Air Products and Chemicals INC.

CALVERT CITY PLANT

INTEROFFICE MEMORANDUM

To: R. C. Lietzau

Date: 18 February 1982

From: B. D. Helms

Copies:

Re: Personnel Monitoring - Vinyl Chloride

G Boyd
Central Safety Approved today 25 Feb 82
Note Joe Smith comment Boyce

G. N. Boyd has completed the field comparison of Reiszner badges versus the pump method for determining employee exposure to VCl. The results (attached) show excellent agreement.

I recommend we change to the badge method of monitoring for VCl. The accuracy will be rechecked in three months with standard gas. Also area field comparisons with the pump method should be made every three months.

Your approval is requested.

B. D. Helms

Joe Smith agrees.

25 Feb 82

Committee Agree

Should recheck on a hot-humid day; Bill

AP00040027

Air Products and Chemicals INC.

ESCAMBIA PLANT

INTEROFFICE MEMORANDUM

To: C. A. Pendergrass

Date: 26 August 1983

Copies: R. H. Sroufe, J. W. Kadlec, T. J. Regan, Tom Noll, B. Rose Calvert, B. Helms, S. Prescott, J. Barr, L. W. Allen, J. Spata

From: D. E. Hoffman

Re: Prepressurizer for VCl Headspace Analysis

SUMMARY

A recent change in the Federal Register method 107 requires pre-pressurizing sample vials with nitrogen prior to analysis on the VCl headspace analyzer. The Escambia QC Lab has fabricated a pre-pressurizer in accordance with the required drawings. Initial testing has shown a minimal change in results. If no comments to the contrary are received by 15 September, the pressurizer will be put into service on that date.

Discussion

The September 7, 1982 Federal Register included a method for pre-pressurization of vials with nitrogen. These changes are underlined in Attachment I. Drawings of the required apparatus were secured, and the Escambia Instrument Department was assigned the task of building it. This proved to be a lengthly job because most of the parts used by B. F. Goodrich to build the prototype are no longer commercially available. APCI attorneys determined equivalent parts were acceptable and in some cases had to be fabricated at Escambia.

The prepressurizer has now been completed, installed, and piped in to the nitrogen supply. Senior Lab Technician R. E. Zeiger has done the necessary cross checking of control samples to determine the effect of the vial prepressurization.

Preliminary analyses showed double peaks appearing at pressures above 19 psig. Method 107 calls for the highest pressure possible without double peaks, so 19 psig was chosen as the prepressure level. Several vials were pressured up, put in the hot bath, and allowed to sit overnight to be certain there was no leakage through the septum or cap.

Finally, several samples of different concentrations were run both with and without prepressurization. The data is tabled below.

PrePressurizer for
VCl Headspace Analysis

FINISHED PRODUCT DRY RESIN - PPM		PROCESS SAMPLES SLURRIES - PPM	
Atmospheric	Prepressurized	Atmospheric	Prepressurized
5	2	436	308
3	2	187	176
4	4	106	93
5	3	154	135
5	4	287	294
5	4	81	86
4	3	79	72
8	5	167	168
2	1	68	63
4	3	69	65
		174	187
1	1	180	190
		456	470
1	1	247	224
		527	486
		124	127
		326	336
		189	180

These initial tests indicate a mean drop of 1 ppm at the normal operating range of below 10 ppm for dried resin and a mean drop of 7 ppm at the higher levels. As expected, there is apparently no significant difference when adding the prepressurizer.

CONCLUSION

The nitrogen prepressurizer is installed in the Escambia QC Lab. Initial results indicate it is operating correctly and will be put in service on September 15, 1983.

D. E. Hoffman

DEH:bg

ATTACHMENT

...procedure ... Appendix C. The ...ended effect of ... is to ... determination of ... chromatographic (GC) column ...ication ... the ...ended effect of ...procedure ... is to provide a method for ...ating GC sample analysis.

EFFECTIVE DATE: September 7, 1982.

Under section 307(b), (1) of the Clean Air Act, judicial review of this rulemaking is available only by the filing of a petition for review in the U.S. Court of Appeals for the District of Columbia Circuit within 60 days of today's publication of this rule. Under section 307(b)(2) of the Clean Air Act, the requirements that are the subject of today's notice may not be challenged later in civil or criminal proceedings brought by EPA to enforce these requirements.

ADDRESSES: *Summary of Comments and Responses.* The summary of comments and responses for the proposed test methods may be obtained from the U. S. EPA Library (MD-35), Research Triangle Park, North Carolina 27711, telephone number (919) 541-2777. Please refer to "Revised Test Methods 106 and 107—Summary of Comments and Responses, EPA 450/3-82-002." The document contains (1) a summary of the changes made to the test methods since proposal and (2) a summary of all the public comments made on the proposed revised methods and the Administrator's responses to the comments.

Docket. A docket, number A-80-50, containing information considered by EPA in the development of the test methods and docket number OAQPS 79-5 Part 2 that contains background information pertaining to Appendix C are available for public inspection between 8:00 a.m. and 4:00 p.m., Monday through Friday, at EPA's Central Docket Section (A-130), West Tower Lobby, Gallery 1, 401 M Street, S.W., Washington, D.C. 20460. A reasonable fee may be charged for copying.

FOR FURTHER INFORMATION CONTACT:

Roger T. Shigehara, Emission Measurement Branch, Emission Standards and Engineering Division (MD-19), U. S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711, telephone (919) 541-[illegible]237.

Public Participation

The revised test methods were proposed and published in the Federal Register on November 18, 1980 (45 FR 76346). Public comments were solicited at the time of proposal. The public comment period was from November 18, 1980, to January 19, 1981, with an extension to February 19, 1981.

Five comment letters were received concerning issues relative to the proposed test methods. The comments have been carefully considered; and where determined to be appropriate by the Administrator, changes have been made in the proposed revisions to the test methods.

Procedures 1 and 2 of Appendix C were proposed and published in the Federal Register April 18, 1980 (45 FR 26660). Public comments were solicited at the time of proposal. The public comment period was from April 18, 1980, to August 21, 1980.

No comment letters were received.

