

SUMMARY SHEET 16
Reduced Sulfur Compounds

		Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 15			
Job No.		FDS 15			
Sampling Location		FDS 15			
Run ID #		FDS 15			
Test Date		FDS 15			
Run Start Time		FDS 15			
Run Finish Time		FDS 15			
Concentration H ₂ S, ppm	H ₂ S	FDS 15			
Concentration MeSH, ppm	MeSH	FDS 15			
Concentration DMS, ppm	DMS	FDS 15			
Concentration DMDS, ppm	DMDS	FDS 15			
Average SO ₂ Equivalent, ppm	SO _{2e}	FDS 15			
Sample Line Loss Ratio	LR	FDS 15			
Corr Avg SO ₂ Equivalent, ppm	SO _{2ec}	SS 16			
Post-test Calibration Checks					
Flow Meter Calibration		FDS 15			
Dilution Factor		FDS 15			

$$SO_{2ec} = \frac{SO_{2e}}{LR}$$

SUMMARY SHEET 16A
Reduced Sulfur Compounds

Run #1 Run #2 Run #3 Avg

Client/Plant Name FDS 16A
 Job No. FDS 16A
 Sampling Location FDS 16A
 Run ID # FDS 16A

Test Date FDS 16A
 Run Start Time FDS 16A
 Run Finish Time FDS 16A

Barometric Pressure, mm Hg P_b FDS 16A
 Average DGM Temperature, °C t_m FDS 16A
 DGM Calibration Factor Y FDS 16A
 Volume of Metered Gas Sample, dcf V_m FDS 16A
 Volume of Metered Gas Sample, dscf $V_{m(std)}$ SS 16A

Sample

Normality of Titrant, meq/mL N LDS 6
 Volume of Aliquot, mL V_a LDS 6
 Volume of Solution, mL V_s LDS 6
 Volume of Titrant, mL V_t LDS 6
 Volume of Titrant for Blank, mL V_{tb} LDS 6

System Performance (H₂S)

DGM Calibration Factor Y_p LDS 16A
 Avg DGM Temperature, °C t_{mp} LDS 16A
 Vol. of Metered Gas, dL V_{mp} LDS 16A
 Std Vol. of Metered Gas, dSL $V_{mp(std)}$ SS 16A
 Normality of Titrant, meq/mL N LDS 6
 Volume of Aliquot, mL V_a LDS 6
 Volume of Solution, mL V_s LDS 6
 Volume of Titrant, mL V_t LDS 6
 Volume of Titrant for Blank, mL V_{tb} LDS 6

Sample Concentration of TRS as SO₂, ppm C_{TRS} SS 16A
 Recovery Gas Ref Concentration, ppm C_{RG} FDS 16A
 Recovered Gas Measured Concentration, ppm C_{RGm} SS 6
 Recovery Efficiency, % R SS 16A
 $R = 1.00 \pm 0.20 ?$

Audit Relative Error, % RE QA1

Post-Test Calibration Checks
 Temperature and Barometer CDS 2d
 Metering System CDS 6

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

$$C_{TRS} = 12025 \frac{(V_t - V_{tb}) N \left(\frac{V_s}{V_a} \right)}{V_{m(std)}}$$

Use the above equation to calculate $V_{mp(std)}$ also; use the appropriate data.

Use the above equation to calculate C_{RGm} also; use the appropriate data.

$$R = \frac{C_{RGm}}{C_{RG}} \times 100$$

FIELD PROCEDURE 16
Reduced Sulfur Compounds

Note: Methods 15 and 16 are identical except for the reduced sulfur compounds being analyzed. Method 15 is used to determine hydrogen sulfide (H₂S), carbonyl sulfide (COS), and carbon disulfide (CS₂) from tail gas control units of sulfur recovery plants. Method 16 is used to determine H₂S, methyl mercaptan (MeSH), dimethyl sulfide (DMS), and dimethyl disulfide (DMDS).

The forms in this section contain the information required by the test method; we are aware that some of the technology specified in the test method is obsolete. In these cases, the user should modify the forms to make them consistent with the technology used.

