

DETERMINATION OF RESIDUAL VINYL CHLORIDE
MONOMER IN VINYL RESINS

1 PURPOSE

A procedure for determining the amount of residual vinyl chloride present in vinyl resins is described. The resin is dissolved in a solvent and the vinyl chloride analyzed using a gas chromatographic technique. The method is applicable for use with poly-(vinyl chloride), vinyl chloride-vinyl acetate copolymers and other vinyl resins in which there are no volatiles which interfere in the determination.

2 EQUIPMENT AND REAGENTS

Gas Chromatograph, Hewlett-Packard (F and M) Model 5750 or equivalent, equipped with a hydrogen flame ionization detector.

Tetrahydrofuran, reagent grade.

Balance with accuracy to 0.10 gram.

2 SAMPLE PREPARATION

Prepare the sample for chromatograph injection by dissolving the test resin in tetrahydrofuran (THF). Weigh 9 grams of THF into a suitable vial. Add 1 gram of the resin to be tested to the THF. Resin addition should be made as quickly as possible. Cap the bottle and place it on a can-roller to attain complete solution. When complete solution has occurred, the sample is ready for injection into the chromatograph.

4 INSTRUMENT PARAMETERS

Instrument	Hewlett-Packard (F and M) Model 5750 (or equivalent) equipped with hydrogen flame ionization detector.
Column	Six-feet by 1/8-inch stainless steel tubing packed with Chromosorb 102, 60/80 mesh.
Temperatures:	
Column	100°C for 4 minutes, manually increased to 250°C
Injector	100°C (maximum)
Detector	300°C (flame ionization)
Sample Size	4 Microliters
Flows:	
Helium Carrier Gas	25 cc/minute
Hydrogen	20 psi at cylinder head.
Compressed Air	40 psi at cylinder head.

4 (Continued)

Elution Times

Vinyl Chloride	2.8 minutes
Tetrahydrofuran	6.3 minutes

5 CALIBRATION

Prepare solution mixtures of varying amounts of vinyl chloride in tetrahydrofuran to cover the expected range of concentration. Obtain from the chromatograph scan the area percent of vinyl chloride for each sample of known monomer content. Prepare a chart plotting the area percent vinyl chloride versus the known weight percent vinyl chloride to establish the relative detector response of the vinyl chloride with respect to tetrahydrofuran.