Significant Comments and Changes to the Proposed Test Methods

Comments on the proposed revisions to the test methods were received from industry, industry counsel, engineering firms, and equipment manufacturers. A detailed discussion of these comments and responses can be found in the summary of comments and responses which is referred to in the ADDRESSES section of this preamble. The summary of comments and responses serves as the basis for the revisions which have been made to the test methods between proposal and promulgation. The major comments and responses are summarized in this preamble. Most of the comment letters contained multiple comments. The comments have been divided into the following areas:

Proposal of Revised Test Methods 105 and 107

One commenter felt that EPA should publish a notice in the Federal Register to clarify the November 18, 1980, notice on Test Methods 105 and 107 (45 FR 76348) as to whether the changes in the methods were proposed or final amendments. The EPA considered the suggestion to be reasonable and a notice was published in the Federal Register on January 6, 1981 (46 FR 1318) to clarify that the changes in Methods 106 and 107 published on November 18, 1980, were proposed changes.

Sample Analysis Procedure—Method 106

One commenter suggested that Section 7.2.2, Preparation of Chromatograph Calibration Curve, be changed to require calibration at least once every 8 hours of continuous operation of the chromatograph, whereas the method requires daily calibration. The EPA has decided it would be an unnecessary burden to arbitrarily set 8 hours as a cutoff point for valid calibration. However, the comment has identified the need for instruction in the method as to the use of multiple calibration curves in data interpretation, and Section 7.2.2 has been revised to provide that instruction.

One commenter questioned the use of Figure 106-2 because it appeared to illustrate a standards preparation procedure different from the one described in the method. Figure 106-2 did illustrate a different sample preparation procedure and has been deleted from the method.

Sample Collection and Analysis Procedure—Method 107

One commenter questioned the need for the sample prepressurization procedure that is included in the revised test method. The Agency believes the prepressurization procedure is valid as prepressurization of sample vials prior to analysis has been shown to produce K_p values which agree with theoretical values. A paper describing a study of this technique has been added to the bibliography section of the method as an aid in the use of this procedure.

PAPER BFG

Quality Assurance—Method 106

One commenter requested that Section 5.2.4, Audit Cylinder Standards, further describe commercial gas manufacturers as an alternative source of these standards. The Agency considered the request to be reasonable, and Section 5.2.4 has been revised to define the acceptability of audit cylinders obtained from commercial gas manufacturers.

Docket

The docket is an organized and complete file of all the information considered by EPA in the development of this rulemaking. The docket is a dynamic file, since material is added throughout the rulemaking development. The docketing system is intended to allow members of the public and industries involved to readily identify and locate documents so that they can intelligently and effectively participate in the rulemaking process. Along with the statement of basis and purpose of the proposed and promulgated test methods and EPA responses to significant comments, the contents of the docket will serve as the record in case of judicial review [Section 307(d)(7)(A)].

Miscellaneous

This rulemaking does not impose any additional emission measurement requirements on facilities affected by this rulemaking. Rather, this rulemaking revises the test methods to which the

AP00040030

Method 107—Determination of Vinyl Chloride Content of Inprocess Wastewater Samples, and Vinyl Chloride Content of Polyvinyl Chloride Resin, Slurry, Wet Cake, and Latex Samples

Introduction

Performance of this method should not be attempted by persons unfamiliar with the operation of a gas chromatograph (GC), nor by those who are unfamiliar with source sampling, because knowledge beyond the scope of this presentation is required. Care must be exercised to prevent exposure of sampling personnel to vinyl chloride, a carcinogen.

1. *Applicability and Principle.*

1.1 Applicability. This method applies to the measurement of the vinyl chloride monomer (VCM) content of inprocess wastewater samples, and the residual vinyl chloride monomer (RVCM) content of polyvinyl chloride (PVC) resins, wet cake, slurry, and latex samples. It cannot be used for polymer in fused forms, such as sheet or cubes. This method is not acceptable where methods from Section 304(h) of the Clean Water Act, 33 U.S.C. 1251 et seq. (the Federal Water Pollution Control Amendments of 1972, as amended by the Clean Water Act of 1977) are required.

1.2 Principle. The basis for this method relates to the vapor equilibrium that is established between RVCM, PVC resin, water, and air in a closed system. The RVCM in a PVC resin will equilibrate rapidly in a closed vessel, provided that the temperature of the PVC resin is maintained above the glass transition temperature of that specific resin.

2. Range and Sensitivity. The lower limit of detection of vinyl chloride will vary according to the chromatograph used. Values reported include 1×10^{-3} mg and 4×10^{-7} mg. With proper calibration, the upper limit may be extended as needed.

3. *Interferences.* The chromatograph columns and the corresponding operating parameters herein described normally provide an adequate resolution of vinyl chloride; however, resolution interferences may be encountered on some sources. Therefore, the chromatograph operator shall select the column and operating parameters best suited to his particular analysis requirements, subject to the approval of the Administrator. Approval is automatic provided that the tester produces confirming data through an adequate supplemental analytical technique, such as analysis with a different column or GC/mass spectroscopy, and has the data available for review by the Administrator.

4. *Precision and Reproducibility.* An interlaboratory comparison between seven laboratories of three resin samples, each split into three parts, yielded a standard deviation of 2.03 percent for a sample with a mean of 2.09 ppm, 4.16 percent for a sample with a mean of 1.66 ppm, and 5.29 percent for a sample with a mean of 62.66 ppm.

5. *Safety.* Do not release vinyl chloride to the laboratory atmosphere during preparation of standards. Venting or purging with VCM/air mixtures must be held to a minimum. When they are required, the vapor must be routed to the outside air. Vinyl chloride, even at low ppm levels, must never be vented inside the laboratory. After vials have been analyzed, they must be vented prior to removal of the vial from the instrument turntable. Vials must be vented through a hypodermic needle connected to an activated charcoal tube to prevent release of vinyl chloride into the laboratory atmosphere. The charcoal must be replaced prior to vinyl chloride breakthrough.

