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cc: Wells + WBB
Letter only
ltr

August 31, 2001

VIA HAND DELIVERY

Mr. Charles B. Cappel
BAGGETT, MCCALL, BURGESS & WATSON
P.O. Box 7820
Lake Charles, LA 70606-7820

Re: Production of Documents by Monsanto Company
in VCM Litigation

Dear Bert:

Enclosed are copies of the documents that you are missing out of the documents produced by Monsanto in the VCM litigation. It appears that the copier failed to copy both sides of some documents. The only document that I was unable to locate is RSV16576. I will make a note of this and if I come across that particular page I will send it to you. However, it may have been another copier error.

Sincerely,

KEVIN POWELL

MKP/lsj

Enclosure

cc: Tom Bistline (w/out enclosure)

VINYL CHLORIDE

DETERMINATION IN AIR BY ADSORPTION ON ACTIVATED CHARCOAL AND ANALYSIS BY GAS CHROMATOGRAPHY

1 PURPOSE AND LIMITATIONS This paper describes a procedure for measuring the exposure of personnel to vinyl chloride in the working environment. The method describe the preparation of standards which will determine vinyl chloride i the range of 9 to 90 parts per million by volume in air, based on 10-minute sample at a flow rate of 100 ml per minute. Lower or hi er ppm ranges can be determined by preparing additional standards cover the range desired. (Vinyl chloride can be determined to at least 0.05 ppm by volume in air by this procedure, using more dilu standards.)

2 PRINCIPLE The sample is collected by passing air through a gla tube containing activated charcoal which adsorbs any vinyl chloride vapors present. The vinyl chloride is then desorbe from the charcoal by carbon disulfide extraction and analyzed by g chromatography.

3 INSTRUMENT PARAMETERS

Chromatograph	AID (Analytical Instrument Developm Inc.) Portable Chromatograph, Mod 511, or equivalent chromatograph, with flame ionization detector.
Column	Six-foot x 1/8-inch stainless steel packed with 10% di(2-ethylhexyl) sebacate on Chromasorb P, 80/100 mesh, NAW.
Column temperature	100°C isothermal
Injection temperature	100°C
Detector	flame ionization detector
Carrier	helium or nitrogen
Hydrogen flow rate	23 cc per minute
Air flow rate	133 cc per minute
Sample size	2 ul solvent flush technique
Retention time	0.5 minute (adjust carrier flow until vinyl chloride elutes at this time)

Alternate Column - Better Resolution

Instrument	Hewlett-Packard 5750 gas chromatograph - dual column, or equivalent.
Column	25-foot x 1/8 inch stainless steel packed with 25% by weight 75% CO880/25% Tergitol E-35 on 40/60 mesh rescreened Chromosorb P.
Detector	flame ionization
Column temperature	40° to 100°C at 4°C per minute
Detector temperature	125°C
Injector temperature	125°C
Carrier gas	helium, 30 ml per minute
Air flow	350 ml per minute
Hydrogen flow	30 ml per minute
Sample size	10 ul
Approximate elution time	6.5 minutes

4 REAGENTS AND APPARATUS

a) Personal sampling pump. MSA Model G or Bendix Micronair. Insert an in-line orifice made from a cut-off and restricted (pinched) hypodermic needle in the sampling line to the pump in order to obtain a 100cc per minute flow on these pumps.

b) Hypo-vials, 15-ml size, Cat. No. 12911; Neoprene septa, Cat. No. 13233; alumina seals, Cat. No. 13214; and hand crimper, Cat. No. 13212, Pierce Chemical Company, Rockford, Illinois.

c) Carbon disulfide, spectrophotometric grade

d) Activated charcoal, Cat. No. 580-26, Barnebey-Cheney, Columbus, Ohio

e) Soap film flow meter, 10-ml size

f) Stop watch

5 PREPARATION OF THE ACTIVATED CARBON ADSORPTION TUBE

a) Prepare the activated charcoal by placing the charcoal on a 40-mesh sieve and wash with demineralized water until no visible fines appear in the washings.

