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Date April 2, 1979
Originating Dept. R & D
Answering letter date

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Subject Determination of VCM in PVC Resins

The items discussed in our phone conversation this morning are attached. The reprint of the article from the Journal of the Association of Official Analytical Chemists emphasizes the necessity of confirming peak assignments. I have underlined sections on pages 813, 815, 816, 818, and 819 which state the importance of a mass spectrometer in proving the presence of residual monomer. It should be noted that this article, which was published by Food and Drug Administration personnel, discusses vinyl chloride homopolymers that are less complex than copolymers.

The other attachment is a partial listing of compounds noted in the literature as being ones that could interfere in the determination of residual monomer in vinyl chloride homopolymer or copolymers. This data was obtained from the American Society for Testing and Materials compilations and various scientific journals.

If we can be of any further help in this matter, let us know.

T. B. Gibb

TBG/fp
Att.

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INDUSTRIAL CHEMICALS

Headspace Sampling and Gas-Solid Chromatographic Determination and Confirmation of ≥ 1 ppb Vinyl Chloride Residues in Polyvinyl Chloride Food Packaging

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The determination and confirmation of residual vinyl chloride (VC) in polyvinyl chloride (PVC) food packaging materials are described. PVC packaging materials are dissolved in dimethylacetamide (DMAC). VC is sparged from solution with helium gas and collected in sealed vials of ethanol. VC is detected and quantitated by using a headspace sampling technique and standard gas-solid chromatography (GSC) with flame ionization detection. GSC peak heights of about 5% full scale deflection are obtained for samples of PVC containing 1 ppb VC. Polymer samples taken from tubing, blood bags, food packaging films, bottles, and unprocessed resin were analyzed. Residual VC levels ranged from 0.3 to 913 ppb. VC was confirmed by GSC-mass spectrometry, using selected ion recording of m/z 62 and 64 in conjunction with full mass scans to identify components eluting near VC.

The detection and quantitation of vinyl chloride (VC) in various matrices have been continuing problems for the past several years. Since the implication of VC as a cause of angiosarcoma of the liver (1) and several other diseases (2), there has existed a need for a highly sensitive and specific analytical method for determining and confirming residual VC in PVC food packaging materials. Previous work in this laboratory (3) was limited to quantitative determination of VC from PVC solutions at the 1 ppm level by direct gas-solid chromatographic (GSC) injection.

Recent work has shown that headspace sampling techniques can increase sensitivities to the low ppb range (4–7). PVC materials or PVC solutions were held in sealed vials and heated for fast equilibration between liquid or solid and vapor phases. A large aliquot of the headspace was withdrawn for GSC or GSC-mass spectrometric (MS) injection.

Apparent sensitivity has been further increased by concentrating VC from the polymer solution into another solvent, using a sparging technique. This was suggested by Bellar and Lichtenberg (8) and has been used by Japanese scientists[1] for several years.

We have modified the sparging and headspace sampling techniques to obtain the described procedures which permit the determination and confirmation of ≥ 1 ppb VC in PVC materials. Polymer samples of tubing, bottles, blood bags, food packaging films, and unprocessed PVC resins were analyzed using flame ionization detection (FID) and confirmed by GSC-MS.

METHOD

Caution: Since VC is a gas at room temperature and has been designated a carcinogen (1, 9, 10) by inhalation, special handling precautions must be taken to preserve the integrity of the standards as well as to ensure the safety of the analyst. Only the septa-sealed vials described under *Apparatus* were found satisfactory for handling VC.

Reagents

(a) *Vinyl chloride.*—(*1*) 3 lb cylinder (99.9% pure) (Matheson Gas Products, 6655 Amberton Dr, Dorsey, MD 21227, or equivalent). (*2*) VC gas standard (0.48 ppm v/v) containing 1.25 ng VC/ml nitrogen (MG Scientific Gases, Kearny, NJ 07029, or equivalent).