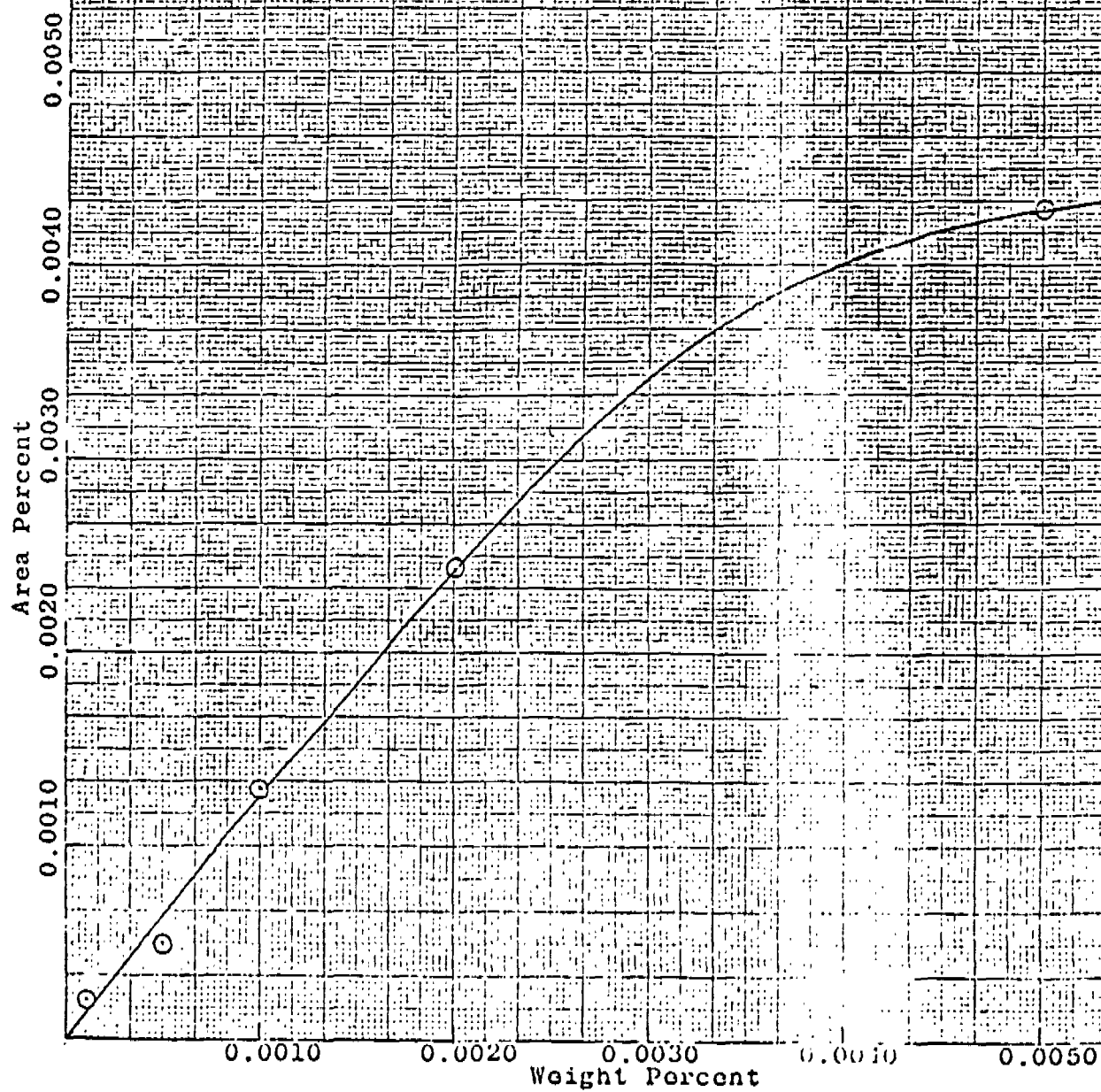
6 CALCULATION

Determine the area percent vinyl chloride from the chromatograph scan for the resin solution to be characterized. Multiply this figure by 10, which converts the data to area percent vinyl chloride based on resin weight. Using the chart developed with the calibration samples, read off the corresponding weight percent of vinyl chloride.

TYPICAL CALIBRATION CURVE

Residual Vinyl Chloride Monomer in Vinyl Resin

Area Percent Conversion to Weight Percent



DUST
DETERMINATION OF TOTAL
AND RESPIRABLE AIRBORNE DUST BY MEMBRANE FILTER
SAMPLING AND GRAVIMETRIC ANALYSIS

- 1 **PURPOSE** This procedure has been written to conform to recommended methods of sampling for dusts as published by the National Institute of Occupational Safety and Health (1) and the American Industrial Hygiene Association. (2)
- 2 **PRINCIPLE** Total dust concentration is measured by drawing a known volume of air through a preweighed, 37-mm polyvinyl chloride membrane filter, sampling with the top cover of the filter holder removed (open faced). The filter is dried by means of vacuum and reweighed to give a measure of the dust concentration. Respirable dust is measured by using a 10-mm nylon cyclone ahead of the membrane filter to remove the larger or non-respirable dust particles before they reach the filter. The filter is treated the same as described for total dust. The table below gives the theoretical deposition of the dust particles.

Aerodynamic diameter (μ m), unit density sphere	Percent passing cyclone
2	90
2.5	75
3.5	50
5.0	25
10.0	0

- 3 **APPARATUS**
- a) Personal sampling pump and charger, Model G, Part No. 456252, Mine Safety Appliance Co., Pittsburgh, Pa., or equivalent. (NOTE - Earlier models of the Model G pump were not equipped with a pulsation damper. If such a model is used, then the pulsation damper kit, MSA Part No. 449614 - should be ordered and installed before taking dust samples.)
 - b) Cyclone and holder; Part No. 456243, MSA holder assembly complete with cyclone and sampling line assemblies.
 - c) Filter holders for 37-mm filters; 2-section, Catalog No. M000037PO, 3-section Catalog No. M000037AO, Millipore Corp., Bedford, Mass.
 - d) Backup pad for 37-mm filter, Catalog No. AP1003700, Millipore Corp.
 - e) Polyvinyl chloride membrane filters; 5.0 μ pore diameter, Vinyl Metricel, VM-1, 37-mm diameter, Catalog No. 60714, Gelman Instrument Co., Ann Arbor, Mich.

- f) Vacuum desiccator (use with a safety shield to contain glass fragments in the event of implosion if a glass desiccator is employed).
- g) Mercury manometer
- h) Analytical balance capable of weighing to the nearest 0.01 mg.
- i) Vacuum pump - capable of reducing pressure in the desiccator to 5 mm of Hg.
- j) Cellulose bands; 2-section holder part No. 625415, Mine Safety Appliances Co. 3-section holder - 41x25 mm, No. 28, Walter H. Jelly and Co., Franklin Park, Illinois.

