [b]	322,361	109,275	382,472	271,369
Emission Rate, lb/hr [c]	7.11E-04	2.41E-04	8.43E-04	5.98E-04
Pounds per Ton of Coal Charged	4.47E-06	1.69E-06	5.37E-06	3.84E-06
Acenaphthylene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	950	290	2,600	1,280
ng to µg	0.95	0.29	2.60	1.28
Concentration, µg/dscm [a]	0.377	0.102	0.837	0.439
Emission Rate, µg/hr [b]	52,928	15,957	121,479	63,455
Emission Rate, lb/hr [c]	1.17E-04	3.52E-05	2.68E-04	1.40E-04
Pounds per Ton of Coal Charged	7.34E-07	2.47E-07	1.70E-06	8.95E-07
Acenaphthene				
Molecular Weight, g/g-mole	154.21	154.21	154.21	
Target Catch, ng	190.00	110.00	450.00	250.00
ng to µg	0.19	0.11	0.45	0.25
Concentration, µg/dscm [a]	0.0754	0.0387	0.1449	0.0863
Emission Rate, µg/hr [b]	10,586	6,053	21,025	12,554
Emission Rate, lb/hr [c]	2.33E-05	1.33E-05	4.64E-05	2.77E-05
Pounds per Ton of Coal Charged	1.47E-07	9.38E-08	2.95E-07	1.79E-07
Fluorene				
Molecular Weight, g/g-mole	166.21	166.21	166.21	
Target Catch, ng	810	230	1,300	780
ng to µg	0.81	0.23	1.30	0.78
Concentration, µg/dscm [a]	0.3216	0.0809	0.4186	0.2737
Emission Rate, µg/hr [b]	45,128	12,655	60,740	39,508
Emission Rate, lb/hr [c]	9.95E-05	2.79E-05	1.34E-04	8.71E-05
Pounds per Ton of Coal Charged	6.26E-07	1.96E-07	8.52E-07	5.58E-07

	B-U-429-1	B-U-429-2	B-U-429-3	Average
Phenanthrene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	1,576	706	1,676	1,319
ng to µg	1.58	0.71	1.68	1.32
Concentration, µg/dscm [a]	0.626	0.248	0.540	0.471
Emission Rate, µg/hr [b]	87,805	38,846	78,307	68,320
Emission Rate, lb/hr [c]	1.94E-04	8.56E-05	1.73E-04	1.51E-04
Pounds per Ton of Coal Charged	1.22E-06	6.02E-07	1.10E-06	9.73E-07
Anthrancene				
Molecular Weight, g/g-mole	178.22	178.22	178.22	
Target Catch, ng	51.00	20.00	86.00	52.33
ng to µg	0.05	0.02	0.09	0.05
Concentration, µg/dscm [a]	0.02025	0.00704	0.02769	0.01833
Emission Rate, µg/hr [b]	2,841	1,100	4,018	2,653
Emission Rate, lb/hr [c]	6.26E-06	2.43E-06	8.86E-06	5.85E-06
Pounds per Ton of Coal Charged	3.94E-08	1.70E-08	5.64E-08	3.76E-08
Fluoranthene				
Molecular Weight, g/g-mole	202.26	202.26	202.26	
Target Catch, ng	430.00	220.00	620.00	423.33
ng to µg	0.43	0.22	0.62	0.42
Concentration, µg/dscm [a]	0.1707	0.0774	0.1996	0.1493
Emission Rate, µg/hr [b]	23,957	12,105	28,968	21,677
Emission Rate, lb/hr [c]	5.28E-05	2.67E-05	6.39E-05	4.78E-05
Pounds per Ton of Coal Charged	3.32E-07	1.88E-07	4.06E-07	3.09E-07
Pyrene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	170.00	80.00	220.00	156.67
ng to µg	0.17	0.08	0.22	0.16
Concentration, µg/dscm [a]	0.0675	0.0282	0.0708	0.0555
Emission Rate, µg/hr [b]	9,471	4,402	10,279	8,051
Emission Rate, lb/hr [c]	2.09E-05	9.70E-06	2.27E-05	1.77E-05
Pounds per Ton of Coal Charged	1.31E-07	6.82E-08	1.44E-07	1.15E-07
Benzo (a) anthrancene				
Molecular Weight, g/g-mole	228.29	228.29	228.29	
Target Catch, ng	45.00	25.00	72.00	47.33
ng to µg	0.05	0.03	0.07	0.05
Concentration, µg/dscm [a]	0.01787	0.00880	0.02318	0.01661
Emission Rate, µg/hr [b]	2,507	1,376	3,364	2,416
Emission Rate, lb/hr [c]	5.53E-06	3.03E-06	7.42E-06	5.33E-06
Pounds per Ton of Coal Charged	3.48E-08	2.13E-08	4.72E-08	3.44E-08

	B-U-429-1	B-U-429-2	B-U-429-3	Average
Chrysene				
Molecular Weight, g/g-mole	228.28	228.28	228.28	
Target Catch, ng	120.00	59.00	160.00	113.00
ng to µg	0.12	0.06	0.16	0.11
Concentration, µg/dscm [a]	0.0476	0.0208	0.0515	0.0400
Emission Rate, µg/hr [b]	6,686	3,246	7,476	5,803
Emission Rate, lb/hr [c]	1.47E-05	7.16E-06	1.65E-05	1.28E-05
Pounds per Ton of Coal Charged	9.27E-08	5.03E-08	1.05E-07	8.26E-08
Benzo (b) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	76.00	45.00	64.00	61.67
ng to µg	0.08	0.05	0.06	0.06
Concentration, µg/dscm [a]	0.0302	0.0158	0.0206	0.0222
Emission Rate, µg/hr [b]	4,234	2,476	2,990	3,234
Emission Rate, lb/hr [c]	9.33E-06	5.46E-06	6.59E-06	7.13E-06
Pounds per Ton of Coal Charged	5.87E-08	3.84E-08	4.20E-08	4.63E-08
Benzo (k) fluoranthene				
Molecular Weight, g/g-mole	252.32	252.32	252.32	
Target Catch, ng	50.00	31.00	39.00	40.00
ng to µg	0.05	0.03	0.04	0.04
Concentration, µg/dscm [a]	0.0199	0.0109	0.0126	0.0144
Emission Rate, µg/hr [b]	2,786	1,706	1,822	2,105
Emission Rate, lb/hr [c]	6.14E-06	3.76E-06	4.02E-06	4.64E-06
Pounds per Ton of Coal Charged	3.86E-08	2.64E-08	2.56E-08	3.02E-08
Benzo (e) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	42.00	32.00	34.00	36.00
ng to µg	0.04	0.03	0.03	0.04
Concentration, µg/dscm [a]	0.0167	0.0113	0.0109	0.0130
Emission Rate, µg/hr [b]	2,340	1,761	1,589	1,896
Emission Rate, lb/hr [c]	5.16E-06	3.88E-06	3.50E-06	4.18E-06
Pounds per Ton of Coal Charged	3.25E-08	2.73E-08	2.23E-08	2.73E-08
Benzo (a) pyrene				
Molecular Weight, g/g-mole	252.30	252.30	252.30	
Target Catch, ng	47.00	24.00	32.00	34.33
ng to µg	0.05	0.02	0.03	0.03
Concentration, µg/dscm [a]	0.0187	0.0084	0.0103	0.0125
Emission Rate, µg/hr [b]	2,619	1,321	1,495	1,811
Emission Rate, lb/hr [c]	5.77E-06	2.91E-06	3.30E-06	3.99E-06
Pounds per Ton of Coal Charged	3.63E-08	2.05E-08	2.10E-08	2.59E-08

	B-U-429-1	B-U-429-2	B-U-429-3	Average
Perylene				
Molecular Weight, g/g-mole	202.24	202.24	202.24	
Target Catch, ng	12.00	ND	ND	4.00
ng to µg	0.01	0.00	0.00	0.00
Concentration, µg/dscm [a]	0.00	0.00	0.00	0.00
Emission Rate, µg/hr [b]	669	0.00	0.00	223
Emission Rate, lb/hr [c]	1.47E-06	0.00E+00	0.00E+00	4.91E-07
Pounds per Ton of Coal Charged	9.27E-09	0.00E+00	0.00E+00	3.09E-09
Indeno (1,2,3-cd) pyrene				
Molecular Weight, g/g-mole	290.34	290.34	290.34	
Target Catch, ng	44.00	32.00	35.00	37.00
ng to µg	0.04	0.03	0.04	0.04
Concentration, µg/dscm [a]	0.0175	0.0113	0.0113	0.0133
Emission Rate, µg/hr [b]	2,451	1,761	1,635	1,949
Emission Rate, lb/hr [c]	5.40E-06	3.88E-06	3.61E-06	4.30E-06
Pounds per Ton of Coal Charged	3.40E-08	2.73E-08	2.29E-08	2.81E-08
Dibenz (a,h) anthracene				
Molecular Weight, g/g-mole	278.33	278.33	278.33	
Target Catch, ng	12.00	ND	ND	4.00
ng to µg	0.01	0.00	0.00	0.00
Concentration, µg/dscm [a]	0.00476	0.00000	0.00000	0.00159
Emission Rate, µg/hr [b]	669	0	0	223
Emission Rate, lb/hr [c]	1.47E-06	0.00E+00	0.00E+00	4.91E-07
Pounds per Ton of Coal Charged	9.27E-09	0.00E+00	0.00E+00	3.09E-09
Benzo (g,h,i) perylene				
Molecular Weight, g/g-mole	276.34	276.34	276.34	
Target Catch, ng	81.00	78.00	63.00	74.00
ng to µg	0.08	0.08	0.06	0.07
Concentration, µg/dscm [a]	0.0322	0.0274	0.0203	0.0266
Emission Rate, µg/hr [b]	4,513	4,292	2,944	3,916
Emission Rate, lb/hr [c]	9.95E-06	9.46E-06	6.49E-06	8.63E-06
Pounds per Ton of Coal Charged	6.26E-08	6.65E-08	4.13E-08	5.68E-08

[a] Milligrams per dry standard cubic meter at 68° F (20° C) and 1 atm.

[b] Micrograms per hour.

[c] Pounds per hour.

ND Not Detectable - Results are below target analyte detection limit. Values are counted as zero (0) in averages.

{ } Estimate - Analyte results are below the quantitation limit and above the detection limit.

Underfire Stack

Trunc'd	CMPD	Quanterra Compound Name		Mol. Wt.	Method Blank	B-U-429-1	Blank Adjusted	B-U-429-2	Blank Adjusted	B-U-429-3	Blank Adjusted
Naphthal	cmpd1	Naphthalene	1	128.16	520	11,000	10,480	31,000	30,480	180,000	179,480
2-Methyl	cmpd2	2-Methylnaphthalene	2	142.2	14	5,800	5,786	2,000	1,986	8,200	8,186
Acenaph	cmpd3	Acenaphthylene	3	154.21	0	950	950	290	290	2,600	2,600
Acenaph	cmpd4	Acenaphthene	4	154.21	0	190	190	110	110	450	450
Fluorene	cmpd5	Fluorene	5	166.21	0	810	810	230	230	1,300	1,300
Phenant	cmpd6	Phenanthrene	6	178.22	24	1,600	1,576	730	706	1,700	1,676
Anthranc	cmpd7	Anthrancene	7	178.22	0	51	51	20	20	86	86
Fluorant	cmpd8	Fluoranthene	8	202.26	0	430	430	220	220	620	620
Pyrene	cmpd9	Pyrene	9	202.24	0	170	170	80	80	220	220
Benzo (a	cmpd10	Benzo (a) anthrancene	10	228.29	0	45	45	25	25	72	72
Chrysen	cmpd11	Chrysene	11	228.28	0	120	120	59	59	160	160
Benzo (b	cmpd12	Benzo (b) fluoranthene	12	252.32	0	76	76	45	45	64	64
Benzo (k	cmpd13	Benzo (k) fluoranthene	13	252.32	0	50	50	31	31	39	39
Benzo (e	cmpd14	Benzo (e) pyrene	14	252.3	0	42	42	32	32	34	34
Benzo (a	cmpd15	Benzo (a) pyrene	15	252.3	0	47	47	24	24	32	32
Perylene	cmpd16	Perylene	16	202.24	0	12	12	ND	ND	ND	ND
Indeno (	cmp17	Indeno (1,2,3-cd) pyrene	17	290.34	0	44	44	32	32	35	35
Dibenz (	cmpd18	Dibenz (a,h) anthracene	18	278.33	0	12	12	ND	ND	ND	ND
Benzo (g	cmpd19	Benzo (g,h,i) perylene	19	276.34	0	81	81	78	78	63	63

APPENDIX E

QA/QC DATA

REPORT OF METHOD AUDIT

Laboratory:
Quanterra Environmental Services
880 Riverside Parkway
West Sacramento, CA 95605

Telephone: 916-373-5600

Project: Indiana Coke (Proj #R012.000)

Audit Date: September 2, 1998

Audit conducted by: Dennis Becvar of PES, and Robert Weidenfeld of Quanterra

Purpose: The purpose of this audit was to verify if the analytical procedures of Quanterra Environmental Services (Quanterra) for Polyaromatic Hydrocarbons were followed in accordance with CARB Method 429 as published August 9, 1996.

Sample Receiving and Log in: Samples are received in a dedicated area (isolated from the analytical laboratories) of the facility. The client name, description of the samples, date and time received, requested methods and analytes are recorded on a sample receiving form directly from the chain of custody form sent with the package containing the samples. In order to minimize contamination or mixing of samples/clients only one package is opened at a time. After the package is opened, each sample is removed from the packing material and the sample container is immediately checked for temperature (the acceptable temperature range is 4±2 °C) with an infrared, unobtrusive, temperature sensor. In addition to the temperature check, each sample is checked for leakage, shipping damage, proper labeling, intact-seals, and volume marks on bottles with liquids. After the samples are logged into the Quanterra sample tracking system, they are stored in a large cold storage room. The Quanterra sample tracking system is interfaced with their Laboratory Information Management System (LIMS), and each "logged in" sample is identified with a computer generated laboratory control number and the location (rack and shelf) the sample will be stored in the refrigerator. The temperature in the refrigerator is maintained at 2-4 °C; also, for documentation purposes, the temperature is continuously recorded on a chart recorder.

If there are any questions regarding the samples the Quanterra project manager is notified, and if necessary, the client is contacted. It should be noted that for the samples delivered by PES for the above project, one of the filters was wet from the ice in the shipping container.

Sample Extraction and Concentration: Within a week or less (the method requires extraction to occur within 21 days of sample collection) the samples are transferred under chain of custody to the extraction laboratory. The XAD-2 cartridges, front half washes and filters are transferred to a precleaned Soxhlet apparatus. All of the Soxhlets are cleaned with methylene chloride over a period of 8-12 hours. The samples are extracted with methylene chloride over a period of sixteen hours at a recylce rate of approximately three cycles per hour. Internal standards are added to the samples before extraction. After the Soxhlet has been allowed to cool the sample is equally divided with one half of the sample retained as an archive sample and the second half is prepared for cleanup with a silica gel column, dried through sodium sulfate, and subsequently concentrated.

Sample Analysis: The extracted and concentrated samples are routed to the high resolution GC/MS laboratory for analysis. The samples are placed in an autosampler tray and the samples are injected into the instrument automatically. The sample injector is purged two times with solvent between sample injections. In addition to internal standards in each sample, the instrument is restandardized after each fifteen samples. The instrument in programmed for control limits and the data in the sample set are flagged if the instrument exceeds the allowable window for drift.

Data Reporting: After the sample analyses are completed the data are checked manually for completeness. If any matrix effects or sample anomalies are found the samples are routed through the analytical laboratory for reanalysis. Each data package sent out by the laboratory is approved by the QA/QC manager and the project manager for completeness.

General Comments: The laboratory is well organized, extremely well maintained and staffed with analysts having many years of experience. Since the analyses are organized by analytical group, i.e. PAH's, metals, etc. each of the analysts has a very thorough understanding of the analysis they are performing. The instrumentation is state of the art and operated by analysts with at least five years of experience. The electronic outputs of the instruments are connected the LIMS. Access to the data in the LIMS is restricted to the analysts, QA/QC staff and project manager, in all cases, the data are in a "read only" format. The quality assurance program appears to be very thorough with sufficient electronic controls and human reviews to flag any data that is outside the acceptable quality control criteria for the analysis. Corrective actions are included in all steps of Quanterra's quality control process.

This auditor did not observe any sample handling or analytical procedures outside the procedures of CARB Method 429 that would result in the invalidation of data for the above referenced project. Mr. Weidenfeld of Quanterra mentioned that the sample matrix for the above samples was expected to be relatively complex and data interpretation would require a significant amount of data reduction time. The schedule for most CARB 429 projects is to issue the laboratory report within 30 days of receiving the samples.

PROCEDURES AUDIT FOR METHOD 315
ERG LABORATORIES

The following is a record of a visit to ERG Labs to evaluate their procedures and handling of Method 315 samples of emission tests from coke ovens. The work was performed by ERG chemist Mr. Linh Nguyen under the direction of Dr. Joan T. Bursey, Laboratory Director.

The first objective was to observe the procedure for weighing the samples. After the samples were logged in, they were transferred to the weighing room, which is temperature and humidity controlled. The samples were then put in desiccators to desiccate for twenty-four hours before being weighed. The samples were then weighed on a laboratory balance. The balance was located on a weighing table, which is especially designed to add stability and provide more accurate weight measurements.

Because the analysis of Method 315 samples for Methylene Chloride Extractable Matter (MCEM) is done over a period of about seven to eight working days, it was not possible to observe the analysis in its entirety. It was decided to observe two segments of the analysis on separate visits. During the first visit the procedures handling of the filters for MCEM analysis, were observed to verify that the analyses were done according to section 11.2.1.2 of the method. The chemist was very meticulate in his work and explained the procedures as they were occurring and answered questions as they were raised. The second visit was about four days later or midway into the analysis. This segment included the MCEM analysis of the impinger solutions. This was done following the procedures in section 11.2.4.1 of the method. This involved the adding of a known quantity of methylene chloride to the impinger water in a separatory funnel and extracting off the phase that had separated. This was done a total of three times. The extracts were then heated to near dryness, transferred to an aluminum dish where the extract was allowed to dry and reach equilibration in the balance room. The sample was then weighed to the nearest 0.1 mg. After analysis was completed and data computed, the data wasreviewed by the laboratory director for accuracy and completeness.

This observer did not notice any sample handling or analytical procedures outside the procedures of Method 315 that would result in the invalidation of data for the above referenced project.

Pacific Environmental Services, Inc.

Memo

To: PES Project File S511.000

From: Franklin Meadows, Project Manager

CC:

Date: 11/13/98

Re: First Analytical Laboratory Visit

The PM and MCEM sample residues were hand delivered by the PES Project Manager to First Analytical Laboratory (FAL) for subsequent analysis for 17 trace metals. The samples were received in a dedicated sample receiving area where they were unpacked and arranged by Run Number and sample fraction. The samples were inspected for integrity, damage, and chain-of-custody documentation. All of the samples were received in good condition and all were accounted for.

Once the samples were accounted for, Dr. William A. Wadlin, FAL Laboratory Manager, assigned a project number to the PES project and logged the samples into FAL's sample tracking system. Each sample and sample fraction was assigned a unique identification number and the samples were again compared to the sample chain-of-custody document.

During the sample preparation stage, each sample digestate was labelled with the original sample identification code. These numbers were used to track the sample through the sample preparation and digestion process. All raw data printouts and calibration curves are labelled with the identification number. These numbers were used throughout the preparation and analysis to the final report of the results, so that a paper trail could be generated for each analytical result that tracks the result back to the original sample.

A walk-through of the laboratory indicated that the laboratory was well organized, clean, and well maintained. The samples were prepared and analyzed by Dr. Wadlin. Standard FAL procedure is to analyze the samples progressively first using the procedure having the least analytical sensitivity followed by the procedure having the next best analytical sensitivity. The laboratory continuously monitors instrument performance and makes necessary instrument repairs in advance of instrument failure so that samples do not need to be re-analyzed due to instrument performance outside the method QC requirements.

FAL has been participating in the EPA's quarterly audit for metals analysis (lead) for the past three years. FAL is also approved by EPA Region I for EPA Method 29 analyses.

NOZZLE CALIBRATION SHEET

DATE: 8-11-98 CALIBRATION BY: D. Schuffel

Nozzle Identification Number	D_1, in.	D_2, in.	D_3, in.	ΔD, in.	D_{avg}
I-315	0.214	0.216	0.215	0.002	0.215
I-429	0.218	0.216	0.217	0.002	0.217
.3.1 Replaced I-429	0.216	0.217	0.217	0.001	0.217
0-315	0.219	0.221	0.220	~~[illegible]~~ DS 0.002	0.220
0-429	0.222	0.219	0.219	0.003	0.220

Where:

$D_{1,2,3}$ = nozzle diameter measured on a different diameter, in.
Tolerance = measure within 0.001 in.

ΔD = maximum difference in any two measurements, in.
Tolerance = 0.004 in.

D_{avg} = average of D_1, D_2, D_3.

NOZZLE CALIBRATION SHEET

DATE: 8/12/98

CALIBRATION BY: RWA

Nozzle Identification Number	D_1, in.	D_2, in.	D_3, in.	ΔD, in.	D_{avg}
B-O-M315-2	0.254	0.255	.254	.001	0.254
B-O-M429-2	0.254	0.254	0.254	.000	0.254
B-O-	0.255	0.254	0.255	0.001	0.254
B-I-429-2	0.215	0.215	0.216	0.001	0.215
B-I-315-2	0.214	0.216	0.215	0.002	0.215

Where:

$D_{1,2,3}$ = nozzle diameter measured on a different diameter, in.
Tolerance = measure within 0.001 in.

ΔD = maximum difference in any two measurements, in.
Tolerance = 0.004 in.

D_{avg} = average of D_1, D_2, D_3.

NOZZLE CALIBRATION SHEET

UNDERFIRE STACK

DATE: 8-14-98

CALIBRATION BY:

Nozzle Identification Number	D_1, in.	D_2, in.	D_3, in.	ΔD, in.	D_{avg}
G .442	0.441	0.443	0.442	0.002	0.442
G .432	0.432	0.431	0.432	0.001	0.432
G .500	0.501	0.499	0.501	0.002	0.500
G .497	0.496	0.499	0.497	0.003	0.497
G .436	0.436	0.436	0.436	0.000	0.436
G .435	0.435	0.436	0.434	0.002	0.435
G .434	0.434	0.434	0.434	0.000	0.434

Where:

$D_{1,2,3}$ = nozzle diameter measured on a different diameter, in.
Tolerance = measure within 0.001 in.

ΔD = maximum difference in any two measurements, in.
Tolerance = 0.004 in.

D_{avg} = average of D_1, D_2, D_3.

NOZZLE CALIBRATION SHEET

DATE: 8/15/98 CALIBRATION BY: RK

Nozzle Identification Number	D_1, in.	D_2, in.	D_3, in.	ΔD, in.	D_{avg}
~~B-v-429-3~~	~~0.497~~	~~0.498~~	~~0.497~~	~~0.001~~	~~0.497~~
B-v-315-3	0.499	0.499	0.498	0.001	0.498
B-v-429-3	0.475	0.474	0.475	0.001	0.475

Where:

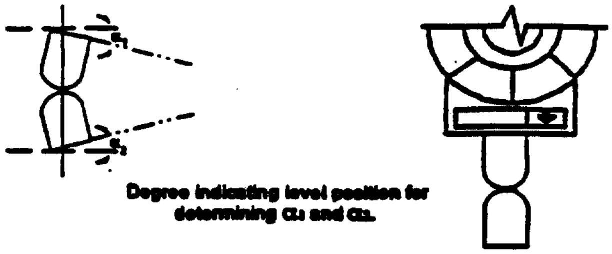
$D_{1,2,3}$ = nozzle diameter measured on a different diameter, in.
Tolerance = measure within 0.001 in.

ΔD = maximum difference in any two measurements, in.
Tolerance = 0.004 in.

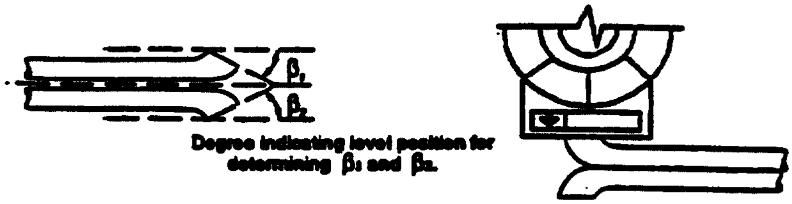
D_{avg} = average of D_1, D_2, D_3.

CALIBRATION DATA SHEET 2
Type S Pitot Tube Inspection

Level and Perpendicular?	YES
Obstruction?	NO
Damaged?	NO
α_1 (-10° ≤ α_1 ≤ +10°)	0
α_2 (-10° ≤ α_2 ≤ +10°)	1
β_1 (-5° ≤ β_1 ≤ +5°)	1
β_2 (-5° ≤ β_2 ≤ +5°)	0
γ	1
Θ	0
$z = A \tan \gamma$ (≤ 0.125")	.0179
$w = A \tan \Theta$ (≤ 0.03125")	0
D_t (3/16" ≤ D_t ≤ 3/8")	3/8
A	1.023
$A/2D_t$ (1.05 ≤ P_A/D_t ≤ 1.5)	1.36

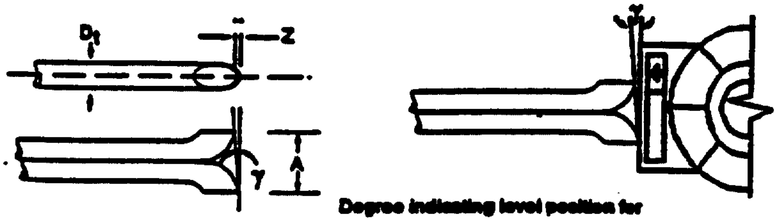


Degree indicating level position for determining α_1 and α_2.



Degree indicating level position for determining β_1 and β_2.

Degree indicating level position for determining Θ



Degree indicating level position for determining γ then calculate Z.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

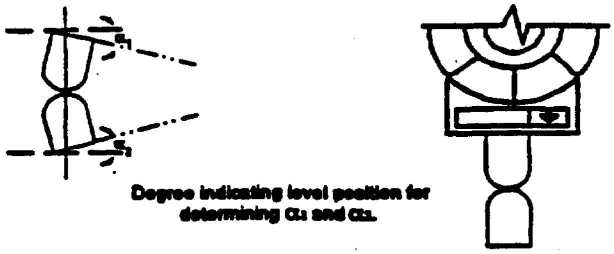
Certification

I certify that the Type S pitot tube/probe ID# RP-21 meets or exceeds all specifications, criteria and/or applicable design features and is hereby assigned a pitot tube calibration factor C_p of 0.84.

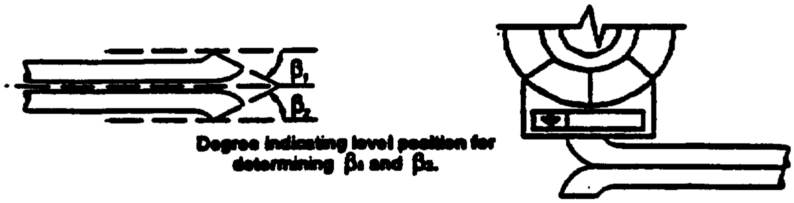
Certified by: 7-14-98 ______________________

Personnel (Signature/Date) Team Leader (Signature/Date)

CALIBRATION DATA SHEET 2
Type S Pitot Tube Inspection

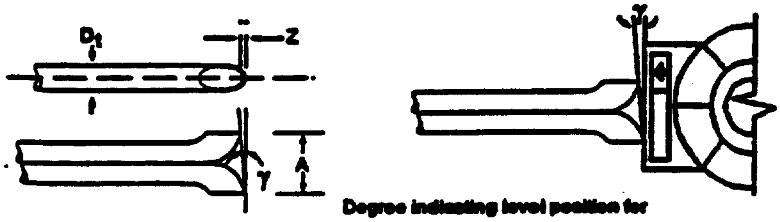


Degree indicating level position for determining α1 and α2.



Degree indicating level position for determining β1 and β2.

Degree indicating level position for determining θ.



Degree indicating level position for determining γ then calculate Z.

Level and Perpendicular?	✓
Obstruction?	N/A
Damaged?	N/A
α_1 ($-10° \le \alpha_1 \le +10°$)	0
α_2 ($-10° \le \alpha_2 \le +10°$)	1
β_1 ($-5° \le \beta_1 \le +5°$)	1
β_2 ($-5° \le \beta_2 \le +5°$)	0
γ	1
θ	0
$z = A \tan \gamma$ ($\le 0.125"$)	.0179
$w = A \tan \theta$ ($\le 0.03125"$)	0
D_t ($3/16" \le D_t \le 3/8"$)	3/8
A	1.023
$A/2D_t$ ($1.05 \le P_A/D_t \le 1.5$)	1.36

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Certification

I certify that the Type S pitot tube/probe ID# RP-20 meets or exceeds all specifications, criteria and/or applicable design features and is hereby assigned a pitot tube calibration factor C_p of 0.84.

Certified by: DDH 11-2-98
Personnel (Signature/Date)

Team Leader (Signature/Date)

5B

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Pitot Tube Number: 5B

Date: 12/22/97

Effective Length: 64"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 3 °(< 10°)

α_2 = 3 °(< 10°)

β_1 = 1 °(< 5°)

β_2 = 3 °(< 5°)

γ = 3

θ = 1

A = 1.124

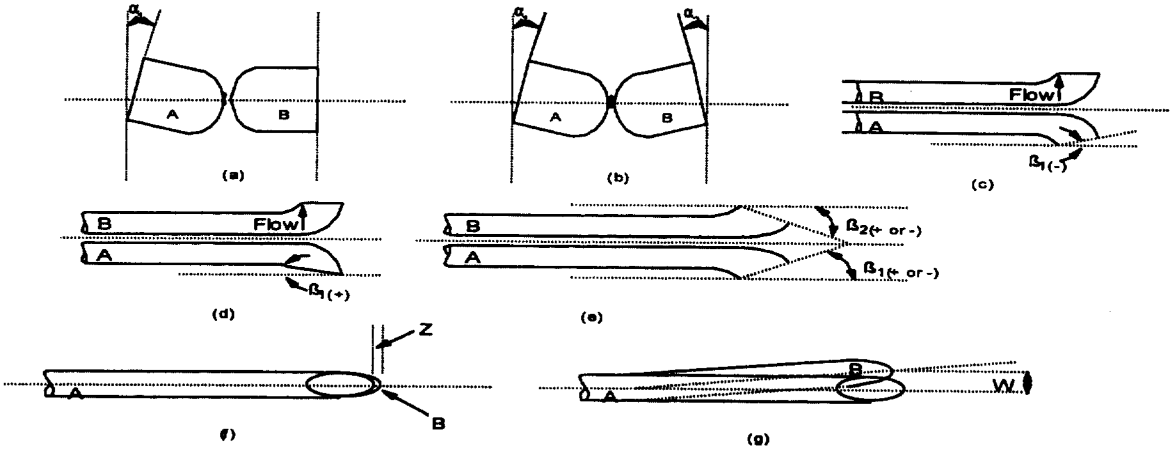
$z = A \sin \gamma$ = 0.059 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta$ = 0.020 cm (in.) 0.08 cm (< 1/32 in.)

P_A = 0.562 cm (in.)

P_B = 0.562 cm (in.)

D_t = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm(1/32 in.) (reference 11.0 in Section 16.0).

5C

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Pitot Tube Number: 5C

Date: 12/23/97

Effective Length: 61"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 0 °(< 10°)

α_2 = 1 °(< 10°)

β_1 = 1 °(< 5°)

β_2 = 1 °(< 5°)

Y = 1

θ = 1

A = 0.948

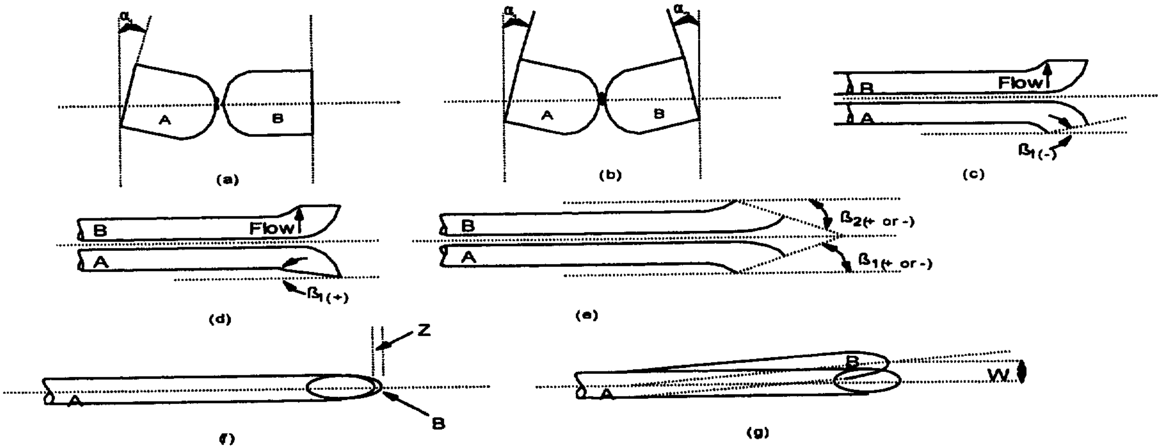
$z = A \sin \gamma$ = 0.017 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta$ = 0.017 cm (in.) 0.08 cm (< 1/32 in.)

P_A = 0.474 cm (in.)

P_β = 0.474 cm (in.)

D_t = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm (1/32 in.) (reference 11.0 in Section 16.0).

5E

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Pitot Tube Number: 5E

Date: 12/22/97

Effective Length: 64"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 1 °(< 10°)

α_2 = 1 °(< 10°)

β_1 = 1 °(< 5°)

β_2 = 3 °(< 5°)

Y = 0

θ = 1

A = 1.122

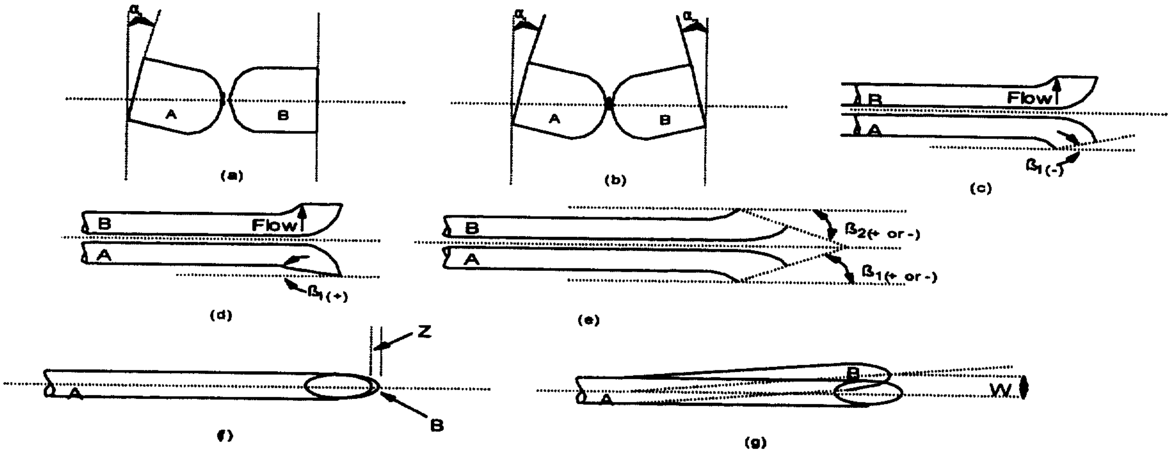
$z = A \sin \gamma$ = 0 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta$ = 0.02 cm (in.) 0.08 cm (< 1/32 in.)

P_A = 0.561 cm (in.)

P_β = 0.561 cm (in.)

D_t = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm (1/32 in.) (reference 11.0 in Section 16.0).

7A

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Pitot Tube Number: 7A

Date: 12/22/97

Effective Length: 86"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 0 °(< 10°)

α_2 = 1 °(< 10°)

β_1 = 3 °(< 5°)

β_2 = 2 °(< 5°)

Y= 4

θ = 1

A = 0.996

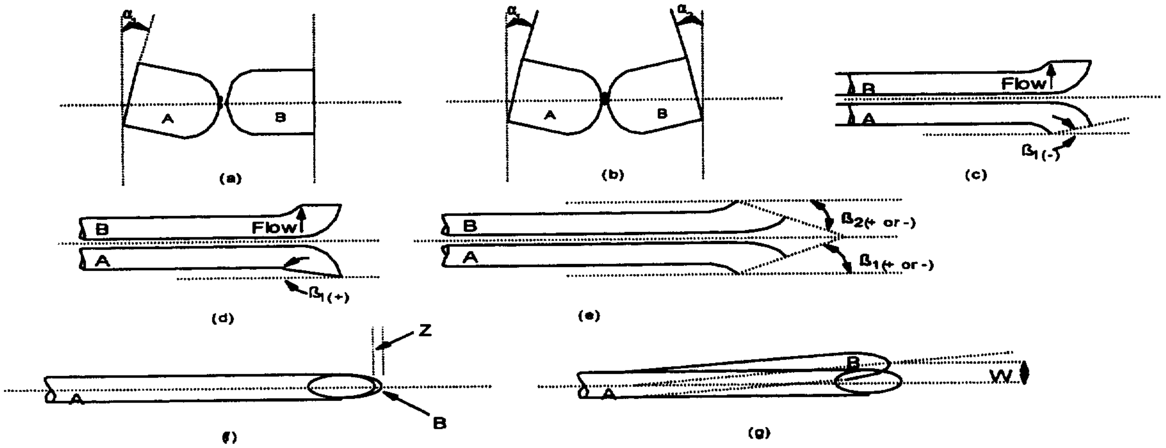
$z = A \sin \gamma$ = 0.069 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta$ = 0.017 cm (in.) 0.08 cm (< 1/32 in.)

P_A = 0.498 cm (in.)

P_β = 0.498 cm (in.)

D_t = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm(1/32 in.) (reference 11.0 in Section 16.0).

7D

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Pitot Tube Number: 7D

Date: 12/23/97

Effective Length: 84.5"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 3 °(< 10°)

α_2 = 3 °(< 10°)

β_1 = 1 °(< 5°)

β_2 = 1 °(< 5°)

Y= 1

θ = 0

A = 0.931

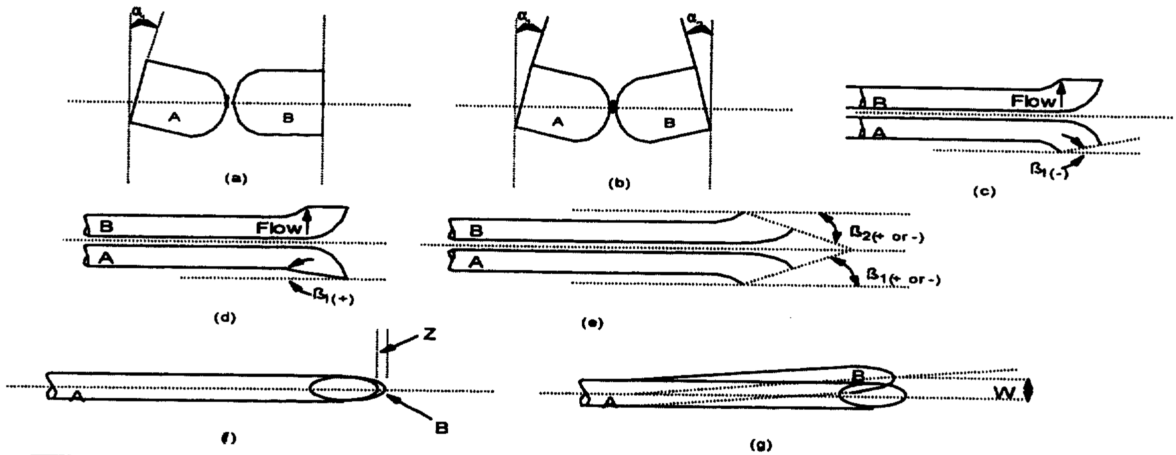
$z = A \sin \gamma$ = 0.016 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta$ = 0 cm (in.) 0.08 cm (< 1/32 in.)

P_A = 0.466 cm (in.)

P_β = 0.465 cm (in.)

D_t = 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm(1/32 in.) (reference 11.0 in Section 16.0).

8C

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Pitot Tube Number: 8C

Date: 12/12/97

Effective Length: 85"

Calibrated By: S. Simon

Pitot Tube Openings Damaged? YES [✓ NO]

Pitot Tube Assembly Level? [✓ YES] NO

α_1 = 3 °(< 10°)

α_2 = 0 °(< 10°)

β_1 = 1 °(< 5°)

β_2 = 1 °(< 5°)

Y = 1

θ = 0

A = 0.939

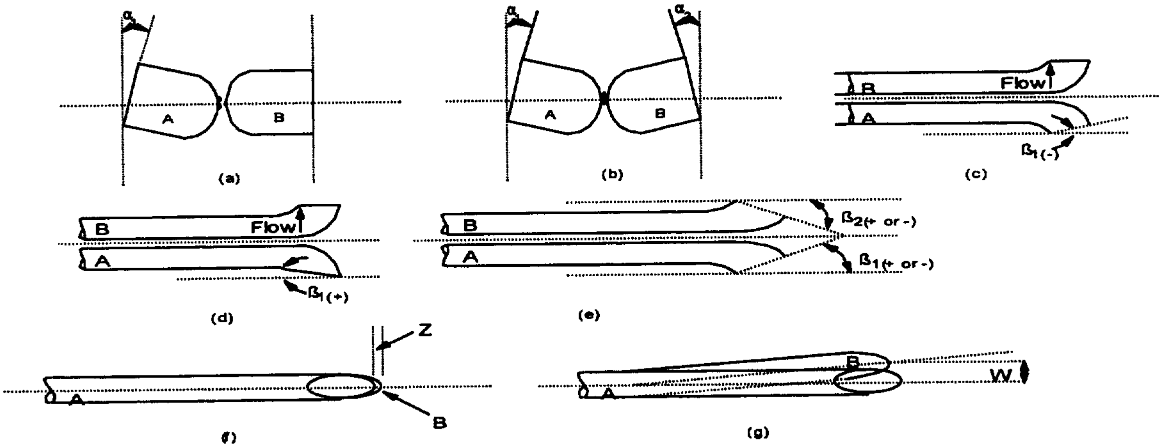
$z = A \sin \gamma =$ 0.016 cm (in.) 0.32 cm (< 1/8 in.)

$w = A \sin \theta =$ 0 cm (in.) 0.08 cm (< 1/32 in.)

$P_A =$ 0.47 cm (in.)

$P_\beta =$ 0.469 cm (in.)

$D_t =$ 0.375 cm (in.)



The types of face-opening misalignment shown above will not affect the baseline value of Cp(s) so long as α_1 and α_2 is less than or equal to 10°, β_1 and β_2 is less than or equal to 5°, z is less than or equal to 0.32 cm (1/8 in.), and w is less than or equal to 0.08 cm (1/32 in.) (reference 11.0 in Section 16.0).

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-2

AMBIENT TEMPERATURE(°F): 77

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:

Mercury-in-glass: ASTM-3

Other:

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	77	76.6	0.4
2	Cold Bath	40	40.6	0.6

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-3

AMBIENT TEMPERATURE(°F): 77

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other: ________

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	77	76.6	0.4
2	Cold Bath	42	42.8	0.8

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-4

AMBIENT TEMPERATURE(°F): 76

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other: ____________

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature, °F	Thermocouple Potentiometer Temperature, °F	Temperature Difference,[b] °F
1	Ambient Air	76	76	0
2	Cold Bath	39	39.2	0.2

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-5

AMBIENT TEMPERATURE(°F): 76

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other:

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	76	76	0
2	Cold Bath	40	40.8	0.8

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-6

AMBIENT TEMPERATURE(°F): 77

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other:

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	77	77.4	0.4
2	Cold Bath	40	40.8	0.8

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-7

AMBIENT TEMPERATURE(°F): 77

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other: ____

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	77	78	1
2	Cold Bath	40	41.2	1.2

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FORM FOR SAMPLE HEAD

DATE: 12/19/97

THERMOCOUPLE NUMBER: SH-8

AMBIENT TEMPERATURE(°F): 76

BAROMETRIC PRES.(In.Hg): 29.33

CALIBRATOR: S.Simon

REFERENCE:
Mercury-in-glass: ASTM-3

Other:

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] °F
1	Ambient Air	76	76.4	0.4
2	Cold Bath	39	38.2	0.8

[a] Type of calibration used.

[b] Allowable tolerance ±2°F

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

THERMOCOUPLE NUMBER: 5B

DATE: 12/22/97

BAROMETRIC PRES.(In.Hg): 29.52

REFERENCE:
Mercury-in-glass: ASTM-3F

Other:

AMBIENT TEMP. °F: 72

CALIBRATOR: J.C.

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] %
1	Ambient Air	72	72	0.00
2	Cold Bath	44	44	0.00
3	Hot Bath	204	204	0.00
4	Hot Oil	400	400	0.00

[a] Type of calibration used.

[b] $\frac{(\text{ref. temp.}^\circ F+460)-(\text{test thermometer temp.}^\circ F+460)}{\text{ref temp,}^\circ F+460}$ X100 100≤1.5%

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

THERMOCOUPLE NUMBER: T5B
DATE: 12/23/97

BAROMETRIC PRES.(In.Hg): 29.52
REFERENCE:
Mercury-in-glass: ASTM-3F

Other:

AMBIENT TEMP. °F: 74
CALIBRATOR: J.C.

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] %
1	Ambient Air	74	74	0.00
2	Cold Bath	46	46	0.00
3	Hot Bath	200	200	0.00
4	Hot Oil	318	318	0.00

[a] Type of calibration used.

[b] $\frac{\text{(ref. temp.°F+460)-(test thermometer temp.°F+460)}}{\text{ref temp,°F+460}}$ X100 100≤1.5%

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

THERMOCOUPLE NUMBER: 7A **DATE:** 12/23/97

BAROMETRIC PRES.(In.Hg): 29.52 **REFERENCE:**
Mercury-in-glass: ASTM-3F

Other:

AMBIENT TEMP. °F: 74

CALIBRATOR: G.Gay

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] %
1	Ambient Air	74	74	0.00
2	Cold Bath	36	36	0.00
3	Hot Bath	198	199	0.15
4	Hot Oil	333	332	0.13

[a] Type of calibration used.

[b] $\frac{(\text{ref. temp,}^\circ F+460)-(\text{test thermometer temp,}^\circ F+460)}{\text{ref temp,}^\circ F+460}$ X100 100≤1.5%

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

THERMOCOUPLE NUMBER: 7D

DATE: 12/23/97

BAROMETRIC PRES.(In.Hg): 29.52

REFERENCE:
Mercury-in-glass: ASTM-3F

Other:

AMBIENT TEMP. °F: 74

CALIBRATOR: G. Gay

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] %
1	Ambient Air	74	74	0.00
2	Cold Bath	40	41	0.20
3	Hot Bath	206	205	0.15
4	Hot Oil	340	341	0.13

[a] Type of calibration used.

[b] $\frac{\text{(ref. temp.°F+460)-(test thermometer temp.°F+460)}}{\text{ref temp,°F+460}}$ X100   100≤1.5%

Comments:

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TEMPERATURE SENSOR CALIBRATION DATA FOR STACK THERMOCOUPLES

THERMOCOUPLE NUMBER: 8C

DATE: 12/15/97

BAROMETRIC PRES.(In.Hg): 29.52

REFERENCE:
Mercury-in-glass: ASTM-3F

Other:

AMBIENT TEMP. °F: 74

CALIBRATOR: G. Gay

Reference point number	Source[a] (Specify)	Reference Thermometer Temperature,°F	Thermocouple Potentiometer Temperature,°F	Temperature Difference,[b] %
1	Ambient Air	74	74	0.00
2	Cold Bath	38	38	0.00
3	Hot Bath	199	199	0.00
4	Hot Oil	339	339	0.00

[a] Type of calibration used.

[b] $\frac{\text{(ref. temp.°F+460)-(test thermometer temp.°F+460)}}{\text{ref temp,°F+460}}$ X100 $\quad$ 100≤1.5%

Comments:

TEMPERATURE SENSOR CALIBRATION FORM

Temperature Sensor No. RP-20 (PROBE) **Sensor Type** K-TC **Length** ______

Ambient Temp. °F 72 **Barometric Pressure, "Hg** 30.21

Reference Temp. Sensor: ______

Date	Ref. Point No.	Temp. Source	Temp. °F		Temp. Diff. %	Within Limits Y/N	Calibrated By
			Ref. Sensor	Test Sensor			
1-22-98	1	ICE H_2O	32	32	0	Y	JWB
"	2	AMB. AIR	72	69	-.65	Y	JWB
"	3	BOIL. H_2O	210	208	-.298	Y	JWB
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						

$$\%\ \text{Temp. Diff} = \frac{(\text{Ref. Temp} + 460) - (\text{Test Temp.} + 460)}{(\text{Ref. Temp.} + 460)} \times 100 \leq 1.5\ \%$$

TEMPERATURE SENSOR CALIBRATION FORM

Temperature Sensor No. 2 (ON PROBE) **Sensor Type** K-TC **Length** 12'
Ambient Temp. °F 74 **Barometric Pressure, "Hg** 29.88"
Reference Temp. Sensor:

Date	Ref. Point No.	Temp. Source	Temp. °F		Temp. Diff. %	Within Limits Y/N	Calibrated By
			Ref. Sensor	Test Sensor			
1-29-98	1	ICE H2O	32	32	0	Y	JWB
"	2	AMB. AIR	74	75	.187	Y	JWB
"	3	BOIL. H2O	208	209	.150	Y	JWB
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						
	1						
	2						
	3						

$$\% \text{ Temp. Diff} = \frac{(\text{Ref. Temp} + 460) - (\text{Test Temp.} + 460)}{(\text{Ref. Temp.} + 460)} \times 100 \leq 1.5\,\%$$

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Fax: (513)398-3342
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Box No.: MB-1
Bar. Press.(Pb): 29.13 in. Hg

Date: 10-27-97
Calibrated By : TAA

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vaccum	10	10	10	10	10	10
Vw_1	Initial RTM	934.200	952.122	963.339	975.415	987.492	999.025
Vw_2	Final RTM	951.896	962.945	975.195	987.207	998.114	1009.583
Vd_1	Initial DGM	448.724	466.737	478.016	490.201	502.365	513.990
Vd_2	Final DGM	466.501	477.617	489.970	502.081	513.078	524.620
Tw	Ave. Temp RTM °F	70	72	71	72	70	71
Td	Ave. Temp DGM °F	79	82	83	84	85	87
t	Time (min.)	46.000	23.000	22.0	18.0	14.0	10.0

[redacted]

$Vw_2 - Vw_1$	Net Volume RTM	17.696	10.823	11.856	11.792	10.622	10.558
$Vd_2 - Vd_1$	Net Volume DGM	17.777	10.880	11.954	11.880	10.713	10.630
	Y	1.011	1.012	1.012	1.011	1.014	1.013
	dH@	1.916	1.925	1.946	1.979	1.949	2.013

AVERAGE Y = 1.012

Average Y Range = 0.992 TO 1.032 **ACCEPT**

AVERAGE dH@ 1.954

Average dH@ Range = 1.754 TO 2.154 **ACCEPT**

Calculations

$$Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw + 460))$$

$$dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw + 460) * t) / Vw)^2$$

Initial Dry Gas Meter Calibration Form (English Units) **1998 Yearly Calibration**

PACIFIC ENVIRONMENTAL SERVICES, INC.

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Box No.:	1	Bar. Press.(Ps):	30.00	in. Hg
Date:	8-17-98	Pretest Gamma:	1.012	
Calibrated By :	gg	Pretest dH@:	1.954	
Plant:	Bethlemem Steel			

[redacted]		RUN 1	RUN 2	RUN 3
DH	Delta H	1.90	1.90	1.90
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	69.112	76.785	84.492
Vw_2	Final RTM	76.785	84.492	91.992
Vd_1	Initial DGM	274.034	281.633	289.274
Vd_2	Final DGM	281.633	289.274	296.735
Tw	Ave. Temp RTM °F	69.0	69.0	69.0
Td	Ave. Temp DGM °F	70.0	76.0	74.0
t	Time (min.)	10.0	10.0	10.0
[redacted]				
$Vw_2 - Vw_1$	Net Volume RTM	7.673	7.707	7.500
$Vd_2 - Vd_1$	Net Volume DGM	7.599	7.641	7.461
	Y	1.007	1.017	1.010
	dH@	1.801	1.765	1.870

AVERAGE Y = 1.011

% Difference from Yearly Y = -0.058

ACCEPT

AVERAGE dH@ = 1.812

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * time) / Vw)^2

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Box No.: 2 Bar. Press.(Pb): 29.50 in. Hg

Date: 10-17-97 Calibrated By : RJK

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vacuum	10	10	10	10	10	10
Vw_1	Initial RTM	558.612	574.095	586.200	598.965	612.242	630.900
Vw_2	Final RTM	569.029	585.906	598.741	612.032	630.414	646.492
Vd_1	Initial DGM	108.182	123.684	135.800	148.548	161.792	180.404
Vd_2	Final DGM	118.634	135.517	148.334	161.596	179.920	195.932
Tw	Ave. Temp RTM °F	75	76	78	78	79	80
Td	Ave. Temp DGM °F	75	78	80	82	83	85
t	Time (min.)	25.5	24.0	22.0	19.0	23.0	14.0
[redacted]							
$Vw_2 - Vw_1$	Net Volume RTM	10.417	11.811	12.541	13.067	18.172	15.592
$Vd_2 - Vd_1$	Net Volume DGM	10.452	11.833	12.534	13.048	18.128	15.528
	Y	0.996	0.999	1.003	1.004	1.006	1.003
	dH@	1.719	1.779	1.769	1.822	1.839	1.852

AVERAGE Y =	**1.002**				
Average Y Range =		0.982	TO	1.022	**ACCEPT**
AVERAGE dH@ =	**1.797**				
Average dH@ Range =		1.597	TO	1.997	**ACCEPT**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * t) / Vw)^2

Initial Dry Gas Meter Calibration Form (English Units) **1998 Yearly Calibration**

PACIFIC ENVIRONMENTAL SERVICES, INC.