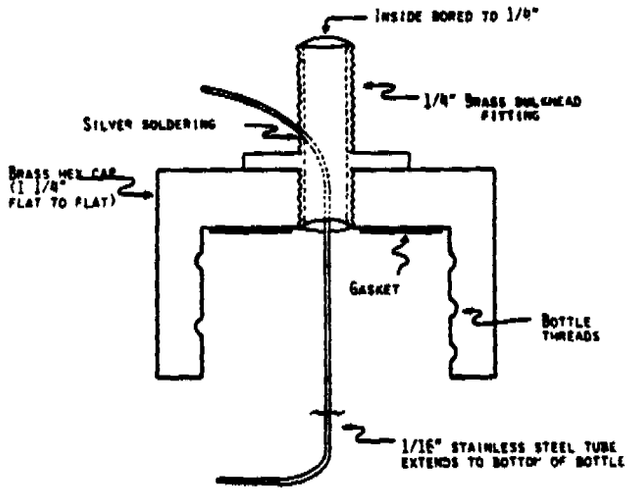
(b) *Ethanol.*—Absolute (Publicker Industries, Inc., 1429 Walnut St, Philadelphia, PA 19102, or equivalent). Before use, test to show absence of co-eluting materials at retention time of VC under conditions of analysis.

C. V. Breder is Associate Referee on Indirect Additives From Food Packages.

This paper was presented at the 91st Annual Meeting of the AOAC, Oct. 17-20, 1977, at Washington, DC.

[1] Analytical method attached to Dec 19, 1975 letter from Sumitomo Bakelite Co., Ltd. 345 Park Ave, New York, NY 10022, to Hearing Clerk, Food and Drug Administration, 5600 Fishers Lane, Rockville, MD 20852.

0004-5756/78/6104-0813$02.10



CAP DETAILS

FIG. 1—Cap details: hexagonal cap machined from solid brass (1⅛ in. flat-to-flat), threaded to same configuration as glass bottle, drilled, and tapped in the top to accommodate ¼ in. Swagelok bulkhead fitting.

(e) *Dimethylacetamide (DMAC).*—3 kg bottle (Eastman Practical Grade, Eastman Chemical Products, PO Box 431, Kingsport, TN 37662, or equivalent). Pre-sparge entire reagent bottle with helium for 2–3 days before use. Before use, test to show absence of co-eluting materials at retention time of VC under conditions of analysis.

Apparatus

(a) *Screw-capped bottles.*—1 oz narrow mouth, 35±0.5 ml (Ace Scientific Supply Co., 1420 East Linden Ave, Linden, NJ 07036, No. 10-4256, or equivalent). Similar bottles with 2 oz capacity. Caps with Teflon-lined septa (Alltech Associates, 202 Campus Dr, Arlington Heights, IL 60004, No. 9522, or equivalent).

(b) *Gas syringe.*—2, 5, and 10 ml (Precision Sampling Corp., PO Box 15119, Baton Rouge, LA 70815, Pressure-Lok, Series A-2, or equivalent).

(c) *Mini-Vials.*—5 ml, with caps and Teflon-lined septa (Alltech Associates, Inc., No. 9500, or equivalent).

(d) *Collection vials.*—Perkin-Elmer F-42 headspace analyzer vials with gray septa and aluminum seals. Capper (P-E No. 105-0106) and decapper (P-E No. 105-0107) for vials, or equivalent.

(e) *Sparging apparatus.*—See descriptive drawings in Figs 1 and 2. For temporary use and preliminary tests, a standard metal cap may be used by removing liner, drilling hole through top, removing surface paint with acetone, and soft-soldering in a bulkhead fitting with sparge tube attached. Sparge tube is attached to bulkhead fitting by drilling hole slightly larger than 1/16 in. downward at angle into bore of fitting (see Fig. 1). Flats of nut portion and upper major part of threads should be avoided. After sparge tube is inserted to proper length (enough to reach bottom of bottle), tube is silver-soldered in place. Thick gasket is then cut to fit inside of cap. Threads on standard metal caps are not sturdy enough for continual use and often strip off after a short time. Plastic caps are unsatisfactory for even temporary usage.

(f) *Forced draft oven.*—Precimon Scientific Co., 3737 W Cortland St, Chicago, IL 60747, Thelco Model 18, or equivalent.

