SUMMARY SHEET 105
Mercury

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 105				
Job No.		FDS 105				
Sample ID #		FDS 105				
Test Date		FDS 105				
Run Start Time		FDS 105				
Run Finish Time		FDS 105				
Sample Time Increment	hr	FDS 105				
Sample Volume per Grab Sample	L	FDS 105				
Solids Content of Blended Sludge	F_{sb}	LDS 105				
Solids Content of Sludge Before Blending	F_{sm}	LDS 105				
Weight Wet Blended Sample, g	S_{wb}	LDS 105				
Digested Sample Volume, mL	V_s	LDS 105				
Digested Aliquot Volume, mL	V_a	LDS 105				
Mass of Hg in Aliquot, μg	m	LDS 101				
Conc. of Hg in Digested Sample, μg/g	C_m	SS 105				
Avg of Three 8-Hr Samples, μg/g	$C_{m(avg)}$	SS 105				
Concentration of Hg, dry, μg/g	M	SS 105				

$$C_m = \frac{m V_s}{V_a S_{wb}}$$

$$M = \frac{C_{m(avg)}}{F_{sb}}$$

FIELD PROCEDURE 105
Mercury in Wastewater Treatment Plant Sewage Sludge

Sampling

Withdraw equal volume increments of sludge, for a total of at least 15 L, at intervals of 30 minutes, over an 8-hr period. Place samples in a rigid plastic container.

FIELD DATA SHEET 105
Mercury

Client/Plant Name _____ Job # _____

City/State _____ Personnel _____

Start Time _____ Stop Time _____ Date _____

Sample ID _____		Sample ID _____		Sample ID _____	
Time (hr)	Sample Vol (L)	Time (hr)	Sample Vol (L)	Time (hr)	Sample Vol (L)
0:00		0:00		0:00	
0:30		0:30		0:30	
1:00		1:00		1:00	
1:30		1:30		1:30	
2:00		2:00		2:00	
2:30		2:30		2:30	
3:00		3:00		3:00	
3:30		3:30		3:30	
4:00		4:00		4:00	
4:30		4:30		4:30	
5:00		5:00		5:00	
5:30		5:30		5:30	
6:00		6:00		6:00	
6:30		6:30		6:30	
7:00		7:00		7:00	
7:30		7:30		7:30	
8:00		8:00		8:00	
Total Vol		Total Vol		Total Vol	

___ Volume of sludge equal increments? ___ Sample containers sealed and labeled?

___ Total volume sample ≥ 15 L?

Comments: _____

_____

_____

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

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

LABORATORY PROCEDURE 105
Mercury in Wastewater Treatment Plant Sewage Sludge

Note: This laboratory procedure is similar to LP 101A, except for the variations below. Use LDS 105.

A. Reagents

1. Aqua Regia. Carefully add one volume conc. HNO_3 to three volumes conc. HCl. Prepare immediately before use.
2. Mercury (II) Stock Solution, 1 mg Hg/mL. Stable for at least one month. Dissolve 135.4 mg ACS reagent-grade $HgCl_2$ in 75 mL water, add 10 mL conc. HNO_3, and adjust the volume to 100.0 mL with water. Mix thoroughly.
3. Nitric Acid, 15%. Dilute 15 mL conc. HNO_3 to 100 mL with water.
4. Intermediate Mercury Standard Solution, 10 μg Hg/mL. Prepare fresh weekly. Pipet 5.0 mL Hg stock solution into a 500-mL volumetric flask, and add 20 mL 15% HNO_3 solution. Adjust the volume to 500 mL with water. Mix thoroughly.
5. Working Mercury Standard Solution, 200 ng Hg/mL. Pipet 5.0 mL "Intermediate Mercury Standard Solution" into a 250-mL volumetric flask. Add 20 mL 15% HNO_3, and adjust the volume to 250 mL with water. Mix thoroughly. Prepare fresh daily.
6. Tin (II) Solution. Dissolve 20 g tin (II) chloride [or 25 g tin (II) sulfate] crystals in 25 mL conc. HCl (do not use other acids for HCl). Dilute to 250 mL with water. Prepare fresh daily, and keep sealed.
7. Sodium Chloride-Hydroxylamine Solution. Dissolve 12 g NaCl and 12 hydroxylamine sulfate (or 12 g hydroxylamine hydrochloride) in water; dilute to 100 mL.
8. Potassium Permanganate, 5%. Dissolve 5 g $KMnO_4$ in water; dilute to 100 mL.

