

**SUMMARY SHEET 6**  
**Sulfur Dioxide**

		Run #1	Run #2	Run #3	Avg
Client/Plant Name	FDS 6				
Job No.	FDS 6				
Sampling Location	FDS 6				
Run ID#	FDS 6				
Test Date	FDS 6				
Run Start Time	FDS 6				
Run Finish Time	FDS 6				
Traverse Points (if applicable)	FDS 1				
Net Run Time, min	$\theta$	FDS 6			
Dry Gas Meter Calibration Factor	Y	CDS 6			
Barometric Pressure, in. Hg	$P_b$	FDS 6			
Average DGM Temperature, °F	$t_m$	FDS 6			
Avg Abs DGM Temperature (460 + $t_m$ ), R	$T_m$	SS 6			
Volume of Metered Gas Sample, dcf	$V_m$	FDS 6			
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6			
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6			
Volume of Sample Solution, mL	$V_s$	LDS 6			
Volume of Sample Aliquot Titrated, mL	$V_a$	LDS 6			
Average Volume Titrant for Sample, mL	$V_t$	LDS 6			
Volume Titrant for Blank, mL	$V_b$	LDS 6			
SO <sub>2</sub> Concentration, lb/dscf	$C_{SO_2}$	SS 6			
Audit Relative Error, %	RE	QA 1			
Post-test Calibration Checks					
Temperature and Barometer		CDS 2d			
Metering System		CDS 6			

$$V_{m(std)} = 17.64 V_m Y \frac{P_b}{T_m}$$

$$C_{SO_2} = 7.061 \times 10^{-5} \frac{(V_t - V_{tb}) N \left( \frac{V_s}{V_a} \right)}{V_{m(std)}}$$

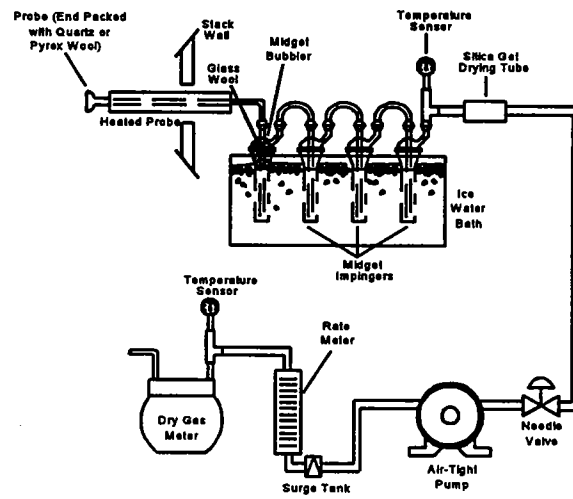


Figure F6-1.  $\text{SO}_2$  Sampling Train.

**FIELD PROCEDURE 6**  
**Sulfur Dioxide**

**A. Pre-test Preparations**

1. Calibrate the metering system (see CP 6).
2. Determine the number and location of sampling points and sampling time.
3. Prepare the sampling train as follows:
  - a. Add 15 mL 80% isopropanol into the midget bubbler.
  - b. Add 15 mL 3% H<sub>2</sub>O<sub>2</sub> into each of the first two midget impingers.
  - c. Leave the final midget impinger dry.
  - d. Assemble the train as shown in Figure F6-1.
  - e. Adjust probe heater to a temperature sufficient to prevent water condensation.
  - f. Place crushed ice and water around the impingers.

**B. Sampling**

1. **Optional:** Leak-check the sampling train (see FP 3c, sections C and D).
2. Record the initial DGM reading and barometric pressure.
3. Position the tip of the probe at the first sampling point, connect the probe to the bubbler, and start the pump.
4. Adjust the sample flow (rotameter) to a constant rate of about 1.0 L/min. Maintain this constant rate ( $\pm 10\%$ ) during the entire sampling run.
5. Traverse, if applicable. Take readings (DGM, temperatures at DGM and at impinger outlet, and rate meter) at least every 5 min.
6. Add more ice during the run to keep the temperature of the gases leaving the last impinger at  $\leq 68^\circ\text{F}$ .
7. At the conclusion of the run, turn off the pump, remove probe from the stack, and record the final readings.
8. **Mandatory:** Leak-check the sampling train (see FP 3c, section C).

**C. Sample Recovery**

1. Drain the ice bath, and purge the remaining part of the train by drawing clean ambient air through the system for 15 min at the sampling rate. Pass air through a charcoal filter or through an extra midget impinger with 15 mL 3% H<sub>2</sub>O<sub>2</sub> or use ambient air without purification.
2. Disconnect the impingers after purging. Discard the contents of the midget bubbler. (Saving this portion until after analysis may be helpful to explain anomalies.)
3. Pour the contents of the midget impingers into a leak-free polyethylene bottle for shipment.
4. Rinse the three midget impingers and the connecting tubes with water, and add the washings to the same storage container.
5. Seal and identify the sample container. Mark the fluid level.

**D. Post-test Calibrations**

Conduct post-test calibration checks of metering system and temperature gauges according to CP 2d and CP 2e (use CDS 2d and CDS 6).

**E. Elimination of Ammonia Interference**

Use FP 6 above, with the following modifications:

1. Use a high efficiency in-stack filter (glass fiber) that is unreactive to SO<sub>2</sub>, e.g., Whatman 934 AH.
2. Maintain the probe at 525°F during sampling.
3. Do not discard the isopropanol solution in the midget bubbler (step C2), but quantitatively recover the solution into container containing the solutions from the midget impingers (step C3).
4. **Alternatives:**
  - a. If SO<sub>3</sub> is expected to be insignificant, the midget bubbler may be deleted from the sampling train.
  - b. If an approximate SO<sub>3</sub> concentration is desired, the midget bubbler contents may be recovered in a separate polyethylene bottle.

**FIELD DATA SHEET 6**  
Gaseous Pollutant Sampling

Method (Circle) 6 6A 6B 7C 7D

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date/Time \_\_\_\_\_

Test Location/Run # \_\_\_\_\_ Personnel \_\_\_\_\_

Train ID#/Sample Box # \_\_\_\_\_ DGM Cal Coef., Y \_\_\_\_\_ Ambient Temp., °F \_\_\_\_\_

Start Time \_\_\_\_\_ End Time \_\_\_\_\_ Bar. Pressure, P<sub>b</sub> \_\_\_\_\_ in. Hg

Trav. Pt.	Samplg time (min)	DGM Rdg (cf)	Rotameter Rdg (cc/min)	Temperature (°F)		Flow Rate Deviation	
				DGM	Imp. Exit	$\Delta V_m$	$\Delta V_m / \bar{\Delta V}_m$
	Total Time, $\theta_s$	Volume, V <sub>m</sub>	Avg	Avg, t <sub>m</sub>	Max $\leq 68^\circ F?$	Avg	0.90 - 1.10?

