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TENNECO CHEMICALS, INC.

A Major Component of TENNECO INC.



TENNECO PLASTICS DIVISION

P.O. Box 116, Burlington, N. J. 08016

November 13, 1969

Mr. James C. Hahn, Technical Assistant
Tenneco Plastics Division
Promociones Industriales Mexicanas, S. A.
KM 2 Carretera La Resurreccion
Arpartado Postal Num. 604
Puebla, Puebla, Mexico

Dear Jim:

Attached is a copy of the gas chromatographic method (B.A.C.M. #201) utilized in Burlington for the analysis of mixed recovered monomer. A typical chromatogram is attached to the method to serve as a guide in optimizing conditions on your model 881 Perkin Elmer gas chromatograph.

As we indicated in earlier correspondence regarding monomer analysis, your Perkin Elmer instrument should perform this job very nicely. However, if you encounter any difficulties, we should be able to readily line these out by an interchanges of samples and chromatograms.

Very truly yours,

TENNECO PLASTICS DIVISION
Tenneco Chemicals, Inc.

H. B. Carr
H. B. Carr, Manager
Quality Control

HBC/rjl

Enclosure

cc: M. G. Caine
E. J. Hourihan
R. H. Marks
M. W. Williams
Primex File

COLORITE 016283

TENNECO CHEMICALS, INC.

TENNECO PLASTICS DIVISION

Burlington, New Jersey

B.A.C.M. #201

DATE ISSUED: November 6, 1969

Determination of Vinyl Chloride Monomer, Vinyl Acetate Monomer, and Trichlorethylene in Recovered Copolymer Monomer

APPLICATION:

The stripped off monomer from the production of polyvinyl chloride/polyvinyl acetate copolymers is recovered and proportionally mixed in with virgin monomers for each batch charged. To achieve the correct ratio of virgin monomer versus recovered monomer, the composition of the recovered monomer is quantitatively analyzed by gas chromatography. The level of trichlorethylene (TCE) is analyzed for at the same time to calculate the additional amount of TCE needed for each batch charged. The sensitivity for this method as written, is in the 0.1% range for each component to be analyzed.

APPARATUS:

1. Hewlett and Packard Gas Chromatograph - F & M 5754A, or equivalent (equipped with flame ionization detector (FID) or therm conductivity detector (TC) and septum covered injection port).
2. Hamilton syringe 50,1 (# 705 LT).
3. Hypodermic needle, luer short hub point style #1, 2" long, 24 gauge (#N-724).
4. Two stage regulators for, helium, air, hydrogen with secondary gauge capacity 0-100 psig.
5. 1/8" OD copper tubing.
6. 1/8" OD stainless steel 316 tubing.
7. 1/8" Swagelok nuts and ferrules.
8. Adjustable wrench 6" and 12".
9. Hoke gas bomb 150 ml, fitted with two valves (Cat. # 4HD150).
10. Vibrator (Scientific Glass Apparatus (SGA) Cat. # M-1425).
11. Glass wool.
12. Glass rod.
13. Evaporating dish, 200 mm diameter
14. Vacuum oven.

15. Analytical balance with 0.1 mg readout.
16. Hot plate.
17. 80 and 100 mesh Tyler screen assembly.
18. Funnel, long stem, 75 mm upper ID.
19. Tygon tubing, 3/16" ID, 3/32" wall thickness.
20. Dry ice chest.
21. 25 ml beaker
22. Planimeter (if available)

REAGENTS:

1. Silicone Gum Rubber UCC-W982 (methyl vinyl) (Hewlett and Packard Cat. #8501-3840).
2. Diatoport S 80 - 100 mesh (Hewlett and Packard Cat. #8501-6302).
3. Methylene chloride, analytical reagent.
4. Vinyl chloride monomer, virgin.
5. Vinyl acetate monomer, virgin.
6. Trichlorethylene, virgin.
7. Dry ice (frozen CO₂).

COLUMN PREPARATION: For column preparation see B.A.C.M. #1 substituting the following reagents, apparatuses and conditions:

Under 2., 5 gm of Silicone Gum Rubber UCC-W982 for 10 gm of Tributyl Phosphate.

Under 3., 50 gm of Diatoport S 80 - 100 mesh for 50 gm of Chromosorb P/AW 60 - 80 mesh.

Under 6., an assembly of 80 and 100 mesh Tyler screens for an assembly of 60 and 80 mesh Tyler screens.

Under 7., transfer the packing on the 100 mesh screen for 80 mesh screen (the size is now 80 - 100 mesh).

Under 8., cut a 12' length of 1/8" SS tubing for a 12' length of 1/4" SS tubing.

Under 12., condition the column with about 50 cc/min. Helium flow overnight at an oven temperature of 250°C for an oven temperature of 50°C.

CONDITIONS:

Column: 12' x 1/8" SS 10% Silicone Gum Rubber UCC-W982 on Diatoport S 80 - 100 mesh
Carrier Gas: Helium 50 cc/min. 40 psig in line
If FID is used:
Hydrogen: 60 cc/min. 40 psig in line
Air: 350 cc/min. 20 psig in line
FID Oven: 310°C

If TC Detector is used:
TC Detector Oven: 350°C
Detector bridge current output: 150 milli-
amperes
Oven: Ambient
Injection Port: 250°C

STANDARDIZATION:

No standardization is required since area normalization is used. Retention times and approximate sensitivities should be established for the components analyzed.

