

SUMMARY SHEET 108
Arsenic

			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 ₂ O	ΔH	FDS 5				
Barometric Pressure, in. Hg	P_b	FDS 5				
Stack Static Pressure, in. H ₂ O	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 ($t_s + 460$), 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 ₂ O	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				
Total Arsenic Mass, μg	m_t	SS 108				
Stack Conc. of Arsenic, g/dscm	C_s	SS 108				
Arsenic Mass Emission Rate, g/hr	E_a	SS 108				
Audit Relative Error, %	RE	QA 1				

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

CDS 2d
CDS 2d
CDS 5

$$C_s = 10^{-6} \left[\frac{m_t}{V_{m(std)}} \right]$$

$$E_a = C_s Q_{sd}$$

FIELD PROCEDURE 108
Particulate and Gaseous Arsenic Emissions

Note: The sampling procedure is similar to that of Method 5. Therefore, follow the general procedure given in FP 5, except for the items noted below (Use FDS 5):

A. Preliminaries

1. The filter does not need to be weighed or be unreactive to SO₂ or SO₃.
2. Select a nozzle size to maintain isokinetic sampling rates below 1.0 cfm.
3. Assemble the train as shown in FP5.

B. Sampling

1. Maintain 225 to 275°F around the filter.
2. Maintain isokinetic sampling below 1.0 cfm.

C. Sample Recovery

Recover the samples as in FP 5, Containers Nos. 1 through 3, except use 0.1 N NaOH for the cleanup solvent instead of acetone. Treat the impinger water as follows:

1. Label the impinger water sample container as Container No. 4 (Impinger Water).
2. Clean each of the first two impingers and connecting glassware in the following manner:
 - a. Wipe the impinger ball joints free of silicone grease, and cap the joints.
 - b. Weigh the impinger and liquid to ±0.5 g (for moisture determination). Note any color or film observed in the impinger catch.

- c. Rotate and agitate each impinger, using the impinger contents as a rinse solution.
 - d. Transfer the liquid to Container No. 4. Remove the outlet ball-joint cap, and drain the contents through this opening without separating the impinger parts.
 - e. *[Note: In step C2e and in step C2f below, measure and record the total amount 0.1 N NaOH used for rinsing.]* Pour about 30 mL of 0.1 NaOH into each of the first two impingers, and agitate. Drain through the outlet of each impinger into Container No. 4. Repeat the operation. Inspect the impingers for any abnormal conditions.
 - f. Rinse each piece of connecting glassware and the back half of the filter holder twice with 0.1 N NaOH; transfer to Container No. 4. *[Do not rinse or brush the glass-fritted filter support.]*
 - g. Mark the height of the fluid level. Label the container.
3. For a blank, take 200 mL 0.1 N NaOH solution directly from the wash bottle being used and place it in a plastic sample container labeled "NaOH blank."
 4. Save a sample of the water, and place it in a container labeled "H₂O blank."

LABORATORY PROCEDURE 108
Particulate and Gaseous Arsenic Emissions

A. Reagent Preparation

1. Sodium Hydroxide, 0.1 N. Dissolve 4.00 g NaOH in ~500 mL water in a 1 L volumetric flask. Dilute to 1.0 L with water.
2. Sodium Borohydride, 5%. Dissolve 5.00 g NaBH₄ in ~500 mL 0.1 N NaOH in a 1-L volumetric flask. Dilute to 1.0 L with 0.1 N NaOH.
3. Potassium Iodide, 30%. Dissolve 300 g KI in 500 mL water in a 1-L volumetric flask. Dilute to 1.0 L with water.
4. Nitric Acid, 0.8 N. Dilute 52 mL conc. HNO₃ to 1.0 L with water.
5. Nitric Acid, 50%. Add 50 mL conc. HNO₃ to 50 mL water.
6. Stock Arsenic Standard, 1 mg/mL. Dissolve 1.3203 g primary standard grade As₂O₃ in 20 mL 0.1 N NaOH in a 150-mL beaker. Slowly add 30 mL conc. HNO₃. Heat the resulting solution and evaporate just to dryness. Transfer the residue quantitatively to a 1 L volumetric flask. Dilute to 1.0 L with water.
7. Working Arsenic Solution, 1.0 µg As/mL. Pipet 1.0 mL stock arsenic standard into an acid-cleaned, 1 L volumetric flask containing ~500 mL water and 5 mL conc. HNO₃. Dilute to 1.0 L with water.
8. Nickel Nitrate, 5%. Dissolve 24.780 g nickel nitrate hexahydrate in water in a 100-mL volumetric flask. Dilute to 100 mL with water.
9. Nickel Nitrate, 1%. Pipet 20 mL 5% nickel nitrate solution into a 100-mL volumetric flask. Dilute to 100 mL with water.
10. Hydrogen Peroxide, 3%. Pipet 50 mL 30% H₂O₂ into a 500 mL volumetric flask. Dilute to 500 mL with water.
11. QA Audit Samples. Obtain from EPA (see QA 1).