A. Optional Pretest Procedures

1. Leak-check all components, sample lines, and connections.
 - a. For components upstream of the sample pump, use FP 3c, section A, except conduct the leak-check at >2 in. Hg vacuum and 1 min.
 - b. For components after the pump, use FP 3c, section E.
2. Observe the response of flowmeters or of the GC output to changes in flow rates or calibration gas concentrations and ascertain the response to be within predicted limits.

B. Calibration

1. Equilibrate the permeation tubes (H₂S, COS, and CS₂) for 24 hr at the calibration temperature ($\pm 0.1^\circ\text{C}$). *(For Method 16, use permeation tubes for H₂S, MeSH, DMS, and DMDS.)*
2. Generate a series of three or more known concentrations spanning the linear range of the FPD (approximately 0.5 to 10 ppm for a 1-mL sample) for each of the sulfur compounds.
3. Bypassing the dilution system, inject the standards into the GC/FPD analyzers until the response of any one of three injects at each concentration varies no more than $\pm 13\%$ from their average (hereafter called precision). *For Method 16, the precision requirement is $\pm 5\%$.*
4. Generate a least squares equation of the concentrations vs. the appropriate GC/FPD response units (log-log relationship).
5. Calibrate each stage of the dilution system using a known concentration of H₂S from the permeation tube system. (See Figure C15-1.) Determine from the GC/FPD the concentration of the diluted calibration in ppm to within $\pm 13\%$ *(or $\pm 5\%$ for Method 16) precision.* Then calculate the dilution factor.

C. Sampling and Analysis Procedure

1. Assemble the apparatus as shown in Figure F15-1. Calibrate the system before the first run as in section B.
2. Insert the sampling probe into the test port; plug off open areas to prevent dilution air from entering the stack. Begin sampling, and dilute the sample approximately 9:1. Condition the entire system with sample for at least 15 min before analyzing.
3. For each sample run, analyze 16 individual injects of the diluted sample on the GC/FPD analyzer over 3 to 6 hr.
4. If sample concentrations decreases during a sample run and the decrease is not due to process conditions, check for clogging in the sample probe. If the probe is clogged, invalidate the test run, and restart the run.
5. After each run, inspect the sample probe.

D. Post-test Procedures

1. Determine the sample line loss as follows:
 - a. Introduce into the sampling system at the probe inlet H₂S of known concentration (using permeation tubes or H₂S/air mixture in a gas cylinder, traceable to permeation tubes) within $\pm 20\%$ of the applicable standard.
 - b. Compare the resulting measured concentration with the known value (must be $\leq 20\%$ loss).
2. After each run, or after a series of runs made within a 24-hr period, recalibrate the GC/FPD analysis and dilution system using only H₂S (or other permeant). Compare against the calibration curve obtained before the test runs. If the means of the triplicates differ $\leq 5\%$, either void the intervening runs or use the calibration data set that gives the highest sample values.

3. After a complete test series, calibrate each flowmeter in the permeation tube flow system with a wet test meter or soap bubble meter (must agree within $\pm 5\%$ of the initial calibration).

3. Section B. Calibrate the GC/FPD system by generating a series of three or more concentrations of each sulfur compound and diluting these samples before injecting them into the GC/FPD system. A separate determination of the dilution factor is not necessary, however, precision of $\pm 13\%$ still applies.

E. Alternatives

1. Step B1. Inject samples of calibration gas at 1-hr intervals until three consecutive hourly samples agree within $\pm 13\%$ of their average.
2. Step B4. Plot the GC/FPD response in current (amperes) vs. their causative concentrations in ppm on log-log coordinate graph paper for each sulfur compound.

FIELD PROCEDURE 16A
Reduced Sulfur Compounds

Note: FP 16A is a procedure in which Method 6 is used after a citrate scrubber to remove SO₂ and an oxidizing system to convert reduced sulfur compounds to SO₂.

