

SUMMARY SHEET 106
Vinyl Chloride

		Run #1	Run #2	Run #3	Avg
Client/Plant Name					FDS 106
Job No.					FDS 106
Sampling Location					FDS 106
Run ID #					FDS 106
Test Date					FDS 106
Run Start Time					FDS 106
Run Finish Time					FDS 106
Barometric Pressure, in. Hg	P_b				FDS 106
Ambient Temperature, °F	t				FDS 106
Velocity Pressure, in. H ₂ O	Δp				FDS 106
% Proportional	%P				FDS 106
Vinyl Chloride Analyzed, ppm	C_c				LDS 106
Bar. Pressure During Cal., mm Hg	P_r				LDS 106
Bar. Pressure During Analysis, mm Hg	P_i				LDS 106
Loop Temp. During Analysis, K	T_i				LDS 106
Loop Temp. During Cal., K	T_r				LDS 106
Lab Ambient Temperature, °C	t_{amb}				LDS 106
Moisture Content in Bag, fraction	B_{wb}				LDS 106
Vinyl Chloride in Bag, ppm	C_b				SS 106

$$C_b = \frac{C_c P_r T_i}{P_i T_r (1 - B_{wb})}$$

FIELD PROCEDURES 106
Vinyl Chloride

A. Pretest Preparation.

1. **Mandatory:** Leak check the bags according to FP 3b. Check the rigid container for leaks in the same manner.
2. For each sample bag in its rigid container, place a rotameter in line between the bag and the pump inlet. Evacuate the bag. A rotameter reading going to zero when the bag appears empty indicates no leaks.
3. Establish the sampling rate at half the bag volume divided by the sampling time.

B. Preparation of Sampling Train

1. Assemble the sample train as shown in Figure 106-1.
2. Join the quick connects as illustrated, and ensure all connections are tight.
3. Place the end of the probe at the centroid of the stack and start the pump with the needle valve adjusted to the desired rate.

4. Allow enough time to purge the line several times, change the vacuum line from the container to the bag and evacuate the bag until the rotameter indicates no flow.

C. Sampling

1. Protect the bag container from sunlight.
2. Reposition the sample and vacuum lines and sample at a rate proportional to the stack velocity. Direct the gas exiting the rotameter away from sampling personnel at all times. Record the information shown on FDS 106.
3. At the end of sampling, shut off the pump, disconnect the sample line from the bag, and disconnect the vacuum line from the bag container.
4. Keep the sample bags out of direct sunlight until analysis.

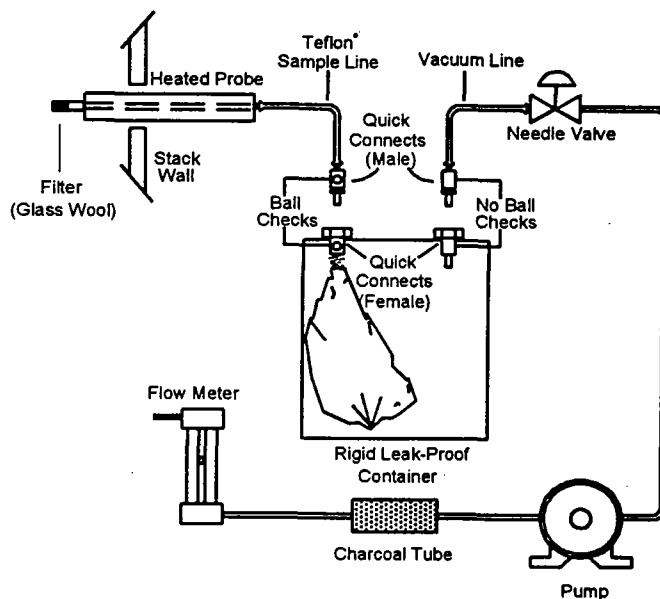


Figure F106-1. Integrated-Bag Sampling Train.

FIELD DATA SHEET 106
Vinyl Chloride

Client/Plant Name _____ Job # _____

City/State _____ Test Location _____

Run # _____ Personnel _____

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

- Pre-test leak check acceptable? Bag evacuated until rotameter reads zero?
 Sample line purged several times before sampling? Container protected from sunlight during sampling?

Time	Rotameter reading	Δp (in. H ₂ O)	√Δp	% Dev
		Avg		

$$\%Dev = \frac{\sqrt{\Delta p}}{(\sqrt{\Delta p})_{avg}} \quad \%Proportional = Highest \%Dev$$

- Sample rate kept proportional to the stack velocity?
 Bag sample at least half full?
 Bag sample stored out of the sunlight?

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

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

LABORATORY PROCEDURE 106
Vinyl Chloride

A. Equipment Preparation

1. Set column to 100°C and detector to 150°C.
2. Determine and maintain optimum H₂ and O₂ flow rates during all chromatography operations.
3. Using zero helium or N₂ as the carrier gas, establish a flow rate in the range consistent with the manufacturer's requirements for satisfactory detector operation. A flow rate of ~40 mL/min should produce adequate separations.
4. Observe the base line periodically and determine that the noise level has stabilized and that base line drift has ceased.
5. Purge the sample loop for 30 sec at 100 mL/min, shut off flow, allow the sample loop pressure to reach atmospheric pressure as indicated by the water manometer, then activate sample valve to analyze the sample.