6. *Apparatus.*

6.1 Sampling. The following equipment is required:

6.1.1 Glass bottles. 60-ml (2-oz) capacity, with wax-lined screw-on tops, for PVC samples.

6.1.2 Glass Vials. 50-ml capacity Hypovial, sealed with Teflon faced Tuf-Bond discs, for water samples.

6.1.3 Adhesive Tape. To prevent loosening of bottle tops.

6.2 Sample Recovery. The following equipment is required:

6.2.1 Glass Vials. With butyl rubber septa, Perkin-Elmer Corporation Nos. 0105–0129 (glass vials), 0001–0720 (gray butyl rubber septum, plug style), 0105–0131 (butyl rubber, septa), or equivalents. The seals must be made from butyl rubber. Silicone rubber seals are not acceptable.

6.2.2 Analytical Balance. Capable of weighing to ±0.0001 gram.

6.2.3 Vial Sealer. Perkin-Elmer No. 105–0106, or equivalent.

6.2.4 Syringe. 100-µl capacity, precision series "A" No. 010025, or equivalent.

6.3 Analysis. The following equipment is required:

6.3.1 Gas Chromatograph. Perkin-Elmer Corporation Model F–40, F–42, or F–45 Head-Space Analyzer, or equivalent. Equipped with backflush accessory.

6.3.2 Chromatographic Columns. Stainless steel 1 m by 3.2 mm and 2 m by 3.2 mm, both, containing 50/80-mesh Porapak Q. The analyst may use other columns provided that the precision and accuracy of the analysis of vinyl chloride standards are not impaired and he has available for review information confirming that there is adequate resolution of the vinyl chloride peak. (Adequate resolution is defined as an area overlap of not more than 10 percent of the vinyl chloride peak by an interferent peak. Calculation of area overlap is explained in Appendix C, Procedure 1: "Determination of Adequate Chromatographic Peak Resolution.") Two 1.83 m columns, each containing 1 percent Carbowax 1500 on Carbopak B, have been suggested for samples containing acetaldehyde.

6.3.3 Thermometer. 0 to 100° C, accurate to ±0.1° C. Perkin-Elmer No. 105–0109, or equivalent.

6.3.4 Sample Tray Thermostat System. Perkin-Elmer No. 105–0103, or equivalent.

6.3.5 Septa. Sandwich type, for automatic dosing, 13 mm. Perkin-Elmer No. 105–1008, or equivalent.

6.3.6 Integrator-Recorder. Hewlett-Packard Model 3380A, or equivalent.

6.3.7 Filter Drier Assembly (3). Perkin-Elmer No. 2230117, or equivalent.

6.3.8 Soap Film Flowmeter. Hewlett Packard No. 0101–0113, or equivalent.

6.3.9 Regulators. For required gas cylinders.

6.3.10 Headspace Vial Pre-Pressurizer. [illegible] 60 pneumatic hypodermic needle inside protective shield. (Blueprint available from Test Support Section, Emission Measurement Branch, Office of Air Quality Planning and Standards, Environmental Protection Agency, Mail Drop 19, Research Triangle Park, N.C. 27711.)

7. *Reagents.* Use only reagents that are of chromatographic grade.

7.1 Analysis. The following items are required for analysis:

7.1.1 Hydrogen. Zero grade.

7.1.2 Nitrogen. Zero grade.

7.1.3 Air. Zero grade.

7.2 Calibration. The following items are required for calibration:

7.2.1 Cylinder Standards (4). Gas mixture standards (50-, 500-, 2000- and 4000-ppm vinyl chloride in nitrogen cylinders). The tester may use cylinder standards to directly prepare a chromatograph calibration curve described in Section 9.2, if the following conditions are met: (a) The manufacturer certifies the gas composition with an accuracy of ±3 percent or better (see Section 7.2.1.1). (b) The manufacturer recommends a maximum shelf life over which the gas concentration does not change by greater than ±5 percent from the certified value. (c) The manufacturer affixes the date of gas cylinder preparation, certified vinyl chloride concentration, and recommended maximum shelf life to the cylinder before shipment to the buyer.

7.2.1.1 Cylinder Standards Certification. The manufacturer shall certify the concentration of vinyl chloride in nitrogen in each cylinder by (a) directly analyzing each cylinder and (b) calibrating his analytical procedure on the day of cylinder analysis. To calibrate his analytical procedure, the manufacturer shall use, as a minimum, a 3-point calibration curve. It is recommended that the manufacturer maintain (1) a high-concentration calibration standard (between 4000 and 8000 ppm) to prepare his calibration curve by an appropriate dilution technique and (2) a low-concentration calibration standard (between 50 and 500 ppm) to verify the dilution technique used. If the difference between the apparent concentration read from the calibration curve and the true concentration assigned to the low-concentration calibration standard exceeds 5 percent of the true concentration, the manufacturer shall determine the source of error and correct it, then repeat the 3-point calibration.

7.2.1.2 Verification of Manufacturer's Calibration Standards. Before using, the manufacturer shall verify each calibration standard by (a) comparing it to gas mixtures prepared (with 99 mole percent vinyl chloride) in accordance with the procedure described in Section 7.1 of Method 106 or by (b) calibrating it against vinyl chloride cylinder Standard Reference Materials (SRM's) prepared by the National Bureau of Standards, if such SRM's are available. The agreement between the initially determined concentration value and the verification concentration value must be within ±5

percent. The manufacturer must recertify all calibration standards on a time interval consistent with the shelf life of the cylinder standards said.

8. *Procedure.*

8.1 Sampling.

8.1.1 PVC Sampling. Allow the resin or slurry to flow from a tap on the tank or silo until the tap line has been well purged. Extend and fill a 60-ml sample bottle under the tap, and immediately tighten a cap on the bottle. Wrap adhesive tape around the cap and bottle to prevent the cap from loosening. Place an identifying label on each bottle, and record the date, time, and sample location both on the bottles and in a log book.

8.1.2 Water Sampling. Prior to use, the 50-ml vials (without the discs) must be capped with aluminum foil and heated in a muffle furnace at 400° C for at least 1 hour to destroy or remove any organic matter that could interfere with analysis. At the sampling location fill the vials bubble-free to overflowing so that a convex meniscus forms at the top. The excess water is displaced as the sealing disc is carefully placed, with the Teflon side down, on the opening of the vial.

Place the aluminum seal over the disc and the neck of the vial, and crimp into place. Affix an identifying label on the bottle, and record the date, time, and sample location both on the vials and in a log book. All samples must be kept refrigerated until analyzed.