PREPARATION OF FILTER

- a) Place the VM-1 polyvinyl chloride filter into the vacuum desiccator and reduce the pressure to 5 mm Hg.
- b) After 15 minutes at the 5-mm pressure, allow the desiccator to return to atmospheric pressure.
- c) Make sure the balance is zeroed before weighing. Holding the filter by the edge with tweezers, place it on the balance pan and record the weight to the nearest 0.01 mg (W_1).
- d) Insert a backup support pad into the bottom section of the filter holder. Carefully place the weighed filter on the support screen.
- e) For total dust sampling (3-section filter holder), place the middle and top sections on the bottom section. Press the sections together firmly until the outer edge of the filter is tightly held against the backup support pad.
- f) For respirable dust sampling (2-section filter holder) place the top section on the bottom section and press firmly.
- g) Insert the red and blue plugs into the inlets of the filter holders and slip a cellulose band from the storage solution over the outside of the holder. Allow the band to dry thoroughly before using.

5 RESPIRABLE DUST AIR SAMPLING PROCEDURE

- a) Remove the red and blue plugs from the 2-section filter holder, and insert the filter holder into the cyclone holder assembly, make sure the filter face is pointing downward toward the cyclone.
- b) Clamp the filter holder securely into the cyclone holder, so that the O-rings are held tightly against the filter holder seats.
- c) Clip the cyclone holder assembly on the shirt collar of the employee. Attach the sampling lines from the cyclone to the inlet of the portable pump.
- d) Adjust the air flow through the cyclone by turning the adjusting screw on the pump until the flowmeter ball on the pump is centered on the red band on the flowmeter (1.8 liters per minute). If samples are to be taken for respirable coal dust, then the recommended flow rate through the cyclone and filter is 2.0 liters per minute. This can be obtained by adjusting the flow on the pump until the flowmeter ball is centered under the orange band on the flowmeter.

- c) Place the pump on a belt around the employee's waist. Record the time the pump was started.
- f) At the end of the working shift, stop the pump and record the time. Note: respirable dust samples need a minimum of 4-hours sampling time to provide enough sample to obtain an adequate amount of dust to weigh.
- g) Carefully remove the sampling assembly from the employee. Remove the filter and holder from the cyclone assembly and replace the red and blue plugs. Do not invert the cyclone assembly during this time as the larger particles of dust from the cyclone can fall onto the filter to give an erroneous weight gain.
- h) In the laboratory, split the cellulose band at the junction of the top and bottom section of the filter holder. Carefully pry off the top section.
- i) Place the bottom section containing the filter into the desiccator and reduce the pressure to 5 mm Hg for 15 minutes to dry the filter.
- j) After the desiccator returns to atmospheric pressure, gently lift the filter out of the bottom section of the filter holder by using a small rod through the hole in the bottom section.
- k) Check the zero setting on the balance to make sure it hasn't changed from previous weighing of the filter. Grasp the edge of the filter with the tweezers and reweigh the filter on the balance. Record the weight to the nearest 0.01 mg (W₂).
- l) Clean the dust from the cyclone body before the next use.

6 TOTAL DUST AIR SAMPLING PROCEDURE

- a) Take a 3-section filter holder which has been prepared according to the directions in Section 4. Slice the cellulose band at the junction of the top section and center retaining ring.
- b) Remove the top section and the plug from the bottom section outlet. Attach a piece of tubing from the bottom outlet and connect the other end of the tubing to the Model G MSA pump.
- c) Adjust the air flow through the filter by turning the adjusting screw on the pump until the flowmeter ball on the pump is centered on the orange band (2.0 liters per minute).
- d) Place the pump on a belt around the employee's waist and clip the filter to the shirt collar of the employee with the face of the filter pointing down.
- e) Record the time the sampling pump was started.
- f) At the end of the working shift, stop the pump and record the time.
- g) Carefully remove the filter from the employee. Replace the top cover and the plug in the bottom outlet.
- h) In the laboratory, split the cellulose band at the junction of the bottom section and the middle retaining ring. Carefully pry off the top and middle sections.
- i) Place the bottom section containing the filter into the desiccator and reduce the pressure to 5 mm Hg for 15 minutes to dry the filter.

