UC 1349-2

VC - FDA

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CHEMICALS AND PLASTICS

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Location Bound Brook, N.J.

Date November 2, 1977
Originating Dept. R & D
Answering letter date
Subject Analysis of VCM in PVC Resins

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Attached is a copy of an analytical procedure for residual VCM in PVC resins sensitive to about 1 ppb which was developed by the FDA. It is quite possible that this may appear as a resin specification in the PVC regulation which is said to be coming out shortly (after two years incubation). We suggest that representative solvent vinyl resins and/or cured coatings be examined for residual VCM by this method so that we do not have any unpleasant surprises later.

Very truly yours,

W B Ackart

WBA/bhm
Att.

W. B. Ackart

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030798

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October 24, 1977

No. 35

To: All members of SPI-VCM/PVC Mailing Lists

Letter Highlights

(1) The Food and Drug Administration has provided a preliminary, unofficial draft of an analytical procedure which it has developed for the determination of residual vinyl chloride (RVCM) in polyvinyl chloride (PVC) plastics. The analytical procedure has a claimed lower detection limit of approximately one part per billion (ppb) in the plastic. Residual vinyl chloride levels ranging from fractional parts per billion (estimated) to the low ppb range were reported for a variety of plasticized PVC packaging materials.

(2) FDA Commissioner Kennedy has stated that the Environmental Protection Agency (EPA) ought to exercise regulatory authority over public water systems and should exercise responsibility for the safety of tap water provided by public water systems. A formal agreement with EPA to this effect would place regulatory jurisdiction of PVC potable water pipe completely under EPA.

Ladies and Gentlemen:

The purpose of this letter is to report on two matters relative to the Food and Drug Administration's (FDA) posture regarding polyvinyl chloride plastics. Activities of other agencies will be the subject of another report.

Analytical Procedure for
Residual Vinyl Chloride

We have previously informed you that FDA had been developing an analytical procedure for the determination of RVCM in polyvinyl chloride food-contact plastics with a claimed lower detection limit near 1 ppb. We have now obtained a preliminary draft version of this procedure and, despite its acknowledged preliminary and unofficial status, we are distributing copies with this letter because of the liklihood of widespread interest in the subject. In addition to a description of the methodology, the report includes the results of analyses conducted on a variety of PVC packaging materials; RVCM levels ranging from fractional ppb (estimated) to low ppb concentrations were reported even for plasticized films. The report further indicates that the presence of vinyl chloride in these samples was confirmed by mass spectroscopy.

Briefly, the new procedure involves the dissolution of the PVC sample in dimethylacetamide (DMAC), sparging the solution so obtained with a stream of helium to volatilize any vinyl chloride obtained in the sample, absorbing the volatilized vinyl chloride in ethanol at dry ice-acetone temperatures, and finally analyzing the ethanol containing the vinyl chloride by means of a headspace procedure utilizing a gas chromatograph with a flame ionization detector.

Although we have not learned enough from our FDA contacts to draw any firm conclusions as to what FDA will do with regard to its final PVC Regulations in light of the development of this methodology, the possibility of strict application of the considerations set forth in the recent acrylonitrile beverage container decision when viewed in the context of the reported detection of vinyl chloride in all the samples listed in the FDA report indicates the need for close attention to this area of regulatory activity.

PVC Potable Water Pipe

Many of you will recall that FDA had proposed (September 19, 1975) that PVC water pipe be the subject of an Interim Food Additive Regulation until questions regarding the possible migration of vinyl chloride to potable water had been resolved. In Comments filed by The Society of

the Plastics Industry, Inc., the argument was presented that responsibility for public water systems including the regulation of potable water pipe had been assigned by Congress to EPA and that, therefore, the proposed Interim Regulation was beyond the scope of FDA's authority.

Shortly thereafter, an Inter-Agency Task Force was set up to resolve the jurisdictional question between FDA and EPA in this and a number of other areas of potential overlap of jurisdiction; however, no final memorandum of understanding has ever been issued despite reported staff agreement that the regulation of PVC potable water pipe should be an EPA responsibility.

More recently, we reported that EPA Administrator Costle had asserted that this regulatory area was one assigned to EPA. Now we are enclosing (with the permission of the editor) reproductions of pages 18-20 from the October 17, 1977 issue of Food Chemical News which indicates that Commissioner Kennedy is likewise in agreement that regulation to assure the safety of public water systems is an EPA responsibility. This report is particularly encouraging at this time since it is generally understood that the Bureau of Foods is making plans to reach some final decision on the long-pending proposed PVC rule making and will begin the preparation of a final Regulation in the "near future."

We will continue to follow these and related developments and will report to you as fully and promptly as we can. In the meantime, if you have any questions, do not hesitate to contact us.

Cordially yours,

Enclosures

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The Determination and Confirmation of ≥ 1 ppb Residual Vinyl Chloride in Polyvinyl Chloride Food Packaging

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ABSTRACT

The determination and confirmation of residual vinyl chloride (VC) polyvinyl chloride (PVC) food packaging materials is described. PVC packaging materials are dissolved in dimethylacetamide (DMAC). VC is sparged from solution with helium gas and collected in sealed vials of ethanol. Detection and quantitation of VC is performed using a headspace sampling technique and standard gas chromatography (GC) with flame-ionization detection. GC peak heights of about 5% full scale deflection (FSD) 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. Confirmation of VC was done by GC/MS 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 the determination and confirmation of 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 GC injection.