8.2 Sample Recovery. Samples must be run within 24 hours.

8.2.1 Resin Samples. The weight of the resin used must be between 3.5 and 4.5 grams. An exact weight must be obtained (±0.0001 g) for each sample. In the case of suspension resins, a volumetric cup can be prepared for holding the required amount of sample. When the cup is used, open the sample bottle, and add the cup volume of resin to the tared sample vial (tared, including septum and aluminum cap). Obtain the exact sample weight, add 100µl or about two equal drops of distilled water, and immediately seal the vial. Report this value on the data sheet; it is required for calculation of RVCM. In the case of dispersion resins, the cup cannot be used. Weigh the sample in an aluminum dish, transfer the sample to the tared vial, and accurately weigh it in the vial. After prepressurization of the samples, condition them for a minimum of 1 hour in the 90° C bath. Do not exceed 5 hours.

Note.—Some aluminum vial caps have a center section that must be removed prior to placing into sample tray. If the cap is not removed, the injection needle will be damaged.

8.2.2 Suspension Resin Slurry and Wet Cake Samples. Decant the water from a wet cake sample, and turn the sample bottle upside down onto a paper towel. Wait for the water to drain, place approximately 0.2 to 4.0 grams of the wet cake sample in a tared vial (tared, including septum and aluminum cap) and seal immediately. Then determine the *sample weight* (±0.0001 g). *All samples must* be prepressurized and then conditioned for 1 hour at 90° C. A sample of wet cake is used to determine total solids (TS). This is required for calculating the RVCM.

8.2.3 Dispersion Resin Slurry and Latex Samples. The materials should not be filtered. Sample must be thoroughly mixed. Using a tared vial (tared, including septum and aluminum cap) add approximately eight drops (0.25 to 0.35 g) of slurry or latex using a medicine dropper. This should be done immediately after mixing. Seal the vial as soon as possible. Determine sample weight (±0.0001 g). After prepressurization, condition the vial for 1 hour at 90° C in the analyzer bath. Determine the TS on the slurry sample (Section 8.3.5).

8.2.4 Inprocess Wastewater Samples. Using a tared vial (tared, including septum and aluminum cap) quickly add approximately 1 cc of water using a medicine dropper. Seal the vial as soon as possible. Determine sample weight (±0.0001 g). Prepressurize the vial, and then condition for 1 to 2 hours as required at 90° C in the analyzer bath.

8.3 Analysis.

8.3.1 Preparation of Equipment. Install the chromatographic column and condition overnight at 160° C. In the first operation, Porapak columns must be purged for 1 hour at 230° C.

Do not connect the exit end of the column to the detector while conditioning. Hydrogen and air to the detector must be turned off while the column is disconnected.

8.3.1.1 Flow Rate Adjustments. Adjust flow rates as follows:

a. Nitrogen Carrier Gas. Set regulator on cylinder to read 50 psig. Set regulator on chromatograph to produce a flow rate of 30.0 cc/min. Accurately measure the flow rate at the exit end of the column using the soap film flowmeter and a stopwatch, with the oven and column at the analysis temperature. After the instrument program advances to the "B" (backflush) mode, adjust the nitrogen pressure regulator to exactly balance the nitrogen flow rate at the detector as was obtained in the "A" mode.

b. Vial Prepressurizer Nitrogen. After the nitrogen carrier is set, solve the following equation and adjust the pressure on the vial prepressurizer accordingly.

$$P = \frac{T_1}{T_2}\left[P_1 - \frac{P_{w1} - P_{w2}}{7.50}\right] - 10 \text{ k Pa}$$

Where:

T_1 = Ambient temperature, °K.

T_2 = Conditioning bath temperature, °K.

P_1 = Gas chromatograph absolute dosing pressure (analysis mode), k Pa.

P_{w1} = Water vapor pressure @ 90° C (525.8 mm Hg).

P_{w2} = Water vapor pressure @ 22° C (19.8 mm Hg).

7.50 = mm Hg per k Pa.

10 k Pa = Factor to adjust the prepressurized pressure to slightly less than the dosing pressure.

Because of gauge errors, the apparatus may *over-pressurize the vial. If the vial pressure is* at or higher than the dosing pressure, an audible double injection will occur. If the vial pressure is too low, errors will occur on resin samples because of inadequate time for headspace gas equilibrium. This condition can be avoided by running several standard gas samples at various pressures around the calculated pressure, and then selecting the highest pressure that does not produce a double injection. All samples and standards must be pressurized for 60 seconds using the vial prepressurizer. The vial is then placed into the 90° C conditioning bath and tested for leakage by placing a drop of water on the septum at the needle hole. A clean, burr-free needle is mandatory.

c. Burner Air Supply. Set regulator on *cylinder to read* 50 psig. *Set regulator on* chromatograph to supply air to burner at a rate between 250 and 300 cc/min. Check with bubble flowmeter.

d. Hydrogen Supply. Set regulator on cylinder to read 30 psig. Set regulator on chromatograph to supply approximately 35 ± 5 cc/min. Optimize hydrogen flow to yield the most sensitive detector response without extinguishing the flame. Check flow with bubble meter and record this flow.

8.3.1.2 Temperature Adjustments. Set temperatures as follows:

a. Oven (chromatograph column), 140° C.

b. Dosing Line, 150° C.

c. Injection Block, 170° C.

d. Sample Chamber, Water Temperature, 90° C ± 1.0° C.

8.3.1.3 Ignition of Flame Ionization Detector. Ignite the detector according to the manufacturer's instructions.

8.3.1.4 Amplifier Balance. Balance the amplifier according to the manufacturer's instructions.

8.3.2 Programming the Chromatograph. Program the chromatograph as follows:

a. I—Dosing or Injection Time. The normal *setting is 2 seconds.*

b. A—"Analysis Time." The normal setting is approximately 70 percent of the VCM retention time. When this timer terminates, the programmer initiates backflushing of the first column.

c. B—Backflushing Time. The normal setting is double the "analysis time."

d. W—Stabilization Time. The normal setting is 0.5 min to 1.0 min.

e. X—Number of Analyses Per Sample. The normal setting is one.