B. Sample Preparation

1. Note the level of liquid in Sample Container Nos. 2 and 4, and determine loss; note this loss, if any, on the laboratory data sheet.
2. **Container No. 2**
 - a. Using a glass fiber filter, filter the contents into a 200-mL volumetric flask. Combine the filtered material with the contents of Container No. 1.
- b. Dilute the filtrate to 200 mL with water. Pipet 50 mL into a 150-mL beaker. Add 10 mL conc. HNO₃, bring to a boil, and evaporate to dryness.
- c. Allow to cool, add 5 mL 50% HNO₃, and then warm and stir.
- d. Allow to cool, transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.

3. Container No. 1

- a. Place the filter and loose particulate matter in a 150-mL beaker. Add the filtered material from Container No. 2.
- b. Add 50 mL 0.1 N NaOH. Stir and warm on a hot plate at low heat (do not boil) for ~15 minutes.
- c. Add 10 mL conc. HNO₃, bring to a boil, then simmer for ~15 min.
- d. Filter the solution through a glass fiber filter. Wash with hot water, and catch the filtrate in a clean 150-mL beaker.
- e. Boil the filtrate, and evaporate to dryness.
- f. Cool, add 5 mL 50% HNO₃, then warm and stir.
- g. Allow to cool. Transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.

4. Container No. 4

- a. Transfer the contents to a 500-mL volumetric flask. Dilute to 500 mL with water.
- b. Pipet 50 mL of the solution into a 150-mL beaker.
- c. Add 10 mL conc. HNO₃, bring to a boil, and evaporate to dryness.
- d. Allow to cool, add 5 mL 50% HNO₃, and then warm and stir.
- e. Allow the solution to cool, transfer to a 50-mL volumetric flask, dilute to volume with water, and mix well.

5. Blanks

- a. Take two filters from each lot of filters used in the sampling. Cut each filter into strips, and treat each filter individually as directed in section B3, beginning with step B3b.
- b. Treat separately 50 mL 0.1 N NaOH and 50 mL water, as directed in section B4, beginning with step b.

C. Calibration

1. Prepare and operate the spectrophotometer according to the manufacturers' instruction manual. The lower limit of flame atomic absorption spectrophotometry is $10\ \mu\text{g As/mL}$. If the arsenic concentration of any sample is $<10\ \mu\text{g/mL}$, use the graphite furnace or vapor generator (either may also be used for sample concentrations up to $30\ \mu\text{g/mL}$).
2. Prepare the standards as follows:
 - a. High Level Procedure. Pipet 1, 3, 5, 8, and 10 mL of the 1.0-mg As/mL stock solution into separate 100-mL volumetric flasks, each containing 5 mL conc. HNO_3 .
 - b. Low Level Vapor Generator Procedure. Pipet 1, 2, 3, and 5 mL of $1.0\ \mu\text{g As/mL}$ standard solution into the separate 100-mL reaction tubes.
 - c. Low Level Graphite Furnace Procedure. Pipet 1, 5, 10, and 15 mL of $1.0\ \mu\text{g As/mL}$ standard solution into the separate 100-mL flasks along with 2 mL 5% nickel nitrate solution and 10 mL 3% H_2O_2 solution.
3. Dilute to the mark with water. Then treat the standards in the same manner as the samples as in section D.
4. Prepare a standard curve of absorbance versus concentration. [*Note:* For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.]

