



Interoffice Communication

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To R. D. Jackson
From D. S. Cox
Date December 12, 1980
Subject EPA VARIANCE STUDY: THE EFFECT OF SAMPLE STORAGE TIME ON
RESIDUAL VCM CONTENT OF PVC WETCAKES

Object The objective was to conduct a study to determine if PVC wetcake samples can be stored for longer than 24 hours without loss of VCM. If the data showed no loss of VCM, they would be used to request a variance from EPA on the mandatory 24-hour limit for analysis after sampling.

Conclusion

The study shows there was no significant loss of residual VCM as the PVC wetcake samples were held over the five days of the study.

Introduction

The samples were collected and prepared at the Aberdeen Chemical Plant September 22, 1980. Twenty-three 8-oz bottles of 5385 PVC slurry were collected in rapid succession during a reactor dump from a D-300 reactor. The bottles were taken to the laboratory where five were randomly chosen from the set and placed in the refrigerator at 40°F to cool to room temperature. When the bottles had cooled sufficiently, the laboratory personnel prepared five wetcake samples from each of the five bottles. This involved filtering the slurry using a large Buchner funnel and weighing plugs of the resulting wetcake into F-42 vials for VCM analysis. The vials were then stored in the refrigerator at 40°F. A set of five vials, one from each of the five bottles, was analyzed each day for five days using the F-42 Headspace Analyzing Chromatograph according to EPA Method 107. After the 25 vials had been analyzed, a total solids was determined on each vial by drying each sample-plus-vial and recording the weight loss relative to the wet sample weight. The residual VCM concentrations in wppm on a dry weight basis obtained over the five-day period are shown in Table I.

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Experimental Conditions

The method as prescribed by EPA for determining residual VCM in PVC slurry (EPA Method 107, Federal Register, Vol. 41, No. 205, October 21, 1976, and amendment in the Federal Register, Vol. 42, No. 109, June 7, 1977) requires that PVC wetcake be analyzed within 24 hours after sampling, using a Perkin-Elmer F-42 Automatic Headspace Analyzer. The method presently being used in the Aberdeen Plant Lab was adopted directly from EPA Method 107. The wetcake sample is equilibrated in a constant temperature bath of 90°C for one hour. The VCM concentration in the headspace of the vial is determined by gas chromatography, using the F-42 and automatic injection, based on analyses of certified VCM cylinder standards. From a headspace concentration, the sample concentration is calculated on a dry resin basis in parts per million by weight (wppm).

Statistical Analysis

A trend analysis test was then performed on the daily mean VCM residual concentrations. Spearman's rank coefficient test was used. A Spearman rank coefficient of 0.10 was determined from the test of the daily mean averages. A coefficient of less than -0.90 would have indicated a significant loss of VCM from the samples during the holding period. Since no significant loss of VCM was determined, the conclusion reached is the holding of residual VCM samples up to five days before analyzing them is an acceptable procedure.

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Attachments

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