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Phone:(513)398-2556
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Box No.:	MB 02	Bar. Press.(Pb):	30.00	in. Hg
Date:	8-17-98	Pretest Gamma:	1.002	
Calibrated By :	sb	Pretest dH@:	1.797	
Plant:	Bethlhem Steel			

[redacted]		RUN 1	RUN 2	RUN 3
DH	Delta H	1.80	1.80	1.80
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	18.042	35.622	53.240
Vw_2	Final RTM	35.622	53.240	68.529
Vd_1	Initial DGM	382.086	399.555	417.231
Vd_2	Final DGM	399.555	417.231	432.629
Tw	Ave. Temp RTM °F	74.0	74.0	74.0
Td	Ave. Temp DGM °F	73.0	77.0	79.0
t	Time (min.)	23.0	23.0	20.0
[redacted]				
$Vw_2 - Vw_1$	Net Volume RTM	17.580	17.618	15.289
$Vd_2 - Vd_1$	Net Volume DGM	17.469	17.676	15.398
	Y	1.000	0.998	0.998
	dH@	1.742	1.721	1.722

AVERAGE Y = 0.999

% Difference from Yearly Y = -0.340 **ACCEPT**

AVERAGE dH@ = 1.728

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * time) / Vw)^2

Posttest Dry Gas Meter Calibration Form (English Units)

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AKA "MB-3"

Box No.:	13	Bar. Press.(Pb):	29.59	in. Hg
Date:	10-16-97	Calibrated By :	R. Kolde	

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vacuum	10	10	10	10	10	10
Vw_1	Initial RTM	474.707	484.860	493.733	506.082	517.525	528.376
Vw_2	Final RTM	484.514	493.318	505.845	517.191	527.985	539.639
Vd_1	Initial DGM	70.442	80.600	89.500	101.900	113.405	124.306
Vd_2	Final DGM	80.267	89.087	101.668	113.061	123.902	135.615
Tw	Ave. Temp RTM °F	90	89	89	89	90	91
Td	Ave. Temp DGM °F	95	95	94	95	98	99
t	Time (min.)	24.0	17.0	21.0	16.0	13.0	10.0

[redacted]

$Vw_2 - Vw_1$	Net Volume RTM	9.807	8.458	12.112	11.109	10.460	11.263
$Vd_2 - Vd_1$	Net Volume DGM	9.825	8.487	12.168	11.161	10.497	11.309
	Y	1.006	1.006	1.002	1.002	1.006	1.000
	dH@	1.749	1.763	1.752	1.810	1.794	1.835

AVERAGE Y =	**1.004**				
Average Y Range =		0.984	TO	1.024	**ACCEPT**
AVERAGE dH@ =	**1.784**				
Average dH@ Range =		1.584	TO	1.984	**ACCEPT**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * t) / Vw)^2

Initial Dry Gas Meter Calibration Form (English Units)

1998 Yearly Calibration

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Box No.:	3(13)	Bar. Press.(Ps):	30.00	in. Hg
Date:	8-17-98	Pretest Gamma:	1.004	
Calibrated By :	gg	Pretest dH@:	1.784	
Plant:	Bethlehem Steel			

[redacted]		RUN 1	RUN 2	RUN 3
DH	Delta H	1.80	1.80	1.80
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	260.644	276.031	291.405
Vw_2	Final RTM	276.031	291.405	307.125
Vd_1	Initial DGM	273.179	288.826	304.526
Vd_2	Final DGM	288.826	304.526	320.620
Tw	Ave. Temp RTM °F	88.0	88.0	88.0
Td	Ave. Temp DGM °F	94.0	96.0	98.0
t	Time (min.)	20.0	20.0	20.5
[redacted]				
$Vw_2 - Vw_1$	Net Volume RTM	15.387	15.374	15.720
$Vd_2 - Vd_1$	Net Volume DGM	15.647	15.700	16.094
	Y	0.990	0.989	0.990
	dH@	1.742	1.739	1.741

AVERAGE Y = **0.990**

% Difference from Yearly Y = **-1.422** **ACCEPT**

AVERAGE dH@ = **1.740**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * time) / Vw)^2

Posttest Dry Gas Meter Calibration Form (English Units)

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AKA "MB-4"

Box No.:	14	Bar. Press.(Pb):	29.20 in. Hg
Date:	11-13-97	Calibrated By :	ggay

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vaccum	10	10	10	10	10	10
Vw_1	Initial RTM	563.413	571.766	583.839	595.357	638.753	614.016
Vw_2	Final RTM	571.445	583.529	595.101	613.720	646.620	638.235
Vd_1	Initial DGM	840.210	848.779	861.129	872.919	917.245	892.031
Vd_2	Final DGM	848.429	860.801	872.660	891.706	925.248	916.654
Tw	Ave. Temp RTM °F	84	84	84	84	75	84
Td	Ave. Temp DGM °F	85	85	85	85	83	85
t	Time (min.)	20.0	24.0	20.0	27.0	10.0	22.0
[redacted]							
$Vw_2 - Vw_1$	Net Volume RTM	8.032	11.763	11.262	18.363	7.867	24.219
$Vd_2 - Vd_1$	Net Volume DGM	8.219	12.022	11.531	18.787	8.003	24.623
	Y	0.978	0.978	0.976	0.976	0.993	0.976
	dH@	1.828	1.840	1.859	1.912	1.849	1.946

AVERAGE Y = 0.979

Average Y Range = 0.959 TO 0.999 **ACCEPT**

AVERAGE dH@ 1.872

Average dH@ Range = 1.672 TO 2.072 **ACCEPT**

Calculations

$$Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw + 460))$$

$$dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw + 460) * t) / Vw)^2$$

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Box No.:	4(14)	Bar. Press.(Pb):	30.00	in. Hg
Date:	8-18-98	Pretest Gamma:	0.979	
Calibrated By :	gg	Pretest dH@:	1.872	
Plant:	Bethlehem Steel			

		RUN 1	RUN 2	RUN 3
DH	Delta H	1.80	1.80	1.80
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	165.419	181.812	198.880
Vw_2	Final RTM	181.812	198.880	213.701
Vd_1	Initial DGM	294.100	310.663	327.945
Vd_2	Final DGM	310.663	327.945	343.025
Tw	Ave. Temp RTM °F	72.0	72.0	72.0
Td	Ave. Temp DGM °F	76.0	78.0	78.0
t	Time (min.)	20.0	20.0	20.0

$Vw_2 - Vw_1$	Net Volume RTM	16.393	17.068	14.821
$Vd_2 - Vd_1$	Net Volume DGM	16.563	17.282	15.080
	Y	0.993	0.994	0.990
	dH@	1.495	1.374	1.822

AVERAGE Y = **0.992**

% Difference from Yearly Y = **1.352** **ACCEPT**

AVERAGE dH@ = **1.564**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * time) / Vw)^2

Posttest Dry Gas Meter Calibration Form (English Units)

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AKA MB-5

Box No.:	MB 15	Bar. Press.(Pb):	29.15	in. Hg
Date:	12-29-97	Calibrated By :	G GAY	

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vaccum	10	10	10	10	10	10
Vw_1	Initial RTM	150.884	157.349	165.074	173.960	184.521	197.212
Vw_2	Final RTM	157.026	164.869	173.741	184.259	196.465	214.019
Vd_1	Initial DGM	564.125	570.758	578.795	587.860	598.779	611.999
Vd_2	Final DGM	570.432	578.496	587.643	598.511	611.127	629.263
Tw	Ave. Temp RTM °F	73	75	75	76	76	78
Td	Ave. Temp DGM °F	76	80	82	84	86	88
t	Time (min.)	15.000	15.000	15.0	15.0	15.0	15.0
[redacted]							
$Vw_2 - Vw_1$	Net Volume RTM	6.142	7.520	8.667	10.299	11.944	16.807
$Vd_2 - Vd_1$	Net Volume DGM	6.307	7.738	8.848	10.651	12.348	17.264
	Y	0.978	0.979	0.990	0.978	0.980	0.982
	dH@	1.719	1.720	1.720	1.827	1.805	1.830

AVERAGE Y =	0.981				
Average Y Range =		0.961	TO	1.001	**ACCEPT**
AVERAGE dH@	1.770				
Average dH@ Range =		1.570	TO	1.970	**ACCEPT**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * t) / Vw)^2

Initial Dry Gas Meter Calibration Form (English Units) **1998 Yearly Calibration**

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Box No.:	15(5)	Bar. Press.(Ps):	30.00	in. Hg
Date:	8-18-98	Pretest Gamma:	0.981	
Calibrated By :	gg	Pretest dH@:	1.710	
Plant:	Bethlehem Steel			

[redacted]		RUN 1	RUN 2	RUN 3
DH	Delta H	1.80	1.80	1.80
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	214.453	230.230	245.365
Vw_2	Final RTM	230.230	245.365	260.368
Vd_1	Initial DGM	608.315	624.400	639.854
Vd_2	Final DGM	624.400	639.854	655.294
Tw	Ave. Temp RTM °F	72.0	72.0	73.0
Td	Ave. Temp DGM °F	81.0	84.0	86.0
t	Time (min.)	20.6	20.0	20.0
[redacted]	[redacted]	[redacted]	[redacted]	[redacted]
$Vw_2 - Vw_1$	Net Volume RTM	15.777	15.135	15.003
$Vd_2 - Vd_1$	Net Volume DGM	16.085	15.454	15.440
	Y	0.993	0.997	0.991
	dH@	1.696	1.728	1.759

AVERAGE Y = **0.994**

% Difference from Yearly Y = **1.296** **ACCEPT**

AVERAGE dH@ = **1.728**

Calculations

$Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw + 460))$

$dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw + 460) * time) / Vw)^2$

Posttest Dry Gas Meter Calibration Form (English Units) 9/18/98

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Phone:(513)398-2556
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AKA MB-6

Box No.:	RMB-13	Bar. Press.(Pb):	29.50	in. Hg
Date:	10-17-97	Calibrated By :	R.Kolde	

[redacted]		RUN 1	RUN 2	RUN 3	RUN 4	RUN 5	RUN 6
DH	Delta H	0.50	0.75	1.00	1.50	2.00	4.00
in Hg	Vacuum	10	10	10	10	10	10
Vw_1	Initial RTM	669.271	680.835	691.078	701.618	713.486	724.113
Vw_2	Final RTM	680.326	690.745	701.245	713.109	723.578	735.099
Vd_1	Initial DGM	23.196	34.797	45.097	55.700	67.635	78.300
Vd_2	Final DGM	34.289	44.775	55.330	67.254	77.774	89.309
Tw	Ave. Temp RTM °F	68.500	67.500	67.000	67.000	67.500	68.500
Td	Ave. Temp DGM °F	73.000	73.500	75.000	75.500	76.500	78.000
t	Time (min.)	27.000	20.000	18.0	17.0	13.0	10.0
[redacted]							
$Vw_2 - Vw_1$	Net Volume RTM	11.055	9.910	10.167	11.491	10.092	10.986
$Vd_2 - Vd_1$	Net Volume DGM	11.093	9.978	10.233	11.554	10.139	11.009
	Y	1.004	1.003	1.006	1.007	1.007	1.006
	dH@	1.680	1.712	1.748	1.830	1.850	1.849

AVERAGE Y =	**1.005**				
Average Y Range =		0.985	TO	1.025	**ACCEPT**
AVERAGE dH@ =	**1.778**				
Average dH@ Range =		1.578	TO	1.978	**ACCEPT**

Calculations

$Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw + 460))$

$dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw + 460) * t) / Vw)^2$

Initial Dry Gas Meter Calibration Form (English Units) **1998 Yearly Calibration**

PACIFIC ENVIRONMENTAL SERVICES, INC.

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Phone:(513)398-2556
Fax: (513)398-3342
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Box No.:	6(Rmb 13)	Bar. Press.(Ps):	30.00	in. Hg
Date:	8-18-98	Pretest Gamma:	1.005	
Calibrated By :	gg	Pretest dH@:	1.778	
Plant:	Bethlehem Steel			

[redacted]		RUN 1	RUN 2	RUN 3
DH	Delta H	1.80	1.80	1.80
in Hg	Vacuum	5.00	5.00	5.00
Vw_1	Initial RTM	105.106	120.425	135.120
Vw_2	Final RTM	120.425	135.120	149.771
Vd_1	Initial DGM	749.253	764.233	779.232
Vd_2	Final DGM	764.233	779.232	794.234
Tw	Ave. Temp RTM °F	70.0	70.0	71.0
Td	Ave. Temp DGM °F	78.0	78.0	81.0
t	Time (min.)	20.0	20.0	20.0
[redacted]				
$Vw_2 - Vw_1$	Net Volume RTM	15.319	14.695	14.651
$Vd_2 - Vd_1$	Net Volume DGM	14.980	14.999	15.002
	Y	1.034	0.990	0.991
	dH@	1.693	1.840	1.847

AVERAGE Y = **1.005**

% Difference from Yearly Y = **-0.024** **ACCEPT**

AVERAGE dH@ = **1.793**

Calculations

Y = (Vw * Pb * (Td + 460)) / (Vd * (Pb + (dHd / 13.6)) * (Tw +460))

dH@ = 0.0317 * dHd / (Pb (Td + 460)) * (((Tw +460) * time) / Vw)^2

Posttest Dry Gas Meter Calibration Form (English Units)

Pacific Environmental Services VOST Box Calibration

Date: 7/19-98
Vost Box Number: V-1

Flow Rate: 1.0 l/min
Rotameter Setting: 1.08
Bubble Meter Temp. : 91

Run 1

Bubble Meter			Meter Box
1012	1007	Initial Volume	9829
1010	1007	Final Volume	9855
1010	1008	Initial Temp.	104
	1009	Final Temp.	104
		Average Temp.	101
Average:	1009.00	Time:	26.25
		QDGM=	972.821
		Y=	1.0372

Run 2

Bubble Meter			Meter Box
1008	1010	Initial Volume	9855.5
1010	1009	Final Volume	9881.5
1011	1007	Initial Temp.	104
	1003	Final Temp.	103
		Average Temp.	103.5
Average:	1008.29	Time:	26.25
		QDGM=	968.505
		Y=	1.0411

Run 3

1009	1005	Initial Volume	9882
1008	1003	Final Volume	9908
1006	1005	Initial Temp.	104
	1008	Final Temp.	103
		Average Temp.	103.5
Average:	1006.29	Time:	26.26
		QDGM=	968.1358553
		Y=	1.0394

Average Y= 1.0392

Pacific Environmental Services VOST Box Calibration

Date: 7/19-98
Vost Box Number: V-2

Flow Rate: 1.0 l/min
Rotameter Setting: 0.95
Bubble Meter Temp. : 91

Run 1

Bubble Meter			Meter Box
1005	1007	Initial Volume	8883.5
1007	1009	Final Volume	8903.5
1007	1012	Initial Temp.	105
	1008	Final Temp.	104
		Average Temp.	104.5
Average:	1007.86	Time:	18.49
		QDGM=	1055.798
		Y=	0.9546

Run 2

Bubble Meter			Meter Box
1007	1008	Initial Volume	8903.5
1006	1009	Final Volume	8923.5
1006	1010	Initial Temp.	105
	1012	Final Temp.	105
		Average Temp.	105
Average:	1008.29	Time:	18.5
		QDGM=	1054.293
		Y=	0.9564

Run 3

1005	1003	Initial Volume	8924
1004	1002	Final Volume	8944
1003	1004	Initial Temp.	105
	1005	Final Temp.	105
		Average Temp.	105
Average:	1003.71	Time:	18.6
		QDGM=	1048.624988
		Y=	0.9572

Average Y= **0.9560**

PACIFIC ENVIRONMENTAL SERVICES, INC.

Central Park West
5001 South Miami Boulevard, P.O. Box 12077
Research Triangle Park, North Carolina 27709-2077
(919) 941-0333 FAX: (919) 941-0234

Posttest Dry Gas Meter Calibration Form (English Units)

Pretest Calibration Factor
System Vacuum Setting, (in Hg)
Reference Meter Correction Factor
Date: 8-18-98 P_{bar}, in Hg ______ Calibrator: JWB Meter Box No. VOST-6

ΔH = 1.8

		VOST	DGM		— Dry Gas Meter —	BUCK	METER	CC/MIN		
Trial	Duration (min)	Initial (ft³) L	Final (ft³)	Net (ft³)	Initial, Inlet ~~(°F)~~	~~Final, Inle~~ (°F)	~~Avg. Inlet (°F)~~	~~Initial, Outlet (°F)~~	~~Final, Outlet (°F)~~	~~Avg. Outlet~~ (°F)
1	10.07	0.0	9.612	9.612	1000	993	979	972	965	961
2	10.08	0.0	9.345	9.345	947	945	943	942	941	940
3	10.07	0.0	9.338	9.338	935	935	936	937	936	934

	BUCK METER	— Reference Meter	CC/MIN				Meter Box Correction Factor	Reference Orifice Press
	Gas Volume			Meter Temperature				
Trial	Initial (ft³)	Final (ft³)	Net (ft³)	Initial (°F)	Final (°F)	Avg. CC/MIN (°F)	Y	ΔH@ (in. H2O)
1	958	954	950	948	945	966	#DIV/0!	#DIV/0!
2	939	939	940	938	937	941	#DIV/0!	#DIV/0!
3	934	935	934	934	935	935	#DIV/0!	#DIV/0!

AVERAGE	[illegible]	[illegible]
% Change	[illegible]	

TOTAL VOL. BUCK METER	Y	Y
9.662	1.005	1.004
9.410	1.007	
9.350	1.001	

BUCK METER READ AT 1 MINUTE INTERVALS -

Baghouse Inlet
Run 2

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana

Methods Run M-Carb 429, M-315
NIOSH 5506

Date August 12, 1998 ~~July~~ , 1998
Page 1 of 2

Quality Control Check	Observation
Prior to Start of Tests	
Keep all cleaned glassware sealed until train assembly	Done
Assemble trains in dust free environment	Done
Visually inspect each train for proper assembly	Done
Level and zero manometer	Done
Calculate proper sampling nozzle size	Done
Visually inspect sampling nozzle for chips	Done
Visually inspect Type S Pitot tube	Done
Leak check each leg of Type S Pitot tube	Done
Leak check entire sampling train	Done
During Testing	
Read temperatures and differential pressures at each traverse point	yes
Sample data and calculations recorded on preformatted data sheets	yes
Unusual occurrences noted in test log	yes
Properly maintain the roll and pitch of axis of Type S Pitots and sampling nozzle	N/A
Leak check train before and after any component changes during test	Done
Maintain the probe and filter temperature	Done
Maintain ice in ice water bath and maintain impinger exit temperature	Done
Calibration forms reviewed for completeness and accuracy	yes
* Data sheets reviewed by ~~PM~~ daily during testing	yes

* Data & Sample Custodian
Operators DDH
M-Carb 429 Meter Box #6
$Y = 1.005$
$\Delta H = 1.778$

Operator G Gay
M-315 Meter Box #1
$Y = 1.012$
$\Delta H = 1.954$

Date ~~July~~ Aug 12, 1998
Page 2 of 2

Quality Control Check	Observation
After Testing	
Visually inspect sampling nozzle	done
Visually inspect Type S Pitot tube	done
Leak check each leg of the Type S Pitot tube	done
Leak check the entire sampling train	done
Record observations if any	yes
Field Log	
Project name/ID and location	Bethleham Steel Chesteton, Ind
Sampling personnel (names/position)	M-Carb 429 M 315 Steve Blaine Nick
Geological observations including map	Flat Terrain approx. 70' off ground
Sample run times and dates	~~10:33AM~~ 9:24 – ~~8:00~~ 7:40 PM 8-11-98
Sample descriptions	M-Carb 429, M-315, NIOSH 5506
Description of QC samples	N/A
Deviations from QAPP	NO
Difficulties in sampling or unusual conditions	Long Sampling period & the large flow chan
Sample Labels	yes
Sample ID	yes
Date and time of collection	8-[illegible]-98 ~~10:33AM – 8:00PM~~ 9:24 AM 7:35
Lab technician initials	RK & RD
Analytical parameter	PAH's EOM Particulate
Preservative required	Sealed, Level marked, Kept cool & handled with Care.

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana

Baghouse Inlet
Run #2

~~July~~ August 12, 1998
Page 1 of 5

I. Test Run Observations

R = Recommended
M = Mandatory

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
Date		M-Conb 429 M-429	M-315 M-315		
1. Train set up	filter ID	N/A	080798-04 ~~0728-05~~ DRB		
	filter weight	N/A	N/A		
	filter checked for holes	✓	✓		
	filter centered	✓	✓		
	nozzle clean	✓	✓		
	nozzle undamaged	✓	✓		
	nozzle diameter (in.)	.215	.215		
	probe liner clean	✓	✓		
	probe markings correct	✓	✓		
	probe heated along entire length	✓	✓		
	impingers charged	✓	✓		
	impingers iced	✓	✓		
	meter box leveled	✓	✓		
	pitot manometer zeroed	✓	✓		
	orifice manometer zeroed	✓	✓		
	filter box or holder at temp.	✓	✓		
	all ball joints lightly greased	N/A	✓		
	all openings capped	yes	yes		
2. Train leak check at nozzle: (<0.02 cfm @ 15 in. Hg initial. Intermediate and final at highest Vacuum during test run.)	initial (R) LC	.008	.006		
	initial (R) VAC	13"Hg	15"Hg		
	* intermediate (R) LC	.004			
	intermediate (R) VAC	13"Hg			
	intermediate (R) LC				
	intermediate (R) VAC				
	intermediate (R) LC				
	intermediate (R) VAC				
	final (M) LC	.005	.005		
	final (M) VAC	10"Hg	5"Hg		
3. Pitot lines leak check: (hold 3 in. H_2O on manometer for (15 sec.)	initial positive line (R)	5.6 ok	7.0 ok		
	initial negative line (R)	8.6 ok	3.5 ok		
	final positive line (M)	>3" ok	>3" ok		
	final negative line (R)	>3" ok	>3" ok		
	pitot tube undamaged	yes	yes		
M-3 bag initial leak check (M) Tedlar bag:	Should hold 2 to 4 in. H_2O pressure for 10 minutes or	yes	yes		
	zero flow meter reading on continuous evacuation or	yes	yes		
	Completely fill bag and let stand overnight—no deflation.	yes	yes		

* Filter changed out about 2 hrs into test.

R = Recommended
M = Mandatory

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
	Date	8-12-98	8-12-98		
		M-429	M-315		
4.	M-3 sampling train check: initial (M)	N/A	N/A		
	(should hold 10 in. vacuum for 1/2 min.) final (M)	N/A	N/A		
		N/A	N/A		
	Purge sample train with stack gas	yes	yes		
	Constant rate sampling 1 pm	yes	yes		
5.	Time test started	9:24A	9:24A		
	Time test ended	2:35	2:11		
6.	Dry gas meter volume: ( ) port initial	528.618	023.672		
	final				
	( ) port initial				
	final				
	( ) port initial				
	final				
	( ) port initial				
	final	617.680	147.673		
7.	Train operation during run — Nozzle changed during run — NOT ALLOWED	NO	NO		
	pitch and yaw of probe o.k.	yes	yes		
	nozzle not scraped on nipple	yes	yes		
	effective seal around probe	yes	yes		
	probe moved at proper time	yes	yes		
	probe heated	yes	yes		
	calculator constants or nomograph changed when TS and/or TM changes significantly	N/A	N/A		
	average time to set isokenetics after probe moved to next point	<10 sec	<10 sec		
	Average values: impinger temperature should be ≤ 70°F	yes	yes		
	Post filter gas streamer or Filter box temperature [✓ 250°F ± 25] ≤320°F, °F circle one	yes	yes		
	stack temperature	147°F	158°F		
	barometric P taken and value	29.8	29.8		
	was probe ever disconnected from filter holder while in stack?	NO	NO		
	was filter changed during run?	yes	NO		

R = Recommended M = Mandatory	Date 8-12-98 Test Run 1 M-429	Date 8-12-98 Test Run 2 M-315	Test Run 3	Test Run 4
Check on filter holder loosening of clamping device holder	yes	yes		
was silica gel changed during run?	NO	NO		
was any particulate lost?	NO	NO		
Accurate reading of: ΔP	.2467	.2964		
ΔH	.239	.291		
meter temperature	79°F	80°F		
stack temperature	147°F	158°F		
meter vacuum	<6" Hg	5.5" Hg		
* time per point	*	*		
impinger temperature	<70°F	<70°F		
filter box temperature	250±25	250±20		
*Minimum sample time of ✓ min met *Minimum sample volume of ✓ dscf collected	yes	yes		
8. Post test: - All openings sealed	yes	yes		
- recovery area clean sheltered	yes	yes		
- filter handled with gloves, forceps	yes	yes		
- petri dish sealed, labeled	yes	yes		
- any sample lost	NO	NO		
water measured mL / grad cyl. weighed gms	54.7	60.9		
- silica gel weighed, net gms	20.1	21.0		
- condition - color Impinges	Clear	clear		
% spent	✓	✓		
- probe cooled sufficiently	yes	yes		
- nozzle removed and brushed	yes	yes		
- probe brushed 6 times	yes	yes		
- nozzle brushes clean	yes	yes		
- wash bottles clean	yes	yes		
- acetone clean	yes	yes		
- M-8 15 minute purge	N/A	N/A		
- water/solution clean	yes	yes		
- blank taken: acetone, water, other Hexane, Methylene Chloride	yes	yes		
Probe brush and extension clean	yes	yes		
Sample container: Clean	yes	yes		
Capped	yes	yes		
Labeled	yes	yes		
Sealed	yes	yes		
Liquid level marked	yes	yes		

* Time varies with each Battery Coke push. Volume also varies from run to run, because of not knowing how many battery pushes will be Sampled.

R = Recommended
M = Mandatory

			Test Run 1	Test Run 2	Test Run 3	Test Run 4
		Date	8-12-98	8-12-98		
			M-429	M-315		
9.	Post test Orsat Analysis of integrated bag sample Orsat analyzer - Analyzer leak check (levels should not fall below cap. tubing and not more than 0.2 mL in burrette for 2 min.)	Initial (M)	yes	yes		
		Final (M)	yes	yes		
			—	—		
	Orsat samples:	Each bag analyzed 3 times	yes	yes		
		% CO_2 agrees within 0.2%	yes	yes		
		% O_2 agrees within 0.2%	yes	yes		
		% CO agrees within 0.2%	N/A	N/A		
	Analysis at end of test. Orsat analyzer checked against air (20.9 ± 0.3)		yes	yes		
	Orsat Analysis:	CO_2%	0.5	.05%		
		O_2%	20.0 ~~N/A~~	20.0%		
		CO%	N/A	N/A		
		$F_o = \frac{20.9 - \% O_2}{\% CO_2}$	N/A	N/A		
		Fuel	N/A	N/A		
		F_o range for fuel	N/A	N/A		
		Orsat analysis valid	yes	yes		
		Orsat solutions changed when calculated F_o exceeds fuel type range	N/A	N/A		
10.	All samples locked up		yes	yes		
	All sampling components clean and sealed		yes	yes		
1 *	All data sheets submitted to observer *		yes	yes		
	- Orsat		yes	yes		
	- Run isokenetic	Team/Observer				
	- Particulate recovery		yes	yes		
2 *	- Process data *		N/A	N/A		
	- Charts		N/A	N/A		
	- Calibration sheets		yes	yes		

1 * Sample & data custodian

2 * Process collected by RTI and will be submitted at later date.

J. **NOTES:** Care should be taken, when sampling for organic compounds, to follow stringent quality control guidelines to avoid contamination of the sample and sampling train. Take note of any occurences which could bias the sample in any manner.

Include: (1) General comments; (2) Changes to pretest agreement with justification; (3) Identify (manufacturer) and describe condition of sampling equipment; (4) any abnormal occurrences during test program. (Additional page(s) attached: Yes ____, No ✓.)

Dennis P Holzschuh
Signature of Observer

P.E.S.
Affiliation of Observer

8-12-98
Date

Baghouse Outlet
Run 2

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana
Methods Run M-Carb 429, M-315
NIOSH 5506

August 12, 1998
Date ~~July~~ , 1998
Page 1 of 2

Quality Control Check	Observation
Prior to Start of Tests	
Keep all cleaned glassware sealed until train assembly	Done
Assemble trains in dust free environment	Done
Visually inspect each train for proper assembly	Done
Level and zero manometer	Done
Calculate proper sampling nozzle size	Done
Visually inspect sampling nozzle for chips	Done
Visually inspect Type S Pitot tube	Done
Leak check each leg of Type S Pitot tube	Done
Leak check entire sampling train	Done
During Testing	
Read temperatures and differential pressures at each traverse point	Done
Sample data and calculations recorded on preformatted data sheets	yes
Unusual occurrences noted in test log	yes
Properly maintain the roll and pitch of axis of Type S Pitots and sampling nozzle	N/A
Leak check train before and after any component changes during test	done
Maintain the probe and filter temperature	yes
Maintain ice in ice water bath and maintain impinger exit temperature	yes
Calibration forms reviewed for completeness and accuracy	yes
Data sheets reviewed by PM daily during testing	yes

M-315 Meter Box NO 4
Y = .979
ΔH = 1.872
K-Factor = 2.13
Pitot # 5E
Thermocouple # T5B
Filter # 080798-23

M-Carb 429 Meter Box #2
Y = 1.02
ΔH 1.797
K Factor
Pitot # 5-B

Date ~~July~~ August 12, , 1998
Page 2 of 2

Quality Control Check	Observation
After Testing	
Visually inspect sampling nozzle	yes
Visually inspect Type S Pitot tube	yes
Leak check each leg of the Type S Pitot tube	yes
Leak check the entire sampling train	yes
Record observations if any	yes
Field Log	
Project name/ID and location	Bethlehem Steel Chesterton Ind
Sampling personnel (names/position)	Nick M-315 Steve Blaine M-429
Geological observations including map	Flat terrain approx 20' off ground
Sample run times and dates	9:29 AM – 7:35 PM 8-12-98
Sample descriptions	M-Carb 429, M315 NIOSh 5506
Description of QC samples	N/A
Deviations from QAPP	NO
Difficulties in sampling or unusual conditions	Long sampling period & Numerous flow chang
Sample Labels	yes
Sample ID	yes
Date and time of collection	8-12-98 9:29 AM – 7:35 PM
Lab technician initials	RK & RD
Analytical parameter	PAH's EOM Particulate
Preservative required	Sealed, Level marked, Kept cool & Handled with care.

Baghouse outlet
Run 2

~~July~~ August 12, 1998, 1998
Page 1 of 5

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana

			Test Run 1	Test Run 2	Test Run 3	Test Run 4
I. Test Run Observations R = Recommended M = Mandatory		Date	M-Carb 429	M-315		
1. Train set up	filter ID		N/A	08079823		
	filter weight		N/A	N/A		
	filter checked for holes		✓	✓		
	filter centered		✓	✓		
	nozzle clean		✓	✓		
	nozzle undamaged		✓ .254	DH .254 DH		
	nozzle diameter (in.)		.235	.235		
	probe liner clean		✓	✓		
	probe markings correct		✓	✓		
	probe heated along entire length		✓	✓		
	impingers charged		✓	✓		
	impingers iced		✓	✓		
	meter box leveled		✓	✓		
	pitot manometer zeroed		✓	✓		
	orifice manometer zeroed		✓	✓		
	filter box or holder at temp.		✓	✓		
	all ball joints lightly greased		N/A	N/A		
	all openings capped		yes	yes		
2. Train leak check at nozzle: (<0.02 cfm @ 15 in. Hg initial. Intermediate and final at highest Vacuum during test run.)	initial (R)	LC	.007	.006		
		VAC	16" Hg	15" Hg		
	intermediate (R)	LC				
		VAC				
	intermediate (R)	LC				
		VAC				
	intermediate (R)	LC				
		VAC				
	final (M)	LC	.001	.007		
		VAC	8" Hg	10" Hg		
3. Pitot lines leak check: (hold 3 in. H_2O) on manometer for (15 sec.)	initial positive line (R)		>3 ok	>3" ok		
	negative line (R)		>3" ok	>3" ok		
	final positive line (M)		>3" ok	>3" ok		
	negative line (R)		>3" ok	>3" ok		
	pitot tube undamaged		yes	yes		
M-3 bag initial leak check (M) Tedlar bag:	Should hold 2 to 4 in. H_2O pressure for 10 minutes or		yes	yes		
	zero flow meter reading on continuous evacuation or		yes	yes		
	Completely fill bag and let stand overnight—no deflation.		yes	yes		

R = Recommended
M = Mandatory

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
	Date	8-12-98	8-12-98		
		M-429	M-315		
4.	M-3 sampling train check: initial (M)	N/A	N/A		
	(should hold 10 in. vacuum for 1/2 min.) final (M)	N/A	N/A		
		N/A	N/A		
	Purge sample train with stack gas	yes	yes		
	Constant rate sampling 1 pm	yes	yes		
5.	Time test started	9:24AM	9:24AM		
	Time test ended	7:40PM	7:40PM		
6.	Dry gas meter volume: ( ) port initial	969.901	895.517		
	final				
	( ) port initial				
	final				
	( ) port initial				
	final				
	( ) port initial				
	final	1172.926	1088.864		
7.	Train operation during run — Nozzle changed during run – NOT ALLOWED	NO	NO		
	pitch and yaw of probe o.k.	N/A	N/A		
	nozzle not scraped on nipple	yes	yes		
	effective seal around probe	yes	yes		
	probe moved at proper time	yes	yes		
	probe heated	yes	yes		
	calculator constants or nomograph changed when TS and/or TM changes significantly	N/A	N/A		
	average time to set isokenetics after probe moved to next point	<10 Sec	<10 Sec		
	Average values: impinger temperature should be ≤ 70°F	yes	yes		
	Post filter gas streamer or Filter box temperature [✓ 250°F + 25] ≤320°F, °F circle one	yes	yes		
	stack temperature	137°F	135°F		
	barometric P taken and value	29.8	29.8		
	was probe ever disconnected from filter holder while in stack?	NO	NO		
	was filter changed during run?	NO	NO		

R = Recommended
M = Mandatory

Date	8-12-98	8-12-98		
	Test Run 1 M429	Test Run 2 M-315	Test Run 3	Test Run 4
Check on filter holder loosening of clamping device holder	yes	yes		
was silica gel changed during run?	NO	NO		
was any particulate lost?	NO	NO		
Accurate reading of: ΔP	.4607	.4106		
ΔH	.693	.586		
meter temperature	80°F	77°F		
stack temperature	137°F	135°F		
meter vacuum	<7"Hg	<5"Hg		
* time per point	✓	✓		
impinger temperature	<70°F	<70°F		
filter box temperature	250±25	250±25		
*Minimum sample time of ✓ min met *Minimum sample volume of ✓ dscf collected	yes	yes		
8. Post test: - All openings sealed	yes	yes		
- recovery area clean sheltered	yes	yes		
- filter handled with gloves, forceps	yes	yes		
- petri dish sealed, labeled	yes	yes		
- any sample lost	yes	yes		
water measured mL grad cyl. / gms weighed	118.0	87.0		
- silica gel weighed, net gms	40.8	35.5		
- condition - color impingers	clear	clear		
% spent	✓	✓		
- probe cooled sufficiently	yes	yes		
- nozzle removed and brushed	yes	yes		
- probe brushed 6 times	yes	yes		
- nozzle brushes clean	yes	yes		
- wash bottles clean	yes	yes		
- acetone clean	yes	yes		
- M-8 15 minute purge	N/A	N/A		
- water/solution clean	yes	yes		
hexane, Methylene Chloride - blank taken: acetone, water, other	yes	yes		
Probe brush and extension clean,	yes	yes		
Sample container: Clean	yes	yes		
Capped	yes	yes		
Labeled	yes	yes		
Sealed	yes	yes		
Liquid level marked	yes	yes		

* Varies with Battery Coke pushes.

August 12

		Date	8-1298	8-12-98		
	R = Recommended M = Mandatory		Test Run 1 M-429	Test Run 2 M-315	Test Run 3	Test Run 4
9.	Post test Orsat Analysis of integrated bag sample Orsat analyzer - Analyzer leak check (levels should not fall below cap. tubing and not more than 0.2 mL in burrette for 2 min.)	Initial (M)	Yes	yes		
		Final (M)	Yes	yes		
	Orsat samples:	Each bag analyzed 3 times	yes	yes		
		% CO_2 agrees within 0.2%	Yes	yes		
		% O_2 agrees within 0.2%	Yes	yes		
		% CO agrees within 0.2%	N/A	N/A		
	Analysis at end of test. Orsat analyzer checked against air (20.9 ± 0.3)		yes	yes		
	Orsat Analysis:	CO_2%	.05%	.05%		
		O_2%	20.1%	20.0%		
		CO%	N A	N/A		
		Fo = $\frac{20.9 - \% O_2}{\% CO_2}$	N/A	N/A		
		Fuel	N/A	N/A		
		F_o range for fuel	N/A	N/A		
		Orsat analysis valid	N/A	N/A		
		Orsat solutions changed when calculated F_o exceeds fuel type range	N/A	N/A		
10.	All samples locked up		Yes	Yes		
	All sampling components clean and sealed		Yes	Yes		
1*	All data sheets submitted to observer		Yes	Yes		
	- Orsat		Yes	yes		
	- Run isokenetic	Team/Observer	Yes	yes		
	- Particulate recovery		yes	yes		
2*	- Process data		N/A	N/A		
	- Charts		N/A	N/A		
	- Calibration sheets		yes	yes		

1* Sample, data custodian

2* Process data collected by RTI, and will be Submitted at later date for final report.

J. NOTES: Care should be taken, when sampling for organic compounds, to follow stringent quality control guidelines to avoid contamination of the sample and sampling train. Take note of any occurences which could bias the sample in any manner.

Include: (1) General comments; (2) Changes to pretest agreement with justification; (3) Identify (manufacturer) and describe condition of sampling equipment; (4) any abnormal occurrences during test program. (Additional page(s) attached: Yes ____, No ✓.)

Dennis P Holzschuh	P.E.S.	8-12-98
Signature of Observer	Affiliation of Observer	Date

Baghouse Inlet
Run 1

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana
Methods run M-315, Carb 429
NIOSH 5506

Date August 11, 1998 ~~July~~ , 1998
Page 1 of 2

Quality Control Check	**Observation**
Prior to Start of Tests	
Keep all cleaned glassware sealed until train assembly	Done
Assemble trains in dust free environment	Done
Visually inspect each train for proper assembly	Done
Level and zero manometer	Done
Calculate proper sampling nozzle size	Done
Visually inspect sampling nozzle for chips	Done
Visually inspect Type S Pitot tube	Done
Leak check each leg of Type S Pitot tube	Done
Leak check entire sampling train	Done
During Testing	
Read temperatures and differential pressures at each traverse point	done
Sample data and calculations recorded on preformatted data sheets	yes
Unusual occurrences noted in test log	yes
Properly maintain the roll and pitch of axis of Type S Pitots and sampling nozzle	yes
Leak check train before and after any component changes during test	done
Maintain the probe and filter temperature	done
Maintain ice in ice water bath and maintain impinger exit temperature	done
Calibration forms reviewed for completeness and accuracy	yes
Data sheets reviewed by ~~BM~~ daily during testing	yes

M-Carb 429 Meter Box # 6
Y = 1.005
ΔH = 1.778

M-315 Meter Box # 1
Y = 1.012
ΔH 1.954

Date ~~July~~ August 11, 1998
Page 2 of 2

Quality Control Check	**Observation**
After Testing	
Visually inspect sampling nozzle	yes
Visually inspect Type S Pitot tube	yes
Leak check each leg of the Type S Pitot tube	yes
Leak check the entire sampling train	yes
Record observations if any	yes
Field Log	
Project name/ID and location	Bethlehem Steel, Chesterton, Ind.
Sampling personnel (names/position)	M-Carb 429 Dennis Holeschuh · M-315 Gary Gay
Geological observations including map	Flat surface about 30 ft over roadway
Sample run times and dates	8-11-98 10:34AM - 8:20PM
Sample descriptions	M-Carb-429, M-315 NIOSH 550
Description of QC samples	N/A
Deviations from QAPP	✓
* **Difficulties in sampling or unusual conditions**	Ten hour sample period - Sampling 9' duct on 2-
Sample Labels	yes
Sample ID	yes
Date and time of collection	8-11-98 10:34AM - 8:20PM
Lab technician initials	R.K & R D
Analytical parameter	PAH's, EOM, Particulate,
Preservative required	Sealed, Marked well, Kept cool, & handled with

Sampling with a 10' probe on 9' duct at two 45°L with heated teflon flex line. This made for a tedious & time consumming port change

~~July~~ --, 1998

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana

Baghouse Inlet
RUN # 2

Page 1 of 5

I. Test Run Observations R = Recommended M = Mandatory			Test Run 1	Test Run 2	Test Run 3	Test Run 4
		Date	M-Carb 429	M-315		
1. Train set up	filter ID		N/A	0228-05		
	filter weight		N/A	✓		
	filter checked for holes		✓	✓		
	filter centered		✓	✓		
	nozzle clean		✓	✓		
	nozzle undamaged		✓	✓		
	nozzle diameter (in.)		.217	.215		
	probe liner clean		✓	✓		
	probe markings correct		✓			
	probe heated along entire length		✓	✓		
	impingers charged		✓	✓		
	impingers iced		✓	✓		
	meter box leveled		✓	✓		
	pitot manometer zeroed		✓	✓		
	orifice manometer zeroed		✓	✓		
	filter box or holder at temp.		✓	✓		
	all ball joints lightly greased		N/A	N/A		
	all openings capped		✓	✓		
2. Train leak check at nozzle: (≤0.02 cfm @ 15 in. Hg initial. Intermediate and final at highest Vacuum during test run.)	initial (R)	LC	.013	.008		
		VAC	15"Hg	15"Hg		
	intermediate (R)	LC				
		VAC				
	intermediate (R)	LC				
		VAC				
	intermediate (R)	LC				
		VAC				
	final (M)	LC	.012	.001		
		VAC	15"Hg	5"Hg		
3. Pitot lines leak check: (hold 3 in. H_2O) on manometer for (15 sec.)	initial	positive line (R)	6.4 ✓	5.6 ✓		
		negative line (R)	6.9 ✓	5.4 ✓		
	final	positive line (M)	>3" ok	>3" ok		
		negative line (R)	>3" ok	>3" ok		
		pitot tube undamaged	yes	yes		
M-3 bag initial leak check (M) Tedlar bag:	Should hold 2 to 4 in. H_2O pressure for 10 minutes or		yes	yes		
	zero flow meter reading on continuous evacuation or		yes	yes		
	Completely fill bag and let stand overnight—no deflation.		yes	yes		

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
R = Recommended M = Mandatory	Date	8-11-98	8-11-98		
		M-429	M-315		
4. M-3 sampling train check:	initial (M)	N/A	N/A		
(should hold 10 in. vacuum for 1/2 min.)	final (M)	N/A	N/A		
		N/A	N/A		
Purge sample train with stack gas		yes	yes		
Constant rate sampling	1 pm	yes	yes		
5. Time test started		10.34	10.34		
Time test ended		8:20 PM	8:20 PM		
6. Dry gas meter volume:	( ) port initial	435.165	895.256		
	final				
	( ) port initial				
	final				
	( ) port initial				
	final				
	( ) port initial				
	final	527.330	027.009		
7. Train operation during run	Nozzle changed during run – NOT ALLOWED	No	No		
pitch and yaw of probe o.k.		yes	yes		
nozzle not scraped on nipple		yes	yes		
effective seal around probe		yes	yes		
probe moved at proper time		yes	yes		
probe heated		yes	yes		
calculator constants or nomograph changed when TS and/or TM changes significantly		N/A ~~yes~~	N/A		
average time to set isokenetics after probe moved to next point		<10 sec	<10 sec		
Average values: impinger temperature should be ≤ 70°F		yes	yes		
Post filter gas streamer or Filter box temperature [✓ 250°F + 25], ≤320°F, °F circle one		yes	yes		
stack temperature		142°F	153°F		
barometric P taken and value		29.7	29.7		
was probe ever disconnected from filter holder while in stack?		No	No		
was filter changed during run?		No	No		

R = Recommended
M = Mandatory

	Test Run 1	Test Run 2	Test Run 3	Test Run 4
Date	8-11-98	8-11-98		
	M-42.9	M 31.5		
Check on filter holder loosening of clamping device holder	yes	yes		
was silica gel changed during run?	no	no		
was any particulate lost?	no	no		
Accurate reading of: ΔP Average Sq Rt in H_2O	.2716	.2880		
ΔH Avg Meter Orifice Pressure	.26	.29		
meter temperature	76°F	80°F		
stack temperature	142°F	153°F		
meter vacuum	<12"	<6"		
time per point	*	*		
impinger temperature	250±25	250±25		
filter box temperature	<70°F	<70°F		
* Minimum sample time of ✓ min met Minimum sample volume of ✓ dscf collected	yes	yes		
8. Post test: - All openings sealed	yes	yes		
- recovery area clean sheltered	yes	yes		
- filter handled with gloves, forceps	yes	yes		
- petri dish sealed, labeled	yes	yes		
- any sample lost	no	no		
water measured mL (grad cyl.) gms (weighed)	65.3	66.6		
- silica gel weighed, net gms	21.5	22.5		
- condition - color Impingers	clear	clear		
% spent	✓	✓		
- probe cooled sufficiently	yes	yes		
- nozzle removed and brushed	yes	yes		
- probe brushed 6 times	yes	yes		
- nozzle brushes clean	yes	yes		
- wash bottles clean	yes	yes		
- acetone clean	yes	yes		
- M-8 15 minute purge	N/A	N/A		
- water/solution clean	yes	yes		
Hexane, Methylene Chloride - blank taken: acetone, water, other	yes	yes		
Probe brush and extension clean,	yes	yes		
Sample container: Clean	yes	yes		
Capped	yes	yes		
Labeled	yes	yes		
Sealed	yes	yes		
Liquid level marked	yes	yes		

* Time varies because of the Battery Coke pushes.

R = Recommended
M = Mandatory

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
	Date	8-11-98	8-11-98		
		M-429	M-315		
9.	Post test Orsat Analysis of integrated bag sample Orsat analyzer - Analyzer leak check (levels should not fall below cap. tubing and not more than 0.2 mL in burrette for 2 min.) Initial (M)	yes	yes		
	Final (M)	yes	yes		
	Orsat samples: Each bag analyzed 3 times	yes	yes		
	% CO_2 agrees within 0.2%	yes	yes		
	% O_2 agrees within 0.2%	yes	yes		
	% CO agrees within 0.2%	N/A	N/A		
	Analysis at end of test. Orsat analyzer checked against air (20.9 ± 0.3)	yes	yes		
	Orsat Analysis: CO_2%	.05%	.05%		
	O_2%	20.05%	20.05%		
	CO%	N/A	N/A		
	$F_o = \frac{20.9 - \%\ O_2}{\%\ CO_2}$	N/A	N/A		
	Fuel	N/A	N/A		
	F_o range for fuel	N/A	N/A		
	Orsat analysis valid	yes	yes		
	Orsat solutions changed when calculated F_o exceeds fuel type range	N/A	N/A		
10.	All samples locked up	yes	yes		
	All sampling components clean and sealed	yes	yes		
1 *	All data sheets submitted to observer	yes	yes		
	- Orsat	yes	yes		
	- Run isokenetic Team/Observer	yes	yes		
	- Particulate recovery	yes	yes		
2 *	- Process data	N/A	N/A		
	- Charts	N/A	N/A		
	- Calibration sheets	yes	yes		

1 * Sample, Data, Custodian

2 * Process data collected by another Contractor (ATI) & will be submitted at later date.

J. **NOTES:** Care should be taken, when sampling for organic compounds, to follow stringent quality control guidelines to avoid contamination of the sample and sampling train. Take note of any occurences which could bias the sample in any manner.

Include: (1) General comments; (2) Changes to pretest agreement with justification; (3) Identify (manufacturer) and describe condition of sampling equipment; (4) any abnormal occurrences during test program. (Additional page(s) attached: Yes ____, No ✓.) *

* Note: The only abnormalities is in the sampling procedure, where the flow varies with each battery push. However this is something we were aware of and was addressed. So for this test it is considered normal.

Dennis P Holzschuh
Signature of Observer

PES
Affiliation of Observer

8-11-98
Date

Baghouse Outlet
Run 1

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana
Methods Run M-315, Carb 429
NIOSH 5506

Date August 11, 1998 ~~July~~ , 1998
Page 1 of 2

Quality Control Check	**Observation**
Prior to Start of Tests	
Keep all cleaned glassware sealed until train assembly	done
Assemble trains in dust free environment	done
Visually inspect each train for proper assembly	done
Level and zero manometer	done
Calculate proper sampling nozzle size	done (M-315, .22) (M-Carb 429, .22)
Visually inspect sampling nozzle for chips	done
Visually inspect Type S Pitot tube	done
Leak check each leg of Type S Pitot tube	done
Leak check entire sampling train	done
During Testing	
Read temperatures and differential pressures at each traverse point	done
Sample data and calculations recorded on preformatted data sheets	yes
Unusual occurrences noted in test log	yes
Properly maintain the roll and pitch of axis of Type S Pitots and sampling nozzle	N/A
Leak check train before and after any component changes during test	done
Maintain the probe and filter temperature	done
Maintain ice in ice water bath and maintain impinger exit temperature	done
Calibration forms reviewed for completeness and accuracy	yes
Data sheets reviewed by PM daily during testing	yes

M-315 Meter Box No 4
y = .979
ΔH = 1.872
K-Factor = 2.13
Pitot # 5E
Thermocouple # T5B
Filter # 080798-06

M-Carb 429 Meter Box #2
y = 1.02
ΔH = 1.797
K-Factor = 2.31
Pitot # 5-B

Date ~~July~~ August 11, 1998
Page 2 of 2

Quality Control Check	Observation
After Testing	~~yes~~
Visually inspect sampling nozzle	yes
Visually inspect Type S Pitot tube	yes
Leak check each leg of the Type S Pitot tube	yes
Leak check the entire sampling train	yes
Record observations if any	yes
Field Log	
Project name/ID and location	Bethlehem Steel Chesterton, Ind.
Sampling personnel (names/position)	(M-315) Nick Nielsen. (M Carb 429) Steve Blaine
Geological observations including map	Flat surface & working at 60-70 ft up
Sample run times and dates	8-11-98
Sample descriptions	M-315, M-Carb 429 N,05A550
Description of QC samples	N/A
Deviations from QAPP	NO
Difficulties in sampling or unusual conditions	Sampling time planned 10 hours
Sample Labels	yes
Sample ID	yes
Date and time of collection	August 11, 1998 10:32AM - 7:58PM
Lab technician initials	RK & RD
Analytical parameter	PAH's, EOM, Particulate
Preservative required	Sealed, Labels Marked, Kept cool & handled with care.

~~July~~ , 1998

Bethlehem Steel Corporation
Coke Oven Emission Test
Chesterton, Indiana

Baghouse Outlet
Run 1

Page 1 of 5

					Date	M-Carb 429	M-315		
I.	**Test Run Observations** R = Recommended M = Mandatory					**Test Run 1**	**Test Run 2**	**Test Run 3**	**Test Run 4**
1.	**Train set up**	**filter ID**			N/A	~~[illegible]~~ ~~N/A~~ DPH	~~N/A~~ ~~08[illegible]~~ DPH	086798-06	
		filter weight				N/A	N/A		
		filter checked for holes				✓	✓		
		filter centered				✓	✓		
		nozzle clean				✓	✓		
		nozzle undamaged				✓	✓		
		nozzle diameter (in.)				.22	.22		
		probe liner clean				✓	✓		
		probe markings correct				✓	✓		
		probe heated along entire length				✓	✓		
		impingers charged				✓	✓		
		impingers iced				✓	✓		
		meter box leveled				✓	✓		
		pitot manometer zeroed				✓	✓		
		orifice manometer zeroed				✓	✓		
		filter box or holder at temp.				✓	✓		
		all ball joints lightly greased				N/A	N/A		
		all openings capped				yes	yes		
2.	**Train leak check at nozzle: (≤0.02 cfm @ 15 in. Hg initial. Intermediate and final at highest Vacuum during test run.)**	**initial (R)**		**LC**		.008	.003		
				VAC		15"Hg	15"Hg		
		intermediate (R)		**LC**					
				VAC					
		intermediate (R)		**LC**					
				VAC					
		intermediate (R)		**LC**					
				VAC					
		final (M)		**LC**		.012	.003		
				VAC		7"Hg	8"Hg		
3.	**Pitot lines leak check: (hold 3 in. H_2O) on manometer for (15 sec.)**	**initial**	**positive line (R)**			>3 ok	>3 ok		
			negative line (R)			>3 ok	>3 ok		
		final	**positive line (M)**			>3" ok	>3" ok		
			negative line (R)			>3" ok	>3" ok		
			pitot tube undamaged			yes	yes		
	M-3 bag initial leak check (M)								
	Tedlar bag:	**Should hold 2 to 4 in. H_2O pressure for 10 minutes or**				yes	yes		
		zero flow meter reading on continuous evacuation or				yes	yes		
		Completely fill bag and let stand overnight—no deflation.				yes	yes		

	Date	M-Carb 429	M-315 8-11-98		
R = Recommended M = Mandatory		Test Run 1 M-429	Test Run 2 M-315	Test Run 3	Test Run 4
4. M-3 sampling train check:	initial (M)	N/A	N/A		
(should hold 10 in. vacuum	final (M)	N/A	N/A		
for 1/2 min.)		N/A	N/A		
Purge sample train with stack gas		yes	yes		
Constant rate sampling	1 pm	yes	yes		
5. Time test started		18:38	18:38		
Time test ended		10:34	10:34		
6. Dry gas meter volume:	( ) port initial	816.865	756.453		
	final				
	( ) port initial				
	final				
	( ) port initial				
	final				
	( ) port initial				
	final	969.483	888.929		
7. Train operation during run	Nozzle changed during run - NOT ALLOWED	NO	NO		
	pitch and yaw of probe o.k.	yes	yes		
	nozzle not scraped on nipple	yes	yes		
	effective seal around probe	✓	✓		
	probe moved at proper time	✓	✓		
	probe heated	✓	✓		
	calculator constants or nomograph changed when TS and/or TM changes significantly	yes	yes		
	average time to set isokenetics after probe moved to next point	<10 sec	<10 sec		
	Average values: impinger temperature should be ≤ 70°F	yes	yes		
	Post filter gas streamer or Filter box temperature [✓ 250°F + 25], ≤320°F, °F circle one	yes	yes		
	stack temperature	132°F	134°F		
	barometric P taken and value	29.7	29.7		
	was probe ever disconnected from filter holder while in stack?	NO	NO		
	was filter changed during run?	NO	NO		

R = Recommended
M = Mandatory

	Test Run 1	Test Run 2	Test Run 3	Test Run 4
Date	8-11-98	8-11-98		
	M-429	M-315		
Check on filter holder loosening of clamping device holder	yes	yes		
was silica gel changed during run?	NO	NO		
was any particulate lost?	NO	NO		
Accurate reading of: ΔP Avg Sq Rt in H_2O	.5851	.4220		
ΔH Avg. Meter Orifice Pressure	.53	.50		
meter temperature	79°F	72°F		
stack temperature	132°F	134°F		
meter vacuum	<7"Hg	5.5"Hg		
* time per point	*	*		
impinger temperature	<70°F	<70°F		
filter box temperature	250±25	250±25		
Minimum sample time of ______ min met Minimum sample volume of ______ dscf collected	ok	ok		
8. Post test: - All openings sealed	yes	yes		
- recovery area clean sheltered	yes	yes		
- filter handled with gloves, forceps	yes	yes		
- petri dish sealed, labeled	yes	yes		
- any sample lost	NO	NO		
water measured (grad cyl. mL / weighed gms)	93.8	49.6		
- silica gel weighed, net gms	35.7	23.0		
- condition - color Impingers	Clear	clear		
% spent	✓	✓		
- probe cooled sufficiently	yes	yes		
- nozzle removed and brushed	yes	yes		
- probe brushed 6 times	yes	yes		
- nozzle brushes clean	yes	yes		
- wash bottles clean	yes	yes		
- acetone clean	yes	yes		
- M-8 15 minute purge	N/A	N/A		
- water/solution clean	yes	yes		
- blank taken: acetone, water, other hexane, Methylene Chloride	yes	yes		
Probe brush and extension clean	yes	yes		
Sample container: Clean	yes	yes		
Capped	yes	yes		
Labeled	yes	yes		
Sealed	yes	yes		
Liquid level marked	yes	yes		

* Time varied with Coke Battery pushes

R = Recommended
M = Mandatory

		Test Run 1	Test Run 2	Test Run 3	Test Run 4
	Date	8-11-98	8-11-98		
		M-429	M-315		
9. Post test Orsat Analysis of integrated bag sample Orsat analyzer - Analyzer leak check (levels should not fall below cap. tubing and not more than 0.2 mL in burrette for 2 min.)	Initial (M)	yes	yes		
	Final (M)	yes	yes		
		—	—		
Orsat samples:	Each bag analyzed 3 times	yes	yes		
	% CO_2 agrees within 0.2%	yes	yes		
	% O_2 agrees within 0.2%	yes	yes		
	% CO agrees within 0.2%	N/A	N/A		
Analysis at end of test. Orsat analyzer checked against air (20.9 ± 0.3)		yes	yes		
Orsat Analysis:	CO_2%	.45%	.45%		
	O_2%	20.55%	20.55%		
	CO%	N/A	N/A		
	$F_o = \frac{20.9 - \%\ O_2}{\%\ CO_2}$	N/A	N/A		
	Fuel	N/A	N/A		
	F_o range for fuel	N/A	N/A		
	Orsat analysis valid	yes	yes		
	Orsat solutions changed when calculated F_o exceeds fuel type range	N/A	N/A		
10.	All samples locked up	yes	yes		
	All sampling components clean and sealed	yes	yes		
1 *	All data sheets submitted to observer	yes	yes		
	- Orsat	Yes	yes		
	- Run isokenetic Team/Observer	yes	yes		
	- Particulate recovery	yes	yes		
2*	- Process data	N/A	N/A		
	- Charts	N/A	N/A		
	- Calibration sheets	yes	yes		

1 * Sample Custodian

2 * Process data collected by (RTI) & will be submitt
@ Later date for Final Report.

J. **NOTES:** Care should be taken, when sampling for organic compounds, to follow stringent quality control guidelines to avoid contamination of the sample and sampling train. Take note of any occurences which could bias the sample in any manner.

Include: (1) General comments; (2) Changes to pretest agreement with justification; (3) Identify (manufacturer) and describe condition of sampling equipment; (4) any abnormal occurrences during test program. (Additional page(s) attached: Yes ____, No ✓.)

Dennis P Holzschuh
Signature of Observer

P E S
Affiliation of Observer

8-11-98
Date

APPENDIX F

PARTICIPANTS

PROJECT PARTICIPANTS

Affiliation	Name	Responsibility
USEPA		
EMC	John C. Bosch, Jr.	Work Assignment Manager
ESD	Alfred E. Vervaert	Group Leader
	Lula H. Melton	Process Monitor and Observer
PES	John T. Chehaske	Program Manager
	Franklin Meadows	Project Manager
	Daniel F. Scheffel	Field Team Leader
	Dennis P. Holzschuh	QA Coordinator
	Ron Kolde	Sample Recovery
	Dennis P. Becvar	Laboratory Audit
	Dennis D. Holzschuh	Field Team Member
	Troy A. Abernathy	Field Team Member
	Gary M. Gay	Field Team Member
	Jason T. Centers	Field Team Member
	Steven B. Blaine	Field Team Member
	Nicky P. Nielsen	Field Team Member
DEECO	Richard Durham	Sample Recovery
	Marc Hamilton	QAPP
ERG	Joan T. Bursey	PM/MCEM Analysis
FAL	William H. Wadlin	Metals Analysis
Quanterra	Robert Weidenfeld	PAH Analysis
LabCorp		NIOSH PAH Analysis

PROJECT PARTICIPANTS (Concluded)

Affiliation	Name	Responsibility
RTI	Marvin Branscome	EPA/ESD Contractor
	Sandy George	EPA/ESD Contractor (Observer)
	Stacy Molinich	EPA/ESD Contractor (Observer)
Bethlehem Steel	Thomas W. Easterly	Superintendent, Environmental Services Dept.
	Larry Mayton	
	Rich Guerra	

APPENDIX G

SAMPLING AND ANALYTICAL PROCEDURES

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 m^2 (113 $in.^2$) in 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:

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

Eq. 1-1

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 2.5.

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 and 24 in.); and (3) nine, for rectangular stacks with equivalent diameters between 0.30 and 0.61 meter (12 and 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 distances 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.

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 used 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 examples, 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 the approval of the Administrator.

In addition, for stacks having diameters greater than 0.61 m (24 in.), no traverse points shall be 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.

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.

2.3.2 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 4 x 3 "minimum number of points" matrix were expanded to 36 points, the final matrix could be 9 x 4 or 12 x 3, and would not necessarily have to be 6 x 6. 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. 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 ±90° 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 α values 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 2.5 may be used to determine the rotation angles in lieu of the procedure described above.

2.5 Alternative Measurement Site Selection Procedure. This alternative applies to sources where measurement locations are less than 2 equivalent or duct diameters downstream or less than one-half 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 the 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, a 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, or other differential pressure gauges (e.g., magnehelic gauges) that meet the specifications described in Method 2, Section 2.2.

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 of three points as specified in Method 2, Section 2.2.

2.5.2 Traverse Points. Use a minimum of 40 traverse points for circular ducts and 42 points for rectangular ducts for the gas flow angle determinations. Follow Section 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 locations for sampling and velocity measurements.

2.5.3 Measurement Procedure.

2.5.3.1 Prepare the directional probe and differential pressure gauges 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 7.6 cm (3 in.) H_2O 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 manometers. Since the manometer level and zero may drift 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, and rotate until zero deflection is indicated for the 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" of the pressure lines and the impact openings prior to measurements of each traverse point.

A post-test check as described in Section 2.5.3.1 is required. If the criteria for a leak-free system are not met, repair the equipment, and repeat the flow angle measurements.

2.5.4 Calculate the resultant angle at each 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 acquired data. Round the values after the final calculations.

2.5.4.1 Calculate the resultant angle at each traverse point:

$$R_i = \text{arc cosine}[(\text{cosine}Y_i)(\text{cosine}P_i)]$$

Eq. 1-2

Where:

R_i = resultant angle at traverse point i, degree.
Y_i = yaw angle at traverse point i, degree.
P_i = pitch angle at traverse point i, degree.

2.5.4.2 Calculate the average resultant for the measurements:

$$\overline{R} = \frac{\Sigma R_i}{n}$$

Eq. 1-3

Where:

R_{avg} = average resultant angle, degree.
n = total number of traverse points.

2.5.4.3 Calculate the standard deviations:

$$S_d = \sqrt{\frac{\sum_{i=1}^{n} (R_i - \bar{R})^2}{(n-1)}}$$

Eq. 1-4

Where:

S_d = standard deviation, degree.

2.5.5 The measurement location is acceptable if $R_{avg} \le 20°$ and $S_d \le 10°$.

2.5.6 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).