Recent work has shown that headspace sampling techniques can increase sensitivities down to the low ppb range (4,5,6,7). 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 GC or GC/MS injection.

Further increase in apparent sensitivity has been achieved by concentration of VC from the polymer solution into another solvent using a sparging technique. This was suggested by Pellar and Lichtenberg (8) and has been used by Japanese scientists (9) 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 GC/MS.

"CTM09

Caution: Since VC is a gas at room temperature and has been designated a carcinogen (1,10,11) 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) Three lb cylinder (99.9% pure), Matheson, or equivalent. (2) VC gas standard (1.00 ppm) containing 2.0 ng VC/ml in nitrogen, EC Scientific Gases, Kearny, NJ 07029 or equivalent.

(b) Ethanol. -- Absolute, Publicker Industries, Inc. or equivalent. Prior to use, test to show absence of co-eluting materials at the retention time of VC under the conditions of analysis.

(c) Dimethylacetamide. -- 3 Kg, Eastman Practical Grade or equivalent. Pre-sparge the entire reagent bottle with helium for 2-3 days before use. Prior to use, test to show absence of co-eluting materials at the retention time of VC under the conditions of analysis.

Apparatus

(a) Screw-capped bottles. -- One oz narrow mouth, 35±0.5 ml, Ace Scientific Supply Co., No. 10-4256, or equivalent. Similar bottles with 2 oz capacity. Caps with Teflon-lined septa (Alltech Associates, No. 9422, or equivalent).

(b) Gas syringe. -- Two, 5, and 10 ml, Precision Sampling, Pressure-Lok, series A-2, or equivalant.

(c) Mini-Vials. -- 5 ml, with caps and Teflon-lined septa. Alltech Associates 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.

(f) Forced draft oven. -- Precision Scientific Company, Thelco Model 18, or equivalent.

(g) Gas chromatograph. -- Hewlett-Packard Model 7620A, or equivalent, equipped with temperature programmer and flame ionization detectors. Operating conditions: Temperature (°C) -- injection port 200, detector 250, column 110. After VC elution (~ 7 min) program column to 180 at fastest rate and hold for 20 min; gas flows (ml/min) -- carrier (He), 35 (0.7 rotameter), makeup gas (He), 30, oxygen, 345, (3.35 rotameter), hydrogen, 35, (0.75 rotameter), electrometer setting, 2×10^{-12} afs.

(h) Chromatographic column. -- 6' x 1/8" O.D. coiled stainless steel (SS), filled with 80/100 mesh Porapak N; retention time of VC approximately 7 min.

Ave Sample 5593

(i) Gas chromatograph-mass spectrometer. -- Finnigan 3200F electron impact quadrupole mass spectrometer equipped with Finnigan 9500 gas chromatograph and Finnigan 6100 data system. Full mass scans and selected ion recording of m/z 62 and 64 were obtained under computer control. Operating conditions: Temperature -- injector 170, column 155, separator 215, transfer line 210; after VC eluted, column vented and programmed to 180°C to clear solvent from column; carrier gas helium 25 ml/min; sample size 2.00 ml headspace at 90°C column listed under (h).

(j) Pint bottles. -- Flint glass 16 oz narrow mouth, screw cap bottles, Fisher Scientific Company Catalog Number 2-0030D or equivalent, with foil lined caps.

Preparation of Standards

Accurately weigh a 2 oz narrow mouth bottle, Mini-Vial cap, and septum. Add 50 ml absolute ethanol to weighed bottle. Cap and reweigh. In a 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 (~ 10,000-40,000 ppm). Dilute this stock solution by withdrawing aliquots through the septum with a syringe and injecting into a weighed, sealed container (1 oz bottle) of ethanol. Reweigh and calculate VC concentration either in weight/weight or in weight/volume units (~ 50 ppm). This solution is similarly diluted to yield a solution containing 1-5 ppm. Make final dilutions into 10 ml of ethanol in a collection vial. Prepare working standards in the 1 - 50 ppb range. If refrigerated, this working standard is stable for a period of up to one week. Multiple septum punctures shorten the working life of the standards.

Determine the actual VC concentration in the headspace of the above working standard under conditions of analysis by direct comparison to the standard gas (1 ppm in N_2) containing 8.6 ng VC/ml. Calculate an average partition coefficient using the following equation:

Equation #1:

$$\text{Partition Coefficient} = \frac{Cv}{CL} = \frac{.29/14\ ml}{.71/10\ ml} = 0.29$$

where Cv - Concentration in vapor (headspace)
CL = Concentration in liquid
.29 = 29% determined to be in headspace
14 ml = Volume of headspace (13 ml) plus volume of syringe (1 ml)
.71 = 71% determined by difference to remain in liquid
10 ml = Volume of ethanol standard

Preparation of Solvents and Samples

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

Drill two holes into the cap of the 3 kg reagent bottle of dimethyl acetamide (DMAC) and run a 1/16" Teflon® or stainless steel tube to the bottom of the bottle through one of the holes. Sparge the DMAC using a slow flow of helium (5-15 ml/min) for several days at room temperature prior to use. After DMAC has been obtained which is free of materials co-eluting at retention time for VC (determined by running a reagent blank), add 200 ml to a 1 pint bottle along with a 2" Teflon® coated magnetic stirring bar.