A. Sampling Train Preparation

1. Set up the sampling train as shown in Figure F16A-1. Prepare the Method 6 part of the train as in FP 6, except use 20 mL H₂O₂. Add 100 mL citrate buffer into the first and second impingers of the SO₂ scrubber; leave the third empty. Keep the Teflon line between the heated filter and citrate scrubber as short as possible.
2. Set the oxidation furnace at 800 ± 100°C and the probe and filter temperature high enough to prevent visible condensation of moisture.
3. Bypassing all sample collection components, draw stack gas into the citrate scrubber for 10 min at 2 L/min. Then assemble the train.
4. *Optional:* Leak-check the sampling train as in FP 3c, sections C and D.
5. *Optional:* Conduct two 30-min system performance checks in the field according to section C.

B. Sample Collection

1. Sample as in Method 6 at 2.0 L/min (±10%) for 1 hr (three 1-hr samples are required for each run) or for 3 hr.
2. *Mandatory:* At the end of sampling, leak-check the sampling train as in FP 3c, section C.
3. Recover the sample as in Method 6, except do not purge the sample.

4. *Mandatory:* Conduct a performance system check after each 3-hr run or after three 1-hr samples. See section C and FDS 16A.
5. *Optional:* Rinse and brush the probe with water, replace the filter, and change the citrate solution.

C. System Performance Check

1. Adjust the flow rates to generate H₂S concentration in the range of the stack gas or within ±20% of the applicable standard and an O₂ concentration >1% at a total flow rate of at least 2.5 L/min. See Figure F16A-2.
2. Calibrate the flow rate from both sources with a soap bubble flow tube.
3. Collect 30-min samples, and analyze in the normal manner. Collect the samples through the probe of the sampling train using a manifold or some other suitable device. Do not replace the particulate filter nor the citrate solution and do not clean the probe before this check.
4. Analyze the samples as in LP 6, except for 1-hr samples, use a 40-mL aliquot, add 160 mL of 100% isopropanol, and four drops of thiorin.
5. Analyze field audit samples, if applicable.

Note: Sample recovery must be 100 ± 20% for data to be valid. Do not use recovery data to correct the test results. However, if the performance check results do not affect the compliance or noncompliance status of the affected facility, the Administrator may accept the results.

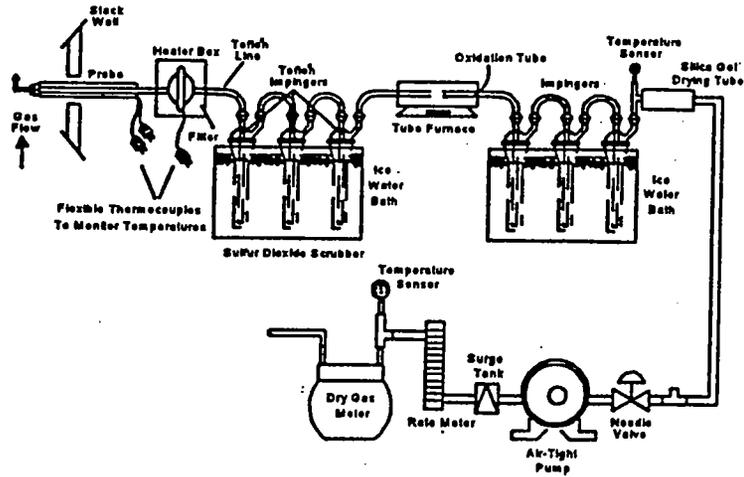


Figure F16A-1. Sampling Train.