(g) *Gas chromatograph.* — Hewlett-Packard Model 7620A, or equivalent, equipped with temperature programmer and flame ionization detectors (using 0.01 in. id flame jets). Operating conditions: Temperatures (°C)—injection port 200, detector 180, column 110 (after VC elution (ca 7 min), program column to 180°C at fastest rate and hold for 20 min); gas flows (ml/min)—helium carrier 35 (0.7 rotameter), helium makeup 30, oxygen 345 (3.35 rotameter), hydrogen 35 (0.75 rotameter); electrometer setting, 2×10^{-12} amp full scale (AFS).

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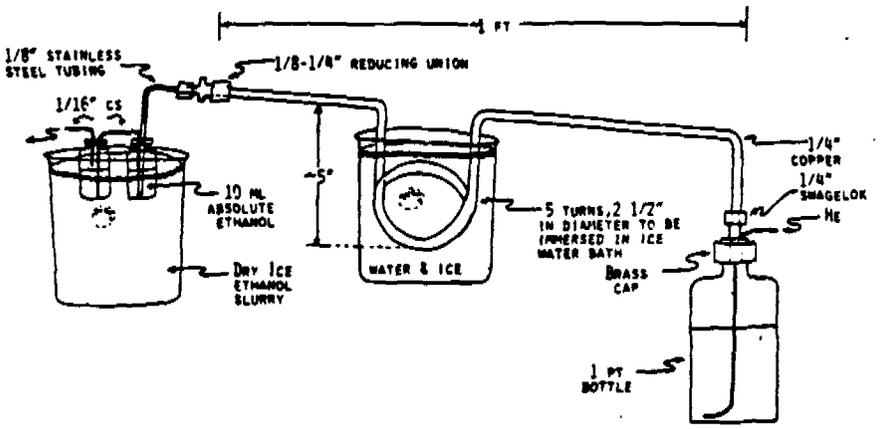

EFFLUENT TUBE DETAILS

FIG. 2—Sparge train details: requires 7 × ⅛ in. stainless steel tubing, ⅛ to ⅛ in. reducing union, 5 ft × ¼ in. copper tubing, and brass cap as shown in Fig. 1.

(h) *Chromatographic column*.—6 ft × ⅛ in. od coiled stainless steel, filled with 80–100 mesh Porapak N; retention time of VC ca 7 min.

(i) *Gas chromatograph-mass spectrometer*.—Finnigan Model 3300F electron impact quadrupole mass spectrometer equipped with Finnigan 9500 gas chromatograph and Finnigan Model 6100 data system. Full mass scans and selected ion recording of m/z 62 and 64 were obtained under computer control. Operating conditions: Temperatures (°C)—injector 170, column 155, separator 215, transfer line 210 (after VC eluted, column was vented and programmed to 180°C to clear solvent from column); helium carrier gas 25 ml/min; sample size 200 ml headspace gas at 99°C; columns tested under (h).

(j) *Pint bottles*.—Flint glass, 16 oz narrow mouth, screw-cap (Fisher Scientific Co., 7722 Fenton St, Silver Spring, MD 20910, No. 2-883DD, or equivalent), with foil-lined caps.

Preparation of Standards

Accurately weigh 2 oz narrow mouth bottle, Mini-Vial cap, and septum. Add 50 ml absolute ethanol to weighed bottle. Cap, and reweigh. In hood, prepare VC stock solution in this bottle by quickly uncapping bottle and adding 0.5–2 g liquid VC from inverted freezer-cooled cylinder of VC. Immediately cap bottle tightly and mix well by shaking. Reweigh and calculate VC concentration by weight (ca 10,000–40,000 ppm). Dilute this stock solution by withdrawing aliquots through septum with syringe and injecting into weighed, sealed container (1 oz bottle) of ethanol. Reweigh, and calculate VC concentration either in w/w or in w/v units (ca 50 ppm). This solution is similarly diluted to yield solution containing 1–5 ppm. Make final dilutions into 10 ml ethanol in collection vial. Prepare working standards in 1–50 ppb range. If refrigerated, these working standards are stable for up to 1 week. Multiple septum punctures shorten working life of standards.

