SUMMARY SHEET 12
Inorganic Lead

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 5				
Job No.		FDS 5				
Sampling Location		FDS 5				
Run ID #		FDS 5				
Test Date		FDS 5				
Run Start Time		FDS 5				
Run Finish Time		FDS 5				
Net Traverse Points		FDS 1				
Traverse Matrix (Rectangular)		FDS 1				
Net Run Time, min	θ	FDS 5				
Nozzle Diameter, in.	D_n	FDS 5				
Dry Gas Meter Calibration Factor	Y	CDS 5				
Average ΔH (orifice meter), in. H_2O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H_2O	P_g	FDS 5				
Absolute Stack Pressure, in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Average Absolute Stack Temperature, R	T_s	FDS 5				
Carbon Dioxide, % dry	$\%CO_2$	FDS 3				
Oxygen, % dry	$\%O_2$	FDS 3				
Carbon Monoxide + Nitrogen, % dry	$\%(CO+N_2)$	FDS 3				
Dry Molecular Weight, lb/lb-mole	M_d	FDS 3				
Average DGM Temperature, °F	t_m	FDS 5				
Volume of Metered Gas Sample, dcf	V_m	FDS 5				
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 5				
Volume Water Condensed, mL	V_{lc}	FDS 5				
Volume of Water Vapor, scf	$V_{w(std)}$	SS 5				
Moisture Content, fraction	B_{ws}	SS 5				
Pitot Tube Coefficient	C_p	CDS 2a				
Average Velocity Pressure, in. H_2O	Δp	FDS 5				
Average $[(t_{si}+460)\,\Delta p]^{1/2}$	$[T_{si}\,\Delta p]^{1/2}$	FDS 5				
Velocity, ft/sec	v_s	SS 5				
Stack Area, ft^2	A	FDS 1				
Volumetric Flow Rate, dscfh	Q_{sd}	SS 5				
Volumetric Flow Rate, wscfh	Q_{sw}	SS 5				
Isokinetic Sampling Rate, %	%I	SS 5				
Pb Concentration from Cal Curve, μg	C_c	LDS 12				
Sample Volume, mL	V_s	LDS 12				
Aliquot Volume, mL	V_a	LDS 12				
Dilution Factor, if applicable	F					
Total Pb in Sample, μg	C^o_{Pb}	SS 12				
Pb Concentration, lb/dscf	C_{Pb}	SS 12				
Post-test Calibration Checks						
Temperature and Barometer		CDS 2d				
Metering System		CDS 5				

$$C^o_{Pb} = C_c \frac{V_s}{V_a} F \qquad C_{Pb} = 2.205 \times 10^{-9} \frac{C^o_{Pb}}{V_{m(std)}}$$

FIELD PROCEDURE 12
Inorganic Lead

Note: The sampling procedure is the same as that in FP 5, except for the following (use FDS 5 for the sampling data).

A. Sampling

1. Use a filter with a lot assay for lead; the filter need not be weighed.
2. Assemble the train as shown in Figure F12-1. Use impingers rather than an alternative condenser system.
3. In each of the first two impingers, place 100 mL 0.1 N HNO_3 (rather than water).
4. Use as sample storage containers 1 L borosilicate glass bottles with screw-cap liners that are either rubber-backed Teflon or leak-free and resistant to chemical attack by 0.1 N HNO_3.