\_\_\_ Proper probe heat (no condensation)?

Purge Rate \_\_\_\_\_ (at avg rotameter rdg)?

Purge Time \_\_\_\_\_ min ( $\geq 15$  min)?

**Sample Recovery**

\_\_\_ Fluid level marked?

\_\_\_ Sample container sealed?

\_\_\_ Sample container identified?

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

**Post-Test Calibrations:**

Attach CDS 2d and CDS 6. Temperature specification for the DGM thermometer is  $\leq \pm 5.4^\circ F$ .

**QA/QC Check**

Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
Personnel (Signature/Date)

\_\_\_\_\_ Team Leader (Signature/Date)

**LABORATORY PROCEDURE 6**  
**Sulfur Oxides**

**A. Reagent Preparation**

1. Isopropanol. Check each lot of isopropanol for peroxide impurities as follows:
  - a. Shake 10 mL isopropanol with 10 mL freshly-prepared 10% potassium iodide solution.
  - b. Prepare a blank by similarly treating 10 mL water.
  - c. After 1 minute, read the absorbance at 352 nanometers on a spectrophotometer, using a 1-cm path length.
  - d. If absorbance  $>0.1$ , do not use the alcohol.
2. Thorin Indicator, 1-(o-arsenophenylazo)-2-naphthol-3,6-disulfonic acid, disodium salt, or equivalent. Dissolve 0.20 g in 100 mL water.
3. Sulfuric Acid Standard, 0.0100 N. Purchase or standardize to  $\pm 0.0002$  N against 0.0100 N NaOH which has previously been standardized against potassium acid phthalate (primary standard grade).
4. Barium Standard Solution, 0.0100 N. Dissolve 1.95 g  $\text{Ba}(\text{ClO}_4)_2 \cdot 3\text{H}_2\text{O}$  in 200 mL water, and dilute to 1 L with isopropanol. Alternatively, 1.22 g  $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$  may be used instead of the perchlorate. Standardize this solution as follows:
  - a. Add 100 mL 100% isopropanol to 25 mL standard sulfuric acid solution.
  - b. Titrate with the barium perchlorate or barium chloride solution.

- c. Run duplicate analyses until titrations agree within  $\pm 1\%$  or  $\pm 0.2$  mL, whichever is larger, and average these titrations.
  - d. Calculate the normality using the average titration volume.
5. QA Audit Samples. Obtain from EPA (see QA 1).

**B. Analysis**

1. Note level of liquid in the sample container, and determine loss; note this loss, if any, on the LDS.
2. Transfer the contents of the storage container to a 100-mL volumetric flask, and dilute to exactly 100 mL with water.
3. Pipette a 20-mL aliquot of this solution into a 250-mL Erlenmeyer flask.
4. Add 80 mL 100% isopropanol and two to four drops thorin indicator.
5. Titrate to a pink endpoint using 0.0100 N barium standard solution.
6. Repeat steps 3 through 5 until duplicates agree within  $\pm 1\%$  or  $\pm 0.2$  mL, whichever is larger, and average the titration volumes.
7. Run a blank with each series of samples.
8. Concurrently analyze the two audit samples and a set of compliance samples.



**CALIBRATION PROCEDURE 6**  
**Metering System****A. Initial Calibration**

1. Leak-check the metering system (drying tube, needle valve, pump, rotameter, and DGM) from the inlet to the drying tube according to FP 3c, section A.
2. Remove the drying tube.
3. Connect a 1 L/rev wet test meter to the inlet of the metering system.
4. Make three independent calibrations runs, using at least five revolutions of the DGM per run.
5. Calculate the calibration factor Y for each run, and average the results (must be  $\leq \pm 2\%$  from the average).

**B. Post-test Calibration Check**

1. Do not conduct a leak-check.

2. Remove the drying tube. Connect a 1 L/rev wet test meter to the inlet of the metering system.
3. Make two or more independent runs, using at least three or more revolutions of the DGM per run.
4. Calculate the calibration factor Y for each run, and average the results (must be  $\leq \pm 5\%$  of  $Y_1$ . If not, recalibrate the metering system and for the calculations, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run.

**C. Alternative**

A dry gas meter calibrated for a standard may be used in place of the wet test meter in step A3. See CP 5c.

**CALIBRATION DATA SHEET 6**  
**Metering System**

Meter Box # \_\_\_\_\_ Date \_\_\_\_\_

Wet Test Meter # \_\_\_\_\_ Barometric Pressure, P<sub>b</sub> \_\_\_\_\_ in. Hg

Initial Calibration \_\_\_\_\_ Recalibration \_\_\_\_\_ Personnel \_\_\_\_\_

Run No.	Rotam. Rdg (cc/min)	WTM			DGM				Time θ (min)	Meter Coeff. Y <sub>i</sub>
		V <sub>w</sub> (L)	t <sub>w</sub> (°F)	Δm (in. H <sub>2</sub> O)	Vol (L)		Temp. (°F)			
					V <sub>d</sub>	V <sub>df</sub>	t <sub>di</sub>	t <sub>do</sub>		
									Avg, Y <sub>d</sub>	

$$Y_i = \frac{V_w \left( \frac{(t_{di} + t_{do})}{2} + 460 \right) \left( P_b + \frac{\Delta m_w}{13.6} \right)}{(V_{df} - V_{di}) (t_w + 460) P_b}$$

DGM Volume/Rev, V<sub>r</sub> = \_\_\_\_\_

Run No.	Initial Calibration		Re-Calibration	
	(V <sub>df</sub> - V <sub>d</sub> )/V <sub>r</sub>	Y <sub>i</sub> /Y <sub>d</sub>	(V <sub>df</sub> - V <sub>d</sub> )/V <sub>r</sub>	Y <sub>d(trc)'/Y<sub>d</sub></sub>
	<b>≥5.0</b>	<b>0.98 to 1.02 ?</b>	<b>≥3.0 ?</b>	<b>0.95 to 1.05 ?</b>