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

Analysis

After the PVC sample is dissolved in the pre-sparged DMAC, add 10.00 ml of absolute ethanol to each of two collection vials. Interconnect the vials with short lengths of 1/16" SS tubing as shown in Fig. 2, and cool in a thick Dry Ice® - ethanol slurry for 30 min before use. Fit the bottle containing the PVC solution with a tight cap equipped with a 1/16" SS sparge tube extending to the bottom of the bottle. The apparatus is shown in Fig. 1.

Immerse the pint bottle containing the PVC solution in a steam bath and add helium sparge gas via a needle valve through the 1/16 SS sparge tube at a rate of ~35 ml/min. Quickly leak check the apparatus by applying Snoop® to all joints and briefly plug the final outlet tube in order to build up pressure. After leak checking, allow the sparge gas to freely pass through the PVC solution, through the bulkhead fitting, into the effluent tube and into the collection vials immersed in the Dry Ice® - ethanol slurry. Sparging apparatus is shown in Fig. 2.

After sparging for 2 to 2 1/2 hours, disconnect the collection vials while still cold, decap using the decapper and re-seal using a fresh unpunctured septum. Analyze vials immediately or store under refrigeration for no longer than 2 days. For analysis, heat vials to 90°C in either a water bath or a forced draft oven and allow to equilibrate at this temperature for a minimum of 1 hour. Heat a gas-tight syringe having an integral valve to 90°C in the forced-draft oven. Wear gloves to use the preheated gas-tight syringe and withdraw 1.00 ml of headspace. Close the syringe valve and return syringe containing the sample to the oven for 5 min to vaporize any condensed solvent. Remove the hot syringe and immediately inject the sample into the gas chromatograph. Make standard VC gas injections (1.00 ml, 2.6 ng) both before and after each headspace sample injection. Quantitate the unknown using the following equation (#2) where

$$\text{ppb VC in PVC} = \frac{\text{Total ng VC in sample}}{20\ \text{g}} - \frac{(\text{ng/ml in HS})14\ \text{ml} + (\text{ng/ml in Liq})10\ \text{ml}}{20\ \text{g}}$$

combined with equation #1 $\frac{Cv}{CL} = 0.29$

where $CL = \frac{Cv}{0.29}$

Equation # 2 giving

$$\text{ppb VC in PVC} = \frac{(Cv)14\ \text{ml} + \frac{Cv}{(0.29)}10\ \text{ml}}{20\ \text{g}}$$

$$= \frac{Cv\left[14\ \text{ml} + \frac{10\ \text{ml}}{0.29}\right]}{20\ \text{g}}$$

$$= 2.42\ Cv$$

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

Calculate the response areas of m/z 62 and 64 using Finnigan Revision 4 software. Calculate the relative abundance of m/z 62 and 64 from the response areas at the retention time of vinyl chloride and compare to theoretical 3:1 ratio.

Results and Discussion

Previous 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 accurate determination of low ppb levels of VC in solid food packaging materials could not be achieved 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 insure 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 transfering 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 of PVC in 200 ml of DMAC without entrainment of the DMAC into the gas stream. Some breakage of bottles occurred 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 Figure 2 shows the assembled sparging apparatus.

GC analysis of the headspaces of several PVC samples following sparging demonstrated that VC could not be adequately resolved from other components using Carbowax 20 M or Silar 10 C 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. GC/MS analysis showed that this component was isobutane. When using the porous polymer columns, from 45 min to 1 hour was required before the baseline was considered adequate for the next analysis.

We were unable to obtain syringes capable of sustained use at 90°C. Syringe leakage problems were occasionally encountered due to sustained useage 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 to clean up the absolute ethanol were made. 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 using the sparging apparatus described. A known amount of VC should then be sparged from the apparatus to check for recovery, leakage, and the GC retention time of VC under operating conditions.

The determination of the partition coefficient (0.29) was made by analyzing VC-ethanol solutions ranging in concentration from 2.5 to 40 ppb. These solutions (10.00 ml) were prepared in collection vials and analyzed under the conditions of the analysis. An average of 37 determinations showed that 29% of the VC partitioned into the headspace. This was demonstrated by direct comparison to the standard gas (1 ppm in N_2) which contained 2.6 ng VC/ml. Equation (1) was used for calculation of the partition coefficient. Standard deviation of all determinations was 0.075.

Spiking and recovery studies at levels of 7 to 26 ppb VC in DMAC and 1% PVC polymer solutions in DMAC were carried out for the entire procedure. The average recovery for 6 analyses of DMAC containing no PVC was 93%. The average recovery for 21 polymer solutions was 93%. 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 of 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 precipitated PVC was filtered onto a large Buchner funnel and air dried. For processing larger quantities of PVC, a large size blender was used for the precipitation. Generally two to three precipitations provided 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 are tabulated in Table 1. A typical FID chromatogram is shown in Fig. 2 along with the chromatogram of the reagent blank. Thirteen samples were analyzed and found to contain VC at levels ranging from 0.3 to 912 ppb in these PVC formulations.