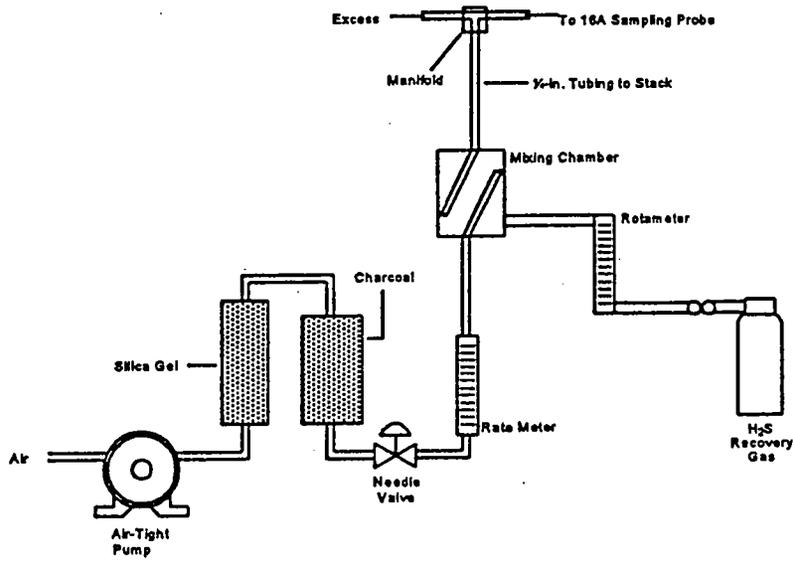


Figure F16A-2. Recovery Gas Dilution System.

FIELD DATA SHEET 16A
Reduced Sulfur Compounds

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
(Sampling Time: Three 1-hr samples or One 3-hr sample?)

Trav Pt.	Samplg time (min)	DGM Rdg (L)	Rotam Rdg (L/min)	Temperature (°C)		Flow Rate Deviation		Furnace Temp (°C)
				DGM	Imp. Exit	ΔV_m	$\Delta V_m / \bar{\Delta V}_m$	
	Total Time, θ_s	Volume, V_m	Avg	Avg, t_m	Max $\pm 20^\circ\text{C}?$	Avg	$2.0 \pm 0.2?$	$800 \pm 100?$

___ Proper probe heat (no condensation)?

Sample Recovery

- ___ No purge?
- ___ Fluid level marked?
- ___ Sample container sealed?
- ___ Sample container identified?

Leak-Checks ± 0.02 Avg Flow Rate at ± 10 in. Hg vac.			
Run #			
Pre (optional) (cc/min)			
Post (mandatory) (cc/min)			
Vacuum (± 10 in. Hg ?)			

Post-Test Calibrations

Attach CDS 2d and CDS 6; temperature specification for DGM is $\pm 5.4^\circ\text{F}$.

QA/QC Check (Include second page)

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

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

FIELD DATA SHEET 16A (Continued)
System Performance Check

Client/Plant Name _____ Job # _____

City/State _____ Date/Time _____

Train ID# _____ Personnel _____

DGM Calibration Factor, Y_p _____ **Note: Check req'd after each 3-hr run or after three 1-hr runs.**

Rotameter Calibration			
Train ID# _____	H ₂ S		Air
Rotameter Rdg, L/min			
Bubble Meter Vol, V_{sb} L			
Time, θ sec			
Bar. Press., P_b mm Hg			
Amb Temp., t_{amb} °C			
Flow Rate, Q_{std} L/min			
Average \bar{Q}_{std} L/min			

Samplg time (min)	H ₂ S Rotam Rdg (L/min)	Air Rotam Rdg (L/min)	DGM Rdg (L)	Temperature (°C)		Flow Rate Deviation	
				DGM	Imp. Exit	ΔV_m	$\Delta V_m / \bar{\Delta V}_m$
0							
5							
10							
15							
20							
25							
30							
Total Time, θ_s	Avg	Avg	Volume, V_{mp}	Avg, t_{mp}	Max ≤ 20 °C?	Avg	0.90 - 1.10?

Reference H₂S Cylinder Concentration, C_{H_2S} _____ ppm (**about stack concentration or $\pm 20\%$ standard?**)

Flow rate: Total = H₂S + Air = _____ L/min (≥ 2 L/min?) Air = _____ L/min (≥ 0.048 Total?)

$$Q_{std} = 21.13 \frac{V_{sb}}{\theta} \frac{P_b}{(t_{amb} + 273)}$$

$$C_{RG} = C_{H_2S} \frac{Q_{H_2S}}{Q_{H_2S} + Q_{Air}}$$

LDS 6 Check: _____ For 1-hr samples, 40 mL aliquot, 160 mL 100% IPA, and 4 drops of thiorin used?

