Air Products and Chemicals INC.

ESCAMBIA PLANT
INTEROFFICE MEMORANDUM

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Escambia
IH

To: L. S. Forester

Date: January 11, 1978

Copies:

From: R. R. Spiegelhalter

Re: Laboratory Monthly Report
December, 1977

Reports by J. H. Williamson and K. J. Femrite covering QC Laboratory operations and working atmosphere monitoring are attached. The results of the working atmosphere monitoring program are also distributed as a separate report to production areas. PVC, amines and methanol were the largest users of laboratory services.

Amines

Standards for all amines products shipped or produced during the month were analyzed on a routine basis to maintain control of the chromatograph data reduction system. The precision of the standards analyzed was normal.

A 60% DMA sample from a July shipment was returned from Calgon for comparative analysis. Problems were encountered in obtaining reproducible results for the pertinent impurities at ppm levels. GC columns were reconditioned or replaced and by use of internal standards acceptable results were obtained. Satisfactory agreement with Calgon on TMA, MEA and DMEA was obtained though we still find more MEA than Calgon (100 ppm vs. 60 ppm). MMA levels reported in July agreed with the Calgon number at the 120 ppm level. Current analyses show 500-800 ppm MMA using an internal standard or external standard. The higher numbers are believed to be the most correct since column performance, sensitivity and sample injection technique have been upgraded over the past weeks. Work to fully evaluate results and upgrade procedures is continuing.

A complaint from Calgon on a shipment in November (A 14987, 11/18) concerning high MMA levels was received. The material was shipped knowing that MMA was out of specs. Reanalysis of the shipment sample and a sample returned from Calgon confirmed MMA at 370-450 ppm (420 reported by Calgon).

An unknown impurity in EBA product eluting before EBA on the Escambia GC column was identified by the Houdry Labs as 2-methyl-pentylamine.(1) The impurity is present at the 0.25% level in current storage material and ranged

from 0 to almost 1% during the production run. The mixed amine was purchased but found to elute after EBA on our column indicating the major impurity is another material. A more likely candidate is 3-methyl-pentylamine. This was not available from the usual suppliers. The only other 6 carbon amine available was 2-amino hexane. This compound eluted on the backside of EBA and at low levels (<0.1%) would probably not be detected.

Work is in progress to upgrade the GC columns used for analysis of TEA product. Adequate separation of unknowns eluting on the backside of the TEA peak is not obtained on a routine basis. The improved analysis will be most useful in future TEA production runs.

Upgrading the GC analysis for DMF to insure detection of other amides is also in progress. Proprionamide and ethyl formamide were detected by GC-mass spec analysis at Houdry. Current DMF product does not contain these impurities but we need to be able to monitor product for other amides as well as the usual low boilers on a routine basis.

PVC

Evaluation of IV and PSD measurements received major emphasis in December as a result of the JM quality problem and in conjunction with R.W. Wolfe. Modifications to the sample weighing and dissolution procedures for IV were implemented at month end as a result of the work. Stainless screens were placed in service about mid month. In order to upgrade IV results significantly technicians must be more precise in preparing samples requiring more work time than has been provided. The precision of IV results in December exemplifies this. Up to mid month the standard deviation for control analysis exceeded 0.01 units. When the work load decreased, the deviation dropped toward 0.005 units, the typical value for control analyses.

The modified procedures should provide further increase in precision. PSD results will be more consistent when riffling of samples is implemented on a routine basis. A riffler should be delivered in January.

A procedure to monitor peroxides in RVCM was established. Contacts with the laboratory at Calvert City concerning the use of sodium nitrite as polymerization inhibitor in the recovery system was made. Adjustment of the pH of the salt solutions between 7 and 8 with bicarbonate may be critical.

AN and Solutions

The precision of analyses for urea in AN in nitrogen solutions was established as ±0.1% absolute at the normal concentrations. A revised procedure will be

issued for laboratory use. Monitoring of solution storage tanks and solution production to provide better control of nitrogen content should be implemented. Production will be contacted.

Quality Complaints

In addition to the quality problem with DMA and Calgon, a DMF problem with a sample shipped to Mexico and a methanol problem were reported. Based on information available, differences in methods of analysis account for the DMF quality problem. Retained material meets the customers requirements. Methanol used for duplicating machine cleaning solvent was analyzed to detect impurities leading to a low flash point. The customer reported a fire occurred during use. The flash point of the returned sample was 60°F, as reported in the literature. GC analysis did not show the presence of low boiling materials.

Literature information concerning the preparation of quaternary ammonium salts from TMA and aromatic halides was provided a customer at the request of Atlanta sales personnel.

Working Atmosphere Monitoring

Working atmosphere monitoring for VCM (area and personnel), for methylamines and IBN, for particulates in Area B and the PVC bagging area, and for alcohols in Area A was conducted throughout the month. Development of a sampling procedure for DMF for personnel monitoring was completed. Specific data are given in K. J. Femrite's report. Problem areas of high levels of air contaminants were not observed.

R. R. Spiegelhalter

(1) A. G. McFarland to R. R. Spiegelhalter, "EBA Product Analysis," October 21, 1977.

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