

MATERIAL

Vinyl Chloride Monomer (VCM)

ANALYSIS

Heavy-end Impurities:

MVA

Ethyl Chloride

2-CPY

Vinylidene Chloride (1, 1-DCY)

t-1,2-DCY

CB

SAMPLE

Liquid Sample in VCM Bomb

APPARATUS

P. E. Model 154-C Vapor Fractometer equipped with gas inlet valve, or equivalent, valved for multi-column operation (See attached drawing.)

Hydrogen Flame Ionization Detector - capable of detecting 2×10^{-10} moles of chlorinated compounds.

Meeco Vaporizing Valve, Type 1S, or equivalent, for flashing liquid VCM into gas sample loop of chromatograph.

Flow-meter for determining rate of purge on sample loop.

Recorder - L. & N. Speedomax Type G, or equivalent, with 1 second pen and 5 mv full-scale deflection. Recorder should be modified to switch to either the thermistor detector or the flame detector.

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REAGENTS AND SUPPLIES

Aluminum Tubing - 3/16 inch O.D.
Helium - 2 regulated supplies.
Chromosorb - red, 30/60 mesh.
Narcoil 40 - dinonyl phthalate from National Research Corp.
Hydrogen - electrolytic grade.
Air - well-filtered, clean supply.
Ethyl chloride - commercial grade.
2-Chloropropene - Columbia Organic Chemical Co., purified
by gas chromatography.
2-Chloro-1,3-butadiene - Monomer-Polymer, Inc., purified
by gas chromatography.
1,1-Dichloroethylene - Monomer-Polymer, Inc., purified
by gas chromatography.
Vinylacetylene - E. I. DuPont Co., purified by gas
chromatography.
Vinyl Chloride - Free of, or very low concentration of,
impurities.
Acetone - for column packing preparation.

PROCEDURE

A. Column Preparation

1. Mix 15 grams of Narcoil 40 with approximately 100 ml of acetone.
2. Pour mixture over 85 grams of 30/60 mesh chromosorb and add more acetone if necessary to make a slurry.
3. Mix continuously while evaporating the acetone. If available, use rotary-film evaporator with baffled flask for mixing. Apply heat from hot air gun and vacuum to flask to aid in evaporation.

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PROCEDURE (cont'd)

4. When the acetone is evaporated, re-screen to remove fines and pack one 20-foot length and one 35-foot length of 3/16-inch tubing with the 30/60 mesh fraction.
5. Install the columns in the instrument as shown on the attached drawing.
6. Adjust the two helium pressures and restrictor valve to give the following flows:
 - a. Column I: 75 ml/minute.
 - b. Column II (with restrictor in flow path): 85 ml/min.
 - c. Columns I & II (in backflush position): 85 ml/min.
7. Adjust temperature to 50° C. and allow instrument and columns to reach equilibrium.
8. Light the hydrogen burner and adjust the hydrogen and air flows so as to yield maximum signal-to-noise ratio when MVA passes through it.
9. Turn on amplifier and recorder and set for maximum sensitivity. The instrument should show no drift for at least five minutes before it is ready for use.

B. Analysis

1. Purge the 5 cc sample loop for at least one minute with sample flashed through the Mecovaporizing Valve, then inject the sample into the flowing carrier stream with the columns in Position A and the recorder turned to the thermal conductivity detector. The attenuator switch of the detector should be set on an attenuation of 2. The valve between the thermal detector and the flame detector should be in the vent position to prevent VCM from going to the flame detector.
2. Allow the sample to flow in Column I as shown in Position A for approximately three minutes (valve should be switched when recorder pen reaches 50% of full scale coming down on VCM peak). This allows the components lighter than VCM and most of the VCM to be vented from Column I.

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PROCEDURE (cont'd)

3. Just before MVA is eluted from Column I, switch the valve to Position B. This allows everything behind the VCM peak to be backflushed onto Column II where they are separated and then flow to the detector. Sometime after backflushing and prior to the backflushed VCM being eluted from Column II, switch the recorder to the flame detector and the valve between the thermal detector and the flame detector to the position such that the flow is through the flame detector.
4. Record all peaks eluted from Column II. A typical chromatogram is attached.