b) Dry the carbon in a drying oven at 110°C until dry and free flowing (overnight). Store in a screw-top, glass bottle until needed.

c) Cut 8-mm O.D. x 6-mm I.D. Pyrex glass tubing into 6-inch lengths and fire polish the ends.

d) Insert a Pyrex glass wool plug two-thirds of the distance into the glass tubes. The longer section of tubing will be designated as the primary end, while the shorter side of the tubing is the back-up end.

e) Pack the tubes with the activated carbon, using a length of 60 mm in the primary section and 35 mm in the back-up section. Retain the carbon with glass wool plugs.

f) Seal the tubes with a red rubber septum or parafilm until ready for use.

6 SAMPLING PROCEDURE

a) Remove the seals from the charcoal tube and attached the back-up section to a portable pump by means of a length of 1/4-inch Tygon tubing.

b) Set the flow rate at 100 ml per minute through the tube with a calibrated rotameter. (NOTE: The 10-ml soap bubble flow meter can be used to obtain a more precise reading of the air flow through the tube if desired).

c) Record the time the air sampling was started and the time when the sampling is completed. A 10-minute sample may be taken with this system.

d) After sampling, recheck the flow rate and reseal the ends of the charcoal tube. Return the tube to the laboratory for analysis.

e) Record the termperature and barometric pressure at the sampling site.

7 ANALYTICAL PROCEDURE

a) Make a small hook at the end of a piece of wire and remove the glass wool plug from the primary end of the charcoal tube. Make sure that no charcoal particles adhere to the glass wool plug.

b) Pour the charcoal into a 10-ml glass-stoppered volumetric flask, pipet 3 ml of carbon disulfide (CS_2) into the flask and stopper securely. (CAUTION: Carbon disufide is toxic and should be handled under a hood.)

c) Agitate the flask periodically for at least 30 minutes.

d) Solvent Flush Injection Technique. This injection technique is designed to eliminate difficulties arising from blow-back or distillation with the needle of the microliter syringe.

e) Flush a 10 µl syringe with CS_2 several times to wet the barrel and plunger.

f) Draw 2 ml of CS_2 into the syringe and remove the tip of the needle from the solvent. Withdraw the plunger an additional 0.5 ml to separate the CS_2 from the sample with a pocket of air.

g) Dip the needle into the sample solution in the volumetric flask and withdraw the plunger until the air bubble between the solvent and the sample has passed the 2 µl mark on the syringe.

h) Remove the tip of the needle from the sample solution and adjust the volume in the syringe until the meniscus of the air bubble rests on the 2 µl mark. Remove the excess sample solution from the tip of the needle.

i) Pull the plunger back an additional 0.5 µl to prevent the sample soluiton from evaporating from the tip of the needle.

j) Inject the entire contents of the syringe into the chromatograph.

k) Measure the peak height and determine the vinyl chloride content from a previously prepared calibration curve.

8 CALIBRATION CURVE

a) Pipet 10 ml of carbon disulfide (CS_2) into each of five 15-ml hypo-vials. Cap the vials with the Neoprene septa and aluminum seals, using the hand crimper.

b) Place the hypo-vials in wet ice to reduce the vapor pressure of CS_2.

c) Cap one of the valves on a steel sample cylinder containing vinyl chloride with a 1/8-inch Swagelok tubing nut which has a chromatographic septum installed in the nut.

d) Insert a hypodermic needle through the septum on the cylinder, open the valve and allow the vinyl chloride to vent through the needle for about 10 seconds to purge the air from the system. Remove the needle.

e) Using the appropriate gas-tight syringe fitted with a number 27 gauge hypodermic needle, insert the needle through the septum on the cylinder and allow the pressure of the vinyl chloride to displace the volume of the syringe. Flush the syringe two times to remove any air which may have been trapped in the needle.

f) Into the respecitve hypo-vials, inject 0.2, 0.3, 0.5, 1.0, and 2.0 cc of vinyl chloride from the syringe.

g) Shake the hypo-vials for one minute to put the vinyl chloride in solution. These standards will contain 56, 82, 133, 260, 515 mgm of vinyl chloride per ml. (NOTE: These calculations are corrected for the dead space in the gas syringe needle. The cold CS_2 solutions in the hypo-vials are under slight negative pressure. Measurements have shown that the dead space in the syringe needle amounts to 0.04 cc, and 0.02 cc of this volume is pulled into the hypo-vial by the negative pressure.)

h) Allow the standards to warm up to room temperature, then inject these standards into the chromatograph using the procedure described in Section 7, paragraphs d through j. Shake the hypo-vials each time just prior to withdrawing a sample.

i) Plot peak height versus micrograms of vinyl chloride per ml.

