SUMMARY SHEET 11
Hydrogen Sulfide

			Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 11				
Job No.		FDS 11				
Sampling Location		FDS 11				
Run ID #		FDS 11				
Test Date		FDS 11				
Run Start Time		FDS 11				
Run Finish Time		FDS 11				
Net Traverse Points		FDS 1				
Traverse Matrix (if rectangular)		FDS 1				
Net Run Time, min	θ	FDS 11				
Barometric Pressure, mm Hg	P_b	FDS 11				
DGM Calibration Factor	Y	CDS 6				
DGM Temperature, °C	t_m	FDS 11				
DGM Sample Volume, L	V_m	FDS 11				
DGM Sample Volume, L	$V_{m(std)}$	SS 11				
<u>Sample</u>						
Normality, Standard Iodine	N_I	LDS 11				
Volume Titrated, 50 mL	V_{IT}	LDS 11				
Normality, Standard Thiosulfate	N_T	LDS 11				
Volume Titrant, mL	V_{TT}	LDS 11				
<u>Blank</u>						
Normality, Standard Iodine	N_I	LDS 11				
Volume Titrated, 50 mL	V_{IT}	LDS 11				
Normality, Standard Thiosulfate	N_T	LDS 11				
Volume Titrant, mL	V_{TT}	LDS 11				
H_2S Concentration, mg/dscm	C_{H2S}	SS 11				
Post-test Calibration Checks						
Temperature		CDS 2d				
Barometer		CDS 2d				
Metering System		CDS 6				

$$V_{m(std)} = 0.3858\ Y\ \frac{V_m\ P_b}{(273 + t_m)}$$

$$C_{H_2S} = 17.04 \times 10^3\ \frac{[V_{IT}N_I - V_{TT}N_T]_{sample} - [V_{IT}N_I - V_{TT}N_T]_{blank}}{V_{m(std)}}$$

FIELD PROCEDURE 11
Hydrogen Sulfide of Fuel Gas Streams in Petroleum Refineries

A. Sampling Preparation

1. Assemble the sampling train as shown in Figure F11-1.
   a. Place 15 mL of 3% H_2O_2 solution in the first impinger.
   b. Leave the second impinger empty.
   c. Place 15 mL of the $CdSO_4$ solution in the third, fourth, and fifth impingers.
   d. Place the impinger assembly in an ice bath container, and place crushed ice around the impingers. Add more ice during the run, if needed.
2. ***Optional:*** Leak-check the sampling train as follows:
   a. Connect the rubber bulb and manometer to the first impinger, as shown in Figure F11-1. Close the petcock on the DGM outlet.
   b. Pressurize the train to 10 in. H_2O with the bulb, and close off the tubing connected to the rubber bulb.
   c. Time pressure drop (must be ≤0.4-in. drop in pressure in 1 min).

B. Sampling

1. Purge the connecting line between the sampling valve and the first impinger as follows:
   a. Disconnect the line from the first impinger, and open the sampling valve.
   b. Allow process gas to flow through the line for 1 to 2 min. Close the sampling valve, and reconnect the line to the impinger train.
2. Open the petcock on the dry gas meter (DGM) outlet. Record the initial DGM reading and the barometric pressure.
3. Open the sampling valve, and then adjust the valve to obtain about 1 L/min. Maintain a constant (±10%) flow rate during the test.
4. Sample for at least 10 min. Take DGM and temperature readings at least every 5 min.
5. At the end of the sampling time, close the sampling valve, and record the final DGM volume and temperature readings.
6. ***Mandatory:*** Leak-check the train (see A2).
7. Disconnect the impinger train from the sampling line, and connect the charcoal tube and the pump, as shown in Figure F11-1.
8. Purge the train at 1 L/min with clean ambient air for 15 min.
9. After purging, cap the open ends, and remove the impinger train to a clean, well-lighted area that is away from sources of heat or direct sunlight.

C. Sample Recovery

Because analysis must immediately follow sample recovery, see LP 11 for sample recovery.

