SUMMARY SHEET 104
Beryllium

			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				
Abs Stack Pressure (P_b + P_g/13.6), in. Hg	P_s	SS 5				
Average Stack Temperature, °F	t_s	FDS 5				
Avg Abs Stack Temperature (460 + t_s), R	T_s	SS 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				
Isokinetic Sampling Rate, %	%I	SS 5				
Total Beryllium, μg	m_{Be}	LDS 104				
Beryllium Emission Rate, g/day	R	SS 104				

		Run #1	Run #2	Run #3	Avg
Post-test Calibration Checks					
Temperature and Barometer	CDS 2d				
Pressure Differential Gauges	CDS 2d				
Metering System	CDS 5				

$$R = 17.64 \frac{m_{Be}\, v_s\, A\, (86{,}400 \times 10^{-6})}{\left[V_{m(std)} + V_{w(std)}\right] \frac{T_s}{P_s}}$$

FIELD PROCEDURE 104
Beryllium

Note: *The field procedure is the same as that in Method 5 except as noted below. Follow the general procedure given in FP 5, except for the items noted below. Be is a hazardous substance; therefore, precautions must be taken to minimize exposure.* ***Use FDS 5.***

A. Preliminaries

1. Soak all glassware (probe, impingers, connections, sample recovery apparatus) in wash acid for 2 hr and rinse with water.
2. Omit the directions for filters, except check them visually against light for irregularities and flaws.
3. Select a nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
4. Select the sampling time (at least 2 hr) accurately determines the maximum emissions that occur in a 24-hr period. For cyclic operations, run sufficient sample runs to accurately represent the emissions over the cycle.

B. Preparation of Sampling Train

1. Assemble the train as shown in FP 5.
   a. Place 100 mL of water in each of the first two impingers, and leave the third impinger empty. Save a portion of the water for a blank analysis.
   b. Place ~200 g of preweighed silica gel in the fourth impinger.
   c. An empty impinger may be inserted between the third impinger and the silica gel to remove excess moisture.
2. Use a Viton A O-ring for the nozzle when stack temperatures are <500°F or a fiberglass string gasket when >500°F. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used.
3. If condensation occurs, use probe and filter heaters set at or above stack temperature to prevent condensation.
4. If temperature affects filter (e.g., Millipore AA is limited to ~225°F), move the filter holder downstream of first impinger if the stack gas is >~200°F.
5. Glassware can be reused for subsequent tests after rinsing twice with water. If not used within 2 days, repeat the initial acid wash procedure.

C. Sample Recovery

1. The cleanup area must be free of Be contamination.
2. <u>Container No. 1</u>. Place the filter and any loose particulate matter from the filter holder in this container.
3. <u>Container No. 2</u> (Impinger/Washings). In this container, place the following:
   a. Contents in the first three impingers. Measure and record volume (to the nearest 1 mL).
   b. Water and acetone (measure amounts of each) rinsings of the probe and all glassware between it and the back half of the third impinger. In cleaning the probe, use acetone and a brush or a long slender rod and cotton balls (include in container).
4. <u>Container No. 3</u> (Silica Gel)

   See FP 5, step E5.
5. <u>Blanks</u>

Save a portion of the water and acetone used in recovery for blank determinations.

LABORATORY PROCEDURE 104
Beryllium

A. Reagent Preparation

1. Hydrochloric Acid, 50%. Add one part HCl to one part water (used as acid wash).
2. Sulfuric Acid, 12 N. Dilute 33 mL conc. H_2SO_4 to 1 L with water.
3. HCl, 25%. Add one part HCl to three parts water.
4. Standard Beryllium Solution, (1 µg Be/mL). Dissolve 10 mg Be in 80 mL 12 N H_2SO_4, and dilute to 1 L with water. Dilute a 10-mL aliquot to 100 mL with 25% HCl. Prepare fresh daily. Equivalent strength Be stock solutions may be prepared from Be salts such as $BeCl_2$ and $Be(NO_3)_2$ (98% minimum purity).

B. Apparatus and Sample Preparation

1. Soak all glassware in wash acid for 2 hr and rinse with water.
2. <u>Container No. 1</u> (Filter)
   a. Transfer the filter and any loose particulate matter from the sample container to a 150-mL beaker.
   b. Add 35 mL conc. HNO_3. Heat on a hotplate until light brown fumes are evident (very important; otherwise, dangerous perchlorates may result from the subsequent $HClO_4$ digestion.
   c. Cool to room temperature, add 5 mL conc. H_2SO_4 and 5 mL conc. $HClO_4$ (only use $HClO_4$ under a hood).
3. <u>Container No. 2</u> (Impinger/Washes)
   a. Place a portion of the contents into a 150-mL beaker, and put on a hotplate. Add portions of the remainder as evaporation proceeds and evaporate to dryness.
   b. Cool the residue, and add 35 mL conc. HNO_3. Heat on a hotplate until light brown fumes are evident.
   c. Cool to room temperature, add 5 mL conc. H_2SO_4 and 5 mL conc. $HClO_4$ (under a hood).
4. <u>Container No. 3</u> (Silica Gel)

   Weigh the spent silica gel, and report to the nearest gram.
5. Combine the samples from Container Nos. 1 and 2 for ease of analysis.
   a. Place on a hotplate, and evaporate to dryness in a $HClO_4$ hood.
   b. Cool and dissolve the residue in 10.0 mL 25% HCl.
   c. If necessary, perform further dilution of sample with 25% HCl to bring within calibration range.

C. Analysis

1. Prepare the atomic absorption spectrophotometer according to the manufacturer's instruction.
2. Analyze the prepared samples at 234.8 nm using a nitrous oxide/acetylene flame. Use LDS 104.

D. Notes

1. Aluminum, silicon and other elements can interfere with this method if present in large quantities. To eliminate these interferences, see B. Fleet, et al., "A Study of Some Matrix Effects in the Determination of Beryllium by Atomic Absorption Spectroscopy in the Nitrous Oxide-Acetylene Flame," Talanta 17:203, 1970.
2. Method 104 has no directions for blanks. Treat a clean filter and the water and acetone blanks according to steps B2 and B3, respectively.

LABORATORY DATA SHEET 104
Beryllium

Client/Plant Name ______________________________ Job # ______________ Date ____________

Spectrophotometer ID# ______________________ Date of Last Calibration ______________ (≤6 months?)

Wavelength (234.8 nm?) ______ Temp. of optical cell ______ °F Analyst ______________________

Working Stds (μg/mL)	Peak Height (H) 1	2	Avg.	H (Blk corr)	C_{Be} (____Be)
0.0					0.0

Note: Repeat each standard until two consecutive peaks agree within 3% of their average value.

Plot calibration curve [H_{avg} (corr) vs. C_{Be}. Best fit straight line must pass through origin ±2% of F.S.

Sample ID#	Vol. Loss, (mL)	Sample Vol., V_f (mL)	Dilution Factor, D.F.	Aliquot Vol., S (mL)	Peak Height, H 1	2	Avg.	H Blk corr	C_{Be} blk corr (___)	m_{Be} (___)
Blank										
Standard										

m_{Be} = μg in the original solution:

$$m_{Be} = \frac{C_{Be}\,(D.F.)\,V_f\,10^{-3}}{S}$$

___ All solutions at room temperature before analysis?

___ Peak maximum of an aliquot greater than 10% of the recorder full scale?

___ A blank and standard run after every 5 samples?

___ One sample checked by the method of standard additions? (Attach LDS).

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: ______________________________ ______________________________

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