9 CALCULATION

$$\frac{A \times 3 \times 24.5}{L \times 62.5} = \text{vinyl chloride, ppm by volume at } 25^{\circ}C \text{ and } 760 \text{ mm}$$

A = mgm per ml of vinyl chloride read from calibration curve

L = total liters of air sample (flow rate x time)

VINYL CHLORIDE

DETERMINATION IN AIR BY GAS CHROMATOGRAPHY

1 PURPOSE AND LIMITATION This method was developed to determi vinyl chloride in air by grab sample taken with a 50-ml hypodermic syringe. Air samples are introduced directly into the gas chromatograph by means of a 5-cc gas sampling valve. The minimum detectable concentration of vinyl chloride is about 0.1 part per million (ppm) by volume in air using this procedure. The method has been written for use with an Analytical Instrument Development portable chromatograph but can be used with other suitably equipped gas chromatographs.

2 INSTRUMENT PARAMETERS

Chromatograph	AID (Analytical Instrument Development, Inc.) Portable Chromatograph, Model 511, or equivalent chromatograph, with flame ionization detector.
Column	eight foot x 1/8-inch stainless steel packed with Poropak Q, 50/80 mesh.
Column temperature	100°C, isothermal
Injection temperature	100°C
Detector	flame ionization detector
Carrier, flow rate	nitrogen, 40 cc per minute
Hydrogen flow rate	26 cc per minute
Air flow rate	120 cc per minute
Sample size	5-cc gas sampling loop
Retention time	approximately 2.8 min.

3 SAMPLING AND ANALYTICAL PROCEDURE

a) Grab samples are taken with a 50-ml hypodermic syringe without needle. After taking samples, cap the opening of the syringe with parafilm or a 7/32 Caplug to prevent diffusion of the vinyl chloride from the syringe. Sampl must be analyzed within 30 minutes after taking, as losse of vinyl chloride will occur in the syringe. Samples ma also be taken with pre-conditioned Saran plastic bag if desired.

b) Turn the gas sampling valve to the load position and connect the 50-ml hypodermic syringe to the inlet. Allow the weight of the plunger to force the contents of the syringe through the 5-cc loop.

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.

3 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/M) Model 5750 (or equivalent) equipped with hydrogen flame ionization detector.
Column	6-ft by 1/8-in. 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

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 per cent of vinyl chloride for each sample of known monomer content. Prepare a chart plotting the area per cent vinyl chloride versus the known weight per cent vinyl chloride to establish the relative detector response of the vinyl chloride with respect to tetrahydrofuran.