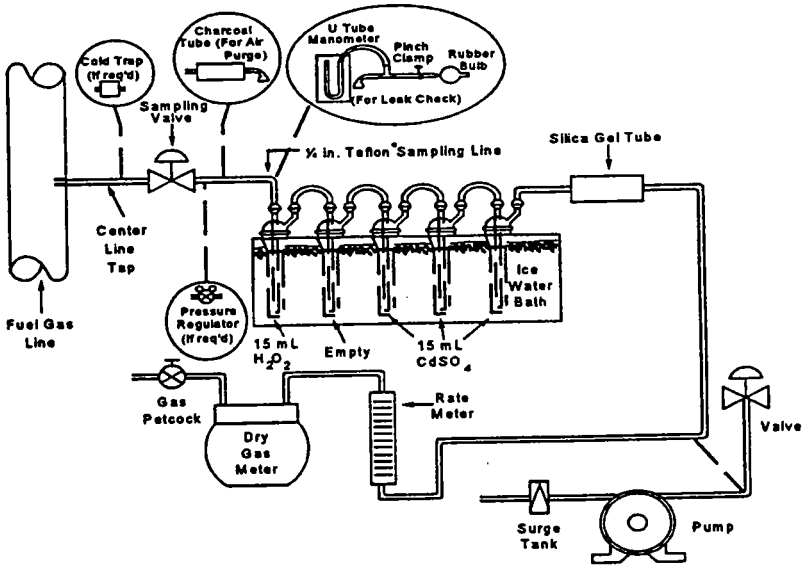

Figure F11-1. H_2S Sampling train.

FIELD DATA SHEET 11
Hydrogen Sulfide

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Test Location/Run # _____ Personnel _____

Train ID#/Sample Box # _____ DGM Cal Coef., Y _____ Ambient Temp., °C_____

Start Time _____ End Time _____ Bar. Pressure, P_b _____ mm Hg

Trav. Pt.	Samplg time (min)	DGM Rdg (L)	Rotameter Rdg (cc/min)	Temperature (°C)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m/\Delta\bar{V}_m$
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max ≤20°C?	Avg	0.90 - 1.10?

Leak-checks ≤0.4 in. H_2O/min			
Run #			
Pre (optional) (in./min)			
Post (mandatory)(in./min)			
Pressure (in. H_2O)			

Purge Rate _____ Purge Time _____ min

$$V_{m(std)} = 0.3858\ V_m\ Y\ \frac{P_b}{(273 + t_m)}$$

Post-Test Calibrations:
Attach CDS 2d and CDS 6 for temperature (≤ ±5.4°F), barometer, and metering system calibration checks.

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

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

LABORATORY PROCEDURE 11
Hydrogen Sulfide

A. *Sample Recovery*

1. Discard the contents of the H_2O_2 impinger.
2. Carefully transfer the contents of the third, fourth, and fifth impingers into a 500-mL iodine flask. Rinse with water the impingers and connecting glassware and quantitatively transfer the rinse into the iodine flask.
3. For a blank, add 45 mL $CdSO_4$ absorbing solution to an iodine flask.
4. Pipette exactly 50 mL 0.01 N I_2 solution into a 125-mL Erlenmeyer flask. Add 10 mL 3 M HCl to the solution.

Note: If Antifoam B was not used or if significant quantities of yellow CdS remain in the impingers, go to step B6 (alternative).

5. Quantitatively transfer the acidified I_2 into each iodine flask. Stopper the flask immediately, and shake briefly.
6. Alternative: Use the acidified I_2 solution (step B4) to extract any remaining CdS from the third, fourth, and fifth impingers and connecting glassware as follows:
   a. Immediately after pouring the acidified I_2 into an impinger, stopper it and shake for a few moments, then transfer the liquid directly to the iodine flask. Do not transfer any rinse portion from one impinger to another. Once the acidified I_2 solution has been poured into any glassware containing CdS, stopper the container at all times except when adding more solution, and do this as quickly and carefully as possible.
   b. After adding any acidified I_2 solution to the iodine flask, allow a few minutes for absorption of the H_2S before adding any further rinses.
   c. Repeat the I_2 extraction until any visible CdS is removed from the impingers.
   d. Quantitatively rinse all the I_2 from the impingers, connectors, and the beaker into the iodine flask using water. Stopper the flask and shake briefly.
7. Allow the iodine flask to stand about 30 min in the dark for absorption of the H_2S into the I_2.
8. Analyze the samples and blank immediately.
9. Recalibrate the metering system and temperature gauges (see FP 2d and CP 6).