B. Sample Recovery

1. The sample recovery procedure for **Containers 1, 2, and 3** is the same as that in FP 5, except for the following:
   a. Use 0.1 N HNO_3 as the rinse rather than water; save a blank of the acid.
   b. Use glass rather than a polyethylene funnel.
2. <u>Container No. 4</u> (Impingers). Several sample containers may be used. Clean each of the first three impingers and connecting glassware in the following manner:
   a. Wipe the impinger ball joints free of silicone grease, and cap the joints.
   b. Rotate and agitate each impinger, so that the impinger contents might serve as a rinse solution.
   c. Remove the outlet ball joint cap, and drain the contents through this opening into a 500-mL graduated cylinder; do not separate the impinger parts (inner and outer tubes) during this operation. Measure the liquid volume to within 2 mL. Alternatively, weigh the liquid to within 0.5 g. Note any color or film observed in the impinger catch.
   d. Transfer the contents to Container No. 4.
   e. Measure and record the total amount of 0.1 N HNO_3 used for rinsing in this step and in step f below. Pour about 30 mL 0.1 N HNO_3 into each of the first three impingers and agitate the impingers. Drain the 0.1 N HNO_3 through the outlet arm of each impinger into Container No. 4. Repeat this operation a second time; inspect the impingers for any abnormal conditions.
   f. Wipe the socket joints of the glassware connecting the impingers free of silicone grease and rinse each piece of glassware twice with 0.1 N HNO_3; transfer this rinse into Container No. 4. <u>(Do not rinse or brush the glass- fritted filter support</u>.)
   g. Mark the height of the fluid level and label and identify the container.

C. Alternatives

1. Simultaneous Determination of Particulate and Lead Emissions. Method 5 (FP 5) may be used to simultaneously determine Pb provided that:
   a. Acetone is used to remove particulate from the probe and inside of the filter holder as specified by Method 5.
   b. 0.1 N HNO_3 is used in the impingers.
   c. A glass fiber filter with a low Pb background is used.
   d. The entire train contents, including the impingers, are treated and analyzed for Pb.
2. Filter Location. A filter may be used between the third and fourth impingers provided that the filter is included for analysis for Pb.
3. In-Stack Filter. An in-stack filter may be used provided that:
   a. A glass-lined probe and at least two impingers, each containing 100 mL 0.1 N HNO_3, are used after the in-stack filter.
   b. The probe and impinger contents are recovered and analyzed for Pb. (Recover sample from the nozzle with acetone if a particulate analysis is to be made.)

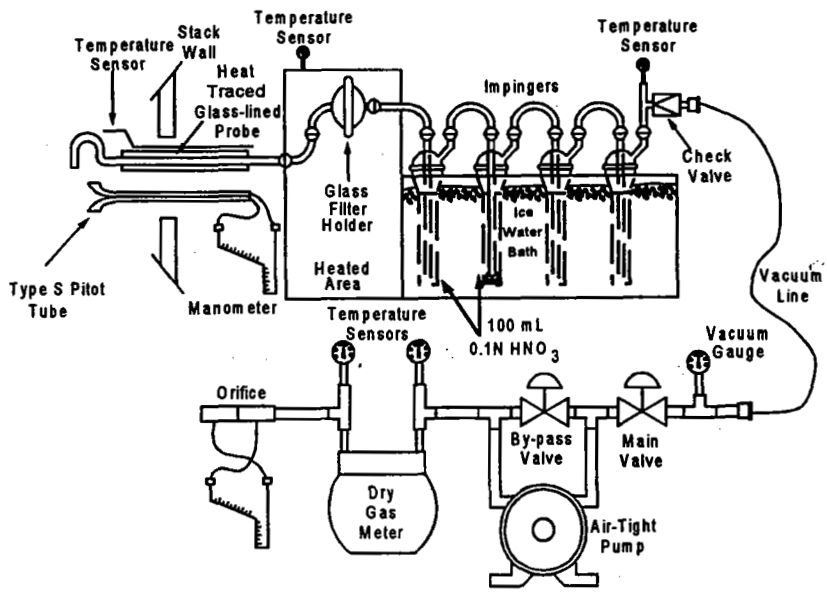

Figure F12-1. Inorganic Lead Sampling Train.