B. Sample Preparation

1. Sludge Mixing
   a. Transfer the entire 15-L sample to a 57-L capacity mortar mixer. Mix the sample for ≥30 min at 30 rpm.
   b. Using a 200-mL beaker, take six 100-mL portions of sludge, and combine in a 2-L blender. Blend the sludge for 5 min; add water as necessary to give a fluid consistency.
   c. Immediately after stopping the blender, use a 50-mL beaker to withdraw four 20-mL portions of blended sludge and place them in separate, tared 125-mL Erlenmeyer flasks.
   c. Reweigh each flask to determine the exact amount of sludge added.
2. Solids Content of Blended Sludge
   a. Dry one of the 20-mL blended samples from step B1c in an oven at 105°C to constant weight.
   b. Cool in a desiccator between weighings; weigh the dry sample.
3. Aqua Regia Digestion of Blended Sludge
   a. To each of the three remaining 20-mL samples from step B1c, add 25 mL aqua regia, and digest the samples on a hot plate at low heat (do not boil) for 30 min, or until samples are a pale yellow-brown color and are void of the dark brown color characteristic of organic matter. Remove from the hot plate, and allow to cool.
   b. Filter each digested sample separately through an S and S No. 588 filter, or equivalent, and rinse the filter contents with 50 mL water.
   c. Transfer the filtrate and filter washing to a 100-mL volumetric flask, and carefully dilute to volume with water.
4. Solids Content of Sludge Before Blending
   a. Using a 200-mL beaker, remove two 100-mL portions of mixed sludge from the mortar mixer, and place in separate, tared 400-mL beakers.
   b. Reweigh each beaker to determine the exact amount of sludge added. Dry in an oven at 105°C, and cool in a desiccator to constant weight.

C. Equipment Preparation

This is the same as that in Method 101A, section C, except calibrate the ***spectrophotometer and recorder*** as follows:

1. Set the spectrophotometer wavelength to 253.7 nm.
2. Make certain the optical cell is at the minimum temperature that will prevent water condensation from occurring.
3. **First** add 25 mL water and 3 drops Antifoam B to the aeration-cell bottle. **Then** pipet 5.0 mL working Hg standard solution (or any Hg-containing solution) into the aeration cell. Never switch the order.

4. Place a Teflon-coated stirring bar in the bottle. Add 5 mL 15% HNO_3 and 5 mL 5% $KMnO_4$ to the aeration bottle, and mix well.

5. Attach the bottle section to the bubbler section of the aeration cell, close stopcock on the aeration cell exit arm, and ensure there is no flow through the bubbler.

6. Add 5 mL sodium chloride-hydroxylamine solution to the aeration bottle and mix. If the solution does not become colorless, add sodium chloride-hydroxylamine solution in 1-mL increments until the solution is colorless.

7. Add 5 mL tin (II) solution to the aeration bottle through the side-arm, and immediately stopper the side arm. Stir the solution for 15 sec, turn on the recorder, open the aeration cell exit arm stopcock, and immediately initiate aeration with continued stirring.

8. Determine the maximum absorbance of the standard, and set this value to read 90% of the recorder full scale.

LABORATORY DATA SHEET 105
Mercury

Client/Plant Name _____ Job # _____ Date_____

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

Analyst _____

Note: *Use **LDS 101** for the analysis of the digested blended sludge and this data sheet for solids content of the sewage sludge samples.*

Solids Content of Blended Sludge

Sample ID#										
Wgt Flask, W_f (g)										
Wgt Flask + Smpl, W_{fs} (g)										
Wgt Flask + Smpl Dried, W_{fd} (g)										
Water, $W_{wb} = W_{fs} - W_{fd}$ (g)										
Wet Smpl, $S_{wb} = W_{fs} - W_f$ (g)										
Solids Content, $F_{sb} = 1 - W_w/S_w$										

Note: *The digested blended sludge sample volume (V_s) is 100 mL (denoted as V_f in LDS 101). The aliquot volume (V_a) is denoted as S in LDS 101. Therefore, for LP 105, make changes accordingly.*

Solids Content of Sludge Before Blending

Sample ID				
Wgt Beaker, W_b (g)				
Wgt Beaker + Smpl, W_{bs} (g)				
Wgt Dried Beaker + Smpl, W_{bd} (g)				
Water, $W_w = W_{bs} - W_{bd}$ (g)				
Wet Sampl, $S_w = W_{bs} - W_b$ (g)				
Solids Content, $F_{sm} = 1 - W_w/S_w$				

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

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