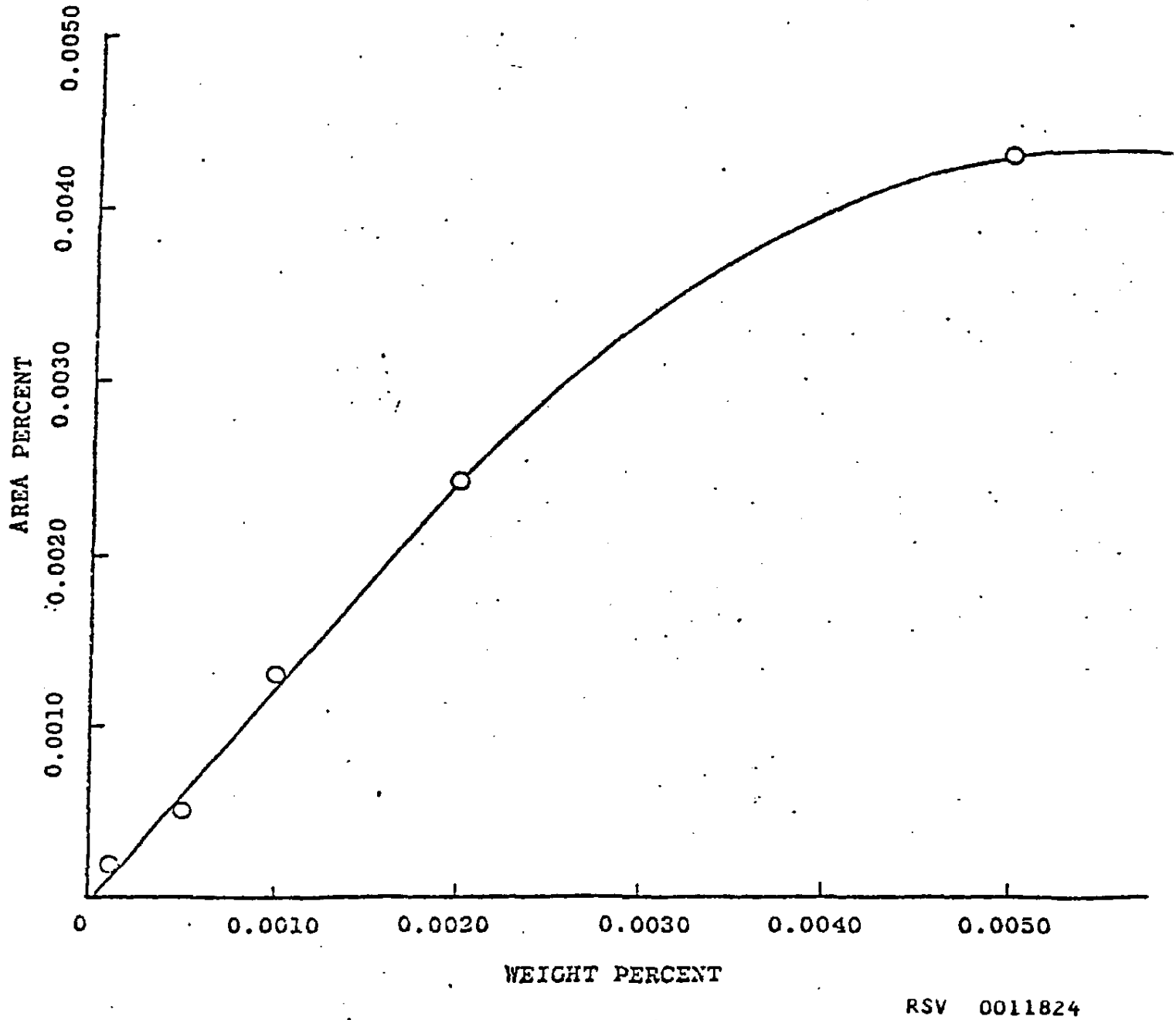
6 CALCULATION

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