8.3.3 Preparation of Sample Turntable. Before placing any sample into turntable, be certain that the center section of the aluminum cap has been removed. All samples and standards must be pressurized for 60 seconds by using the vial prepressurizer. The numbered sample vials should be placed in the corresponding numbered positions in the turntable. Insert samples in the following order:

Position 1 and 2—Old 2000-ppm standards for conditioning. These are necessary only after the analyzer has not been used for 24 hours or longer.

Position 3—50-ppm standard, freshly prepared.

Position 4—500-ppm standard, freshly prepared.

Position 5—2000-ppm standard, freshly prepared.

Position 6—4000-ppm standard, freshly prepared.

Position 7—Sample No. 7 (This is the first sample of the day, but is given as 7 to be consistent with the turntable and the integrator printout.)

After all samples have been positioned, insert the second set of 50-, 500-, 2000-, and 4000-ppm standards. Samples, including standards, must be conditioned in the bath of 90° C for 1 hour (not to exceed 5 hours).

8.3.4 Start Chromatograph Program. When all samples, including standards, have been conditioned at 90° C for 1 hour, start the analysis program according to the manufacturer's instructions. These instructions must be carefully followed when starting and stopping a program to prevent damage to the dosing assembly.

8.3.5 Determination of TS. For wet cake, slurry, resin solution, and PVC latex samples, determine TS for each sample by accurately weighing approximately 3 to 4 grams of sample in an aluminum pan before and after placing in a draft oven (105 to 110° C). Samples must be dried to constant weight. After first weighing, return the pan to the oven for a short period of time, and then reweigh to verify complete dryness. The TS are then calculated as the final sample weight divided by initial sample weight.

9. *Calibration.* Calibration is to be performed each 8-hour period when the instrument is used. Each day, prior to running samples, the column should be conditioned by running two 2000-ppm standards from the previous day.

9.1 Preparation of Standards. Calibration standards are prepared as follows: Pipet 100μl or about two equal drops of distilled water in the sample vial, then fill the vial with the VCM/nitrogen standard, rapidly seal the septum, and seal with the aluminum cap. Use a ¼-in. stainless steel line from the cylinder to the vial. Do not use rubber or tygon tubing. The sample line from the cylinder must be purged (into a properly vented hood) for several minutes prior to filling the vials. After purging, reduce the flow rate to 500 to 1000 cc/min. Place end of tubing into vial (near bottom). Position a septum on top of the vial, pressing it against the ¼-in. filling tube to minimize the size of the vent opening. This is necessary to minimize mixing air with the standard in the vial. Each vial is to be purged with standard for 90 seconds, during which time the filling tube is gradually slid to the top of the vial. After the 90 seconds, the tube is removed with the septum, simultaneously sealing the vial. Practice will be necessary to develop good technique. Rubber gloves should be worn during the above operations. The sealed vial must then be pressurized for 60 seconds using the vial pressurizer. Test the vial for leakage by placing a drop of water on the septum at the needle hole.

9.2 Preparation of Chromatograph Calibration Curve.

Prepare two 50-, 500-, 2000-, and 4000-ppm standard samples. Run the calibration samples in exactly the same manner as regular samples. Plot A_s, the integrator area counts for each standard sample, versus C_c, the concentration of vinyl chloride in each standard sample. Draw a straight line through the points derived by the least squares method.

10. *Calculations.*

10.1 Response Factor. If the calibration curve described in Section 9.2 passes through zero, a response factor, R_f, may be used to compute vinyl chloride concentrations. To compute a response factor, divide any particular A_s by the corresponding C_c.

$$R_f = \frac{A_s}{C_c} \qquad \text{Eq. 107-1}$$

Where:

A_s = Chromatograph area counts of vinyl chloride for the sample.

P_a = Ambient atmospheric pressure, mm Hg.

R_f = Response factor in area counts per ppm VCM.

T_1 = Ambient laboratory temperature, °K.

M_v = Molecular weight of VCM, 62.5 g/mole.

V_g = Volume of the vapor phase, cm^3.

R = Gas constant, (62360 cm_3) (mm Hg/mole) (°K).

m = Sample weight, g.

K_p = Henry's Law Constant for VCM in PVC @ 90° C, 6.52×10^{-6} g/g/mm Hg.

If the calibration curve does not pass through zero, the calibration curve must be employed to calculate each sample concentration unless the error introduced by using a particular R_f is known.

10.2 Residual Vinyl Chloride Monomer Concentration, (C_{rvc}) or Vinyl Chloride Monomer Concentration. Calculate C_{rvc} in ppm or mg/kg as follows:

$$C_{rvc} = \frac{A_s P_a}{R_f T_1}\left[\frac{P_v V_g}{R m} + K_p (TS) T_2 + K_w (1 - TS) T_2\right] \qquad \text{Eq. 107-2}$$

Results calculated using these equations represent concentration based on the total sample. To obtain results based on dry PVC content, divide by TS.

TS = Total solids expressed as a decimal fraction.

T_2 = Equilibrium temperature, °K.

K_w = Henry's Law Constant for VCM in water (at 90° C, 7×10^{-7} g/g/mm Hg.

Assuming the following conditions are met, these values can be substituted into Equation 107-2:

P_a = 750 mm Hg.

V_g = Vial volume—sample volume (Fisher vials are 22.0 cm^3 and Perkin-Elmer vials are 21.0 cm^3).

T_1 = 23° C or 296° K.

T_2 = 90° C or 363° K.

$$C_{rvc} = \frac{A_s}{R_f} \frac{750}{296}\left[\frac{65.5\left(21.3 - \frac{m(TS)}{1.38} - \frac{m(1-TS)}{0.38V_g}\right)}{62360\ m} + 6.25 \times 10^{-6}(TS)(363) + 7.0 \times 10^{-7}(1-TS)(363)\right]$$

11. *References.*

1. B.F. Goodrich, Residual Vinyl Chloride Monomer Content of Polyvinyl Chloride Resins, Latex, Wet Cake, Slurry and Water Samples. B.F. Goodrich Chemical Group Standard Test Procedure No. 1005–E. B.F. Goodrich Technical Center, Avon Lake, Ohio. October 8, 1979.

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40 CFR Part 61 is amended by adding Appendix C as follows:

Appendix C.—Quality Assurance Procedures

* * * * *

Procedure 1—Determination of Adequate Chromatographic Peak Resolution

In this method of dealing with resolution, the extent to which one chromatographic peak overlaps another is determined.