B. *Reagent Preparation*

1. $CdSO_4$ Absorbing Solution. Dissolve 41 g $3CdSO_4 \cdot 8H_2O$ and 15 mL 0.1 M H_2SO_4 in a 1-L volumetric flask containing about 0.75 L water. Dilute to volume with water. Mix thoroughly. The pH should be 3 ± 0.1. (Optional: Add 10 drops Dow-Corning Antifoam B.) Shake well before use. Do not use after 1 month.
2. H_2O_2, 3%. Dilute 30% H_2O_2 1:9 by volume, as needed. Prepare fresh daily.
3. Hydrochloric Acid Solution, 3 M. Add 240 mL conc. HCl (s.g. 1.19) to 500 mL water in a 1-L volumetric flask. Dilute to 1 L with water. Mix thoroughly.
4. Iodine Solution, 0.1 N. Dissolve 24 g KI in 30 mL water. Add 12.7 g resublimed I_2 to the KI solution. Shake the mixture until the I_2 is completely dissolved. If possible, let the solution stand overnight in the dark. Slowly dilute the solution to 1 L with water, with swirling. Filter the solution if it is cloudy. Store solution in a brown-glass reagent bottle.
5. Standard I_2 Solution, 0.01 N. Pipette 100.0 mL 0.1 N iodine solution into a 1 L volumetric flask, and dilute to volume with water. Standardize daily. Protect this solution from light. Keep reagent bottles and flasks tightly stoppered.
6. Standard Sodium Thiosulfate Solution, 0.1 N. Dissolve 24.8 g sodium thiosulfate pentahydrate ($Na_2S_2O_3 \cdot 5H_2O$) or 15.8 g anhydrous sodium thiosulfate ($Na_2S_2O_3$) in 1 L water, and add 0.01 g anhydrous sodium carbonate (Na_2CO_3) and 0.4 mL chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for about 15 min, and store in a glass-stoppered, reagent bottle.
7. Standard Sodium Thiosulfate Solution, 0.01 N. Pipette 50.0 mL the standard 0.1 N $Na_2S_2O_3$ solution into a volumetric flask, and dilute to 500 mL with water.
8. Alternative to A7: Standard Phenylarsine Oxide Solution, 0.01 N. Dissolve 1.80 g C_6H_5AsO in 150 mL 0.3 N sodium hydroxide. After settling, decant 140 mL of this solution into 800 mL water. Bring the solution to pH 6-7 with 6 N HCl, and dilute to 1 L with water.

9. Starch Indicator Solution. Suspend 10 g soluble starch in 100 mL water, and add 15 g KOH pellets. Stir until dissolved, dilute with 900 mL water, and let stand for 1 hr. Neutralize the alkali with conc. HCl, using an indicator paper similar to Alkacid test ribbon, then add 2 mL glacial acetic acid as a preservative.

C. 0.1 N $Na_2S_2O_3$ Reagent Standardizations

1. Weigh and transfer 2 g dried potassium dichromate ($K_2Cr_2O_7$) to a 500-mL volumetric flask. Dissolve in water and dilute to exactly 500 mL.
2. In a 500-mL iodine flask, dissolve about 3 g KI in 45 mL water, then add 10 mL 3 M HCl solution. Pipette 50 mL dichromate solution into this mixture. Gently swirl the solution once, and allow it to stand in the dark for 5 min. Dilute the solution with 100 to 200 mL water, washing down the sides of the flask with part of the water. Titrate with 0.1 N $Na_2S_2O_3$ until the solution is light yellow.
3. Add 4 mL starch indicator and continue titrating slowly to a green end point.
4. Repeat titrations until replicate analyses agree within 0.05 mL, and average these values.
5. Calculate the normality. Repeat each week, or after each test series, whichever time is shorter.