**QA/QC Check**  
 Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
 Personnel (Signature/Date) \_\_\_\_\_ Team Leader (Signature/Date) \_\_\_\_\_



**SUMMARY SHEET 6a**  
**Sulfur Dioxide (Alternative)**

		Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6			
Job No.		FDS 6			
Sampling Location		FDS 6			
Run ID#		FDS 6			
Test Date		FDS 6a			
Run Start Time		FDS 6a			
Run Finish Time		FDS 6a			
Traverse Points (if applicable)		FDS 1			
Net Run Time, min	$\theta$	FDS 6a			
Avg Cal Flow Rate, cfm	$\bar{Q}_{std}$	FDS 6a			
Barometric Pressure, in. Hg	$P_b$	FDS 6a			
Critical Orifice Inlet Vac during Cal, in. Hg	$P_c$	FDS 6a			
Critical Orifice Inlet Vac during Sampling, in. Hg	$P_{sr}$	FDS 6a			
Ambient Air Moisture Content, fraction	$B_{wa}$	FDS 6a			
Impinger Outlet Moisture Content, fraction	$B_{wo}$	FDS 6a			
Volume of Metered Gas Sample, dscf	$V_{m(std)}$	SS 6a			
Normality, Ba Perchlorate Titrant, meq/mL	$N$	LDS 6			
Volume of Sample Solution, mL	$V_s$	LDS 6			
Volume of Sample Aliquot Titrated, mL	$V_a$	LDS 6			
Average Volume Titrant for Sample, mL	$V_t$	LDS 6			
Volume Titrant for Blank, mL	$V_b$	LDS 6			
SO <sub>2</sub> Concentration, lb/dscf	$C_{SO_2}$	SS 6a			
Post-test Calibration Checks					
Temperature and Barometer		CDS 2d			
Metering System		FDS 6a			

$$V_{m(std)} = Q_{std} \theta (1 - B_{wa}) \sqrt{\frac{M_a (1 - B_{wa})}{M_d (1 - B_{wo})} \left( \frac{P_b + P_{sr}}{P_b + P_c} \right)}$$

$$C_{SO_2} = 7.061 \times 10^{-5} \frac{(V_t - V_{tb}) N \left( \frac{V_s}{V_a} \right)}{V_{m(std)}}$$

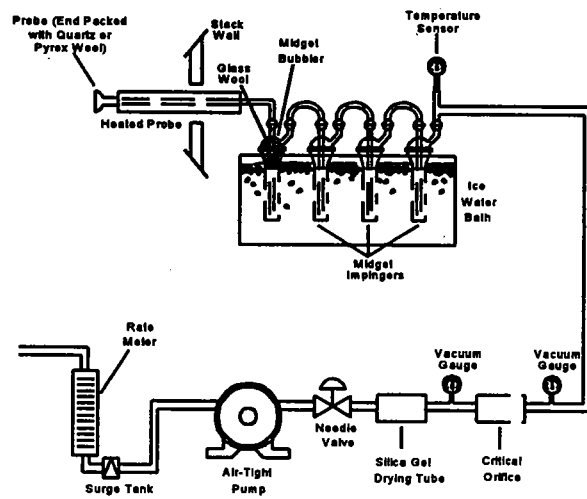


Figure F6a-1. SO<sub>2</sub> Sampling Train using a Critical Orifice.

**FIELD PROCEDURE 6a**  
**Critical Orifice Sampling Train**

**Note:** This procedure describes the technique for sampling trains using critical orifices. The midjet impinger trains are as specified, e.g., Method 6.

1. Prepare the sampling train as shown in Figure F6a-1.
2. *Optional:* Leak-check the sampling train (see FP 3c, sections C and D); add surge tank before rotameter).
3. Determine the %moisture of the ambient air using the wet and dry bulb temperatures or, if appropriate, a relative-humidity meter.
4. Calibrate the entire sampling train as follows:
  - a. Attach a 500-cc soap bubble meter to the inlet of the probe.
  - b. Set the outlet vacuum 1 to 2 in. Hg above the critical vacuum.
  - c. Determine the volumetric flow rate (see FDS 6a).
  - d. Calculate the standard volume of air measured by the soap bubble meter and standard volumetric flow rate.
5. Use the same vacuum used during the calibration run. Start the watch and pump simultaneously.
6. Take readings as shown in FDS 6a at least every 5 min.
7. At the end of the sampling run, stop the watch and pump simultaneously.
8. Conduct a post-test calibration run as in step 4 (see FDS 6a).
9. Average  $Q_{std}$  from both calibration runs.
10. Calculate the sample gas volume  $V_{m(std)}$ .
11. Determine the ratio of the molecular weights of air to stack gas,  $M_a/M_g$ . If this ratio is 0.97 to 1.03, the term  $(M_a/M_g)^{1/2}$  may be dropped from the equation (see SS 6a).
12. Drain the ice bath, and purge the sampling train by drawing clean ambient air through the system for 15 min. Pass air through a charcoal filter or through an extra midjet impinger with 15 mL 3%  $H_2O_2$  or use ambient air without purification.

**FIELD DATA SHEET 6a**  
**Critical Orifice Sampling Train**

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date/Time \_\_\_\_\_

Train ID# \_\_\_\_\_ Personnel \_\_\_\_\_

Crit. Orifice ID# _____	Crit. Vac. _____ in. Hg			
	1	2	3	4
Run No.				
Bubble Meter Vol, $V_{sb}$ cc				
Time, $\theta$ sec				
Bar. Press., $P_b$ in. Hg				
Amb Temp., $t_{amb}$ °F				
Inlet Vac. $P_c$ in. Hg $\geq$ Crit. Vac.??				
Outlet Vac., in. Hg				
Flow Rate, $Q_{std}$ cc/min				
Average $\bar{Q}_{std}$ cc/min				
Post $\bar{Q}_{std}$ /Pre $\bar{Q}_{std}$	<i>(0.95 to 1.05?)</i>			
Average Pre/Post $Q_{std}$ , cfm				

Elapsed Time (min)	Outlet Temp (°F)	Vacuum (in. Hg)		Rotam Rdg (cc/min)
		Inlet	Outlet	

Dry Molecular Weight of Stack Gas,  $M_d$  \_\_\_\_\_ lb/lb-mole

Moisture Content of Amb. Air,  $B_{wa}$  \_\_\_\_\_ (fraction)

Moisture Content of Impinger Outlet,  $B_{wo}$  \_\_\_\_\_ (fraction)

$$Q_{std} = 1059 \frac{V_{sb}}{\theta} \frac{P_b}{(t_{amb} + 460)}$$

$$\text{Avg Pre/Post } Q_{std} = 3.531 \times 10^{-5} \bar{Q}_{std}$$

**QA/QC Check**

Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_

Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
 Personnel (Signature/Date)

\_\_\_\_\_  
 Team Leader (Signature/Date)

**CALIBRATION PROCEDURE 6a**  
**Critical Orifice**

**Note:** Critical orifices used in midget type impinger trains are calibrated in the field. This CP covers the selection and check for suitability.