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____
Personnel (Signature/Date)

Team Leader (Signature/Date)

FIELD PROCEDURE 16Aa
H₂S Content in Cylinder Gases

A. Pre-test Preparations

1. Calculate gas sample volumes in liters. Divide the cylinder gas value by the ppm-liters factor provided below:

H ₂ S cylinder gas concentration (ppm)	Factor ppm-L
5 to <30 ppm	650 ppm-L
30 to <500 ppm	800 ppm-L
500 to <1500 ppm	1000 ppm-L

2. Select a critical orifice within the following flow rate range :

H ₂ S cylinder gas conc. (ppm)	Critical orifice flow rate, (mL/min)
5 to <50 ppm	1500 ± 500 ppm
50 to <250 ppm	500 ± 250 ppm
250 to <1000 ppm	200 ± 50 ppm
> 1000 ppm	75 ± 25 ppm

3. Calibrate the critical orifice with the sampling train according to FP 6a.
4. Determine the approximate sampling time for a cylinder of known concentration. Divide the gas sample volume times 1000 by the critical orifice flow rate.

B. Sampling Train Preparation

1. Connect the Teflon tubing, Teflon tee, and rotameter to the flow control needle valve as shown in Figure F16Aa-1. Vent the rotameter to an exhaust hood. Plug the open end of the tee.
2. Approximate the critical orifice flow rate by connecting the critical orifice to the sampling system as shown in Figure F16Aa-1 without the H₂S cylinder. Connect a rotameter to the inlet of the first impinger. Turn on the pump, and increase vacuum to about half atmosphere. Slowly increase the vacuum until a constant flow rate is reached. Record the vacuum reading as the critical vacuum. Ensure that this flow rate is in the range shown in step A2 before proceeding.

C. Sample Collection

1. Five to 10 min prior to sampling, open the cylinder valve while keeping the flow control needle valve closed. Adjust the delivery pressure to 20 psi. Open the needle valve slowly until the rotameter

shows a flow rate ~ 50 to 100 mL above the flow rate of the critical orifice being used in the system.

2. Place 50 mL zinc acetate solution in the first two impingers, leave the third impinger empty and assemble as shown in Figure F16Aa-1. Make sure the ground-glass fittings are tight. Connect the Teflon sample line to the first impinger. Protect the absorbing solution from light during sampling by covering the impingers with a dark cloth or piece of plastic.
3. Record the information on the data sheet. Open the closed end of the tee. Connect the sampling tube to the tee, ensure a tight connection. Start the sample pump and stopwatch simultaneously. Sample for the period determined in step A4.
4. Turn off the pump and stopwatch. Disconnect the sampling line from the tee and plug it. Close the needle valve followed by the cylinder valve. Record the sampling time.
5. Conduct a post-test critical orifice calibration run using the calibration procedures outlined in step A3. The Q_{std} obtained before and after the test cannot differ by >5%.

D. Sample Recovery

1. Do not detach the stems from the bottoms of the impingers. Add 20.0 mL 0.01 N iodine solution through the stems of the first two impingers, dividing it between the two (add ~ 15 mL to the first impinger and the rest to the second).
2. Add 2 mL HCl solution through the stems, dividing it between the two impingers.
3. Disconnect the sampling line and store the impingers.

E. Post-test Calibration Checks

Calibrate barometer according to FP 2d.