TYPICAL CALIBRATION CURVE

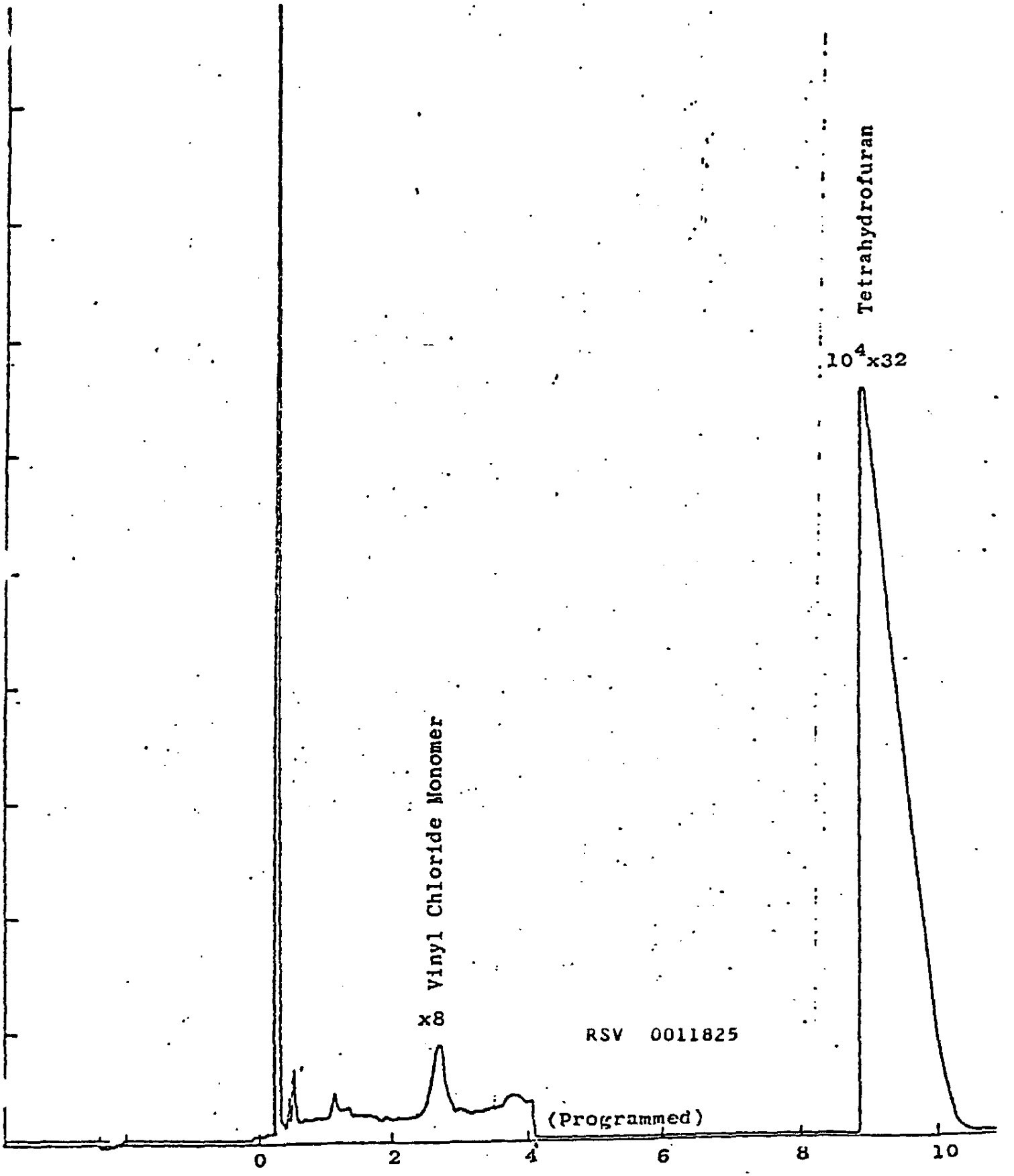
Residual Vinyl Chloride Monomer in Vinyl Resin