Mass spectrometry was the confirmation step in the method. It proved invaluable in the development of the method by identifying components eluting near the retention time of vinyl chloride. This insured in the final procedure that there were no components co-eluting with the vinyl chloride that would have invalidated the VC quantitation. Full GC/MS scans (m/z 16-200) of 12 of the samples chromatographed on Peak 3 were obtained to identify the components eluting near vinyl chloride. Figure 3 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 were found to contain carbonyl sulfide, propene, water and acetaldehyde. Chloroethane isobutane and butene were identified in most of the samples. The retention times of the components identified in each of the samples relative to vinyl chloride are listed in Table 2. Identification of all components was based on spectral interpretation and comparison with reference compounds analyzed under the identical GC/MS conditions. The sample represented by Fig 4 contained 2 ppb VC. At this level, a VC response was not evident in the total ion current profile but was readily apparent in Fig 4 which is the selected ion current profile of this sample.

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 this m/z 62 and 64 ratio insured 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 mass spectrometry 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 interferences to the mass spectrometry confirmation. Analysis of standards of epichlorohydrin and dichloroethane demonstrated that they elute after VC. The chlorobutenes being heavier than butene would also elute later than VC (12).

The lack of co-eluting materials that would have interferred with the FID results made it unnecessary to quantitate the samples by GC/MS. Good quantitation by GC/MS requires strict control of the analytical conditions and the construction of an analytical curve from standards analyzed simultaneously with the samples. Such an effort was considered redundant in this case.

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Table 1. PVC Sample Summary

Sample No.	Sample Description	VC found[a] (ppb)
1	Rigid calendared 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

[b] Estimated value - below method's quantitation limit

[c] None detected

Table 2. - Retention time of identified components relative to VC

Sample No.[a]	Temp.	Carbon-dioxide	Carbonyl-sulfide	Propene	Propane	Chloro-methane	Water	Vinyl Chloride	Isobutane	Butene	Acetalde-hyde	Butane	cis-2-Butene	1,3-Ruta-diene	Ethanol
1	155		.42	None		.69	.71	1.00	1.07	1.20	1.22				1.76
2	155		.44	.50		.71	.72	1.00			1.19				
3	155		.47	.55		.73	.74	1.00		1.17	1.17				
4-tube 1	155		.46	.55			.67	1.00	1.05	1.21	1.21				
4-tube 2	155		.42				.72				1.21				
5	155		.44	.51		.70	.70	1.00		1.20	1.20				1.86
6	155		.44	.52		.71	.72	1.00		1.20	1.26				
6	120		.32	.42		.59	.65	1.00		1.20	1.22				
7	155		.44	.52		.70	.72	1.00		1.20	1.22				
8	155		.44	.53		.70	.72	1.00	1.06	1.16	1.17				
11	155		.43			.72	.70	1.00		1.20	1.20				
12	155		.50			.69	.69	1.00		1.20	1.21				
13	155		.46	.52		.70	.70	1.00	1.07	1.20	1.20				
13	120		.31	.40		.64		1.00		1.20	1.20				
2 ml Blank (spiked)	155		.43	.49		.70	.70	1.00			1.23				
2 ml Blank (tube 2	155		.43				.70				1.17				
2 ml Blank (spiked)	110	.10	.31	.42		.66	.66	1.00			1.28				
Dichloroethane	155	- elutes after ethanol													
1,3-Butadiene	155				.51					1.18				1.27	
10 µl Butane lighter	120				.43				1.11			1.04			
cis-2-Butene	155												1.38		
Acetaldehyde	155										1.21				

Figure Captions:

Fig. 1 - Can Details

Fig. 2 - Effluent Tube Details

Fig. 3 - Gas Chromatograms of 1.0 ml Headspace

Fig. 4 - Total ion current profile of sample #3 containing 2.1 ppb VC

Fig. 5 - Selected ion recording profiles at m/z 62 and 64 of sample #3 containing 2.1 ppb VC