For convenience, consider the range of the elution curve of each compound as running from -2σ to $+2\sigma$. This range is used in other resolution criteria, and it contains 95.45 percent of the area of a normal curve. If two

peaks are separated by a known distance, it one can determine the fraction of the area of one curve that lies within the range of the other. The extent to which the elution curve of a contaminant compound overlaps the curve of a compound that is under analysis is found by integrating the contaminant curve over the limits $b-2\sigma_s$ to $b+2\sigma_s$, where σ_s is the standard deviation of the sample curve.

This calculation can be simplified in several ways. Overlap can be determined for curves of unit area; then actual areas can be introduced. Desired integration can be resolved into two integrals of the normal distribution function for which there are convenient calculation programs and tables. An example would be Program 15 in Texas Instruments Program Manual ST1, 1978, Texas Instruments, Inc., Dallas, Texas 75222.

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The standards for calibration of the gas chromatograph should be prepared by injecting 40 uL of vapor into the headspace above 5 mL of carbon disulfide. A teflon lined silicone septum (Cat. No. 0-26) is recommended for sealing the vials and for preparation of standards.

REAGENTS

Only activated charcoal Cat. No. 0-21 (or 0-22 in vials) or equivalent should be used. Carbon disulfide (reagent or spectroscopic grade) should be as pure as possible to speed analysis time.

CALIBRATION OF THE MONITOR

The monitor should be calibrated before use if not purchased calibrated. Expose the device to a known concentration (10 ppm) of vinyl chloride for a given period of time (2 hr). This can be done conveniently by placing the monitor in an aluminum or glass chamber (Cat. No. 0-25) with the calibration gas moving through the chamber at about 1 L per minute per monitor being exposed. Permeation tubes are probably the most accurate source of vinyl chloride while compressed gas standards are more convenient to use. The recommended procedures above should be used for charging the device and analyzing the charcoal. The calibration constant can be calculated from the equation:

$$k = \frac{c\ t}{w}$$

** The same sampling and desorption procedures can be used for Freon-12.

REAL

REISZNER ENVIRONMENTAL & ANALYTICAL LABS, INC.

MINIMONITOR

RECOMMENDED OPERATING PROCEDURE
FOR MEASURING VINYL CHLORIDE IN AIR
Also Freon 12**

1. Pour* 1.35 g of activated charcoal (Cat. No. 0-21 or 0-22) into the MiniMonitor and attach the monitor to an individual as close to the breathing zone as is practical.

2. After exposure, normally 1 to 8 hr, pour the charcoal into a 7 mL glass vial.* Seal the vial with a teflon faced silicone septum (Cat. No. 0-26) and refrigerate the sample at -20°C if it is to be stored for more than 8 hrs.

ANALYSIS

1. Cool the sample to -20°C (in a freezer) and add 5 mL of cold (dry ice) carbon disulfide. The desorption apparatus (Cat. No. 0-24) in figure 1 is particularly convenient for this purpose because it allows the carbon disulfide to be measured at room temperature.

2. Recap the vial immediately after adding the carbon disulfide and allow to stand 30 minutes.

3. Shake the vials at 10 and 20 minutes after solvent addition.

4. Inject 5 uL into a gas chromatograph equipped with a flame ionization detector. The column used should be chosen to separate possible interfering species that occur in the environment being sampled; however, a 6 ft 1/8 in. Chromosorb 102 (Johns Mansville) or a 20 ft. 1/8 in. FFAP column are commonly used. An internal standard that is not removed from solution by the charcoal is recommended.

5. Calculate the concentration from the equation:

$$c = \frac{k\,w}{t}$$

where
k = constant
c = concentration of vinyl chloride, ppmv
t = time of exposure, hr
w = volume of vinyl chloride, µL @ 25°C

* To facilitate rapid addition and removal of charcoal a vibrator (engraver) may be touched to the badge clip or the MiniMonitor may be thumped during the process.

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REAL
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CERTIFICATE OF CALIBRATION

The MiniMonitor has been calibrated at least two times to assure a guaranteed maximum error of 5%. The charcoal used in calibration was PCB 20-40 mesh and was reactivated at 350° under a stream of nitrogen. To calculate the concentration of vinyl chloride use the equation:

$$C = \frac{w\,k}{t}$$

where C = concentration
w = amount of gaseous pollutant in sample (µℓ at 25°C)
t = time, (hr)
k = constant $(\frac{ppm,hr}{\mu\ell})$

MiniMonitor No.	k*
641	0.84
642	0.88
647	0.71

Certifying Agent _____
Kenneth D. Reiszner
Ph.D., President
12/10/81

* The charge of PCB should be 1.35 g.

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$$C = \frac{w\,k}{t}$$

where C = concentration
w = amount of gaseous pollutant in sample (μℓ at 25°C)
t = time, (hr)
k = constant $\left(\frac{ppm,hr}{\mu\ell}\right)$

MiniMonitor No.	k*
622	0.72
638	0.79
639	0.76

Certifying Agent _____
Kenneth D. Reiszner
Ph.D., President
10/19/81

* The charge of PCB should be 1.35 g.

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REISZNER ENVIRONMENTAL & ANALYTICAL LABS., INC.

CERTIFICATE OF CALIBRATION

The MiniMonitor has been calibrated at least two times to assure a guaranteed maximum error of 5%. The charcoal used in calibration was PCB 20-40 mesh and was reactivated at 350° under a stream of nitrogen. To calculate the concentration of vinyl chloride use the equation:

$$C = \frac{w\,k}{t}$$

where C = concentration
w = amount of gaseous pollutant in sample (μℓ at 25°C)
t = time, (hr)
k = constant $\left(\frac{ppm,hr}{\mu\ell}\right)$

MiniMonitor No.	k*
552	0.82
546	0.85
553	0.84

Certifying Agent

Kenneth D. Reiszner
Ph.D., President
7/13/81

* The charge of PCB should be 1.35 g.

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ENVIRONMENTAL & ANALYTICAL LABS, INC.