Area Percent Conversion to Weight Percent



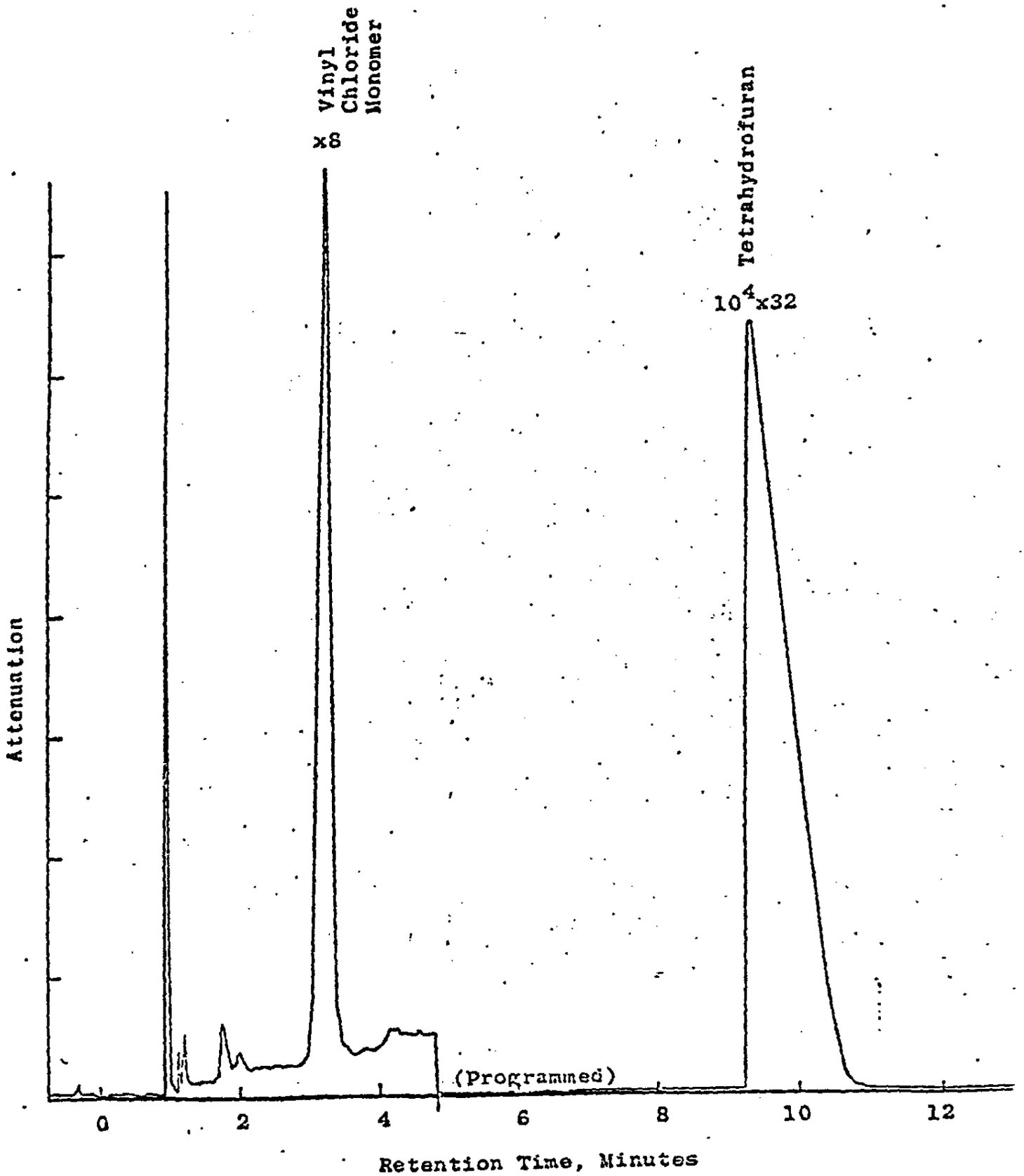
RSV 0011824

SYNTHETIC SAMPLE OF 1 PPM VINYL CHLORIDE IN THE SOLVENT
(Sample Volume = 2 Microliters)