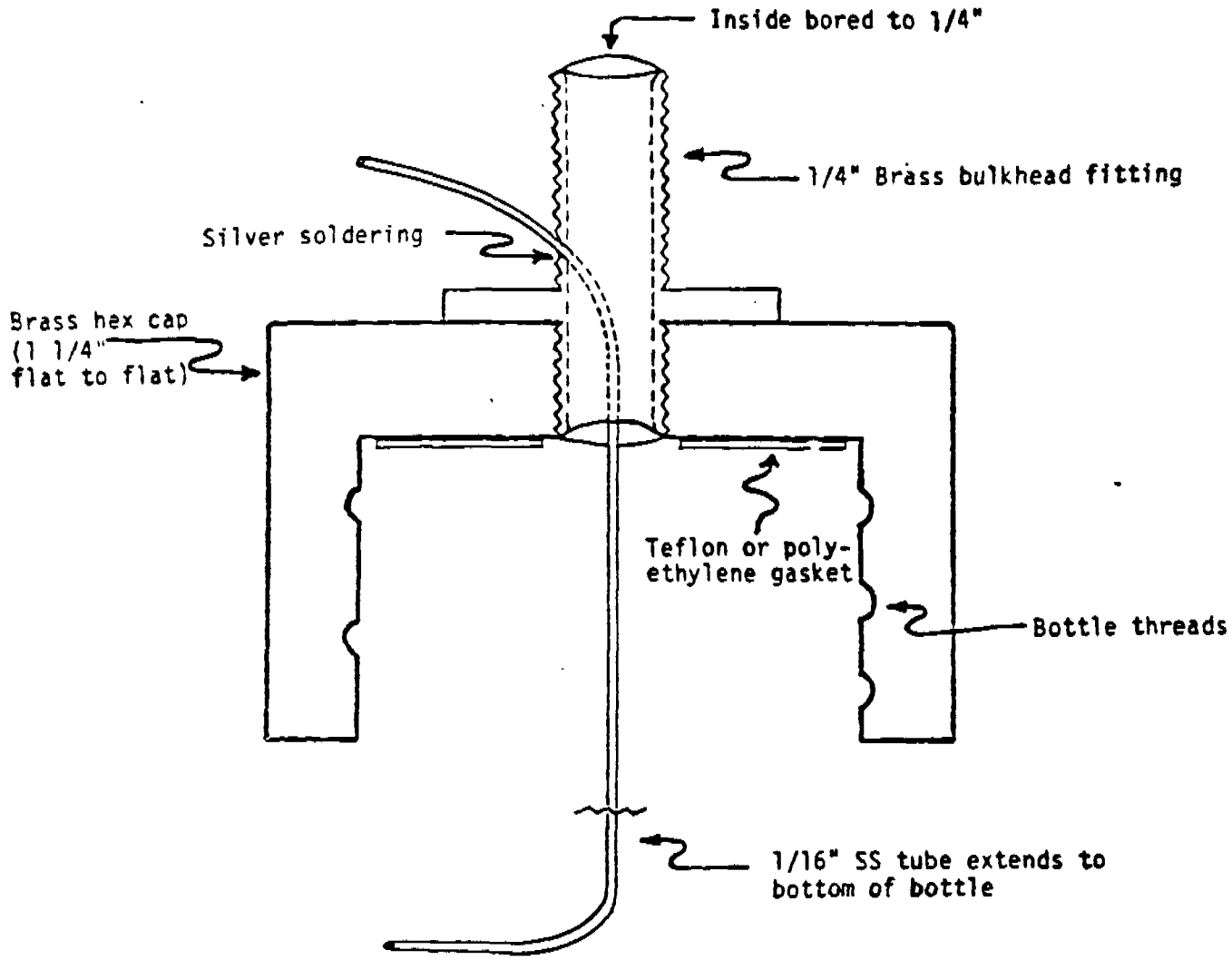


Fig. #1 - CAP DETAILS

A. For temporary use and preliminary tests a standard metal cap may be used by removing the liner, drilling a hole through the top, removing the surface paint with acetone, and soft soldering in a bulk head fitting with the sparge tube attached. The sparge tube is attached to the bulkhead fitting by drilling a hole slightly larger than 1/16 inch downward at an angle into the bore of the filling (See diagram). The flats of the nut portion and the upper major part of the threads should be avoided. After insertion of the sparge tube to the proper length (enough to reach the bottom of the bottle), the tube is silver soldered in place. A thick teflon or polyethylene gasket is then cut to fit the inside of the cap.

The threads on standard metal caps are not sturdy enough for continual use and often strip off after a short time. Plastic caps were found to be unsatisfactory for even temporary usage.

B. A more permanent cap for continual use has been designed. Machined from solid brass, the sparging cap consists of a hex-shaped cap (1 1/4 inch flats) threaded to the same configuration as the bottle and drilled and tapped in the top to accommodate a Swagelok 1/4 inch bulkhead fitting.