B. Calibration

1. Obtain three manufacturer-certified cylinder gas standards of vinyl chloride (VC) having concentrations (C_c) of 5, 10, and 50 ppm.
2. Analyze the zero gas and each gas standard. Record the information indicated in LDS 106.
3. Measure VC peak area A_m by a disc integrator, electronic integrator, or a planimeter.
4. Calculate $A_c = A_m A_f$ (attenuator setting). Repeat until two consecutive injection areas are within 5%, then plot the average of those two values versus C_c. Draw a straight line through the points derived by the least squares method.
5. Determine the retention time (the distance on the chart from the time of injection time to the time at which the peak maximum occurs divided by the chart speed).
6. Perform calibration daily, or before and after the analysis of each emission test set of bag samples, whichever is more frequent. For each group of sample analyses, use the average of the two calibration curves which bracket that group to determine the respective sample concentrations.
7. If the two calibration curves differ by more than 5% from their mean value, then report the final results by both calibration curves.

8. Immediately after preparing the calibration curve and before analyzing the samples, Analyze the audit samples described in Appendix C, Procedure 2: "Procedure for Field Auditing GC Analysis."

C. Sample Preparation

1. With a new piece of Teflon tubing identified for that bag, connect a bag inlet valve to the GC sample valve. Switch the valve to receive gas from the bag through the sample loop.
2. Arrange the equipment so the sample gas passes from the sample valve to a 100-mL/min rotameter with flow control valve followed by a charcoal tube and a 1-in. H₂O pressure gauge.
3. Maintain sample flow by a vacuum pump or container pressurization if the collection bag remains in the rigid container.
4. After purging the sample loop, allow the pressure gauge to return to zero before activating the gas sampling valve.

D. Sample Analysis

1. Record the data indicated in LDS 106. Mark the position of the pen on the chart at the time of sample injection.
2. From the chart, note the peak having the retention time corresponding to vinyl chloride as determined in step B5.
3. Measure and record the peak heights, H_m.
4. Record A_m and retention time.
5. Repeat the injections until two consecutive values for the total area of the VC peak do not vary more than ±5%.
6. Use the average value for these two total areas to compute the bag concentration.
7. Compare the ratio of H_m to A_m for the VC sample with the same ratio for the standard peak that is closest in height. If these ratios differ by more than 10%, the VC peak may not be pure (possibly acetaldehyde is present) and the secondary column should be employed.

E. Moisture Determination

1. Measure the ambient temperature and barometric pressure near the bag.
2. From a water saturation vapor pressure table, determine and record the water vapor content of the bag as a decimal figure, assuming a relative humidity of 100%.

F. Preparation of Standard Mixtures (Alternative)

1. Leak-check the 16-inch square Tedlar bag according to FP 3b.
2. Evacuate the bag, and meter in 5.0 L of N₂.
3. For a 50-ppm vinyl chloride concentration,
 - a. While the bag is filling, use the 0.5 mL syringe to inject 250 μ L of 99.9 + % vinyl chloride gas through the wall of the bag.
 - b. After withdrawing the syringe, immediately cover the resulting hole with a piece of adhesive tape.
4. For 10- and 5-ppm concentrations, repeat step E3, except use the 50- μ L syringe to inject in 50 μ L and 25 μ L, respectively.
5. Place each bag on a smooth surface and alternately depress opposite sides of the bag 50 times to further mix the gases. Do not use the gas mixture standards after 10 days.

6. Do not reuse a bag if the new gas mixture standard is a lower concentration than that of the previous gas mixture standard.

G. Alternatives

1. Other column and operating parameters may be used, provided that adequacy is confirmed through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and the data are available for review by the Administrator.
2. Other chromatographic columns may be used provided that the precision and accuracy specifications are met in the analysis of vinyl chloride standards and resolution of the vinyl chloride peak is adequate, i.e., the area overlap of the vinyl chloride peak and an interferant peak is not more than 10% (see 40 CFR Part 61, Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution").
3. GC system must be capable of producing a response to 0.1-ppm vinyl chloride that is at least as great as the average noise level. (Response is measured from the average value of the base line to the maximum of the wave form, while standard operating conditions are in use.)

LABORATORY DATA SHEET 106
Vinyl Chloride

Client/Plant Name _____ Job No. _____ Date _____

City/State _____ Sampling Location _____ Gas Chromatograph ID # _____

Bar. Pressure During Cal, P_r _____ mm Hg During Sample Analyses, P_i _____ mm Hg Amb. Lab Temp. _____ °C

Sample No.	Sample ID #	Injection Time	Sample Loop Temp. (K)	Column Temp. (°C)	Carrier Gas Flowrate (mL/min)	Chart Speed	Attenuator Setting A_f	Measured Peak Area A_m	Sample Peak Area [$A_m A_f$] A_c	Peak Height H_m	Ratio H_m/A_m	Retention time (sec)	Conc. C_c
	5 ppm Std												
	10 ppm Std												
	50 ppm Std												
	Audit												

_____ Sample loop purged for 30 seconds at 100 mL/min? _____ All injections duplicated until two consecutive peak areas do not vary >5%?

_____ Average area of the two peaks used to compute the bag concentration?

Note: Compare the ratio (H_m/A_m) for the sample with the same ratio for the standard peak that is closest in height. If it is >10% then the sample may not be pure and the secondary column should be used.

Plot calibration curve [A_c versus C_c].

Saturation vapor pressure at lab temp., P_w _____ mm Hg $B_{wb} = P_w/P_i$

QA/QC Check

Completeness _____ Legibility _____ Accuracy _____ Specifications _____ Reasonableness _____

Checked by: _____

Personnel (Signature/Date)

Team Leader (Signature/Date)