CALCULATIONS

The concentration of each component is calculated as follows:

1. Measure the peak height and width at one-half the peak height using a scale of 50 divisions to 1 inch.
2. Calculate the area of each peak according to the following equation:

area =

peak height x peak width at one-half peak height x attenuation

3. Calculate the concentration of each component as follows:

Concentration(ppm) = peak area x factor

PREPARATION OF REAGENT SOLUTIONS

None necessary.

CALIBRATION

1. Prepare synthetics of MVA, ethyl chloride, 2-CPY, 1,1-DCY, trans-1,2-DCY, and CB in VCM to cover the concentration range of interest. At least two synthetics should be prepared in each concentration range. Synthetics should be prepared in clean, stainless steel bombs of any convenient size. The pressure should be sufficient to permit extended use of the mixtures. The blending system used must be completely free of leaks and all kinds of foreign materials.

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CALIBRATION (cont'd)

2. Analyze the synthetic mixtures as described under the Procedure for Analysis.
3. Calculate the factor for each component according to the following equations.

factor =

$$\frac{\text{concentration (ppm) of component in synthetic}}{\text{peak height x peak width at one-half peak height x attenuation}}$$

SAFETY PRECAUTIONS

No precautions need be observed other than those normally observed when handling volatile chlorinated compounds under pressure.

ANALYTICAL TIME

The average analytical time for this analysis should be 1.0 hour.

PRECISION OF ANALYSIS AND SIGNIFICANT FIGURES FOR REPORTING RESULTS

95% confidence limits of this method is $\pm 5\%$ of the amount present.

Report results to two significant figures.

SCOPE

This method is designed only for heavy-end impurities in VCM, although the components analyzed for could be detected in other materials if the proper point is selected for backflushing.

EDC may be determined in VCM using the front column only. In this case allow the sample to run through Column I with the eluted components being vented until after VCM has been almost completely eluted, then switch the flow through the flame detector. Calibration for this should be made separately.

REFERENCES

Monsanto Research Method TC-1157 (GC).

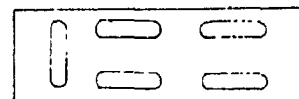
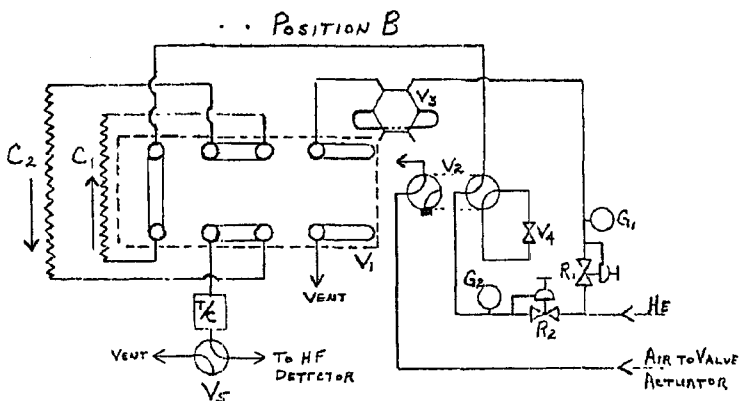
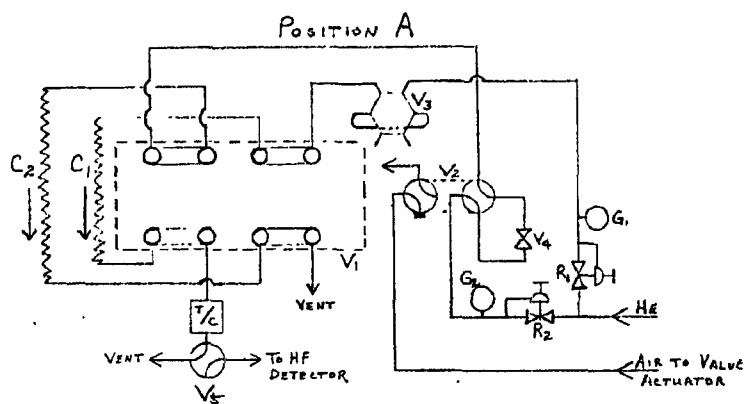
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VIEW OF CHANNEL LOCATIONS
IN TEFLON SLIDE FOR
WALL 8-PORT VALVE