D. Analysis

1. Measure absorbance of standards, blanks, and samples against 0.8 N HNO_3 . If the sample concentration falls outside the range of the calibration curve, make an appropriate dilution with 0.8 N HNO_3 .
2. Using the appropriate standard curve, determine the arsenic concentration in each sample fraction and blank. For the arsenic concentration in the filter blank, use the average of the two blank values from each lot.

3. Vapor Generator Procedure

- a. If necessary, screen the samples by conventional atomic absorption to determine the approximate concentration.
- b. Place a sample containing between 0 and $5\ \mu\text{g}$ arsenic in the reaction tube, dilute to 15 mL with water.
- c. Pipet 15 mL conc. HCl into each tube. Add 1 mL 30% KI solution. Place the reaction tube into a 50°C water bath for 5 min.
- d. Cool to room temperature. Connect the reaction tube to the vapor generator assembly. When the instrument response has returned to baseline, inject 5.0 mL 5% NaBH_4 , and integrate the resulting spectrophotometer signal over a 30-sec time period.

4. Graphite Furnace Procedure

- a. Dilute the digested sample so that a 5-mL aliquot contains $<1.5\ \mu\text{g}$ of arsenic.
 - b. Pipet 5 mL of this digested solution into a 10-mL volumetric flask. Add 1 mL 1% nickel nitrate solution, 0.5 mL 50% HNO_3 , and 1 mL 3% H_2O_2 , and dilute to 10 mL with water.
 - c. Inject the sample in the furnace for analysis.
5. Check absorbance of standards frequently against 0.8 N HNO_3 (reagent blank) during the analysis to ensure that base-line drift has not occurred.
 6. **Mandatory:** Check for matrix effects on the arsenic results (see LP 12, section D).
 7. Weigh the silica gel contents of Container No. 3 (see FP 5, step E5).
 8. Analyze the audit samples, if applicable.

LABORATORY DATA SHEET 108
Arsenic

Method (circle) 108, 108A, 108B

Client/Plant Name _____ Job # _____

City/State _____ Date Last Calibration _____

Spectrophotometer ID # _____ Date _____ Analyst _____

Std Vol (mL)	Std Conc ()	Absorbance
		108: High Level Std Vol = 0.0, 1.0, 3.0, 5.0, 8.0, 10.0 mL Std Conc = 0, 10, 30, 50, 80, 100 µg/mL
		108: Vapor Generator = 0.0, 1.0, 2.0, 3.0, 5.0 mL Std Conc = 0, 1.0, 2.0, 3.0, 5.0 µg
		108: Graphite Furnace = 0.0, 1.0, 5.0, 10.0, 15.0 mL Std Conc = 0, 10, 50, 100, 150 ng/mL
		108A: Std Vol = 0.0, 1.0, 5.0, 10.0, 25.0 mL Std Conc = 0, 10, 50, 100, 250 µg/mL
		108B: Std Vol = 0.0, 1.0, 5.0, 10.0, 25.0 mL Std Conc = 0, 10, 50, 100, 250 µg/mL

Plot Absorbance vs. Concentration and attach graph (not necessary for direct readout instruments).

Sample Number	Sample ID #	Sample Volume, V _n (mL)	Dilution Factor, F _d	Absorbance	Concentration, C _a (µg/mL)	Mass, m (µg)
	0.8N HNO ₃					
	Filter blank					
	Filter blank					
	Reagent blank					
	Audit #1					
	Audit #2					
	Ore Sample Wgt, W (mg)					

$m_n = C_a F_d V_n$ $m_t = m_n(\text{filters}) + m_n(\text{probe}) + m_n(\text{impingers}) - m_n(\text{filter blank}) - m_n(\text{NaOH}) - m_n(\text{H}_2\text{O}) = \text{---} \mu\text{g}$