LABORATORY PROCEDURE 12
Inorganic Lead

A. Reagent Preparation

1. Nitric Acid, 0.1 N. Dilute 6.5 mL conc. HNO_3 to 1 L with water.
2. HNO_3, 6 N. Dilute 390 mL conc. HNO_3 to 1 L with water.
3. HNO_3, 50% (v/v). Dilute 500 mL conc. HNO_3 to 1 L with water.
4. Stock Lead Standard Solution, 1000 μg Pb/mL. Dissolve 0.1598 g $Pb(NO_3)_2$ in about 60 mL water, add 2 mL conc. HNO_3, and dilute to 100 mL with water.
5. Working Lead Standards. Pipet 0.0, 1.0, 2.0, 3.0, 4.0, and 5.0 mL stock lead standard solution into 250-mL volumetric flasks. Add 5 mL conc. HNO_3 to each flask, and dilute to volume with water. These working standards contain 0.0, 4.0, 8.0, 12.0, 16.0, and 20.0 μg Pb/mL, respectively. Prepare, as needed, additional standards at other concentrations in a similar manner.
6. Hydrogen Peroxide, 3%. Dilute 10 mL 30% H_2O_2 to 100 mL with water.

B. Sample Preparation

1. <u>Container No. 1</u> (Filter)
   a. Cut the filter into strips and transfer the strips and all loose particulate matter into a 125-mL Erlenmeyer flask. If the estimated particulate catch is greater than 800 mg, use a 250-mL flask (see step B3).
   b. Rinse the petri dish with 10 mL 50% HNO_3 to insure a quantitative transfer, and add to the flask.
2. <u>Containers No. 2 and No. 4</u> (Probe and Impingers)
   a. Check the liquid level in Containers No. 2 and No. 4, and determine and record loss (if any) on LDS 12.
   b. Combine the contents of Containers No. 2 and No. 4, and take to dryness on a hot plate.
3. Sample Extraction for Lead
   a. Based on the approximate stack gas particulate concentration and the total volume of stack gas sampled, estimate the total weight of particulate sample collected.
   b. Then transfer the residue from Containers No. 2 and No. 4 to the 125-mL Erlenmeyer flask that contains the filter using rubber policeman and 10 mL 50% HNO_3 for every 100 mg of sample collected in the train or a minimum of 30 mL 50% HNO_3, whichever is larger.
   c. Place the Erlenmeyer flask on a hot plate, and heat with periodic stirring for 30 min at just below the boiling point. If the sample volume falls below 15 mL, add more 50% HNO_3. Add 10 mL 3% H_2O_2, and continue heating for 10 min. Add 50 mL hot (80°C) water, and heat for 20 min. Remove the flask from the hot plate, and allow to cool.
   d. Filter the sample through a Millipore membrane filter, or equivalent, and transfer the filtrate to a 250-mL volumetric flask. Dilute to volume with water.
4. Filter Blank
   a. Take two filters from each lot of filters used in the sampling train.
   b. Cut each filter into strips, and place each filter in a separate 125-mL Erlenmeyer flask.
   c. Add 15 mL 50% HNO_3, and treat as described in step B using 10 mL 3% H_2O_2 and 50 mL hot water. Filter and dilute to a total volume of 100 mL with water.
5. HNO_3 Blank
   a. Take the entire 200 mL 0.1 N HNO_3 to dryness on a steam bath.
   b. Add 15 mL 50% HNO_3, and treat as described in section B3 using 10 mL 3% H_2O_2 and 50 mL hot water. Dilute to a total volume of 100 mL with water.

C. Analysis

1. Calibrate the spectrophotometer as follows:
   a. Measure the absorbance of the standard solutions using the instrument settings recommended by the spectrophotometer manufacturer. Repeat until good agreement ($\leq \pm 3\%$) is obtained between two consecutive readings.

b. Plot the absorbance (y-axis) versus concentration in μg Pb/mL (x-axis). Draw or compute a straight line through the linear portion of the curve. Do not force the calibration curve through zero, but if the curve does not pass through the origin or $\leq \pm 0.003$ absorbance units, check for incorrectly prepared standards and for curvature in the calibration curve.

c. To determine stability of the calibration curve, run a blank and a standard after every five samples, and recalibrate, as necessary.