1. Select a critical orifice for the desired flow rate. The needle sizes and tubing lengths shown below give the following approximate flow rates.

Approximate Sizes/Flow Rates for Critical Orifices			
Gauge/cm	cc/min	Gauge/cm	cc/min
21/7.6	1100	23/3.8	500
22/2.9	1000	23/5.1	450
22/3.8	900	24/3.2	400

2. To adapt these needles to a Method 6 type sampling train, do the following.
  - a. Insert sleeve type, serum bottle stoppers into two reducing unions.
  - b. Insert the needle into the stoppers as shown in Figure F6a-1.

3. Determine suitability and the appropriate operating vacuum of the critical orifices as follows:
  - a. Temporarily attach a rotameter and surge tank to the outlet of the sampling train.
  - b. Turn on the pump, and adjust the valve to give an outlet vacuum reading corresponding to about half of the atmospheric pressure.
  - c. Observe the rotameter reading. Slowly increase the vacuum until a stable reading (critical vacuum) is obtained on the rotameter and record this value.
  - d. Do not use orifices that do not reach a critical value.
4. Identify the critical orifice.

**SUMMARY SHEET 6A**  
**Sulfur Dioxide, Carbon Dioxide, and Moisture**

Method (circle) 6A 6B

		Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6			
Job No.		FDS 6			
Sampling Location		FDS 6			
Run ID#		FDS 6			
Test Date		FDS 6			
Run Start Time		FDS 6			
Run Finish Time		FDS 6			
Traverse Points (if applicable)		FDS 1			
Net Run Time, min	0	FDS 6			
Dry Gas Meter Calibration Factor	Y	CDS 6			
Barometric Pressure, in. Hg	P <sub>b</sub>	FDS 6			
Average DGM Temperature, °F	t <sub>m</sub>	FDS 6			
Absolute Average DGM Temperature, R	T <sub>m</sub>	SS 6			
Volume of Metered Gas Sample, dcf	V <sub>m</sub>	FDS 6			
Volume of Metered Gas Sample, dscf	V <sub>m(std)</sub>	SS 6			
CO <sub>2</sub> Absorber, Initial Weight, g	m <sub>ai</sub>	FDS 6A			
CO <sub>2</sub> Absorber, Final Weight, g	m <sub>af</sub>	FDS 6A			
Volume CO <sub>2</sub> , scf	V <sub>CO2(std)</sub>	SS 6A			
CO <sub>2</sub> Concentration, % dry	C <sub>CO2</sub>	SS 6A			
Moisture, Initial Weight, g	m <sub>wi</sub>	FDS 6A			
Moisture, Final Weight, g	m <sub>wf</sub>	FDS 6A			
Volume Moisture, scf	V <sub>w(std)</sub>	SS 6A			
Moisture Concentration, %	C <sub>w</sub>	SS 6A			
Normality, Ba Perchlorate Titrant, meq/mL	N	LDS 6			
Volume of Sample Solution, mL	V <sub>s</sub>	LDS 6			
Volume of Sample Aliquot Titrated, mL	V <sub>a</sub>	LDS 6			
Average Volume Titrant for Sample, mL	V <sub>t</sub>	LDS 6			
Volume Titrant for Blank, mL	V <sub>b</sub>	LDS 6			
SO <sub>2</sub> Concentration, lb/dscf	C <sub>SO2</sub>	SS 6			
Carbon F-factor, scf/mmBtu	F <sub>c</sub>	M-19			
SO <sub>2</sub> Emission Rate, lb/mmBtu	E <sub>SO2</sub>	SS 6A			
Post-test Calibration Checks					
Temperature and Barometer		CDS 2d			
Metering System		CDS 6			

$$V_{\text{CO}_2(\text{std})} = 5.467 \times 10^{-4} (m_{\text{af}} - m_{\text{ai}})$$

$$C_w = \frac{V_{w(\text{std})}}{V_{m(\text{std})} + V_{w(\text{std})} + V_{\text{CO}_2(\text{std})}} \times 100$$

$$C_{\text{CO}_2} = \frac{V_{\text{CO}_2(\text{std})}}{V_{m(\text{std})} + V_{\text{CO}_2(\text{std})}} \times 100$$

$$m_{\text{SO}_2} = 32.03 (V_t - V_{\text{tb}}) N \left( \frac{V_s}{V_a} \right)$$

$$V_{w(\text{std})} = 1.336 \times 10^{-3} (m_{\text{wf}} - m_{\text{wi}})$$

$$E_{\text{SO}_2} = F_c (1.829 \times 10^9) \frac{m_{\text{SO}_2}}{(m_{\text{af}} - m_{\text{ai}})}$$

**FIELD PROCEDURE 6A**  
**Sulfur Dioxide, Moisture, and Carbon Dioxide**

**A. Pre-test Preparation**

1. Prepare the sampling train as shown in Figure F6A-1.
  - a. Add 15 mL 80% isopropanol into the first midget bubbler. Insert glass wool into the top of the isopropanol bubbler.
  - b. Add 15 mL 3% H<sub>2</sub>O<sub>2</sub> into each of the first two midget impingers.
  - c. Add about 25 g Drierite to fourth vessel.
2. Clean the outsides of the bubblers and impingers, and weigh simultaneously all four vessels at room temperature (20°C) to ±0.1 g.
3. Prepare the CO<sub>2</sub> absorber as shown in Figure F6A-2. Check the absorber by rotating the cylinder in a horizontal position. The CO<sub>2</sub> absorbing material should not shift or have open spaces or channels.
4. Clean and dry the outside of the cylinder, and weigh at room temperature to ±0.1 g. Assemble the train as shown in Figure F6A-1.
5. Adjust the probe heater to a temperature sufficient to prevent condensation.
  - a. Downstream of wet scrubbers, use a heated out-of-stack filter (either borosilicate glass wool or glass fiber mat). The filter may be within the heated section of the sampling probe, but not within 15 cm of the probe inlet or any unheated section of the probe.
  - b. Heat the probe and filter to ≥20°C above the source temperature, but not >120°C.
6. Place crushed ice and water around the impingers and bubblers to cover at least two-thirds of their length.
7. Mount the CO<sub>2</sub> absorber outside the water bath in a vertical flow position with the sample gas inlet at the bottom.