Matrix effects checked? (Attach LDS.) _____ Baseline drift checked? _____

QA/QC Check
 Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

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

SUMMARY SHEET 108A
Arsenic

Method (circle) 108A 108B

		Run #1	Run #2	Run #3	Avg
Client/Plant Name					
Job No.					
Run ID #					
Test Date					
Weight of Ore Sample, mg	W				
Dilution Factor	F _d				
Sample Conc. of Arsenic, µg/mL	C _a				
Arsenic in Ore, %	%As				
Audit Relative Error, %	RE				QA 1

$$\%As = \frac{5 C_a F_d}{W}$$

LABORATORY PROCEDURE 108A
Inorganic Arsenic

Note: Use LDS 108.

A. Reagent Preparation

The reagents, 0.1 N NaOH (prepare half the amount), 5% sodium borohydride, 5% nickel nitrate, 1 mg As/mL stock arsenic standard (except rather than evaporating just to dryness, heat in an oven at 105°C for 2 hr), and QA audit samples, are the same as that in Method 108. In addition, prepare the following:

1. Nitric Acid, 0.5 N. Add 32 mL conc. HNO₃ to a 1-L volumetric flask with water, dilute to volume with water.
2. Potassium Chloride Solution, 10%. Dissolve 10 g KCl in water, add 3 mL conc. HNO₃, and dilute to 100 mL.
3. Standard Arsenic Solutions. Pipet 1, 5, 10, and 25 mL stock As solution into separate 100-mL volumetric flasks. Add 10 mL KCl solution and dilute to the mark with 0.5 N HNO₃ to obtain 10, 50, 100, and 250 µg As/mL.

B. Sample Preparation

1. Obtain a sample that is representative of the ore lot (representative samples routinely collected for metals analysis may be used). Grind the sample to a finely pulverized state.

2. Weigh 50 to 500 mg of finely pulverized sample to the nearest 0.1 mg.
3. Transfer the sample into the Teflon cup of the digestion bomb. Add 2 mL each of conc. HNO₃ and HF. Seal the bomb immediately to prevent the loss of any volatile arsenic compounds that may form.
3. Heat in an oven 105°C for 2 hr. Remove from the oven and cool.
4. Using a Teflon filter, quantitatively filter the digested sample into a 50-mL polypropylene volumetric flask.
5. Rinse the bomb three times with small portions of 0.5 N HNO₃, filter the rinses into the flask, add 5 mL 10% KCl solution to the flask, and dilute to 50 mL with 0.5 N HNO₃.

C. Analysis

1. Dilute 10 mL 10% KCl solution to 100 mL with 0.5 N HNO₃ and use this as a reagent blank.
2. Analyze the samples as in FP 108, except use the reagent in step C1 of this procedure as the reagent blank and make appropriate dilutions with 0.5 N HNO₃.

LABORATORY PROCEDURE 108B
Arsenic Content in Ore Samples from Nonferrous Smelters

A. Reagents and Spectrophotometer Preparation

1. Prepare the spectrophotometer as in LP 108, section C.
2. Prepare stock arsenic standard (1.0 mg As/mL) as follows:
 - a. Dry some primary grade As_2O_3 at 105°C .
 - b. Dissolve 1.3203 g in a 400-mL beaker with 10 mL HNO_3 and 5 mL HCl. Cover with a watch glass and heat gently until dissolution is complete.
 - c. Add 10 mL HNO_3 and 25 mL HClO_4 , evaporate to strong fumes of HClO_4 and reduce to about 20 mL.
 - d. Cool, add 100 mL of water and 100 mL HCl, and transfer quantitatively to a 1 L volumetric flask. Dilute to volume with water and mix.
3. Prepare standard solutions as follows:
 - a. Pipet 1, 5, 10, and 25 mL stock As solution into separate 100-mL flasks.
 - b. Add 2 mL HClO_4 , 10 mL HCl, and dilute to the mark with water to obtain 10, 50, 100, and 250 μg As/mL. For lower level arsenic samples, use Method 108C.
4. Measure the standard absorbances against the reagent blank. Check these absorbances frequently against the blank during the analysis to ensure that baseline drift has not occurred.
5. Prepare a standard curve of absorbance versus concentration. (*Note:* For instruments equipped with direct concentration readout devices, preparation of a standard curve will not be necessary.) In all cases, follow calibration and operational procedures in the manufacturer's instruction manual. Maintain a laboratory log of all calibrations.
6. Obtain QA Audit Samples. See QA 1.

