

TO Route List AT DATE September 25, 1975
FROM W.C. Champion AT Burlington COPY TO ☒ File
SUBJECT Revised Test Methods

Attached are two Test Methods which are to be inserted into your Quality Control Manuals. They are as follows:

<u>Test Method</u>	<u>Title</u>	<u>Manual Section</u>
2-31	Determination of Hard Particles in Record Resin	2
4-104	Quantitative Analysis of Copolymer Recovered Monomer	4

Method 2-31 supersedes the issue of January, 1970. Please discard the obsolete procedure which this replaces. Method 4-104 is currently being used at Flemington. This technique will be employed at Burlington at a later date. In the meantime, please retain the copy of Method 4-104 issued January, 1975 in your Manual.

W.C. Champion
(jst)
W.C. Champion

Attachments (2)

WCC/jst

Route List

W.D. Bord
H.B. Carr
D. Hershkowitz
H.R. Horton
F.W. Kanzler
D.J. Krzywicki
W. Miringoff
G.I. Rozand
P.R. Scarito
A.C. Siegel
J.T. Sweeney
R&D - Flemington

TENNECO CHEMICALS, INC.
ORGANICS & POLYMERS DIVISION
TESTING METHOD

DATE: September, 1975

METHOD NO: 2-31

TITLE: Determination of Hard Particles in Record Resin

PURPOSE: This method is for determining the possibility of hard particles causing stamper damage.

APPARATUS: Balance, 1,000 gm capacity
Paper Cups
Timer, calibrated in seconds
Hydraulic Press, steam heated (60,000 lb capacity)
Q-Panels, aluminum
Spatula, stainless steel, 6" length

CHEMICALS: Resin to be tested
Standard resin (315 type)

PROCEDURE:

1. Weigh 500 gms of resin to be tested and screen through a 40 mesh screen (.0165" in opening).
2. Press oversize particles with a spatula to determine if they are hard particles.
3. If the particles are found to be hard, mix the oversize particles with 3 gms of the standard resin and place between two aluminum Q-Panels.
4. Place in hydraulic press heated to 300°F and close as rapidly as possible to 60,000 lbs pressure and hold for two minutes.
5. Remove aluminum Q-Panels from press to cool.

REPORT: Carefully examine surfaces of aluminum Q-Panels for any small dents or scratches. Report number and nature of defect (e.g. scratches, nicks, dents).

SAFETY: Gloves should be worn when removing Q-Panels from the press.

WRITTEN BY: D.J. Krzywicki

ISSUED BY: 

W.C. Champion
Supervisor - Quality Control

Supersedes Issue Dated 1/27/70

COLORITE 009881

TENNECO CHEMICALS, INC.

ORGANICS & POLYMERS DIVISION

TESTING METHOD

DATE: September, 1975

METHOD NO: 4-104

TITLE: Quantitative Analysis of Copolymer Recovered Monomer

PURPOSE: To quantitatively determine the composition of copolymer recovered monomer

APPARATUS: Gow-Mac Gas Chromatograph (Model 69-100)
Carle Liquid Sampling Valve, 5 μ l inject (Model 2015)
Whitey Sampling Cylinders, 150 ml capacity

CHEMICALS: Sample to be tested
Helium, high purity

INSTRUMENT CONDITIONS: Gow-Mac: 50-55°C (temperature control set 25)
Inject Port Heater: - off
Detector Current: 150 maximum
Liquid Sample Valve
Oven Temperature: 30-40°C
Carrier Gas: helium 80 psig in line pressure

Note: The instrument is never to be turned off except during the change of helium cylinders or when a malfunction is noted.

SAFETY: Vinyl chloride is a Cancer Suspect Agent. Avoid physical contact or breathing of vapor. The sample valve must be installed in such a way that it provides venting of the sample purge to a hooded area.

PROCEDURE:

1. Attach one end of the sample cylinder to the quick connect on the inlet side of the sampling valve.
2. Attach the other end of the sample cylinder to the quick connect on the helium pressurization line.
3. Open helium pressurization valve.
4. Open upper valve on sample cylinder (this will pressurize the sample cylinder with 80 psig of helium).

5. Check helium flow rate through the gas chromatograph column using a soap bubble meter and a stop watch (flow rate should be 13 ± 2 seconds per 50 ml).
6. Open vent valve completely.
7. Open lower valve on sample cylinder and allow liquid to purge through the liquid sample valve.
8. When there is complete flow through the vent line, close the vent valve completely and reopen the valve approximately $1/8$ of a turn. (The liquid monomer flow rate, through the vent line, is properly adjusted when small droplets can be observed at the end of the line.
9. Start the chart drive and set the attenuation to 256x for VCM.
10. Turn the liquid sample valve handle to either the left or right position. (Each turn of the handle will automatically inject 5 μ l of sample into the gas chromatograph column.)
11. After the elution of the vinyl chloride, change the attenuation to 128x to accommodate the vinyl acetate monomer.
12. When the VAcM has eluted, change the attenuation to 8x and wait for the TCE peak.

Note: The order of elution is:

- a. vinyl chloride monomer
- b. vinyl acetate monomer
- c. trichloroethylene

13. After the completion of the test, close the upper valve on the sample cylinder and the helium pressurization valve. Remove the pressurization line quick connect and open the upper valve on the sample cylinder. Allow the residual monomers to purge from the cylinder and the liquid sample valve.
14. Remove the sample cylinder from the unit and install the special adapter between the pressurization line and the inlet side of the liquid sample valve.
15. Open the vent valve completely and open the helium pressurization valve and purge the complete system by rotating the liquid sample valve back and forth several times.
16. Leave the adapter in position to prevent any foreign material from entering the lines and shut the helium pressurization valve off.

CALCULATIONS: $(X) \times (Y) \times \text{attenuation} = \text{peak area}$

X = height of peak

Y = width of peak at half the height

B = MVC peak area

C = MVAc peak area

D = TCE peak area

Percentage of components by weight:

$$\text{MVC} = \frac{B}{B+C+D} \times 100 \times \text{RF}$$

$$\text{MVAc} = \frac{C}{B+C+D} \times 100 \times \text{RF}$$

$$\text{TCE} = \frac{D}{B+C+D} \times 100 \times \text{RF}$$

RESPONSE FACTORS:

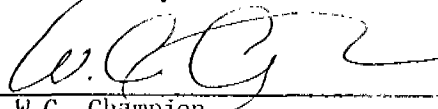
vinyl chloride monomer x 1

vinyl acetate monomer x 1

TCE x 1.6

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