2.5.6.1 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.

2.5.6.2 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 ±2° of 0°. Straightening vanes should be installed, if necessary, to meet this criterion.

2.5.6.3 Pitch Angle Calibration. Perform a calibration traverse according to the manufacturer's recommended protocol in 5° increments for angles from -60° to +60° 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 datavalues 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°.

2.5.6.4 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 variations in the pitot markings used to indicate pitot head positions.

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

Number of traverse points	Matrix layout
9	3x3
12	4x3
16	4x4
20	5x4
25	5x5
30	6x5
36	6x6
42	7x6
49	7x7

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	14.6	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	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3		75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5
4		93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9
5			85.4	67.7	34.2	25.0	20.1	16.9	14.6	112.9	11.6	10.5
6			95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2
7				89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1
8				96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4
9					91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0
10					97.4	88.2	79.9	71.7	61.8	38.8	31.5	27.2
11						93.3	85.4	78.0	70.4	61.2	39.3	32.3
12						97.9	90.1	83.1	76.4	69.4	60.7	39.8
13							94.3	87.5	81.2	75.0	68.5	60.2

14							98.2	91.5	85.4	79.6	73.8	67.7
15								95.1	89.1	83.5	78.2	72.8
16								98.4	92.5	87.1	82.0	77.0
17									95.6	90.3	85.4	80.6
18									98.6	93.3	88.4	83.9
19										96.1	91.3	86.8
20										98.7	94.0	89.5
21											96.5	92.1
22											98.9	94.5
23												96.8
24												98.9

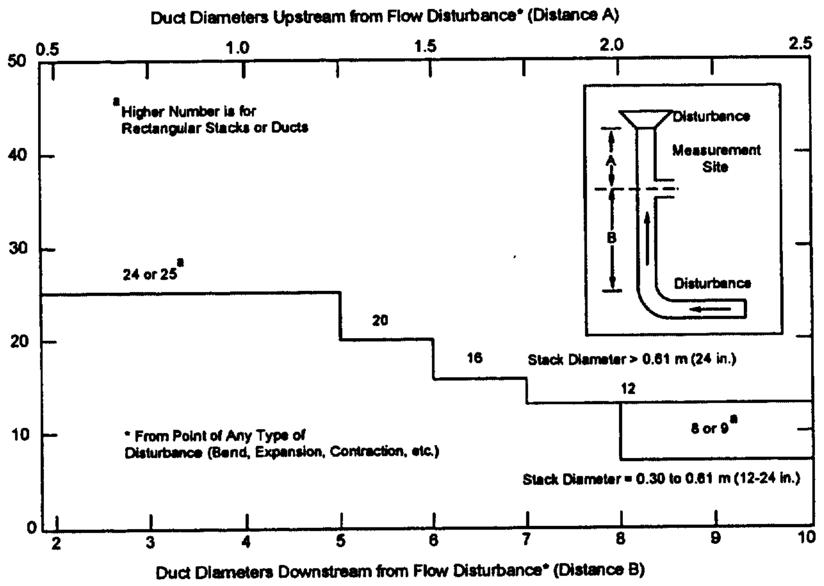

Figure 1-1. Minimum number of traverse points for particulate traverses.

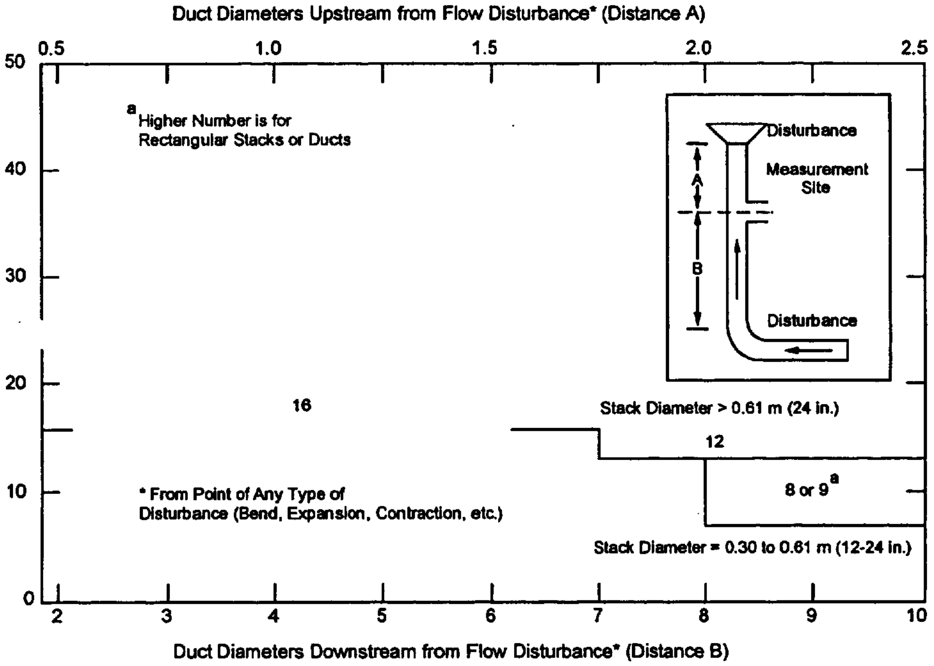

Figure 1-2. Minimum number of traverse points for velocity (nonparticulate) traverses.

Traverse Point	Distance % of diameter
1	4.4
2	14.7
3	29.5
4	70.5
5	85.3
6	95.6

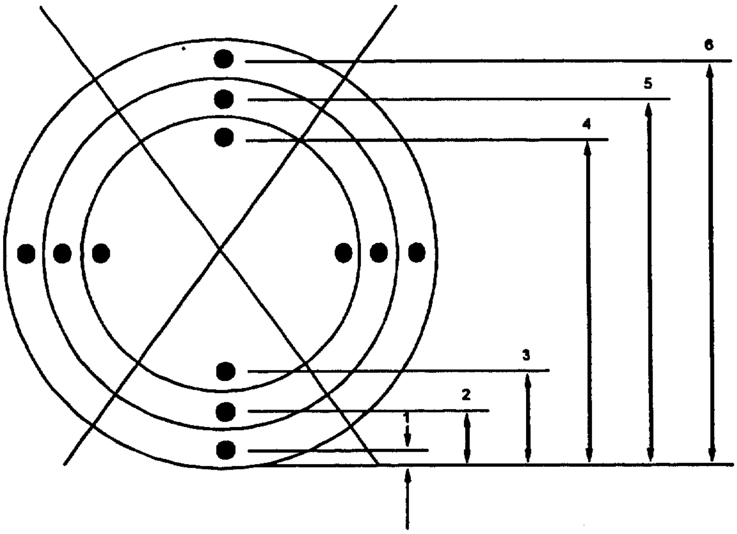

Figure 1-3. Example showing circular stack cross section divided into 12 equal areas, with location of traverse points indicated.

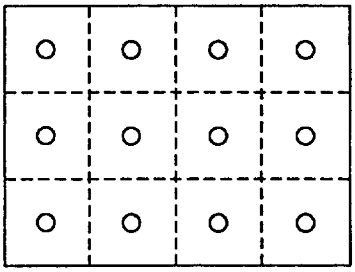

Figure 1-4. Example showing rectangular stack cross section divided into 12 equal areas, with a traverse point at centroid of each area.

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 (Stausscheibe 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 that 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. Pitot tube made of metal tubing (e.g., stainless steel) as shown in Figure 2-1. It is recommended that the external tubing diameter (dimension D_t, Figure 2-2b) be between 0.48 and 0.95 cm (3/16 and 3/8 inch). There shall be an equal distance from the base of each leg of the pitot tube to its face-opening plane (dimensions P_A and P_B, 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. 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. Most sampling trains are equipped with a 10-in. (water column) inclined-vertical manometer, having 0.01-in. H_2O divisions on the 0- to 1-in. inclined scale, and 0.1-in. H_2O 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.) H_2O. 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.) H_2O; (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.) H_2O; (3) for traverses of fewer than 12 points, more than one Δp reading is below 1.3 mm (0.05 in.) H_2O. Citation 18 in the 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 (in.) H_2O.

n = Total number of traverse points.

K = 0.13 mm H_2O when metric units are used and 0.005 in. H_2O 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., magnehelic 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. 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. 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 (0.1 in.) Hg. See **NOTE** in Method 5, Section 2.1.9.

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 4 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 for a reference. The standard pitot tube shall, preferably, have a known coefficient, obtained either (1) directly from the National Bureau of Standards, Route 70 S, Quince Orchard 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 in Sections 2.7.1 through 2.7.5 below and illustrated in Figure 2-4 (see also Citations 7, 8, and 17 in the 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 2-4) 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 the static pressure holes.

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

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

2.7.5 Ninety-degree bend, with curved or mitered junction.

2.8 Differential Pressure Gauge for Type S Pitot Tube Calibration. An inclined manometer or equivalent. If the single-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 (0.005 in.) H_20. For multivelocity calibrations, the gauge shall be readable to the nearest 0.13 mm (0.005 in.) H_20 for Δp values between 1.3 and 25 mm (0.05 and 1.0 in.) H_20, and to the nearest 1.3 mm (0.05 in.) H_20 for Δp values above 25 mm (1.0 in.) H_20. A special, more sensitive gauge will be required to read Δp values below 1.3 mm (0.05 in.) H_20 (see Citation 18 in the Bibliography).

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.) H_20 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.) H_20. 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 2-5).

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.

3.6 Determine the stack gas dry molecular weight. For combustion processes or processes that emit essentially $C0_2$, 0_2, CO, and N_2, 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.

3.7 Obtain the moisture content from Reference Method 4 (or equivalent) or from Method 5.

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. CALIBRATION

4.1 Type S Pitot Tube. Before its initial use, carefully examine the Type S 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_t, Figure 2-2b); and (b) the base-to-opening plane distances (dimensions P_A and P_B, Figure 2-2b). If D_t is between 0.48 and 0.95 cm (3/16 and 3/8 in.), and if P_A and P_B are equal and between 1.05 and 1.50 D_t, there are two possible options: (1) the pitot tube may be calibrated according to the procedure outlined in Sections 4.1.2 through 4.1.5 below, or (2) a baseline (isolated tube) 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 knowledge of the baseline coefficient value (see Section 4.1.1).

If D_t, P_A, and P_B are outside the specified limits, the pitot tube must be calibrated as outlined in Sections 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 the Bibliography); therefore an assigned (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 4.1.2 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).

4.1.2 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:

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:

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

Eq. 2-1

Where:

D_e = 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.

4.1.2.3 The flow system shall have the capacity to generate a test-section velocity around 915 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 915 m/min (3,000 ft/min) will generally be valid to ±3 percent for the measurement of velocities above 305 m/min (1,000 ft/min) and to ±5 to 6 percent for the measurement of velocities between 180 and 305 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 1,525 m/min (600 to 5,000 ft/min), and calibration data shall be taken at regular velocity intervals over this range (see Citations 9 and 14 in the Bibliography for details).

4.1.2.4 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.

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 4.1.5.1), 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 and is pointed directly into the flow. Make sure that the entry port surrounding the tube is properly sealed.

4.1.3.6 Read Δp_s, and enter its value in the data table. Remove the Type S pitot tube from the duct, and disconnect it from the manometer.

4.1.3.7 Repeat Steps 4.1.3.3 through 4.1.3.6 above until three pairs of Δp readings 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 Type S pitot tube.

4.1.3.9 Perform calculations, as described in Section 4.1.4 below.

4.1.4 Calculations.

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

$$C_{p(s)} = C_{p(std)} \sqrt{\frac{\Delta p_{std}}{\Delta p_s}} \qquad \text{Eq. 2-2}$$

Where:

$C_{p(s)}$ = Type S pitot tube coefficient.

$C_{p(std)}$ = 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_{std} = Velocity head measured by the standard pitot tube, cm (in.) H_2O.

Δp_s = Velocity head measured by the Type S pitot tube, cm (in.) H_2O.

4.1.4.2 Calculate $\overline{C}_p$ (side A), the mean A-side coefficient, and $\overline{C}_p$ (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 $C_{p(s)}$ from $\overline{C}_p$ (side A), and the deviation of each B-side values of $C_{p(s)}$ from $\overline{C}_p$ (side B). Use the following equation:

$$\text{Deviation} = C_{p_{(s)}} - \overline{C}_p(\text{A or B})$$

Eq. 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:

$$\sigma(\text{side A or B}) = \frac{\sum_{1}^{3} \left| C_{p(s)} - \overline{C}_p(\text{A or B}) \right|}{3}$$

Eq. 2-4

4.1.4.5 Use the Type S pitot tube only if the values 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 $\overline{C}_p$ (A) and $\overline{C}_p$ (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., $\overline{C}_p$ (side A) and $\overline{C}_p$ (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 9 in the 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 2-6A), the value of $C_{p(s)}$ 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 the Bibliography).

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.)

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 or in an 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, 30.5 to 91.4 cm (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 $C_{p(s)}$. Consult Citation 9 in the Bibliography for details. Conventional pitot-sampling probe assemblies are not recommended for use in ducts having inside diameters smaller than 30.5 cm (12 in.) (see Citation 16 in the Bibliography).

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 2-2 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 the Figure 2-2 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 Figure 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 dial 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 alternative reference, subject to the approval of the Administrator.

If, during calibration, the absolute temperature 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, m^2 (ft^2).

B_{ws} = Water vapor in the gas stream (from Method 5 or Reference Method 4), proportion by volume.

C_p = Pitot tube coefficient, dimensionless.

K_p = Pitot tube constant,

$$34.97 \frac{m}{sec}\left[\frac{(g/g\text{-mole})(mmHg)}{(^\circ K)(mmH_2O)}\right]^{1/2}$$

for the metric system.

$$85.49\frac{ft}{sec}\left[\frac{\text{lb/lb-mole})\ (\text{in.Hg})}{(^{\circ}R)\ (\text{in.}H_2O)}\right]^{1/2}$$

for the English system.

M_d = Molecular weight of stack gas, dry basis (see Section 3.6), g/g-mole (lb/lb-mole).

M_s = Molecular weight of stack gas, wet basis, g/g-mole (lb/lb-mole).

$$= M_d(1-B_{ws}) + 18.0B_{ws}$$

Eq. 2-5

P_{bar} = Barometric pressure at measurement site, mm Hg (in. Hg).

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

P_s = Absolute stack pressure, mm Hg (in. Hg),

$$= P_{bar} + P_g$$

Eq. 2-6

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

Q_{sd} = Dry volumetric stack gas flow rate corrected to standard conditions, dsm³/hr (dscf/hr).

t_s = Stack temperature, °C (°F).

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

$$= 273 + t_s$$

Eq. 2-7

for metric.

$$= 460 + t_s$$

Eq. 2-8

for English.

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

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

Δp = Velocity head of stack gas, mm H_2O (in. H_2O).

3,600= Conversion factor, sec/hr.

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

5.2 Average Stack Gas Velocity.

$$v_s = K_p C_p \left(\sqrt{\Delta p}\right)_{avg} \sqrt{\frac{T_{s(avg)}}{P_s M_s}}$$

Eq. 2-9

5.3 Average Stack Gas Dry Volumetric Flow Rate.

$$Q_{sd} = 3,600(1 - B_{ws}) v_s A \frac{T_{std}}{T_{s(avg)}} \frac{P_s}{P_{std}}$$

Eq. 2-10

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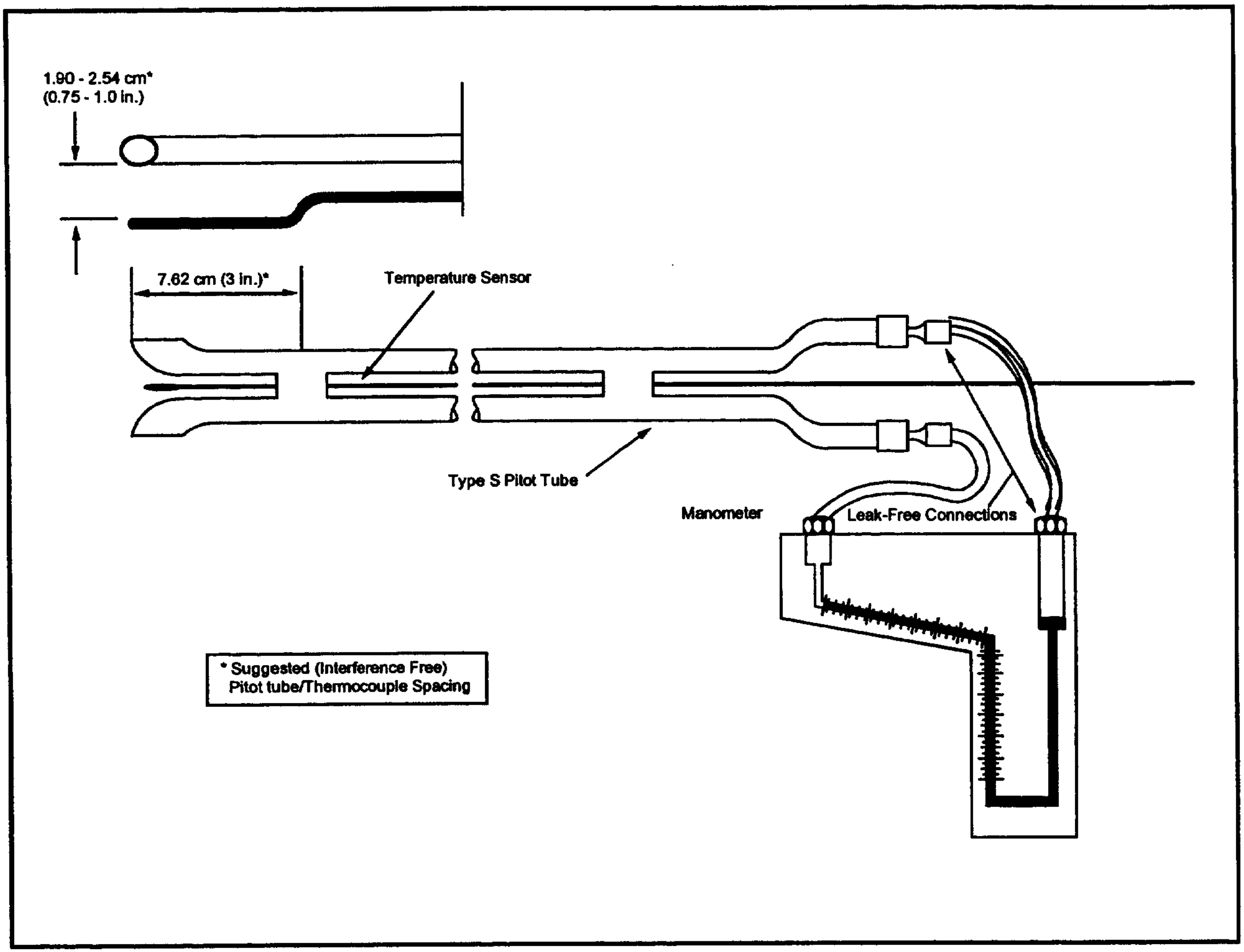
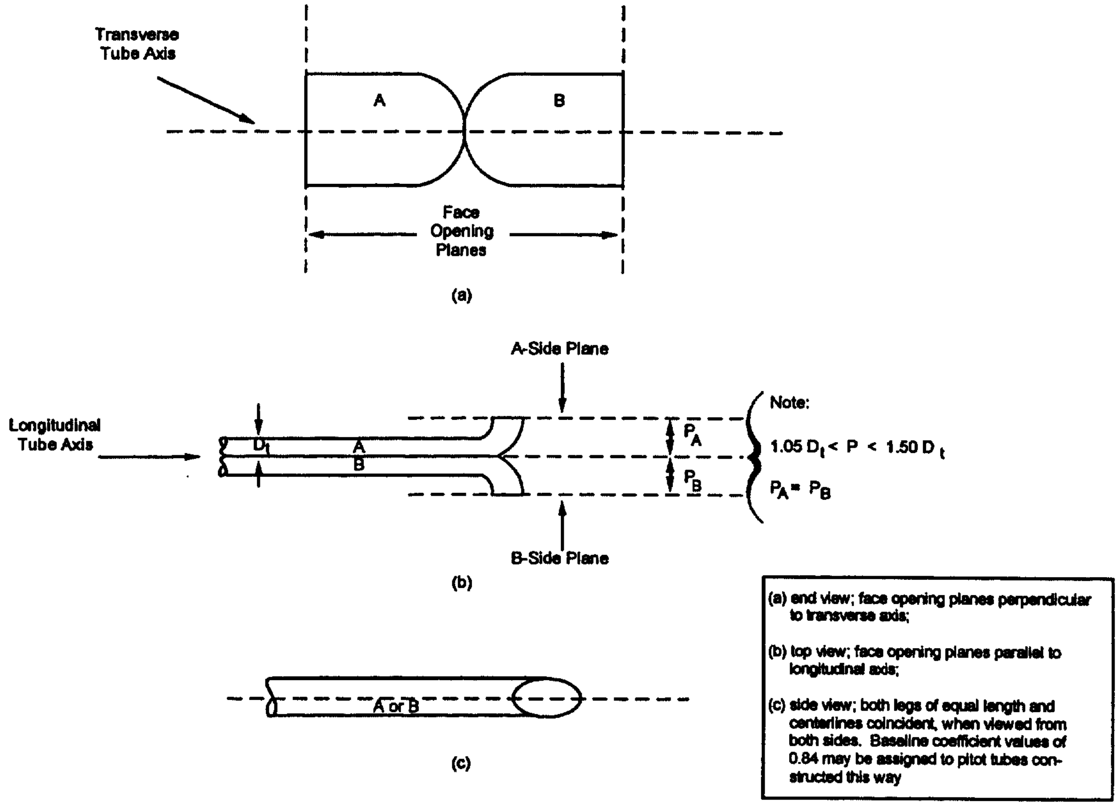


Figure 2-1. Type S pitot tube manometer assembly.



Note:

$1.05\ D_t < P < 1.50\ D_t$

$P_A = P_B$

(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 2-2. Properly constructed Type S pitot tube.

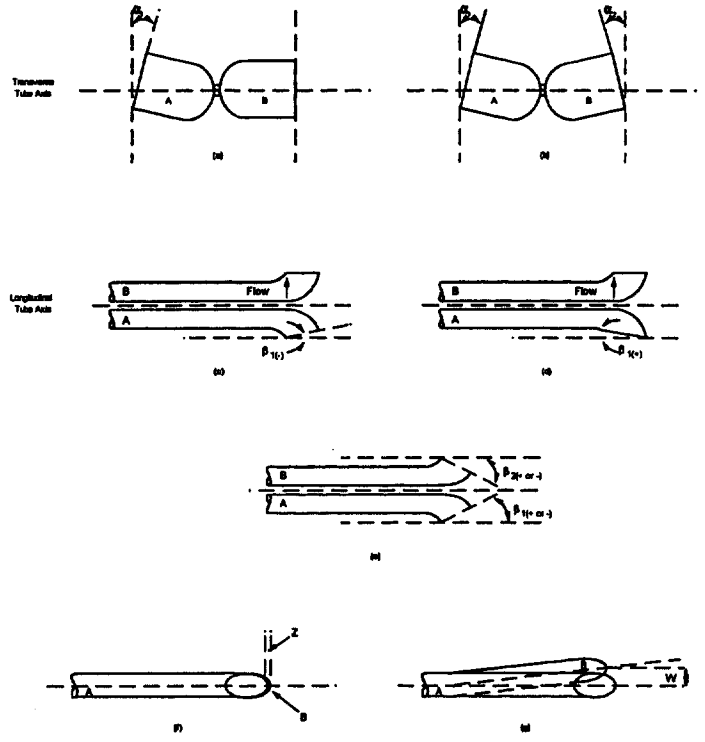


Figure 2-3. Types of face-opening misalignment that can result from field use or improper construction of Type S pitot tubes. These will not affect the baseline value of Cp(s) so long as α^1 and $\alpha^2 \leq 10°$, β^1 and $\beta^2 \leq 5°$, $z \leq 0.32$ cm (1/8 in.) and $w \leq 0.08$ cm (1/32 in.) (citation 11 in Bibliography).

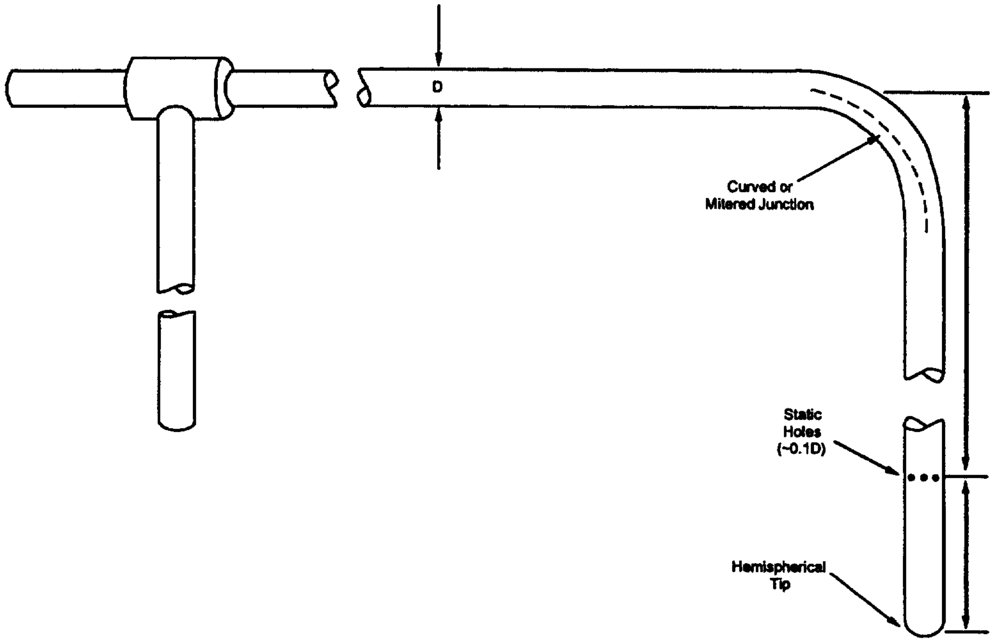


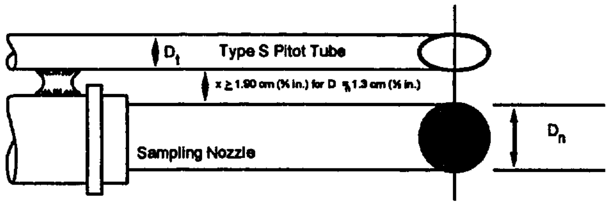
Figure 2-4. Standard pitot tube design specifications.

PLANT ________________________________
DATE __________ RUN NO. ________________STACK DIA. OR
DIMENSIONS, m (in.) _____ BAROMETRIC PRESS., mm Hg
(in. Hg) _____CROSS SECTIONAL AREA, m^2 (ft^2)________
OPERATORS ____________________________
PITOT TUBE I.D. NO. ____________________
AVG. COEFFICIENT, Cp = _____________
LAST DATE CALIBRATED ________________

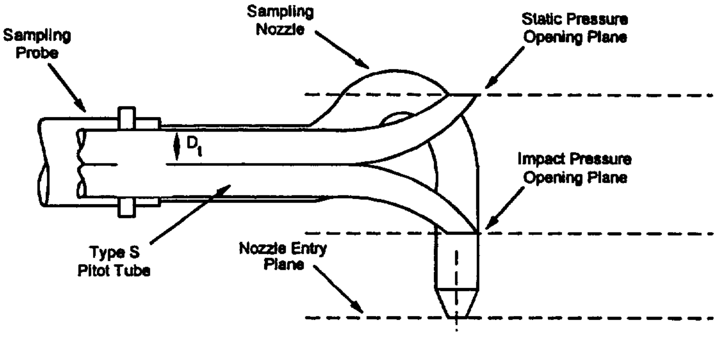
SCHEMATIC OF STACK
CROSS SECTION

Traverse Pt. No.	Vel. Hd., Δp mm (in.) H_2O	Stack Temperature		P_g mm Hg (in.Hg)	$(\Delta p)^{1/2}$
		T_s, °C (°F)	T_s, °K (°R)		
		Average			

Figure 2-5. Velocity traverse data.



A. Bottom View; showing minimum pitot tube-nozzle separation.



B. Side View; to prevent pitot tube from interfering with gas flow streamlines approaching the nozzle, the impact pressure opening plane of the pitot tube shall be even with or above the nozzle entry plane.

Figure 2-6. Proper pitot tube-sampling nozzle configuration to prevent aerodynamic interference; button-hook type nozzle; centers of nozzle and pitot opening aligned; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

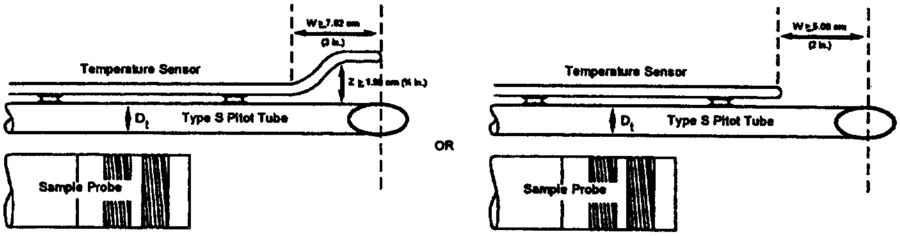


Figure 2-7. Proper thermocouple placement to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

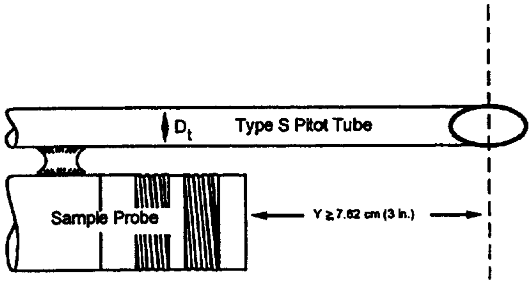


Figure 2-8. Minimum pitot-sample probe separation needed to prevent interference; D_t between 0.48 and 0.95 cm (3/16 and 3/8 in.).

PITOT TUBE IDENTIFICATION NUMBER: ________ DATE: ____________CALIBRATED BY: ______________________________________

	"A" SIDE CALIBRATION			
RUN NO.	ΔP_{std} cm H_2O (in H_2O)	$\Delta P_{(s)}$ cm H_2O (in H_2O)	$C_{p(s)}$	Deviation $C_{p(s)} - C_p(A)$
1				
2				
3				
		$C_{p,avg}$ (SIDE A)		

	"B" SIDE CALIBRATION			
RUN NO.	ΔP_{std} cm H_2O (in H_2O)	$\Delta P_{(s)}$ cm H_2O (in H_2O)	$C_{p(s)}$	Deviation $C_{p(s)} - C_p(B)$
1				
2				
3				
		$C_{p,avg}$ (SIDE B)		

$$\text{Average Deviation} = \sigma_{(A\,or\,B)} = \frac{\sum_{i=1}^{3}\left|C_{p(s)} - \overline{C}_{p(A\,or\,B)}\right|}{3} \leftarrow \text{Must Be} \le 0.01$$

$$\left|\overline{C}_p(\text{Side A}) - \overline{C}_p(\text{Side B})\right| \leftarrow \text{Must Be} \le 0.01$$

Figure 2-9. Pitot tube calibration data.

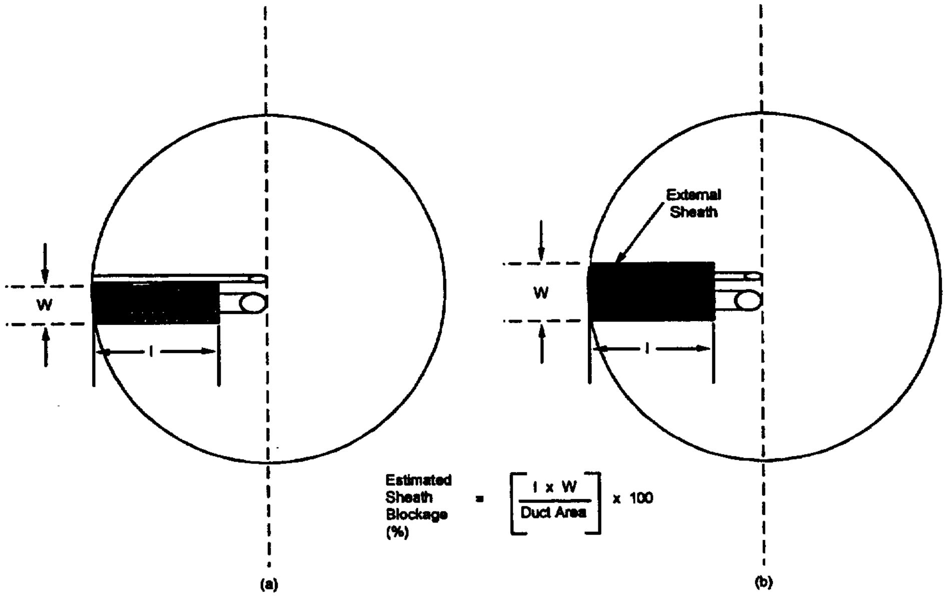


Figure 2-10. Projected-area models for typical pitot tube assemblies.

Method 3B - Gas Analysis for the Determination of Emission Rate Correction Factor or Excess Air

1. APPLICABILITY AND PRINCIPLE

1.1 Applicability.

1.1.1 This method is applicable for determining carbon dioxide (CO_2), oxygen (O_2), and carbon monoxide (CO) concentrations of a sample from a gas stream of a fossil-fuel combustion process for excess air or emission rate correction factor calculations.

1.1.2 Other methods, as well as modifications to the procedure described herein, are also applicable for all of the above determinations. Examples of specific methods and modifications include: (1) a multi-point sampling method using an Orsat analyzer to analyze individual grab samples obtained at each point, and (2) a method using CO_2 or O_2 and stoichiometric calculations to determine excess air. These methods and modifications may be used, but are subject to the approval of the Administrator, U.S. Environmental Protection Agency (EPA).

1.1.3 **Note.** Mention of trade names or specific products does not constitute endorsement by EPA.

1.2 Principle. A gas sample is extracted from a stack by one of the following methods: (1) single-point, grab sampling; (2) single-point, integrated sampling; or (3) multi-point, integrated sampling. The gas sample is analyzed for percent CO_2, percent O_2, and, if necessary, percent CO. An Orsat analyzer must be used for excess air or emission rate correction factor determinations.

2. APPARATUS

The alternative sampling systems are the same as those mentioned in Section 2 of Method 3.

2.1 Grab Sampling and Integrated Sampling. Same as in Sections 2.1 and 2.2, respectively, of Method 3.

2.2 Analysis. An Orsat analyzer only. For low CO_2 (less than 4.0 percent) or high O_2 (greater than 15.0 percent) concentrations, the measuring burette of the Orsat must have at least 0.1 percent subdivisions. For Orsat maintenance and operation procedures, follow the instructions recommended by the manufacturer, unless otherwise specified herein.

3. PROCEDURES

Each of the three procedures below shall be used only when specified in an applicable subpart of the standards. The use of these procedures for other purposes must have specific prior approval of the Administrator.
Note: A Fyrite-type combustion gas analyzer is not acceptable for excess air or emission rate correction factor determinations, unless approved by the Administrator. If both percent CO_2 and percent O_2 are measured, the analytical results of any of the three procedures given below may also be used for calculating the dry molecular weight (see Method 3).

3.1 Single-Point, Grab Sampling and Analytical Procedure.

3.1.1 The sampling point in the duct shall be as described in Section 3.1 of Method 3.

3.1.2 Set up the equipment as shown in Figure 3-1 of Method 3, making sure all connections ahead of the analyzer are tight. Leak check the Orsat analyzer according to the procedure described in Section 6 of Method 3. This leak check is mandatory.

3.1.3 Place the probe in the stack, with the tip of the probe positioned at the sampling point; purge the sampling line long enough to allow at least five exchanges. Draw a sample into the analyzer. For emission rate correction factor determinations, immediately analyze the sample, as outlined in Sections 3.1.4 and 3.1.5, for percent CO_2 or percent O_2. If excess air is desired, proceed as follows: (1) immediately analyze the sample, as in Sections 3.1.4 and 3.1.5, for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent, and (3) calculate percent excess air as outlined in Section 4.2.

3.1.4 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, make repeated passes through each absorbing solution until two consecutive readings are the same. Several passes (three or four) should be made between readings. (If constant readings cannot be obtained after three consecutive readings, replace the absorbing solution.) Note: Since this single-point, grab sampling and analytical procedure is normally conducted in conjunction with a single-point, grab sampling and analytical procedure for a pollutant, only one analysis is ordinarily conducted. Therefore, great care must be taken to obtain a valid sample and analysis. Although in most cases, only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 3.4 be used to validate the analytical data.

3.1.5 After the analysis is completed, leak check (mandatory) the Orsat analyzer once again, as described in Section 6 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis.

3.2 Single-Point, Integrated Sampling and Analytical Procedure.

3.2.1 The sampling point in the duct shall be located as specified in Section 3.1.1.

3.2.2 Leak check (mandatory) the flexible bag as in Section 2.2.6 of Method 3. Set up the equipment as shown in Figure 3-2 of Method 3. Just before sampling, leak check (mandatory) the train as described in Section 4.2 of Method 3.

3.2.3 Sample at a constant rate, or as specified by the Administrator. The sampling run must be simultaneous with, and for the same total length of time as, the pollutant emission rate determination. Collect at least 30 liters (1.00 ft^3) of sample gas. Smaller volumes may be collected, subject to approval of the Administrator.

3.2.4 Obtain one integrated flue gas sample during each pollutant emission rate determination. For emission rate correction factor determination, analyze the sample within 4 hours after it is taken for percent CO_2 or percent O_2 (as outlined in Sections 3.2.5 through 3.2.7). The Orsat analyzer must be leak checked (see Section 6 of Method 3) before the analysis. If excess air is desired, proceed as follows: (1) within 4 hours after the sample is taken, analyze it (as in Sections 3.2.5 through 3.2.7) for percent CO_2, O_2, and CO; (2) determine the percentage of the gas that is N_2 by subtracting the sum of the percent CO_2, percent O_2, and percent CO from 100 percent; and (3) calculate percent excess air, as outlined in Section 4.2.

3.2.5 To ensure complete absorption of the CO_2, O_2, or if applicable, CO, follow the procedure described in Section 3.1.4. Note: Although in most instances only CO_2 or O_2 is required, it is recommended that both CO_2 and O_2 be measured, and that Section 3.4.1 be used to validate the analytical data.

3.2.6 Repeat the analysis until the following criteria are met:

3.2.6.1 For percent CO_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when CO_2 is greater than 4.0 percent or (b) 0.2 percent by volume when CO_2 is less than or equal to 4.0 percent. Average three acceptable values of percent CO_2, and report the results to the nearest 0.1 percent.

3.2.6.2 For percent O_2, repeat the analytical procedure until the results of any three analyses differ by no more than (a) 0.3 percent by volume when O_2 is less than 15.0 percent or (b) 0.2 percent by volume when O_2 is greater than or equal to 15.0 percent. Average the three acceptable values of percent O_2, and report the results to the nearest 0.1 percent.

3.2.6.3 For percent CO, repeat the analytical procedure until the results of any three analyses differ by no more than 0.3 percent. Average the three acceptable values of percent CO, and report the results to the nearest 0.1 percent.

3.2.7 After the analysis is completed, leak check (mandatory) the Orsat analyzer once again, as described in Section 6 of Method 3. For the results of the analysis to be valid, the Orsat analyzer must pass this leak test before and after the analysis.

3.3 Multi-Point, Integrated Sampling and Analytical Procedure.

3.3.1 The sampling points shall be determined as specified in Section 5.3 of Method 3.

3.3.2 Follow the procedures outlined in Sections 3.2.2 through 3.2.7, except for the following: Traverse all sampling points, and sample at each point for an equal length of time. Record sampling data as shown in Figure 3-3 of Method 3.

3.4 Quality Control Procedures.

3.4.1 Data Validation When Both CO_2 and O_2 Are Measured. Although in most instances, only CO_2 or O_2 measurement is required, it is recommended that both CO_2 and O_2 be measured to provide a check on the quality of the data. The following quality control procedure is suggested. <u>Note</u>: Since the method for validating the CO_2 and O_2 analyses is based on combustion of organic and fossil fuels and dilution of the gas stream with air, this method does not apply to sources that (1) remove CO_2 or O_2 through processes other than combustion, (2) add O_2 (e.g., oxygen enrichment) and N_2 in proportions different from that of air, (3) add CO_2 (e.g., cement or lime kilns), or (4) have no fuel factor, F_o, values obtainable (e.g., extremely variable waste mixtures). This method validates the measured proportions of CO_2 and O_2 for fuel type, but the method does not detect sample dilution resulting from leaks during or after sample collection. The method is applicable for samples collected downstream of most lime or limestone flue-gas desulfurization units as the CO_2 added or removed from the gas stream is not significant in relation to the total CO_2 concentration. The CO_2 concentrations from other types of scrubbers using only water or basic slurry can be significantly affected and would render the F_o check minimally useful.

3.4.1.1 Calculate a fuel factor, F_o, using the following equation:

$$F_o = \frac{20.9 - \%O_2}{\%CO_2} \qquad \text{Eq. 3B-1}$$

where:

$\%O_2$ = Percent O_2 by volume, dry basis.

$\%CO_2$ = Percent CO_2 by volume, dry basis.

20.9 = Percent O_2 by volume in ambient air.

If CO is present in quantities measurable by this method, adjust the O_2 and CO_2 values before performing the calculation for F_o as follows:

$$\%CO_2(adj) = \%CO_2 + \%CO$$

$$\%O_2(adj) = \%O_2 - 0.5\ \%CO$$

where:

$\%CO$ = Percent CO by volume, dry basis.

3.4.1.2 Compare the calculated F_o factor with the expected F_o values. The following table may be used in establishing acceptable ranges for the expected F_o if the fuel being burned is known. When fuels are burned in combinations, calculate the combined fuel F_d and F_c factors (as defined in Method 19) according to the procedure in Method 19, Section 5.2.3. Then calculate the F_o factor as follows:

$$F_o = \frac{0.209\ F_d}{F_c} \qquad \text{Eq. 3B-2}$$

	Fuel type	F_o range
Coal:	Anthracite and lignite	1.016 - 1.130
	Bituminous	1.083 - 1.230
Oil:	Distillate	1.260 - 1.413
	Residual	1.210 - 1.370
Gas:	Natural	1.600 - 1.836
	Propane	1.434 - 1.586
	Butane	1.405 - 1.553
Wood		1.000 - 1.120
Wood bark		1.003 - 1.130

3.4.1.3 Calculated F_0 values, beyond the acceptable ranges shown in this table, should be investigated before accepting the test results. For example, the strength of the solutions in the gas analyzer and the analyzing technique should be checked by sampling and analyzing a known concentration, such as air; the fuel factor should be reviewed and verified. An acceptability range of ±12 percent is appropriate for the F_0 factor of mixed fuels with variable fuel ratios. The level of the emission rate relative to the compliance level should be considered in determining if a retest is appropriate, i.e.; if the measured emissions are much lower or much greater than the compliance limit, repetition of the test would not significantly change the compliance status of the source and would be unnecessarily time consuming and costly.

4. CALCULATIONS

4.1 Nomenclature. Same as Section 5 of Method 3 with the addition of the following:

%EA = Percent excess air.

0.264 = Ratio of O_2 to N_2 in air, v/v.

4.2 Percent Excess Air. Calculate the percent excess air (if applicable) by substituting the appropriate values of percent O_2, CO, and N_2 (obtained from Section 3.1.3 or 3.2.4) into Equation 3B-3.

$$\%EA = \frac{\%O_2 - 0.5\ \%CO}{0.264\ \%N_2 - (\%O_2 - 0.5\ \%CO)} \times 100 \qquad \text{Eq. 3B-3}$$

Note: The equation above assumes that ambient air is used as the source of O_2 and that the fuel does not contain appreciable amounts of N_2 (as do coke oven or blast furnace gases). For those cases when appreciable amounts of N_2 are present (coal, oil, and natural gas do not contain appreciable amounts of N_2) or when oxygen enrichment is used, alternative methods, subject to approval of the Administrator, are required.

5. BIBLIOGRAPHY

Same as Method 3.

Method 4 - Determination of Moisture Content in Stack Gases

1. PRINCIPLE AND APPLICABILITY

1.1 Principle. A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

1.2 Applicability. This method is applicable for determining the moisture content of stack gas.

1.2.1 Two procedures are given. The first is a reference method, for accurate determinations of moisture content (such as are needed to calculate emission data). The second is an approximation method, which provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content, e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc., are also acceptable.

1.2.2 The reference method is often conducted simultaneously with a pollutant emission measurement run; when it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent; these calculations shall not be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, U.S. Environmental Protection Agency, to be capable of yielding results within 1 percent H_2O of the reference method.

1.2.3 Note: The reference method may yield questionable results when applied to saturated gas streams or to streams that contain water droplets. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to within 1°C (2°F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see Section 2.2.1) during the reference method traverse; calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) using a psychrometric chart and making appropriate corrections if stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternative methods, subject to the approval of the Administrator, shall be used.

2. REFERENCE METHOD

The procedure described in Method 5 for determining moisture content is acceptable as a reference method.

2.1 Apparatus. A schematic of the sampling train used in this reference method is shown in Figure 4-1. All components shall be maintained and calibrated according to the procedures in Method 5.

2.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and equipped with a filter, either in-stack (e.g., a plug of glass wool inserted into the end of the probe) or heated out-stack (e.g., as described in Method 5), to remove particulate matter. When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

2.1.2 Condenser. See Method 5, Section 2.1.7, for a description of an acceptable type of condenser and for alternative measurement systems.

2.1.3 Cooling System. An ice bath container and crushed ice (or equivalent), to aid in condensing moisture.

2.1.4 Metering System. Same as in Method 5, Section 2.1.8, except do not use sampling systems designed for flow rates higher than 0.0283 m^3/min (1.0 cfm). Other metering systems, capable of maintaining a constant sampling rate to within 10 percent and determining sample gas volume to within 2 percent, may be used, subject to the approval of the Administrator.

2.1.5 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg. See **NOTE** in Method 5, Section 2.1.9.

2.1.6 Graduated Cylinder and/or Balance. To measure condensed water and moisture caught in the silica gel to within 1 ml or 0.5 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. These balances are suitable for use here.

2.2 Procedure. The following procedure is written for a condenser system (such as the impinger system described in Section 2.1.7 of Method 5) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

2.2.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point. Place known volumes of water in the first two impingers. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

2.2.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m^3/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

2.2.3 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of about 120°C (248°F), to prevent water condensation ahead of the condenser; allow time for the temperatures to stabilize. Place crushed ice in the ice bath container. It is recommended, but not required, that a leak check be done, as follows: Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder), and pull a 380 mm (15 in.) Hg vacuum; a lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.02 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

2.2.4 During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each run, record the data required on the example data sheet shown in Figure 4-2. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point, at least once during each time increment.

2.2.5 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the silica gel outlet.

2.2.6 After collecting the sample, disconnect the probe from the filter holder (or from the first impinger), and conduct a leak check (mandatory) as described in Section 2.2.3. Record the leak rate. If the leakage rate exceeds the allowable rate, the tester shall either reject the test results or shall correct the sample volume as in Section 6.3 of Method 5. Next, measure the volume of the moisture condensed to the nearest ml. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-3), and calculate the moisture percentage, as described in 2.3 below.

2.2.7 A quality control check of the volume metering system at the field site is suggested before collecting the sample following the procedure in Method 5, Section 4.4.

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

2.3.1 Nomenclature.

B_{ws} = Proportion of water vapor, by volume, in the gas stream.

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

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

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

R = Ideal gas constant, 0.06236 (mm Hg)(m^3)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft^3)/(lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

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

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

$V_{wsg(std)}$ = Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).

V_f = Final volume of condenser water, ml.

V_i = Initial volume, if any, of condenser water, ml.

W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.

Y = Dry gas meter calibration factor.

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

2.3.2 Volume of Water Vapor Condensed.

$$V_{wc(std)} = (V_f - V_i)\,\rho_w \frac{RT_{std}}{P_{std}M_w} = K_1\,(V_f - V_i) \qquad \text{Eq. 4-1}$$

Where:

K_1 = 0.001333 m^3/ml for metric units,

= 0.04707 ft^3/ml for English units.

2.3.3 Volume of Water Collected in Silica Gel.

$$V_{wsg(std)} = \frac{(W_f - W_i)\,RT_{std}}{P_{std}M_w} = K_2\,(W_f - W_i) \qquad \text{Eq. 4-2}$$

Where:

K_2 = 0.001335 m^3/g for metric units,

= 0.04715 ft^3/g for English units.

2.3.4 Sample Gas Volume.

$$V_{m(std)} = V_m Y \frac{(P_m)(T_{std})}{(P_{std})(T_m)} = K_3 Y \frac{V_m P_m}{T_m} \qquad \text{Eq. 4-3}$$

Where:

K_3 = 0.3858 °K/mm Hg for metric units,

= 17.64 °R/in. Hg for English units.

NOTE: If the post-test leak rate (Section 2.2.6) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in Section 6.3 of Method 5.

2.3.5 Moisture Content.

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} \qquad \text{Eq. 4-4}$$

NOTE: In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see Section 1.2), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

2.3.6 Verification of Constant Sampling Rate. For each time increment, determine the ΔV_m. Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results, and repeat the run.

3. APPROXIMATION METHOD

The approximation method described below is presented only as a suggested method (see Section 1.2).

3.1 Apparatus. See Figure 4-4.

3.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation and equipped with a filter (either in-stack or heated out-stack) to remove particulate matter. A plug of glass wool, inserted into the end of the probe, is a satisfactory filter.

3.1.2 Impingers. Two midget impingers, each with 30-ml capacity, or equivalent.

3.1.3 Ice Bath. Container and ice, to aid in condensing moisture in impingers.

3.1.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

3.1.5 Valve. Needle valve, to regulate the sample gas flow rate.

3.1.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

3.1.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to within 2 percent, and calibrated over the range of flow rates and conditions actually encountered during sampling.

3.1.8 Rate Meter. Rotameter, to measure the flow range from 0 to 3 liters/min (0 to 0.11 cfm).

3.1.9 Graduated Cylinder. 25-ml.

3.1.10 Barometer. Mercury, aneroid, or other barometer, as described in Section 2.1.5 above.

3.1.11 Vacuum Gauge. At least 760-mm (30-in.) Hg gauge, to be used for the sampling leak check.

3.2 Procedure.

3.2.1 Place exactly 5 ml water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet; then, plug the probe inlet, and pull a vacuum of at least 250 mm (10 in.) Hg. Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0 to 40 cc/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable. **NOTE:** Carefully release the probe inlet plug before turning off the pump.

3.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 liters/min (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft^3) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as required by Figure 4-5.

3.2.3 After collecting the sample, combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.

3.3 Calculations. The calculation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content, for the purpose of determining isokinetic sampling rate settings.

3.3.1 Nomenclature.

B_{wm} = Approximate proportion by volume of water vapor in the gas stream leaving the second impinger, 0.025.

B_{ws} = Water vapor in the gas stream, proportion by volume.

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

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

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

R = Ideal gas constant, 0.06236 [(mm Hg)(m^3)]/[(g-mole)(°K)] for metric units and 21.85 [(in. Hg)(ft^3)]/[(lb-mole)(°R)] for English units.

T_m = Absolute temperature at meter, °K (°R).

T_{std} = Standard absolute temperature, 293°R (528°R).

V_f = Final volume of impinger contents, ml.

V_i = Initial volume of impinger contents, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

Y = Dry gas meter calibration factor.

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

3.3.2 Volume of Water Vapor Collected.

$$V_{wc} = \frac{(V_f - V_i)\rho_w R T_{std}}{P_{std} M_w} = K_1 (V_f - V_i) \qquad \text{Eq. 4-5}$$

Where:

K_1 = 0.001333 m^3/ml for metric units,

= 0.04707 ft^3/ml for English units.

3.3.3 Gas Volume.

$$V_{m(std)} = V_m\left(\frac{P_m}{P_{std}}\right)\left(\frac{T_{std}}{T_m}\right) = K_2\ V_m \frac{P_m}{T_m} \qquad \text{Eq. 4-6}$$

Where:

K_2 = 0.03858 °K/mm Hg for metric units,

= 17.64 °R/in. Hg for English units.

3.3.4 Approximate Moisture Content.

$$B_{ws} = \frac{V_{wc}}{V_{wc}+V_{m(std)}} + B_{wm} = \frac{V_{wc}}{V_{wc}+V_{m(std)}} + (0.025) \qquad \text{Eq. 4-7}$$

4. CALIBRATION

4.1 For the reference method, calibrate the metering system, temperature gauges, and barometer according to Sections 5.3, 5.5, and 5.7, respectively, of Method 5. The recommended leak check of the metering system (Section 5.6 of Method 5) also applies to the reference method. For the approximation method, use the procedures outlined in Section 5.1.1 of Method 6 to calibrate the metering system, and the procedure of Method 5, Section 5.7, to calibrate the barometer.

5. BIBLIOGRAPHY

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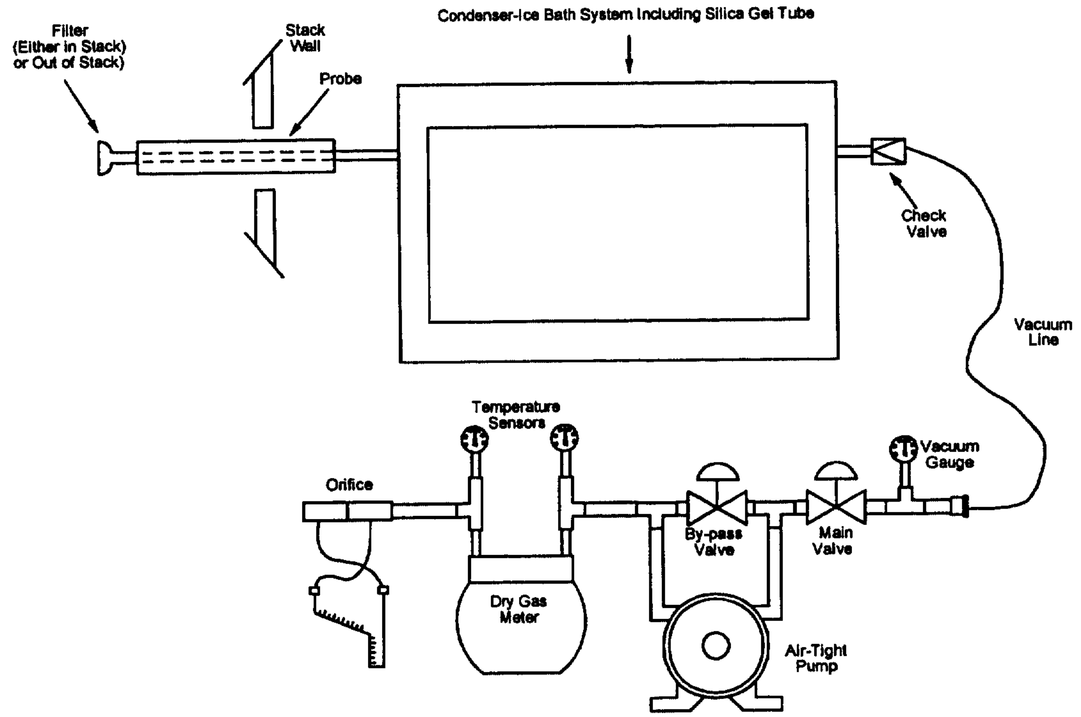

Figure 4-1. Moisture sampling train reference method.

Figure 4-2. Field Moisture Determination Reference Method.

Plant ______________________________
Location______________________________
Operator______________________________
Date______________________________
Run No. ______________________________
Ambient temperature____________________
Barometric pressure____________________
Probe Length_________________________

SCHEMATIC OF STACK CROSS SECTION

Traverse Pt. No.	Sampling Time (θ), min	Stack Temperature °C (°F)	Pressure differential across orifice meter ΔH mm (in.) H_2O	Meter Reading gas sample volume m^3 (ft^3)	ΔV_m m^3 (ft^3)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger °C(°F)
						Inlet Tm_{in} °C(°F)	Outlet Tm_{out} °C(°F)	
Average								

Figure 4-3. Analytical data - reference method.

	Impinger volume, ml	Silica gel weight, g
Final	________	________
Initial	________	________
Difference	________	________

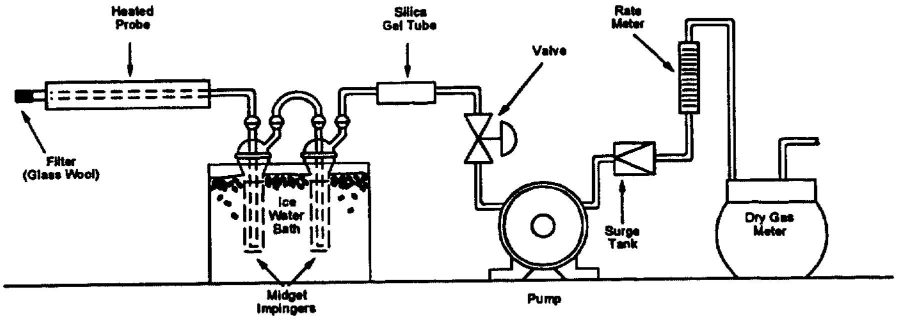

Figure 4-4. Moisture Samping Train - Approximation Method.

Figure 4-5. Field Moisture Determination - Approximation Method.

Location________________________________Comments:
Test____________________________________
Date____________________________________
Operator________________________________
Barometric pressure____________________

Clock Time	Gas volume through meter, (V_m), m^3 (ft^3)	Rate meter setting m^3/min (ft^3/min)	Meter temperature ° C (° F)

APPENDIX A TO PART 63--TEST METHODS

* * * * *

METHOD 315 - DETERMINATION OF PARTICULATE AND METHYLENE CHLORIDE EXTRACTABLE MATTER (MCEM) FROM SELECTED SOURCES AT PRIMARY ALUMINUM PRODUCTION FACILITIES

NOTE: This method does not include all of the specifications (e.g., equipment and supplies) and procedures (e.g., sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5 of 40 CFR part 60, appendix A.

1.0 Scope and Application.

1.1 Analytes. Particulate matter (PM). No CAS number assigned. Methylene chloride extractable matter (MCEM). No CAS number assigned.

1.2 Applicability. This method is applicable for the simultaneous determination of PM and MCEM when specified in an applicable regulation. This method was developed by consensus with the Aluminum Association and the U.S. Environmental Protection Agency (EPA) and has limited precision estimates for MCEM; it should have similar precision to Method 5 for PM in 40 CFR part 60, appendix A since the procedures are similar for PM.