D. 0.01 N C_6H_5AsO Standardization (if applicable)

1. Weigh and transfer 2 g $K_2Cr_2O_7$ to a 500-mL volumetric flask. Dissolve in water, and dilute to exactly 500 mL.
2. In a 500 mL iodine flask, dissolve approximately 0.3 g KI in 45 mL water; add 10 mL 3 M HCl. Pipette 5 mL dichromate solution into the iodine flask. Gently swirl the contents of the flask once allow to stand in the dark for 5 min. Dilute the solution with 100 to 200 mL water, washing down the sides of the flask with part of the water. Titrate with 0.01 N C_6H_5AsO until the solution is light yellow.
3. Add 4 mL starch indicator, and continue titrating slowly to a green end point.
4. Repeat titrations until replicate analyses agree within 0.05 mL, and average these values.
5. Calculate the normality. Repeat each week or after each test series, whichever time is shorter.

E. 0.01 N I_2 Reagent Standardization

1. Pipette 25 mL standard I_2 solution into a 125-mL Erlenmeyer flask. Add 2 mL 3 M HCl. Titrate rapidly with standard 0.01 N $Na_2S_2O_3$ solution or with 0.01 N C_6H_5AsO until the solution is light yellow, using gentle mixing.
2. Add four drops starch indicator solution, and continue titrating slowly until the blue color just disappears.
3. Repeat titrations until replicate values agree within 0.05 mL, then average these values.
4. Calculate normality of the I_2 solution. Repeat daily.

F. Analysis

1. Test starch indicator solution for decomposition by titrating with 0.01 N I_2 solution, 4 mL starch solution in 200 mL water that contains 1 g KI. If more than 4 drops of 0.01 N I_2 standard solution are required to obtain the blue color, prepare a fresh solution.
2. Conduct titration analyses immediately after recovery to prevent loss of I_2 from the sample. Avoid direct sunlight. **(See LDS 11).**
3. Rapidly titrate each sample with 0.01 N $Na_2S_2O_3$ solution (or 0.01 N C_6H_5AsO, if applicable), in an iodine flask, to a light yellow color.
4. Add 4 mL starch indicator solution, and continue titrating slowly until the blue color just disappears.
5. Titrate the blanks in the same manner as the samples.
6. Run blanks each day until replicate values agree within 0.05 mL, and average them.

LABORATORY DATA SHEET 11
Hydrogen Sulfide

Client/Plant Name _____ Job # _____

City/State _____ Sampling Location _____

Analyst _____ Date Analyzed _____ Time Analyzed _____

Run No.	Sample			Sample Titration		
	Total, V (mL)	Aliquot, A (mL)	Factor, F = V/A	T_1 (mL)	T_2 (mL)	Avg, V_{TT} (mL)
Blank # 1						
Blank # 2						

No.	$K_2Cr_2O_7$, W (g)	Thiosulfate Standard Titration		Iodine Standard Titration		
		Volume, V_S (mL)	Normality, N_S	Aliquot, V_I (25 mL)	Volume, V_T (mL)	Normality, N_I
1						
2						
Avg						

____ Analyses started within 1 hr of sampling?

____ Titrations done 30 min after adding acidified Iodine solution?

____ All replicate titrations agree within 0.05 mL?

____ Starch indicator tested for decomposition?

$$N_S = 2.039 \frac{W}{V_S} \qquad N_I = \frac{N_T V_T}{V_I}$$

$$N_T = 0.10\, N_S$$

Note: This data sheet is designed to be used with standard thiosulfate solution; if standard phenylarsine is used, make the necessary changes according to Method 11.

QA/QC Check

Completeness ____ Legibility ____ Accuracy ____ Specifications ____ Reasonableness ____

Checked by: _____ _____

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