Determine actual VC concentration in headspace of above working standards under conditions of analysis by direct comparison to standard gas (0.48 ppm in nitrogen) containing 1.25 ng VC/ml. Calculate average partition coefficient, using the following equation.

$$\text{Partition coefficient} = C_v/C_l = (0.13/14\ \text{ml})/(0.87/10\ \text{ml}) = 0.11 \quad (1)$$

where C_v = concentration in vapor (headspace); C_l = concentration in liquid; 0.13 = 13% determined to be in headspace; 14 ml = volume of headspace (13 ml) plus volume of syringe (1 ml); 0.87 = 87% determined by difference to remain in liquid; 10 ml = volume of ethanol standard.

Preparation of Solvents and Samples

Check absolute ethanol to be used in collection vials for materials co-eluting with VC by carefully pipetting 10.00 ml into one of the vials and sealing the vial with vial capper. Analyse as described under *Analysis*.

Drill 2 holes in cap of 3 kg reagent bottle of dimethylacetamide (DMAC) and run ⅛ in. Teflon or stainless steel tube to bottom of bottle through 1 of the holes. Sparge DMAC, using slow

flow of helium (5–15 ml/min) for several days at room temperature before use. After DMAC free of materials co-eluting at retention time for VC has been obtained (determined by analyzing reagent blank), add 200 ml to 1 pt bottle along with 2 in. Teflon-coated magnetic stirring bar.

Cut thick PVC samples such as tubing and bottles into small pieces which will fit through neck of narrow mouth pint bottles. Prepare film by rolling sample into long narrow tube which can be cut into short sections to fit into bottle. Accurately weigh 20.00 g PVC material and add to bottle containing 200 ml DMAC and stirring bar. Tightly cap bottle, using cap with foil liner, and stir contents until PVC is completely dissolved. Complete solution requires stirring from 2 hr to overnight, depending on PVC formulation involved. Highly plasticized samples dissolve more readily than do more rigid samples. (*Note:* Opaque solutions are generally obtained when the PVC composition contains impact modifiers or pigments.)

Analysis

After PVC sample is dissolved in pre-sparged DMAC, add 10.00 ml absolute ethanol to each of 2 collection vials. Interconnect vials with short lengths of ⅛ in. stainless steel tubing as shown in Fig. 2, and cool in thick Dry Ice-ethanol slurry for 30 min before use. Fit bottle containing PVC solution with tight cap equipped with ⅛ in. stainless steel sparge tube extending to bottom of bottle. The apparatus is shown in Fig. 1.

Immerse pint bottle containing PVC solution in steam bath and add helium sparge gas via needle valve through ⅛ in. stainless steel sparge tube at ca 85 ml/min. Quickly leak check apparatus by applying Snoop (Potomac Valve and Fitting, Inc., 1500 E Jefferson St, Rockville, MD 20852) to all joints and briefly plug final outlet tube to build up pressure. After leak checking, allow sparge gas to freely pass through PVC solution, through bulkhead fitting, into effluent tube and into collection vials immersed in Dry Ice-ethanol slurry. Sparging apparatus is shown in Fig. 2.

After sparging for 2–2½ hr, disconnect collection vials while still cold, decap using decapper, and re-seal using fresh unpunctured septum. Analyze vials immediately or store under refrigeration for not more than 2 days. For analysis, heat vials to 90°C in either water bath or forced draft oven and let equilibrate at this temperature for ≥1 hr. Heat gas-tight syringe having integral valve to 90°C in forced-draft oven. Wear gloves to use preheated gas-tight syringe and withdraw 1.00 ml headspace. Close syringe valve and return syringe containing sample to oven for 5 min to vaporise any condensed solvent. Remove hot syringe and immediately inject sample into gas chromatograph. Make standard VC gas injection (1.00 ml, 1.25 ng) both before and after each headspace sample injection. Quantitate unknown using the following equation:

$$\begin{aligned}\text{VC in PVC, ppb} &= \text{total ng VC in sample}/20\ \text{g} \\ &= [(\text{ng/ml in HS}) \times 14\ \text{ml} \\ &\quad + (\text{ng/ml in liq.}) \times 10\ \text{ml}]/20\ \text{g} \qquad (2)\end{aligned}$$