1.3 Data quality objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method.

Particulate matter and MCEM are withdrawn isokinetically from the source. PM is collected on a glass fiber filter maintained at a temperature in the range of 120 ± 14°C (248 ± 25°F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The PM mass, which includes any material that condenses on the probe and is subsequently removed in an acetone rinse or on the filter at or above the filtration temperature, is determined gravimetrically after removal of uncombined water. MCEM is then determined by adding a methylene chloride rinse of the probe and filter holder, extracting the condensable hydrocarbons collected in the impinger water, adding an acetone rinse followed by a methylene chloride rinse of the sampling train components after the filter and before the silica gel impinger, and determining residue gravimetrically after evaporating the solvents.

3.0 Definitions. [Reserved]

4.0 Interferences. [Reserved]

5.0 Safety.

This method may involve hazardous materials, operations, and equipment. This method does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this method to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies.

NOTE: Mention of trade names or specific products does not constitute endorsement by the EPA.

6.1 Sample collection. The following items are required for sample collection:

6.1.1 Sampling train. A schematic of the sampling train used in this method is shown in Figure 5-1, Method 5, 40 CFR part 60, appendix A. Complete construction details are given in APTD-0581 (Reference 2 in section 17.0 of this method); commercial models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, Method 5, 40 CFR part 60, appendix, A see the following subsections.

NOTE: The operating and maintenance procedures for the sampling train are described in APTD-0576 (Reference 3 in section 17.0 of this method). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein. The use of grease for sealing sampling train components is not recommended because many greases are soluble in methylene chloride. The sampling train consists of the following components:

6.1.1.1 Probe nozzle. Glass or glass lined 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. Other materials of construction may be used, subject to the approval of the Administrator. A range of nozzle sizes suitable for isokinetic sampling should be available. Typical nozzle sizes range from 0.32 to 1.27 cm (1/8 to 1/2 in.) inside diameter (ID) in increments of 0.16 cm (1/16 in.). Larger nozzle sizes are also available if higher volume sampling trains are used. Each nozzle shall be calibrated according to the procedures outlined in section 10.0 of this method.

6.1.1.2 Probe liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a probe gas temperature at the exit end during sampling of 120 ± 14°C (248 ± 25°F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Because the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and using the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz liners shall be used for temperatures between 480 and 900°C (900 and 1,650°F). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate glass is 820°C (1,500°F) and for quartz glass it is 1,500°C (2,700°F).

6.1.1.3 Pitot tube. Type S, as described in section 6.1 of Method 2, 40 CFR part 60, appendix A, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in Figure 5-1 of Method 5, 40 CFR part 60, appendix A) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-6b, 40 CFR part 60, appendix A) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in section 10.0 of Method 2, 40 CFR part 60, appendix A.

6.1.1.4 Differential pressure gauge. Inclined manometer or equivalent device (two), as described in section 6.2 of Method 2, 40 CFR part 60, appendix A. One manometer shall be used for velocity head (Dp) readings, and the other, for orifice differential pressure readings.

6.1.1.5 Filter holder. Borosilicate glass, with a glass frit filter support and a silicone rubber gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

6.1.1.6 Filter heating system. Any heating system capable of maintaining a temperature around the filter holder of 120 ± 14°C (248 ± 25°F) during sampling, or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. Alternatively, the tester may opt to operate the equipment at a temperature lower than that specified. A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling. Heating systems other than the one shown in APTD-0581 may be used.

6.1.1.7 Temperature sensor. A temperature sensor capable of measuring temperature to within ±3°C (5.4°F) shall be installed so that the sensing tip of the temperature sensor is in direct contact with the sample gas, and the temperature around the filter holder can be regulated and monitored during sampling.

6.1.1.8 Condenser. The following system shall be used to determine the stack gas moisture content: four glass impingers connected in series with leak-free ground glass fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm (1/2 in.) ID glass tube extending to about 1.3 cm (1/2 in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard tip. The first and second impingers shall contain known quantities of water (section 8.3.1 of this method), the third shall be empty, and the fourth shall contain a known weight of silica gel or equivalent desiccant. A temperature sensor capable of measuring temperature to within 1°C (2°F) shall be placed at the outlet of the fourth impinger for monitoring.

6.1.1.9 Metering system. Vacuum gauge, leak-free pump, temperature sensors capable of measuring temperature to within 3°C (5.4°F), dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1 of Method 5, 40 CFR part 60, appendix A. 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 allow periodic checks of isokinetic rates.

6.1.1.10 Sampling trains using metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm (0.1 in.) Hg.

NOTE: The barometric reading may be obtained from a nearby National Weather Service station. In this 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 made at a rate of minus 2.5 mm (0.1 in) Hg per 30 m (100 ft) elevation increase or plus 2.5 mm (0.1 in) Hg per 30 m (100 ft) elevation decrease.

6.1.3 Gas density determination equipment. Temperature sensor and pressure gauge, as described in sections 6.3 and 6.4 of Method 2, 40 CFR part 60, appendix A, and gas analyzer, if necessary, as described in Method 3, 40 CFR part 60, appendix A. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-4, 40 CFR part 60, appendix A). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature sensor need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

6.2 Sample recovery. The following items are required for sample recovery:

6.2.1 Probe-liner and probe-nozzle brushes. Nylon or Teflon® bristle brushes with stainless steel wire handles. The probe brush shall have extensions (at least as long as the probe) constructed of stainless steel, nylon, Teflon®, or similarly inert material. The brushes shall be properly sized and shaped to brush out the probe liner and nozzle.

6.2.2 Wash bottles. Glass wash bottles are recommended. Polyethylene or tetrafluoroethylene (TFE) wash bottles may be used, but they may introduce a positive bias due to contamination from the bottle. It is recommended that acetone not be stored in polyethylene or TFE bottles for longer than a month.

6.2.3 Glass sample storage containers. Chemically resistant, borosilicate glass bottles, for acetone and methylene chloride washes and impinger water, 500 ml or 1,000 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 or methylene chloride. (Narrow-mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

6.2.4 Petri dishes. For filter samples, glass, unless otherwise specified by the Administrator.

6.2.5 Graduated cylinder and/or balance. To measure condensed water, acetone wash and methylene chloride wash used during field recovery of the samples, 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 such balance is suitable for use here and in section 6.3.4 of this method.

6.2.6 Plastic storage containers. Air-tight containers to store silica gel.

6.2.7 Funnel and rubber policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

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

6.3 Sample analysis. The following equipment is required for sample analysis:

6.3.1 Glass or Teflon® weighing dishes.

6.3.2 Desiccator. It is recommended that fresh desiccant be used to minimize the chance for positive bias due to absorption of organic material during drying.

6.3.3 Analytical balance. To measure to within 0.1 mg.

6.3.4 Balance. To measure to within 0.5 g.

6.3.5 Beakers. 250 ml.

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

6.3.7 Temperature sensor. To measure the temperature of the laboratory environment.

6.3.8 Buchner fritted funnel. 30 ml size, fine (<50 micron)-porosity fritted glass.

6.3.9 Pressure filtration apparatus.

6.3.10 Aluminum dish. Flat bottom, smooth sides, and flanged top, 18 mm deep and with an inside diameter of approximately 60 mm.

7.0 Reagents and Standards.

7.1 Sample collection. The following reagents are required for sample collection:

7.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Method D 2986-95A (incorporated by reference in § 63.841 of this part). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing $S0_2$ or $S0_3$, the filter material must be of a type that is unreactive to $S0_2$ or $S0_3$. Reference 10 in section 17.0 of this method may be used to select the appropriate filter.

7.1.2 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.

7.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water shall be used. Run blanks prior to field use to eliminate a high blank on test samples.

7.1.4 Crushed ice.

7.1.5 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. [Caution: Many stopcock greases are methylene chloride-soluble. Use sparingly and carefully remove prior to recovery to prevent contamination of the MCEM analysis.]

7.2 Sample recovery. The following reagents are required for sample recovery:

7.2.1 Acetone. Acetone with blank values < 1 ppm, by weight residue, is required. Acetone blanks may be run prior to field use, and only acetone with low blank values may be used. In no case shall a blank value of greater than 1E-06 of the weight of acetone used be subtracted from the sample weight.

NOTE: This is more restrictive than Method 5, 40 CFR part 60, appendix A. At least one vendor (Supelco Incorporated located in Bellefonte, Pennsylvania) lists <1 mg/l as residue for its Environmental Analysis Solvents.

7.2.2 Methylene chloride. Methylene chloride with a blank value <1.5 ppm, by weight, residue. Methylene chloride blanks may be run prior to field use, and only methylene chloride with low blank values may be used. In no case shall a blank value of greater than 1.6E-06 of the weight of methylene chloride used be subtracted from the sample weight.

NOTE: A least one vendor quotes <1 mg/l for Environmental Analysis Solvents-grade methylene chloride.

7.3 Sample analysis. The following reagents are required for sample analysis:

7.3.1 Acetone. Same as in section 7.2.1 of this method.

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

7.3.3 Methylene chloride. Same as in section 7.2.2 of this method.

8.0 Sample Collection, Preservation, Storage, and Transport.

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

8.1 Pretest preparation. It is suggested that sampling equipment be maintained according to the procedures described in APTD-0576.

8.1.1 Weigh several 200 g to 300 g portions of silica gel in airtight containers to the nearest 0.5 g. Record on each container the total weight of the silica gel plus 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.

8.1.2 A batch of glass fiber filters, no more than 50 at a time, should placed in a soxhlet extraction apparatus and extracted using methylene chloride for at least 16 hours. After extraction, check filters visually against light for irregularities, flaws, or pinhole leaks. Label the shipping containers (glass or plastic petri dishes), and keep the filters in these containers at all times except during sampling and weighing.

8.1.3 Desiccate the filters at 20 ± 5.6°C (68 ± 10°F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 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 longer than 2 minutes and a relative humidity above 50 percent. Alternatively (unless otherwise specified by the Administrator), the filters may be oven-dried at 104°C (220°F) for 2 to 3 hours, desiccated 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.

8.2 Preliminary determinations.

8.2.1 Select the sampling site and the minimum number of sampling points according to Method 1, 40 CFR part 60, appendix A or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2, 40 CFR part 60, appendix A; it is recommended that a leak check of the pitot lines (see section 8.1 of Method 2, 40 CFR part 60, appendix A) be performed. Determine the moisture content using Approximation Method 4 (section 1.2 of Method 4, 40 CFR part 60, appendix A) or its alternatives to make isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in section 8.6 of Method 2, 40 CFR part 60, appendix A; 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 particulate sample run.

8.2.2 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 8.2 of Method 2, 40 CFR part 60, appendix A).

8.2.3 Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the required probe length.

8.2.4 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 as 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.

8.2.5 The sampling time at each point shall be the same. 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 eliminate timekeeping errors.

8.2.6 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.

8.3 Preparation of sampling train.

8.3.1 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. 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.

8.3.2 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 to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

8.3.3 When glass liners are used, install the selected nozzle using a Viton A 0-ring when stack temperatures are less than 260°C (500°F) and an asbestos string gasket when temperatures are higher. See APTD-0576 for details. Mark the probe with heat-resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.3.4 Set up the train as in Figure 5-1 of Method 5, 40 CFR part 60, appendix A, using (if necessary) a very light coat of silicone grease on all ground glass joints, greasing only the outer portion (see APTD-0576) to avoid possibility of contamination by the silicone grease. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

8.3.5 Place crushed ice around the impingers.

8.4 Leak-check procedures.

8.4.1 Leak check of metering system shown in Figure 5-1 of Method 5, 40 CFR part 60, appendix A. 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 5-2 of Method 5, 40 CFR part 60, appendix A): 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 1 minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

8.4.2 Pretest leak check. A pretest leak-check is recommended but not required. If the pretest leak-check is conducted, the following procedure should be used.

8.4.2.1 After the sampling train has been assembled, turn on and set the filter and probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A 0-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum.

NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.

8.4.2.2 If an asbestos string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm (15 in.) Hg vacuum. (See NOTE in section 8.4.2.1 of this method). Then connect the probe to the train and perform the leak check at approximately 25 mm (1 in.) Hg vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm (15 in.) Hg vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m^3/min (0.02 cfm), whichever is less, are unacceptable.

8.4.2.3 The following leak check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, as this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak check as shown below and start over.

8.4.2.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable) and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and the silica gel from being entrained backward into the third impinger.

8.4.3 Leak checks during sample run. If, during the sampling run, a component (e.g., filter assembly or impinger) change becomes necessary, a leak check shall be conducted immediately before the change is made. The leak check shall be done according to the procedure outlined in section 8.4.2 of this method, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, either record the leakage rate and plan to correct the sample volume as shown in section 12.3 of this method or void the sample run.

NOTE: Immediately after component changes, leak checks are optional; if such leak checks are done, the procedure outlined in section 8.4.2 of this method should be used.

8.4.4 Post-test leak check. A leak check is mandatory at the conclusion of each sampling run. The leak check shall be performed in accordance with the procedures outlined in section 8.4.2 of this method, except that it shall 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 m^3/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, either record the leakage rate and correct the sample volume, as shown in section 12.4 of this method, or void the sampling run.

8.5 Sampling train operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a temperature around the filter of 120 ± 14°C (248 ± 25°F), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

8.5.1 For each run, record the data required on a data sheet such as the one shown in Figure 5-2 of Method 5, 40 CFR part 60, appendix A. Be sure to record the initial reading. Record the DGM 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 indicated by Figure 5-2 of Method 5, 40 CFR part 60, appendix A 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.

8.5.2 Clean the portholes prior to the test run to minimize the chance of sampling deposited material. To begin sampling, remove the nozzle cap and verify that the filter and probe heating systems are up to temperature and that the pitot tube and probe 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 of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 ± 0.02 and the stack gas equivalent density (dry molecular weight) is 29 ± 4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above-stated ranges, do not use the nomographs unless appropriate steps (see Reference 7 in section 17.0 of this method) are taken to compensate for the deviations.

8.5.3 When the stack is under significant negative pressure (height of impinger stem), close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

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

8.5.5 Traverse the stack cross-section, as required by Method 1, 40 CFR part 60, appendix A 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 through the portholes; this minimizes the chance of extracting deposited material.

8.5.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level; add more ice and, if necessary, salt to maintain a temperature of less than 20°C (68°F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

8.5.7 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 the sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak check (see section 8.4.3 of this method). The total PM weight shall include the summation of the filter assembly catches.

8.5.8 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: When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used in all trains, in which case the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of the front-half catch and one analysis of the impinger catch may be performed.

8.5.9 At the end of the sample run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final DGM reading, and then conduct a post-test leak check, as outlined in section 8.4.4 of this method. Also leak-check the pitot lines as described in section 8.1 of Method 2, 40 CFR part 60, appendix A. The lines must pass this leak check in order to validate the velocity head data.

8.6 Calculation of percent isokinetic. Calculate percent isokinetic (see Calculations, section 12.12 of this method) to determine whether a run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult the Administrator for possible variance on the isokinetic rates.

8.7 Sample recovery.

8.7.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

8.7.2 When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the filter holder, thus drawing water from the impingers into the filter holder.

8.7.3 Before moving the sample train to the cleanup site, remove the probe from the sample train, wipe off the silicone grease, and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Wipe off the silicone grease from the filter inlet where the probe was fastened and cap it. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger or condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser. After wiping off the silicone grease, cap off the filter holder outlet and impinger inlet. Ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

8.7.4 Transfer the probe and filter-impinger assembly 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.

8.7.5 Save a portion of the acetone and methylene chloride used for cleanup as blanks. Take 200 ml of each solvent directly from the wash bottle being used and place it in glass sample containers labeled "acetone blank" and "methylene chloride blank," respectively.

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

8.7.6.1 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 PM cake is inside the fold. Using a dry nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Seal the container.

8.7.6.2 Container No. 2. Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Perform the acetone rinse as follows:

8.7.6.2.1 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 the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

8.7.6.2.2 Brush and rinse the inside parts of the Swagelok fitting with acetone in a similar way until no visible particles remain.

8.7.6.2.3 Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces are wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe, hold a sample container under the lower end of the probe, and catch any acetone and PM that is brushed from the probe. Run the brush through the probe three times or more until no visible PM is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above-described manner at least six times, since metal probes have small crevices in which PM can be entrapped. Rinse the brush with acetone and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe as described above.

8.7.6.2.4 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

8.7.6.2.5 After ensuring that all joints have been wiped clean of silicone grease, 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 a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone also (if applicable).

8.7.6.2.6 After rinsing the nozzle, probe, and front half of the filter holder with acetone, repeat the entire procedure with methylene chloride and save in a separate No. 2M container.

8.7.6.2.7 After acetone and methylene chloride washings and PM have been collected in the proper sample containers, tighten the lid on the sample containers so that acetone and methylene chloride will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to determine whether leakage occurs during transport. Label each container to identify clearly its contents.

8.7.6.3 Container No. 3. Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container and seal the container. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall 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 in section 11.2.3 of this method.

8.7.6.4 Impinger water. Treat the impingers as follows:

8.7.6.4.1 Make a notation of any color or film in the liquid catch. Measure the liquid that is in the first three impingers to within 1 ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas.

8.7.6.4.2 Following the determination of the volume of liquid present, rinse the back half of the train with water, add it to the impinger catch, and store it in a container labeled 3W (water).

8.7.6.4.3 Following the water rinse, rinse the back half of the train with acetone to remove the excess water to enhance subsequent organic recovery with methylene chloride and quantitatively recover to a container labeled 3S (solvent) followed by at least three sequential rinsings with aliquots of methylene chloride. Quantitatively recover to the same container labeled 3S. Record separately the amount of both acetone and methylene chloride used to the nearest 1 ml or 0.5g.

NOTE: Because the subsequent analytical finish is gravimetric, it is okay to recover both solvents to the same container. This would not be recommended if other analytical finishes were required.

8.8 Sample transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

9.0 Quality Control.

9.1 Miscellaneous quality control measures.

Section	Quality Control Measure	Effect
8.4, 10.1-10.6	Sampling and equipment leak check and calibration	Ensure accurate measurement of stack gas flow rate, sample volume

9.2 Volume metering system checks. The following quality control procedures are suggested to check the volume metering system calibration values at the field test site prior to sample collection. These procedures are optional.

9.2.1 Meter orifice check. Using the calibration data obtained during the calibration procedure described in section 10.3 of this method, determine the $\Delta H_@$ for the metering system orifice. The $\Delta H_@$ is the orifice pressure differential in units of in. H_20 that correlates to 0.75 cfm of air at 528°R and 29.92 in. Hg. The $\Delta H_@$ is calculated as follows:

$$\Delta H_@ = 0.0319\ \Delta H\ \frac{T_m\ \Theta^2}{P_{bar}\ Y^2\ V_m^2}$$

where

0.0319 = (0.0567 in. Hg/°R)(0.75 cfm)^2;

ΔH = Average pressure differential across the orifice meter, in. H_20;

T_m = Absolute average DGM temperature, °R;

Θ = Total sampling time, min;

P_{bar} = Barometric pressure, in. Hg;

Y = DGM calibration factor, dimensionless;
V_m = Volume of gas sample as measured by DGM, dcf.

9.2.1.1 Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (i.e., pump, volume meter, and orifice) at the $\Delta H_@$ pressure differential for 10 minutes. Record the volume collected, the DGM temperature, and the barometric pressure. Calculate a DGM calibration check value, Y_c, as follows:

$$Y_c = \frac{10}{V_m}\left[\frac{0.0319\ T_m}{P_{bar}}\right]^{\frac{1}{2}}$$

where

Y_c = DGM calibration check value, dimensionless;
10 = Run time, min.

9.2.1.2 Compare the Y_c value with the dry gas meter calibration factor Y to determine that: $0.97\,Y < Y_c < 1.03Y$. If the Y_c value is not within this range, the volume metering system should be investigated before beginning the test.

9.2.2 Calibrated critical orifice. A calibrated critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box, may be used as a quality control check by following the procedure of section 16.2 of this method.

10.0 Calibration and Standardization.

NOTE: Maintain a laboratory log of all calibrations.

10.1 Probe nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID 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.

10.2 Pitot tube assembly. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in section 10.1 of Method 2, 40 CFR part 60, appendix A.

10.3 Metering system.

10.3.1 Calibration prior to use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5-5 of Method 5, 40 CFR part 60, appendix A. The wet test meter should have a capacity of 30 liters/revolution (1 ft^3/rev). A spirometer of 400 liters (14 ft^3) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading, as expected in field use, to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the DGM. Also note the barometric pressure and the temperatures of the wet test meter, the inlet of the DGM, and the outlet of the DGM. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.15 m^3 (5 cf) at all orifice settings. Record all the data on a form similar to Figure 5-6 of Method 5, 40 CFR part 60, appendix A, and calculate Y (the DGM calibration factor) and $\Delta H_@$ (the orifice calibration factor) at each orifice setting, as shown on Figure 5-6 of Method 5, 40 CFR part 60, appendix A. Allowable tolerances for individual Y and $\Delta H_@$ values are given in

Figure 5-6 of Method 5, 40 CFR part 60, appendix A. Use the average of the Y values in the calculations in section 12 of this method.

10.3.1.1. 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 m^3/min (0.02 cfm); at the end of the run, take the difference of the measured wet test meter and DGM volumes; divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m^3/min (0.02 cfm).

10.3.2 Calibration after use. 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 DGM calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as previously detailed.

NOTE: Alternative procedures, e.g., rechecking the orifice meter coefficient, may be used, subject to the approval of the Administrator.

10.3.3 Acceptable variation in calibration. If the DGM coefficient values obtained before and after a test series differ by more than 5 percent, either the test series shall 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.

10.4 Probe heater calibration. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

NOTE: The probe heating system shall be calibrated before its initial use in the field.

10.5 Temperature sensors. Use the procedure in section 10.3 of Method 2, 40 CFR part 60, appendix A to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-in-glass thermometers.

10.6 Barometer. Calibrate against a mercury barometer.

11.0 Analytical Procedure.

11.1 Record the data required on a sheet such as the one shown in Figure 315-1 of this method.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1.

11.2.1.1 PM analysis. Leave the contents in the shipping container or transfer the filter and any loose PM 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, 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 (overnight desiccation is a common practice). If a third weighing is required and it agrees within ±0.5 mg, then the results of the second weighing should be used. For quality assurance purposes, record and report each individual weighing; if more than three weighings are required, note this in the results for the subsequent MCEM results.

11.2.1.2 MCEM analysis. Transfer the filter and contents quantitatively into a beaker. Add 100 ml of methylene chloride and cover with aluminum foil. Sonicate for 3 minutes then allow to stand for 20 minutes. Set up the filtration apparatus. Decant the solution into a clean Buchner fritted funnel. Immediately pressure filter the solution through the tube into another clean, dry beaker. Continue decanting and pressure filtration until all the solvent is transferred. Rinse the beaker and filter with 10 to

20 ml methylene chloride, decant into the Buchner fritted funnel and pressure filter. Place the beaker on a low-temperature hot plate (maximum 40°C) and slowly evaporate almost to dryness. Transfer the remaining last few milliliters of solution quantitatively from the beaker (using at least three aliquots of methylene chloride rinse) to a tared clean dry aluminum dish and evaporate to complete dryness. Remove from heat once solvent is evaporated. Reweigh the dish after a 30-minute equilibrium in the balance room and determine the weight to the nearest 0.1 mg. Conduct a methylene chloride blank run in an identical fashion.

11.2.2 Container No. 2.

11.2.2.1 PM analysis. Note the level of liquid in the container, and confirm on the analysis sheet whether 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.

11.2.2.2 MCEM analysis. Add 25 ml methylene chloride to the beaker and cover with aluminum foil. Sonicate for 3 minutes then allow to stand for 20 minutes; combine with contents of Container No. 2M and pressure filter and evaporate as described for Container 1 in section 11.2.1.2 of this method.

NOTES FOR MCEM ANALYSIS:

1. Light finger pressure only is necessary on 24/40 adaptor. A Chemplast adapter #15055-240 has been found satisfactory.

2. Avoid aluminum dishes made with fluted sides, as these may promote solvent "creep," resulting in possible sample loss.

3. If multiple samples are being run, rinse the Buchner fritted funnel twice between samples with 5 ml solvent using pressure filtration. After the second rinse, continue the flow of air until the glass frit is completely dry. Clean the Buchner fritted funnels thoroughly after filtering five or six samples.

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

11.2.4 Container 3W (impinger water).

11.2.4.1 MCEM analysis. Transfer the solution into a 1,000 ml separatory funnel quantitatively with methylene chloride washes. Add enough solvent to total approximately 50 ml, if necessary. Shake the funnel for 1 minute, allow the phases to separate, and drain the solvent layer into a 250 ml beaker. Repeat the extraction twice. Evaporate with low heat (less than 40°C) until near dryness. Transfer the remaining few milliliters of solvent quantitatively with small solvent washes into a clean, dry, tared aluminum dish and evaporate to dryness. Remove from heat once solvent is evaporated. Reweigh the dish after a 30-minute equilibration in the balance room and determine the weight to the nearest 0.1 mg.

11.2.5 Container 3S (solvent).

11.2.5.1 MCEM analysis. Transfer the mixed solvent to 250 ml beaker(s). Evaporate and weigh following the procedures detailed for container 3W in section 11.2.4 of this method.

11.2.6 Blank containers. Measure the distilled water, acetone, or methylene chloride in each container either volumetrically or gravimetrically. Transfer the "solvent" to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. (Conduct a solvent blank on the distilled deionized water blank in an identical fashion to that described in section 11.2.4.1 of this method.) Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE: The contents of Containers No. 2, 3W, and 3M as well as the blank containers 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 swirled occasionally to maintain an even temperature. Use extreme care, as acetone and methylene chloride are highly flammable and have a low flash point.

12.0 Data Analysis and Calculations.

12.1 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.

12.2 Nomenclature.

A_n = Cross-sectional area of nozzle, m^3 (ft^3).
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_a = Acetone blank residue concentration, mg/g.
C_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
I = Percent of isokinetic sampling.
L_a = Maximum acceptable leakage rate for either a pretest leak check or for a leak check following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.
L_i = Individual leakage rate observed during the leak check conducted prior to the "ith" component change (I = 1, 2, 3...n), m^3/min (cfm).
L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).
m_a = Mass of residue of acetone after evaporation, mg.
m_n = Total amount of particulate matter collected, mg.
M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
P_{bar} = 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, 0.06236 [(mm Hg)(m^3)]/[(°K)(g-mole)] {21.85 [(in. Hg)(ft^3)]/[(°R)(lb-mole)]}.
T_m = Absolute average dry gas meter (DGM) temperature (see Figure 5-2 of Method 5, 40 CFR part 60, appendix A), °K (°R).
T_s = Absolute average stack gas temperature (see Figure 5-2 of Method 5, 40 CFR part 60, appendix A), °K(°R).
T_{std} = Standard absolute temperature, 293°K (528°R).
V_a = Volume of acetone blank, ml.
V_{aw} = Volume of acetone used in wash, ml.
V_t = Volume of methylene chloride blank, ml.
V_{tw} = Volume of methylene chloride used in wash, ml.
V_{lc} = Total volume liquid collected in impingers and silica gel (see Figure 5-3 of Method 5, 40 CFR part 60, appendix A), ml.
V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).
$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).
$V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
V_s = Stack gas velocity, calculated by Equation 2-9 in Method 2, 40 CFR part 60, appendix A, using data obtained from Method 5, 40 CFR part 60, appendix A, m/sec (ft/sec).
W_a = Weight of residue in acetone wash, mg.
Y = Dry gas meter calibration factor.
ΔH = Average pressure differential across the orifice meter (see Figure 5-2 of Method 5, 40 CFR part 60, appendix A), mm H_20 (in H_20).
ρ_a = Density of acetone, 785.1 mg/ml (or see label on bottle).
ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).
ρ_t = Density of methylene chloride, 1316.8 mg/ml (or see label on bottle).
θ = Total sampling time, min.
θ_1 = Sampling time interval, from the beginning of a run until the first component change,

min.

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

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

13.6 = Specific gravity of mercury.

60 = Sec/min.

100 = Conversion to percent.

12.3 Average dry gas meter temperature and average orifice pressure drop. See data sheet (Figure 5-2 of Method 5, 40 CFR part 60, appendix A).

12.4 Dry gas volume. Correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in Hg) by using Equation 315-1.

$$V = V_m Y \frac{T_{std}\left(P_{bar} + \frac{\Delta H}{13.6}\right)}{T_m P_{std}} \qquad \text{Eq. 315-1}$$

$$= V = K_1 V_m Y \frac{P_{bar} + \left(\frac{\Delta H}{13.6}\right)}{T_m}$$

where

K_1 = 0.3858 °K/mm Hg for metric units,

= 17.64 °R/in Hg for English units.

NOTE: Equation 315-1 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_a. If L_p or L_i exceeds L_a, Equation 315-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 315-1 with the expression:

$$[V_m - (L_p - L_a)\,\Theta]$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation
315-1 by the expression:

$$[V_m - (L_1 - L_a)\,\Theta_1 - \sum_{i=2}^{n} (L_i - L_a)\,\Theta_i - (L_p - L_a)\,\Theta_p]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a.

12.5 Volume of water vapor condensed.

$$V_{w(std)} = V_{lc}\frac{\rho_w R T_{std}}{M_w P_{std}} = K_2 V_{lc}$$

Eq. 315-2

where

K_2 = 0.001333 m^3/ml for metric units;

= 0.04706 ft^3/ml for English units.

12.6 Moisture content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}}$$

Eq. 315-3

NOTE: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger analysis (Equation 315-3), and a second from the assumption of saturated conditions. The lower of the two values of B_{ws} shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in section 4.0 of Method 4, 40 CFR part 60, appendix A. For the purposes of this method, the average stack gas temperature from Figure 5-2 of Method 5, 40 CFR part 60, appendix A may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is ±1°C (2°F).

12.7 Acetone blank concentration.

$$C_a = \frac{M_a}{V_a \rho_a}$$

Eq. 315-4

12.8 Acetone wash blank.

$$W_a = C_a V_{aw} \rho_a$$

Eq. 315-5

12.9 Total particulate weight. Determine the total PM catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank associated with these two containers (see Figure 315-1).

NOTE: Refer to section 8.5.8 of this method to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

12.10 Particulate concentration.

$$c_s = K_3 m_n / V_{m(std)}$$

Eq. 315-6

where

K = 0.001 g/mg for metric units;

= 0.0154 gr/mg for English units.

12.11 Conversion factors.

From	To	Multiply by
ft^3	m^3	0.02832
gr	mg	64.80004
gr/ft^3	mg/m3	2288.4
mg	g	0.001
gr	lb	1.429×10^{-4}

12.12 Isokinetic variation.

12.12.1 Calculation from raw data.

$$I = \frac{100\ T_s \left[K_4\ V_{lc} + \left(\frac{V_m\ Y}{T_m}\right)\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{60\ \Theta\ v_s\ P_s\ A_n} \qquad \text{Eq. 315-7}$$

where

K_4 = 0.003454 [(mm Hg)(m^3)]/[(ml)(°K)] for metric units;
= 0.002669 [(in Hg)(ft^3)]/[(ml)(°R)] for English units.

12.12.2 Calculation from intermediate values.

$$I = \frac{T_s\ V_{m(std)}\ P_{std}\ 100}{T_{std}\ v_s\ \Theta\ A_n\ P_s\ 60\ (1-B_{ws})} \qquad \text{Eq. 315-8}$$

$$= K_5 \frac{T_s\ V_{m(std)}}{P_s\ v_s\ A_n\ \Theta\ (1-B_{ws})}$$

where

K_5 = 4.320 for metric units;
= 0.09450 for English units.

12.12.3 Acceptable results. If 90 percent ≤ I ≤ 110 percent, the results are acceptable. If the PM or MCEM results are low in comparison to the standard, and "I" is over 110 percent or less than 90 percent, the Administrator may opt to accept the results. Reference 4 in the Bibliography may be used to make acceptability judgments. If "I" is judged to be unacceptable, reject the results, and repeat the test.

12.13 Stack gas velocity and volumetric flow rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in sections 5.2 and 5.3 of Method 2, 40 CFR part 60, appendix A.

12.14 MCEM results. Determine the MCEM concentration from the results from Containers 1, 2, 2M, 3W, and 3S less the acetone, methylene chloride, and filter blanks value as determined in the following equation:

$$m_{mcem} = \Sigma m_{total} - w_a - w_t - f_b$$

13.0 Method Performance. [Reserved]
14.0 Pollution Prevention. [Reserved]

15.0 Waste Management. [Reserved]

16.0 Alternative Procedures.

16.1 Dry gas meter as a calibration standard. A DGM may be used as a calibration standard for volume measurements in place of the wet test meter specified in section 16.1 of this method, provided that it is calibrated initially and recalibrated periodically as follows:

16.1.1 Standard dry gas meter calibration.

16.1.1.1. The DGM to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity, e.g., 3 liters/rev (0.1 ft^3/rev). A spirometer (400 liters or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft^3/rev) and be capable of measuring volume to within 1.0 percent; wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained.

16.1.1.2 Set up the components as shown in Figure 5-7 of Method 5, 40 CFR part 60, appendix A. A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the DGM should be minimized (no greater than 100 mm H_2O [4 in. H_2O] at a flow rate of 30 liters/min [1 cfm]). This can be accomplished by using large- diameter tubing connections and straight pipe fittings.

16.1.1.3 Collect the data as shown in the example data sheet (see Figure 5-8 of Method 5, 40 CFR part 60, appendix A). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

16.1.1.4 Calculate flow rate, Q, for each run using the wet test meter volume, V_w, and the run time, q. Calculate the DGM coefficient, Y_{ds}, for each run. These calculations are as follows:

$$Q = K_1 \frac{P_{bar} V_w}{(t_w + t_{std}) \Theta} \qquad \text{Eq. 315-9}$$

$$Y_{ds} = \frac{V_w (T_{ds} + T_{std}) P_{bar}}{V_{ds} (T_w + T_{std})(P_{bar} + \frac{\Delta p}{13.6})} \qquad \text{Eq. 315-10}$$

where

K_1 = 0.3858 for international system of units (SI);
17.64 for English units;
P_{bar} = Barometric pressure, mm Hg (in Hg);
V_w = Wet test meter volume, liter (ft^3);
t_w = Average wet test meter temperature, °C (°F);
t_{std} = 273°C for SI units; 460°F for English units;
Θ = Run time, min;
t_{ds} = Average dry gas meter temperature, °C (°F);
V_{ds} = Dry gas meter volume, liter (ft^3);
Δp = Dry gas meter inlet differential pressure, mm H_2O (in H_2O).

16.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Y_{ds} values at each flow rate resulting in five average meter coefficients, Y_{ds}.

16.1.1.6 Prepare a curve of meter coefficient, Y_{ds}, versus flow rate, Q, for the DGM. This curve shall be used as a reference when the meter is used to calibrate other DGMs and to determine whether recalibration is required.

16.1.2 Standard dry gas meter recalibration.

16.1.2.1 Recalibrate the standard DGM against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard DGM is kept in a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

16.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates (suggested rates are 14 and 28 liters/min [0.5 and 1.0 cfm]). Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within 1.5 percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

16.2 Critical orifices as calibration standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in section 10.3 of this method, provided that they are selected, calibrated, and used as follows:

16.2.1 Selection of critical orifices.

16.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubing that has been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices; i.e., a critical vacuum can be obtained, as described in section 7.2.2.2.3 of Method 5, 40 CFR part 60, appendix A. Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min or the expected operating range. Two of the critical orifices should bracket the expected operating range. A minimum of three critical orifices will be needed to calibrate a Method 5 DGM; the other two critical orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown in Table 315-1 give the approximate flow rates indicated in the table.

16.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13 x 20 mm sleeve type, into a 0.5 in Swagelok quick connect. Insert the needle into the stopper as shown in Figure 5-9 of Method 5, 40 CFR part 60, appendix A.

16.2.2 Critical orifice calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in section 6.1.1.9 of this method to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

16.2.2.1 Calibration of meter box. The critical orifices must be calibrated in the same configuration as they will be used; i.e., there should be no connections to the inlet of the orifice.

16.2.2.1.1 Before calibrating the meter box, leak-check the system as follows: Fully open the coarse adjust valve and completely close the bypass valve. Plug the inlet. Then turn on the pump and determine whether there is any leakage. The leakage rate shall be zero; i.e., no detectable movement of the DGM dial shall be seen for 1 minute.

16.2.2.1.2 Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See section 5.6 of Method 5, 40 CFR part 60, appendix A for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn 0-rings, etc. and make the necessary repairs.

16.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in section 5.3 of Method 5, 40 CFR part 60, appendix A. Make sure that the wet test meter meets the requirements stated in section 7.1.1.1 of Method 5, 40 CFR part 60, appendix A. Check the water level in the wet test meter. Record the DGM calibration factor, Y.

16.2.2.2 Calibration of critical orifices. Set up the apparatus as shown in Figure 5-10 of Method 5, 40 CFR part 60, appendix A.

16.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

16.2.2.2.2 Leak-check the system as in section 7.2.2.1.1 of Method 5, 40 CFR part 60, appendix A. The leakage rate shall be zero.

16.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: turn on the pump, fully open the coarse adjust valve, and adjust the bypass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, DH. Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice. Orifices that do not reach a critical value shall not be used.

16.2.2.2.4 Obtain the barometric pressure using a barometer as described in section 6.1.2 of this method. Record the barometric pressure, P_{bar}, in mm Hg (in. Hg).

16.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve ±0.5 percent in K'. Record the information listed in Figure 5-11 of Method 5, 40 CFR part 60, appendix A.

16.2.2.2.6 Calculate K' using Equation 315-11.

$$K' = \frac{K_1 \; V_m \; Y \; (P_{bar} + \frac{\Delta H}{13.6}) \; T_{amb}^{\frac{1}{2}}}{P_{bar} \; T_m \; \Theta} \qquad \text{Eq. 315-11}$$

where

K' = Critical orifice coefficient, $[(m^3)(°K)^{½}]/[(mm\ Hg)(min)]$ $\{[(ft^3)(°R)^{½}]/[(in.\ Hg)(min)]\}$;

T_{amb} = Absolute ambient temperature, °K (°R).

16.2.2.2.7 Average the K' values. The individual K' values should not differ by more than ±0.5 percent from the average.

16.2.3 Using the critical orifices as calibration standards.

16.2.3.1 Record the barometric pressure.

16.2.3.2 Calibrate the metering system according to the procedure outlined in sections 7.2.2.2.1 to 7.2.2.2.5 of Method 5, 40 CFR part 60, appendix A. Record the information listed in Figure 5-12 of Method 5, 40 CFR part 60, appendix A.

16.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y, using the equations below:

$$V_{m(std)} = K_1 \, V_m \, [P_{bar} + (\Delta H/13.6)]/T_m \qquad \text{Eq. 315-12}$$

$$V_{cr(std)} = K' \, (P_{bar} \, \Theta)/T_{amb}^{1/2} \qquad \text{Eq. 315-13}$$

$$Y = V_{cr(std)}/V_{m(std)} \qquad \text{Eq. 315-14}$$

where

$V_{cr(std)}$ = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dscm (dscf).

K' = 0.3858 °K/mm Hg for metric units

= 17.64 °R/in Hg for English units.

16.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration factor, Y, at each of the flow rates should not differ by more than ±2 percent from the average.

16.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking orifice 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to section 7.2.2.2 of Method 5, 40 CFR part 60, appendix A.

17.0 References.

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18.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 315-1. Flow Rates for Various Needle Sizes and Tube Lengths.

Gauge/length (cm)	Flow rate (liters/min)	Gauge/length (cm)	Flow rate (liters/min)
12/7.6	32.56	14/2.5	19.54
12/10.2	30.02	14/5.1	17.27
13/2.5	25.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.61
13/10.2	20.67	15/10.2	10.48

Particulate analysis			
Plant			
Date			
Run No.			
Filter No.			
Amount liquid lost during transport			
Acetone blank volume (ml)			
Acetone blank concentration (Eq.315-4) (mg/mg)			
Acetone wash blank (Eq.315-5) (mg)			
	Final weight (mg)	Tare weight (mg)	Weight gain (mg)
Container No. 1			
Container No. 2			
Total			
Less Acetone blank			
Weight of particulate matter			
Moisture analysis			
	Final volume (mg)	Initial volume (mg)	Liquid collected (mg)
Impingers	Note 1	Note 1	
Silica gel			
Total			

FIGURE 315-1. Particulate and MCEM Analyses

Note 1: Convert volume of water to weight by multiplying by the density of water (1 g/ml).

MCEM analysis					
Container No.	Final weight (mg)	Tare of aluminum dish (mg)	Weight gain	Acetone wash volume (ml)	Methylene chloride wash volume (ml)
1					
2+2M					
3W					
3S					
Total			$\sum m_{total}$	$\sum v_{aw}$	$\sum v_{tw}$
Less acetone wash blank (mg) (not to exceed 1 mg/l of acetone used)			$w_a = c_a \rho_a \sum v_{aw}$		
Less methylene chloride wash blank (mg) (not to exceed 1.5 mg/l of methylene chloride used)			$w_t = c_t \rho_t \sum v_{tw}$		
Less filter blank (mg) (not to exceed.... (mg/filter)			F_b		
MCEM weight (mg)			$m_{MCEOM} = \sum m_{total} - w_a - w_t - f_b$		

FIGURE 315-1 (Continued). Particulate And MCEM Analyses

* * * * *

State of California
California Environmental Protection Agency
Air Resources Board

Method 429

Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions from Stationary Sources

Adopted: September 12, 1989
Amended: [insert date of amendment]

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APPENDIX A

Method 429

Determination of Polycyclic Aromatic Hydrocarbon (PAH) Emissions From Stationary Sources

1. INTRODUCTION

1.1 APPLICABILITY

This method applies to the determination of nineteen polycyclic aromatic hydrocarbons (PAH) in emissions from stationary sources. These are listed in Table 1. The sensitivity which can ultimately be achieved for a given sample will depend upon the types and concentrations of other chemical compounds in the sample as well as the original sample size and instrument sensitivity.

Any modification of this method beyond those expressly permitted shall be considered a major modification subject to approval by the Executive Officer of the California Air Resources Board or his or her authorized representative.

1.2 PRINCIPLE

Particulate and gaseous phase PAH are extracted isokinetically from the stack and collected on XAD-2 resin, in impingers, or in upstream sampling train components (filter, probe, nozzle). Only the total amounts of each PAH in the stack emissions can be determined with this method. It has not been demonstrated that the partitioning in the different parts of the sampling train is representative of the partitioning in the stack gas sample for particulate and gaseous PAH.

The required analytical method is isotope dilution mass spectrometry combined with high resolution gas chromatography. This entails the addition of internal standards to all samples in known quantities, matrix-specific extraction of the sample with appropriate organic solvents, preliminary fractionation and cleanup of extracts and analysis of the processed extract for PAH using high-resolution capillary column gas chromatography coupled with either low resolution mass spectrometry (HRGC/LRMS), or high resolution mass spectrometry (HRGC/HRMS). To ensure comparable results, the same MS method must be used for samples collected at all tested locations at those sources where more than one location is tested.

Minimum performance criteria are specified herein which must be satisfied to ensure the quality of the sampling and analytical data.

1.3 DEFINITIONS AND ABBREVIATIONS

1.3.1 Internal Standard

An internal standard is a ^{2}H-labelled PAH which is added to all field samples, blanks and other quality control samples before extraction. It is also present in the calibration solutions. Internal standards are used to measure the concentration of the analyte and surrogate compounds. There is one internal standard assigned to each of the target analytes and surrogates.

1.3.2 Surrogate Standard

A surrogate standard is a labelled compound added in a known amount to the XAD-2 resin of the sampling train, and allowed to equilibrate with the matrix before the gaseous emissions are sampled. The surrogate standard has to be a component that can be completely resolved, is not present in the sample, and does not have any interference effects. Its measured concentration in the extract is an indication of the how effectively the sampling train retains PAH collected on the XAD-2 resin. The recovery of the surrogate standards in the field blanks can be used to determine whether there are any matrix effects caused by time or conditions under which the sample is transported and stored prior to analysis.

1.3.3 Alternate Standard

An alternate standard is a ^{2}H-labelled PAH compound which is added to the impinger contents prior to extraction to estimate the extraction efficiency for PAHs in the impinger sample.

1.3.4 Recovery Standard

A recovery standard is a ^{2}H-labelled PAH compound which is added to the extracts of all field samples, blanks, and quality control samples before HRGC/MS analysis. It is also present in the calibration solution. The response of the internal standards relative to the recovery standard is used to estimate the recovery of the internal standards. The internal standard recovery is an indicator of the overall performance of the analysis.

1.3.5 Relative Response Factor

The relative response factor is the response of the mass spectrometer to a known amount of an analyte or labelled compound (internal standard or surrogate standard) relative to a known amount of an internal standard or another labelled compound (recovery standard or internal standard).

1.3.6 Performance Standard

A performance standard is a mixture of known amounts of selected standard compounds. It is used to demonstrate continued acceptable performance of the GC/MS system. These checks include system performance checks, calibration checks, quality checks, matrix recovery, and surrogate recoveries.

1.3.7 Performance Evaluation Sample

A performance evaluation sample is one prepared by EPA or other laboratories that contains known concentrations of method analytes, and has been analyzed by multiple laboratories to determine statistically the accuracy and precision that can be expected when a method is performed by a competent analyst. Concentrations must be in the same range as typical field samples. Analyte concentrations are not known by the analyst.

1.3.8 Laboratory Control Sample

A laboratory control sample is one that contains known concentrations of method analytes that is analyzed by a laboratory to demonstrate that it can obtain acceptable identifications and measurements with procedures to be used to analyze field samples containing the same analytes. Analyte concentrations are known by the analyst. The laboratory must prepare the control sample from stock standards prepared independently from those used for calibration.

1.3.9 End User

The regulating agency shall be considered the end user if this test method is conducted for regulatory purposes, or the regulating agency shall designate the end user for the purposes of this method. Otherwise the end user shall be the party who defrays the cost of performing this test method. In any case, the pre-test protocol (Section 2) must identify the end user.

1.3.10 Tester

Usually the tester is a contract engineering firm that performs the sampling procedures and delegates responsibility for specific analytical procedures to an analytical group (usually part of a subcontracting laboratory firm). In some cases, the tester may be part of the regulating agency. The tester shall be the party ultimately responsible for the performance of this test method whether directly or indirectly through the co-ordination of the efforts of the analytical group and the efforts of the sampling group.

1.3.11 Analyst

This term refers to the analytical group that performs the analytical procedures to generate the required analytical data.

1.3.12 Source Target Concentration

This is the target concentration for each emitted PAH of interest specified by the end user of the test results. The target concentration shall be expressed in units of mass of target substance per volume of emissions; typical units are nanograms per dry standard cubic meter or micrograms per dry standard cubic meter (ng/dscm or μg/dscm)

1.3.13 The Method Detection Limit

The method detection limit (MDL) is based on the precision of detection of the analyte concentration near the detection limit. It is the product of the standard deviation of seven replicate analyses of resin samples spiked with low concentrations of the analyte and Student's t value for 6 degrees of freedom at a confidence level of 99 %.

1.3.14 The Practical Quantitation Limit

The practical quantitation limit (PQL) is a limit for each compound at or below which data must not be reported. It is the minimum sample mass that must be collected in the sampling train to allow detection during routine laboratory operation within the precision limits established by the MDL determination. The PQLs will be estimated at 5 times the MDL for those PAH that are not contaminants of the resin. The PQL for the remainder will be estimated at 5 times the blank XAD-2 resin level.

2. THE SOURCE TEST PROTOCOL

Every performance of this test method shall have an identified operator of the source to be tested, an identified end user of the test method results, and an identified tester who performs this test method. Figure 1 is a summary of the responsibilities of the parties involved in the coordination and performance of the source test. The protocol for the entire test procedure should be understood and agreed upon by the responsible parties prior to the start of the test.

2.1 RESPONSIBILITIES OF THE END USER AND THE TESTER

2.1.1 The End User

Before testing may begin, the end user of the test results (1.3.9) shall specify a source target concentration for each of the PAH to be determined by this method using the guidelines of Section 2.2.1.

The end user shall approve the source test protocol only after reviewing the document and determining that the minimum pre-test requirements (Sections 2.2 to 2.5) have been met.

2.1.2 The Tester

The tester (1.3.10) shall have the primary responsibility for the performance of the test method, and shall co-ordinate the efforts of the analytical group and the efforts of the sampling group.

The tester shall be responsible for the selection of an analyst with documented experience in the satisfactory performance of the method. The tester shall obtain from the analyst all of the analytical data (Section 2.3) that are required for pre-test calculations of sampling parameters.

Before performing the rest of this method, the tester shall develop and write a source test protocol (Section 2.2) to help ensure that useful test method results are obtained. The tester shall plan the test based on the information provided by the end user, the results of pre-test surveys of the source, and the tester's calculations of target source testing parameters (Section 2,2).

The tester shall be responsible for ensuring that all of the sampling and analytical reporting requirements (Section 10) are met.

2.1.3 The Analyst

The analyst shall be responsible for performing all of the required analytical procedures described in this test method and reporting the results as required by Sections 2.3, 4.2.1, 4.2.2, 10.1.1, 10.1.2, 10.1.3, and 10.2).

2.2 PRE-TEST REQUIREMENTS

The source test protocol shall specify the test performance criteria of the end user and all assumptions, required data and calculated targets for the following testing parameters:

(1) source target concentration of each emitted PAH of interest (2.2.1),

(2) preliminary analytical data (2.3) for each target PAH, and

(3) planned sampling parameters (2.5.4, 2.5.5, and 2.5.6).

The protocol must demonstrate that the testing parameters calculated by the tester will meet the needs of the end user. The source test protocol shall describe the procedures for all aspects of the source test including information on supplies, logistics, personnel and other resources necessary for an efficient and coordinated test.

The source test protocol shall identify the end user of the results, the tester, the analytical group, and the sampling group, and the protocol shall be signed by the end user of the results and the tester.

The tester shall not proceed with the performance of the remainder of this method unless the source test protocol is signed by the tester and the end user.

2.2.1 Source Target Concentration (STC)

The tester shall not proceed with the test unless a target concentration has been chosen. This will be the primary reporting objective of the emissions test. The end user shall select a basis for determining each target concentration from: a) regulatory limits, b) environmental risk assessments, and (c) the interests of the end user, the tester, and the stationary source.

2.2.1.1 Regulatory Limits

The regulatory limit shall be the basis for determining a target concentration for stationary source emissions in those cases where the purpose of the emissions test is to demonstrate compliance with the established regulatory limit.

2.2.1.2 Environmental Risk Assessments

In some cases testing is conducted for an environmental risk assessment. A pre-test estimate of the permissible risk shall then be used to determine the target concentration for stationary source emissions.

Note that some risk assessment methodologies will assume that a PAH is present at the detection limit or one half of the detection limit even when the compound is not detected. This is inappropriate for planning for the performance of the test method because by definition a substance cannot be detected at one half of its detection limit. In such cases, the target sampling parameter must be the maximum practical sample volume.

2.2.1.3 Interests of the End User, the Tester and the Stationary Source

In cases where the emissions test is not being performed to demonstrate compliance with a regulation, nor is it required for a risk assessment, the end user may use emissions results from previous tests of the facility or from similar facilities.

If estimates of the emissions are not availble, the tester must conduct a preliminary test at each emissions point of interest. This target concentration is necessary for the calculation of the target sampling parameters required by Section 2.5. Therefore, the emissions measured during the preliminary test must be representative of source operation. The tester must document operating conditions, and know from historical data, the extent to which the results of this preliminary run are representative of emissions from the source. This will require documentation of operating conditions during the preliminary test, and a knowledge of the potential variability in emissions with differences in source operation.

As an alternative to conducting a preliminary test, the end user may specify, as a sampling target, the longest practical sampling time so as to obtain the lowest practically achievable source reporting limit (Section 2.5.6).

2.3 REQUIRED PRELIMINARY ANALYTICAL DATA

2.3.1 Results of Blank Contamination Checks

The tester must obtain from the analyst the results of the PAH contamination checks. The analytical report must satisfy the reporting requirements of Sections 10 and 10.1.

The analyst shall use the procedures described in Sections 4.2.1 and 4.2.2 to clean the sampling media (filters and XAD-2 resin) and check for PAH contamination.

Table 3 shows the results of analyses of different lots of re-cleaned XAD-2 resin. The purpose of this table is to show typical variability. Actual results may vary from one test to another.

2.3.2 The Method Detection Limit

The method detection limit (MDL) must be determined by the same analyst (1.3.11) that will perform the analyses subsequent to sampling. Before estimating the method detection limit (MDL), the analyst shall identify those PAH that are contaminants of the XAD-2 resin using the procedures described in Sections 4.2.2.1 to 4.2.2.4. The analyst shall determine the MDL as described in Section 8.3 and Appendix A.

2.3.3 The Practical Quantitation Limit

The analyst shall calculate the practical quantitation limits (PQLs) for the target PAH. This value will be 5 times the MDL or 5 times the XAD-2 background level for those compounds that have been identified by the analyst as contaminants.

Table 2 lists practical quantitation limits obtained during ARB's development of this method. The values for the PQLs will vary with the performance of individual laboratories. Therefore, the tester must obtain PQL values for all of the target analytes from the analyst .

2.4 EXPECTED RANGE IN TARGET CONCENTRATIONS OF INDIVIDUAL PAHs

The PAH compounds in a source test sample can show large differences in concentrations. A sample that might provide sufficient analyte for the detection and quantitation of the lowest concentration PAH could contain levels of other PAHs that exceed the upper limit of the method.

In some cases the solution is two GC/MS injections - first with the undiluted extract, and then again after appropriate dilution of the extract. At other times the required minimum dilution might be so large as to result in the reduction of the internal standard response below the minimum required by the method. With prior notification of expected levels of the target analytes, the analyst can modify the preparation of the samples so that useful results might be obtained. All major modifications must be approved by the Executive Officer.

2.5 SAMPLING RUNS, TIME, AND VOLUME

2.5.1 Sampling Runs

A test shall include at least three sampling runs in series and a blank sampling train.

2.5.2 Minimum Sample Volume (MSV)

This is the minimum sample volume that must be collected in the sampling train to provide the minimum reportable mass of PAH for quantitation. It must be based on a) the practical quantitation limit (2.3.3), b) the source target concentration (2.2.1), and c) sampling limitations. Use Equation 429-1 to calculate the target MSV for each PAH analyte.

$$MSV(dscm) = PQL \times \frac{1}{STC} \qquad 429\text{-}1$$

Where:

PQL = The practical quantitation limit, ng/sample (Section 2.3.3)
STC = The source target concentration, ng/dscm (Section 2.2.1)

2.5.3 Minimum Sampling Time (MST)

This is the minimum time required to collect the minimum sample volume at the expected average volumetric sampling rate (VSR). Use Equation 429-2 to calculate the minimum sampling time (MST) required to collect the minimum sample volume calculated in Section 2.5.2. The tester must use an average volumetric sampling rate (VSR) appropriate for the source to be tested.

$$MST(hours) = \frac{MSV}{VSR} \times \frac{1}{0.028317} \times \frac{1}{60} \qquad 429\text{-}2$$

Where:

VSR = Expected average volumetric sampling rate, dscfm
60 = Factor to convert minutes to hours
0.028317 = Factor to convert dscf to dscm

The end user must decide whether the MSTs are all practically feasible and whether they can be increased to allow for any deviation from the sampling and analytical conditions assumed by the test plan. Based on this decision, the tester must use either Section 2.5.4 (a) or 2.5.4 (b) to calculate a planned sample volume (PSV).

2.5.4 Planned Sample Volume (PSV)

This is the volume of emissions that must be sampled to provide the target analytes at levels between the PQL and the limit of linearity. The planned sample volume is the primary sampling target whenever practically feasible. The PSV is calculated according to either 2.5.4 (a) or 2.5.4 (b).

(a) If the end user has decided that the MSTs can be increased, the tester must use Equation 429-3 to calculate the PSV using the largest of the 19 MSV values calculated in Section 2.5.2. and the largest value for F that will give a practically achievable sample volume that provides the target analytes at levels between the PQL and the limit of linearity. Use this PSV to calculate the planned sampling time (Section 2.5.5 a) and Equation 429-6.

(b) If the MSTs are not all practically achievable, the tester and the end user must agree on a maximum practical sampling time (Section 2.5.5b). This value must then be used for the PST in Equation 429-4 to calculate the PSV. The tester must identify in the source test protocol the target analytes for which the PSV is lower than the MSV. The primary reporting objective of the test cannot be achieved for those analytes. If the primary reporting objective cannot be achieved for all of the target analytes, it must be discussed in the protocol and the alternative reporting objective (Section 2.5.6) must be approved by the end user of the results.

The volume of sample that is actually collected will be determined by practical sampling limitations, the intended use of the data and the level of uncertainty that the end user can tolerate in the measurement of the target concentrations. This uncertainty will decrease as the value of F (Equation 429-5) increases.

$$PSV(dscm) = MSV \times F \qquad 429\text{-}3$$

$$PSV(dscm) = PST \times VSR \qquad 429\text{-}4$$

$$F = \frac{PSV}{MSV} \qquad 429\text{-}5$$

Where:

PST = Planned sampling time from Section 2.5.5
F = A safety factor (> 1) that allows for deviation from ideal sampling and analytical conditions

2.5.5 Planned Sampling Time (PST)

Two options are available for calculating the planned sampling time depending on whether the primary objective can be achieved for all of the target analytes.

(a) The planned sampling time (PST) shall be long enough to 1) collect the planned sample volume with reportable levels of the target analytes and 2) sample representative operating conditions of the source. If the average sampling rate (VSR) used to estimate the planned sampling time cannot be achieved in the field (Section 4.4.4.1), the sampling time must be recalculated using the actual VSR and the target PSV in equation 429-6.

(b) The planned sampling time shall be a practical maximum approved by the end user and it shall be long enough to sample representative operating conditions of the source.

$$PST(hours) = \frac{PSV}{VSR} \times \frac{1}{0.028317} \times \frac{1}{60} \qquad 429\text{-}6$$

2.5.6 Preliminary Estimate of Source Reporting Limit (SRL)

Before the test proceeds, the end user and the tester shall agree on a preliminary estimate of the source reporting limit for each target PAH. The SRL shall be calculated using Equation 429-7. The planned sample volume will contain reportable levels of a given analyte if that analyte is present in the emissions at a concentration that is equal to or greater than the calculated SRL.

$$SRL(ng/dscm) = \frac{PQL}{PSV} \qquad 429\text{-}7$$

Where:

SRL = Preliminary estimate of source reporting limit, ng/dscm
PQL = Practical quantitation limit, ng
PSV = Planned sample volume, dscm

2.5.7 Example Calculations

Figure 9 B is an example of the minimum required calculations of sampling parameters for the source test protocol.

3. INTERFERENCES

Interferences may be caused by contaminants in solvents, reagents, sorbents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the ions monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 6.1.1.