**B. Sample Concentration Sampling**

1. Collect the sample following FP 6, section B. Remove the CO<sub>2</sub> absorber after the leak-check and before purging of the sampling train.
2. After purging, disconnect the isopropanol bubbler and the impingers.
  - a. Allow about 10 min for them to reach room temperature.
  - b. Clean and dry the outsides, and weigh them simultaneously.

**C. Sample Recovery**

1. Discard (or save, if desired) the contents of the isopropanol bubbler. Transfer the contents of the midget impingers into a leak-free polyethylene bottle for shipping.
  - a. Rinse the two midget impingers and connecting tubes with water, and add to the same storage container.
  - b. Mark the fluid level. Seal and identify the sample container.
2. Allow about 10 min for the CO<sub>2</sub> absorber to warm to room temperature, clean and dry the outside, and weigh to ±0.1 g. Discard used Ascarite II material.

**D. Post-test Calibrations**

Conduct post-test calibration checks of metering system and temperature gauges. (See CP 2d, CP 2e, and CP6).

**E. Emission Rate Sampling for FP 6A and 6B**

When only the emission rate of SO<sub>2</sub> (ng/J) is needed, use the same procedure as that in FP 6a, except for the following;

1. A dry gas meter is not needed (see Figure F6A-1).
2. The weighing steps of the isopropanol bubbler, the SO<sub>2</sub> absorbing impingers or the moisture absorber (steps A2 and B2 of FP 6A) may be omitted.
3. During sampling, dry gas meter readings, barometric pressure, and dry gas meter temperatures need not be recorded.

**F. Alternatives/Suggestions**

1. Other types of impingers and bubblers, such as Mae West for SO<sub>2</sub> collection and rigid cylinders for moisture absorbers containing Drierite, may be used with proper attention to reagent volumes and levels.
2. Flexible tubing, e.g., Tygon, may be used to connect the last SO<sub>2</sub> absorbing bubbler to the Drierite absorber and to connect the Drierite absorber to the CO<sub>2</sub> absorber.
3. A second, smaller CO<sub>2</sub> absorber containing Ascarite II may be added in line downstream of the primary CO<sub>2</sub> absorber as a breakthrough indicator. Ascarite II turns white when CO<sub>2</sub> is absorbed.
4. A heated Teflon connector may be used to connect the filter holder or probe to the first impinger.

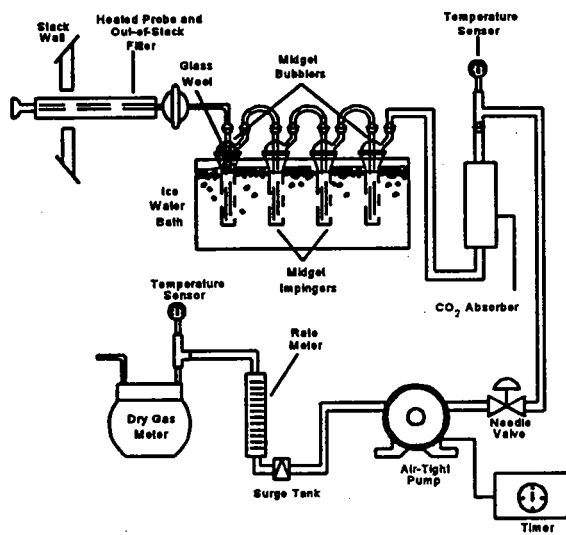


Figure F6A-1. Sampling Train.



**FIELD DATA SHEET 6A  
Moisture and Carbon Dioxide**

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

Test Location/Run # \_\_\_\_\_ Personnel \_\_\_\_\_

*Note: Use FDS 6 or 6B for SO<sub>2</sub> data and attach this data sheet.*

Moisture Determination: Bubblers/Impingers			
Initial wgt, m <sub>wi</sub> (g)			
Final wgt, m <sub>wf</sub> (g)			
CO <sub>2</sub> Determination: CO <sub>2</sub> Absorber			
Initial wgt, m <sub>ai</sub> (g)			
Final wgt, m <sub>af</sub> (g)			

QA/QC Check  
 Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
 Personnel (Signature/Date) Team Leader (Signature/Date)

**FIELD PROCEDURE 6B**  
**Sulfur Dioxide and Carbon Dioxide Daily Average Emissions**

*Note: This procedure is a modification of Method 6A to obtain a 24-hr sample.*

**A. Sample Preparation**

Prepare the sample train in the same way as that in FP 6A, except for the following:

1. Do not include the isopropanol bubbler. An empty bubbler may be used in its place.
2. Include a filter (either in-stack, out-of-stack, or both).
3. For the probe heat, use a continuous electric supply that is separate from the timed operation of the sample pump.
4. Include a timer switch.

**B. Sampling**

Follow the procedure in FP 6A, except for the following:

1. Adjust the timer-switch to operate in the "on" position from 2 to 4 min on a 2-hr repeating cycle or other specified cycle.
2. Cover and protect the impingers and bubbler tank from intense heat and direct sunlight. In freezing conditions, protect the impinger solution and the water bath from becoming frozen.
3. When the pump is started, start the timer also. Ensure that the timer is operating as intended, i.e., in the "on" position for the desired period and the cycle repeats as required.
4. During the 24-hr sampling period, record the dry gas meter temperature one time between 9:00 a.m. and 11:00 a.m., and the barometric pressure.
5. At the conclusion of the run, turn off the timer and the sample pump, remove the probe from the stack, and record the final gas meter volume reading.
6. After 30 days of operation of the test train, check the calibration of the metering system according to CP 6, section B. If the recalibration factor has deviated from its previous calibration by  $\geq \pm 5\%$ , for the preceding 30 days of data, use the calibration factor (initial or recalibration) that yields the lower gas volume for each test run. Use the latest calibration factor for succeeding tests.