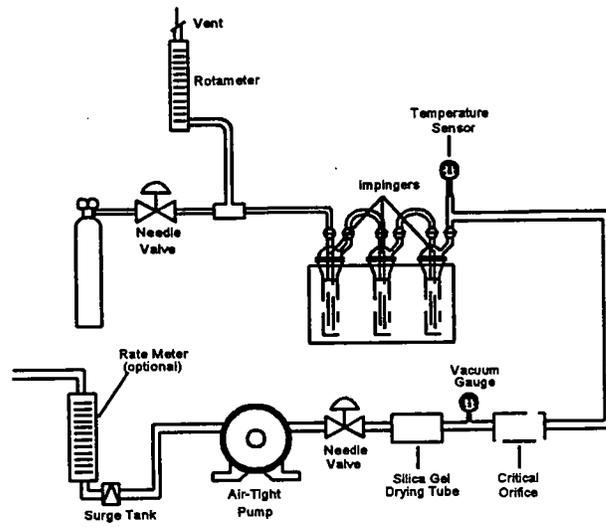


Figure F16Aa-1. Recovery Check Gas Sampling Train.

FIELD DATA SHEET 16Aa
Hydrogen Content in Cylinder Gases

Client/Plant Name _____ Job # _____

City/State _____ Test Location _____

Run # _____ Personnel _____

Bar Press, P_b _____ in. Hg Amb Temperature _____ °F Date _____

Cylinder tag value _____ ppm Calculated sample volume _____ L Critical orifice flow rate _____ mL/min

Calculated sample time _____ min. Sample Time = $\frac{\text{Sample Vol.} \times 1000}{\text{Critical Orifice Flow Rate}}$

Start time _____ End time _____

Use FDS 6a to collect the necessary data (attach to this data sheet). In addition, note the following:

Vent Rotameter Reading:

Before pump is on _____ After pump is on _____ Difference = critical orifice meter flow rate?

Using the information from FDS 6a, calculate $V_{m(\text{std})}$ using the equation below:

$$V_{m(\text{std})} = \bar{Q}_{\text{std}} \theta_s (1 - B_{\text{wa}}) \frac{M_a}{M_s}$$

 M_a = molecular weight of ambient air saturated at impinger temperature. At 25°C, use $M_a = 28.5$ g/g-mole. M_s = molecular weight of sample gas (nitrogen) saturated at impinger temperature. At 25°C, use $M_s = 27.7$ g/g-mole.**QA/QC Check**

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

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

LABORATORY PROCEDURE 16Aa
H₂S Content in Cylinder Gases

A. Reagents:

1. Zinc Acetate Absorbing Solution. Dissolve 20 g zinc acetate in water and dilute to 1 L.
2. Standard Potassium Bi-iodate [KH(IO₃)₂], 0.100 N. Dissolve 3.249 g anhydrous KH(IO₃)₂ in water and dilute to 1 L.
3. Standard Sodium Thiosulfate (Na₂S₂O₃), 0.1 N. Dissolve 24.8 g sodium thiosulfate pentahydrate (Na₂S₂O₃·5H₂O) or 15.8 g anhydrous sodium thiosulfate (Na₂S₂O₃) in 1 L water, and add 0.01 g anhydrous sodium carbonate (Na₂CO₃) and 0.4 mL chloroform (CHCl₃) to stabilize. Shake thoroughly or aerate with nitrogen for about 15 min, and store in a glass-stoppered, reagent bottle. Standardize according to step B1.
4. Standard Na₂S₂O₃, 0.01 N. Pipette 100.0 mL 0.1 N Na₂S₂O₃ solution into a 1-L volumetric flask, and dilute to the mark with water.
5. Iodine, 0.1 N. Dissolve 24 g KI in 30 mL water. Add 12.7 g resublimed I₂ to the KI solution. Shake the mixture until the I₂ 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.
6. Standard I₂, 0.01 N. Pipette 100.0 mL 0.1 N I₂ into a 1 L volumetric flask, and dilute to volume with water. Standardize following step B2 daily. Protect this solution from light. Keep reagent bottles and flasks tightly stoppered.
7. HCl, 10%. Add 230 mL conc. HCl to 770 mL water.
8. Starch Indicator. To 5 g starch (potato, arrowroot, or soluble), add a little cold water, and grind in a mortar to a thin paste. Pour into 1 L of boiling water, stir, and let settle overnight. Use the clear supernatant. Preserve with 1.25 g salicylic acid, 4 g zinc chloride, or a combination of 4 g sodium propionate and 2 g sodium azide per liter of starch solution. Some commercial starch substitutes are satisfactory.