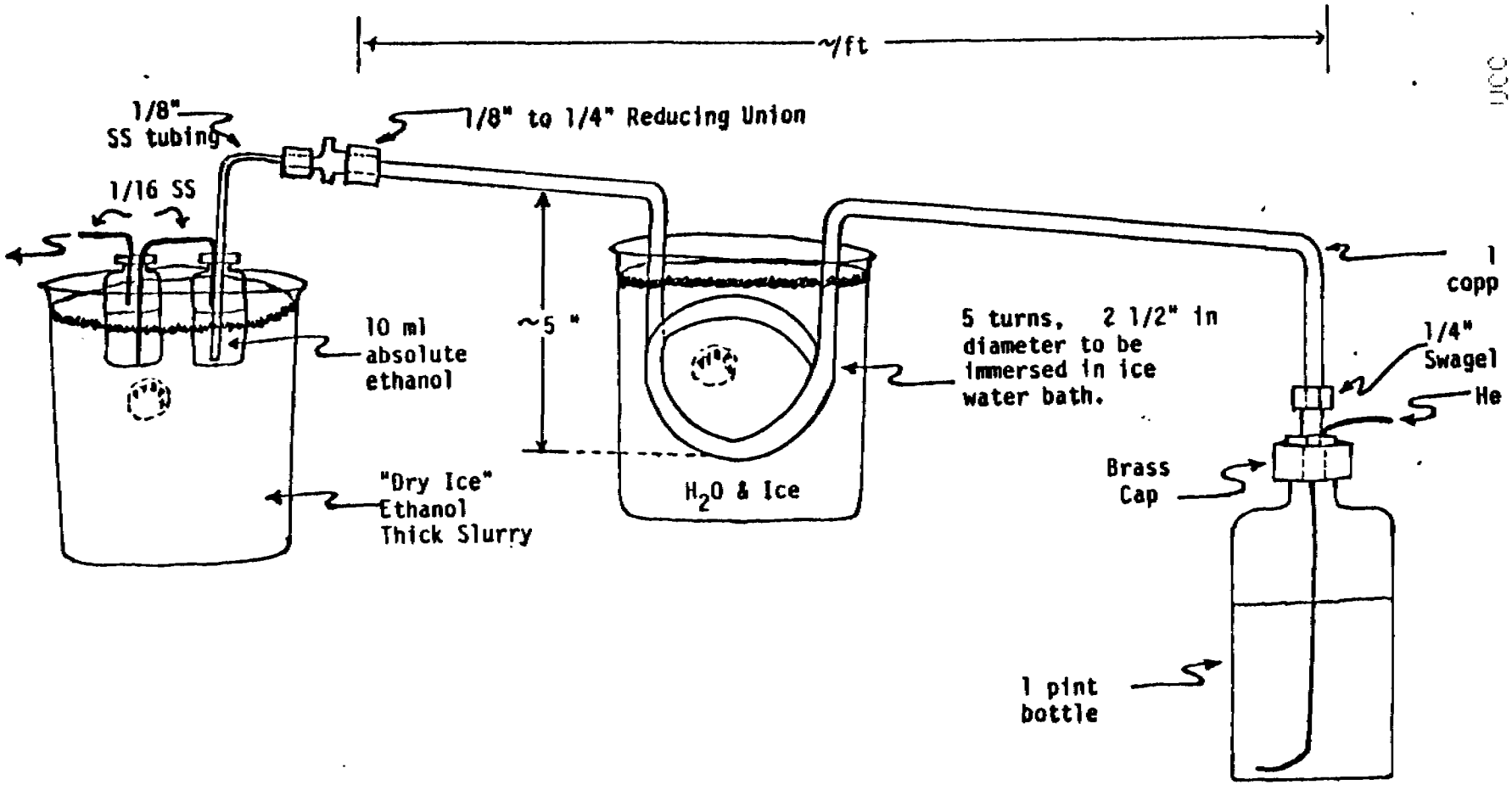
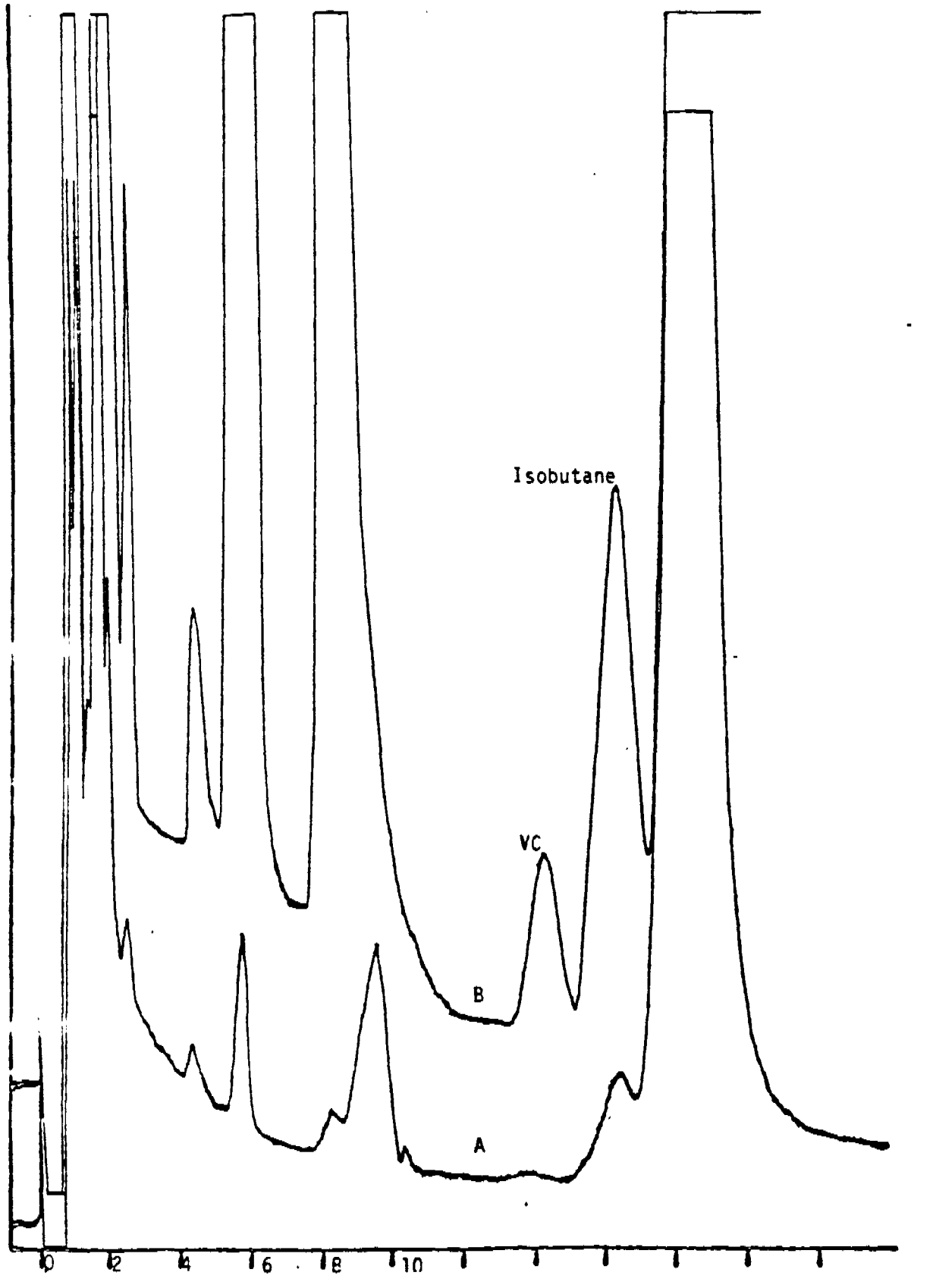