combined with Eq. (1), $C_v/C_l = 0.11$, where $C_l = C_v/0.11$, then

$$\begin{aligned}&\text{VC in PVC, ppb} \\ &\quad = [C_v \times 14\ \text{ml} + (C_v/0.11) \times 10\ \text{ml}]/20\ \text{g} \\ &\quad = [C_v(14\ \text{ml} + 10\ \text{ml}/0.11)]/20\ \text{g} \\ &\quad = 5.25\ C_v\end{aligned}$$

The remainder of the sample in the collection vials after GSC determination of residual VC is used to confirm the presence of VC by GSC-MS. Equilibrate samples at 90°C for 1 hr in constant temperature bath and withdraw 2.00 ml aliquot of headspace with preheated gas-tight syringe having integral valve. Inject sample quickly into GSC-MS to minimise cooling of syringe and condensation of vapor.

Calculate response areas of m/z 62 and 64, using Finnigan Revision H software. Calculate relative abundance of m/z 62 and 64 from response areas at retention time of VC and compare with theoretical 3:1 ratio.

Results and Discussion

Previous unpublished work in this laboratory indicated that quantitative determinations of VC in PVC at very low levels would require a combination of headspace sampling and sparging. Headspace techniques have the advantage of allowing larger portions of the VC present in the sample to be injected into the gas chromatograph than could be done by direct injection of solutions. Undesirable components can often be reduced or eliminated. Unfortunately, the low ppb levels of VC in solid food packaging materials could not be determined accurately by using headspace sampling alone. This is due to differing and unknown equilibration times from sample to sample. Direct headspace sampling has been shown to reliably detect low levels of VC in several food simulating solvents (6). If the polymer is dissolved in a suitable solvent, a favorable partitioning of VC into the headspace may be obtained. Our desire to use a relatively dilute polymer solution to ensure rapid equilibration resulted in a low total VC concentration in

the headspace for samples containing VC at the ppb level. Therefore, an additional concentration step was needed. Sparging provided a means of transferring VC from a solution of low concentration to one of higher concentration. The combination of sparging and headspace sampling techniques produced the desired sensitivity.

DMAC was chosen to dissolve the PVC because of its excellent solvent properties and its low volatility. Absolute ethanol was chosen as collection solvent because it remains a liquid at Dry Ice temperatures and is an excellent solvent for VC. Helium was chosen as the sparge gas because of its low solubility in ethanol.

The sparge train was designed to use readily available components, the pint bottle being chosen for its convenience in holding 20 g PVC in 200 ml DMAC without entrainment of the DMAC into the gas stream. Some bottles broke at the threads, possibly due to overtightening, machining mismatch, or differences in coefficient of expansion between the glass and brass used in the cap construction. A larger bottle (1 qt) could be used for the determination of VC at lower levels where a larger sample would be required. Figure 1 shows the details of the cap and Fig. 2 shows the assembled sparging apparatus.

GSC analysis of the headspaces of several PVC samples, following sparging, demonstrated that VC could not be adequately resolved from other components by using Carbowax 20M or Silar 10C liquid phases in conventional packed columns. Chromosorb 101, Chromosorb 108, Porapak T, Porapak R, and Tenax GC also provided inadequate resolution. Chromosorb 104 was marginally useful but is not recommended. The Porapak N column was selected for its excellent resolution of VC from all encountered interferences except one component eluting after VC which sometimes precluded baseline resolution. GSC-MS analysis showed that this component was isobutane. With the porous polymer columns, from 45 min to 1 hr was required before the FID baseline was considered adequate for the next analysis. Attempts to shorten these turn-around times by backflushing were unsuccessful. The Hall electrolytic conductivity detector and the Dohrmann microcoulometric detector were evaluated in an attempt to provide interference-free detection of VC. These detectors were difficult to use and suffered from a lack of reproducibility on a day-to-day basis. Since VC interferences were not eliminated by using these detectors, the FID remained the detector of choice.