The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

Transformation of PAH and the formation of artifacts can occur in the sampling train. PAH degradation and transformation on sampling train filters have been demonstrated. Certain reactive PAH such as benzo[a]pyrene, benzo[a]anthracene, and fluoranthene when trapped on filters can readily react with stack gases. These PAH are transformed by reaction with low levels of nitric acid and higher levels of nitrogen oxides, ozone, and sulfur oxides.

PAH degradation may be of even greater concern when they are trapped in the impingers. When stack gases such as sulfur oxides and nitrogen oxides come in contact with the impinger water they are converted into sulfuric acid and nitric acid respectively. There is evidence that under such conditions certain PAH will be degraded. It is recommended that the PAH levels in the impingers be used as a qualitative tool to determine if breakthrough has occurred in the resin.

4. SAMPLING APPARATUS, MATERIALS AND REAGENTS

4.1 SAMPLING APPARATUS

The sampling train components listed below are required. The tester may use an alternative to the required sampling apparatus only if, after review by the Executive Officer, it is deemed equivalent for the purposes of this test method.

Mention of trade names or specific products does not constitute endorsement by the California Air Resources Board. In all cases, equivalent items from other suppliers may be used.

A schematic of the sampling train is shown in Figure 2. The train consists of nozzle, probe, heated particulate filter, condenser, and sorbent module followed by three impingers and a silica gel drying cartridge. An in-stack filter may not be used because at the in-stack temperatures the filter material must be of a material other than the Teflon required by the method. A cyclone or similar device in the heated filter box may be used for sources emitting a large amount of particulate matter.

For sources with a high moisture content, a water trap may be placed between the heated filter and the sorbent module. Additional impingers may also be placed after the sorbent module. If any of these options are used, details must be provided in the test report. The train may be constructed by adaptation of an ARB Method 5 train. Descriptions of the train components are contained in the following sections.

4.1.1 Probe Nozzle

Quartz, or borosilicate 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 approved by the Executive Officer.

A range of sizes suitable for isokinetic sampling should be available, e.g., 0.32 to 1.27 cm (1/8 to 1/2 in.) - or larger if higher volume sampling trains are used - inside diameter (ID) nozzles in increments of 0.16 cm (1/16 in.). Each nozzle shall be calibrated according to the procedures outlined in Section 5.1 of ARB method 5.

4.1.2 Probe

The probe should be lined or made of Teflon, quartz, or borosilicate glass. The liner or probe is to provide an inert surface for the PAH in the stack gas. The liner or probe extends past the retaining nut into the stack. A temperature-controlled jacket provides protection of the liner or probe. The liner shall be equipped with a connecting fitting that is capable of forming a leak-free, vacuum tight connection without the use of sealing greases.

4.1.3 Preseparator

A cyclone, a high capacity impactor or other device may be used if necessary to remove the majority of the particles before the gas stream is filtered. This catch must be used for any subsequent analysis. The device shall be constructed of quartz or borosilicate glass. Other materials may be used subject to approval by the Executive Officer.

4.1.4 Filter Holder

The filter holder shall be constructed of borosilicate glass, with a Teflon frit or Teflon coated wire support and glass-to-glass seal or Teflon gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe, cyclone, or nozzle depending on the configuration used. Other holder and gasket materials may be used subject to approval by the Executive Officer.

4.1.5 Sample Transfer Line

The sample transfer line shall be Teflon (1/4 in. O.D. x 1/32 in. wall) with connecting fittings that are capable of forming leak-free, vacuum tight connections without using sealing greases. The line should be as short as possible.

4.1.6 Condenser

The condenser shall be constructed of borosilicate glass and shall be designed to allow the cooling of the gas stream to at least 20°C before it enters the sorbent module. Design for the normal range of stack gas conditions is shown in Figure 3.

4.1.7 Sorbent Module

The sorbent module shall be made of glass with connecting fittings that are able to form leak-free, vacuum tight seals without the use of sealant greases (Figure 3). The vertical resin trap is preceded by a coil-type condenser, also oriented vertically, with circulating cold water. Gas entering the sorbent module must have been cooled to 20 °C (68°F) or less. The gas temperature shall be monitored by a thermocouple placed either at the inlet or exit of the sorbent trap. The sorbent bed must be firmly packed and secured in place to prevent settling or channeling during sample collection. Ground glass caps (or equivalent) must be provided to seal the sorbent-filled trap both prior to and following sampling. All sorbent modules must be maintained in the vertical position during sampling.

4.1.8 Impinger Train

Connect three or more impingers in series with ground glass fittings able to form leak-free, vacuum tight seals without sealant greases. All impingers shall be of the Greenburg-Smith design modified by replacing the tip with a

1.3 cm (1/2 in.) I.D. glass tube extending to 1.3 cm (1/2 in.)from the bottom of the flask.

The first impinger may be oversized for sampling high moisture streams. The first and second impingers shall contain 100 mL of 3 mM sodium bicarbonate ($NaHCO_3$) and 2.4 mM sodium carbonate Na_2CO_3) (Section 4.2.5). This is intended to neutralize any acids that might form in the impingers. The third impinger shall be empty. Silica gel shall be added to the fourth impinger. A thermometer which measures temperatures to within 1°C (2°F), shall be placed at the outlet of the third impinger.

4.1.9 Silica Gel Cartridge

This may be used instead of a fourth impinger. It shall be sized to hold 200 to 300 gm of silica gel.

4.1.10 Pitot Tube

Type S, as described in Section 2.1 of ARB Method 2 or other devices approved by the Executive Officer. The pitot tube shall be attached to the probe extension to allow constant monitoring of the stack gas velocity as required by Section 2.1.3 of ARB Method 5. When the pitot tube occurs with other sampling components as part of an assembly, the arrangements must meet the specifications required by Section 4.1.1 of ARB Method 2. Interference-free arrangements are illustrated in Figures 2-6 through 2-8 of ARB Method 2 for Type S pitot tubes having external tubing diameters between 0.48 and 0.95 cm (3/16 and 3/8 in.).

Source-sampling assemblies that do not meet these minimum spacing requirements (or the equivalent of these requirements) may be used only if the pitot tube coefficients of such assemblies have been determined by calibration procedures approved by the Executive Officer.

4.1.11 Differential Pressure Gauge

Two inclined manometers or equivalent devices, as described in Section 2.2 of ARB Method 2. One manometer shall be used for velocity head (ΔP) readings and the other for orifice differential pressure readings.

4.1.12 Metering System

Vacuum gauge, leak-free pump, thermometers accurate 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 2. Other metering systems must meet the requirements stated in Section 2.1.8 of ARB Method 5.

4.1.13 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.

4.1.14 Gas Density Determination Equipment

Temperature sensor and pressure gauge, as described in Section 2.3 and 2.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The preferred configuration and alternative arrangements of the temperature sensor shall be the same as those described in Section 2.1.10 of ARB Method 5.

4.1.15 Filter Heating System

The heating system must be capable of maintaining a temperature around the filter holder during sampling of (120 ± 14°C) (248 ± 25°F). A temperature gauge capable of measuring temperature to within 3°C (5.4°F) shall be installed so that the temperature around the filter holder can be regulated and monitored during sampling.

4.1.16 Balance

To weigh the impingers and silica gel cartridge to within 0.5 g.

4.2 SAMPLING MATERIALS AND REAGENTS

4.2.1 Filters

The filters shall be Teflon coated glass fiber filters without organic binders, or Teflon membrane filters, and shall exhibit at least 99.95 percent efficiency (0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM standard Method D 2986-71. Test data from the supplier's quality control program are sufficient for this purpose. Record the manufacturer's lot number.

4.2.1.1 Contamination Check of Filter

The tester must have the filters cleaned by the analyst and checked for contamination prior to use in the field. The contamination check must confirm that there are no PAH contaminants present that will interfere with the analysis of the sample PAHs of interest at the target reporting limits. The analyst must record the date the filter was cleaned.

The filters shall be cleaned in batches not to exceed 50 filters. To clean the filters, shake for one hour in methylene chloride in a glass dish that has been cleaned according to Section 6.2. After extraction, remove the filters and dry them under a clean N_2 stream. Analyze one filter using the same extraction, clean-up and analysis procedures to be used for the field samples (Sections 6.5.1.2, 6.6, and 7.5).

$$\text{Blank value per filter} = \frac{\text{Total mass (ng) of analyte}}{\text{No. filters extracted}} \tag{429-8}$$

The acceptance criteria for filter cleanliness depends on 1) the method reporting limit, 2) the expected field sample volume and 3) the desired reporting limit for the sampled emissions stream. Filters with PAH levels equal to or greater than the target reporting limit for the analyte(s) of concern shall be rejected for field use.

If the filter does not pass the contamination check, re-extract the batch and analyze a clean filter from the re-extracted batch. Repeat the re-extraction and analysis until an acceptably low background level is achieved. Store the remainder tightly wrapped in clean hexane-rinsed aluminum foil as described in Section 4.3.3.

Record the date of the last cleaning of the filters and the date of the PAH analysis, and prepare a laboratory report of the analytical results that includes all of the information required by Section 10.2.

The tester shall obtain this laboratory report with the date of cleaning of the filters, and the date of the filter contamination check from the analyst, and report them in the source test protocol and the test report as required by Sections 10.1 and 10.3.

4.2.2 Amberlite XAD-2 Resin

The XAD-2 resin must be purchased precleaned and then cleaned again as described below before use in the sampling train.

4.2.2.1 Cleaning XAD-2 Resin

This procedure must be carried out in a giant Soxhlet extractor which will hold enough XAD-2 for several sorbent traps, method blanks and QC samples. Use an all glass thimble containing an extra coarse frit for extraction of the XAD-2. The frit is recessed 10 to 15 mm above a crenelated ring at the bottom of the thimble to facilitate drainage. The resin must be carefully retained in the extractor cup with a glass wool plug and stainless steel screen to prevent floating on the methylene chloride.

Clean the resin by two sequential 24 hour Soxhlet extractions with methylene chloride. Replace with fresh methylene chloride after the first 24 hour period.

4.2.2.2 Drying Cleaned XAD-2 Resin

The adsorbent must be dried with clean inert gas. Liquid nitrogen from a standard commercial liquid nitrogen cylinder has proven to be a reliable source of large volumes of gas free from organic contaminants. A 10.2 cm ID Pyrex pipe 0.6 m long with suitable retainers as shown in Figure 4 will serve as a satisfactory column. Connect the liquid nitrogen cylinder to the column by a length of cleaned 0.95 cm ID copper tubing, coiled to pass through a heat source. A convenient heat source is a water bath heated from a steam line. The final nitrogen temperature should only be warm to the touch and not over 40 °C.

Continue the flow of nitrogen through the adsorbent until all the residual solvent is removed. The rate of flow should be high enough that the particles are gently agitated but not so high as to cause the particles to break up.

4.2.2.3 Residual Methylene Chloride Check.

Extraction: Weigh a 1.0 g sample of dried resin into a small vial, add 3 mL of hexane, cap the vial and shake it well.

Analysis: Inject a 2 µL sample of the extract into a gas chromatograph operated under the following conditions:

Column:	6 ft x 1/8 in stainless steel containing 10% OV-101 on 100/120 Supelcoport.
Carrier Gas:	Helium at a rate of 30 mL/min.
Detector:	Flame ionization detector operated at a sensitivity of 4 X 10^{-11} A/mV.
Injection Port Temperature:	250 °C.
Detector Temperature:	305 °C.
Oven Temperature:	30 °C for 4 min; programmed to rise at 40 °C per min until it reaches 250 °C; return to 30 °C after 1000 seconds.

Compare the results of the analysis to the results from a reference solution prepared by adding 2.5 µL of methylene chloride into 100 mL of hexane. This corresponds to 100 µg of methylene chloride per g of adsorbent. The maximum acceptable concentration is 1000 µg/g of adsorbent. If the methylene chloride in the adsorbent exceeds this level, drying must be continued until the excess methylene chloride is removed.

4.2.2.4 Contamination Check of XAD-2 Resin

The cleaned, dried XAD-2 resin must be checked for PAH contamination. Analyze a sample of the resin equivalent in size to the amount required to charge one sorbent cartridge for a sampling train. The extraction, concentration, cleanup and GC/MS analytical procedures shall be the same for this sample as for the field samples (Sections 6.5.1.2, 6.6, and 7.5).

The acceptance limit will depend on the PQL, the expected concentration in the sampled gas stream, and the planned sample volume. The contamination level must be less than the PQL or no more than 20 percent of the expected sample level.

If the cleaned resin yields a value for a target analyte which is not acceptable for the end user's intended application of the test results, repeat the extraction unless the analyst has historical data that demonstrate that re-extraction cannot reasonably be expected to further reduce the contamination levels. The tester must obtain these data from the analyst and include them in both the source test protocol and the emissions test report.

The contamination check shall be repeated if the analyst does not have such historical data. The analyst shall reclean and dry the resin (4.2.2.1, 4.2.2.2, and 4.2.2.3) and repeat the PAH analysis of the re-cleaned resin. If the repeat analysis yields a similar result to the first, record the contamination level for both the initial cleaning and the re-cleaning.

The analyst shall record the dates of the cleaning and extraction of the resin, and prepare a laboratory report of the analytical results that includes all of the information required by Section 10.2.

The tester shall obtain the dates of cleaning and the laboratory report of the results of the contamination check from the analyst, and report them in both the source test protocol and the emissions test report as required by Sections 10.1 and 10.3.

The tester shall identify the analytes for which the PQLs will be based on a blank contamination value, and calculate the PQLs as required by Section 2.3.3.

4.2.2.5 Storage of XAD-2 Resin

After cleaning, the resin may be stored in a wide mouth amber glass container with a Teflon-lined cap, or placed in one of the glass adsorbent modules wrapped in aluminum foil and capped or tightly sealed with Teflon film at each end. The containers and modules shall then be stored away from light at temperatures 4 °C or lower until the resin is used in the sampling train.

The adsorbent must be used within twenty one (21) days of cleaning. If the adsorbent is not used within 21 days, it must be re-checked for contamination before use.

4.2.3 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 desiccants (equivalent or better) may be used, subject to approval by the Executive Officer.

4.2.4 Reagent Water

Deionized, then glass-distilled, and stored in hexane- and methylene chloride-rinsed glass containers with TFE-lined screw caps.

4.2.5 Impinger Solution

Sodium bicarbonate 3 mM, and sodium carbonate 2.4 mM. Dissolve 1.0081 g sodium bicarbonate ($NaHCO_3$) and 1.0176 g of sodium carbonate (Na_2CO_3) in reagent water (4.2.4), and dilute to 4 liters.

4.2.6 Crushed Ice

Place crushed ice in the water bath around the impingers.

4.2.7 Glass Wool

Cleaned by sequential rinsing in three aliquots of hexane, dried in a 110 °C oven, and stored in a hexane-washed glass jar with TFE-lined screw cap.

4.2.8 Chromic Acid Cleaning Solution

Dissolve 200 g of sodium dichromate in 15 mL of reagent water, and then carefully add 400 mL of concentrated sulfuric acid.

4.3 PRE-TEST PREPARATION

The positive identification and quantitation of PAH in an emissions test of stationary sources are strongly dependent on the integrity of the samples received and the precision and accuracy of all analytical procedures employed. The QA procedures described in Sections 4.3.7 and 8 are to be used to monitor the performance of the sampling methods, identify problems, and take corrective action.

4.3.1 Calibration

All sampling train components shall be maintained and calibrated according to the procedure described in APTD-0576 (Section 11.7), unless otherwise specified herein. The tester shall maintain a record of all calibration data.

4.3.1.1 Probe Nozzle

Probe nozzles shall be calibrated according to the procedure described in ARB Method 5.

4.3.1.2 Pitot Tube

Calibrate the Type S pitot tube assembly according to the procedure described in Section 4 of ARB Method 2.

4.3.1.3 Metering System

Calibrate the metering system before and after use according to the requirements of Section 5.3 of ARB Method 5.

4.3.1.4 Temperature Gauges

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

4.3.1.5 Leak Check of Metering System Shown in Figure 1

The tester shall use the procedure described in Section 5.6 of ARB Method 5

4.3.1.6 Barometer

Calibrate against a mercury barometer.

4.3.2 Cleaning Glassware for Sampling and Recovery

All glass parts of the train upstream of and including the sorbent module and the first impingers shall be cleaned as described in Section 3A of the 1974 issue of Manual of Analytical Methods for Analysis of Pesticide Residues in Human and Environmental Samples (Reference 11.4). Take special care to remove residual silicone grease sealants on ground glass connections of used glassware. These greasy residues shall be removed by soaking several hours in a chromic acid cleaning solution (4.2.8) prior to routine cleaning as described above. Other cleaning procedures may be used as long as acceptable blanks are obtained. Acceptance criteria for blanks are stated in Section 8.2.

Rinse all glassware with acetone, hexane, and methylene chloride prior to use in the PAH sampling train.

Glassware used in sample recovery procedures must be rinsed as soon as possible after use with the last solvent used in it. This must be followed by detergent washing with hot water, and rinses with tap water, deionized water, acetone, hexane, and methylene chloride. Other cleaning procedures may be used as long as acceptable blanks are obtained. Acceptance criteria for blanks are stated in Section 8.2.

4.3.3 Preparation of Filter

The clean dry filter (4.2.1) must be kept tightly wrapped in hexane-rinsed aluminum foil and stored at 0 to 4°C in a container away from light until sampling. Before inserting the filter in the sampling train, check visually against light for irregularities and flaws or pinhole leaks.

4.3.4 Preparation of Sorbent Cartridge, Method Blank, and Laboratory Control Samples

Sorbent Cartridge

Use a sufficient amount (at least 30 gms or 5 gms/m^3 of stack gas to be sampled) of cleaned resin to completely fill the glass sorbent cartridge which has been thoroughly cleaned as prescribed (4.2.2).

Add the required surrogate standards (Table 7) to the sorbent cartridges for all of the sampling and blank trains for each series of test runs. Follow the resin with hexane-rinsed glass wool, cap both ends, and wrap the cartridge in aluminum foil. Store the prepared cartridges as required by Section 4.3.5.

The sorbent cartridges must be loaded, and the surrogate standards must be added to the resin in a clean area in the laboratory. There must be no turnaround of a used cartridge in the field.

The analyst shall record the date that the surrogate standards were added to the resin and the amount of each compound. The tester shall obtain these data from the analyst and report them in the source test protocol and the test report.

The appropriate levels for the surrogate standards are given in Table 7 which shows the spiking plan for surrogate standards, internal standards, alternate standards, and recovery standards. All of these required compounds are generally available. Additional labelled PAH may also be used if available. The labelled compounds used as surrogate standards must be different from the internal standards used for quantitation, and from the alternate and recovery standards. If the spiking scheme (Table 7) is modified, the tester must demonstrate that the proposed modification will generate data of satisfactory quality. Table 7A shows an approved modification that has been used in ARB's method development. All modifications must be approved by the Executive Officer before the emissions test is performed.

Laboratory Method Blank

Take a sample of XAD-2 resin from the same batch used to prepare the sampling cartridge. This will serve as the laboratory method blank (Section 8.1.1). The mass of this sample must be the same as that used in the sampling train. Spike with the same surrogate standards at the same levels used in the sampling cartridges.

Laboratory Control Sample

Set aside two samples of XAD-2 resin from the same batch used to prepare the sampling cartridge. These will serve as the laboratory control samples. (Section 8.1.3). The mass of each sample must be the same as that used in the sampling train.

4.3.5 Storage of Prepared Cartridges, Method Blank and Laboratory Control Sample

Store the aluminum foil wrapped sorbent cartridges away from light at 4 °C or lower until they are fitted into the sampling trains. Do not remove the caps before the setup of the sampling train.

The maximum storage time from cleaning of the resin to sampling with the spiked resin cartridge must not exceed 21 days (4.2.2.5).

Store the laboratory method blank and laboratory control samples in amber glass jars with Teflon lined lids at temperatures no higher than 4 °C.

4.4 SAMPLE COLLECTION

Because of the complexity of this method, testers must be experienced with the test procedures in order to ensure reliable results.

4.4.1 Preliminary Field Determinations

Select the sampling site and the minimum number of sampling points according to ARB Method 1 or as specified by the Executive Officer.

Determine the stack pressure, temperature, and the range of velocity heads using ARB Method 2. Conduct a leak-check of the pitot lines according to ARB Method 2, Section 3.1.

Determine the moisture content using ARB Method 4 or its alternatives for the purpose of making isokinetic sampling rate settings.

Determine the stack gas dry molecular weight, as described in ARB Method 2, Section 3.6. If integrated sampling (ARB Method 3) 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 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. Do not change the nozzle size during the run. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see Section 2.2 of ARB 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.

The target sample volume and sampling time must already have been calculated for the source test protocol and approved by the end user as required by Sections 2.2 and 2.5. The total sampling time must be such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Executive Officer), and (2) the total gas sample volume collected (corrected to standard conditions) will not be less than the target value calculated for the source test protocol (Section 2.5.5).

To avoid timekeeping errors, the number of minutes sampled at each point should be an integer or an integer plus one-half minute.

4.4.2 Preparation of Collection Train

Keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Caution: Do not use sealant greases in assembling the sampling train.

Record the performance of the setup procedures for the sampling train. Figure 10 is an example of a form for recording the sampling train setup data. The tester must record all of the routine information indicated on this form as well as any additional data which are necessary for documenting the quality of any reported results.

Place 100 ml of the impinger solution (4.2.5) in the first impinger and weigh. Record the total weight. Repeat the procedure for the second impinger. Leave the third impinger empty. Weigh the empty third impinger and record the weight.

Weigh 200 to 300 g of silica gel to the nearest 0.5 g directly into a tared impinger or silica gel cartridge just prior to assembly of the sampling train. The tester may optionally measure and record in advance of test time the weights of several portions of silica gel in air-tight containers. One portion of the preweighed silica gel must then be transferred from its container to the silica gel cartridge or fourth impinger. Place the container in a clean place for later use in the sample recovery.

Using tweezers or clean disposable surgical gloves, place a filter in the filter holder. Be sure that the filter is properly centered and the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly of the filter holder is completed.

Mark the probe extension with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

Assemble the train as in Figure 2. Place crushed ice around the impingers.

4.4.3 Leak Check Procedures

4.4.3.1 Pretest Leak Check

After the sampling train has been assembled, turn on and set the filter and probe heating systems at the desired operating temperatures. Allow time for the temperature to stabilize. Leak-check the train at the sampling site by plugging the nozzle with a TFE plug and pulling a vacuum of at least 380 mm Hg (15 in. Hg).

Note: A lower vacuum may be used, provided that it is not exceeded during the test.

The following leak-check instructions for the sampling train are described in Section 4.1.4.1 of ARB Method 5. Start the pump with by-pass valve fully open and coarse adjust valve completely closed. Partially open the coarse adjust valve and slowly close the by-pass valve until the desired vacuum is reached. Do not reverse the direction of the by-pass valve. This will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum or end the leak-check as described below and start over.

Determine the leakage rate. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m^3 per min. (0.02 cfm), whichever is less, is unacceptable. Repeat the leak check procedure until an acceptable leakage rate is obtained. Record the leakage rate on the field data sheet (Figure 5).

When the leak-check is completed, first slowly remove the plug from the inlet to the probe nozzle and immediately turn off the vacuum pump. This prevents water from being forced backward and keeps silica gel from being entrained backward.

4.4.3.2 Leak Checks During Sample Run

If, during the sampling run, it becomes necessary to change a component (e.g., filter assembly or impinger), a leak check shall be conducted immediately before the change is made. The leak-check shall be done according to the procedure described in Section 4.4.3.1 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, the tester shall either (1) record the leakage rate and correct the volume of gas sampled since the last leak check as shown in Section 4.4.3.4 below, or (2) void the sampling run. Record the leakage rate.

Immediately after component changes, leak-checks must be conducted according to the procedure outlined in Section 4.4.3.1 above. Record the leakage rate on the field data sheet (Figure 5).

4.4.3.3 Post Test Leak Check

A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be done in accordance with the procedures outlined in Section 4.4.3.1 except that it shall be conducted at a vacuum equal to or greater than the maximum value recorded during the sampling run. Record the leakage rate on the field data sheet (Figure 5). If the leakage rate is found to be no greater than 0.00057 m^3/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, (1) record the leakage rate and correct the sample volume as shown in Section 4.4.3.4 below, or (2) void the sampling run.

4.4.3.4 Correcting for Excessive Leakage Rates

If the leakage rate observed during any leak-check after the start of a test exceeds the maximum leakage rate L_a (see definition below), replace V_m in Equation 429-9 with the following expression.

$$V_m - \sum_{i=1}^{n} (L_i - L_a)\theta_i - (L_p - L_a)\theta_p \qquad 429\text{-}9$$

Where:

V_m = Volume of gas sampled as measured by the dry gas meter (dscf).

L_a = Maximum acceptable leakage rate equal to 0.00057 m^3/min (0.02 ft^3/min) or 4% of the average sampling rate, whichever is smaller.

L_p = Leakage rate observed during the post-test leak-check, m^3/min (ft^3/min).

L_i = Leakage rate observed during the leak-check performed prior to the "ith" leakcheck (i = 1,2,3...n), m^3/min (ft^3/min).

θ_i = Sampling time interval between two successive leak-checks beginning with the interval between the first and second leak-checks, min.

θ_p = Sampling time interval between the last (n^{th}) leak-check and the end of the test, min.

Substitute only for those leakage rates (L_i or L_p) which exceed L_a.

4.4.4 Train Operation

4.4.4.1 Sampling Train

During the sampling run maintain a sampling rate within 10 percent of true isokinetic, unless otherwise specified or approved by the Executive Officer. The actual sampling rate must be at or above the VSR (Equation 429-4) to collect the target sample mass in the estimated sampling time. If the target sampling rate cannot be achieved, adjust the planned sampling time to achieve the target sample volume (PSV).

For each run, record the data required on the sample data sheet shown in Figure 5. The operator must record the dry gas meter reading at the beginning of the test, 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.

Record other readings required by Figure 5 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 of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 ± 0.02, and the stack gas equivalent density (dry molecular weight) (M_d) is equal to 29 ± 4. APTD-0576 (Reference 11.7) details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Reference 11.8) 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.

Turn on the recirculating pump for the adsorbent module and the condenser, and begin monitoring the temperature of the gas entering the adsorbent trap. Ensure that the temperature of the gas is 20 °C or lower before sampling is started.

Traverse the stack cross section, as required by ARB Method 1 or as specified by the Executive Officer, 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. This minimizes the chance of extracting deposited material.

During the test run, take appropriate steps (e.g., adding crushed ice to the impinger ice bath) to maintain the temperature at the condenser outlet below 20°C (68°F). 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 during a sample run. Another complete filter assembly must be used rather than changing the filter itself. Before a new filter assembly is installed, conduct a leak-check as outlined in Section 4.4.3.2. The total PAH analysis shall include the combined catches of all filter assemblies.

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 approval by the Executive Officer.

Note that when two or more trains are used, a separate analysis of each train shall be performed, unless identical nozzle sizes were used on all trains, in which case the 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 4.4.3.3. Also, leak-check the pitot lines as described in ARB Method 2; the lines must pass this leak-check, in order to validate the velocity head data. Record leakage rates.

Record any unusual events during the sampling period.

4.4.4.2 Blank Train

There shall be at least one blank train for each series of three or fewer test runs. For those sources at which emissions are sampled at more than one sampling location, there shall be at least one blank train assembled at each location for each set of three or fewer runs.

Prepare and set up the blank train in a manner identical to that described above for the sampling trains. The blank train shall be taken through all of the sampling train preparation steps including the leak check without actual sampling of the gas stream. Recover the blank train as described in Section 5.3. Follow all subsequent steps specified for the sampling train including extraction, analysis, and data reporting.

4.4.5 Calculation of Percent Isokinetic

Calculate percent isokinetic (Section 4.5.7) to determine whether the run should be repeated. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Executive Officer for possible variance on the isokinetic rates.

4.5 CALCULATIONS

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

4.5.1 Nomenclature

A = Cross-sectional area of stack, ft^2.

A_n = Cross-sectional area of nozzle, ft^2.

B_{ws} = Water vapor in the gas stream, proportion by volume.

C_s = Concentration of PAH in stack gas, ng/dscm, corrected to standard conditions of 20°C, 760 mm Hg (68°F, 29.92 in. Hg) on dry basis.

G_s = Total mass of PAH in stack gas sample, ng.

ΔH = Average pressure differential across the orifice meter, mm H_2O (in. H_2O).

I = Percent isokinetic sampling.

L_a = Maximum acceptable leakage rate for either a pretest leak-check or for a leak check following a component change; equal to 0.00057 m^3/min (0.02 cfm) or 4 percent of the average sampling rate, whichever is less.

L_i = Individual leakage rate observed during the leak-check conducted prior to the "ith" component change ($i = 1, 2, 3, ...n$), m^3/min (cfm).

L_p = Leakage rate observed during the post-test leak check, m^3/min (cfm).

M_d = Molecular weight of stack gas, dry basis, lb/lb-mole (g/g-mole).

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

M_s = Molecular weight of stack gas, wet basis, lb/lb-mole (g/g-mole).

P_{bar} = 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).

Q_{std} = Dry volumetric stack gas flow rate corrected to standard conditions, dscf/min (dscm/min).

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

R = Ideal gas constant 0.06236 mm Hg-m^3/°K-g-mole (21.85 in Hg-ft^3/R-lb-mole).

T_m = Absolute average dry gas meter temperature, °K (°R).

T_s = Absolute average stack gas temperature °K (°R).

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

V_{1c} = Total volume of liquid collected in impingers and silica gel, mL.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

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

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

v_s = Stack gas velocity, calculated by ARB Method 2, Equation 2-9, ft/sec (m/sec).

Y = Dry gas meter calibration factor.

θ = Total sampling time, min.

θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.

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

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

φ_w = Sampling time interval, from the final (nth) component change until

13.6 = Specific gravity of mercury.

60 = Conversion factor, sec/min.

100 = Conversion to percent.

4.5.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop

See sampling run record (Figure 5).

4.5.3 Dry Gas Volume

Use Equation 429-10 to correct the sample volume measured by the dry gas meter to standard conditions (20°C, 760 mm Hg or 68°F, 29.92 in Hg).

$$V_{m(std)} = V_m\, Y\, \frac{T_{std}}{T_m} \frac{\left(P_{bar} + \frac{\Delta H}{13.6}\right)}{P_{std}} = K_1\, V_m\, Y \frac{\left(P_{bar} + \frac{\Delta H}{13.6}\right)}{T_m} \qquad 429\text{-}10$$

Where:

$$K_1 = \frac{T_{std}}{P_{std}} = 0.3858\ ^{o}\mathrm{K/mm\ Hg}$$ for metric units

$$= 17.65\ ^{o}\mathrm{R/in\ Hg}$$ for English units

NOTE: Equation 429-10 may 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_a. If L_p or L_i exceeds L_a, V_m in Equation 429-10 must be modified as described in Section 4.4.3.4.

4.5.4 Average Stack Gas Velocity

Calculate the average stack gas velocity, v_s, as specified in ARB Method 2, Section 5.2.

4.5.5 Volume of Water Vapor

Calculate the volume of water vapor using Equation 429-11 and the weight of the liquid collected during sampling (Sections 5.3.6 and 5.3.8).

$$V_{w(std)} = V_{1c} \frac{\rho_w}{M_w} \frac{RT_{std}}{P_{std}} = K_2\, V_{1c} \qquad 429\text{-}11$$

Where:

K_2 = 0.001333 m_3/mL for metric units, or
= 0.04707 ft_3/mL for English units.

4.5.6 Moisture Content

Calculate the moisture content of the gas, B_{ws}.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad 429\text{-}12$$

NOTE: In saturated or water-droplet laden streams, the procedure for determining the moisture content is given in the note to Section 1.2 of Method 4. For the purpose of this method, the average stack-gas temperature from Figure 5 may be used for this determination, provided that the accuracy of the in-stack temperature sensor is ± 1^{o}C (2^{o}F)

4.5.7 Isokinetic Variation

4.5.7.1 Calculation from Raw Data

$$I = \frac{100T_s\left[K_3V_{1c} + \frac{V_mY}{T_m}\left(P_{bar} + \frac{\Delta H}{13.6}\right)\right]}{60\ \theta\ v_s\ P_s\ A_n} \qquad 429\text{-}13$$

Where:

K_3 = 0.003454 mm Hg-m^3/mL-°K for metric units

= 0.002669 in Hg-ft^3/mL-°R for English units

4.5.7.2 Calculation from Intermediate Values

$$I = \frac{100T_s\ V_{m(std)}\ P_{std}}{T_{std}\ v_s\ \theta\ A_n\ P_s\ 60\ (1 - B_{ws})} \qquad 429\text{-}14$$

$$= K_4 \frac{T_s\ V_{m(std)}}{P_s\ v_s\ \theta\ A_n\ (1 - B_{ws})}$$

Where:

K_4 = 4.320 for metric units.

= 0.09450 for English units.

4.5.8 Average stack gas dry volumetric flow rate

Use Equation 429-15 to calculate the average dry volumetric flow rate of the gas.

$$Q_{std} = 60\ K_1\ (1 - B_{ws})\ v_s\ A\left(\frac{P_s}{T_s}\right) \qquad 429\text{-}15$$

Where:

$$K_1 = \frac{T_{std}}{P_{std}}$$ = 0.3858 °K/mm Hg for metric units

= 17.65 °R/in Hg for English units

4.6 ISOKINETIC CRITERIA

If 90 percent < I < 110 percent, the isokinetic results are acceptable. If there is a bias to the results because I < 90 percent or I > 110 percent, then the results must be rejected and the test repeated, unless the test results are accepted by the Executive Officer.

5 SAMPLE RECOVERY

5.1 SAMPLE RECOVERY APPARATUS

5.1.1 Probe Nozzle Brush

Inert bristle brush with stainless steel wire handle. The brush shall be properly sized and shaped to brush out the probe nozzle.

5.1.2 Wash Bottles

Teflon wash bottles are required; Teflon FEP®.

5.1.3 Glass Sample Storage Containers

Precleaned narrow mouth amber glass bottles, 500 mL or 1000 mL. Screw cap liners shall be Teflon.

5.1.4 Filter Storage Containers

Sealed filter holder or precleaned, wide-mouth amber glass containers with Teflon lined screw caps.

5.1.5 Balance

To measure condensed water to within 0.5 g.

5.1.6 Silica Gel Storage Containers

Air tight metal containers to store silica gel.

5.1.7 Funnel and Rubber Policeman

To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

5.1.8 Funnel

To aid in sample recovery. Glass or Teflon® must be used.

5.1.9 Ground Glass Caps or Hexane Rinsed Aluminum Foil

To cap off adsorbent tube and the other sample-exposed portions of the aluminum foil.

5.1.10 Aluminum Foil

Heavy-duty, precleaned with methylene chloride.

5.2 SAMPLE RECOVERY REAGENTS

5.2.1 Reagent Water

Deionized (DI), then glass distilled, and stored in hexane and methylene chloride-rinsed glass containers with TFE-lined screw caps.

5.2.2 Acetone

Nanograde quality. "Distilled in Glass" or equivalent, stored in original containers. A blank must be screened by the analytical detection method.

5.2.3 Hexane

Nanograde quality. "Distilled in Glass" or equivalent, stored in original containers. A blank must be screened by the analytical detection method.

5.2.4 Methylene Chloride

Nanograde quality or equivalent. A blank must be screened by the analytical detection method.

5.3 SAMPLE RECOVERY PROCEDURE

Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period and a post test leak check has been performed (4.4.3.3). Allow the probe to cool.

When the probe can be safely handled, wipe off all external particulate matter near the tip of the probe nozzle. Conduct the post test leak check as described in Section 4.4.3.3. Remove the probe from the train and close off both ends of the probe with precleaned aluminum foil (5.1.10). Seal off the inlet to the train with a ground glass cup or precleaned aluminum foil.

Transfer the probe and impinger assembly to the cleanup area. This area must be clean, and enclosed so that the chances of contaminating the sample will be minimized.

No smoking is allowed.

Inspect the train prior to and during disassembly and note any abnormal conditions, broken filters, color of the impinger liquid, etc. Figure 6 summarizes the recovery procedure described in Sections 5.3.1 to 5.3.8.

Figure 11 is an example of a form for recording the performance of the sample recovery procedure. The tester must record all of the routine information indicated on this form as well as any additional data which are necessary for documenting the quality of any reported results.

5.3.1 Sample Container No. 1 (front half rinses)

Quantitatively recover material deposited in the nozzle, probe, the front half of the filter holder, and the cyclone, if used, first by brushing and then by sequentially rinsing with acetone, hexane, and methylene chloride three times each. Place all these rinses in Container No.1. Mark the liquid level.

5.3.2 Cyclone Catch

If the optional cyclone is used, quantitatively recover the particulate matter by sequentially rinsing the cyclone with acetone, hexane, and methylene chloride. Store in a clean sample container and cap.

5.3.3 Sample Container No. 2 (filter)

Carefully remove the filter from the filter holder and place it in its identified container. Use a pair of precleaned tweezers to handle the filter. Do not wrap the filter in aluminum foil. If it is necessary to fold the filter, make sure that the particulate cake is inside the fold. Carefully transfer to the container any particulate matter and/or filter fibers which adhere to the filter holder gasket by using a dry inert bristle brush and/or a sharp-edged blade. Seal the container.

5.3.4 Sorbent Module

Remove the sorbent module from the train and cap it.

5.3.5 Sample Container No. 3 (back half rinses)

Rinse the back half of the filter holder, the transfer line between the filter and the condenser, and the condenser (if using the separate condenser-sorbent trap) three times each with acetone, hexane and methylene chloride, and collect all rinses in Container No. 3. If using the combined condenser/sorbent trap, the rinse of the condenser shall be performed in the laboratory after removal of the XAD-2 portion. If the optional water knockout trap has been employed, the contents and rinses shall be placed in Container No. 3. Rinse it three times each with acetone, hexane, and methylene chloride. Mark the liquid level.

The back half rinses may also be combined in a single container with the front half rinses (Section 5.3.1).

5.3.6 Sample Container No. 4 (Impinger contents)

Wipe off the outside of each of the first three impingers to remove excess water and other material. Weigh the impingers and contents to the nearest ±0.5 g using a balance. Record the weight. Calculate and then record the weight of liquid collected during sampling. Use this weight and the weight of liquid collected in the silica gel (Section 5.3.8) to calculate the moisture content of the effluent gas (Sections 4.5.5 and 4.5.6). Pour the impinger catch directly into Container No. 4. Mark the liquid level.

5.3.7 Sample Container No. 5 (Impinger rinses)

Rinse each impinger sequentially three times with acetone, hexane, and methylene chloride and pour rinses into Container No. 5. Mark the liquid level. These rinses may be combined with the previously weighed impinger contents in Container No. 4.

5.3.8 Weighing Silica Gel

Weigh the spent silica gel to the nearest 0.5 g using a balance. Record the weight. Calculate and then record the weight of liquid collected during sampling. Use this weight and the weight of liquid collected in the impingers (Section 5.3.6) to calculate the moisture content of the effluent gas (Sections 4.5.5 and 4.5.6). .

5.4 SAMPLE PRESERVATION AND HANDLING

From the time of collection to extraction, maintain all samples (Sections 5.3.1 to 5.3.7) at 4°C or lower and protect from light. All samples must be extracted as soon as practically feasible, but within 21 days of collection; and all extracts must be analyzed as soon as practically feasible, but within 40 days of extraction. Success in meeting the holding time requirement will depend on pre-test planning by the tester and the laboratory.

6 ANALYTICAL PREPARATION

This method is restricted to use only by or under the supervision of analysts experienced in the use of capillary column gas chromatography/mass spectrometry and skilled in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedures described in Sections 7.3, 8.2.6, and 8.3.1.

6.1 SAFETY

The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined. Nevertheless, each chemical compound should be treated as a potential health hazard and exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Reference 11.9 describes procedures for handling hazardous chemicals in laboratories.

The following method analytes have been classified as known or suspected human or mammalian carcinogens: benzo(a)anthracene and dibenzo-(a,h,)anthracene. A guideline for the safe handling of carcinogens can be found in Section 5209 of Title 8 of the California Administrative Code.

6.2 CLEANING OF LABORATORY GLASSWARE

Glassware used in the analytical procedures (including the Soxhlet apparatus and disposable bottles) must be cleaned as soon as possible after use by rinsing with the last solvent used in it. This must be followed by detergent washing with hot water, and rinses with tap water, deionized water, acetone, hexane, and methylene chloride. Other cleaning procedures may be used as long as acceptable blanks are obtained. Acceptance criteria for blanks are given in Section 8.2.

Clean aluminum foil with acetone followed by hexane and methylene chloride.

6.3 APPARATUS

6.3.1 Grab Sample Bottle

Amber glass, 125-mL and 250-mL, fitted with screw caps lined with Teflon. The bottle and cap liner must be acid washed and solvent rinsed with acetone and methylene chloride, and dried before use.

6.3.2 Concentrator Tube, Kuderna-Danish

10-mL, graduated (Kontes-K-570050-1025 or equivalent). Calibration must be checked at the volumes employed in the test. A ground glass stopper must be used to prevent evaporation of extracts.

6.3.3 Evaporation Flask, Kuderna-Danish

500-mL (Kontes K-570001-0500 or equivalent). (Attached to concentrator tube with springs).

6.3.4 Snyder Column, Kuderna-Danish

Three-ball macro (Kontes K-569001-0121 or equivalent).

6.3.5 Snyder Column, Kuderna-Danish

Two-ball micro (Kontes K-569001-0219 or equivalent).

6.3.6 Minivials

1.0 mL vials; cone-shaped to facilitate removal of very small samples; heavy wall borosilicate glass; with Teflon-faced rubber septa and screw caps.

6.3.7 Soxhlet Apparatus

1 liter receiver, 1 heating mantle, condenser, Soxhlet extractor.

6.3.8 Rotary Evaporator

Rotovap R (or equivalent), Brinkmann Instruments, Westbury, NY.

6.3.9 Nitrogen Blowdown Apparatus

N-Evap Analytical Evaporator Model 111 (or equivalent), Organomation Associates Inc., Northborough, MA.

6.3.10 Analytical Balance

Analytical. Capable of accurately weighing to the nearest 0.0001 g.

6.3.11 Disposable Pipet

5 3/4 inch x 7.0 mm OD.,

6.4 SAMPLE PREPARATION REAGENTS

6.4.1 Reagent water

Same as 5.2.1.

6.4.2 Acetone

Same as 5.2.2.

6.4.3 Hexane

Same as 5.2.3.

6.4.4 Methylene Chloride

Same as 5.2.4.

6.4.5 Sulfuric Acid

ACS. Reagent grade. Concentrated, sp. gr. 1.84.

6.4.6 Sodium Sulfate

ACS. Reagent grade. Granular, anhydrous. Purify prior to use by extracting with methylene chloride and oven drying for 4 or more hours in a shallow tray. Place the cleaned material in a glass container with a Teflon lined screw cap, and store in a desiccator.

6.4.7 Silica Gel

For column chromatography, type 60, EM reagent, 100-200 mesh, or equivalent. Soxhlet extract with methylene chloride, and activate by heating in a foil covered glass container for longer than 16 hours at 130 °C, then store in a desiccator. The storage period shall not exceed two days.

NOTE: The performance of silica gel in the column cleanup procedure varies with manufacturers and with the method of storage. The analyst shall establish a procedure that satisfies the performance criteria of Section 6.6.1.

6.4.8 Alumina: Acidic

Soxhlet extract with methylene chloride, and activate in a foil covered glass container for 24 hours at 190 °C.

NOTE: The performance of alumina in the column cleanup procedure varies with manufacturers and with the method of storage. The analyst shall establish a procedure that meets the performance criteria of Section 6.6.1.

6.4.9 Nitrogen

Obtained from bleed from liquid nitrogen tank.

6.5 SAMPLE EXTRACTION

WARNING: Stack sampling will yield both liquid and solid samples for PAH analysis. Samples must not be split prior to extraction even when they appear homogeneous as in the case of single liquid phase samples. Solid samples such as the resin are not homogeneous and particulate matter may not be uniformly distributed on the filter. In addition, filter samples are generally so small that the desired detection limit might not be achieved if the sample were split.

The recovered samples may be combined as follows:

1) Particulate filter and particulate matter collected on the filter (Section 5.3.3), cyclone catch (Section 5.3.2) and sample container No. 1 (Section 5.3.1).

2) Sample container No. 3 (Section 5.3.5), resin (Section 5.3.4) and rinse of resin cartridge.

3) Sample container No.4 (Section 5.3.6) and sample container No.5 (Section 5.3.7)

Two schemes for sample preparation are described in Sections 6.5.1 and 6.5.2 below. One of these must be used.

Section 6.5.1 describes sample preparation procedures for separate GC/MS analyses of impingers and the remainder of the sampling train. Figure 7 is a flowchart of the extraction and cleanup procedures.

Section 6.5.2 describes sample preparation procedures for GC/MS analysis of a single composite extract from each sampling train. The recovered samples are combined as shown in Figure 8.

6.5.1 Separate Analysis of Impingers

A separate analysis of the impingers can be used to determine whether there has been breakthrough of PAHs past the resin.

6.5.1.1 Extraction of Liquid Samples

A. **Sample Container No. 1 (Front half rinses)**

Concentrate the contents of sample container No. 1 (Section 5.3.1) to a volume of about 1-5 mL using the nitrogen blowdown apparatus. Rinse the sample container three times with small amounts of methylene chloride and add these rinses to the concentrated solution. Concentrate further to about 1-5 mL. This residue will likely contain particulate matter which was removed in the rinses of the probe and nozzle. Transfer the residue (along with three rinses of the final sample vessel) to the Soxhlet apparatus with the filter and particulate catch and proceed as described under Section 6.5.1.2 below.

B. **Sample Container No. 3 (Back half rinses)**

Concentrate the contents of sample container No. 3 (Section 5.3.5) to a volume of about 1-5 mL using the nitrogen blowdown apparatus. Rinse the sample container three times with small amounts of methylene chloride and add these rinses to the concentrated solution. Concentrate further to about 1-5 mL. Combine this residue (along with three rinses of the final sample vessel) in the Soxhlet apparatus with the resin sample, and proceed as described under Section 6.5.1.2 below.

C. **Containers No. 4 and No. 5 (Impinger contents and rinses)**

Place the contents of Sample Containers No. 4 and No. 5 (Sections 5.4.6 and 5.4.7) in a separatory funnel. Add the appropriate amount of ^{2}H-labelled alternate standard solution (Section 7 and Table 7 or 7A) to achieve the final extract concentrations indicated in Table 8 or 8A. The amounts required by Section 7.2.4 are based on a final volume of 500 μL for analysis (450 μL of sample extract and 50 μL of recovery standard solution). Extract the sample three times with 60 mL aliquots of methylene chloride. Combine the organic fractions. Divide the extract in two - one half to be archived, and the other for cleanup and GC/MS analysis. Store the archive sample at 4°C away from light.

Pour the remaining extract through Na_2SO_4 into a round bottom flask. Add 60 to 100 mL hexane and evaporate to about 10 mL. Repeat three times or less if the methylene chloride can be removed with less hexane. Add the appropriate amount of alternate standard (Section 7.2.7) to achieve the final extract concentrations shown in Table 6 or 6A. This standard must be used to monitor the efficiency of the cleanup procedure.

Concentrate the remaining sample to 2 mL with a Kuderna-Danish concentrator or rotary evaporator, then transfer the extract to a 8-mL test tube with hexane. Proceed with sample cleanup procedures below (Section 6.6).

6.5.1.2 Extraction of Solid Samples

Filter, Particulate matter, and Resin

The Soxhlet apparatus must be large enough to allow extraction of the sample in a single batch. Clean the Soxhlet apparatus by a 4 to 8-hr Soxhlet with methylene chloride at a cycling rate of 3 cycles per hour. Discard the solvent. Add 20 g Na_2SO_4 to the thimble. Combine the filter, resin, glass wool, and concentrated front and back half rinses (6.5.1.1A and 6.5.1.1B) and place on top of the Na_2SO_4. Add the appropriate amount of internal standard (Section 7.2.4 and Table 7) to achieve the final extract concentrations indicated in Table 8.

Place the thimble in the Soxhlet apparatus, and add about 700 mL of methylene chloride to the receiver. Assemble the Soxhlet, turn on the heating controls and cooling water, and allow to reflux for 16 hours at a rate of 3 cycles per hour. After extraction, allow the Soxhlet to cool. Divide the sample in two - one half to be archived, and the other for cleanup and GC/MS analysis. Store the archive sample at 4°C away from light.

Exchange the remaining extract to hexane. Add 60 to 100 mL hexane and evaporate to about 10 mL. Repeat three times or as necessary to remove the methylene chloride. Add the appropriate amount of alternate standard (Section 7.2.7 and Table 7 or 7A) to achieve the final extract concentrations shown in Table 8 or 8A. This alternate standard must be used to monitor the efficiency of the cleanup procedure when the impingers are analyzed separately from the remainder of the sampling train.

Concentrate the remaining sample to about 2 mL with a Kuderna-Danish concentrator or rotoevaporator, then transfer the extract to a 8-mL test tube with hexane. Proceed with sample cleanup procedures below (Section 6.6).

6.5.2 Single Composite Extract For Analysis

6.5.2.1 Extraction of Aqueous Samples

Containers No. 4 and No. 5 (Impinger contents and rinses)

Pour the contents of Sample Containers No. 4 and No. 5 (Sections 5.3.6 and 5.3.7) into an appropriate size separatory funnel. Do not add internal standards. Instead, add the appropriate amount of alternate standard spiking solution (Section 7 and Table 7 or 7A) to achieve the final extract concentrations indicated in Table 8 or 8A.

Extract the sample three times with 60 mL aliquots of methylene chloride. Combine the organic fractions with the solid samples and concentrated rinses (6.5.2.2) in a Soxhlet extractor.

6.5.2.2 Extraction of Solid Samples

Concentrate the front and back half rinses as described in Sections 6.5.1.1A and 6.5.1.1B. Clean the Soxhlet apparatus as in Section 6.5.1.2. Place the filter and resin in the Soxhlet apparatus along with the concentrated front and back half rinses and the impinger extract. Add the internal standards, extract the sample, and concentrate the extract as described in Section 6.5.1.2. Divide the extract into two equal portions. Store one of these, the archive sample, at 4 °C away from light. The remaining extract must be exchanged to hexane as described in Section 6.5.1.2. Do not add the alternate standard to this composite extract. It has already been added to the impinger sample (6.5.2.1).

Concentrate the extract to 2 mL with a Kuderna-Danish concentrator or rotary evaporator, then transfer to a 8-mL test tube with hexane or equivalent non-polar solvent such as isooctane. Proceed with sample cleanup procedures below (Section 6.6)

6.6 COLUMN CLEANUP

Several column chromatographic cleanup options are available. Either of the two described below may be sufficient. Before using a procedure for the cleanup of sample extracts, the analyst must demonstrate that the requirements of Sections 8.1.3.1 and 8.2.6 can be met using the cleanup procedure. Acceptable alternative cleanup procedures may also be used provided that the analyst can demonstrate that the performance requirements of Sections 8.1.3.1 and 8.2.6 can be met. Compliance with the requirements of Sections 8.1.1.1 and 8.2.6 must also be demonstrated whenever there is a change in the column cleanup procedure used for the initial demonstration.

The sample extract obtained as described in Sections 6.5.1C and 6.5.1.2 or 6.5.2.2 is concentrated to a volume of about 1 mL using the nitrogen blowdown apparatus, and this is transferred quantitatively with hexane rinsings to at least one of the columns described below.

6.6.1 Column Preparation

A. Silica Gel Column

Pack a glass gravity column (250 mm x 10 mm) in the following manner:

Insert a clean glass wool plug (Section 4.2.7) into the bottom of the column and add 10 grams of activated silica gel (Section 6.4.7) in methylene chloride. Tap the column to settle the silica gel, and then add a 1 cm layer of anhydrous sodium sulfate (Section 6.4.6)

Variations among batches of silica gel may affect the elution volume of the various PAH. Therefore, the volume of solvent required to completely elute all of the PAH must be verified by the analyst. The weight of the silica gel can then be adjusted accordingly. Satisfactory recovery (as defined in Section 6.6) of each native PAH in the LCS (8.1.3) must be demonstrated whenever there is a change in the method of preparing the silica gel columns.

B. Acid Alumina Column

Pack a 250 mm x 10 mm glass gravity column as follows:

Insert a clean glass wool plug (Section 4.2.7) into the bottom of the column. Add 6 g of acid alumina prepared as described in Section 6.4.8. Tap the column gently to settle the alumina, and add 1 cm of anhydrous sodium sulfate to the top.

Satisfactory recovery (as defined in Section 6.6) of each native PAH in the LCS (8.1.3) must be demonstrated whenever there is a change in the method of preparing the acid alumina columns.

6.6.2 Column Chromatography Procedure

A. Silica Gel Column

Elute the column with 40 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 1 mL sample extract onto the column using two additional 2 mL rinses of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, begin elution of the column with 25 mL of hexane followed by 25 mL of methylene chloride/hexane (2:3)(v/v). Collect the entire eluate. Concentrate the collected fraction to about 5 mL using the K-D apparatus or a rotary evaporator. Do not allow the extract to go to dryness.

Transfer to a minivial using a hexane rinse and concentrate to 450 μL using a gentle stream of nitrogen. Store the extracts in a refrigerator at 4 ^{o}C or lower away from light until GC/MS analysis (Section 7).

B. Alumina Column

Elute the column with 50 mL of hexane. Let the solvent flow through the column until the head of the liquid in the column is just above the sodium sulfate layer. Close the stopcock to stop solvent flow.

Transfer 1 mL of the sample extract onto the column. Rinse out extract vial with two 1 mL rinses of hexane and add it to the top of the column immediately. To avoid overloading the column, it is suggested that no more than 300 mg of extractable organics be placed on the column.

Just prior to exposure of the sodium sulfate to the air, elute the column with a total of 15 mL of hexane. If the extract is in 1 mL of hexane, and if 2 mL of hexane was used as a rinse, then 12 mL of additional hexane should be used. Collect the effluent and concentrate to about 2 mL using the K-D apparatus or a rotary evaporator.

Transfer to a minivial using a hexane rinse and concentrate to 450 μL using a gentle stream of nitrogen. Store the extracts at 4°C or lower away from light until GC/MS analysis.

7 GC/MS ANALYSIS

7.1 APPARATUS

7.1.1 Gas Chromatograph

An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The GC injection port must be designed for capillary columns. Splitless injection is recommended.

7.1.2 Column

Fused silica columns are required.

A. 30 M long x 0.32 mm ID fused silica capillary column coated with a crosslinked phenyl methyl silicone such as DB-5.

B. Any column equivalent to the DB-5 column may be used as long as it has the same separation capabilities as the DB-5.

7.1.3 Mass Spectrometer

7.1.3.1 Low Resolution

A low resolution mass spectrometer (LRMS) equipped with a 70 eV (nominal) ion source operated in the electron impact ionization mode, and capable of monitoring all of the ions in each Selected Ion Monitoring (SIM) group (Table 13) with a total cycle time of 1 second or less.

7.1.3.2 High Resolution

The high resolution mass spectrometer (HRMS) must be capable of operation in the SIM mode at a resolving power of 8,000. Electron impact ionization must be used. The mass spectrometer must be capable of monitoring all of the ions listed in each of the three SIM descriptors (Table 14) with a total cycle time of 1 second or less.

7.1.4 GC/MS Interface

Any gas chromatograph to mass spectrometer interface may be used as long as it gives acceptable calibration response for each analyte of interest at the desired concentration and achieves the required tuning performance criteria (Sections 7.3.5 and 7.3.6). All components of the interface must be glass or glass-lined materials. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the mass spectrometer ion source without being exposed to the ionizing electron beam.

7.1.5 Data Acquisition System

A computer system must be interfaced to the mass spectrometer. The system must allow the continuous acquisition and storage on machine-readable media of all data obtained throughout the duration of the chromatographic program. The computer must have software that can search any GC/MS data file for ions of a specific mass and plot a Selected Ion Current Profile or SICP (a plot of the abundances of the selected ions versus time or scan number). Software must also be able to integrate, in any SICP, the abundance between specified time or scan-number limits.

The data system must provide hard copies of individual ion chromatograms for selected gas chromatographic time intervals.

The data system must also be able to provide hard copies of a summary report of the results of the GC/MS runs. Figures 14A to 14C show the minimum data that the system must be available to provide.

7.2 REAGENTS

7.2.1 Stock Standard Solution (1.00 μg/μL)

Standard solutions can be prepared from pure standard materials or purchased as certified solutions.

7.2.2 Preparation of Stock Solutions

A. Calibration standards. Prepare stock calibration standard solutions of each of the PAH analytes by accurately weighing the required amount of pure material. Dissolve the material in isooctane and dilute to volume. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard.

Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

B. Internal standards. Prepare stock solutions in isooctane of the fourteen internal standards listed in Table 4 or 4A at concentrations of 1000 ng/μL.

C. Recovery standards. Prepare stock solutions in isooctane of the three recovery standards listed in Table 4 or 4A at concentrations of 1000 ng/μL.

D. Alternate standard. Prepare a stock solution in isooctane of the alternate standard listed in Table 4 or 4A at a concentration of 1000 ng/μL.

E. Surrogate standards. Prepare stock solutions in isooctane of the surrogate standards listed in Table 4 or 4A at a concentration of 1000 ng/µL.

Store stock standard solutions in Teflon®-sealed screw-cap bottles at 4°C and protect from light. Stock standard solutions must be checked frequently for signs of degradation or evaporation, especially just before using them to prepare calibration standard solutions or spiking solutions.

Replace stock standard solutions every 12 months or more frequently if comparison with quality control check samples according to Section 7.4.1 indicates a problem.

7.2.3 Calibration Standards

Prepare calibration standards at a minimum of five concentration levels. One of the calibration standards should be at a concentration near, but above, the method detection limit. The others should include the range of concentrations found in real samples but should not exceed the linear range of the GC/MS system.

Prepare calibration working standard solutions by combining appropriate volumes of individual or mixed calibration standards with internal standard, recovery standards, and alternate standard spiking solution and making up to volume with hexane to obtain the solution concentrations given in Tables 5, 6, and 6A. The suggested ranges are 0.25 ng/µL to 5.0 ng/µL for LRMS and 10 pg/µL to 500 pg/µL for HRMS.

All standards must be stored at 4°C or lower and must be freshly prepared if the check according to Section 7.4.1 indicates a problem.

7.2.4 Internal Standard (IS) Spiking Solution

The concentration of internal standard in the IS spiking solution must be such that the amount of solution added to the calibration standard solution and the sample is at least 2 mL.