SYNTHETIC SAMPLE OF 10 PPM VINYL CHLORIDE IN THE SOLVENT

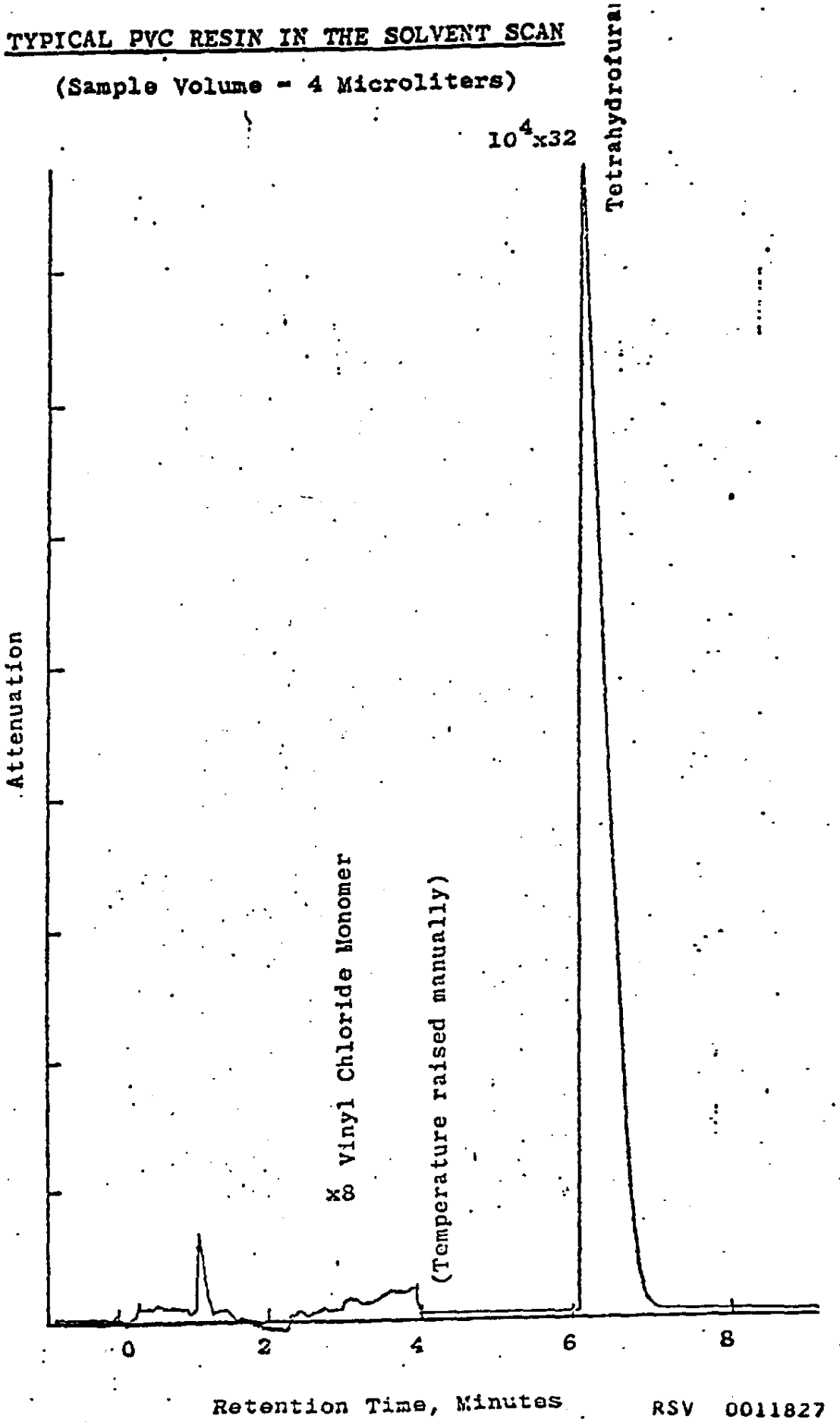
(Sample Volume = 2 Microliters)



RSV 0011826

TYPICAL PVC RESIN IN THE SOLVENT SCAN

(Sample Volume = 4 Microliters)



RSV 0011827

SAMPLING GUIDELINES

WORKPLACE AIR MONITORING

Sampling procedures should meet the following general requirements:

1. Samples collected shall be representative of the individual worker's exposur or of the concentration in a particular work area.

2. Sample records shall include:

   a) The date and time of the sample collection.

   b) Duration of the sample collection.

   c) Volumetric flow rate of sample collection.

   d) Description of the sampling location.

   e) Other pertinent information.

3. Breathing zone samples shall be collected as near as practicable to the worker's face without interfering with his freedom of movement. These samples shall characterize the exposure from each job or operation.

4. Work area samples shall be collected at the normal breathing zone level of an average worker. These samples may be used to characterize the exposure from each job or operation, if the time a worker spends in this area is identifiable.

5. Sampling methods and analytical procedures shall be calibrated at least weekly to ensure accuracy.

RNWheelerJr/ra

May 8, 1974