I-184 (50M)

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO H.B. Carr AT Burlington DATE December 8, 1972

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File

SUBJECT Analyses of Vinyl Chloride Monomer (VCM) in the Stripper Headspace and Recovery Slurry

Gas chromatographic analyses were performed on a stripper headspace sample and a slurry sample to determine the VCM level after recovery. The following is a summary of the analyses work and results:

I. Sampling

Two medium pressure sample containers were used to obtain the samples:

A. Headspace Sample

An evacuated sample container was attached to a sample line normally used for Ionol addition. At the time of sampling the stripper had been pressurized to 20 psig with nitrogen just prior to transfer of the slurry to the blend tank.

B. Slurry Sample

A sample container was attached to the slurry transfer line on a sample tap just before leading to the chip basket. A slow sample flow was noted during the filling of the sample container. This may be attributable to the fact that the sample tap is in a right angle to the slurry transfer line.

II. Analyses

Two different analyses conditions were utilized for the samples:

A. Headspace Sample

1. Sample Preparation

A septum covered Swaglok fitting was attached to one valve outlet of the sample container. The sample was withdrawn from the container with a Hamilton gas syringe. Two ml of this sample was then transferred onto the gas chromatographic column.

2. Instrument Conditions and Calibration

The gas phase analyses were accomplished with a 12' x $\frac{1}{4}$" SS column packed with 20% tributyl phosphate on Chromosorb P(AW), an inject port temperature of 130°C, oven temperature of 60°C and a flame ionization detector (FID) temperature of 260°C.

2. Instrument Conditions and Calibration (continued)

Calibration was made by injection of 0.5 ml of VCM in the same manner as mentioned for the sample in 11.A.1.. The detector response of this inject was used as a 25% by mol VCM standard.

3. Results

The VCM level was calculated to be 21.9% in the stripper headspace (see Table I, attached).

B. Slurry Sample

1. Sample Preparation

a. Slurry Liquid

As before a septum-covered Swaglok fitting was attached to one valve outlet of the sample container. After sampling with a syringe, five µl of the liquid was injected onto the column for analyses.

b. Slurry Sample

After analysis of the slurry liquid, a slurry sample was transferred directly from the sample container into an Erlenmeyer flask containing 87.9 gms tetrahydrofuran (THF). The resin contained in the slurry was allowed to dissolve for about fifteen minutes. A 5 µl aliquot of the solution was subsequently transferred onto the column for analysis.

2. Instrument Conditions and Calibration

For the analysis of the liquid, a 12' x ¼" SS column packed with 10% Diethylene Glycol Succinate (DEGS) on Chromosorb W(AW). The instrument conditions were set as follows: inject port 180°C, oven 70°C and the FID 260°C.

The calibration standard was prepared by saturating 100 ml of water with VCM. This was accomplished by bubbling about 1000 ml of VCM through the 100 ml of water. The VCM content in the saturated water was calculated to be 0.73% by weight.

3. Results

The dissolved VCM level in the slurry water was found to be 0.15% by weight. The total VCM level in the resin slurry THF sample was 1.6% by weight based on the slurry sample weight in the THF.

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Attachment.

FWK/jt

TABLE I

VINYL CHLORIDE MONOMER

IN STRIPPER HEADSPACE AND RECOVERY SLURRY

SAMPLE	VCM
Stripper Headspace @20 psig	21.9% by mol
B-5212-3120M30-225/16R	
Slurry Sample	
B-5212-3120-225/16R	
a. Liquid	0.15% by weight
b. Slurry (Total)	1.56% by weight