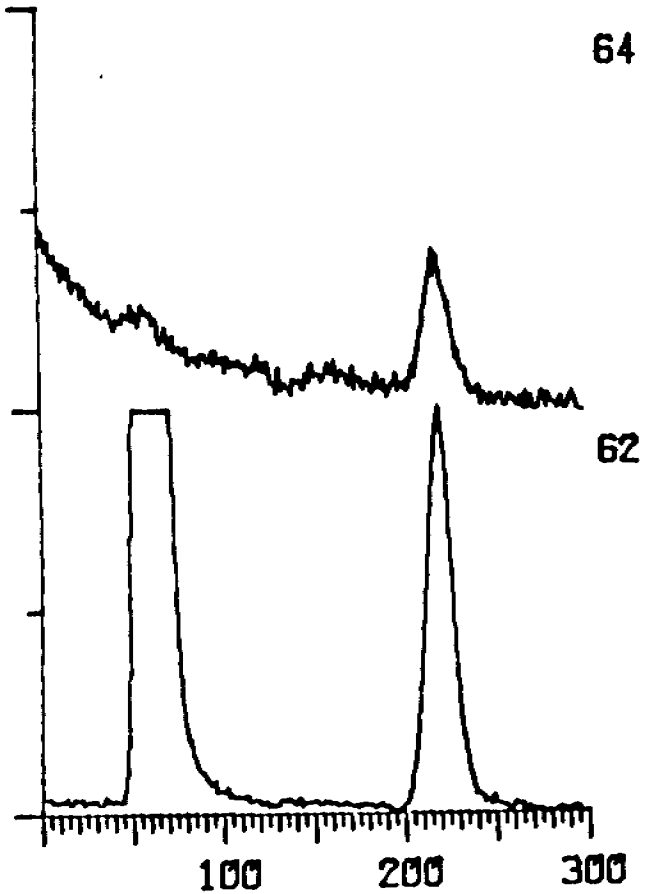
Fig. #2 - Effluent Tube Details:

Requires: (a) 7 inches of 1/8 inch stainless steel tubing
(b) 1/8 inch to 1/4 inch reducing union
(c) 5 feet of 1/4 inch OD copper tubing

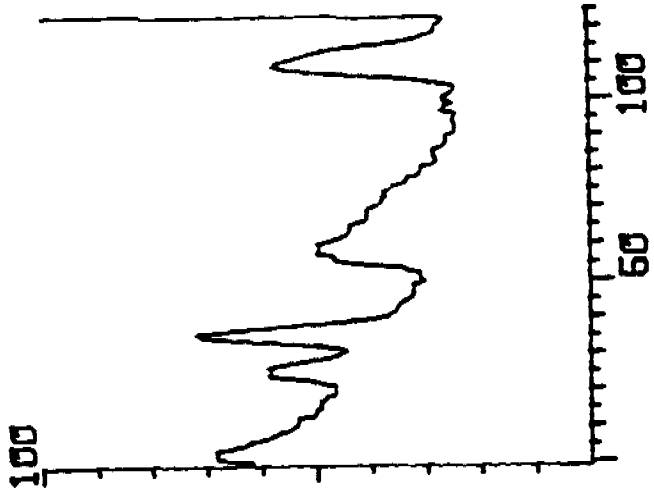
A second collection vial is required since as much as 10% of the VC may pass through the first vial. At levels above 10 ppb a third vial is recommended.



UCC
030821



UCC 030822



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FDA has been compiling data on sodium intake through its marketbasket studies, Vanderveen said, forecasting an assessment of salt intake. He predicted it will show that most people are consuming an adequate amount of salt, but that some consumers with unusual eating patterns may consume more than 20 grams a day. If so, Vanderveen continued, there may be a "great debate" on whether FDA should take any action.

Noting that FDA's enrichment-restoration-fortification regulations are on the "verge" of being finalized, Vanderveen said fortification "will become more and more important." The new regulations, which he said spell out fortification practices, will take a balanced nutrient approach. Such an across-the-board policy is needed rather than consideration of fortification on an item-by-item basis, he said.

The FDA-er predicted that new foods "will receive the brunt" of FDA's regulatory effort in the future, because it is an area in which "we can make changes."

Amendment of Law Placed Emphasis on Nutrient Toxicity, FDA-er Says

A "big concern" of FDA will be nutrient toxicities, Vanderveen said, explaining that the amendment to FDA's law sponsored by Sen. Proxmire (D-Wis.), which limited FDA's authority to regulate vitamins and minerals, puts pressure on the agency to regulate on the basis of toxicity rather than need.

The protein quality standard is a "matter of concern," Vanderveen said, noting that FDA recognizes the weaknesses in use of the Protein Efficiency Ratio but has found no good alternative. He said the agency is also interested in quality assessment of other nutrients.

The FDA-er indicated that his agency is seeking new terms to use in place of the term "fiber," using this as an example of definition problems in nutrition.

FDA's efforts in the field of nutrition were summarized by Vanderveen as dealing with: nutritional needs, especially regarding disease; application of dietary management of diseases; nutritional interactions between nutrients and other substances, such as food additives; nutritional toxicities; bioavailability; and nutritional information and how to communicate it.