We were unable to obtain syringes capable of continued use at 90°C. Syringe leakage problems were occasionally encountered due to sustained use at 90°C. Syringes were tested by pulling back the plunger, closing the integral valve, and pushing on the plunger while holding the syringe under water. A stream of bubbles indicated leakage and the syringe was discarded.

Pre-sparging of the DMAC satisfactorily removed possible interferences contained therein. No attempts were made to clean up the absolute ethanol. If materials co-eluting with VC were present in the chromatograms of the blank ethanol, a new container was opened and checked. In general, interferences were not a problem.

Before attempting the analysis of unknown PVC materials, a complete analysis of a reagent blank should be obtained by using the sparging apparatus described. A known amount of VC should be sparged from the apparatus to check for recovery, leakage, and the GSC retention time of VC under operating conditions.

The partition coefficient was determined by analyzing 17 VC-ethanol solutions ranging in concentration from 1.8 to 12 ppb (polymer basis). These solutions (10.00 ml) were prepared in collection vials, analyzed under the conditions of the analysis, and quantitated by direct comparison to the standard gas (0.48 ppm in N_2) which contained 1.25 ng VC/ml. By using Eq. 1, the partition coefficient was calculated to be 0.11 with a standard deviation of 0.015.

Spiking and recovery studies at levels of 0.7–26 ppb VC in DMAC and in 10% PVC polymer solutions in DMAC were carried out for the entire procedure. The average recovery for 6 analyses of DMAC containing no PVC was 91% with a standard deviation of 3.7. The average recovery for 21 polymer solutions containing 10% PVC was 93% with a standard deviation of 11.7. The PVC for the recovery studies was free of residual VC. (*Note:* PVC containing no VC was obtained by several precipitations from tetrahydrofuran (THF) into methanol. About 50 g PVC from tubing or film was dissolved in a minimum amount of THF. This solution was slowly poured into a vigorously stirred beaker containing methanol. The ratio of the THF solution to methanol was about 1:10. The precipi-

Table 1. Residual VC found in various PVC samples

Sample	Sample description	VC found,[a] ppb
1	rigid calendered food grade sheet	1.2
2	thin plasticized food film	2.5
3	thin plasticized food film	2.1
4	plasticized blood bag	1.2
5	thin plasticized copolymer food film	2.1
6	thin plasticized food film	0.6[b]
7	plasticized food and milk tubing	0.7[b]
8	plasticized beverage tubing	0.7[b]
9	rigid vegetable oil bottle	120
10	rigid French water bottle	913
11	French bottle molding compound	0.6[b]
12	thick plasticized canning sheet	0.4[b]
13	plasticized blood bag	0.3[b]
14	reagent blank	ND[c]

[a] Quantitated by tangent skimming VC peak and measuring peak height—single determination.
[b] Estimated value—below method quantitation limit.
[c] None detected.

tated PVC was filtered onto a large Buchner funnel and air-dried. For processing larger quantities of PVC, a large blender was used for the precipitation. Generally 2–3 precipitations provided clean PVC if the starting PVC contained no more than about 1 ppm VC.

A variety of PVC materials were analyzed using the method described and results are given in Table 1. A typical FID chromatogram is shown in Fig. 3 along with the chromatogram of the reagent blank. Thirteen samples were analyzed and contained VC at levels ranging from 0.3 to 913 ppb. Identity was confirmed by mass spectrometry. It proved invaluable in the development of the method by identifying components eluting near the retention time of vinyl chloride. This ensured in the final procedure that there were no components co-eluting with vinyl chloride that would invalidate the GSC quantitation. Full GSC-MS scans (m/z 16–200) of 12 of the samples chromatographed on Porapak N were obtained to identify the components eluting near vinyl chloride. Figure 4 is the total ion current profile of a typical sample recorded from the elution of carbon dioxide until the elution of ethanol. The reagent blank and the samples contained carbonyl sulfide, propene, water, and acetaldehyde. Chloromethane, isobutane, and butene were identified in most of the samples. Identification of all components was based on spectral interpretation and comparison with reference compounds analyzed under identical conditions. The sample represented by Fig. 4 contained 2.1 ppb VC. At this level, a VC response was not evident in the total ion current profile but was readily apparent in the selected ion current profile of this sample (Fig. 5).