7. Recalibrate temperature gauges and field barometer at 30-day intervals. (See FP 3c and 3d.)

**C. Alternatives/Suggestions**

1. Other sampling equipment, such as Mae West bubblers and rigid cylinders for moisture absorption, which requires sample or reagent volumes other than those specified in this procedure for full effectiveness may be used.
2. Rather than intermittent operation, Method 6B may be operated at low flow rates (<100 mL/min). In this case, molecular sieve material may be substituted for Ascarite II as the CO<sub>2</sub> absorbing material, e.g., Union Carbide 1/16 inch pellets, 5 Å, or equivalent. Do not discard the molecular sieve material, but regenerate it per the manufacturer's instruction.
3. Sampling may be conducted continuously if a low flow-rate sample pump (20 to 40 mL/min for the reagent volumes described in this method) is used. Then the timer-switch is not necessary. In addition, if the sample pump is designed for constant rate sampling, the rate meter may be deleted. The total gas volume collected should be between 25 and 60 L for the amounts of sampling reagents prescribed in this method.
4. Use glass probes or corrosion resistant types of stainless steel, e.g., Hasteloy or Carpenter 20.
5. The emission rate procedure FP 6A is also applicable to this method.

**FIELD DATA SHEET 6B**  
**24-Hour Sampling Train**

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date \_\_\_\_\_

Test Location/Run # \_\_\_\_\_ Personnel \_\_\_\_\_

Train ID#/Sample Box # \_\_\_\_\_ DGM Cal Coef., Y \_\_\_\_\_

Trav. Pt.	Clock Time	DGM		Rotameter Rdg (cc/min)	Bar. Press. P <sub>b</sub> (in. Hg)
		Vol Rdg, V <sub>d</sub> (cf)	Temp., t <sub>d</sub> (°F)		

\_\_\_ Proper probe heat (no condensation)?

**Sample Recovery**

\_\_\_ Leak rate at  $\leq 10$  in. Hg vacuum = \_\_\_\_\_  
 ( $\leq 2\%$  of sampling rate)?

\_\_\_ Fluid level marked?

\_\_\_ Purge rate = \_\_\_\_\_ (at avg rotam. rdg)?

\_\_\_ Sample container sealed?

\_\_\_ Purge time = \_\_\_\_\_ ( $\leq 15$  min)?

\_\_\_ Sample container identified?

**Post-Test Calibrations** (at 30-day intervals)

Attach CDS 2d and CDS 6. Temperature specification is  $\pm 5.4$  °F?

**QA/QC Check**

Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
 Personnel (Signature/Date)

\_\_\_\_\_ Team Leader (Signature/Date)

**SUMMARY SHEET 6C**  
**Sulfur Dioxide**

		Run #1	Run #2	Run #3	Avg
Client/Plant Name		FDS 6C			
Job No.		FDS 6C			
Sampling Location		FDS 6C			
Run ID #		FDS 6C			
Test Date		FDS 6C			
Run Start Time		FDS 6C			
Run Finish Time		FDS 6C			
Average Gas Concentration, dry basis, ppm	$\bar{C}$	FDS 6C			
Avg System Cal Bias Check Responses for Zero Gas, ppm	$C_o$	FDS 6C			
Avg System Cal Bias Check Responses for Upscale Cal Gas, ppm	$C_m$	FDS 6C			
Actual Conc of Upscale Cal Gas, ppm	$C_{ma}$	FDS 6C			
Effluent Gas Concentration, dry basis, ppm	$C_{gas}$	SS 6C			
Interference Check Value from FP 6Ca	$C_{ic}$	SS 6			
Post-test Calibration Check					
System calibration bias check		FDS 6C			

$$C_{gas} = (\bar{C} - C_o) \frac{C_{ma}}{C_m - C_o}$$

**FIELD PROCEDURE 6C**  
**Sulfur Dioxide**  
**(Instrumental Analyzer Procedure)**

**A. Preparations**

1. Obtain SO<sub>2</sub> calibration gases (SO<sub>2</sub> in N<sub>2</sub> or SO<sub>2</sub> in air or gas mixtures of SO<sub>2</sub>/CO<sub>2</sub>, SO<sub>2</sub>/O<sub>2</sub> or SO<sub>2</sub>/CO<sub>2</sub>/O<sub>2</sub> in N<sub>2</sub>).
2. For fluorescence-based analyzers, use calibration gases that contain concentrations of O<sub>2</sub> and CO<sub>2</sub> within  $\pm 1\%$  O<sub>2</sub> and  $\pm 1\%$  CO<sub>2</sub> of that of the effluent samples introduced to the analyzer or, alternatively, use SO<sub>2</sub> in air and correction factors for O<sub>2</sub>/CO<sub>2</sub> quenching.
3. Use three calibration gases as specified below:
  - a. High-Range. 80 to 100% of span.
  - b. Mid-Range. 40 to 60% of span.
  - c. Zero. SO<sub>2</sub> concentration  $< 0.25\%$  of span.
4. For Protocol 1 gases, obtain a certification from the gas manufacturer that Protocol 1 was followed.
5. For non-Protocol 1 gases, obtain gases within manufacturer's tolerance of  $\leq \pm 2\%$  of tag value. Using Method 6, analyze the gases in triplicate until each run is  $\leq \pm 5\%$  or  $\pm 5$  ppm, whichever is greater, of average. See CDS 6Ca.
  - a. If  $C_{avg} \leq \pm 5\%$  of manufacturer's tag value, use tag value.
  - b. If  $C_{avg} > \pm 5\%$ , conduct at least three additional analyses until the results of six *consecutive* runs agree within  $\pm 5\%$  or  $\pm 5$  ppm, whichever is greater, of the average. Then use this average for the cylinder value.
6. Prepare and calibrate the gas analyzer and data recorder. Adjust system components as necessary.

**B. Analyzer Calibration Error**

*Conduct this test initially and each time the system exceeds the system bias and drift specifications.*

1. Introduce the zero, mid-range, and high-range gases to the measurement system at any point upstream of the gas analyzer. Do not make any adjustments to the system except those necessary to adjust the calibration gas flow rate at the analyzer.
2. Record the analyzer responses to each calibration gas on CDS 6C.

**C. Sampling System Bias Check**

Conduct this bias check *initially* and *after each sampling run*.