B. Standardizations

1. Na₂S₂O₃, 0.1 N. Standardize the 0.1 N Na₂S₂O₃ as follows: To 80 mL water, stirring constantly, add 1 mL conc. H₂SO₄, 10.0 mL 0.100 N KH(IO₃)₂ and 1 g KI. Titrate immediately with 0.1 N NaS₂O₃,

until the solution is light yellow. Add 3 mL starch solution and titrate until the blue color just disappears. Repeat the titration until replicate analyses agree within 0.05 mL. Take the average volume of Na₂S₂O₃ consumed, and calculate the normality to three decimal figures (see LDS).

2. Iodine, 0.01 N. Standardize the 0.01 N I₂ as follows:
 - a. Pipet 20.0 mL 0.01 N I₂ into a 125-mL Erlenmeyer flask. Titrate with standard 0.01 N Na₂S₂O₃ until the solution is light yellow. Add 3 mL starch solution, and continue titrating until the blue color just disappears.
 - b. If the normality of the iodine tested is not 0.010, add a few mL 0.1 N I₂ if it is low, or a few mL water if it is high, and standardize again. Repeat the titration until replicate values agree to ±0.05 mL. Calculate the normality to three decimal places.

C. Blank Analysis

During sample collection, run a blank as follows:

1. Add 100 mL zinc acetate solution, 20.0 mL 0.01 N I₂, and 2 mL 10% HCl to a 250-mL Erlenmeyer flask. Titrate, while stirring, with 0.01 N Na₂S₂O₃ until the solution is light yellow. Add starch, and continue titrating until the blue color disappears.
2. Some difficulties in the titration include:
 - a. The solution will turn slightly white in color near the end point, and the disappearance of the blue color is hard to recognize.
 - b. A blue color may reappear in the solution about 30 to 45 sec after the titration endpoint is reached.

D. Sample Analysis

1. After the sample has been stored in the impingers for 30 min rinse the impinger stems into the impinger bottoms.
2. Titrate the impinger contents with 0.01 N Na₂S₂O₃. Do not transfer the contents of the impinger to a flask because this may result in a loss of iodine and cause a positive bias.
3. Analyze a blank with each sample, as the blank titer has been observed to change over the course of a day.

LABORATORY DATA SHEET 16Aa
Hydrogen Sulfide Content in Cylinder Gases

Client/Plant Name _____ Job # _____

City/State _____ Sampling Location _____

Analyst _____ Date Analyzed _____ Time Analyzed _____

Standardizations:

No.	Thiosulfate Standard Titration		Iodine Standard Titration		
	Volume, V _S (mL)	Normality, N _T	Aliquot, V _I (20 mL)	Volume, V _T (mL)	Normality, N _I
1					
2					
Avg					

$$N_T = \frac{1}{V_S}$$

$$N_I = \frac{N_T V_T}{V_I}$$

Sample Analysis:

Run No.	Total Sample Vol. (mL)	First Impinger, V ₁ (mL)	Second Impinger, V ₂ (mL)	Total Standard used, V _T (mL)	H ₂ S Conc., C _{H2S}
	100				
	100				
	100				
	100				
	100				
	100				
Blank # 1	100			(V _{TB})	
Blank # 2	100			(V _{TB})	
Blank # 3	100			(V _{TB})	

_____ Titrations done 30 min after adding acidified Iodine solution?

_____ All replicate standardization titrations agree within 0.05 mL?

_____ Starch indicator tested for decomposition?

$$C_{H_2S} = \frac{12025 N_T (V_{TB} - V_T)}{V_{m(std)}}$$

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____
 Personnel (Signature/Date)