B. Sample Preparation

1. Weigh 100 to 1000 mg of finely pulverized sample to the nearest 0.1 mg. Transfer the sample to a 150-mL Teflon beaker.
2. Dissolve the sample by adding (in this order) 15 mL HNO_3 , 10 mL HCl, 10 mL HF, and 10 mL HClO_4 , and let stand for 10 min.
3. In a HClO_4 fume hood, heat on a hot plate until 2-3 mL HClO_4 remain, then cool. Add 20 mL water and 10 mL HCl. Cover and warm until the soluble salts are in solution. Cool, and transfer quantitatively to a 100-mL volumetric flask. Dilute to the mark with water.

C. Analysis

1. Determine the absorbance of each sample using the blank as a reference.
2. If the sample concentration falls outside the range of the calibration curve, appropriately dilute with 2% HClO_4 /10% HCl (prepared by diluting 2 mL conc. HClO_4 and 10 mL conc. HCl to 100 mL with water).
3. Determine the As concentration in each sample from the calibration curve.
4. **Mandatory:** Check for matrix effects according to LP 12, section D.
5. If applicable, analyze the audit samples.

LABORATORY PROCEDURE 108C
Arsenic Content in Ore Samples from Nonferrous Smelters

Note: This method is applicable to samples having an analytical concentration less than 10 µg As/mL.

A. Reagent Preparation

1. Dilute Hydrochloric Acid. Add one part conc. HCl to nine parts water.
2. Ammonium Molybdate Solution, 5 g/L. Dissolve 0.5 g $(\text{NH}_4)_6\text{Mo}_7\text{O}_{24}\cdot 4\text{H}_2\text{O}$ in water in a 100-mL volumetric flask, and dilute to the mark. Use freshly-prepared.
3. Standard Arsenic Solution, 10 µg As/mL. Dissolve 0.1320 g of As_2O_3 in 100 mL HCl in a 1-L volumetric flask. Add 200 mL water, cool, dilute to the mark with water, and mix. Transfer 100 mL of this solution to a 1-L volumetric flask, add 40 mL HCl, cool, dilute to the mark, and mix.
4. Hydrazine Sulfate Solution, 1 g/L. Dissolve 0.1 g $(\text{NH}_2)_2\cdot\text{H}_2\text{SO}_4$ in water, and dilute to 100 mL in a volumetric flask. Use freshly-prepared.
5. Potassium Bromate (KBrO_3) Solution, 0.03%. Dissolve 0.3 g KBrO_3 in water, and dilute to 1 L with water.
6. 1:1 HCl:Water. Slowly add one part conc. HCl to one part water.
7. Obtain QA audit samples, if applicable (see QA 1).
6. Distill until the vapor in the flask reaches 107°C. When distillation is complete, remove the flask from the hot plate, and simultaneously wash down the side arm with water as it is removed from the cylinder.
7. If the expected arsenic content is from 0.0020 to 0.10%,
 - a. Dilute the distillate to the 50-mL mark of the cylinder with water, stopper, and mix.
 - b. Transfer a 5.0-mL aliquot to a 50-mL volumetric flask. Add 10 mL water and a boiling granule. Place the flask on a hot plate, and heat gently until the bromine is expelled and the color of methyl orange indicator persists upon the addition of 1-2 drops. Cool the flask to room temperature.
 - c. Neutralize just to the yellow color of the indicator with dropwise additions of NH_4OH . Bring back to the red color by dropwise addition of dilute HCl, and add 10 mL excess.
8. If the expected arsenic content is from 0.0002 to 0.0010%,
 - a. Transfer either the entire initial distillate or the measured remaining distillate from above to a 250-mL beaker. Wash the cylinder with two successive portions of conc. HNO_3 , adding each portion to the distillate in the beaker.
 - b. Add 4 mL conc. HClO_4 , a boiling granule, and cover with a flat watch glass placed slightly to one side. Boil gently on a hot plate until the volume is reduced to about 10 mL.
 - c. Add 3 mL HNO_3 , and continue the evaporation until HClO_4 is refluxing on the beaker cover. Cool briefly, rinse the underside of the watch glass and the inside of the beaker with about 3-5 mL water, cover, and continue the evaporation to expel all but 2 mL of the HClO_4 .