1. Introduce the calibration gases at the calibration valve installed at the outlet of the sampling probe. Operate the system at the normal sampling rate, and make no adjustments to the measurement system other than those necessary to adjust the calibration gas flow rates at the analyzer. Wait until a stable response is achieved before taking readings.
2. Introduce either the mid-range or high-range gas, whichever is closest to the effluent concentrations, and record the analyzer response and the time it took to reach a stable response on FDS 6C.
3. Introduce zero gas, and record the analyzer response and the time it took to reach a stable response.

**D. Emission Test Procedure**

1. Select the sampling site and sampling points as in Method 6. Set up the sampling system as shown in Figure F6C-1.
2. Sample at each measurement point using the same sampling rate as that used during the sampling system bias check. Maintain constant sampling rate (i.e.,  $\pm 10\%$ ) during the entire run.
3. Use the same sampling time per run as that used for Method 6 plus twice the stable response time for the instrument. Then determine the average effluent concentration.
4. Use the following options to determine the average gas concentration.
  - a. By integration of the area under the curve for chart recorders.
  - b. By averaging measurements recorded at equally spaced intervals over the entire run: Runs  $\leq 1$  hr must have recorded measurements at 1-minute intervals or a minimum of 30 measurements, whichever is less restrictive and runs  $> 1$  hr must have measurements at 2 min intervals or a minimum of 96 measurements, whichever is less restrictive.

**E. Post-Run Tests**

1. Following each run, or before adjustments are made to the measurement system during the run, determine the sampling system bias. Do not make any adjustments to the measurement system until after the drift checks are completed. Record the system responses on FDS 6C.
2. If the sampling system does not pass the bias test at either the zero or upscale calibration values, void the run. Repeat the calibration error and bias tests before the next run.
3. If the sampling system passes the bias check, calculate the zero and upscale calibration drift to determine whether the calibration error and system bias tests must be conducted before the next run.

**F. Alternatives**

1. Step A3c. For zero gas, ambient air may be used by purifying the air through a charcoal filter or through one or more impingers containing a solution of 3%  $H_2O_2$ .
2. A calibration curve established prior to the analyzer calibration error check may be used to convert the analyzer response to the equivalent gas concentration introduced to the analyzer. However, the same correction procedure shall be used for all effluent and calibration measurements obtained during the test.

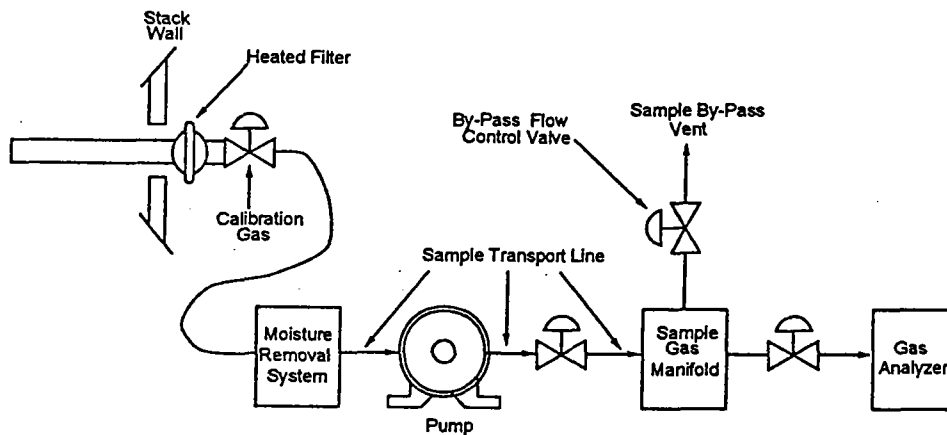


Figure F6C-1. Measurement System Schematic.

**FIELD DATA SHEET 6C**  
Analyzer Calibration Bias and Drift

Method \_\_\_\_\_

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date \_\_\_\_\_

Test Location \_\_\_\_\_ Personnel \_\_\_\_\_

**Note:** Indicate units. Analyzer ID# \_\_\_\_\_ Span \_\_\_\_\_ Upscale Value,  $C_{ma}$  \_\_\_\_\_

**Note:** Conduct this test initially and after each sampling run. Introduce gas at probe outlet.

If interference test is required, attach appropriate data sheets from Method 6. Avg Syst Resp = (Pre + Post)/2  
(M 6C results  $\leq$  7% of M 6 results?)  $C_m$  = Upscale,  $C_o$  = Zero Avg Syst Resp

Run No.	Level	Time	Analyzer Resp	Pre		Post		% Drift	Stable Resp Time	Avg Syst Resp
				System Resp	System %Bias	System Resp	System %Bias			
1	Upscale									
	Zero									
2	Upscale									
	Zero									
3	Upscale									
	Zero									
4	Upscale									
	Zero									
5	Upscale									
	Zero									
6	Upscale									
	Zero									
7	Upscale									
	Zero									
8	Upscale									
	Zero									
9	Upscale									
	Zero									
10	Upscale									
	Zero									
11	Upscale									
	Zero									
12	Upscale									
	Zero									

\_\_\_ Normal operation and no adjustments to system except to adjust calibration gas flow rates at analyzer?  
 \_\_\_ %Syst Bias = 100 (Syst Resp - Anal Resp)/Span ( $\leq \pm 5\%$  of span?) \_\_\_ %Drift = Post - Pre ( $\leq \pm 3\%$  of span?)  
 \_\_\_ Failure of bias test (or exceeding cal drift spec) requires repeat of cal error (CDS 6C) and bias tests before next run.

**QA/QC Check**  
 Completeness \_\_\_ Legibility \_\_\_ Accuracy \_\_\_ Specifications \_\_\_ Reasonableness \_\_\_

Checked by: \_\_\_\_\_ Personnel (Signature/Date) \_\_\_\_\_ Team Leader (Signature/Date) \_\_\_\_\_

**FIELD DATA SHEET 6C (Continued)**  
**Sample Concentration**

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date \_\_\_\_\_

Test Location \_\_\_\_\_ Personnel \_\_\_\_\_

**Note: Indicate units.**

***Void sample runs that fail the bias test (see FDS 6C, pg 1).***

Run No.	Meas Pt	Time	Sampl Rate	Syst Resp	Conc	Run No.	Meas Pt	Time	Sampl Rate	Syst Resp	Conc

\_\_\_\_ Sampling rate same (within ± 10%) as that used during the sampling system bias check at each measurement point?