2. Lead Determination

a. Determine the absorbance for each source sample, the filter blank, and 0.1 N HNO_3 blank. Analyze each sample three times in this manner. Make appropriate dilutions, as required, to bring all sample Pb concentrations into the linear absorbance range of the spectrophotometer.

b. If the Pb concentration of a sample is at the low end of the calibration curve and high accuracy is required, take the sample to dryness on a hot plate and dissolve the residue in the appropriate volume of water to bring it into the optimum range of the calibration curve.

c. If high concentrations of copper are present, analyze the samples at 283.3 nm.

3. Container No. 3 (Silica Gel). If not done in the field, weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g.

D. Check for Matrix Effects

Check at least one sample from each source using the Method of Additions as follows:

1. Add or spike an equal volume of standard solution to an aliquot of the sample solution, then measure the absorbance of the resulting solution and the absorbance of an aliquot of unspiked sample.

2. Calculate the Pb concentration C_s in μg/mL of the sample solution. Volume corrections are not required if the solutions as analyzed are made to the same final volume. Therefore, C_s and C_a represent Pb concentrations before dilutions.

3. Method of Additions procedures described on pages 9-4 and 9-5 of the section entitled "General Information" of the Perkin Elmer Corporation Atomic Absorption Spectrophotometry Manual, No. 303-0152 may also be used.

4. If the results of the Method of Additions procedure used on the single source sample is $> \pm 5\%$ of the value obtained by the routine atomic absorption analysis, then reanalyze all samples from the source using the Method of Additions procedure.

LABORATORY DATA SHEET 12
Inorganic Lead

Client/Plant Name _____ Job # _____ Date/Time _____

Spectrophotometer ID# _____ Wavelength _____nm Analyst _____

Working Standards (μg Pb/mL)	0.0	4.0	8.0	12.0	16.0	20.0
Absorbance 1, A_1						
Absorbance 2, A_2						
Q/C chk $(A_1 - A_2)/A_1$ ($\leq \pm 3\%$) (✓)						

____ Plot of calibration curve attached? ____ Curve $\leq \pm 0.003$ absorbance units of the origin?.

Note: If copper is present in high concentrations, use 283.3 nm to analyze the samples.

Sample ID#	Volume (mL)			Absorbance, A (OD)					Pb Conc, C_C (μg/mL)
	Loss, V_l	Smpl, V_s	Aliqt, V_a	A_1	A_2	A_3	Avg	Corr**	
Filter Blank									
0.1 N HNO_3 Blank									
Spiked Sample									
Unspiked Sample									
Cal Blank*									
Cal Standard*									

• Run these calibration checks (blank and standard) every 5 samples.
** Subtract filter and 0.1N HNO_3 blanks from average absorbance. Dilutions? _____

Matrix Check Spike:

$$C_s = C_a \frac{A_s}{A_t - A_s}$$

C_s = Pb concentration
C_a = Pb standard concentration, μg/mL = _____
A_s = Absorbance, unspiked sample
A_t = Absorbance, spiked sample

____ ***$C_s \leq \pm 0.05$ unspiked concentration?***

Note: *If the 5% specification is not met, run all samples using Method of Addition.*

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ___

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)

LABORATORY DATA SHEET 12a
Matrix Analysis

Client/Plant Name _____ Job # _____

Date/Time _____ Analyst _____

Note: This is a generic form for the Methods of Addition. Add the proper units. Make any adjustments as appropriate.

	Measurement Units					
Sample ID						
Spiked Sample, S						
Unspiked Sample, U						
Difference, D						
Standard, R						
%R = (D - R)/R						

QA/QC Check
Completeness ____ Legibility ____ Accuracy ____ Specifications____ Reasonableness ____

Checked by: _____ _____
Personnel (Signature/Date) Team Leader (Signature/Date)