CERTIFICATE OF CALIBRATION

The MiniMonitor has been calibrated at least two times to assure a guaranteed maximum error of 5%. The charcoal used in calibration was PCB 20-40 mesh and was reactivated at 350° under a stream of nitrogen. To calculate the concentration of vinyl chloride use the equation:

$$C = \frac{w\,k}{t}$$

where C = concentration
w = amount of gaseous pollutant in sample (µℓ at 25°C)
t = time, (hr)
k = constant $\left(\frac{ppm,hr}{\mu\ell}\right)$

MiniMonitor No.	k*
648	0.67
649	0.77
650	0.78

Certifying Agent

Kenneth D. Reiszner
Ph.D., President
12/18/81

* The charge of PCB should be 1.35 g.

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ANALYSES OF VINYL CHLORIDE MONOMER BY METHOD 107 USING THE SIGMA 2000 WITH HS 100 HEADSPACE SAMPLER

SCOPE:

The Sigma 2000 and HS 100 Headspace Sampler are used to analyze the vinyl chloride monomer concentration in polyvinyl chloride resins, slurries, and water samples according to Method 107 of the Federal Register, Subpart F- National Emission Standard for Vinyl Chloride, 7/30/80, pages 121:0504 - 121:0506. Method 107, Section 6.3.1 allows the use of a GC equivalent to the Perkin Elmer F-40, F-42, or F-45 Headspace Analyzers.

PRINCIPLE:

The vinyl chloride monomer, as a gas, in equilibrium with resin, slurry or water in a heated sealed vial is measured and this amount measured in the headspace is mathematically related to the amount present in the solid or liquid samples.

APPARATUS:

1. Sigma 2000 gas chromatograph, Perkin Elmer
2. HS-100 headspace sampler, Perkin Elmer
3. Glass vials with butyl rubber septa, Perkin Elmer
4. Vial sealer, Perkin Elmer, part #105-0106
5. Stainless steel GC columns, 6' x 1/8", 3' x 1/8" bath containing 0.2% carbowax 1500 on Carbopak C.
6. Analytical balance: capable of weighing to .0001 gms.

REAGENTS:

1. Hydrogen, zero grade
2. Nitrogen, zero grade
3. Air, zero grade
4. Calibration standard of 50, 500, 2000 and 4000 ppm of VCM in Nitrogen, Matheson Scientific

INSTRUMENT CONDITIONS:

1. Set hydrogen and air flow at rates to optimize detector sensitivity as specified in the Sigma 2000 manual.
2. Oven temperature: 70°C
3. Analyses time: 6 minutes
4. Range: 1
5. Attenuation: X 8

6. Sample Temp.: 90°C

7. Transfer Temp.: 140°C

8. Thermostatting Time: 60 minutes

9. Pressurization Time: 0.5 minutes

10. Injection Time: 0.04 minutes

11. Withdrawal Time: 0.2 minutes

12. Precut Off Time: 0.00

13. Backflush on/off: 1.00 2.00

14. Vent 1 = YES/-1 = NO: -1

PROCEDURE:

1. Prepare samples and calibration standards for analyses.

   a. Weigh 3.5 to 4.5 gms of resin into tared sample vial. Record exact weight to .0001 gms.

   b. Filter slurry samples and place 0.2 to 4 gms of the wet slurry into a tared sample vial. Determine solids on wet slurry by placing 3 to 4 grams of filtered sample in a tared aluminum weighing pan and determining initial and final weight after drying to a constant weight at 105-110°C.

   c. Place approximately 1 cc of water in tared sample vial and record exact weight to 0.0001 gms.

   d. Place 100 ul of water into each of 8 sample vials and crimp septa on tops. Purge sample line from calibration gas cylinder and place needle thru septum of sample vial. Purge vial at 500-1000cc/min. for 90 seconds. By this procedure fill 2 sample vials with each of the 4 calibration gas mixtures.

2. Load Magazine

   a. Position 1 and 2 should contain old 2000 ppm standards for conditioning if the instrument has not been used for 24 hours or longer.

   b. Position 3 - 50 ppm standard, freshly prepared.

   c. Position 4 - 500 ppm standard, freshly prepared.

   d. Position 5 - 2000 ppm standard, freshly prepared.

   e. Position 6 - 4000 ppm standard, freshly prepared.

   f. Position 7 thru 95 (as many spaces as required) should contain the samples to be analyzed.

   g. A second set of standards, 50, 500, 2000, and 4000 ppm should be inserted immediately following the last sample.

3. GC and HS-100 Conditions

a. Get a list of chromatographic functions.
Press SECTION and ENTER
Display should read:

```
CHROMATOGRAPHY FUNCTIONS
SECTION 1 INSTR STATUS
SECTION 2 ACTIVE METHOD
SECTION 3 METHOD CONTROL
SECTION 4 HS-100
```

b. List the present GC status.
Press #1 and ENTER
Display should read:

```
METH #1 (Active Method)
CHAN:  A  FID     B (Channel Designation)
RANGE:  1
ATTN:  8    X2
(If the cursor is positioned next to the X2 pressing
or   keys change the attenuation values.)
ZERO:  A          B
EQUTIME:  0.5     ACT (Status Displayed)
OVEN:  SET        ACT
Temp 1  70        (Status Displayed)
Time 1   6.00
Rate 1   0
HS-100 (Status Displayed)
```

c. List the active method.
Press #2 and ENTER
Display should read:

```
METH #(normally 1)
OVEN
  TEMP:  70
  TIME:   6.00
  RATE:   0
FLOWS  SET  ACT  ZONES  SET  ACT
   A1        0   INS A  140  140
   A2        2   DET A  160  160
                 INJ B  140  140
AUX 1     45
RUNTIME:  6.0   ACT:  (status displayed)
OVEN ACT:  70.1  STATUS:  (status displayed)
```

Press ADVANCE & ENTER
Should display:

```
OVEN MAX TEMP:  150
EQUILIBRATION TIME:  0.50
CHAN:  A FID     RANGE 1
(won't use RUN TIMED EVENTS)
```

d. List the number of the currently set up method.
Press SECTION and ENTER. Press #3 and ENTER.
The display should read:

	ENTER
SET UP/VOID	1
GENERATE/EDIT	2
DELETE	3
COPY	4
STORE SET UP	5
TRANSFER	6
PRINT	7

Press No. 1 and ENTER.
Display should show:

