

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

To H.B. Carr

At Burlington

DATE March 10, 1972

FROM E.L. Kanyok

AT Burlington

COPY TO P.R. Scarito
M.W. Williams
File

SUBJECT Burlington Quality Control Manpower

This memo is to advise you that the Burlington Quality Control testing workload has become increasingly backlogged, particularly in the area of CRE's and special testing support.

The reasons for this situation are:

1. Preparation of Columbia Records preshipment samples which is time-consuming (screening 5-lb. samples through 50 mesh screen, counting contamination and checking bulk density).
2. Pilot Plant is submitting special requests (8 to 10 weekly) for testing of packed bulk density, loose bulk density, PTU (using DOP) and PTU (using G-54).
3. Resins Group is submitting CRE's (21 in the first 7 days of March) for complete analysis.

Personnel

4. M. McGargile transferred to Production on 3/6/72 and has a 14-working-day trial period. At that time, another man will be brought into the lab who will also have a 14-working-day trial period which means we will not be up to maximum efficiency until 4/17/72.
5. In the past month, A. Gentilini was out sick for 9 days and J. Duffy for 7 days.
6. Of course, we were cut back 1 technician in mid-December, 1971.

It should be noted that raw materials, in-process testing, finished product testing and bulk shipments have priority over CRE's and special requests.

I would recommend that:

1. PTU's, bulk density, etc. be run at the Flemington site.
2. We review current test frequency for standard products (in-process and finished products) and reduce where appropriate and if feasible.
3. Our manpower be increased to include the technician we lost in December if the addition to the budget can be approved. This man was assigned to day shift and handled most of the CRE's and special requests in addition to running testing rechecks, raw material tests and equipment calibration check-outs.

ELN/jt

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TENNECO CHEMICALS, INC.
TENNECO INTERMEDIATES DIVISION

ANALYTICAL & PHYSICAL TESTING MANUAL INDEX

SECTION 300

	<u>Method No.</u>
Determination of Bound Vinyl Acetate in Vinyl Acetate/Vinyl Chloride Copolymers by Infrared Spectroscopy	300-1
Determination of Residual Vinyl Acetate Monomer in Polyvinyl Chloride/Polyvinyl Acetate Monomer	300-2

TENNECO CHEMICALS, INC.

TENNECO INTERMEDIATES DIVISION

T.A.M. 300-2

Specific Use: Burlington/Flemington

Date Issued: March, 1972

DETERMINATION OF RESIDUAL VINYL ACETATE MONOMER IN POLYVINYL CHLORIDE/
POLYVINYL ACETATE MONOMER

APPLICATION

The level of residual vinyl acetate monomer is suspected to have a direct influence on the processability of the Copolymer.

The analysis is accomplished by dissolving the polymer in tetrahydrofuran (THF) followed by precipitation with methanol, followed by gas chromatographic analysis of the supernatant liquid. The sensitivity for this method, as written, is to the 0.001% (10 ppm) level.

APPARATUS:

1. Hewlett-Packard gas chromatograph, F&M-5754A, or equivalent (equipped with a hydrogen flame ionization detector (FID) and septum covered, on column injection port).
2. Hamilton fixed needle syringe, 50 μ l (Cat. No. 705N, Hamilton Company, P.O. Box 307, Whittier, California 90608).
3. Screw cap Erlenmeyer flask, 250 ml.
4. Burrell wrist-action shaker (Cat. No. 21-6055, Ace Scientific, 1420 East Linden Ave., Linden, New Jersey 07036).
5. Aluminum foil.
6. Analytical balance with readout to 0.1 mg.
7. Volumetric flask, 50 ml.
8. Transfer pipette, 30 ml, 20 ml.
9. Graduated cylinder, 50 ml.
10. 1/8" Swagelok nuts and ferrules.

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11. 1/8" OD 316 stainless steel tubing.
12. 2-stage regulators for helium, hydrogen, air with secondary gauge capacity 0-100 psig.
13. Glass wool.
14. Glass rod.
15. Evaporating disc, 200 mm diameter.
16. Vacuum oven.
17. Hot plate.
18. 60- and 80-mesh Tyler screen assembly.
19. Funnel, long stem, 75 mm upper ID.
20. Tygon tubing, 3/16" ID, 3/32" wall thickness.

REAGENTS:

1. Silicone gum rubber, UCC-W-982 (methyl vinyl) (Hewlett-Packard, Cat. No. 8501-3840).
2. Diatoport S, 80-100 mesh (Hewlett-Packard, Cat. No. 8501-6302).
3. Methylene chloride, analytical reagent.
4. Vinyl acetate, monomeric.
5. Tetrahydrofuran, chromatography quality (THF).
6. Water, demineralized.

COLUMN PREPARATION:

For column preparation see T.A.M. 2-1 except use an 8-ft. length of 1/4" stainless steel tubing as the column.

CONDITIONS:

Column:	8-ft. x 1/4" stainless steel 10% silicone gum rubber, UCC-W-982 on Diatoport S 60-80 mesh
Carrier Gas:	Helium, 47 cc/min, 50 psig in line
Air:	350 cc/min, 20 psig in line
Hydrogen:	60 cc/min, 40 psig in line
Injection Port:	70°C
FID:	120°C

CALCULATION: (continued)

ppm of residual vinyl acetate = $\frac{\text{ppm in standard} \times \text{peak height of sample}}{\text{peak height of standard}}$

(A typical chromatogram is shown in Figure 1.)

Written By: F.W. Kanzler

Issued By: H.B. Carr, Supervisor
Quality Control

STANDARDIZATION:

1. Using a 50 μ l syringe, weigh approximately 15-30 mg of vinyl acetate monomer into a 50 ml volumetric flask. Record to the nearest ± 0.1 mg. (The vinyl acetate level should be close to the anticipated residual monomer level in the sample to be analyzed. The weights given above represent a residual monomer level from 3,750 to 7,500 ppm (4 gm sample)).
2. Pipette 30 ml of THF into the 50 ml volumetric flask. Shake well.
3. Make up to volume with water.
4. With a 50 μ l syringe, inject 5 μ l of the standard into the gas chromatograph and attenuate the peaks as required. (The standard must be run at the same attenuation as the sample.)

PROCEDURE:

1. Into a 250 ml Erlenmeyer flask, weigh 4.0 gms of sample. Record the weight to the nearest ± 0.1 mg.
2. Pipette 30 ml of THF into the Erlenmeyer flask. Cover with aluminum foil and cap.
3. Place the flask on the Burrell wrist-action shaker and allow it to shake for 4 hours. Make certain that the sample is completely dissolved.
4. Pipette 20 ml of water into the Erlenmeyer flask and place on the shaker for 1 hour to facilitate precipitation of the polymer.
5. After 10 minutes of standing, inject 5 μ l of the supernatant liquid into the gas chromatograph. Attenuate the peaks as required.

CALCULATION:

1. Measure the peak height of the standard vinyl acetate peak (in millimeters).
2. Measure the peak height of the sample vinyl acetate peak.
3. Record the values from steps 1. and 2. and the ppm concentration of the vinyl acetate standard.