- V₁ - WALL 8-PORT VALVE
- V₂ - CIRCLE SEAL DOUBLE 4-PORT VALVE
- V₃ - 6-WAY GAS CHARGING VALVE
- V₄ - NEEDLE VALVE
- C₁ - COLUMN I
- C₂ - COLUMN II
- R_{1,2} - HELIUM REGULATORS
- G_{1,2} - PRESSURE GAUGES, 0-60 #
- V₅ - CIRCLE SEAL 4-PORT VALVE

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FIGURE I

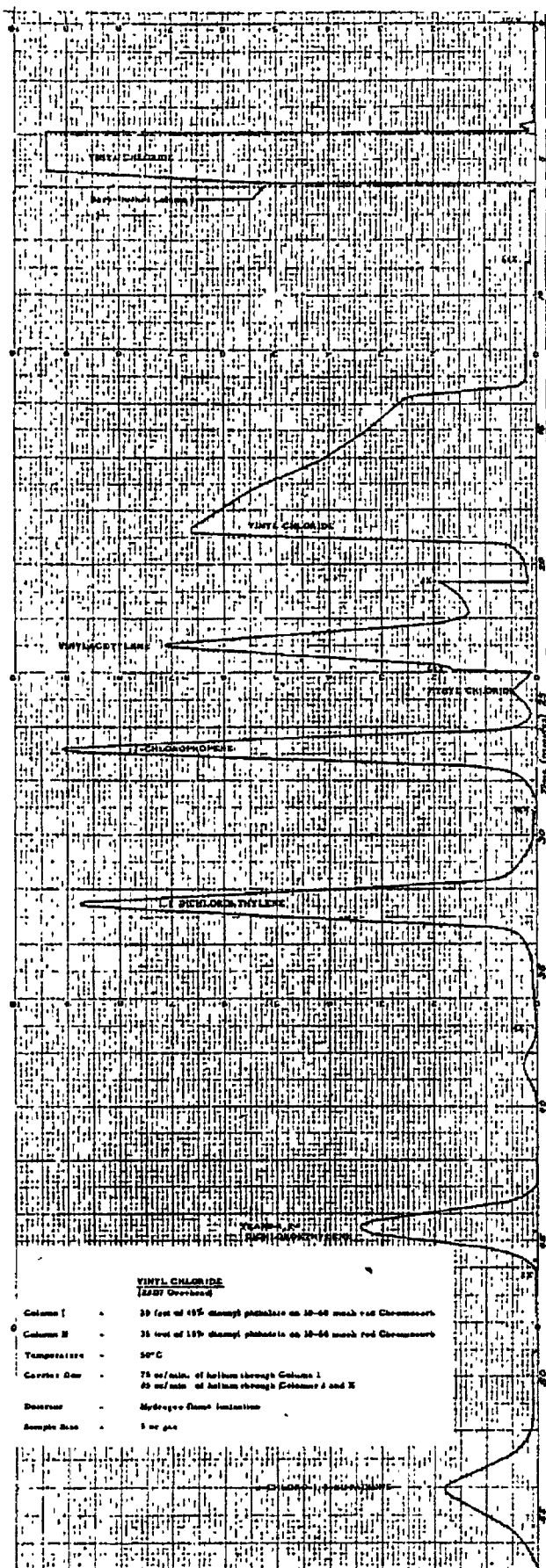
METHOD 602.296 (G.C.)
April 26, 1962
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