B. Sample Preparation

1. Weigh 1.0 g of finely pulverized sample to the nearest 0.1 mg.
2. Transfer the sample to a 300-mL Erlenmeyer flask and add (in this order) 15 mL HNO_3 , 4 mL HCl, 2 mL HF, 3 mL HClO_4 , and 15 mL H_2SO_4 .
3. In a HClO_4 fume hood, heat on a hot plate to decompose the sample. Then heat while swirling over an open flame until dense, white fumes evolve.
4. Cool, add 15 mL water, swirl to hydrate the H_2SO_4 completely, and add several boiling granules. Cool to room temperature.
5. Add 1 g KBr, 1 g hydrazine sulfate, and 50 mL HCl. Immediately attach the distillation head with thermometer and dip the side arm into a 50-mL graduated cylinder containing 25 mL water and 2 mL bromine water. Keep the graduated cylinder immersed in a beaker of cold water during distillation.

- d. **Note:** If the solution appears cloudy due to a small amount of antimony distilling over, add 4 mL 1:1 HCl:water and 5 mL water, cover, and warm gently until clear. If cloudiness persists, add 5 mL HNO₃ and 2 mL H₂SO₄. Continue the evaporation of volatile acids to solubilize the antimony until dense white fumes of H₂SO₄ appear. Retain at least 1 mL of the H₂SO₄.
- e. To the 2 mL HClO₄ solution or 1 mL H₂SO₄ solution, add 15 mL water, boil gently for 2 min, and then cool.
- f. Proceed with the molybdenum blue color development by neutralizing the solution directly in the beaker just to the yellow indicator color by dropwise addition of NH₄OH. Just bring back the red color by dropwise addition of dilute HCl.
- g. Transfer the solution to a 50-mL volumetric flask, and rinse the beaker successively with 10 mL dilute HCl, followed by several small portions of water. At this point the volume of solution in the flask should ≤ 40 mL.

C Calibration

1. Transfer 1.0, 2.0, 4.0, 8.0, 12.0, 16.0, and 20.0 mL of standard arsenic solution (10 $\mu\text{g}/\text{mL}$) to each of seven 50-mL volumetric flasks. Dilute to 20 mL with dilute HCl.
2. Add one drop of methyl orange solution and neutralize to the yellow color with dropwise addition of NH₄OH. Just bring back to the red color by dropwise addition of dilute HCl, and add 10 mL in excess.

3. Proceed with the color development as described in section D.. Plot the photometric readings of the calibration solutions against $\mu\text{g As per 50 mL}$ of solution. From the curve, determine the As concentration in each sample.

D. Analysis

1. Add 1 mL KBrO₃ solution to the flask and heat on a low-temperature hot plate to about 50°C to oxidize the arsenic and methyl orange.
2. Add 5.0 mL ammonium molybdate solution to the warm solution and mix. Add 2.0 mL of hydrazine sulfate solution, dilute until the solution comes within the neck of the flask, and mix.
3. Place in a 400-mL beaker, 80% full of boiling water, for 10 min. Supply enough heat to prevent the water bath from cooling much below the boiling point upon inserting the volumetric flask. Remove the flask, cool to room temperature, dilute to the mark, and mix.
4. Transfer a suitable portion of the reference solution to an absorption cell, and adjust the photometer to the initial setting, using a light band centered at 660 nm. While maintaining this photometer adjustment, take the photometric readings of the calibration solutions followed by the samples.
5. If applicable, analyze the audit samples.

