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CHEMICALS AND PLASTICS

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To Name	Mr. R. N. Wheeler	Date	April 17, 1975
Division		Originating Dept	Research and Development
Location	514	Subject	Sampling of Vinyl Resins for Residual Vinyl Chloride Monomer Analysis
Copy to	R. M. Arnold, 515 G. P. Bigelow, NYO M. E. Eisenhour, 515 T. L. Dawson, 511 C. E. Fry, 514 M. E. Griffith, 514 D. E. Hardman, NYO L. P. McMaster, 312 G. F. Tacquard, 515 W. E. Whitehurst, 511		

Dear Nick:

Attached for review of the recipients of this letter is a "Recommended Procedure for Sampling and Storage of Test Samples of Vinyl Resins for Residual Vinyl Chloride Monomer Analysis". After discussions with the Plant Control Laboratory personnel we concluded that the documentation of a procedure which would yield data essentially the same as that of samples received by customers is a difficult task because of the highly volatile nature of vinyl chloride monomer. Consequently, the procedure attached is intended to define the residual vinyl chloride monomer level of the product at the point of transfer to the final container prior to shipment (bags or hopper cars).

Ideally, sampling procedures are based on analysis of material withdrawn from a large lot by a continuous sampling scheme; because of the volatility of vinyl chloride monomer, however, we do not believe such a procedure conducted in a typical manner would be appropriate. Consequently, the procedure described involves essentially "spot" sampling during packaging with precautions listed to insure that the sample taken from a lot and the sample analyzed are essentially the same.

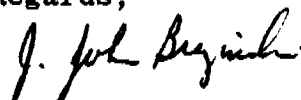
Unquestionably, depending on the level of vinyl chloride monomer present and with specific resins, other sampling procedures than those described may also be suitable. The method written allows for use of such variations, provided that comparable results are obtained.

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Comments from the recipients of this letter are welcomed. The intent is to publish the recommended procedure as an attachment to the Standard Method "Determination of Residual Vinyl Chloride Monomer In Vinyl Resins", WC-326-G-1.

Regards,



J. John Brezinski

JJB:noh

Attachment

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RECOMMENDED PROCEDURE
FOR SAMPLING AND STORAGE
OF TEST SAMPLES OF VINYL RESIN
POWDER FOR RESIDUAL VINYL CHLORIDE MONOMER ANALYSIS

1. Purpose

This method describes a recommended procedure for the sampling and storage of test samples of vinyl resin powders prior to analysis for contained vinyl chloride monomer. The principle objective of the procedure is to insure that the analysis of free monomer in the sample is representative of the sample taken at the producing or packaging unit.

2. Limitation

Containment of residual vinyl chloride monomer (b.p. of -13.9°C) requires storage of the resin powder or granules in an air-tight container. Opening of the container at any point prior to withdrawal of a sample for contained monomer analysis will result in the escape of monomer present and thus the data will not be representative of the lot or blend.

As the containers/lids recommended are not hermetically sealed, slight loss of monomer can occur particularly over a prolonged storage period: to minimize this possible source of error, the time interval between the taking of the sample and analysis for vinyl chloride monomer should not exceed 24 hours.

3. Equipment

- a) Glass jar, 4-oz., wide-mouth, fitted with a metal or phenolic based aluminum-lined cap.
- b) Electrical tape, 1/2 inch wide, elastic
- c) Sample identification labels
- d) Sample scoop
- e) Small brush

4. Procedure

- a) Transfer the vinyl resin powder or granules into the clean glass jar, filling the jar.
- b) Brush the top surface of the glass and the side threads to remove any particles of resin.
- c) Immediately seal the jar with the aluminum-lined lid, applying adequate pressure to insure a good seal.

- d) Stretch a single thickness of electrical tape completely around the jar at the juncture of the lower end of the lid and the glass jar. Press along the surface of the tape to insure a good adhesive bond.
- e) Place an appropriate sample label on the jar indicating the resin designation, blend or lot number, date and time of sampling.

5. Sampling Frequency and Timing

The number of samples taken and frequency of sampling will depend on the lot or blend size. At a minimum, sampling must be made as the resin powder or granules are being packaged or transferred into the final shipment container, i.e. during the bagging operation or during the filling of a hopper car.

Bagging Operation

Samples for vinyl chloride monomer should be taken at the beginning, in the middle and near the conclusion of bagging of a definable blend or lot of resin.

Hopper Car Sampling

Samples should be taken at the outset, in the middle and near the end of the filling of the hopper car from the resin stream being transferred to the car.

Frequency of Sampling

Frequency of sampling of a specific resin product with a consistent value of contained monomer, i.e. <1.0 ppm, can be reduced following the guidelines of the Skip-Lot Sampling Plan (Union Carbide Informational Test Method, 23-204-21).

Variations In Sampling Procedure

Variations in sampling procedure and technique can be utilized provided they yield results comparable to those obtained by the procedure described.

6. Analysis of Sample For Residual Monomer

- a) Analysis of the sample of powdered or granular vinyl resin for contained monomer should be conducted as soon as possible after the sample container is filled, preferable within 4-hours after sampling and in no case later than 24-hours after sampling.
- b) Samples analyzed for residual monomer should be taken from near the center portion of the contents of the jar as soon as possible after the jar is opened and in no case later than 1 minute after such opening*.

* Caution: Removal of the sample jar lids and withdrawal of sample for testing should be conducted in an effectively hooded area to minimize the possibility of inhalation of contained vinyl chloride monomer.