PROCEDURE:

1. Cool the bomb sample of the recovered MVC in the dry ice chest together with the 50 μ l syringe (hypodermic needle mounted) and the 25 ml beaker.
2. After at least 30 minutes, transfer about 10 ml of the liquid in the bomb into the cooled 25 ml beaker.
3. Rapidly withdraw about 20 μ l of the sample in the 25 ml beaker and inject the sample into the gas chromatograph. (Note 1)
4. Attenuate the three component peaks as they elute.

CALCULATION:

1. Determine the individual peak areas with either a planimeter or by measuring the width of the peak at half the peak height. (The planimeter area is direct reading.) The area for the half width of peak height determination is found by:
Total peak height in mm x width of the peak at half the peak height in mm = Area
2. Normalize all peak areas to one attenuation.

% component in recovered copolymer monomer =

$$\frac{\text{Area of component analyzed} \times 100}{\text{Total Peak Areas}}$$

Note: All steps described in 1.) through 3.) should be performed as fast as possible and in the dry ice chest. To best accomplish the inject, the dry ice box should be in close proximity of the gas chromatograph.

Figure 1 shows a typical chromatogram.
Analysis run with FID.

Written by: F. W. Kanzler

Issued by: H. B. Carr
H. B. Carr
Manager - Quality Control

Attachments

COLORITE 016288

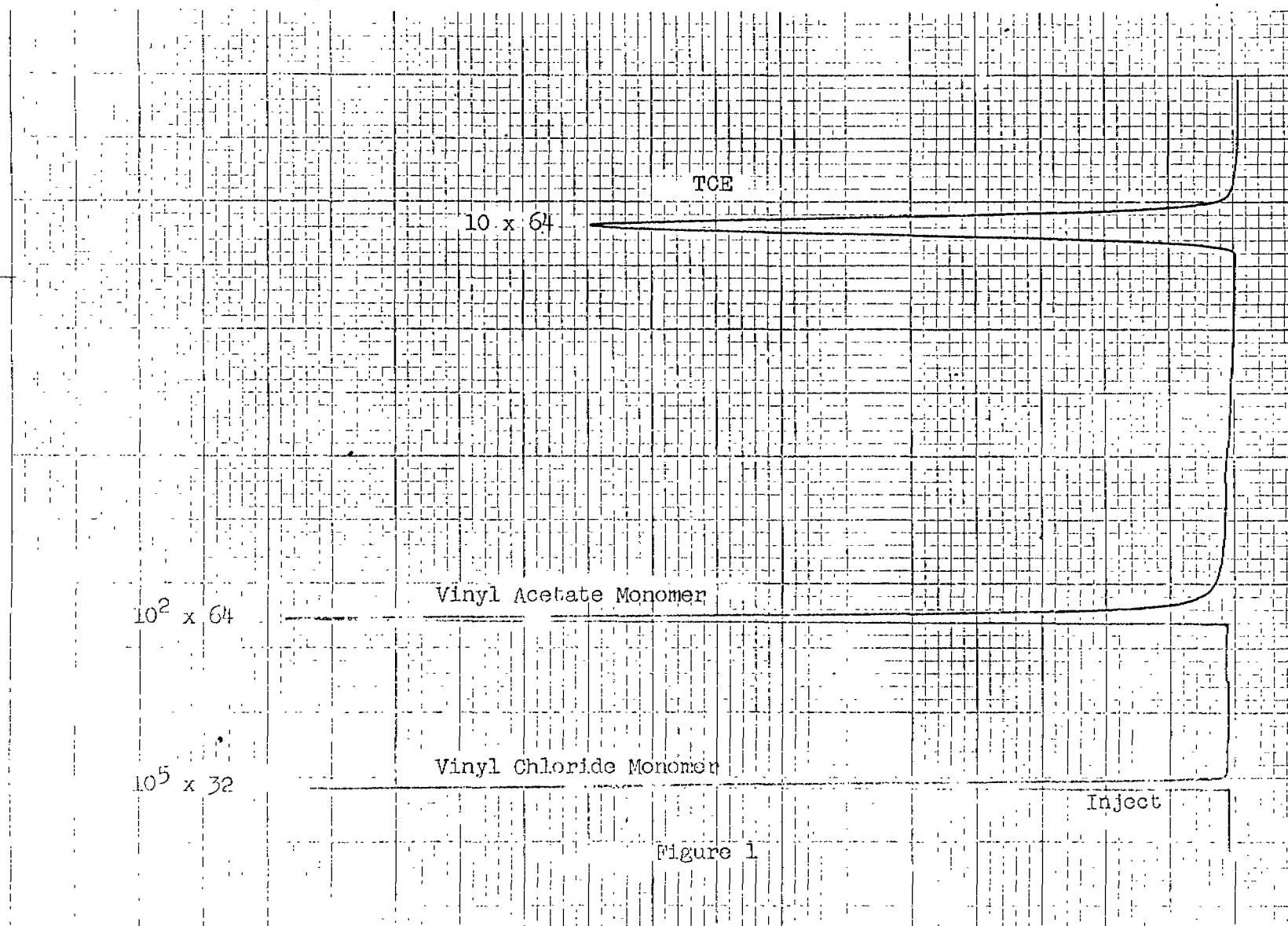


Figure 1