THE FOLLOWING METHODS EXIST:
1 (current method set up to run)
THE SET UP METHOD IS: 1 (Must agree with method number in section 1 and 2.)
ENTER METHOD NUMBER:
Use if setting up a new method.

e. Display conditions Set Up for HS-100
Press SECTION and ENTER.
Press No. 4 and ENTER.
Press No. 3 and ENTER.
Press method no. to be used and ENTER.
Press 1 (constant mode) and ENTER.
The H-100 conditions for the method specified should be displayed. Compare with the values listed for instrument conditions in this method (assuming only one method exists). This same procedure may be used to create new HS-100 methods.

f. Actually start analyses by specifying 1st and last vial to be analyzed.
Press SECTION and ENTER.
Press No. 4 and ENTER.
Press No. 2 and ENTER.
The display should read:

FIRST VIAL LAST HS-PROGRAM #
1
START AT VIAL # oo
STATUS HS-100 (status displayed)

Enter the number of the first vial to be analyzed. The cursor will move below the word LAST. Enter the no. of the last vial to be analyzed. The cursor will move below HS-PROGRAM #. Enter the number of the HS program being used. Normally will be no. 1. The cursor will move to a position below the first vial entered so that a second sequence of vial number may be entered if desired (nine sequences may be entered). Press ENTER to move the cursor to the right of START AT VIAL #. Enter the first vial in the first sequence from top of screen. The display will change to read STOP AT VIAL #. Enter the last vial which will be analyzed.

When this is entered the sample positions in the magazine of the HS-100 will shift to position the vial # listed as first to be analyzed below the carousel.

The current vial rotating past the sampling position of the carousel will automatically be displayed on the screen.

The STATUS of the HS-100 will be displayed on the screen.

The sequence will automatically start after entering the first and last vials to be analyzed.

g. Setting up a new GC Method.
If it is necessary to generate new GC conditions, a new GC method must be generated by pressing SECTION and ENTER.
Press No. 3 and ENTER.
Press No. 2 and ENTER.
Enter method number: Enter the number of the method you are creating.
A menu will appear with the method number you specify. Enter the values you desire for GC control.

4. Delete old sequence and prepare new sequence.

The sequence can be prepared while the samples are thermostating in HS-100.

a. Get into LAS by typing BYE if the terminal says BASIC >. If the terminal reads PLEASE LOG ON when RETURN is hit, type in JK.JK and RETURN followed by PASSWORD? JK. The terminal will be in file manager: Type in LAS to get into LAS which is designated by a READY*.

b. Type: C1,2 and hit RETURN.

c. Type: DE,S,SEQ2 and hit RETURN.

d. Type: PR,S,SEQ2 and hit RETURN.

e. In response to channel #? Type in 2 and hit RETURN.

f. The program will ask for method? Type in ZERO2. This method has been previously prepared for channel 2 and is simply an area percent method.

g. In response to the next 3 questions the program asks simply hit RETURN and don't answer.

h. The next input required is for SAMPLES. The appearance will be:

SAMPLES
SAMPLE-NAME BLT# PROC-FILE RAW-FILE %DIL-F STD-AMT SMP-AMT
1*
The 1st response should be to type in the 1st sample name which should be the COND SAMPLE. Type in COND1.Then type in 2 commas, , to bypass BTL#. Type in PROC-FILE name (ex: P1) and a comma. Type in RAW-FILE name (ex: R1) and a comma. Type in 100.00 and a comma for % DIL-F. Type in 1.00 and a comma for STD-AMT. Type in 1.00 for SMP-AMT.

The last three responses for %DIL-F, STD-AMT, and SMP-AMT only have to be typed in on the first line. They will automatically be copied for subsequent lines. Example of 1st line:

COND1, , P1, R1, 100.00, 1.00, 1.00

Example of 2nd line:

COND 2, , P2, R2,

Example of 3rd line:

S51, , P3, R3,

etc. until all samples and standards have been entered. The names entered should be descriptive of the sample or std. A different process and raw file name must be associated with each name so that each chromatogram may be stored in different raw and process files. If the raw and process file names were the same for 2 different sample names the data from the second chromatogram would simply over-write the first and the data from the 1st chromatogram would be lost.

After the last sample name is entered, (should be the 4000 ppm std.) type /E and hit RETURN to end the sequence.

It will respond with SUBSEQUENCE 2? Type in NO, hit RETURN.

It will respond with DONE. Hit RETURN to get READY*.

5. Associate sequence with a channel.

   a. Type in CH, 2, SEQ2 and hit RETURN.

   b. In response to DONE, hit RETURN.

   The sequence is now ready to start putting all area slices from each chromatogram into the RAW files specified in the sequence to integrate the area slices into peaks and store this data in the specified process files. The contents of the process files will automatically appear on the 5880 printer as each sample is analyzed.

6. Turn on the Perkin Elmer recorder so the peaks may be observed as they elute.

7. Calculation of VCM results.

   a. Obtain areas of VCM peak at RT of 1.35 min. on each printout for the 8 calibration standards.

   b. Type BA on the 588C keyboard.

   c. Type RU, P2088

   d. Answer questions as follows:

   NUMBER OF DATA PAIRS? 8
   TYPE IN DATA PAIRS. (X, Y - RETURN)
   Enter each AREA, then the conc (ppm)
   ex: 42,000, 51
   Do Not Enter the Conc First.
   Modifications (Y/N)? Type N if all data was entered correctly.
   OUTDVC? T2

DETAILS FOR CURVE TYPE? 0
Take values of A and B from equation 1 Y = A + (B*X)
A = Y intercept
B = slope

e. Type RU, HS.
Program will ask for value of A determined above.
Type in value for A.
Program will ask for value of B determined above.
Type in value for B.
Program will ask for area of peak for first sample to be calculated.
Type in area determined in step 6.a.

Program will ask for weight of sample used. Type in value obtained in step 1, a, b, or c depending upon the type sample. Use uncorrected solids weight for slurry samples. Program will ask which type sample is being analyzed (1) slurry, (2) resin, (3) water. Type in a 1, 2, or 3. If the sample is a slurry the program will ask for the percent solids expressed as a decimal and it will correct for solids.