Prepare the internal standard spiking solution by using appropriate volumes of stock solutions of Section 7.2.2B to give the concentrations shown in Table 4 or 4A. A volume of 2 mL of either the LRMS or HRMS spiking solution will provide the amount of the internal standards that must be added to the sample (Table 7 or 7A) before extraction to achieve, in a final volume of 500 µL, the sample extract concentrations shown in Table 8 for LRMS and Table 8 or 8A for HRMS analysis. The target concentrations in Tables 8 and 8A are based on a final volume of 500 µL and 100 percent recovery of the internal standards added to the sample.

7.2.5 Recovery Standard Spiking Solution

The concentration of recovery standard in this spiking solution must be such that the amount of solution added to the concentrated sample extract is 50 µL to give a final extract volume of 500 µL.

Use an appropriate volume of stock solution of Section 7.2.2C to prepare a recovery standard spiking solution with the concentrations shown in Table 4or 4A. Store at 4 °C or lower.

A volume of 50 μL of the recovery standard spiking solution shown in Table 4 or 4A will provide the amount of each recovery standard required by Table 7 or 7A to achieve the target sample concentration of Table 8 or 8A. Final volumes, may be adjusted depending on the target detection limit.

7.2.6 Surrogate Standard Spiking Solution

The concentration of surrogate standard in this spiking solution must be such that the amount of solution added to the calibration standard solution and the sorbent module is at least 2 mL.

Prepare the surrogate standard spiking solution by using the appropriate volume of stock solution of Section 7.2.2E to give the concentration shown in Table 4 or 4A. A volume of 2 mL of either the LRMS or HRMS spiking solution will provide the amount of the surrogate standards that must be added to the sample (Table 7 or 7A) before sampling to achieve the sample extract concentrations shown in Table 8 or 8A in a final sample volume of 500 μL.

7.2.7 Alternate Standard Spiking Solution

The concentration of alternate standard in this spiking solution must be such that the amount of solution added to the calibration standard solution and the sample extracts is at least 2 mL.

Prepare the alternate standard spiking solution by using the appropriate volume of stock solution of Section 7.2.2D to give the concentration shown in Table 4 or 4A. A volume of 2 mL of either the LRMS or HRMS spiking solution will provide the amount of the alternate standard that must be added to the sample (Table 7 or 7A) before extraction to achieve the sample extract concentrations shown in Table 8 or 8A in a final sample volume of 500 μL.

7.2.8 Calibration Check Standard

The calibration check standard shall be used for column performance checks, and for continuing calibration checks. Solution #3 from Table 5 shall be the calibration check standard for LRMS, while Solution #3 from Table 6 or 6A shall be the calibration check standard for HRMS.

7.3 INITIAL CALIBRATION

An acceptable initial calibration (7.3.8) is required before any samples are analyzed, and then intermittently throughout sample analyses as dictated by results of the continuing calibration procedures described in Section 7.4. The GC/MS system must be properly calibrated and the performance documented during the initial calibration.

7.3.1 Retention Time Windows

Before sample analysis, determine the retention time windows during which the selected ions will be monitored. Determine Relative Retention Time (RRTs) for each analyte by using the corresponding ^{2}H - labelled standard.

7.3.2 GC Operating Conditions

The GC column performance (Section 7.3.5) must be documented during the initial calibration. Table 10 summarizes GC operating conditions known to produce acceptable results with the column listed. The GC conditions must be established by each analyst for the particular instrumentation by injecting aliquots of the calibration check standard (7.2.8). It may be necessary to adjust the operating conditions slightly based on observations from analysis of these solutions. Other columns and/or conditions may be used as long as column performance criteria of Section 7.3.5 are satisfied.

Thereafter the calibration check standard must be analyzed daily to verify the performance of the system (Section 7.4).

7.3.3 GC/MS Tuning Criteria

A. Low Resolution Mass Spectrometry

Use a compound such perfluorotributylamine (PFTBA) to verify that the intensity of the peaks is acceptable. If PFTBA is used, mass spectral peak profiles for m/z 69, 219 and 264 must be recorded, plotted, and reported. The scan should include a minimum of +/- two peaks (i.e, m/z 67-71 for the m/z 69 profile).

B. High Resolution Mass Spectrometry

Tune the instrument to meet the minimum required resolving power of 8,000 at 192.9888 or any other PFK reference signal close to 128.0626 (naphthalene). Use peak matching and the chosen PFK reference peak to verify that the exact mass of m/z 242.9856 is within 5 ppm of the required value. The selection of the low and high mass ions must be such that they provide the largest voltage jump performed in any of the three mass descriptors.

7.3.4 MS Operating Conditions

A. Low Resolution Mass Spectrometry

Analyze standards and samples with the mass spectrometer operating in the Selected Ion Monitoring (SIM) mode with a total cycle time of 1 second or less.

B. High Resolution Mass Spectrometry

Analyze standards and samples with the mass spectrometer operating in the SIM mode with a total cycle time (including the voltage reset time) of one second or less.

A reference compound such as Perfluorokerosene (PFK) must be used to calibrate the SIM mass range. One PFK ion per mass descriptor is used as a lock-mass ion to correct for mass drifts that occur during the analysis. In addition to the lock-mass ion, several ions characteristic of PFK are monitored as QC check ions (Table 13).

7.3.5 GC Column Performance Criteria

A. The height of the valley between anthracene and phenanthrene at m/z 178 or the ^{2}H-analogs at m/z 188 shall not exceed 50 percent of the taller of the two peaks.

B. The height of the valley between benzo(b)fluoranthene and benzo(k)fluoranthene shall not exceed 60 percent of the taller of the two peaks.

If these criteria are not met and normal column maintenance procedures are not successful, the column must be replaced and the initial calibration repeated.

7.3.6 Mass Spectrometer Performance

A. Low Resolution Mass Spectrometry

Verify acceptable sensitivity during initial calibration. Demonstrate that the instrument will achieve a minimum signal-to-noise ratio of 10:1 for the quantitation and confirmation ions when the calibration standard with the lowest concentration is injected into the GC/MS system.

B. High Resolution Mass Spectrometry

Record the peak profile of the high mass reference signal (m/z 242.9856) obtained during peak matching by using the low-mass PFK ion at m/z 192.9888 (or lower in mass) as a reference. The minimum resolving power of 8,000 must be demonstrated on the high-mass ion while it is transmitted at a lower accelerating voltage than the low-mass reference ion, which is transmitted at full sensitivity.

The format of the peak profile representation must allow manual determination of the resolution, that is, the horizontal axis must be a calibrated mass scale (amu or ppm per division).

The peak width of the high mass ion at 5 percent of the peak height must not exceed 125 ppm in mass.

7.3.7 Calibration Procedure

Using stock standards, prepare at least five calibration standard solutions, using the same solvent that was used in the final sample extract. Keep the recovery standards and the internal standards at fixed concentrations. Adjust the concentrations recommended in Tables 5 and 6, if necessary, to ensure that the sample analyte concentration falls within the calibration range. The calibration curve must be described within the linear range of the method. Calibrate the mass spectrometer response using a 2 μL aliquot of each calibration solution. Analyze each solution once.

Calculate:

A. the relative response factors (RRFs) for each analyte as described in Sections 7.7.1.1, 7.7.1.2, and 7.7.1.3.

B. the mean RRFs as required by Section 7.7.1.4.

C. the standard deviation (SD) and relative standard deviation (RSD) as required by Section 7.7.2.

Report all results as required by Section 10.2.

7.3.8 Criteria for Acceptable Initial Calibration

An acceptable initial calibration must satisfy the following performance criteria:

A. The requirements of Sections 7.3.5 and 7.4.6 must be met.

B. The signal to noise ratio (S/N) for the GC signals present in every selected ion current profile (SICP) must be > 10:1 for the labelled standards and unlabelled analytes.

C. The percent relative standard deviation for the mean relative response factors must be no greater than 30 percent for both the unlabelled analytes and internal standards (Section 7.7.2). Otherwise, take corrective action as required by Section 7.7.2.

7.4 CONTINUING CALIBRATION

The continuing calibration consists of an analysis of the calibration check standard (Section 7.2.8) once during each 12-hour shift as described in Section 7.4.1.

The criteria for acceptable continuing calibration are given in Section 7.4.2. These must be satisfied or else corrective action must be taken as required by Section 7.4.2.

7.4.1 Calibration Check

The calibration check standard (Section 7.2.8) must be analyzed at the beginning and end of each analysis period, or at the beginning of every 12-hour shift if the laboratory operates during consecutive 12 hour shifts.

Inject a 2-μL aliquot of the calibration check standard (Section 7.2.8) into the GC/MS. Use the same data acquisition parameters as those used during the initial calibration.

Check the retention time windows for each of the compounds. They must satisfy the criterion of Section 7.4.2C

Check for GC resolution and peak shape. Document acceptable column performance as described in Section 7.3.5. If these criteria are not met, and normal column maintenance procedures are unsuccessful, the column must be replaced and the calibration repeated.

Calculate the continuing RRF and ΔRRF, the relative percent difference (RPD) between the daily RRF and the initial calibration mean RRF as described in Section 7.7.1.5.

Report the results as required by Section 10.2.

7.4.2 Continuing Calibration Performance Criteria

An acceptable continuing calibration must satisfy the following performance criteria:

A. The signal to noise ratio (S/N) for the GC signals present in the selected ion current profile (SICP) for all labelled and unlabelled standards must be ≥ 10:1.

B. The measured RRFs of all analytes (labelled and unlabelled) must be within 30 percent of the mean values established during the initial calibration. If this criterion is not satisfied, a new initial calibration curve must be established before sample extracts can be analyzed.

C. The retention time for any internal standard must not change by more than 30 seconds from the most recent calibration check. Otherwise, inspect the chromatographic system for malfunctions and make the necessary corrections. Document acceptable performance with a new initial calibration curve.

7.5 GC/MS ANALYSIS

The laboratory may proceed with the analysis of samples and blanks only after demonstrating acceptable performance as specified in Sections 7.3 and 7.4.

Analyze standards, field samples and QA samples (Section 8.1) with the gas chromatograph and mass spectrometer operating under the conditions recommended in Sections 7.3.2 and 7.3.4.

Approximately l hr before HRGC/LRMS or HRGC/HRMS analysis, adjust the sample extract volume to approximately 500 µL. This is done by adding 50 µL of the recovery standard spike solution (Section 7.2.5, and Table 4 or 4A) to the 450 µL final volume (Section 6.6.2) of the concentrated sample extract give the sample extract concentration required by Table 8 or 8A. If the sample volume must be changed to achieve a desired detection limit, the recovery spike solution concentration must be adjusted accordingly to achieve the target concentrations of Table 8 or 8A.

Inject a 2 µL aliquot of the sample extract (Section 6.6.2) on to the DB-5 column. Use the same volume as that used during calibration. Recommended GC/MS operating conditions are described in Section 7.3.

The presence of a given PAH is qualitatively confirmed if the criteria of Section 7.6.1 are satisfied.

The response for any quantitation or confirmation ion in the sample extract must not exceed the response of the highest concentration calibration standard.

Collect, record, and store the data for the calculations required by Sections 9.1.7, 9.1.8, 9.1.9, and 9.1.10. Report the results as required by Section 10.2.

7.6 QUALITATIVE ANALYSIS

7.6.1 Identification Criteria

7.6.1.1 Ion Criteria

For LRMS analysis, all quantitation and confirmation ions (Table 13) must be present.

7.6.1.2 Relative Retention Time (RRT) Criteria

The relative retention time (RRT) of the analyte compared to the RRT for the ^{2}H-standards must be within ±0.008 RRT units of the relative retention times obtained from the continuing calibration (or initial calibration if this applies).

7.6.1.3 Signal to Noise Ratio

The signal to mean noise ratio must be 10:1 for the internal standards. This ratio for the unlabelled compounds must be greater than 2.5 to 1 for the quantitation ions for HRMS and for both quantitation and confirmation ions for LRMS.

If broad background interference restricts the sensitivity of the GC/MS analysis, the analyst must employ additional cleanup on the archive sample and reanalyze.

7.7 QUANTITATIVE ANALYSIS

7.7.1 Relative Response Factors (RRFs)

7.7.1.1 RRF for Unlabelled PAH and Surrogate Standards from Initial Calibration Data

Use the results of the calibration and Equation 429-13 to calculate the relative response factors (RRFs) for each calibration compound and surrogate standard in each calibration solution (Tables 5 or 5A). Table 11 shows the assignments of the internal standards for calculation of the RRFs for the calibration solution shown in Table 5. Table 11A shows the assignments of the internal standards for calculation of the RRFs for the calibration solution shown in Table 5A. Report the results as required by Section 10.2.

7.7.1.2 RRF for Determining Internal Standard Recovery

Use the results of the calibration in Equation 429-18 to calculate the relative response factor for each internal standard relative to an appropriate recovery standard. Table 11 shows the assignments of the recovery standards for calculating internal standard recoveries for the calibration solution shown in Table 5. Table 11A shows the assignments of the recovery standards for calculating internal standard recoveries for the calibration solution shown in Table 5A. Report the results as required by Section 10.2.

7.7.1.3 RRF for Determining Alternate Standard Recovery

Use the calibration results and Equation 429-19 to calculate the response factor for the alternate standard relative to the appropriate recovery standard. Table 11 shows the assignment of the recovery standards for calculating alternate standard recovery for the calibration solution shown in Table 5. for the calibration solution shown in Table 5. Report the results as required by Section 10.2.

7.7.1.4 Mean Relative Response Factor

Use Equation 429-20 to calculate the mean RRF for each compound (unlabelled calibration standards, surrogate standards, internal standards and alternate standard). This is the average of the five RRFs calculated for each compound (one RRF calculated for each calibration solution). The mean RRF may be used if the linearity criterion of Section 7.7.2 is satisfied.
Report the results as required by Section 10.2.

7.7.1.5 RRF from Continuing Calibration Data

Analyze one or more calibration standards (one must be the medium level standard) on each work shift of 12 hours or less. Use Equations 429-17, 429-18, and 429-19 to calculate the RRFs for each analyte. Use Equation 429-22 to calculate ΔRRF, the relative percent difference between the daily RRF and the mean RRF calculated during initial calibration. Check whether the performance criterion of Section 7.4.2B is satisfied. Report the results as required by Section 10.2.

7.7.2 Relative Standard Deviation of Relative Response Factors

For each analyte, calculate the sample standard deviation (SD) of the RRFs used to calculate the mean RRF. Use Equation 429-21 to calculate the percent relative standard deviation (%RSD) for each analyte. The analyst may use the mean RRF if the percent relative standard deviation of the RRFs is 30% or less. If the RSD requirement is not satisfied, analyze additional aliquots of appropriate calibration solutions to obtain an acceptable RSD of RRFs over the entire concentration range, or take action to improve GC/MS performance. Otherwise, use the complete five point calibration curve for that compound.

8 QUALITY ASSURANCE/QUALITY CONTROL

Each laboratory that uses this method is required to operate a formal quality control program. The minimum quality control requirements of this program consists of an initial demonstration of laboratory capability (according to Sections 7.3 and 8.1.3.1), and periodic analysis of blanks and spiked samples as required in Sections 8.1.1 and 8.1.3.2 as a continuing check on performance.

The laboratory must maintain performance records to document the quality of data that are generated. The results of the data quality checks must be compared with the method performance criteria to determine if the analytical results meet the performance requirements of the method. The laboratory must generate accuracy statements as described in Section 8.4.1.

8.1 QA SAMPLES

8.1.1 Laboratory Method Blank

The analyst must run a laboratory method blank with each set of 15 or fewer samples. The method blank must be a resin sample from the same batch used to prepare the sampling cartridge and the laboratory control samples. The method blank must be prepared and stored as described in Sections 4.3.4 and 4.3.5.

The analyst shall perform all of the same procedures on the method blank as are performed on the solid samples (Section 6.5.2.1) from the beginning of sample extraction through to the end of the GC/MS analytical procedures.

8.1.2 Performance Evaluation Samples

The laboratory should analyze performance evaluation samples quarterly when these samples become available. These samples must be prepared and analyzed by the same methods used for the field samples. Performance for the most recent quarter should be reported with the results of the sample analysis.

8.1.3 Laboratory Control Sample (LCS)

8.1.3.1 Initial Demonstration of Laboratory Capability

Before performing sample analyses for the first time, the analyst shall demonstrate the ability to generate results of acceptable precision and accuracy by using the following procedures.

Prepare spiking solutions from stock standards prepared independently from those used for calibration. Spike at least four resin samples cleaned as described in Section 4.2.2 with each of the target unlabelled analytes as indicated in Table 9. Blank resin contamination levels must be no greater than 10 percent of the levels of the spiked analytes. Add the amounts of internal standards required by Table 7 or 7A. Add the alternate standard to the extract to monitor the efficiency of the cleanup procedure.

The LCS spikes shall undergo all of the same procedures as are performed on the solid samples (Section 6.5.1.2) from the beginning of sample extraction through to the end of the GC/MS analytical procedures.

<u>Calculate:</u>

(A) percent recoveries for the internal standards and alternate standard,
(B) the mass of each target analyte in μg/sample or ng/sample,
(C) the average of the results for the four analyses in μg/sample or ng/sample,
(D) the average recovery (R) as a percentage of the amount added, and
(E) the relative standard deviation S_R.
Report the results as required by Section 10.2.4.

If all the acceptance criteria of Section 8.2.6 are satisfied for all of the target PAH, the analyst may begin analysis of blanks and samples. Otherwise, corrective action must be taken as required by Section 8.2.6.

8.1.3.2 Ongoing Analysis of LCS

The analyst must run two laboratory control samples with each set of 15 or fewer samples. The resin for the LCS must be taken from the same batch used to prepare the sampling cartridge and the laboratory method blank. The LCS resin must be prepared and stored as described in Sections 4.3.4 and 4.3.5.

Prepare spiking solutions from stock standards prepared independently from those used for calibration. Spike each resin sample with each of the target unlabelled analytes as indicated in Table 9. Blank resin contamination levels must be no greater than 10 percent of the levels of the spiked analytes. Add the amounts of internal standards required by Table 7 or 7A. Add the alternate standard to the extract to monitor the efficiency of the cleanup procedure.

The LCS spikes shall undergo all of the same procedures as are performed on the solid samples (Section 6.5.1.2) from the beginning of sample extraction through to the end of the GC/MS analytical procedures.

Calculate:

(A) percent recoveries for the internal standards and alternate standard,
(B) the mass of each target analyte in μg/sample or ng/sample,
(C) the average of the results for the two analyses in μg/sample or ng/sample,
(D) the average recovery as a percentage of the amount added, and
(E) the relative percent difference for the two analyses.

Report the results as required by Section 10.2.

Add the results which satisfy the performance requirements of Section 8.2.6 to the results of the initial LCS analyses (8.1.3.1) and previous ongoing data for each compound in the LCS sample.

Update the charts as described in Section 8.4.1.

8.2 ACCEPTANCE CRITERIA

8.2.1 Blank Trains

The levels of any unlabelled analyte quantified in the blank train must not exceed 20 percent of the level of that analyte in the sampling train. If this criterion cannot be met, calculate a reporting limit that is five times the blank value (Equations 429-32 and 429-33). Do not subtract the blank value from the sample value.

8.2.2 Surrogate Standard Recovery

Acceptable surrogate (field spike) recoveries should range from 50 to 150 percent. If field spike recoveries are not within the acceptable range, this must be clearly indicated in the laboratory report. The affected sampling run must be identified in the report of the calculated emissions data.

8.2.3 Internal Standard Recovery

Recoveries for each of the internal standards must be greater than 50 percent and less than 150 percent of the known value.

If internal standard recoveries are outside of the acceptable limits, the signal to noise ratio of the internal standard must be greater than 10. Otherwise the analytical procedure must be repeated on the stored portion of the extract.

NOTE: This criterion is used to assess method performance. As this is an isotope dilution technique, it is, when properly applied, independent of internal standard recovery. Lower recoveries do not necessarily invalidate the analytical results for PAH, but they may result in higher detection limits than are desired.

If low internal standard recoveries result in detection limits that are unacceptable, the cleanup and GC/MS analysis must be repeated with the stored portion of the extract. If the analysis of the archive sample gives low recoveries and high detection limits, the results of both analyses must be reported.

8.2.4 Laboratory Method Blank

The laboratory method blank must not contain any of the target analytes listed in Table 1 at levels exceeding the PQL or 5 percent of the analyte concentration in the field sample.

If the method blank is contaminated, check solvents, reagents, standard solutions apparatus and glassware to locate and eliminate the source of contamination before any more samples are analyzed. Table 3 shows those compounds that commonly occur as contaminants in the method blank, and the ranges of concentrations that have been reported.

If field samples were processed with a laboratory method blank that showed PAH levels greater than 5 percent of the field sample, they must be re-analyzed using the archived portion of the sample extract.

Recoveries of the internal standards must satisfy the requirements of 8.2.3. If the internal standard recoveries are less than 50%, the S/N ratio must be greater than 10 for the internal standard.

8.2.5 Performance Evaluation Sample

The following will be a requirement when performance evaluation samples become available, and performance criteria have been established:

Performance for the most recent quarter must be reported with the results of the sample analysis. If the performance criteria (to be established) are not achieved, corrective action must be taken and acceptable performance demonstrated before sample analysis can be resumed.

8.2.6 Laboratory Control Samples

8.2.6.1 Initial and Ongoing Analysis

The signal of each analyte in the initial and ongoing laboratory control samples must be at least 10 times that of the background.

Acceptable accuracy is a percent recovery between 50 and 150 percent.
Acceptable precision for the initial LCS samples is a relative standard deviation (RSD) of 30 percent or less.
Acceptable precision for the ongoing analysis of duplicate samples is a relative percent difference of 50 percent or less.

If the RSD for the initial demonstration exceeds the precision limit, or any calculated recovery falls outside the range for accuracy, the laboratory performance for that analyte is unacceptable.
If the RPD for any ongoing duplicate analyses exceeds the precision limit, or any calculated recovery falls outside the range for accuracy, the laboratory performance for that analyte is unacceptable.

Beginning with Section 8.1.3.1, repeat the test for those analytes that failed to meet the performance criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.1.3.1 for the initial analysis and Section 8.3.1.2 for the ongoing analysis.

8.3 ESTIMATION OF THE METHOD DETECTION LIMIT (MDL) AND PRACTICAL QUANTITATION LIMIT (PQL)

8.3.1 Initial Estimate of MDL and PQL

The analyst shall prepare a batch of XAD-2 resin as described in Sections 4.2.2.1 to 4.2.2.3, then check for contamination as required by Section 4.2.2.4. Identify those PAH analytes present at background levels that are too high for the MDL determination. Use the procedure of Appendix A to calculate MDLs for the remaining target PAH compounds. A suggested initial spike level for the MDL determination is 5 times a theoretical method quantitation limit (TMQL) estimated according to Equation 429-16.

$$TMQL = C \times \frac{V}{P} \times 100 \times 2 \qquad 429\text{-}16$$

Where:

C = the concentration of the PAH in the lowest concentration calibration standard used in the initial calibration, (ng/μL)

V = the final extract volume, (μL)

P = the assumed percent recovery (50%) of the internal standard

2 = a factor to account for the fact that the final extract volume (V) contains one half of the analyte in the sample. The other half is archived.

8.3.2 Ongoing Estimation of MDL and PQL

Once every quarter in which this method is used, the analytical laboratory must analyze one spiked resin sample as described in Appendix A. Include all initial and quarterly results in the calculation of the standard deviation and MDL for each analyte that has not been identified as a common contaminant of the XAD-2 resin.

If the MDL for any analyte exceeds the MDL established during the initial determination, take corrective action as necessary, and repeat the monthly analysis. If any MDL still exceeds the initial MDL, then the initial standard deviation estimation procedure (Appendix A) must be repeated.

8.4 LABORATORY PERFORMANCE

The analyst must have documented standard operating procedures (SOPs) that contain specific stepwise instructions for carrying out this method. The SOPs must be readily available and followed by all personnel conducting the work. The SOP must be made available for review upon request by the Executive Officer, the tester or reviewer of the analytical results. The analyst may impose restrictions on the dissemination of the information in the SOP.

The analyst must have documented precision and accuracy statements (Section 8.4.1) readily available.

The analyst must have results of the initial and ongoing estimates of the MDL (Sections 8.3.1 and 8.3.2) readily available.

8.4.1 Precision and Accuracy Statement

The precision and accuracy statements for the analytical procedure shall be based on the results of the initial and ongoing LCS analyses. The frequency of analysis is stated in Section 8.1.3.

Prepare a table of the recoveries and the relative percent difference for each ongoing analysis of the LCS and LCS duplicate. Figure 15A is an example of such a table. This must be included in the analytical data package submitted for each set of sample analyses.

Prepare a quality control chart for each target analyte that provides a graphic representation of continued laboratory performance for that target analyte. Figure 15B is an example QC chart for benzo(a)pyrene.

9. CALCULATIONS

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

9.1 ANALYST'S CALCULATIONS

The analyst shall carry out the calculations described in Sections 9.1.1 to 9.1.11.

9.1.1 Relative Response Factors (RRF) for Unlabelled PAH and Surrogate Standards

Calculate the RRF for each target unlabelled PAH analyte and surrogate standard in each calibration solution . Use Equation 429-17 and the data obtained during initial calibration (7.3.7) or continuing calibration (7.4.1).

$$RRF_s = \frac{A_s \times Q_{is}}{A_{is} \times Q_s} \qquad 429\text{-}17$$

Where:

A_s = Area of the response for characteristic ions of the unlabelled analyte or surrogate standard (Tables 11 or 11A, 13, and 14).

A_{is} = Area of the response for characteristic ions of the appropriate internal standard (Tables 11 or 11A, 13, and 14).

Q_s = Amount of the unlabelled PAH calibration analyte or surrogate standard injected on to GC column, ng.

Q_{is} = Amount of the appropriate internal standard injected on to GC column, ng.

9.1.2 RRF for Determination of Internal Standard Recovery

Calculate RRF_{is} according to Equation 429-18, using data obtained from the analysis of the calibration standards.

$$RRF_{is} = \frac{A_{is} \times Q_{rs}}{A_{rs} \times Q_{is}} \qquad 429\text{-}18$$

Where:

A_{rs} = Area of the response for characteristic ions of the appropriate recovery standard (Tables 11 or 11A, 13, and 14).

Q_{rs} = Amount of the appropriate recovery standard injected on to GC column, ng.

9.1.3 RRF for Determination of Alternate Standard Recovery

Calculate RRF_{as} according to Equation 429-19, using data obtained from the analysis of the calibration standards.

$$RRF_{as} = \frac{A_{as} \times Q_{rs}}{A_{rs} \times Q_{as}} \qquad 429\text{-}19$$

Where:

A_{as} = Area of the response for characteristic ions of the alternate standard (Tables 13 and 14).

Q_{as} = Amount of alternate standard injected on to the GC column, ng.

9.1.4 Mean Relative Response Factors ($\overline{RRF}$)

Calculate the mean RRF for each target unlabelled PAH, surrogate standard, internal standard and alternate standard using Equation 429-20 and the RRFs calculated according to Sections 9.1.1, 9.1.2, and 9.1.3.

$$\overline{RRF} = \frac{1}{n} \sum_{i=1}^{n} (RRF)_i \qquad 429\text{-}20$$

Where:

RRF_i = RRF calculated for calibration solution "i" using one of Equations 429-17, 429-18 or 429-19.

n = The number of data points derived from the calibration. The minimum requirement is a five-point calibration (Section 7.2.3, Tables 5 and 6 or 6A)

9.1.5 Percent Relative Standard Deviation (%RSD) of Relative Response Factors

Use Equation 429-21 to calculate the relative standard deviation of the Relative Response Factors for each analyte.

$$\%RSD = \frac{SD}{\overline{RRF}} \times 100\% \qquad 429\text{-}21$$

Where:

$\overline{RRF}$ = Mean relative response factor of a given analyte as defined in Sections 7.7.1.4 and 9.1.4.

SD = The sample standard deviation of the relative response factors used to calculate the mean RRF.

9.1.6 Continuing Calibration ΔRRF

Use Equation 429-22 to calculate ΔRRF, the relative percent difference (RPD) between the daily RRF and the mean RRF calculated during initial calibration.

$$\Delta RRF = \frac{RRF_c - \overline{RRF}}{\overline{RRF}} \times 100\% \qquad 429\text{-}22$$

Where:

RRF_c = The RRF of a given analyte obtained from the continuing calibration (Section 7.4).

9.1.7 Percent Recovery of Internal Standard, R_{is}

Calculate the percent recovery, R_{is} for each internal standard in the sample extract, using Equation 429-23.

$$R_{is} = \frac{A_{is} \times Q_{rs}}{A_{rs} \times \overline{RRF_{is}} \times Q_{is}} \times 100\% \qquad 429\text{-}23$$

Where:

$\overline{RRF}_{is}$ = Mean relative response factor for internal standard (Equations 429-18 and 429-20).

9.1.8 Percent Recovery of Surrogate Standard, R_{ss}

Calculate the percent recovery, R_{ss} for each surrogate standard in the sample extract, using Equation 429-24.

$$R_{ss} = \frac{A_{ss} \times Q_{is}}{A_{is} \times \overline{RRF_{s}} \times Q_{ss}} \times 100\% \qquad 429\text{-}24$$

Where:

A_{ss} = Area of the response for characteristic ions of the surrogate standard (Tables 13 and 14).

Q_{ss} = Amount of the surrogate standard added to resin cartridge before sampling, ng.

$\overline{RRF}_{s}$ = Mean relative response factor for surrogate standard (Equations 429-17 and 429-20).

9.1.9 Percent Recovery of Alternate Standard, R_{as}

Calculate the percent recovery, R_{as} for the alternate standard in the sample extract, using Equation 429-25.

$$R_{as} = \frac{A_{as} \times Q_{rs}}{A_{rs} \times \overline{RRF_{as}} \times Q_{as}} \times 100\% \qquad 429\text{-}25$$

Where:

$\overline{RRF}_{as}$ = Mean relative response factor for alternate standard (Equations 429-19 and 429-20).

9.1.10 Mass of the Target Analytes and Surrogate Standards in Emissions Sample or Blank Train

Use Equation 429-26 to determine the total mass of each PAH compound or surrogate standard in the sample:

Report the PQL (9.1.11) for those analytes that were not present at levels higher than the PQL provided to the tester prior to testing (2.3.3).

$$M = \frac{Q_{is} \times A_s}{A_{is} \times \overline{RRF}} \qquad 429\text{-}26$$

Where:

M = Mass (ng) of surrogate standard (M_s) or target analyte (M_t) detected in the sample.

Q_{is} = Amount of internal standard or surrogate standard added to each sample.

A_s = Area of the response for characteristic ions of the unlabelled analyte or surrogate standard (Tables 13 and 14).

A_{is} = Area of the response for characteristic ions of the appropriate internal standard (Tables 13, and 14).

$\overline{RRF}$ = Mean relative response factor of a given analyte calculated as required by Sections 7.7.1.4 and 9.1.4.

9.1.11 Analytical Reporting Limit

The analyst shall report the PQL (Section 2.3.3) for those analytes that were not present in the emissions sample or blank train at levels higher than the pre-test estimate of the PQL.

9.2 TESTER'S CALCULATIONS

9.2.1 Sample/Blank Train PAH Mass Ratio

Use Equation 429-27 to calculate the sample/blank train mass ratio for each PAH detected at levels above the MDL in both the field sample and the blank train.

$$\text{RATIO} = \frac{M_t}{M_{BT}} \qquad 429\text{-}27$$

Where:

M_t = Mass of target PAH analyte detected in the sampling train (Equation 429-26).

M_{BT} = Mass of the same PAH analyte detected in the blank train.

If the sample to blank train PAH mass ratio is less than five, calculate the reporting limit for the tested source as required by Section 9.2.4.2. Do not calculate M_c (Section 9.2.2) or M_e (Section 9.2.3) for the emissions report.

9.2.2 PAH Concentration in Emissions

Use Equation 429-28 to calculate the concentration in the emissions of 1) the PAH analytes detected in the sampling train but not in the blank train, and 2) the PAH analytes that satisfy the minimum sample to blank train mass ratio required by Section 9.2.1.

$$M_c = \frac{M_t}{V_{m(std)}} \times \frac{1}{0.028317} \qquad 429\text{-}28$$

Where:

M_c = Concentration of PAH in the gas, ng/dscm, corrected to standard conditions of 20°C, 760 mm Hg (68°F, 29.92 in. Hg) on dry basis.

M_t = Mass of PAH compound in gas sample, ng (Equation 429-26)

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscf (Equation 429-10)

0.028317 = Factor for converting dscf to dscm.

9.2.3 PAH Mass Emission Rate

Use Equation 429-29 to calculate the mass emission rate for each PAH compound that satisfies the minimum sample/blank train PAH mass ratio (Section 9.2.1).

$$M_e = \frac{M_s}{V_{m(std)}} \times \frac{Q_{std}}{60} \qquad 429\text{-}29$$

Where:

M_e = Mass emission rate for PAH analyte, ng/second

M_t = Mass of PAH compound in the gas sample, ng (Equation 429-26)

Q_{std} = Average stack gas dry volumetric flow rate corrected to standard conditions, dscf/min.

60 = Factor for converting minutes to seconds

9.2.4 Source Reporting Limit

9.2.4.1 PAH Not Detected in Either Sampling or Blank Train

Use Equation 429-30 or 429-31 to calculate the reporting limit for those analytes that were not detected at levels above the PQL in either the sampling or blank train.

$$RL_{cs} = \frac{PQL}{V_{m(std)}} \times \frac{1}{0.028317} \qquad 429\text{-}30$$

$$RL_{es} = \frac{PQL}{V_{m(std)}} \times \frac{Q_{std}}{60} \qquad 429\text{-}31$$

Where:

RL_{cs} = Reporting limit for the tested source, (ng/dscm), corrected to standard conditions of 20°C, 760 mm Hg (68°F, 29-92 in. Hg) on dry basis.

RL_{es} = Reporting limit for the tested source, (ng/sec.).

0.028317 = Factor for converting dscf to dscm.

60 = Factor for converting minutes to seconds.

9.2.4.2 PAH Detected in Blank Train and Sample/Blank Train Ratio <5

If the sample to blank train PAH mass ratio is less than five, then Equation 429-32 or 429-33 shall be used to calculate the reporting limit for that PAH.

$$RL_{cb} = \frac{5 \times M_{BT}}{V_{m(std)}} \times \frac{1}{0.028317} \qquad 429\text{-}32$$

$$RL_{eb} = \frac{5 \times M_{BT}}{V_{m(std)}} \times \frac{Q_{std}}{60} \qquad 429\text{-}33$$

Where:

RL_{cb} = Reporting limit for the tested source, (ng/dscm), corrected to standard conditions of 20°C, 760 mm Hg (68°F, 29-92 in. Hg) on dry basis.

RL_{eb} = Reporting limit for the tested source, (ng/sec.).

M_{BT} = The total mass of that PAH analyte in the field blank train.

10. REPORTING REQUIREMENTS

The source test protocol must contain all the sampling and analytical data required by Sections 2.2 to 2.5, 4.2.1.1, and 4.2.2.4, as well as the information listed in Sections 10.1 and 10.2 that pertain to identification and quantitation of the samples.

The emissions test report must contain all of the sampling and analytical data necessary to calculate emissions values for the target analytes or to demonstrate satisfactory performance of the method.

The end user or reviewer should be able to obtain from the source test report all information necessary to recalculate all reported test method results or to verify that all required procedures were performed.

Any deviations from the procedures described in this method must be documented in the analytical and sampling report.

10.1 SOURCE TEST PROTOCOL

At a minimum, the source test protocol must include all of the data required by Section 2.2 and the information listed in Sections 10.1.1 through 10.1.4.

10.1.1 Preparation of Filters

A. Manufacturer's lot number for the batch of filters to be used in the test.

B. Contamination check of filter (Section 4.2.1.1)

- (i) Date of cleaning.
- (ii) Date of PAH analysis.
- (iii) Table of results of PAH analysis required by Section 4.2.1. The analytical report must include all of the data listed in Section 10.2.

C. Storage conditions prior to the test (4.3.3)

10.1.2 Preparation of XAD-2 resin

A. ID for the batch to be used in the test. The same batch must be used for the sampling train and the laboratory QC samples.

B. Contamination check of resin (Sections 4.2.2.1 to 4.2.2.4)

- (i) Date of cleaning.
- (ii) Date of PAH analysis.
- (iii) Table of results of PAH analysis required by Secton 4.2.2.4. The analytical report must include all of the data listed in Section 10.2.

C. Addition of surrogate standards to the resin cartridge.

- (i) Amount of each compound.
- (ii) Date of spiking.

D. Storage conditions prior to the test (Section 4.3.3)

10.1.3 Method Detection Limits and Practical Quantitation Limits

The MDL and PQL for each target analyte determined as required by Sections 2.3.2 and 2.3.3.

10.1.4 Target Sampling Parameters

A. Source target concentration of each emitted PAH of interest.

B. Results of calculations required by Sections 2.5.2 to 2.5.5.

Figure 9 shows the minimum required calculations of target sampling parameters.

10.2 LABORATORY REPORT

The analyst must generate a laboratory report for each pre-test analysis of the sampling media (Sections 2.3, 4.2.2.1, and 4.2.2.4) and each post-test analysis of the sampling trains and laboratory QC samples.

A minimum of 7 post-test analyses are required to determine the emissions from the source and to document the quality of the emissions data. These are the analyses of three sampling runs, one blank train, one laboratory method blank and two laboratory control samples.

At a minimum, any report (data package) from the analyst to the tester shall contain the information listed in Sections 10.2.1 to 10.2.6 pertaining to identification and PAH quantitation of all samples.

10.2.1 Five-point Initial Calibration

The report of the results of the initial five-point calibration must include the data listed in A, B, and C below:

A. Mass chromatograms for each initial calibration solution that show at a minimum:

(i) Instrument ID,
(ii) laboratory sample ID on each chromatogram.
(iii) date and time of GC/MS analysis,
(iv) mass of monitored ions for each compound in the calibration solution - unlabelled PAH, internal standard, surrogate standard, alternate standard and recovery standard,
(v) retention time for each compound in the calibration solution, and
(vi) either peak height or area of the signals observed for the monitored ion masses.

B. A summary table of the data obtained for each initial calibration solution that shows at a minimum:

(i) Instrument ID,
(ii) laboratory sample ID,
(iii) date and time of GC/MS analysis,
(iv) retention time for each compound - unlabelled PAH, internal standard, surrogate standard, alternate standard and recovery standard,
(v) relative retention time for each unlabelled PAH,
(vi) either peak height or area of the signals observed for the monitored ion masses,
(vii) the relative response factors for each unlabelled PAH, internal standard, surrogate standard, and alternate standard, and
(viii) analyst's signature

Figure 14A is an example of a summary table that contains the minimum required information for the analysis of a single calibration solution.

C. A summary table that shows at a minimum:

(i) Instrument ID,
(ii) the date and time of the GC/MS analysis,
(iii) the relative response factor (RRF) calculated for each unlabelled PAH, internal standard, surrogate standard, and alternate standard in each calibration solution,
(iv) the average relative response factor ($\overline{RRF}$) calculated for the five point calibration,
(v) the relative standard deviation of the relative response factors, and
(vi) the recovery of each internal standard in percent.

Figure 14B is an example of a report that contains the minimum required information for a five point calibration summary.

10.2.2 Continuing Calibration

The report of the results of a continuing calibration must include the data listed in 10.2.2 A, B, and C below:

A. Mass chromatogram that shows at a minimum the information listed in 10.2.1 A.

B. A summary table of the raw data obtained for the continuing calibration that shows at a minimum, the information listed in 10.2.1 B.

C. A summary table that shows at a minimum:

(i) the relative response factor (RRF) for each unlabelled PAH, internal standard, surrogate standard, and alternate standard in the continuing calibration solution,
(ii) the average relative response factor ($\overline{RRF}$) for each compound calculated for the five point calibration,

(iii) ΔRRF for each unlabelled PAH, internal standard, surrogate standard, and alternate standard in the continuing calibration solution,
(iv) the recovery of each internal standard in percent.

Figure 14C is an example of a summary report that contains the minimum information required by Section 10.2.2C for the analysis of the continuing calibration solution.

10.2.3 Laboratory Method Blank

The laboratory report of the results of the analysis of the method blank must include at a minimum the data listed in 10.2.3 A, B, and C below:

A. Mass chromatograms that show at a minimum the information listed in 10.2.1 A.

B. A summary table of the data obtained for each method blank that shows at a minimum, the information listed in 10.2.5 B.

C. A summary table that reports the same data as listed in 10.2.5 C below.

10.2.4 Laboratory Control Samples

The report of the results of the analysis of the LCS samples must include at a minimum the data listed in 10.2.4 A, B, and C below:

A. Mass chromatograms that show at a minimum the information listed in 10.2.1 A.

B. A summary table of the raw data for each sample that shows at a minimum, the information listed in 10.2.1 B, and in addition:

(i) Client's sample ID
(ii) mass of each analyte,
(iii) the recovery of each internal standard, and alternate standard,

Figure 16A is an example of a summary table that contains the minimum information required by 10.2.4 B.

C. A summary table that reports for the two LCS analyses:

(i) client's sample ID,
(ii) sample matrix description,
(iii) date of cleaning of the XAD-2 resin,
(iv) lot number for the resin (resin for all field samples and QA samples must come from the same lot),
(v) date of extraction of LCS samples,

Figure 15A is an example of a summary table that contains the minimum information required by 10.2.4 C.

10.2.5 Emissions Samples

The report of the results of the analyses of the three sampling trains and the blabk train must include the data listed in 10.2.5 A, B, and C below:

A. Mass chromatograms that show at a minimum the information listed in 10.2.1 A, and in addition,

(i) client's sample ID

B. A summary table of the data for the analysis of each sample that shows at a minimum, the information listed in 10.2.1 B, and in addition,

(i) client's sample ID
(ii) Date of five point initial calibration (ICAL)
(iii) ICAL ID,
(iv) mass of each analyte,
(v) the recovery of each internal standard, alternate standard and surrogate standards in percent.

Figure 16A is an example of a summary table that contains the minimum information required by 10.2.5 B.

C. A summary table that reports:

(i) client's sample ID (from a chain of custody record submitted by the tester),
(ii) sample matrix description,
(iii) date of cleaning of the XAD-2 resin,
(iv) lot number for the resin (resin for all field samples and QA samples must come from the same lot),
(ii) date of submittal of the tester's samples
(v) date of extraction of samples,
(vi) Initial calibration Run ID,
(vii) Continuing calibration ID

Figure 16B is an example of a summary table that contains the minimum information required by 10.2.5C.

10.2.6 Data Flags

The laboratory report must include an explanation of any qualifiers that are used to indicate specific qualities of the data.

10.3 EMISSIONS TEST REPORT

The emissions test report should include narrative that describes how the test was done. The tester's report must also include all the appropriate sections used in a report from a Method 5 test such as a description of the plant process, sampling port locations, control equipment, fuel being used, generai plant load conditions during the test (description of plant production equipment problems, etc.), and anything else necessary to characterize the condition being tested.

The tester's report must also include all of the information listed in Sections 10.3.1 to 10.3.4.

10.3.1 Tester's Summary of Analytical Results

The tester must summarize the results of the minimum seven analyses required for each source test. At a minimum, the summary must contain the information listed in Figure 17A including all data flags.

The tester must obtain the detailed analytical results (Section 10.2) from the laboratory and include them in the appendices as required below.

10.3.2 Field Data Summary

The report from the tester to the end user must contain a field data summary. This summary must include at a minimum a table of the results of the calculations required by Section 4.5. as well as the values which were used to calculate the reported results. Figure 17B is an example of a field data summary that contains the minimum required information.

10.3.3 PAH Emissions Results

Figure 17C show the calculations of the concentrations and mass emission rates of the target PAH. The reviewer should be able to use the data in Figures 17A and 17B to check the calculations in Figure 17C. The reviewer should also be able to check the appendix to the report to determine the accuracy and the quality of the data summarized by the tester in Figures 17A and 17B.

10.3.4 Appendix to the Emissions Test Report

At a minimum, the following raw data or signed copies must be included in an appendix to the emissions test report.

A. Record of data for sample site selection and minimum number of traverse points.

B. Moisture determination for isokinetic settings.

C. Velocity traverse data.

D. Gas analysis for determination of molecular weight.

E. Calibration records.

F. Method 429 sampling run sheets.

G. PAH laboratory reports listed in Section 10.2

The information listed above is to be considered as the minimum that should be included to characterize a given operating condition. The end user or the executive officer may require additional information for any given project.

11. BIBLIOGRAPHY

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11.3 U.S. Environmental Protection Agency/Office of Solid Waste, Washington D.C., Method 3630B. Silica Gel Cleanup. In "Test Methods for Evaluating Solid Waste-Physical/Chemical Methods" SW-846 (1986).

11.4 Thomason, J.R., ed., Cleaning of Laboratory Glassware. Section 3, A, pp 1-7 in "Analysis of Pesticide Residues in Human and Environmental Samples", Environmental Protection Agency, Research Triangle Park, N.C. (1974).

11.5 ARB Method 428. Determination of Polychlorinated Dibenzo-p-dioxin (PCDD) and Polychlorinated Dibenzofuran (PCDF) Emissions From Stationary Sources. September, 1990.

11.6 U. S. Environmental Protection Agency, Method 1625 Revision B - Semivolatile Organic Compounds by Isotope Dilution. 40 CFR Ch.1 (7-1-95 Edition) Pt. 136, App. A.

11.7 Rom, Jerome J., Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency. Research Triangle Park, NC. APTD-0576. March, 1972.

11.8 Shigehara, R.T., Adjustments in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News, 2: 4-11. October, 1974

11.9 "Prudent Practices in the Laboratory. Handling and Disposal of Chemicals," National Academy Press. Washington D.C. 1995.

TABLE 1

METHOD 429 TARGET ANALYTES

Naphthalene
2-Methylnaphthalene
Acenaphthene
Acenaphthylene
Fluorene
Phenanthrene
Anthracene
Fluoranthene
Pyrene
Benzo(a)anthracene
Chrysene
Benzo(b)fluoranthene
Benzo(k)fluoranthene
Benzo(e)pyrene
Benzo(a)pyrene
Perylene
Indeno(1,2,3-cd)pyrene
Dibenz(a,h)anthracene
Benzo(ghi)perylene

TABLE 2

PRACTICAL QUANTITATION LIMITS FOR TARGET PAHs

	LRMS (µg/sample)	HRMS (ng/sample)	
Naphthalene	1.00 244	480	370
2-Methylnaphthalene	1.25	66	19
Acenaphthene	0.210	5.0	5.0
Acenaphthylene	0.104	5.0	5.0
Fluorene	0.207	16.5	5.5
Phenanthrene	0.85	22	14
Anthracene	0.146	5.0	5.0
Fluoranthene	0.346	5.0	5.0
Pyrene	0.191	5.0	5.0
Benzo(a)anthracene	0.167	5.0	5.0
Chrysene	0.272	5.0	5.0
Benzo(b)fluoranthene	1.119	5.0	5.0
Benzo(k)fluoranthene	0.738	5.0	5.0
Benzo(e)pyrene	0.146	5.0	5.0
Benzo(a)pyrene	0.191	5.0	5.0
Perylene	0.143	5.0	5.0
Indeno(1,2,3-cd)pyrene	0.798	5.0	5.0
Dibenz(a,h)anthracene	0.465	5.0	5.0
Benzo(ghi)perylene	0.305	5.0	5.0

TABLE 3

PAH ANALYSIS BY HRMS OF DIFFERENT LOTS OF CLEANED RESIN

PAH ANALYTES	CONCENTRATION (ng/sample)												
	SAMPLE IDENTIFICATION												
	A1	A2	A3	A4	A5	A6	A7	A8	A9	A10	A11	A12	A13
Naphthalene	480	220	198	120	350	340	320	360	370	380	340	520	220
2-Methylnaphthalene	65	32	38	15.6	32	15.6	32	26	19	45	15	32	48
Acenaphthylene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Acenaphthene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Fluorene	16.5	9.8	13	< 5.0	5.7	5.4	7.4	5.8	5.5	10	5.5	6.8	5.0
Phenanthrene	22	16	32	<12.5*	14	14.8	16	12	14	24	13	<13.0*	14
Anthracene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Fluoranthene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Pyrene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(a)anthracene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Chrysene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(b)fluoranthene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(k)fluoranthene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(e)pyrene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(a)pyrene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Perylene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Indeno(1,2,3-cd)pyrene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Dibenzo(a,h)anthracene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0
Benzo(g,h,i)perylene	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0	< 5.0

* 5 x the concentration of the lowest calibration standard

TABLE 4

COMPOSITION OF THE SAMPLE SPIKING SOLUTIONS

Spiking Solutions	Analytes	Concentration ng/µl LRMS	Concentration pg/µl HRMS
1.	**Surrogate Standards**		
	d_{10}-Fluorene	1.0	250
	d_{14}-Terphenyl	1.0	250
2.	**Internal Standards**		
	d_8-Naphthalene	1.0	100
	d_{10}-2-Methylnaphthalene	1.0	100
	d_8-Acenaphthylene	1.0	100
	d_{10}-Phenanthrene	1.0	100
	d_{10}-Fluoranthene	1.0	100
	d_{12}-Benzo(a)anthracene	1.0	100
	d_{12}-Chrysene	1.0	100
	d_{12}-Benzo(b)fluoranthene	1.0	200
	d_{12}-Benzo(k)fluoranthene	1.0	200
	d_{12}-Benzo(a)pyrene	1.0	200
	d_{12}-Perylene	1.0	200
	d_{12}-Indeno(1,2,3,c-d)pyrene	1.0	200
	d_{14}-Dibenz(a,h)anthracene	1.0	200
	d_{12}-Benzo(ghi)perylene	1.0	200
3.	**Alternate Standard**		
	d_{10}-Anthracene	1.0	100
4.	**Recovery Standards**		
	d_{10}-Acenaphthene	20.0	2000
	d_{10}-Pyrene	20.0	2000
	d_{12}-benzo(e)pyrene	20.0	2000

TABLE 4A

COMPOSITION OF ALTERNATIVE SAMPLE SPIKING SOLUTIONS

Spiking Solutions	Analytes	Concentration pg/μl HRMS
1A.	Surrogate Standards	
	d_{12}-Benzo(e)pyrene	250
	d_{14}-Terphenyl	250
2A.	Internal Standards	
	d_8-Naphthalene	100
	d_8-Acenaphthylene	100
	d_{10}-Acenaphthene	100
	d_{10}-Fluorene	100
	d_{10}-Phenanthrene	100
	d_{10}-Fluoranthene	100
	d_{12}-Benzo(a)anthracene	100
	d_{12}-Chrysene	100
	d_{12}-Benzo(b)fluoranthene	200
	d_{12}-Benzo(k)fluoranthene	200
	d_{12}-Benzo(a)pyrene	200
	d_{12}-Indeno(1,2,3,c-d)pyrene	200
	d_{14}-Dibenz(a,h)anthracene	200
	d_{12}-Benzo(ghi)perylene	200
3A.	Alternate Standard	
	d_{10}-Anthracene	100
4A.	Recovery Standards	
	d_{10}-2-Methylnaphthalene	2000
	d_{10}-Pyrene	2000
	d_{12}-Perylene	2000

TABLE 5

CONCENTRATIONS OF PAHs IN WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR LOW RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (ng/μL)				
	Solutions				
	1	2	3	4	5
Calibration Standards					
Naphthalene	0.25	0.5	1.0	2.5	5.0
2-Methylnaphthalene	0.25	0.5	1.0	2.5	5.0
Acenaphthene	0.25	0.5	1.0	2.5	5.0
Acenaphthylene	0.25	0.5	1.0	2.5	5.0
Fluorene	0.25	0.5	1.0	2.5	5.0
Phenanthrene	0.25	0.5	1.0	2.5	5.0
Anthracene	0.25	0.5	1.0	2.5	5.0
Fluoranthene	0.25	0.5	1.0	2.5	5.0
Pyrene	0.25	0.5	1.0	2.5	5.0
Benzo(a)anthracene	0.25	0.5	1.0	2.5	5.0
Chrysene	0.25	0.5	1.0	2.5	5.0
Benzo(b)fluoranthene	0.25	0.5	1.0	2.5	5.0
Benzo(k)fluoranthene	0.25	0.5	1.0	2.5	5.0
Benzo(e)pyrene	0.25	0.5	1.0	2.5	5.0
Benzo(a)pyrene	0.25	0.5	1.0	2.5	5.0
Perylene	0.25	0.5	1.0	2.5	5.0
Indeno(1,2,3-cd)pyrene	0.25	0.5	1.0	2.5	5.0
Dibenz(a,h)anthracene	0.25	0.5	1.0	2.5	5.0
Benzo(ghi)perylene	0.25	0.5	1.0	2.5	5.0
Internal Standards					
d_8-Naphthalene	1.0	1.0	1.0	1.0	1.0
d_{10}-2-Methylnaphthalene	1.0	1.0	1.0	1.0	1.0
d_8-Acenaphthylene	1.0	1.0	1.0	1.0	1.0
d_{10}-Phenanthrene	1.0	1.0	1.0	1.0	1.0
d_{10}-Fluoranthene	1.0	1.0	1.0	1.0	1.0
d_{12}-Benzo(a)anthracene	1.0	1.0	1.0	1.0	1.0
d_{12}-Chrysene	1.0	1.0	1.0	1.0	1.0
d_{12}-Benzo(b)fluoranthene	1.0	1.0	1.0	1.0	1.0
d_{12}-Benzo(k)fluoranthene	1.0	1.0	1.0	1.0	1.0
d_{12}-Benzo(a)pyrene	1.0	1.0	1.0	1.0	1.0
d_{12}-Perylene	1.0	1.0	1.0	1.0	1.0
d_{12}-Indeno(1,2,3,c-d)pyrene	1.0	1.0	1.0	1.0	1.0
d_{14}-Dibenz(a,h)anthracene	1.0	1.0	1.0	1.0	1.0
d_{12}-Benzo(ghi)perylene	1.0	1.0	1.0	1.0	1.0

TABLE 5 (CONT)

CONCENTRATIONS OF PAHs IN WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR LOW RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (ng/µL)				
	Solutions				
	1	2	3	4	5
Surrogate Standards					
d_{10}-Fluorene	1.0	1.0	1.0	1.0	1.0
d_{14}-Terphenyl	1.0	1.0	1.0	1.0	1.0
Alternate Standard					
d_{10}-Anthracene	1.0	1.0	1.0	1.0	1.0
Recovery Standards					
d_{10}-Acenaphthene	1.0	1.0	1.0	1.0	1.0
d_{10}-Pyrene	1.0	1.0	1.0	1.0	1.0
d_{12}-benzo(e)pyrene	1.0	1.0	1.0	1.0	1.0

TABLE 6

CONCENTRATIONS OF PAHs IN WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR HIGH RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (pg/μL)				
	Solutions				
	1	2	3	4	5
Calibration Standards					
Naphthalene	10	50	100	200	500
2-Methylnaphthalene	10	50	100	200	500
Acenaphthylene	10	50	100	200	500
Acenaphthene	10	50	100	200	500
Fluorene	10	50	100	200	500
Phenanthrene	10	50	100	200	500
Anthracene	10	50	100	200	500
Fluoranthene	10	50	100	200	500
Pyrene	10	50	100	200	500
Benzo(a)anthracene	10	50	100	200	500
Chrysene	10	50	100	200	500
Benzo(b)fluoranthene	10	50	100	200	500
Benzo(k)fluoranthene	10	50	100	200	500
Benzo(e)pyrene	10	50	100	200	500
Benzo(a)pyrene	10	50	100	200	500
Perylene	10	50	100	200	500
Indeno(1,2,3-cd)pyrene	10	50	100	200	500
Dibenz(a,h)anthracene	10	50	100	200	500
Benzo(ghi)perylene	10	50	100	200	500
Internal Standards					
d_8-Naphthalene	100	100	100	100	100
d_8Methylnaphthalene	100	100	100	100	100
d_8-Acenaphthylene	100	100	100	100	100
d_{10}-Phenanthrene	100	100	100	100	100
d_{10}-Fluoranthene	100	100	100	100	100
d_{12}-Benzo(a)anthracene	100	100	100	100	100
d_{12}-Chrysene	100	100	100	100	100
d_{12}-Benzo(b)fluoranthene	200	200	200	200	200
d_{12}-Benzo(k)fluoranthene	200	200	200	200	200
d_{12}-Benzo(a)pyrene	200	200	200	200	200
d_{12}-Perylene	200	200	200	200	200
d_{12}-Indeno(1,2,3,c-d)pyrene	200	200	200	200	200
d_{14}-Dibenz(a,h)anthracene	200	200	200	200	200
d_{12}-Benzo(ghi)perylene	200	200	200	200	200

TABLE 6 (CONT)

CONCENTRATIONS OF PAHs IN WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR HIGH RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (pg/µL)				
	Solutions				
	1	2	3	4	5
Surrogate Standards					
d_{10}-Fluorene	100	100	100	100	100
d_{14}-Terphenyl	100	100	100	100	100
Alternate Standard					
d_{10}-Anthracene	100	100	100	100	100
Recovery Standards					
d_{10}-Acenaphthene	200	200	200	200	200
d_{10}-Pyrene	200	200	200	200	200
d_{12}-benzo(e)pyrene	200	200	200	200	200

TABLE 6A

CONCENTRATIONS OF PAHs IN ALTERNATIVE WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR HIGH RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (pg/μL)				
	Solutions				
	1	2	3	4	5
Calibration Standards					
Naphthalene	10	50	100	200	500
2-Methylnaphthalene	10	50	100	200	500
Acenaphthylene	10	50	100	200	500
Acenaphthene	10	50	100	200	500
Fluorene	10	50	100	200	500
Phenanthrene	10	50	100	200	500
Anthracene	10	50	100	200	500
Fluoranthene	10	50	100	200	500
Pyrene	10	50	100	200	500
Benzo(a)anthracene	10	50	100	200	500
Chrysene	10	50	100	200	500
Benzo(b)fluoranthene	10	50	100	200	500
Benzo(k)fluoranthene	10	50	100	200	500
Benzo(e)pyrene	10	50	100	200	500
Benzo(a)pyrene	10	50	100	200	500
Perylene	10	50	100	200	500
Indeno(1,2,3-cd)pyrene	10	50	100	200	500
Dibenz(a,h)anthracene	10	50	100	200	500
Benzo(ghi)perylene	10	50	100	200	500
Internal Standards					
d_8-Naphthalene	100	100	100	100	100
d_8-Acenaphthylene	100	100	100	100	100
d_{10}-Acenaphthene	100	100	100	100	100
d_{10}-Fluorene	100	100	100	100	100
d_{10}-Phenanthrene	100	100	100	100	100
d_{10}-Fluoranthene	100	100	100	100	100
d_{12}-Benzo(a)anthracene	100	100	100	100	100
d_{12}-Chrysene	100	100	100	100	100
d_{12}-Benzo(b)fluoranthene	200	200	200	200	200
d_{12}-Benzo(k)fluoranthene	200	200	200	200	200
d_{12}-Benzo(a)pyrene	200	200	200	200	200
d_{12}-Indeno(1,2,3,c-d)pyrene	200	200	200	200	200
d_{14}-Dibenz(a,h)anthracene	200	200	200	200	200
d_{12}-Benzo(ghi)perylene	200	200	200	200	200

TABLE 6A (CONT)

CONCENTRATIONS OF PAHs IN ALTERNATIVE WORKING GC/MS CALIBRATION STANDARD SOLUTIONS FOR HIGH RESOLUTION MASS SPECTROMETRY

	CONCENTRATIONS (pg/μL)				
	Solutions				
	1	2	3	4	5
Surrogate Standards					
d_{12}-benzo(e)pyrene	100	100	100	100	100
d_{14}-Terphenyl	100	100	100	100	100
Alternate Standard					
d_{10}-Anthracene	100	100	100	100	100
Recovery Standards					
d_{10}-2-Methylnaphthalene	200	200	200	200	200
d_{10}-Pyrene	200	200	200	200	200
d_{12}-Perylene	200	200	200	200	200

TABLE 7

SPIKE LEVELS FOR LABELLED STANDARDS

Time of Addition	Analyte	LRMS (µg/sample)	HRMS (ng/sample)
Before sampling	**Surrogate Standards**		
	d_{10}-Fluorene	2.0	500
	d_{14}-Terphenyl	2.0	500
Before extraction	**Internal Standards**		
	d_8-Naphthalene	2.0	200
	d_{10}-2-Methylnaphthalene	2.0	200
	d_8-Acenaphthylene	2.0	200
	d_{10}-Phenanthrene	2.0	200
	d_{10}-Fluoranthene	2.0	200
	d_{12}-Benzo(a)anthracene	2.0	200
	d_{12}-Chrysene	2.0	200
	d_{12}-Benzo(b)fluoranthene	2.0	400
	d_{12}-Benzo(d)fluoranthene	2.0	400
	d_{12}-Benzo(a)pyrene	2.0	400
	d_{12}-Perylene	2.0	400
	d_{12}-Indeno(1,2,3,c-d)pyrene	2.0	400
	d_{14}-Dibenz(a,h)anthracene	2.0	400
	d_{12}-Benzo(ghi)perylene	2.0	400
Before extraction	**Alternate Standard**		
	d_{10}-Anthracene	2.0	200
Before GC/MS	**Recovery Standards**		
	d_{10}-Acenaphthene	1.0	100
	d_{10}-Pyrene	1.0	100
	d_{12}-benzo(e)pyrene	1.0	100

TABLE 7A

SPIKE LEVELS FOR LABELLED STANDARDS FOR ALTERNATIVE HRMS SPIKING SCHEME

Time of Addition	Analyte	HRMS (ng/sample)
Before sampling	<u>**Surrogate Standards**</u>	
	d_{12}-benzo(e)pyrene	500
	d_{14}-Terphenyl	500
Before extraction	<u>**Internal Standards**</u>	
	d_8-Naphthalene	200
	d_8-Acenaphthylene	200
	d_{10}-Acenaphthene	200
	d_{10}-Fluorene	200
	d_{10}-Phenanthrene	200
	d_{10}-Fluoranthene	200
	d_{12}-Benzo(a)anthracene	200
	d_{12}-Chrysene	200
	d_{12}-Benzo(b)fluoranthene	400
	d_{12}-Benzo(d)fluoranthene	400
	d_{12}-Benzo(a)pyrene	400
	d_{12}-Indeno(1,2,3,c-d)pyrene	400
	d_{14}-Dibenz(a,h)anthracene	400
	d_{12}-Benzo(ghi)perylene	400
Before extraction	<u>**Alternate Standard**</u>	
	d_{10}-Anthracene	200
Before GC/MS	<u>**Recovery Standards**</u>	
	d_{10}-2-Methylnaphthalene	100
	d_{10}-Pyrene	100
	d_{12}-Perylene	100

TABLE 8

TARGET CONCENTRATIONS FOR LABELLED STANDARDS IN SAMPLE EXTRACT[1]

	ng/µl LRMS	pg/µl HRMS
Surrogate Standards		
d_{10}-Fluorene	2.0	500
d_{14}-Terphenyl	2.0	500
Internal Standards		
d_8-Naphthalene	2.0	200
d_{10}-2-Methylnaphthalene	2.0	200
d_8-Acenaphthylene	2.0	200
d_{10}-Phenanthrene	2.0	200
d_{10}-Fluoranthene	2.0	200
d_{12}-Benzo(a)anthracene	2.0	200
d_{12}-Chrysene	2.0	200
d_{12}-Benzo(b)fluoranthene	2.0	400
d_{12}-Benzo(k)fluoranthene	2.0	400
d_{12}-Benzo(a)pyrene	2.0	400
d_{12}-Perylene	2.0	400
d_{12}-Indeno(1,2,3,c-d)pyrene	2.0	400
d_{14}-Dibenz(a,h)anthracene	2.0	400
d_{12}-Benzo(ghi)perylene	2.0	400
Alternate Standard		
d_{10}-Anthracene	1.0	200
Recovery Standards		
d_{10}-Acenaphthene	1.0	200
d_{10}-Pyrene	1.0	200
d_{12}-benzo(e)pyrene	1.0	200

---

[1] Assuming 100 percent recovery.