University of Florida's Dr. Howard Appledorf, who presented nutrient data on various "fast foods," attacked Assistant Agriculture Secretary Carol Tucker Foreman for stating that "fast foods are junk foods." He predicted a USDA "blitz" against fast foods, but predicted that fast foods will prevail. He stressed the possibilities of fast food items being used in school lunch programs to assure that students eat the food and receive the nutrition, and to reduce waste.

FDA PREFERS REGULATING ONLY WATER USED AS A FOOD INGREDIENT

The Food and Drug Administration would prefer regulating water only as it is used as an ingredient in commercial food products, Food and Drugs Commissioner Dr. Donald Kennedy told the Environmental Protection Agency recently (See FOOD CHEMICAL NEWS, Sept. 5, Page 33).

In an Oct. 4 letter to EPA Administrator Douglas M. Costle, the Commissioner said discussions between FDA and EPA seem to lead to a choice between two options:

-- "FDA's role in regulating water could be limited to water used as an ingredient in commercial food products (e.g. in bread, canned punch, bottled water). EPA would assume plenary responsibility for safety of tap water provided by public water systems. This option would generally result in our agencies having mutually exclusive areas of responsibility with respect to water.

-- FDA could assume an active role in regulating water provided by public water systems in addition to water used in commercial food products. This option might require FDA to promulgate food additive regulations for water treatment chemicals and would probably result in FDA establishment of poisonous or deleterious substance tolerance regulations for unavoidable contaminants in drinking water provided by public water systems. This option would probably result in substantial overlap of our agencies' programs for regulation of water."

On balance, the first option appears to FDA to be the preferable choice, Kennedy said, explaining that FDA believes Congress intended EPA to have full authority over public water systems when it passed the Safe Drinking Water Act.

In recognition of the Congressional intent, he continued, FDA would defer to EPA to regulate drinking water, and would not regard water meeting EPA, State and local standards as "actionable" when used as a food ingredient, pointing out that FDA has deferred in the past to the drinking water program of the Public Health Service. The concerns with this approach are, the Commissioner observed:

"(1) It is uncertain whether EPA has sufficient statutory authority to bring about adequate control; and (2) there may be a lack of resources to undertake all the evaluations that ultimately would be required."

Kennedy said he believed the new Toxic Substances Control Act (TSCA) would probably give EPA the necessary authority to "pre-clear" the application of new chemicals to production of water, but he added there would still be doubt as to the extent of EPA's authority to require submissions for evaluating substances that have been in use for some time.

The Commissioner said he recognized that control of a new substance under TSCA may present some difficulties because of the need first to issue a regulation requiring that the substance be tested. To regulate substances already in use, he continued, "EPA would need to conduct an inventory and itself undertake the burden of testing, which leads to resource questions."

He pointed out, however, that a need for additional resources to carry out an effective program of safety assurance for water treatment substances is a common problem under either option and probably should not be the primary factor in reaching a decision.

He acknowledged that the second option offers greater ability under the Food, Drug and Cosmetic Act to exercise control over substances used in public water supplies, but he noted that FDA could only use the §406 approach for unavoidable contaminants.

"This approach would also carry with it a commitment to undertake a comprehensive review program in order to establish permanent tolerances for water treatment substances," he wrote.

Kennedy said the second option appears to prevent "a substantial prospect of wasteful duplication of effort by our agencies and of confusion arising out of overlapping regulation by two government agencies."

If the first option is adopted, the Commissioner said steps would be needed to coordinate responsibilities in an effort to avoid regulatory gaps, pointing out, for example, food and food additives are exempt from TSCA.

In cases of uncertainty as to which agency has responsibility, Kennedy advocated joint action, saying, however, it would be preferable to work under the Safe Drinking Water Act.

He added that a review of authority under this act would be helpful in determining a strategy that would minimize the workload of this option and increase the likelihood of its success.

Meanwhile, Resources for the Future announced that it had begun planning a Safe Drinking Water Conference for Feb. 15-17 in Washington as proposed by Foremost Foods (See FOOD CHEMICAL NEWS, Aug. 8, Page 53; and Aug. 22, Page 2).

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FDA TO PUBLISH TETRACYCLINE PROPOSAL OCT. 18

The Food and Drug Administration will publish its proposal to ban some feed uses of tetracyclines in the Oct. 18 Federal Register, the agency informed Congressional committees in an Oct. 3 letter.

The House Appropriations Committee had directed FDA to inform interested groups on Capitol Hill at least 15 days in advance of proposals to ban uses of low-level antibiotics in feed (See FOOD CHEMICAL NEWS, Sept. 19, Page 7).

In his letters to Appropriations subcommittee chairman Whitten (D-Miss.) and other members of Congress, FDA Commissioner Donald Kennedy also revealed that a proposal on "distribution controls" for antibiotics "will be published shortly after the tetracycline proposal."

The Commissioner previewed the tetracycline proposal, as follows:

> "... The subtherapeutic uses of tetracyclines intended to be eliminated are those uses for which there are effective alternative drugs. Nontherapeutic uses of tetracyclines for which there are no alternative drugs available will be retained for use by the livestock industry.
>
> "Examples of effective alternativ drugs available for the uses of penicillin and the tetracyclin s include bacitracin (zinc and methyl ne disalicylat), erythromycin,