- j) After the desiccator returns to atmospheric pressure, gently lift the filter out of the bottom section of the filter holder by using a small rod through the hole in the bottom section.
 - k) Check the zero setting on the balance to make sure it hasn't changed from previous weighing of the filter. Grasp the edge of the filter with the tweezers and reweigh the filter on the balance. Record the weight to the nearest 0.1 (W_2).
- 7 CALIBRATION OF MSA SAMPLING PUMP
- a) Assemble the apparatus as shown in Figure 1. Tape the cap on the gallon bottle with vinyl electrical tape to insure a tight seal.
 - b) Start the pump and adjust the flow until the ball in the flowmeter is centered on the red band (1.8 liters per minute).

APPARATUS FOR CALIBRATION OF MSA PERSONAL SAMPLING PUMP

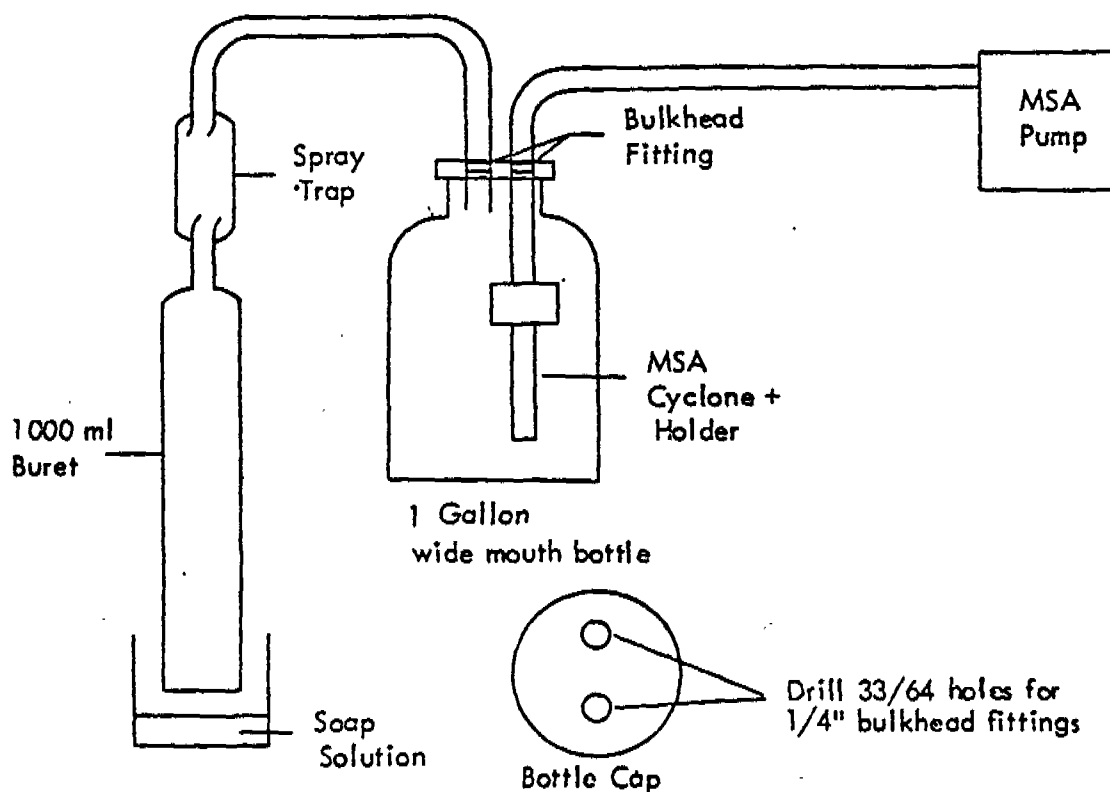


Figure 1

- c) Moisten the interior surface of the buret with soap solution.
- d) Dip the end of the buret into the soap solution to start a bubble moving up the buret.
- e) With a stopwatch, measure the time in seconds that it takes for the bubble to move from the zero graduation mark on the buret to the 1000-ml mark.
- f) Divide the time in seconds into 60 to obtain the flow rate in liters per minute.
- g) Repeat the determination, centering the flowmeter ball on the pump on the orange band (2.0 liters per minute).
- h) A wet test meter may be used in place of the soap-film buret if desired.

8 CALCULATIONS

a) Respirable dust

$$\frac{(W_2 - W_1) \times 10^6}{F \times T} = \text{respirable dust concentration in milligrams per cubic meter (mg/M}^3\text{)}$$

F = Flow rate in liters per minute through the cyclone and filter

T = Total sampling time in minutes

W₁ = Initial weight of the polyvinyl chloride filter in grams

W₂ = Final weight of the polyvinyl chloride filter in grams

b) Total dust

Use identical calculation except to report the value as total dust concentration in mg/M³. (F = flow rate in liters through the filter)

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