

BUSINESS CONFIDENTIAL

TRIP REPORT

MEMORANDUM

SOLVENT VINYL RESINS FOR COATINGS
MEETING WITH FDA ON RVCM CONTENT
DECEMBER 1, 1978

AUTHORS: G. S. Peacock

DATE: December 12, 1978

PROJECT NO.: 326N02

SUPERVISOR: C. N. Merriam

FILE NO.: 6107

A meeting was held with FDA personnel in Washington, December 1, 1978, to review our progress in measurement of residual vinyl chloride monomer (RVCM) in UCC polymers.

We presented the results of our testing via the FDA method, presented our evidence for use of the Hall detector to resolve interferences inherent in the FDA method and presented the very recent results we obtained using the Hall detector. Our results obtained with the FDA method are summarized in Tables I and II attached. Our results using the FDA method and the Hall detector are shown in the letter from T. B. Gibb to G. S. Peacock, 11/28/78, attached. This data was presented to FDA at the meeting.

FDA responded by submitting a handout, attached, comparing FDA GC data to UCC data. FDA did not measure VAGH or solvent solutions and has not seen the interferences we have been plagued with. In addition, the FDA Porapak N column does a much better job of resolving VCM. They stated that some lots perform better than others and provided us with lot numbers for our use.

The FDA results with VYHH are quite interesting (Figure 5A and 5B). They obtained an RVCM content of 0.4 ppb. This sample (VYHH B-7418) was taken directly off the bag line and shipped to FDA in March, 1978 (letter R. M. Arnold to FDA, 3/16/78). FDA tested the sample June 20, 1978.

FDA is aiming to have sufficient data to draft a regulation by mid-1979. They appear to be thinking in terms of a catch-all regulation that would impose one RVCM limit on the polymer or food contact item rather than having different limits for each end use.

Research and Development Department
Chemicals and Plastics
Union Carbide Corporation
Bound Brook, New Jersey

UCC
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We agreed to submit a suggested protocol which FDA would critique that would provide the data needed to draft a regulation. FDA requested and received permission from us to discuss the use of the Hall detector with other interested parties. FDA will summarize the results of this meeting and publish the results in the Public Record. We requested permission to prepare our summary of the meeting to be submitted to our customers to keep them posted of our progress. FDA agreed to this but wants to review and approve our statement before we issue it.

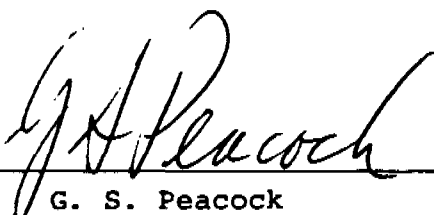
Present fro FDA were:

Dr. Arthur Lipman - Regulatory Officer
Dr. Felci - "
Dr. John Dennison - Chemist

and several others whose names and titles we did not record.

Present for UCC were:

R. W. Lasher
E. J. Mendlow
J. G. Kucsma
F. G. Willeboordse
W. B. Ackart
T. B. Gibb
R. N. Wheeler
G. S. Peacock


G. S. Peacock

GSP:ld
Attachments

TABLE I

APPARENT MONOMER CONTENT⁽¹⁾ IN VINYL CHLORIDE COPOLYMERS

<u>Resin Sample</u>	<u>Apparent Monomer (parts-per-billion)</u>	
	<u>Bound Brook</u>	<u>Texas City⁽²⁾</u>
VYHH 11405-72-1 As Received	260	310
Exposed 5 Days	12	
11405-72-3	110, 130, 180	200
11405-89-1	370, 320	--
11405-89-2	90	--
VMCH 11405-72-4	50	120
VAGH 11405-88-3	160	--
FDA Referee Sample	2, 1	(3)
(Plasticized VC Homopolymer)		

(1) Area of peak eluting at or near the retention time for known vinyl chloride calculated as if it were that monomer.

(2) Determined by Union Carbide Quality Control Method.

(3) FDA reported a value of 6 parts-per-billion for this sample.

TABLE II

CHANGE OF APPARENT MONOMER CONTENT(1)
DURING EVAPORATION OF METHYL ETHYL KETONE SOLVENT

<u>Solvent Fraction Remaining</u>	<u>Apparent Monomer (Parts-Per-Billion)</u>
0 (Original VYHH)	140
1.0 (Initial Solution)(2)	420
0.9	220
0.8	80
0.5	190
0.25	130
0.025 (Baked)	100

(1) Area of peak eluting at or near the retention time of known vinyl chloride calculated as if it were that monomer.

(2) Initial solution 20% resin by weight.



INTERNAL CORRESPONDENCE

CHEMICALS AND PLASTICS

RIVER ROAD, BOUND BROOK, NEW JERSEY 08805

To (Name) Mr. G. S. Peacock
 Division Chemicals & Plastics
 Location Bound Brook, N. J.

Date November 29, 1978

Originating Dept. R & D

Answering letter date

Copy to Dr. W. B. Ackart - BB/98
 Dr. L. E. Brydia - BB/200
 Ms. O. M. Garty - BB/200
 Dr. R. A. Gregory - BB/203
 Dr. D. L. Kemp - BB/200
 Dr. G. T. Kwiatkowski - BB/98
 Mr. R. W. Lasher - NY/33
 Dr. C. N. Merriam - BB/98
 Dr. F. G. Willeboordse - BB/98

Subject Determination of VCM
 in Solvent Vinyls with
 a Hall Detector

In recent weeks we have evaluated an electrolytic conductivity (Hall) detector for determination of residual vinyl chloride monomer in vinyl chloride copolymers. The detector, purchased from Tracor, Inc., was installed and checked out on the HP 5754 gas chromatograph on which we have done all of the FDA related VCM work in recent months. The detector performs according to specifications and gives a typical response of 2.6 mv/mg for VCM as compared to 0.17 mv/mg response with the flame ionization detector (FID). This indicates a 15 fold increase in sensitivity, but we do not anticipate that much of an improvement because of noise and other interferences (see Figure 1). The linearity of the detector was determined over the range of greatest interest, 0.5 to 50 ppb based on a theoretical resin weight, and was found to be at least as good as the FID used under FDA conditions (see Figure 2). The expected selectivity was also realized. When a copolymer sample (VAGH) is analyzed under the FDA conditions using the Hall detector, the isobutylene peak which interferes with FID detection is not observed and the VCM peak can be measured (see Figure 3).

Using this Hall detector modification of the original FDA headspace/GC method we have analyzed samples of three vinyl chloride copolymers and the FDA referee sample. The copolymer samples had been stored as dry powders in glass jars with screw caps during the 6 to 9 months since their arrival at Bound Brook. Therefore, the low levels found for these samples do not necessarily reflect their original monomer contents (see Table I).

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Mr. G. S. Peacock

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November 29, 1978

Determination of VCM
in Solvent Vinyls with
a Hall Detector

With the resolving power of the system cited above, we have found some evidence that the porous polymer chromatographic column is a limiting factor. It appears that the column will adsorb small amounts of VCM which may desorb again during later injections (see Figure 4). We plan to determine the practicality of using the column system specified in the Carbide QC method (Tergitol E-35 on Chromosorb W/switching valve/SP-1000 on Carbopack C) instead of the Porapak N.



T. B. Gibb



P. H. Wolfe

fp
Att.

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