\_\_\_\_ Sampling time includes twice the average stable system response time before average concentration determined?

**QA/QC Check**

Completeness \_\_\_\_ Legibility \_\_\_\_ Accuracy \_\_\_\_ Specifications \_\_\_\_ Reasonableness \_\_\_\_

Checked by: \_\_\_\_\_  
Personnel (Signature/Date)

\_\_\_\_\_   
Team Leader (Signature/Date)



**CALIBRATION DATA SHEET 6C**  
Analyzer Calibration Error

Method \_\_\_\_\_

Client/Plant Name \_\_\_\_\_ Job # \_\_\_\_\_

City/State \_\_\_\_\_ Date/Time \_\_\_\_\_

Test Location \_\_\_\_\_ Personnel \_\_\_\_\_

Type of Calibration Gas:  Protocol 1 (attach manufacturer's certification)  Analysis (attach CDS 6Ca)

**Fluorescence-based Analyzers:**  Cal gas SO<sub>2</sub>/CO<sub>2</sub>/O<sub>2</sub> in N<sub>2</sub> with O<sub>2</sub> and CO<sub>2</sub> within ±1% O<sub>2</sub> or ±1% CO<sub>2</sub> of effluent concentration; or.  Correction factors for O<sub>2</sub>/CO<sub>2</sub> attached.

*Conduct this test initially and each time system fails system bias/drift specs. Introduce gas at any point upstream of analyzer.*

**Note:** Indicate units. Analyzer ID# \_\_\_\_\_ Span \_\_\_\_\_

Run No.	Level	Cylinder Value	Analyzer Response	Absolute Difference	%Cal Error (of span) (≤2%?)
1	Zero				
	Mid-range				
	High-range				
2	Zero				
	Mid-range				
	High-range				
3	Zero				
	Mid-range				
	High-range				
4	Zero				
	Mid-range				
	High-range				
5	Zero				
	Mid-range				
	High-range				
6	Zero				
	Mid-range				
	High-range				

$$\%Cal Error = \frac{Absolute\ Difference}{Span} \times 100$$

No adjustments made to system except for adjusting flow rate of calibration gases at the analyzer?

**QA/QC Check**

Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_

Personnel (Signature/Date)

\_\_\_\_\_

Team Leader (Signature/Date)

**FIELD PROCEDURE 6Ca**  
Interference Check

**Note:** For each individual analyzer, conduct this interference check for at least three runs during the initial field test on a particular source category. Retain the results, and report them with each test performed on that source category. Use SS 6, FDS 6, and LDS 6.

1. Assemble the modified Method 6 train as shown in Figure 6Ca-1, and install the sampling train to obtain a sample at the by-pass discharge vent of measurement system.
2. Record the initial dry gas meter reading.
3. Open the flow control valve concurrent with the initiation of the sampling period, and adjust the flow to 1 L/min ( $\pm 10\%$ ).  
(**Note:** Avoid over-pressurizing the impingers and causing leakage.)
4. Record appropriate data as shown in FDS 6.
5. At the end of the test run, record the final dry gas meter reading.
6. Recover and analyze the contents of the midget impingers, and determine the  $\text{SO}_2$  gas concentration using the procedures of Method 6 (see LDS 6). Determine the average gas concentration exhibited by the analyzer for the run (see SS 6).

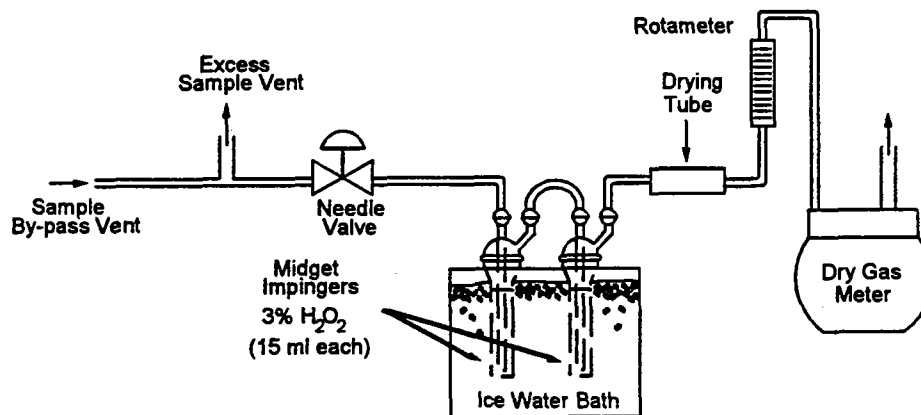


Figure F6Ca-1. Interference Check Sampling Train.

**CALIBRATION DATA SHEET 6Ca**  
**Analysis of Calibration Cylinder Gases**

Date \_\_\_\_\_ (Must be  $\leq 6$  months before test) Span \_\_\_\_\_

Cylinder ID#: Zero: \_\_\_\_\_ Mid: \_\_\_\_\_ High: \_\_\_\_\_

Methods \_\_\_\_\_ Personnel \_\_\_\_\_

Attach appropriate field, laboratory, calibration data sheets (List): \_\_\_\_\_

Run No.	Zero	Mid-Range	High-Range
1			
2			
3			
4			
5			
6			
Average	(<0.25% of span?)	(40%-60% of span?)	(80%-100% of span?)
Max % Dev			
Tag Value, ppm			

\_\_\_\_ Runs in triplicate or sextuplet sets are consecutive?

Specification	Method 6	Method 7	Method 3
Max % Dev from Average*	$\leq \pm 5\%$ or $\pm 5$ ppm	$\leq \pm 10\%$ or $\pm 10$ ppm	$\leq \pm 5\%$ or $\pm 0.2\%$ abs
Average Diff from Tag Value*	$\leq \pm 5\%$ or $\pm 5$ ppm	$\leq \pm 10\%$ or $\pm 10$ ppm	$\leq \pm 5\%$ or $\pm 0.2\%$ abs

\* Whichever is greater.  
 If avg diff from tag value > specification, use the avg of the 6 runs as the cylinder value.

**QA/QC Check**  
 Completeness \_\_\_\_\_ Legibility \_\_\_\_\_ Accuracy \_\_\_\_\_ Specifications \_\_\_\_\_ Reasonableness \_\_\_\_\_

Checked by: \_\_\_\_\_  
 Personnel (Signature/Date) Team Leader (Signature/Date)