_____ Team Leader (Signature/Date)

SUMMARY SHEET 16B
Reduced Sulfur Compounds

		Run #1	Run #2	Run #3	Avg
Client/Plant Name					
Job No.	FDS 16B				
Sampling Location	FDS 16B				
Run ID #	FDS 16B				
Test Date	FDS 16B				
Run Start Time	FDS 16B				
Run Finish Time	FDS 16B				
Avg TRS Concentration, ppm	C _{TRS}	FDS 16B			
Sample Line Loss Ratio	LR	FDS 16B			
Corr Avg TRS Concentration, ppm	C _{TRSc}	SS 16B			

$$C_{TRSc} = \frac{C_{TRS}}{LR}$$

FIELD PROCEDURE 16B
Reduced Sulfur Compounds

Note: FP 16B is a combination of Methods 16 (same as Method 15) and 16A. The oxidized sulfur compounds are measured using gas chromatography/flame photometric detection. The O₂ content in the flue gas must be $\geq 1\%$. Use FDS 16B.

A. Sampling Train Preparation

1. Set up the sampling train as shown in Figure F16B-1. Prepare the sampling train according to section A of FP 16A.
2. Set up the GC/FPD system according to FP 15, section B (Methods 15 and 16 are identical).

B. Sample Collection

1. Sample according to FP 15, section C.
2. If the sample is diluted determine the precise dilution factor.

C. System Performance Check

Conduct this check according to FP 16A, section C, except use measurements of the GC/FPD to determine the precision.

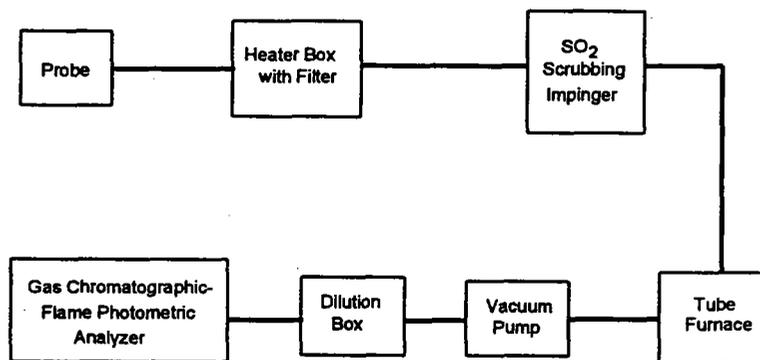


Figure F16B-1. Sampling Train.

FIELD DATA SHEET 16B
Reduced Sulfur Compounds

Client/Plant Name _____ Date _____ Job # _____

City/State _____ Personnel _____

Calibration (✓) Initial _____ Post-Test _____ ($\leq \pm 5\%$ Diff?) Sampling Location _____

Conc. Level	SO ₂ Conc., C (ppm)	GC/FPD Response: %Dev = $\leq \pm 5\%$				
		Inject #1	Inject #2	Inject #3	Average	High % Dev
1						
2						
3						

Note: Plot response vs. concentration; attach graph. Fyrite O₂ _____ (1% ?)

Use only if dilution is necessary.

Stage	SO ₂ Conc. (ppm)	GC/FPD Resp: % Dev = $\leq \pm 5\%$					Meas. Conc. (ppm)	Dilution Factor
		Inject #1	Inject #2	Inject #3	Average	% Dev		
1								
2								

Sample Line Loss:

Ref Conc, C_r _____ ppm H₂S Meas. Conc, C_m _____ ppm LR = C_m/C_r = _____ (0.80 to 1.20)

Post-test Flow Meter Calibration (permeation tube flow system):

Initial Cal Factor, Y_i _____ Post-Test Cal Factor, Y_f _____ Y_f/Y_i = _____ (0.95 to 1.05 ?)

Run No.	Resp	Meas. Conc. (ppm)	D.F.	Conc., C _S (ppm)	Run No.	Resp	Meas. Conc. (ppm)	D.F.	Conc., C _S (ppm)
1					9				
2					10				
3					11				
4					12				
5					13				
6					14				
7					15				
8					16				
							Avg SO _{2e} , C _{TRS}		

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____
Personnel (Signature/Date)

Team Leader (Signature/Date)