TABLE 8A

TARGET CONCENTRATIONS FOR LABELLED STANDARDS IN SAMPLE EXTRACT OBTAINED WITH ALTERNATIVE HRMS SPIKING SCHEME[1]

	pg/μl HRMS
Surrogate Standards	
d_{12}-benzo(e)pyrene	500
d_{14}-Terphenyl	500
Internal Standards	
d_8-Naphthalene	200
d_8-Acenaphthylene	200
d_{10}-Acenaphthene	200
d_{10}-Fluorene	200
d_{10}-Phenanthrene	200
d_{10}-Fluoranthene	200
d_{12}-Benzo(a)anthracene	200
d_{12}-Chrysene	200
d_{12}-Benzo(b)fluoranthene	400
d_{12}-Benzo(k)fluoranthene	400
d_{12}-Benzo(a)pyrene	400
d_{12}-Indeno(1,2,3,c-d)pyrene	400
d_{14}-Dibenz(a,h)anthracene	400
d_{12}-Benzo(ghi)perylene	400
Alternate Standard	
d_{10}-Anthracene	200
Recovery Standards	
d_{10}-2-Methylnaphthalene	200
d_{10}-Pyrene	200
d_{12}-Perylene	200

---

[1] Assuming 100 percent recovery.

TABLE 9

CONCENTRATIONS OF COMPOUNDS IN LABORATORY CONTROL SPIKE SAMPLE

	ng/sample	
	LRMS	HRMS
Unlabelled Compounds		
Naphthalene	2.0	1000
2-Methylnaphthalene	2.0	200
Acenaphthylene	2.0	200
Acenaphthene	2.0	200
Fluorene	2.0	200
Phenanthrene	2.0	500
Anthracene	2.0	200
Fluoranthene	2.0	200
Pyrene	2.0	200
Benzo(a)anthracene	2.0	200
Chrysene	2.0	200
Benzo(b)fluoranthene	2.0	200
Benzo(k)fluoranthene	2.0	200
Benzo(e)pyrene	2.0	200
Benzo(a)pyrene	2.0	200
Perylene	2.0	200
Indeno(1,2,3,c-d)pyrene	2.0	200
Dibenz(a,h)anthracene	2.0	200
Benzo(ghi)perylene	2.0	200
Alternate Standard		
d_{10}-Anthracene	2.0	200

TABLE 10

RECOMMENDED GAS CHROMATOGRAPHIC OPERATING CONDITIONS FOR PAH ANALYSIS

Column Type	DB-5
Length (m)	30
ID (mm)	0.25
Film Thickness (μm)	0.32
Helium Linear Velocity (cm/sec)	30
Injection mode	Splitless
Splitless Time (sec)	30
Initial Temperature (°C)	45
Initial Time (min)	4
Program Rate (°C/min)	8
Final Temperature (°C)	300
Final Hold Time	until benzo(ghi) perylene has eluted
Injector Temperature (°C)	320

TABLE 11

ASSIGNMENTS OF INTERNAL STANDARDS FOR CALCULATION OF RRFs AND QUANTITATION OF TARGET PAHs AND SURROGATE STANDARDS

Analyte	Internal Standards
Unlabeled PAH	
Naphthalene	d_8-Naphthalene
2-Methylnaphthalene	d_{10}-2-Methylnaphthalene
Acenaphthylene	d_8-Acenaphthylene
Acenaphthene	d_8-Acenaphthylene
Fluorene	d_{10}-Phenanthrene
Phenanthrene	d_{10}-Phenanthrene
Anthracene	d_{10}-Phenanthrene
Fluoranthene	d_{10}-Fluoranthene
Pyrene	d_{10}-Fluoranthene
Benzo(a)anthracene	d_{12}-Benzo(a)anthracene
Chrysene	d_{12}-Chrysene
Benzo(b)fluoranthene	d_{12}-Benzo(b)fluoranthene
Benzo(k)fluoranthene	d_{12}-Benzo(k)fluoranthene
Benzo(e)pyrene	d_{12}-Benzo(a)pyrene
Benzo(a)pyrene	d_{12}-Benzo(a)pyrene
Perylene	d_{12}-Perylene
Indeno(1,2,3-cd)pyrene	d_{12}-Indeno(1,2,3,c-d)pyrene
Dibenz(a,h)anthracene	d_{14}-Dibenz(a,h)anthracene
Benzo(ghi)perylene	d_{12}-Benzo(ghi)perylene
Surrogate Standards	
d_{10}-Fluorene	d_{10}-Phenanthrene
d_{14}-Terphenyl	d_{10}-Fluoranthene

TABLE 11A

ASSIGNMENTS OF INTERNAL STANDARDS FOR CALCULATION OF RRFs AND QUANTITATION OF TARGET PAHs AND SURROGATE STANDARDS USING ALTERNATIVE HRMS SPIKING SCHEME

Analyte	Internal Standards
Unlabeled PAH	
Naphthalene	d_8-Naphthalene
2-Methylnaphthalene	d_{10}-Acenaphthene
Acenaphthylene	d_8-Acenaphthylene
Acenaphthene	d_{10}-Acenaphthene
Fluorene	d_{10}-Fluorene
Phenanthrene	d_{10}-Phenanthrene
Anthracene	d_{10}-Phenanthrene
Fluoranthene	d_{10}-Fluoranthene
Pyrene	d_{10}-Fluoranthene
Benzo(a)anthracene	d_{12}-Benzo(a)anthracene
Chrysene	d_{12}-Chrysene
Benzo(b)fluoranthene	d_{12}-Benzo(b)fluoranthene
Benzo(k)fluoranthene	d_{12}-Benzo(k)fluoranthene
Benzo(e)pyrene	d_{12}-Benzo(a)pyrene
Benzo(a)pyrene	d_{12}-Benzo(a)pyrene
Perylene	d_{12}-Benzo(a)pyrene
Indeno(1,2,3-cd)pyrene	d_{12}-Indeno(1,2,3,c-d)pyrene
Dibenz(a,h)anthracene	d_{14}-Dibenz(a,h)anthracene
Benzo(ghi)perylene	d_{12}-Benzo(ghi)perylene
Surrogate Standards	
d_{14}-Terphenyl	d_{10}-Fluoranthene
d_{12}-Benzo(e)pyrene	d_{12}-Benzo(a)pyrene

TABLE 12

ASSIGNMENTS OF RECOVERY STANDARDS FOR DETERMINATION OF PERCENT RECOVERIES OF INTERNAL STANDARDS AND THE ALTERNATE STANDARD

Analyte	Recovery Standard
Internal Standards	
d_8-Naphthalene	d_{10}-Acenaphthene
d_{10} -2-Methylnaphthalene	d_{10}-Acenaphthene
d_8-Acenaphthylene	d_{10}-Acenaphthene
d_{10}-Phenanthrene	d_{10}-Pyrene
d_{10}-Fluoranthene	d_{10}-Pyrene
d_{12}-Benzo(a)anthracene	d_{10}-Pyrene
d_{12}-Chrysene	d_{10}-Pyrene
d_{12}-Benzo(b)fluoranthene	d_{12}-Benzo(e)pyrene
d_{12}-Benzo(k)fluoranthene	d_{12}-Benzo(e)pyrene
d_{12}-Benzo(a)pyrene	d_{12}-Benzo(e)pyrene
d_{12}-Perylene	d_{12}-Benzo(e)pyrene
d_{12}-Indeno(1,2,3,c-d)pyrene	d_{12}-Benzo(e)pyrene
d_{14}-Dibenz(a,h)anthracene	d_{12}-Benzo(e)pyrene
d_{12}-Benzo(ghi)perylene	d_{12}-Benzo(e)pyrene
Alternate Standard	
d_{10}-Anthracene	d_{10}-Pyrene

TABLE 12A

ASSIGNMENTS OF RECOVERY STANDARDS FOR DETERMINATION OF PERCENT RECOVERIES OF INTERNAL STANDARDS AND THE ALTERNATE STANDARD USING ALTERNATIVE HRMS SPIKING SCHEME

Analyte	Recovery Standard
Internal Standards	
d_8-Naphthalene	d_{10}-2-Methylnaphthalene
d_{10} -2-Methylnaphthalene	d_{10}-2-Methylnaphthalene
d_8-Acenaphthylene	d_{10}-2-Methylnaphthalene
d_{10}-Phenanthrene	d_{10}-Pyrene
d_{10}-Fluoranthene	d_{10}-Pyrene
d_{12}-Benzo(a)anthracene	d_{10}-Pyrene
d_{12}-Chrysene	d_{10}-Pyrene
d_{12}-Benzo(b)fluoranthene	d_{12}-Perylene
d_{12}-Benzo(k)fluoranthene	d_{12}-Perylene
d_{12}-Benzo(a)pyrene	d_{12}-Perylene
d_{12}-Perylene	d_{12}-Perylene
d_{12}-Indeno(1,2,3,c-d)pyrene	d_{12}-Perylene
d_{14}-Dibenz(a,h)anthracene	d_{12}-Perylene
d_{12}-Benzo(ghi)perylene	d_{12}-Perylene
Alternate Standard	
d_{10}-Anthracene	d_{10}-Pyrene

TABLE 13

QUANTITATION AND CONFIRMATION IONS FOR SELECTED ION MONITORING OF PAHs BY HRGC/LRMS

Analyte	Quant. Ion	Confirm. Ion	%Relative Abundance of Confirm. Ion
Naphthalene	128	127	10
d_8-Naphthalene	136	68	80
2-Methylnaphthalene	142	141	80
d_{10}-2-Methylnaphthalene	152		
Acenaphthylene	152	153	15
d_8-Acenaphthylene	160		
Acenaphthene	154	153	86
d_{10}-Acenaphthene	164		
Fluorene	166	165	80
d_{10}-Fluorene	176		
Phenanthrene	178	176	15
d_{10}-Phenanthrene	188	94	
Anthracene	178	176	15
d_{10}-Anthracene	188	94	
Fluoranthene	202	101	15
d_{10}-Fluoranthene	212	106	
Pyrene	202	101	15
d_{10}-Pyrene	212	106	
Benzo(a)anthracene	228	114	15
d_{12}-Benzo(a)anthracene	240	120	
Chrysene	228	114	15
d_{12}-Chrysene	240	120	
d_{14}-Terphenyl	244	122	15

TABLE 13 (CONT)

QUANTITATION AND CONFIRMATION IONS FOR SELECTED ION MONITORING OF PAHs BY HRGC/LRMS

Analyte	Quant. Ion	Confirm. Ion	%Relative Abundance of Confirm. Ion
Benzo(b)fluoranthene	252	126	25
d_{12}-Benzo(b)fluoranthene	264	132	
Benzo(k)fluoranthene	252	126	25
d_{12}-Benzo(k)fluoranthene	264	132	
Benzo(e)pyrene	252	126	25
d_{12}-Benzo(e)pyrene	264	132	
Benzo(a)pyrene	252	126	25
d_{12}-Benzo(a)pyrene	264	132	
Perylene	252	126	26
d_{12}-Perylene	264	132	
Indeno(1,2,3-cd)pyrene	276	138	28
d_{12}-Indeno(1,2,3-cd)pyrene	288		
Dibenz(ah)anthracene	278	139	24
d_{14}-Dibenz(ah)anthracene	292		
Benzo(ghi)perylene	276	138	37
d_{12}-Benzo(ghi)perylene	288		

TABLE 14

MASS DESCRIPTORS USED FOR SELECTED ION MONITORING FOR HRGC/HRMS

Descriptor No.	Analyte	Ion Type	Accurate m/z
1	Naphthalene	M	128.0626
	PFK	LOCK	130.9920
	d_8-Naphthalene	IS	136.1128
	2-Methylnaphthalene	M	142.0782
	d_{10}-2-Methylnaphthalene	IS	152.1410
	Acenaphthylene	M	152.0626
	d_8-Acenaphthylene	IS	160.1128
	Acenaphthene	M	154.0782
	d_{10}-Acenaphthene	RS	164.1410
	PFK	QC	169.9888
2	Fluorene	M	166.0782
	d_{10}-Fluorene	SS	176.1410
	Phenanthrene	M	178.0782
	d_{10}-Phenanthrene	IS	188.1410
	Anthracene	M	178.0782
	d_{10}-Anthracene	AS	188.1410
	Fluoranthene	M	202.0782
	d_{10}-Fluoranthene	IS	212.1410
	Pyrene	M	202.0782
	PFK	QC	204.9888
	d_{10}-Pyrene	RS	212.1410
	Benzo(a)anthracene	M	228.0939
	d_{12}-Benzo-a-Anthracene	IS	240.1692
	Chrysene	M	228.0939
	d_{12}-Chrysene	IS	240.1692
	PFK	LOCK	230.9856
	d_{14}-Terphenyl	SS	244.1974

IS = Internal Standard
SS = Surrogate Standard
AS = Alternate Standard
RS = Recovery Standard
LOCK = Lock-Mass Ion
QC = Quality Control Check Ion

TABLE 14 (CONT)

MASS DESCRIPTORS USED FOR SELECTED ION MONITORING FOR HRGC/HRMS

Descriptor No.	Analyte	Ion Type	Accurate m/z
3	Perylene	M	252.0939
	d_{12}-Perylene	IS	264.1692
	PFK	QC	268.9824
	Benzo(b)fluoranthene	M	252.0939
	d_{12}-Benzo(b)fluoranthene	IS	264.1692
	Benzo(k)fluoranthene	M	252.0939
	d_{12}-Benzo-k-fluoranthene	IS	264.1692
	Benzo(e)pyrene	M	252.0939
	d_{12}-Benzo(e)pyrene	RS	264.1692
	Benzo(a)pyrene	M	252.0939
	d_{12}-Benzo(a)pyrene	IS	264.1692
	Benzo(ghi)perylene	M	276.0939
	d_{12}-Benzo(ghi)perylene	IS	288.1692
	Indeno(1,2,3-cd)pyrene	M	276.0939
	d_{12}-Indeno(1,2,3-cd)pyrene	IS	288.1692
	Dibenzo(ah)anthracene	M	278.1096
	PFK	LOCK	280.9824
	d_{14}-Dibenzo(ah)anthracene	IS	292.1974

The following nuclidic masses were used:

H = 1.007825 ^{2}H = 2.014102 C = 12.000000

IS = Internal Standard
SS = Surrogate Standard
AS = Alternate Standard
RS = Recovery Standard
LOCK = Lock-Mass Ion
QC = Quality Control Check Ion

FIGURE 1

METHOD 429 FLOWCHART

1

§1.3.9 *The end user is identified*
§1.3.10 *The tester is designated*

2

The end user chooses:
§2.1.1 • source target concentration

3

§2.1.2 *The tester selects analyst with documented*
§8.4 *experience in satisfactory performance of analytical*
§8.4.1 *procedures*

4

Tester and laboratory coordinate:
§4.3.2 • pre-test cleaning of glassware
§4.2 • pre-test cleaning, contamination checks, and
§4.3.3 storage of sampling materials and reagents
§4.3.4 • preparation of filter, sorbent cartridges, method blanks, and LCS

5

Tester requests pre-test analytical results from laboratory:
§10.1.1 • contamination check of filters
§10.1.2 • contamination check of XAD-2 resin
§10.1.3 • Method detection limits (MDLs) and Practical quantitation limits (PQLs)

6

Tester calculates and plans:
§2.5 • ≥3 sampling runs and ≥1 blank sampling train
• sample volume
• sampling time
• source reporting limit
• chain of custody

7

Tester performs:
§4.3.1 • calibration of equipment

8

Tester writes:
§2.2 • pre-test protocol

9

Tester performs:
§4.4.1 • preliminary field sampling determinations
§4.4.2 • sampling train preparation
§4.4.3 • leak checks
§4.4.4 • sampling procedure
  • ≥3 sampling runs
  • ≥1 blank sampling train
§5 • recovery of all runs and blank sampling train

10

Tester delivers:
§5.3 • recovered sampling runs and blank train(s)
§5.4 • chain of custody record

11

Laboratory performs:
§6 • extraction of field samples
§7 • analyses
§8 • QA/QC procedures
§9 • chain of custody
§10.2 • reporting requirements

12

Tester performs:
§4.3.1 • post-test calibrations
§9.2 • calculations
§10.3 • data recording and chain of custody
• reporting requirements

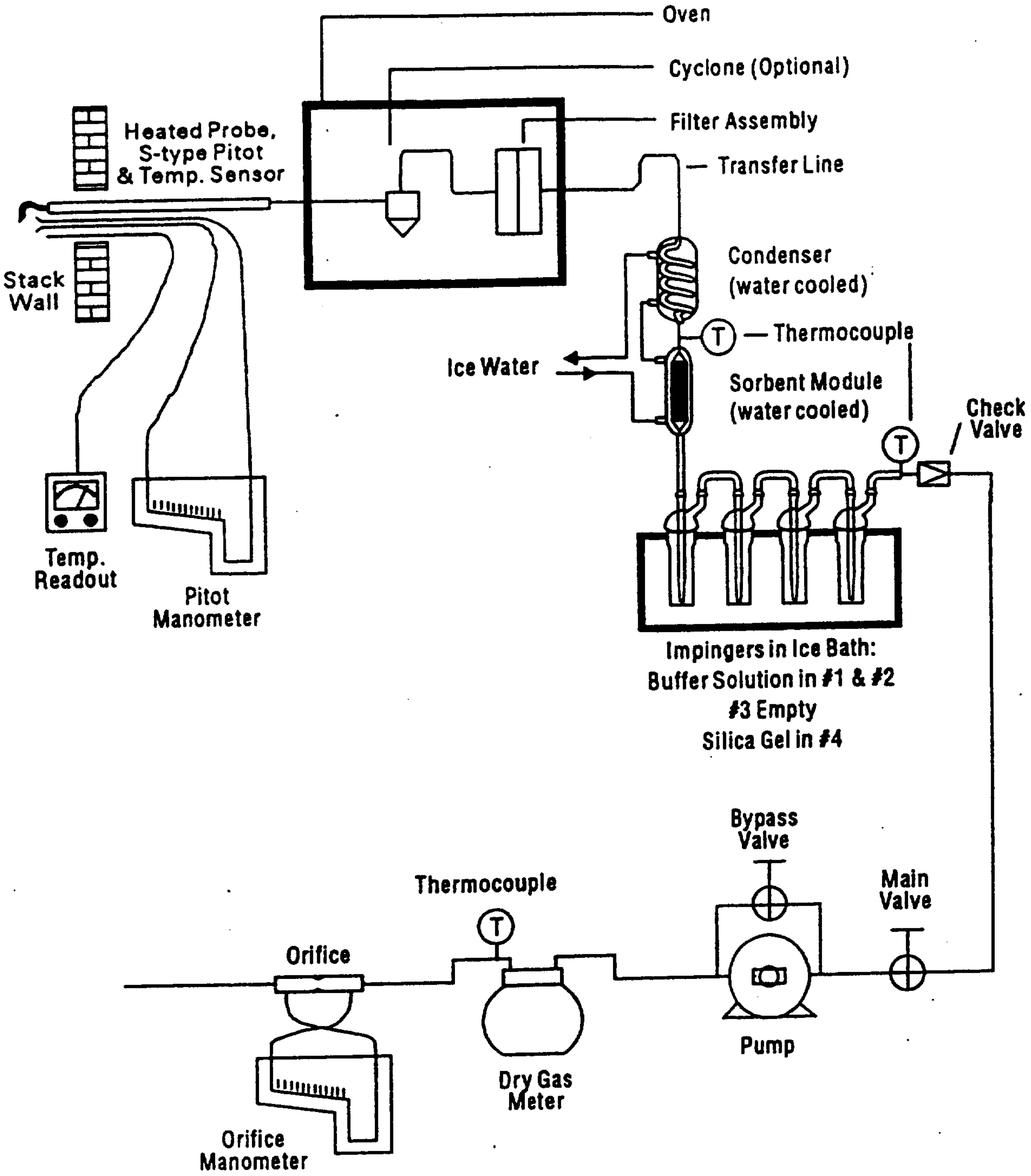


Figure 2

PAH Sampling Train

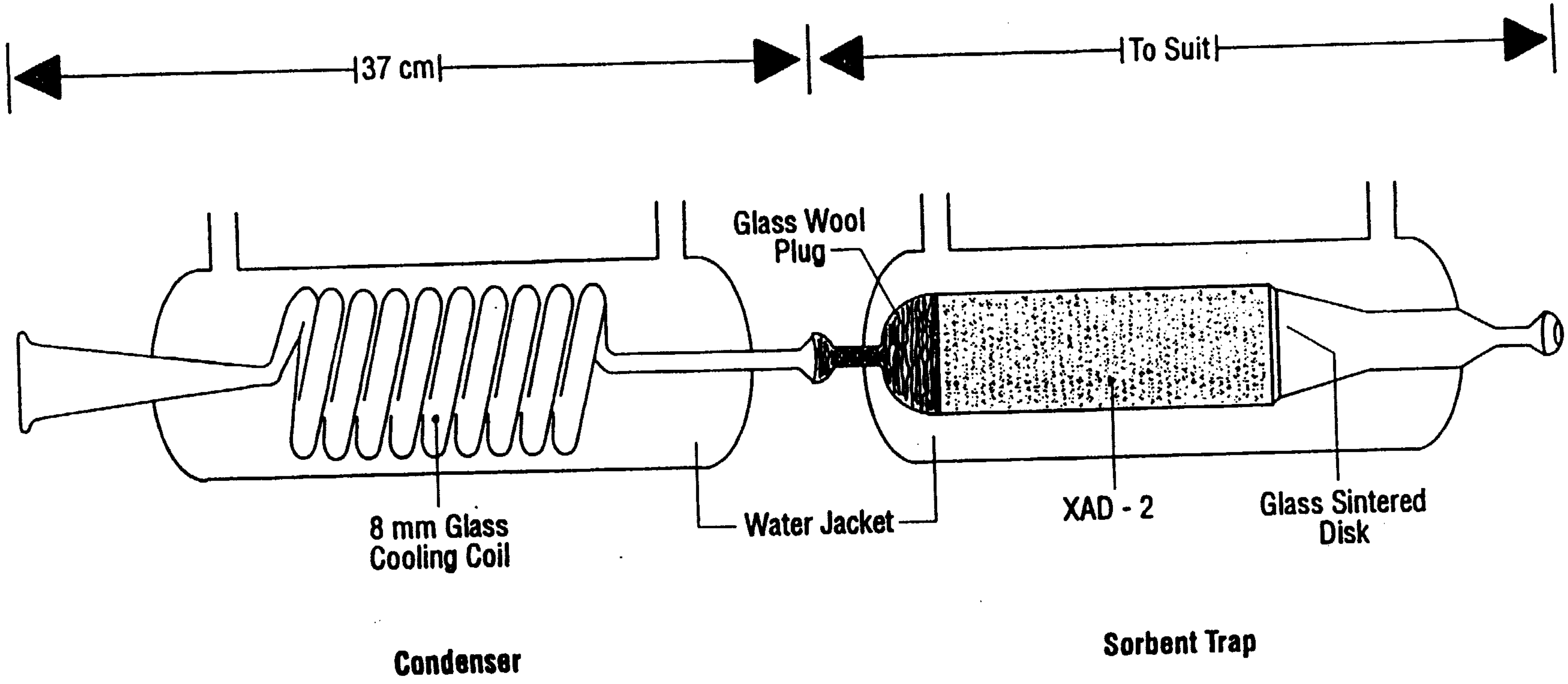


Figure 3

Condenser and Sorbent Trap for Collection of Gaseous PAHs

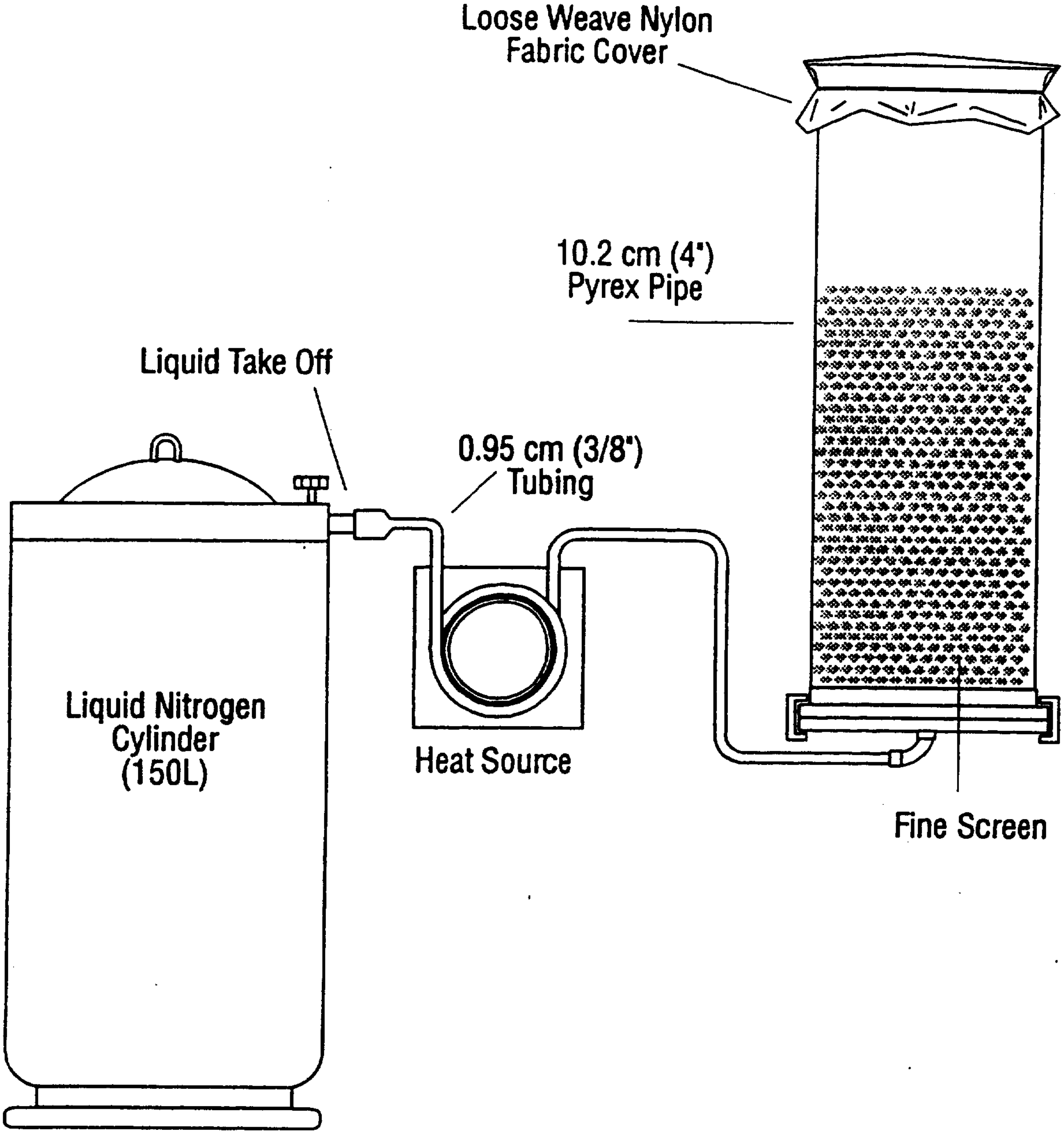


Figure 4

XAD-2 Fluidized Bed Drying Apparatus

FIGURE 5

METHOD 429 FIELD DATA RECORD

Run No. ______
Location ______
Date ______
Operator ______
Meter Box No. ______
Local Time
  Start/Stop ______
ΔH@ ______
Stack Diameter ______
Meter Box Calibration
Factor (Y) ______

Pitot Tube Factor ______
Probe Tip Dia, in. ______
Probe Length ______
Sampling Train Leak Test   Leak Rate
Before ______ in. Hg ______ cu.ft/min
After ______ in. Hg ______ cu.ft/min
Leak Check Volume ______ cu. ft.
Pitot Tube Leak Check
Before ______ After ______

Project No. ______
Plant Name ______
Ambient Temp °F ______
Meter Temp °F ______
Bar. Press, "Hg ______
Stack Press, "H_2O ______
Assumed Moisture, % ______
Heater Box Setting, °F ______
Probe Heater Setting, °F ______
Assumed M.W. (wet%) ______
Assumed M.W. (dry%) ______

Sampling Point	Clock Time	Dry Gas Meter, cu, ft.	Pitot ΔP in. H_2O	Orifice ΔH "H_2O		Temperature (°F)			Pump Vacuum in. Hg
				Desired	Actual	Impinger	Filter box	Stack	
Start									

Figure 6
Recovery of PAH Sampling Train

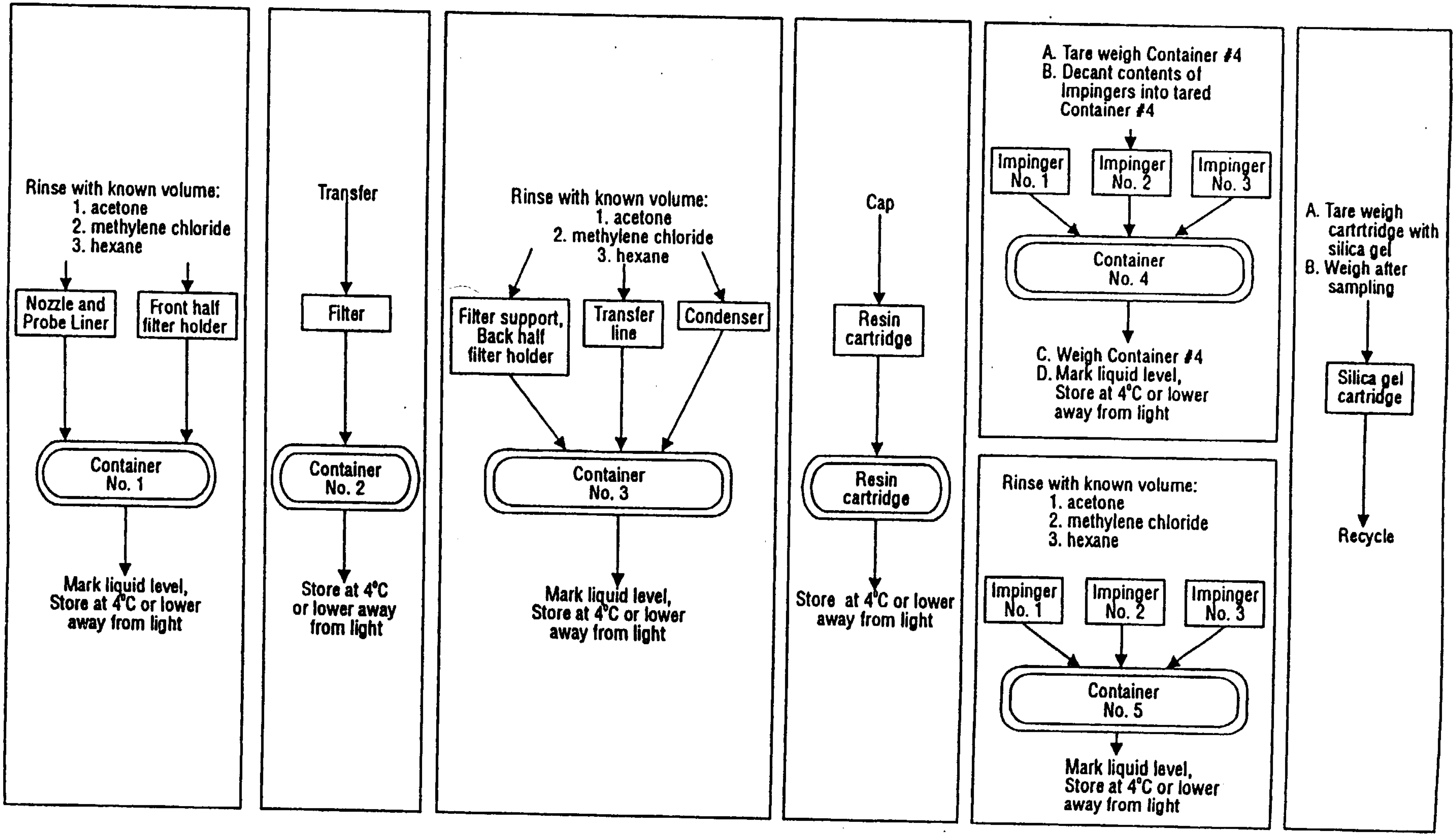
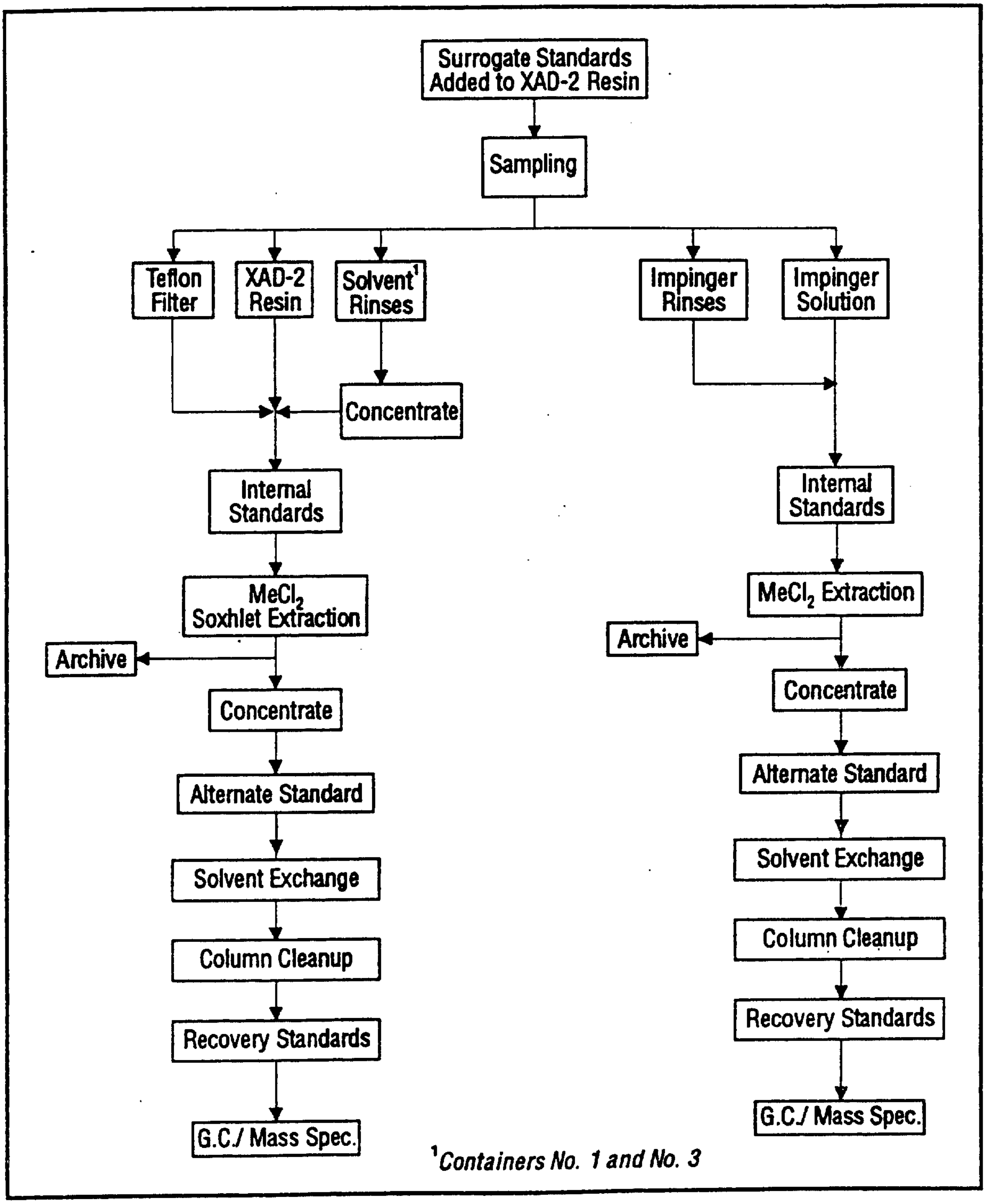


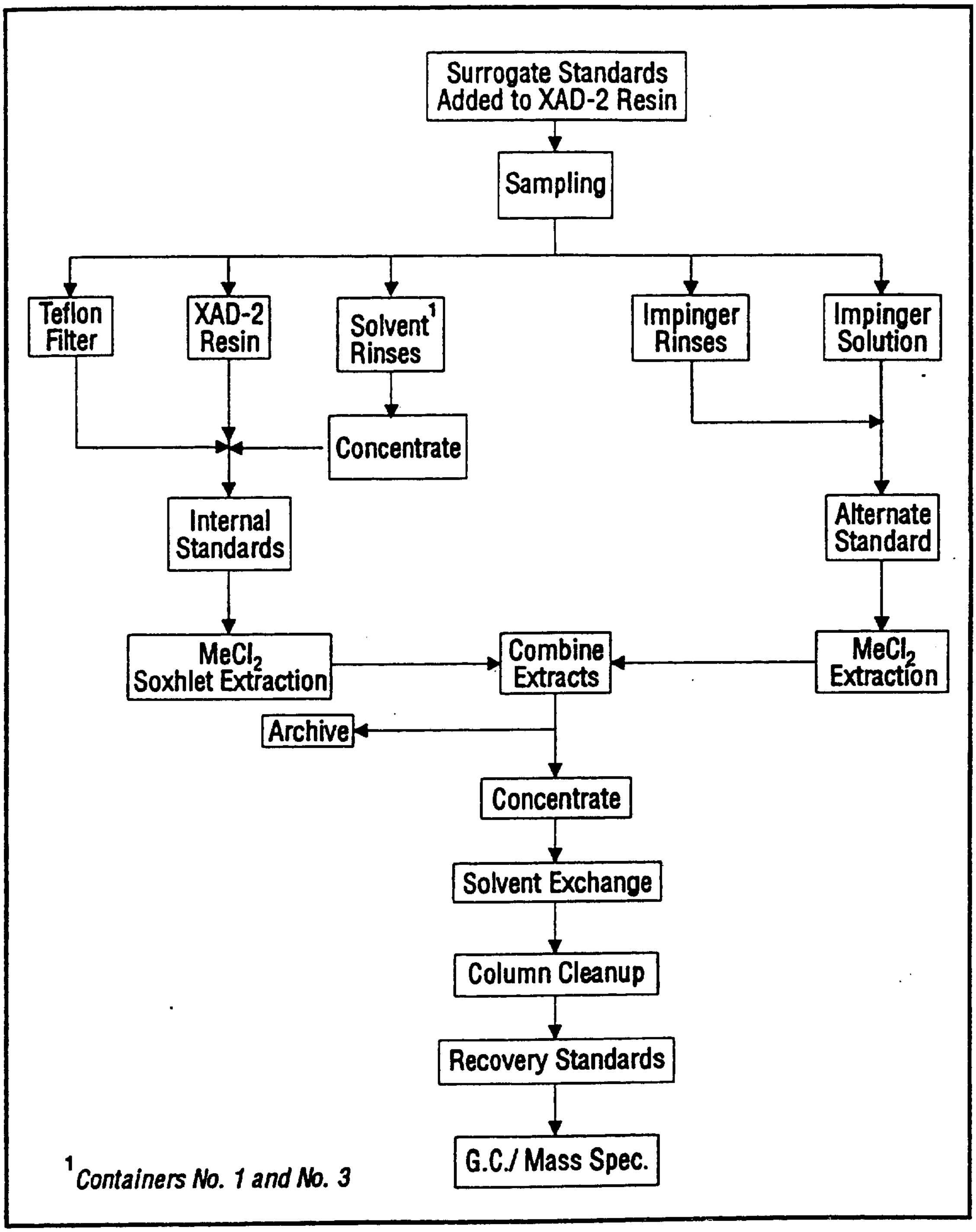
Figure 7

Flow Chart for Sampling, Extraction and Cleanup for Determination of PAH in a Split Sample



[1] *Containers No. 1 and No. 3*

Figure 8
Flow Chart for Sampling, Extraction and Cleanup for Determination of PAH in a Composite Sample



[1] *Containers No. 1 and No. 3*

FIGURE 9

EXAMPLE OF PRE-TEST CALCULATIONS FOR PAH EMISSIONS TEST

					PST = 6 hours PSV = 180 dscf	
	PQL (ng/sample)	STC (ng/dscm)	MSV (dscf)	MST (hours)	F	SRL (ng/dscm)
Naphthalene	2400	<1500	>56.5	>1.89	NA	471
2-Methylnaphthalene	330	NA	NA	NA	NA	64.7
Acenaphthylene	5.0	180	0.98	0.03	183	0.98
Acenaphthene	5.0	6	29.4	0.98	6	0.98
Fluorene[1]	83	<6	>489	>16.3	NA	16.3
Phenanthrene	110	120	32.4	1.08	6	21.6
Anthracene	5.0	<6	>29.4	>0.98	NA	0.98
Fluoranthene	5.0	46	3.8	0.13	47	0.98
Pyrene	5.0	46	3.8	0.13	47	0.98
Benzo(a)anthracene	5.0	<6	>29.4	>0.98	NA	0.98
Chrysene	5.0	42	4.2	0.14	43	0.98
Benzo(b)fluoranthene	5.0	50	3.5	0.12	51	0.98
Benzo(k)fluoranthene	5.0	50	3.5	0.12	51	0.98
Benzo(e)pyrene	5.0	NA	NA	NA	NA	0.98
Benzo(a)pyrene	5.0	<6	>29.4	>0.98	NA	0.98
Perylene	5.0	NA	NA	NA	NA	0.98
Indeno(1,2,3-c,d)pyrene	5.0	<6	>29.4	>0.98	NA	0.98
Dibenzo(a,h)anthracene	5.0	<6	>29.4	>0.98	NA	0.98
Benzo(g,h,i)perylene	5.0	<6	>29.4	>0.98	NA	0.98
Average Volumetric Sampling Rate (VSR) = 0.5 dscfm = 30 dscf/hr						

PQL = Practical quantitation limit for analyte (based on pre-test analysis of XAD-2 resin)

STC = Source target concentration for analyte. (From previous emissions test. Samples were analyzed by HRGC/LRMS).

MSV = Minimum sample volume required to collect detectable levels of target analyte. (MSV = PQL ÷ STC) *Equation 429-1*

MST = Minimum sample time required to collect detectable levels of target analyte at VSR. (MST = MSV ÷ VSR) *Equation 429-2*

PST = Planned sampling time (6 hours chosen as the longest practical sampling time for the planned emissions test)

PSV = Planned sample volume (PSV = PST × VSR) *Equation 429-4*

F = Safety factor (>1) that allows for deviation from ideal sampling and analytical conditions. (F = PSV ÷ MSV) *Equation 429-5*

SRL = Source reporting limit if the target analyte cannot be detected with the planned test parameters. (SRL = PQL ÷ PSV) *Equation 429-7*

NA This calculation is not applicable either because there is no STC value available or the STC is a detection limit.

[1] PSV is lower than the MSV. Therefore, the analyte is not expected to be detected if it is present at the target concentrations. It will only be detected if the actual concentration is lower than the indicated SRL.

FIGURE 10

CARB METHOD 429 (PAHs) SAMPLING TRAIN SET-UP RECORD

RUN NO. ____________________
PLANT NAME ____________________
SET-UP DATE ____________________
RECEIVED BY ____________________

PROJECT NO. ____________________
PLANT LOCATION ____________________
SET-UP BY ____________________
DATE/TIME ____________________

	COMPONENTS	COMPONENT ID	OTHER INFORMATION	
1.	NOZZLE	____________	Material	____________
			Diameter	____________
2.	PROBE	____________	Liner material	____________
			Length	____________
3.	FILTER HOLDER	____________	Before set-up, all openings sealed with	____________
			Filter support type	____________
4.	FILTER	Lot # ____________	Filter Type	____________
			Size	____________
			Contamination check?	____________
5.	TRANSFER LINE AND CONDENSER	____________	Transfer line material	____________
	Fittings	____________		____________
6.	XAD-2 RESIN CARTRIDGE	____________	Both ends sealed in lab prior to set-up	____________
			Fittings	____________
			Contamination check?	____________
			Spiked?	____________
7.	IMPINGERS: No. 1	____________	Charge with 100 mL impinger solution and weigh	____________ g
	U-Connector	____________		
	No. 2	____________	Charge with 100 mL impinger solution and weigh	____________ g
	U-Connector	____________		
	No. 3	____________		
	U-Connector	____________	Weigh empty	____________ g
8.	SILCA GEL CARTRIDGE	____________	Tare weight	____________ g

Appearance ________________________

FIGURE 11

CARB METHOD 429 (PAHs) SAMPLING TRAIN RECOVERY RECORD

RUN NO. ____________________
PLANT NAME ____________________
RECOVERY DATE ____________________
PROJECT NO. ____________________
PLANT LOCATION ____________________
RECOVERED BY ____________________

1. **CHECK whether openings were covered.** **RINSE 3x each with Acetone, $MeCl_2$, Hexane.**
MARK liquid level and STORE containers at temp. <4°C away from light.

Component	Openings covered?	Rinse volume (mL)			Storage Container(s) IDs
		Acetone	$MeCl_2$	Hexane	
Nozzle					
Probe liner					
Filter holder front					

2. **STORE filter(s) at temp. <4°C away from light.** **RECORD ALL sample storage information.**

Component	Appearance after sampling	Storage (Temperature & light)	Storage Container(s) ID
Filter			
Filter			
Filter			

3. **CHECK whether openings were covered.** **RINSE 3x each with Acetone, $MeCl_2$, Hexane.**
MARK liquid level and STORE containers at temp. <4°C away from light.

Component	Openings covered?	Rinse volume (mL)			Storage Temp. & light	Storage Container ID
		Acetone	$MeCl_2$	Hexane		
Filter support and filter holder back						
Transfer line						
Condenser						

4. **STORE Resin cartridges at temp. <4°C away from light.** **RECORD ALL storage information.**

ID	Appearance after sampling	Storage temperature & light conditions

5. **WEIGH impinger contents and silica gel cartridge.**
MARK liquid level and STORE impinger contents at temp. <4°C away from light.

Weight	No. 1	No. 2	No. 3	Additional impingers No. 4	Additional impingers No. 5	Silica gel cartridge
Final (g)						
Before sampling (g)						
Gain (g)	(A)	(B)	(C)	(D)	(E)	(F)

Total condensate (A) + (B) + (C) + (D) + (E) + (F) ____________________ (g)

STORAGE CONTAINER ID(s) ________ ________ ________ ________

6. **RINSE impingers 3x each with Acetone, $MeCl_2$, Hexane.**
MARK liquid level and STORE impinger rinses at temp. <4°C away from light.

Rinse volumes (mL)	Acetone					
	$MeCl_2$					
	Hexane					

STORAGE CONTAINER ID(s) ________ ________ ________ ________ ________

FIGURE 12

CHAIN OF CUSTODY SAMPLE RECORD

Project # ____________ Date: ____________

Start: ____________

Stop: ____________

Source name: ____________

Sampling location: ____________

Chain of Custody Log Record # (s) ____________

Sample/Run # : ____________

Sample type: ____________

Operator: ____________

SAMPLE STORAGE INFORMATION

SAMPLE PRESERVATION	Comments
Ice/Dry ice?	

CHAIN OF CUSTODY

ACTION	DATE	TIME	GIVEN BY	TAKEN BY

RELATED IDs	DESCRIPTION/COMMENTS	Log #s
FR	Front rinse (nozzle, probe,filter holder front)	
F	Filter in sealed storage container	
BR	Back rinse (filter support, filter holder, sample line & condenser	
C	Resin cartridge	
I	Impinger contents	
IR	Impinger rinses	

FIGURE 13

CHAIN OF CUSTODY LOG RECORD

PROJECT NO. ________________ **Page** _____ **of** _______

Log #	Sample ID	Date	Time	Comments	Given by	Taken by

Sample Identifier	**Sample Description**
FR	Rinses of probe and front half of filter holder
F	Filter in sealed storage container
BR	Rinses of filter support, back half of filter holder, sample transfer line and condenser
C	Aluminum foil wrapped, capped resin cartridge
I	Impinger contents
IR	Impinger rinses

FIGURE 14A

EXAMPLE GC/MS SUMMARY REPORT (HRMS) FOR INITIAL CALIBRATION SOLUTION #1 CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

ICAL ID: ST1120A1
RUN #: PAHCS1

ACQUIRED: 12/3/94 16:23:24
PROCESSED: 12/3/94

INSTRUMENT: W
OPERATOR: MPA

	RT	RRT	Area	RRF
Naphthalene	8:20	1.006	6.66 E+07	0.75
2-Methylnaphthalene	9:42	1.007	1.44 E+07	1.30
Acenaphthylene	11:04	1.003	1.57 E+07	1.44
Acenaphthene	11:20	1.004	1.05 E+07	0.94
Fluorene	12:06	1.003	8.15 E+06	1.05
Phenanthrene	13:20	1.003	1.99 E+07	1.15
Anthracene	13:23	1.001	7.07 E+06	1.02
Fluoranthene	14:38	1.001	3.18 E+07	1.26
Pyrene	14:55	1.001	3.31 E+07	1.31
Benzo(a)anthracene	16:34	1.002	2.08 E+07	1.13
Chrysene	16:39	1.003	2.26 E+07	1.13
Benzo(b)fluoranthene	18:54	1.004	2.35 E+07	1.69
Benzo(k)fluoranthene	18:58	1.004	2.50 E+07	1.24
Benzo(e)pyrene	19:42	1.004	2.41 E+07	1.20
Benzo(a)pyrene	19:51	1.003	2.11 E+07	1.07
Perylene	20:06	1.004	1.38 E+07	0.70
Indeno(1,2,3-c,d)pyrene	23:60	1.006	2.07 E+07	2.19
Dibenzo(a,h)anthracene	24:01	1.006	1.49 E+07	1.66
Benzo(g,h,i)perylene	25:15	1.005	1.84 E+07	2.23
d_8-Naphthalene	8:17	1.000	3.54 E+08	4.22
d_8-Acenaphthylene	11:02	1.000	1.09 E+08	1.29
d_{10}-Acenaphthene	11:17	1.000	1.11 E+08	1.32
d_{10}-Fluorene	12:04	1.000	7.78 E+07	0.93
d_{10}-Phenanthrene	13:18	1.000	6.92 E+07	0.82
d_{10}-Fluoranthene	14:37	1.000	2.53 E+08	1.03
d_{12}-Benzo(a)anthracene	16:32	1.000	1.83 E+08	0.75
d_{12}-Chrysene	16:36	1.000	2.00 E+08	0.82
d_{12}-Benzo(b)fluoranthene	18:50	1.000	2.77 E+08	1.35
d_{12}-Benzo(k)fluoranthene	18:54	1.000	4.03 E+08	1.95
d_{12}-Benzo(a)pyrene	19:47	1.000	3.93 E+08	1.91
d_{12}-Indeno(1,2,3-c,d)pyrene	23:52	1.000	1.89 E+08	0.92
d_{14}-Dibenzo(a,h)anthracene	23:52	1.000	1.80 E+08	0.87
d_{12}-Benzo(g,h,i)perylene	25:07	1.000	1.65 E+08	0.80
d_{14}-Terphenyl	14:59		2.65 E+08	0.52
d_{12}-Benzo(e)pyrene	19:37	1.000	1.44 E+08	0.37
d_{10}-Anthracene	13:22	1.000	5.82 E+07	0.69
d_{10}-2-Methylnaphthalene	9:38	1.000	8.40 E+07	---
d_{10}-Pyrene	14:54	1.000	2.45 E+08	---
d_{12}-Perylene	20:01	1.000	1.03 E+08	---

FIGURE 14B

EXAMPLE OF INITIAL CALIBRATION (ICAL) RRF SUMMARY
CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

ICAL ID: ST1120
RUN #: NA

ACQUIRED: 3-DEC-94
PROCESSED: 3-DEC-94

INSTRUMENT: W
OPERATOR: MPA

	RRF #1	RRF #2	RRF #3	RRF #4	RRF #5	Mean RRF	SD	%RSD
Naphthalene	0.75	0.66	0.61	0.64	0.71	0.67	0.056	8.29%
2-Methylnaphthalene	1.30	1.15	1.10	1.12	1.26	1.19	0.089	7.47%
Acenaphthylene	1.44	1.27	1.24	1.28	1.43	1.33	0.096	7.19%
Acenaphthene	0.94	0.84	0.80	0.83	0.94	0.87	0.067	7.72%
Fluorene	1.05	0.94	0.88	0.92	1.07	0.97	0.082	8.43%
Phenanthrene	1.15	1.06	1.01	1.05	1.23	1.10	0.088	8.00%
Anthracene	1.02	1.00	0.98	0.95	1.14	1.02	0.074	7.25%
Fluoranthene	1.26	1.15	1.08	1.13	1.28	1.18	0.085	7.21%
Pyrene	1.31	1.27	1.13	1.15	1.41	1.25	0.115	9.22%
Benzo(a)anthracene	1.13	1.05	1.05	1.04	1.23	1.10	0.082	7.43%
Chrysene	1.13	1.02	0.97	0.98	1.11	1.04	0.073	7.00%
Benzo(b)fluoranthene	1.69	1.45	1.46	1.42	1.86	1.58	0.194	12.33%
Benzo(k)fluoranthene	1.24	1.25	1.14	1.18	1.26	1.21	0.052	4.32%
Benzo(e)pyrene	1.20	1.12	1.06	1.06	1.19	1.12	0.066	5.89%
Benzo(a)pyrene	1.07	0.99	0.96	0.96	1.14	1.02	0.080	7.81%
Perylene	0.70	0.63	0.58	0.60	0.70	0.64	0.059	9.12%
Indeno(1,2,3-c,d)pyrene	2.19	2.01	1.92	1.99	2.26	2.07	0.143	6.90%
Dibenzo(a,h)anthracene	1.66	1.60	1.56	1.61	1.87	1.66	0.122	7.35%
Benzo(g,h,i)perylene	2.23	2.05	1.96	2.00	2.32	2.11	0.154	7.28%
d_8-Naphthalene	4.22	4.15	4.16	4.18	4.10	4.16	0.044	1.05%
d_8-Acenaphthylene	1.29	1.29	1.28	1.27	1.30	1.29	0.012	0.91%
d_{10}-Acenaphthene	1.32	1.34	1.32	1.30	1.32	1.32	0.013	1.00%
d_{10}-Fluorene	0.93	0.95	0.94	0.95	0.95	0.94	0.011	1.21%
d_{10}-Phenanthrene	0.82	0.82	0.82	0.86	0.88	0.81	0.026	3.09%
d_{10}-Fluoranthene	1.03	1.00	1.07	1.07	0.99	1.03	0.038	3.71%
d_{12}-Benzo(a)anthracene	0.75	0.70	0.70	0.72	0.70	0.71	0.022	3.09%
d_{12}-Chrysene	0.82	0.79	0.81	0.83	0.84	0.82	0.021	2.56%
d_{12}-Benzo(b)fluoranthene	1.35	1.39	1.46	1.27	1.32	1.36	0.072	5.32%
d_{12}-Benzo(k)fluoranthene	1.95	1.95	2.14	1.84	2.11	2.00	0.124	6.23%
d_{12}-Benzo(a)pyrene	1.91	1.96	2.11	1.82	1.99	1.96	0.107	5.46%
d_{12}-Indeno(1,2,3-c,d)pyrene	0.92	0.88	0.98	0.85	0.98	0.92	0.059	6.40%
d_{14}-Dibenzo(a,h)anthracene	0.87	0.84	0.91	0.78	0.89	0.86	0.049	5.71%
d_{12}-Benzo(g,h,i)perylene	0.80	0.76	0.83	0.73	0.80	0.78	0.042	5.36%
d_{14}-Terphenyl	0.52	0.52	0.49	0.48	0.51	0.51	0.018	3.59%
d_{12}-Benzo(e)pyrene	0.37	0.37	0.37	0.36	0.36	0.36	0.005	1.50%
d_{10}-Anthracene	0.69	0.73	0.74	0.80	0.90	0.77	0.080	10.40%
d_{10}-2-Methylnaphthalene	---	---	---	---	---	---	---	---
d_{10}-Pyrene	---	---	---	---	---	---	---	---
d_{12}-Perylene	---	---	---	---	---	---	---	---

FIGURE 14C

EXAMPLE OF CONTINUING CALIBRATION (CONCAL) SUMMARY CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

CONCAL ID: CC1202
CONCAL DATE: 12/3/94

ICAL ID: ST1120
ICAL DATE: 3-DEC-94

INSTRUMENT: W
OPERATOR: MPA

	RRF	ICAL RRF	ΔRRF	RPD %
Naphthalene	0.68	0.67	0.01	1.5
2-Methylnaphthalene	1.42	1.19	0.23	17.6
Acenaphthylene	1.42	1.33	0.09	6.6
Acenaphthene	0.91	0.87	0.04	4.5
Fluorene	0.98	0.97	0.01	1.0
Phenanthrene	1.10	1.10	0.00	0.0
Anthracene	0.98	1.02	-0.04	4.0
Fluoranthene	1.12	1.18	-0.06	5.2
Pyrene	1.18	1.25	-0.07	5.8
Benzo(a)anthracene	1.08	1.10	-0.02	1.8
Chrysene	1.04	1.04	0.00	0.0
Benzo(b)fluoranthene	1.46	1.58	-0.12	7.9
Benzo(k)fluoranthene	1.12	1.21	-0.09	7.7
Benzo(e)pyrene	1.04	1.12	-0.08	7.4
Benzo(a)pyrene	0.95	1.02	-0.07	7.1
Perylene	0.62	0.64	-0.02	3.2
Indeno(1,2,3-c,d)pyrene	2.04	2.07	-0.03	1.5
Dibenzo(a,h)anthracene	1.61	1.66	-0.05	3.1
Benzo(g,h,i)perylene	2.11	2.11	0.00	0.0
d_8-Naphthalene	4.78	1.16	0.68	15.3
d_8-Acenaphthylene	1.20	1.29	-0.09	7.2
d_{10}-Acenaphthene	1.25	1.32	-0.07	5.5
d_{10}-Fluorene	0.85	0.94	-0.09	10.1
d_{10}-Phenanthrene	0.79	0.81	-0.02	2.5
d_{10}-Fluoranthene	1.05	1.03	0.02	1.9
d_{12}-Benzo(a)anthracene	0.69	0.71	-0.02	2.9
d_{12}-Chrysene	0.82	0.82	0.00	0.0
d_{12}-Benzo(b)fluoranthene	1.24	1.36	-0.12	9.2
d_{12}-Benzo(k)fluoranthene	1.91	2.00	-0.09	4.6
d_{12}-Benzo(a)pyrene	1.87	1.96	-0.09	4.7
d_{12}-Indeno(1,2,3-c,d)pyrene	0.84	0.92	-0.08	9.1
d_{14}-Dibenzo(a,h)anthracene	0.80	0.86	-0.06	7.2
d_{12}-Benzo(g,h,i)perylene	0.76	0.78	-0.02	2.6
d_{14}-Terphenyl	0.50	0.51	-0.01	2.0
d_{12}-Benzo(e)pyrene	0.37	0.36	0.01	2.7
d_{10}-Anthracene	0.71	0.77	-0.06	8.1
d_{10}-2-Methylnaphthalene	---	---		
d_{10}-Pyrene	---	1.000		
d_{12}-Perylene	---	1.000		

FIGURE 15A.