Selected ion recording of m/z 62 and 64 was used to confirm the presence of VC. The characteristic 3:1 ratio of these ions at the retention time of VC produced by the natural abundance of ^{35}Cl and ^{37}Cl in the molecular ion of VC increased the specificity of the method. This ratio was reproducible to within 2% of the theoretical value when the level of residual monomer in the polymer was 1 ppb.

Monitoring the m/z 62 and 64 ratio ensured that only compounds containing one chlorine atom and thus the elemental composition of vinyl chloride as a molecular ion or fragment are possible interferences to the GSC-MS confirmation. Chlorobutenes, epichlorohydrin, and dichloroethane are low molecular weight compounds that produce a fragment at m/z 62 containing one chlorine atom and are potential interferents. Analysis of samples of epichlorohydrin

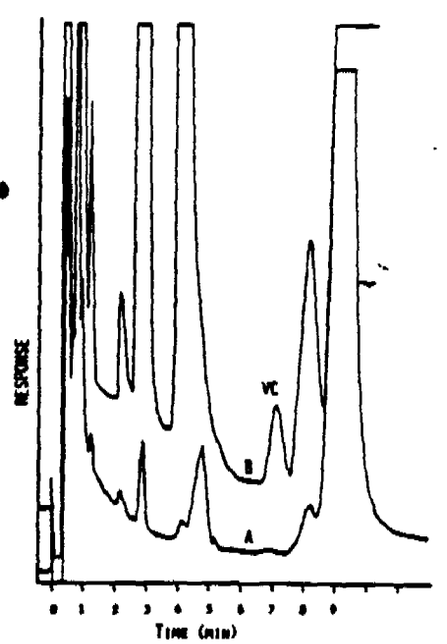

FIG. 3—Typical gas chromatograms: A, 1 ml headspace of reagent blank; B, 1 ml headspace of Sample 3 containing 2.1 ppb VC, polymer basis (0.4 ng VC injected).

FIG. 4—Total ion current profile of Sample 3 containing 2.1 ppb VC, polymer basis (0.8 ng VC injected); retention time of VC indicated by arrow.

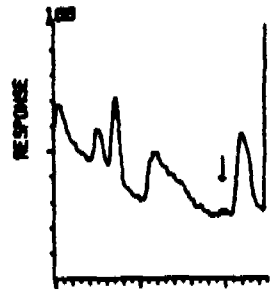

FIG. 5—Selected ion recording profiles of m/z 62 and 64 of Sample 3 containing 2.1 ppb VC, polymer basis (0.8 ng VC injected).

dichloroethane demonstrated that they elute later VC. The heavier chlorobutenes would also elute later than VC (11).

The lack of interfering co-eluting materials made quantitation by GSC-MS unnecessary. Good quantitation by GSC-MS requires strict control of the analytical conditions and the construction of an analytical curve from standards analyzed simultanously. Such an effort was considered redundant in this case.

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POSSIBLE INTERFERENCES WITH VCM

Porous Polymer Type

Butane
Cyclobutane
Isobutane
2,2-Dimethyl propane
Ethyl chloride
Methanol
Methyl chloride
Propane
Ethylene Oxide
Butene-1
Butene-2-cis
Isobutene
Propylene
2-Methyl butane

Polyether Type Columns

Toluene
2,3-Pentane dione
2-Ethyl-3-methyl dioxolane
3-Hexanone
Di-isopentyl ether
1-Butene-2-ol
2,4-Dimethyl-1,3-dioxane
2-Methyl propanol
3-Methyl-2-butanol
2,3-Dimethyl butanol
n-Butyl acetate
2-Methyl pentanol
3-Pentyl acetate
2-Pentyl acetate
3-Methyl-4,6-dioxanone
3-Pentanol
2,5,8-Trimethyl-4,6-dioxanone
2-Methyl decane
2-Pentanol
Isobutyl propionate
3-Methyl-3-pentanol
3-Ethyl nonane