EXAMPLE OF SUMMARY REPORT OF LCS RESULTS
CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

Client ID CARB	Sample Matrix: XAD-2	ICAL ID: ST1120	Resin Lot #: LC1130M
Lab ID: 14129/LCS1/LCS2	Date Received: NA	ICAL DATE: 12/3/94	LCS IDs: NA
Instrument: W	Date Extracted: 11/30/94	CONCAL ID: NA	LCS DATE: NA
Operator: MPA	Date Analyzed: 12/3/94	CONCAL DATE: NA	
Reviewer: JCM	Sample amount: Sample	Units: NA	

COMPOUND:	LCS1 %R	LCS2 %R	RPD %
Naphthalene	100	103	3.0
2-Methylnaphthalene	96	95	1.0
Acenaphthylene	95	97	2.1
Acenaphthene	92	94	2.2
Fluorene	94	96	2.1
Phenanthrene	93	94	1.1
Anthracene	91	89	2.2
Fluoranthene	90	92	2.2
Pyrene	87	89	2.3
Benzo(a)anthracene	87	86	1.2
Chrysene	83	89	7.0
Benzo(b)fluoranthene	92	93	1.1
Benzo(k)fluoranthene	92	95	3.2
Benzo(e)pyrene	97	99	2.0
Benzo(a)pyrene	89	92	3.3
Perylene	89	89	0.0
Indeno(1,2,3-c,d)pyrene	87	90	3.4
Dibenzo(a,h)anthracene	88	90	2.2
Benzo(g,h,i)perylene	89	91	1.2
Internal Standards (%R)			
d_8-Naphthalene	67	64	
d_8-Acenaphthylene	73	70	
d_{10}-Acenaphthene	76	75	
d_{10}-Fluorene	79	81	
d_{10}-Phenanthrene	88	93	
d_{10}-Fluoranthene	84	80	
d_{12}-Benzo(a)anthracene	96	98	
d_{12}-Chrysene	96	91	
d_{12}-Benzo(b)fluoranthene	88	85	
d_{12}-Benzo(k)fluoranthene	85	84	
d_{12}-Benzo(a)pyrene	92	90	
d_{12}-Indeno(1,2,3-c,d)pyrene	104	105	
d_{14}-Dibenzo(a,h)anthracene	96	96	
d_{12}-Benzo(g,h,i)perylene	102	103	
Alternate Standard (%R)			
d_{10}-Anthracene	83	85	

FIGURE 15B

LCS RECOVERIES FOR BENZO(a)PYRENE

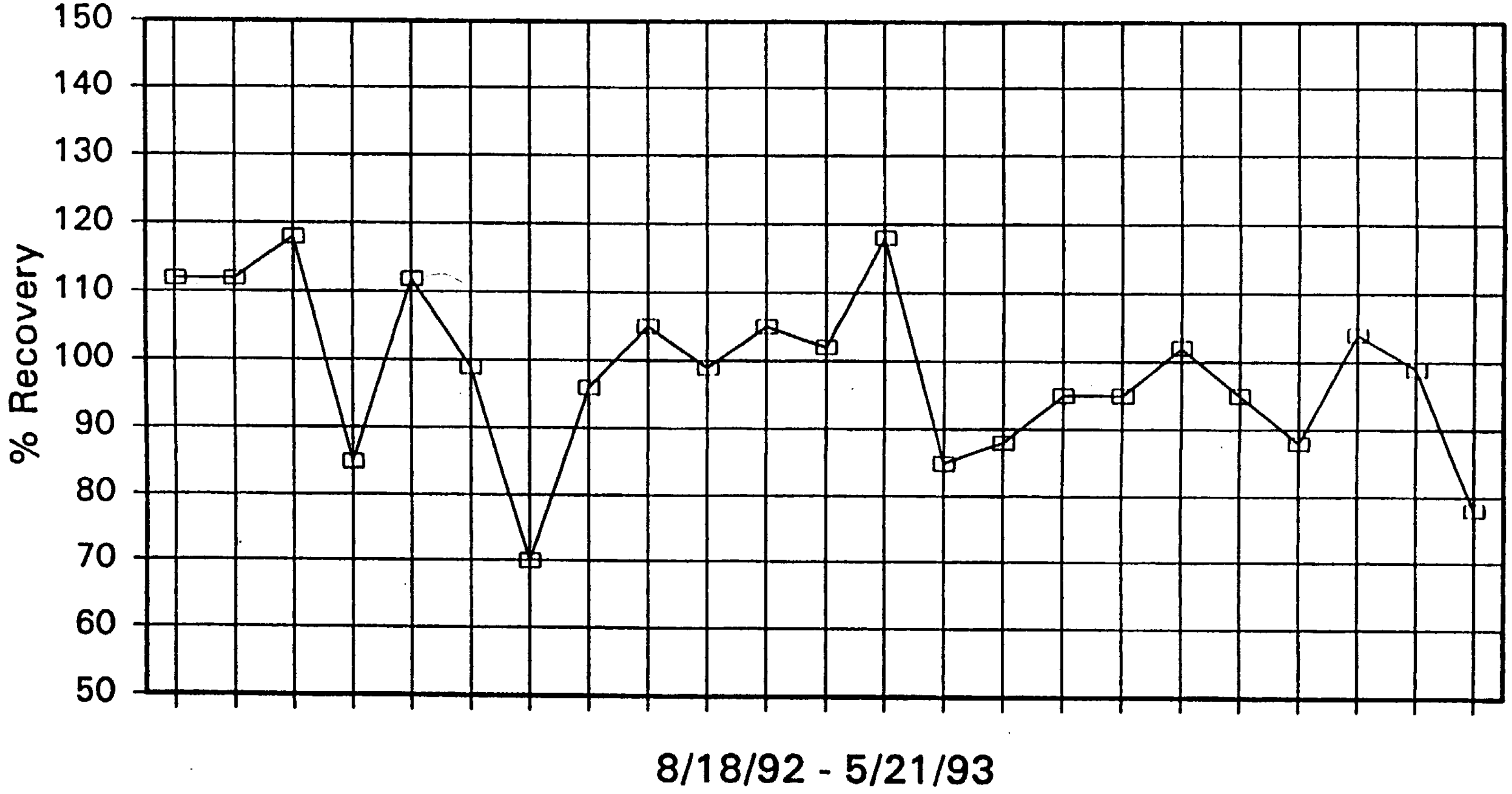


FIGURE 16A

EXAMPLE GC/MS SUMMARY REPORT (HRMS) FOR SAMPLE RUN #32
CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

Lab ID: 14129-02
Acquired: 12/3/94 16:23:40
Client ID: M429-32

ICAL ID: 12/3/94 16:23:40
ICAL DATE: 12/3/94

Instrument: W
Operator: MPA
Reviewer: JCM

	RT	RRT	Area	RRF	Amt. (ng)	% REC
Naphthalene	8:21		1.053 E+10	0.67	10,478.37	
2-Methylnaphthalene	9:41		1.790 E+08	1.19	140.98	
Acenaphthylene	11:03		9.371 E+08	1.33	712.59	
Acenaphthene	11:19		7.649 E+06	0.87	8.21	
Fluorene	12:05		2.417 E+07	0.97	30.02	
Phenanthrene	13:17		8.402 E+08	1.10	925.53	
Anthracene	13:21		2.905 E+07	1.02	34.54	
Fluoranthene	14:36		5.932 E+08	1.18	254.36	
Pyrene	14:52		7.611 E+08	1.25	307.62	
Benzo(a)anthracene	16:32		3.120 E+06	1.10	1.9	
Chrysene	16:32		9.620 E+06	1.04	6.2	
Benzo(b)fluoranthene	18:49		1.030 E+06	1.58	7.6	
Benzo(k)fluoranthene	Not found		0.0	1.21		
Benzo(e)pyrene	19:36		1.646 E+07	1.12	13.61	
Benzo(a)pyrene	19:46		4.936 E+06	1.02	3.95	
Perylene	20:01		1.823 E+06	0.64	2.32	
Indeno(1,2,3-c,d)pyrene	23:54		5.728 E+06	2.07	4.37	
Dibenzo(a,h)anthracene	23:56		5.875 E+05	1.66	0.59	
Benzo(g,h,i)perylene	25:09		1.584 E+07	2.11	14.95	
d_8-Naphthalene	8:18	1.000	4.794 E+08	1.16	124.92	62.5
d_8-Acenaphthylene	11:01	1.000	1.972 E+08	1.29	166.07	83.0
d_{10}-Acenaphthene	11:16	1.000	2.142 E+08	1.32	176.19	88.1
d_{10}-Fluorene	12:02	1.000	1.658 E+08	0.94	190.71	95.4
d_{10}-Phenanthrene	13:16	1.000	1.652 E+07	0.81	213.39	106.7
d_{10}-Fluoranthene	14:34	1.000	3.955 E+08	1.03	116.22	58.1
d_{12}-Benzo(a)anthracene	16:28	1.000	2.835 E+08	0.71	121.18	60.6
d_{12}-Chrysene	16:31	1.000	2.987 E+08	0.82	111.08	55.5
d_{12}-Benzo(b)fluoranthene	18:45	1.000	3.439 E+08	1.36	165.79	41.4
d_{12}-Benzo(k)fluoranthene	18:50	1.000	4.304 E+08	2.00	141.02	35.3
d_{12}-Benzo(a)pyrene	19:41	1.000	4.895 E+08	1.96	163.67	40.9
d_{12}-Indeno(1,2,3-c,d)pyrene	23:46	1.000	2.529 E+08	0.92	179.71	44.9
d_{14}-Dibenzo(a,h)anthracene	23:45	1.000	2.400 E+08	0.86	182.65	45.7
d_{12}-Benzo(g,h,i)perylene	24:60	1.000	2.006 E+08	0.78	167.24	41.8
d_{14}-Terphenyl	14:55		7.988 E+08	0.51	523	105
d_{12}-Benzo(e)pyrene	19:32	1.000	3.011 E+08	0.36	676.33	135.3
d_{10}-Anthracene	13:20	1.000	6.795 E+07	0.77	95.29	47.6
d_{10}-2-Methylnaphthalene	9:38	1.000	1.844 E+07	---	100	
d_{10}-Pyrene	14:51	1.000	6.576 E+08	---	100	
d_{12}-Perylene	19:56	1.000	3.057 E+08	---	100	

FIGURE 16B

EXAMPLE LABORATORY REPORT OF PAH RESULTS FOR SAMPLE RUN #32
CALIFORNIA AIR RESOURCES BOARD METHOD 429 POLYCYCLIC AROMATIC HYDROCARBONS

Client ID M429-32
Lab ID: 14129-02
Instrument: W
Operator: MPA
Reviewer: JCM

Sample Matrix: M429
Date Received: 11/18/94
Date Extracted: 11/30/94
Date Analyzed: 12/3/94
Sample amount: Sample

ICAL ID: ST1120
ICAL DATE: 12/3/94
CONCAL ID: NA
CONCAL DATE: NA
Units: ng/sample

Resin Lot #: LC1130M
LCS IDs: 14129-LCS1/LCS2
LCS DATE: 12/3/94

COMPOUND:	Conc.	R.L.	Flags
Naphthalene	10478	1600	
2-Methylnaphthalene	141	94	
Acenaphthylene	712	5.0	
Acenaphthene	8.2	5.0	
Fluorene	30	27	
Phenanthrene	930	80	
Anthracene	35	5.0	
Fluoranthene	254	5.0	
Pyrene	307	5.0	
Benzo(a)anthracene	ND	5.0	
Chrysene	6.2	5.0	
Benzo(b)fluoranthene	7.6	5.0	
Benzo(k)fluoranthene	ND	5.0	
Benzo(e)pyrene	14	5.0	
Benzo(a)pyrene	ND	5.0	
Perylene	ND	5.0	
Indeno(1,2,3-c,d)pyrene	ND	5.0	
Dibenzo(a,h)anthracene	ND	5.0	
Benzo(g,h,i)perylene	15	5.0	
Internal Standards (%R)			
d_8-Naphthalene	62		
d_8-Acenaphthylene	83		
d_{10}-Acenaphthene	88		
d_{10}-Fluorene	95		
d_{10}-Phenanthrene	107		
d_{10}-Fluoranthene	58		
d_{12}-Benzo(a)anthracene	61		
d_{12}-Chrysene	56		H
d_{12}-Benzo(b)fluoranthene	41		H
d_{12}-Benzo(k)fluoranthene	35		H
d_{12}-Benzo(a)pyrene	41		H
d_{12}-Indeno(1,2,3-c,d)pyrene	45		H
d_{14}-Dibenzo(a,h)anthracene	46		H
d_{12}-Benzo(g,h,i)perylene	42		
Alternate Standard (%R)			
d_{10}-Anthracene	48		
Surrogate Standard (%R)			
d_{14}-Terphenyl	105		
d_{12}-Benzo(e)pyrene	135		

FIGURE 17A
EXAMPLE OF TESTER'S SUMMARY OF LABORATORY REPORTS

Run #:	31	32	33	Field Blank	Method Blank	LCS #1	LCS #2
	ng/sample					percent recovery	
Naphthalene	4300	10000	460000 *	< 1600	< 1700	100	103
2-Methylnaphthalene	< 94	140	6400 *	< 94	<78	96	95
Acenaphthylene	140	710	85000 *	9.1	< 5.0	95	97
Acenaphthene	9.2	8.2	500	< 5.0	< 5.0	92	94
Fluorene	27	30	180	< 27	< 27	94	96
Phenanthrene	310	930	43000 *	< 80	< 74	93	94
Anthracene	26	35	2400	5.3	< 5.0	91	89
Fluoranthene	83	250	16000 *	16	< 5.0	90	92
Pyrene	110	310	20000 *	19	< 5.0	87	89
Benzo(a)anthracene	< 5.0	< 5.0	170	< 5.0	< 5.0	87	86
Chrysene	< 5.0	6.2	300	< 5.0	< 5.0	83	89
Benzo(b)fluoranthene	< 5.0	7.6	340	< 5.0	< 5.0	92	93
Benzo(k)fluoranthene	< 5.0	< 5.0	89	< 5.0	< 5.0	92	95
Benzo(e)pyrene	35	< 35	530	6.9	< 5.0	97	99
Benzo(a)pyrene	< 5.0	< 5.0	240	< 5.0	< 5.0	89	92
Perylene	< 5.0	< 5.0	110	< 5.0	< 5.0	89	89
Indeno(1,2,3-c,d)pyrene	< 5.0	< 5.0	100	< 5.0	< 5.0	87	90
Dibenzo(a,h)anthracene	< 5.0	< 5.0	6.4	< 5.0	< 5.0	88	90
Benzo(g,h,i)perylene	< 85	< 85	440	17.0	< 5.0	89	91
Internal Standards (%R)							
d_8-Naphthalene	66	62	57 *	53	55	67	64
d_8-Acenaphthylene	82	83	85 *	73	69	73	70
d_{10}-Acenaphthene	85	88	80 *	81	75	76	75
d_{10}-Fluorene	91	95	102	90	82	79	81
d_{10}-Phenanthrene	106	107	79 *	107	93	88	93
d_{10}-Fluoranthene	79	58	75 *	83	80	84	80
d_{12}-Benzo(a)anthracene	100	61	108	114	93	96	98
d_{12}-Chrysene	91	56	99	102	88	96	91
d_{12}-Benzo(b)fluoranthene	69	41 H	60	85	84	88	85
d_{12}-Benzo(k)fluoranthene	62	35 H	50	78	84	85	84
d_{12}-Benzo(a)pyrene	70	41 H	58	86	89	92	90
d_{12}-Indeno(1,2,3-c,d)pyrene	82	45 H	58	106	106	104	105
d_{14}-Dibenzo(a,h)anthracene	72	42 H	58	92	92	96	96
d_{12}-Benzo(g,h,i)perylene	84	46 H	58	107	104	102	103
Surrogate Standards (%R)							
d_{14}-Terphenyl	125	105	90	123	130		
d_{12}-Benzo(e)pyrene	72	135	112	103	112		
Alternate Standard (%R)							
d_{10}-Anthracene	67	48 H	115	116	101	83	85
Test Date	11/15/94	11/16/94	11/17/94	11/16/94	NA	NA	NA
Date received by lab.	11/18/94	11/18/94	11/18/94	11/18/94	NA	NA	NA
Date extracted	11/30/94	11/30/94	11/30/94	11/30/94	11/30/94	11/30/94	11/30/94
Date analyzed	12/3/94	12/3/94	12/3/94	12/3/94	12/3/94	12/3/94	12/3/94

"<" denotes that the compound was not detected at levels above the indicated reporting limit.
"H" indicates internal Standard Recovery Results below 50%, but signal-to-noise greater than 10:1.
"*" indicates compounds reanalyzed at 1:50 dilution due to saturation.

FIGURE 17B
FIELD DATA SUMMARY FOR PAH EMISSIONS TEST

RUN ID	31	32	33
DATE	11-15-95	11-16-95	11-17-95
START/STOP TIME	1015/1435	1020/1645	0855/1525
LOCATION	STACK	STACK	STACK
STACK DIAMETER	35.5 in.	35.5 in.	35.5 in.
NOZZLE DIAMETER	0.3105	0.313 in.	0.3125 in.
METER BOX ID	5419	5419	5419

STANDARD DRY GAS VOLUME	$V_{m(std)}$	145.19	235.57	250.76	DSCF(68° F)
	V_m	132.65	213.67	228.10	cubic ft
	P_{bar}	29.78	29.98	29.88	inches Hg
	ΔH_{avg}	1.15	1.35	1.56	inches H_2O
	T_m	60.0	60.0	60.0	° F
	K_1	17.64	17.64	17.64	
	Y	1.08	1.08	1.08	
PERCENT MOISTURE	B_{ws}	12.9	15.0	18.4	percent
	Impinger + tare	2183.3	2092.3	2063	grams
	Final wt.	2609.8	2934.9	3210.2	grams
	Net imp. catch	426.5	842.6	1147.2	grams
	Silica gel tare	1561.8	1788.8	1585.7	grams
	Post sampling wt.	1590.0	1826.9	1536.2	grams
	Moisture gain	28.2	38.1	49.5	grams
	Total moisture (V_{1c})	454.7	880.7	1196.7	grams
	$V_{w(std)}$	21.43	41.50	56.39	DSCF(68° F)
	$V_{m(std)}$	145.19	235.57	250.76	DSCF(68° F)
	K_2	0.0471	0.0471	0.0471	
MOLECULAR WEIGHT	M_d	29.93	29.95	30.08	lb/lbmole
	M_s	28.40	28.16	27.86	lb/lbmole
	O_2	11.25	10.75	10.00	percent
	CO	0.00	0.00	0.00	percent
	CO_2	9.25	9.50	10.50	percent
	N_2	79.50	79.75	79.50	percent
	B_{ws}	12.86	14.98	18.36	percent
GAS VELOCITY	v_s	38.4	40.88	43.2	feet/second
	Δp	0.530	0.56	0.59	inches H_2O
	T_s	420	428	427	° F
	P_g	-0.27	-0.27	-0.27	inches H_2O
	P_s	29.76	29.96	29.86	inches Hg
	M_s	28.40	28.16	27.86	lb/lbmole
	K_p	85.49	85.49	85.49	
	C_p	0.83	0.83	0.83	
VOLUMETRIC FLOW RATE	Q_{std}	8241	8531	8641	DSCF(68° F)
	B_{ws}	12.86	14.98	18.36	percent
	v_s	38.38	40.88	43.23	feet/second
	A	6.8736	6.8736	6.8736	sq. feet
	sec/min	60	60	60	
	K_1	17.64	17.64	17.64	
ISOKINETIC RATIO	I	96	99	104	percent
	T_s	420	428	427	° F
	$V_{m(std)}$	145.19	235.57	250.76	DSCFM(68° F)
	P_s	29.76	29.96	29.86	inches Hg
	v_s	38.38	40.88	43.23	feet/second
	θ	240	360	360	minutes
	B_{ws}	12.86	14.98	18.36	percent
	A_n	0.00053	0.00053	0.00053	sq. feet
	K_4	0.09450	0.09450	0.09450	

FIGURE 17C

EXAMPLE OF EMISSIONS TEST REPORT

	Run #31	Run #32	Run #33
(ng/dscm)			
Naphthalene	1046	1499	64782
2-Methylnaphthalene	<23	21.0	901
Acenaphthylene	34	106	11971
Acenaphthene	2.2	1.2	70
Fluorene	6.6	4.5	25
Phenanthrene	75	139	6056
Anthracene	<6.3	5.3	338
Fluoranthene	20	38	2253
Pyrene	27	47	2817
Benzo(a)anthracene	<1.2	<0.75	24
Chrysene	<1.2	0.92	42
Benzo(b)fluoranthene	<1.2	1.1	48
Benzo(k)fluoranthene	<1.2	<0.75	13
Benzo(e)pyrene	<8.5	<5.3	75
Benzo(a)pyrene	<1.2	<0.75	34
Perylene	<1.2	<0.75	16
Indeno(1,2,3-c,d)pyrene	<1.2	<0.75	14
Dibenzo(a,h)anthracene	<1.2	<0.75	0.90
Benzo(g,h,i)perylene	<21	<13	62
(ng/sec)			
Naphthalene	4068	6036	264180
2-Methylnaphthalene	<89	85	3676
Acenaphthylene	132	429	48816
Acenaphthene	8.7	5.0	287
Fluorene	26	18	103
Phenanthrene	293	561	24695
Anthracene	<25	21	1378
Fluoranthene	79	151	9189
Pyrene	104	187	11486
Benzo(a)anthracene	<4.7	<3.0	99
Chrysene	<4.7	3.7	172
Benzo(b)fluoranthene	<4.7	4.6	195
Benzo(k)fluoranthene	<4.7	<3.0	51
Benzo(e)pyrene	<33	<21	304
Benzo(a)pyrene	<4.7	<3.0	138
Perylene	<4.7	<3.0	63
Indeno(1,2,3-c,d)pyrene	<4.7	<3.0	57
Dibenzo(a,h)anthracene	<4.7	<3.0	3.7
Benzo(g,h,i)perylene	<80	<51	253

Standard Conditions: 68 deg.F (20 deg.C) & 29.92 in. Hg. (760 mm Hg)
"<" indicates that the compound was not detected above the reporting limit.

METHOD 429 - APPENDIX A

DETERMINATION OF THE METHOD DETECTION LIMIT

This procedure is based on the approach adopted by the EPA and included as Appendix B to Title 40, Part 136 of the Code of Federal Regulations (40 CFR 136). The samples shall be subjected to the same extraction, concentration, cleanup, and analytical procedures as those required for the field samples.

A1 Procedure

A1.1 Make an estimate of the detection limit (MDL) of each target compound using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent methylene chloride.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

(e) The concentration equivalent to five times the theoretical quantitation limit (Section 8.3.1 of the test method)

The experience of the analyst is important to this process, but one of the above considerations must be included in the initial estimate of the detection limit.

A1.2 Prepare according to the procedures described in Sections 4.2.2.1 to 4.2.2.4 enough XAD-2 resin to provide, at a minimum, eight aliquots each with mass equal to that required to pack a Method 429 sorbent cartridge. A contamination check must be conducted to identify those PAH for which a MDL cannot be determined by this method.

A1.3 To each of seven (7) aliquots of the clean resin, add an amount of each target analyte equal to the estimated detection limit. The mass of each resin aliquot must be known, and should be approximately 40 grams, the amount required to pack a Method 429 sorbent cartridge. The eighth aliquot shall be a blank.

A1.4 Process each of the eight samples through the entire PAH analytical method. All quality criteria requirements of the analytical method must be satisfied.

A1.5 Report the analytical results. The laboratory report must satisfy all of the reporting requirements of Section 10 of the test method.

A1.6 It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with step A1.3. This will: (1) prevent repeating this entire procedure and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure a good estimate of the method detection, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each through the entire method, including blank measurements as described above in step A1.3. Evaluate these data:

(1) If the sample levels are in a desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL according to Section A2.

(2) If these measurements indicate the selected analyte level is not in correct range, reestimate the MDL with a new sample as in A1.2 and repeat steps A1.3 to A1.5.

A2 Calculation

A2.1 Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} x_i^2\right)}{n}\right] \qquad \text{429-(A)-(34)}$$

$$S = \sqrt[2]{S^2}$$

Where:

X_i, i = 1 to n, are the analytical results in the final method reporting units obtained from the n sample aliqouts and Σ refers to the sum of the X values from i = 1 to n.

A2.2 (a) Compute the MDL as follows:

$$MDL = t_{(n-1,\ 1-\alpha = 0.99)} \times (S) \qquad 429(A)\text{-}(35)$$

Where:

MDL = the method detection limit

$t_{(n-1,\ 1-\alpha\ =\ 0.99)}$ = Students' t-value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table 429(A)-1.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in A2.2(a) are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL = 0.64 MDL
UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

A3 Optional Iterative Procedure

A3.1 This is to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step A1.1, take the MDL as calculated in Step A2.2, spike the matrix at this calculated MDL and repeat the procedure starting with Step A1.3.

(b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A/S^2_B < 3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S_A^2 + 6S_B^2}{12}\right] \qquad 429(A)\text{-}(36)$$

if $S^2_A/S^2_B > 3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step A1.3. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the S_{pooled} as calculated in Equation 429(A)-3 to compute the final MDL according to the following equation:

$$MDL = 2.681\,(S_{pooled}) \qquad 429(A)\text{-}(37)$$

Where: 2.681 is equal to $t_{(12,\ 1-\alpha\ =\ .99)}$.

(d) The 95% confidence limits for MDL calculated using Equation 429(A)-4 are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

$$LCL = 0.72\ MDL$$
$$UCL = 1.65\ MDL$$

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLE 429(A)-1

SELECTED STUDENT'S t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of Replicates	Degrees of Freedom (n-1)	$t_{(n-1,\ .99)}$
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390

FORMULA: Table 1

M.W.: Table 1

POLYNUCLEAR AROMATIC HYDROCARBONS

METHOD: 5506
ISSUED: 5/15/85

OSHA: proposed for B[a]P: $0.2\ \mu g/m^3$
ACGIH: suspect carcinogen (B[a]P)

PROPERTIES: Table 1

COMPOUNDS:
- acenaphthene
- acenaphthylene
- anthracene
- benz[a]anthracene
- benzo[b]fluoranthene
- benzo[k]fluoranthene
- benzo[ghi]perylene
- benzo[a]pyrene
- benzo[e]pyrene
- chrysene
- dibenz[a,h]anthracene
- fluoranthene
- fluorene
- indeno[1,2,3-cd]pyrene
- naphthalene
- phenanthrene
- pyrene

SYNONYMS: PAH; PNA; also see Table 2.

SAMPLING

SAMPLER: FILTER + SORBENT
(2-µm, 37-mm PTFE + washed XAD-2, 100 mg/50 mg)

FLOW RATE: 2 L/min

VOL-MIN: 200 L
-MAX: 1000 L

SHIPMENT: transfer filters to culture tubes; wrap sorbent and culture tubes in Al foil; ship @ 0 °C

SAMPLE STABILITY: unknown; protect from heat and UV radiation

FIELD BLANKS: 10% (≥3) of samples
MEDIA BLANKS: 6 to 10

AREA SAMPLES: 8 replicates on preweighed filters for solvent selection

ACCURACY

RANGE STUDIED, BIAS, AND OVERALL PRECISION (s_r): not measured

MEASUREMENT

METHOD: HPLC, FLUORESCENCE/UV DETECTION

ANALYTE: compounds above

EXTRACTION: 5 mL organic solvent appropriate to sample matrix (step 7)

COLUMN: 15 cm x 4.6 mm, reverse phase, 5-µm C_{18}

INJECTION VOLUME: 10 to 50 µL

MOBILE PHASE: H_2O/CH_3CN gradient @ ambient temperature

FLOW RATE: 1.0 mL/min

DETECTORS: UV @ 254 nm; fluorescence @ 340 nm (excitation), 425 nm (emission)

CALIBRATION: external standards in CH_3CN

RANGE, LOD AND PRECISION (s_r): EVALUATION OF METHOD

APPLICABILITY: The working range for B[a]P is 1 to 50 $\mu g/m^3$ for a 400-L air sample. Specific sample sets may require modification in filter extraction solvent, choice of measurement method, and measurement conditions (see EVALUATION OF METHOD).

INTERFERENCES: Any compound which elutes at the same HPLC retention time may interfere. Heat, ozone, NO_2, or UV light may cause sample degradation.

OTHER METHODS: This revises P&CAM 206 and 251 [1]. The spectrophotometric methods, P&CAM 184 and 186 [1], have not been revised. Also see Method 5515 (GC).

REAGENTS:

1. Filter extraction solvent: benzene,* cyclohexane, methylene chloride, or other appropriate solvents, pesticide grade grade (step 7).
2. Water, distilled, deionized, degassed.
3. Acetonitrile, HPLC grade, degassed.
4. PAH reference standards,* appropriate to the PAH-containing matrix sampled.
5. Calibration stock solution, 0.25 mg/mL.* Check purity of each PAH reference standard by GC/FID, HPLC/fluorescence and/or melting point. Purify, if necessary, by recrystallization. Weigh 25 mg of each PAH into a 100-mL volumetric flask; dilute to volume with acetonitrile. Stable six months if refrigerated and protected from light.

*See SPECIAL PRECAUTIONS.

EQUIPMENT:

1. Sampler:
   a. Filter. PTFE-laminated membrane filter, 2-µm pore size, 37-mm diameter (ZEFLOUR, Membrana, Pleasanton, CA or equivalent), backed by a gasket (37-mm OD, 32-mm ID) cut from a cellulose support pad, in cassette filter holder.
      NOTE 1: If sampling is to be done in bright sunlight, use opaque or foil-wrapped cassettes to prevent sample degradation.
      NOTE 2: Take filters to be preweighed from the filter package and allow to equilibrate 24 hrs with laboratory atmosphere before taring.
   b. Sorbent tube, connected to filter with minimum length PVC tubing. Plastic caps are required after sampling. Washed XAD-2 resin (front = 100 mg; back = 50 mg) (Supelco ORBO 43 or equivalent). Pressure drop at 2 L/min airflow 1.6 to 2 kPa (15 to 20 cm H_2O).
2. Personal sampling pump capable of operating for 8 hrs at 2 L/min, with flexible connecting tubing.
3. Aluminum foil.
4. Vial, scintillation, 20-mL, glass, PTFE-lined cap.
5. Refrigerant, bagged.
6. Culture tubes, PTFE-lined screw cap, 13-mm x 100-mm.
7. Forceps.
8. Filters, 0.45-µm, PTFE or nylon (for filtering sample solutions).
9. Pipet, 5-mL.
10. Syringe or micropipets, 1- to 100-µL.
11. Ultrasonic bath.
12. HPLC, with gradient capability, fluorescence (excitation @ 240 nm, emission @ 425 nm) and UV (254 nm) detectors in series, electronic integrator, and column [HC-ODS-SILX (Perkin-Elmer Corp.), Vydac 201TP (The Separations Group) or equivalent; see page 5506-1].
13. Volumetric flasks, 10- and 100-mL.
14. Lighting in laboratory: incandescent or UV-shielded fluorescent.
15. Kuderna-Danish extractor.

SPECIAL PRECAUTIONS: Treat benzene and all polynuclear aromatic hydrocarbons as carcinogens. Neat compounds should be weighed in a glove box. Spent samples and unused standards are toxic waste. Regularly check counter tops and equipment with "black light" for fluorescence as an indicator of contamination by PAH.

SAMPLING:

1. Calibrate each personal sampling pump with a representative sampler in line.
2. Take personal samples at 2 L/min for a total sample size of 200 to 1000 L. Take a concurrent set of eight replicate area samples at 2 to 4 L/min on preweighed, 2-µm PTFE filters in an area of highest expected PAH concentration.
   NOTE: The area samples are needed for solvent selection (step 7).
3. Immediately after sampling, transfer the filter carefully with forceps to a scintillation vial. Hold filter at edge to avoid disturbing the deposit. Cap the scintillation vial and wrap it in aluminum foil.
   NOTE: This step is necessary to avoid loss of analytes due to sublimation and degradation by light.
4. Cap the sorbent tube and wrap it in aluminum foil.
5. Ship to laboratory in insulated container with bagged refrigerant.

SAMPLE PREPARATION:

NOTE: UV light may degrade PAH. Use yellow, UV-absorbing shields for fluorescent lights or use incandescent lighting.

6. Refrigerate samples upon receipt at laboratory.
7. Determine optimum extraction solvent.
   a. Allow the preweighed area filter samples to equilibrate 24 hrs with the laboratory atmosphere.
   b. Weigh the area filters. Determine total weight collected on each.
   c. Extract the first pair of area filters with acetonitrile, the second with benzene, the third with cyclohexane, and the fourth with methylene chloride, according to step 8.
      NOTE: Use alternate solvents, if appropriate. PAH of interest may be entrained within, and adsorbed by, particulate matter collected on the filter. It is necessary to determine the solvent which maximizes recovery of the PAH from each sample matrix. For example, methylene chloride [2,3] and benzene:ethanol (4:1 v/v) [4] have been recommended for extraction of PAH from diesel exhaust particulate.
   d. Analyze the extracts for the PAH of interest (steps 10 through 18). Normalize the total mass of PAH found to the mass of sample collected.
   e. Choose the solvent which gives the highest recovery of PAH of interest. Use the solvent chosen to extract the personal filter samples.
8. Extract filters.
   a. Add 5.0 mL of the solvent chosen in step 7 to each scintillation vial containing a filter. Start media and reagent blanks at this step.
   b. Cap and let sit 15 to 20 min in an ultrasonic bath.
      NOTE 1: Soxhlet extraction may be required when large amounts of highly adsorptive particulate matter (e.g., fly ash or diesel soot) are present.
      NOTE 2: The sample must be dissolved in acetonitrile for chromatography. If needed, perform solvent exchange as follows:
      CAUTION: To avoid loss of volatile components, do not allow the sample to go to dryness at any time.
      (1) After filtration (step 10), take the sample to near dryness in a Kuderna-Danish extractor.
      (2) Add ca. 1 mL acetonitrile, take to near dryness, and adjust final volume to 1.0 mL with acetonitrile and filter again.
9. Desorb PAH from sorbent.
   a. Score each sorbent tube with a file in front of the front (larger) sorbent section. Break tube at score line.

   b. Transfer glass wool plug and front sorbent section to a culture tube. Discard the foam plug. Transfer back sorbent section to a second culture tube.
   c. Add 5.0 mL acetonitrile to each culture tube. Cap the culture tubes.
   d. Allow samples to sit for 30 min. Swirl occasionally.

10. Filter all sample extracts through an 0.45-µm membrane filter.

CALIBRATION AND QUALITY CONTROL:

11. Calibrate daily with at least five working standards.
    a. Dilute aliquots of calibration stock solution with acetonitrile in 10-mL volumetric flasks (e.g., to 2.5, 0.5, 0.1, 0.02, and 0.002 µg/mL).
    b. Intersperse working standards and samples in the measurements.
    c. Prepare calibration graphs (peak area vs. µg of each PAH per sample).
12. Recovery and desorption efficiency.
    a. Determine recovery (R) from filters and desorption efficiency (DE) from sorbent tubes at least once for each lot of filters and sorbent tubes used in the range of interest.
       (1) Filters. Using a microliter syringe or micropipette, spike four filters at each of five concentration levels with a mixture of the analytes. Allow the filters to dry in the dark overnight. Analyze the filters (steps 8, 10, and 14 through 16. Prepare graphs of R vs. amounts found.
           NOTE: This step may not be used for some highly adsorptive particulate matrices for which calibration by the method of standard additions may be more accurate.
       (2) Sorbent tubes. Transfer an unused front sorbent section to a culture tube. Prepare a total of 24 culture tubes in order to measure DE at five concentration levels plus blanks in quadruplicate. Using a microliter syringe or micropipette, add calibration stock solution directly to sorbent. Cap culture tubes and allow to stand overnight. Analyze (steps 9, 10, and 14 through 16). Prepare graphs of DE vs. amounts found.
    b. Check R and DE at two levels for each sample set, in duplicate. Repeat determination of R and DE graphs if checks do not agree to within ±5% of DE graph.
13. Analyze at least three field blanks for each sample medium.

MEASUREMENT:

14. Set HPLC according to manufacturer's recommendations and to conditions on page 5506-1. Equilibrate column at 60% CH_3CN/40% H_2O at 1.0 mL/min for 15 min before injecting first sample.
15. Inject sample aliquot. Start mobile phase gradient:
    a. Linear gradient 60% CH_3CN to 100% CH_3CN, 20 min.
    b. Hold at 100% CH_3CN for 20 min.
       NOTE: Hold longer if necessary to prevent carryover of background, e.g., from coal dust.
    c. Linear gradient to initial condition, 5 min.
16. Measure peak areas.
    NOTE 1: Approximate retention times appear in Table 3.
    NOTE 2: If peak area is above the calibration range, dilute with appropriate solvent, reanalyze, and apply dilution factor in calculations.
    NOTE 3: If sample has many interferences, additional sample cleanup may be necessary. Many cleanup procedures have been published. Liquid-liquid partitioning between cyclohexane and nitromethane [5,6] is widely used, but other techniques may be more appropriate for specific samples.

CALCULATIONS:

17. Read the mass, µg (corrected for R or DE) of each analyte found on the filter (W) and front sorbent (W_f) and back sorbent (W_b) sections, and on the average media blank filter (B) and front sorbent (B_f) and back sorbent (B_b) sections from the calibration graphs.
18. Calculate concentration, C (µg/m³), in air as the sum of the particulate concentration and the vapor concentration using the actual air volume sampled, V (L).

$$C = \frac{(W - B + W_f + W_b - B_f - B_b) \cdot 10^3}{V}, \ \mu g/m^3.$$

NOTE: W_f and W_b include analyte originally collected on the filter as particulate, then volatilized during sampling. This can be a significant fraction for many PAH (e.g., fluoranthane, naphthalene, fluorene, anthracene, phenanthrene).

EVALUATION OF METHOD:

The fluorescence detector used in this method is both sensitive and selective. The detector can "see" as little as 50 pg of many PAH injected on the column. LODs for the 17 analytes range from 50 to 350 ng per sample. It does not respond to non-fluorescent molecules such as aliphatics. The method is, therefore, most amenable to determination of trace amounts of PAH in mixtures of aliphatic compounds. Successful applications include: aluminum reduction facilities, asphalt fume, coal gasification plants, coal liquefaction plants, coal tar pitch, coke oven emissions, creosote treatment facilities, diesel exhaust, graphite electrode manufacturing, petroleum pitch, and roofing tearoff operations.

This method has been evaluated by analyzing spiked filters, spiked sorbent tubes, and complete spiked sampling trains through which were drawn 500 L of air [7]. Each of the three groups was spiked with each analyte at two concentration levels in sextuplicate. Particular note should be made that the effect of particulate matter has not been evaluated, and every sampling matrix is unique. The data on the following page were obtained on spiked samplers stored refrigerated in the dark for three months followed by measurement with HPLC.

COMPOUND	CALIBRATION RANGE (µg per sample)	LOD (µg per sample)	MEASUREMENT PRECISION	
			SPIKED[a]	SPIKED + AIR[b]
1. ACENAPHTHENE	2.0 - 13	0.8	.058 S	.093 (50)
2. ACENAPHTHYLENE	1.0 - 100	0.35	.032 S	.075 (100)
3. ANTHRACENE	0.4 - 13	0.05	.039 S	.037 (5)
4. BENZ[a]ANTHRACENE	0.4 - 13	0.15	.032 F	.084 (5)
5. BENZO[b]FLUORANTHENE	0.4 - 12	0.1	.027 F	.028 (10)
6. BENZO[k]FLUORANTHENE	0.4 - 13	0.15	.025 F	.027 (1)
7. BENZO[ghi]PERYLENE	0.5 - 25	0.2	.031 F	.029 (10)
8. BENZO[a]PYRENE	0.4 - 14	0.2	.027 F	.029 (5)
9. BENZO[e]PYRENE	0.5 - 13	0.2	(c)	(c)
10. CHRYSENE	0.4 - 12	0.15	.039 F	.024 (5)
11. DIBENZ[a,h]ANTHRACENE	0.5 - 25	0.2	.026 F	.029 (10)
12. FLUORANTHENE	0.4 - 13	0.15	.026 S	.050 (10)
13. FLUORENE	0.7 - 13	0.25	.031 S	.090 (10)
14. INDENO[1,2,3-cd]PYRENE	0.5 - 12	0.2	.044 F	.032 (10)
15. NAPHTHALENE	0.6 - 13	0.25	.041 S	.125 (50)
16. PHENANTHRENE	0.4 - 13	0.1	.036 S	.070 (2)
17. PYRENE	0.5 - 13	0.2	(c)	(c)

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[a] RSD for filter (F) where volatilization is nil or for sorbent (S) where substantial volatilization may occur during sampling.

[b] RSD determined at the µg level shown in parenthesis for a spiked filter followed by a sorbent tube. After spiking, laboratory air was drawn through the sampling train at 2 L/min for 4 hrs.

[c] Not determined.

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METHOD REVISED BY: B. R. Belinky and E. J. Slick, NIOSH/DPSE.

Table 1. Formulae and physical properties.

COMPOUND (by M.W.)	EMPIRICAL FORMULA	MOLECULAR WEIGHT	DETECTOR	MELTING POINT (°C)	BOILING POINT (°C)*	REF.
1. NAPHTHALENE	$C_{10}H_8$	128.17	UV	80	218	[9]
2. ACENAPHTHYLENE	$C_{12}H_8$	152.20	UV	92-93	265-275	[10]
3. ACENAPHTHENE	$C_{12}H_{10}$	154.21	UV	96.2	279	[10]
4. FLUORENE	$C_{13}H_{10}$	166.22	UV	116	293-295	[9]
5. ANTHRACENE	$C_{14}H_{10}$	178.23	UV	218	340	[9]
6. PHENANTHRENE	$C_{14}H_{10}$	178.23	UV	100	340	[9]
7. FLUORANTHENE	$C_{16}H_{10}$	202.26	FL	110	—	[9]
8. PYRENE	$C_{16}H_{10}$	202.26	FL	156	399	[9]
9. BENZ[a]ANTHRACENE	$C_{18}H_{12}$	228.29	FL	158-159	—	[9]
10. CHRYSENE	$C_{18}H_{12}$	228.29	UV	255-256	—	[9]
11. BENZO[b]FLUORANTHENE	$C_{20}H_{12}$	252.32	FL	168	—	[9]
12. BENZO[k]FLUORANTHENE	$C_{20}H_{12}$	252.32	FL	217	480	[10]
13. BENZO[a]PYRENE	$C_{20}H_{12}$	252.32	FL	177	—	[9]
14. BENZO[e]PYRENE	$C_{20}H_{12}$	252.32	FL	178-179	—	[9]
15. BENZO[ghi]PERYLENE	$C_{22}H_{12}$	276.34	FL	273	—	[9]
16. INDENO[1,2,3-cd]PYRENE	$C_{22}H_{12}$	276.34	FL	161.5-163	—	[8]
17. DIBENZ[a,h]ANTHRACENE	$C_{22}H_{14}$	278.35	FL	262	—	[9]

*Many of these compounds will sublime.

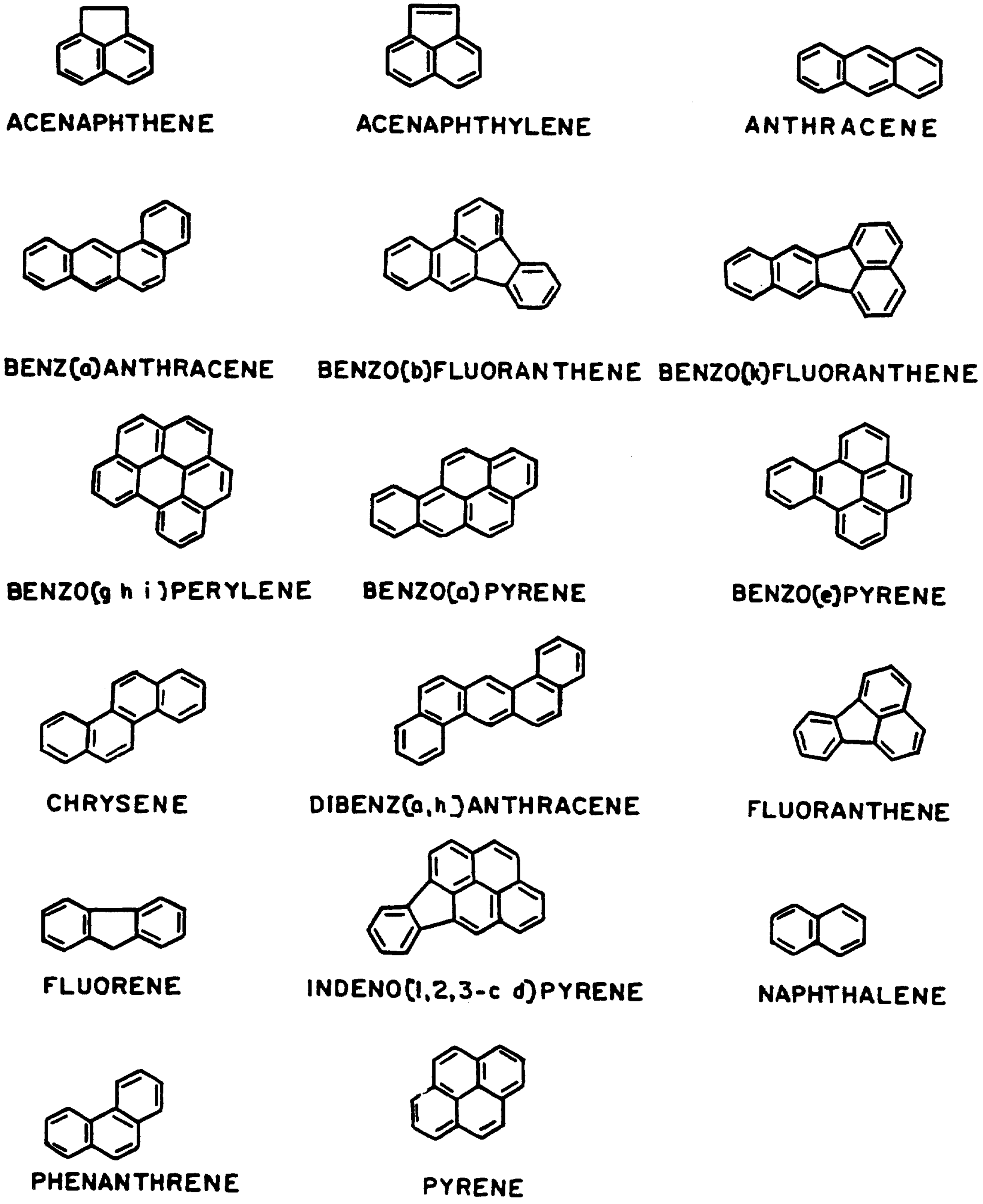
Table 2. Synonyms.

COMPOUND (alphabetically)	SYNONYMS
1. ACENAPHTHENE	CAS# 83-32-9
2. ACENAPHTHYLENE	CAS# 208-96-8
3. ANTHRACENE	CAS# 120-12-7
4. BENZ[a]ANTHRACENE	1,2-benzanthracene; benzo[b]phenanthrene; 2,3-benzophenanthrene; tetraphene; CAS# 56-55-3
5. BENZO[b]FLUORANTHENE	3,4-benzofluoranthene; 2,3-benzofluoranthene; benz[e]acephenanthrylene; B[b]F; CAS# 205-99-2
6. BENZO[k]FLUORANTHENE	11,12-benzofluoranthene; CAS# 207-08-9
7. BENZO[ghi]PERYLENE	1,12-benzoperylene; CAS# 191-24-2
8. BENZO[a]PYRENE	3,4-benzopyrene; 6,7-benzopyrene; B[a]P; BP; CAS# 50-32-8
9. BENZO[e]PYRENE	1,2-benzopyrene; 4,5-benzopyrene; B[e]P; CAS# 192-97-2
10. CHRYSENE	1,2-benzophenanthrene; benzo[a]phenanthrene; CAS# 218-01-9
11. DIBENZ[a,h]ANTHRACENE	1,2,5,6-dibenzanthracene; CAS# 53-70-3
12. FLUORANTHENE	benzo[jk]fluorene; CAS# 206-44-0
13. FLUORENE	CAS# 86-73-7
14. INDENO[1,2,3-cd]PYRENE	2,3-phenylenepyrene; CAS# 193-39-5
15. NAPHTHALENE	naphthene; CAS# 91-20-3
16. PHENANTHRENE	CAS# 85-01-8
17. PYRENE	benzo[def]phenanthrene; CAS#129-00-0

Table 3. Approximate PAH retention times.

COMPOUND	RETENTION TIME (min)*
1. NAPHTHALENE	2.4
2. ACENAPHTHALENE	2.8
3. ACENAPHTHENE	3.6
4. FLUORENE	3.9
5. PHENANTHRENE	4.7
6. ANTHRACENE	5.8
7. FLUORANTHENE	6.8
8. PYRENE	7.7
9. BENZ[a]ANTHRACENE	11.2
10. CHRYSENE	12.1
11. BENZO[e]PYRENE	14.0
12. BENZO[b]FLUORANTHENE	14.8
13. BENZO[k]FLUORANTHENE	16.5
14. BENZO[a]PYRENE	17.3
15. DIBENZ[a,h]ANTHRACENE	20.0
16. BENZO[ghi]PERYLENE	20.0
17. INDENO[1,2,3-cd]PYRENE	21.2

*NOTE: Determined with a Perkin-Elmer HC-ODS-SILX column. Actual retention times will vary with individual columns and column age.



ACENAPHTHENE

ACENAPHTHYLENE

ANTHRACENE

BENZ(a)ANTHRACENE

BENZO(b)FLUORANTHENE

BENZO(k)FLUORANTHENE

BENZO(g h i)PERYLENE

BENZO(a)PYRENE

BENZO(e)PYRENE

CHRYSENE

DIBENZ(a,h)ANTHRACENE

FLUORANTHENE

FLUORENE

INDENO(1,2,3-c d)PYRENE

NAPHTHALENE

PHENANTHRENE

PYRENE

Figure 1. Structures of PAH.

TECHNICAL REPORT DATA

Please read instructions on the reverse before completing

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4. TITLE AND SUBTITLE Final Report - Emissions Testing of Combustion Stack and Pushing Operations at Coke Battery No. 2 at Bethlehem Steel Corporation's Burns Harbor Division in Chesterton, Indiana Volume III of III	5. REPORT DATE February 1999 6. PERFORMING ORGANIZATION CODE
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15. SUPPLEMENTARY NOTES

16. ABSTRACT

The United States Environmental Protection Agency (EPA) is investigating the coke making industry to characterize hazardous air pollutants (HAPs) emitted from coke pushing operations and combustion (underfire) stacks. This test report addresses pushing emissions from a coke oven, and emissions from the combustion (underfire) stack that serves Coke Battery No. 2 at Bethlehem Steel Corporation's Burns Harbor Division in Chesterton, Indiana. The purpose of this test program was to quantify emissions from the inlet and outlet of the baghouse controlling emissions from the coke pushing operation and to quantify emissions from the combustion outlet stack. The data may be used by the EPA in the future to support a residual risk assessment for coke oven facilities.

The testing was performed to quantify uncontrolled and controlled air emissions of filterable particulate matter (PM), methylene chloride extractable matter (MCEM) and 19 polycyclic aromatic hydrocarbons (PAHs) including acenaphthene, acenaphthylene, anthracene, benzo(a)anthracene, benzo(a)pyrene, benzo(b)fluoranthene, benzo(e)pyrene, benzo(k)fluoranthene, benzo(ghi)perylene, chrysene, dibenzo(a,h)anthracene, fluoranthene, fluorene, indeno(1,2,3-cd)pyrene, 2-methylnapthalene, napthalene, perylene, phenanthrene, and pyrene. In addition, following the PM and MCEM analyses, the samples were analyzed to screen for the presence of 17 trace metals. Baghouse dust samples were also collected and analyzed for 16 trace metals. Simultaneous testing was performed at the inlet and outlet of the baghouse controlling emissions from the coke pushing operation. Sampling was also performed on the combustion outlet stack. In addition to pollutant testing, oxygen (O_2) and carbon dioxide (CO_2) were measured at each location. During the sampling program, Research Triangle Institute (RTI), another EPA contractor, monitored and recorded process and emission control system operating parameters.

This volume (Volume III) is comprised of 363 pages and includes Appendices: D (Calculations), E (QA/QC Data), F (Participants), and G (Sampling and Analytical Procedures).

17. KEY WORDS AND DOCUMENT ANALYSIS

a. DESCRIPTIONS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COASTI Field/Group
Baghouse Coke Ovens Emission Measurements Hazardous Air Pollutants Metals Methylene Chloride Extractable Matter Particulate Matter Polycyclic Aromatic Hydrocarbons		

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