



CALVERT CITY PLANT
INTEROFFICE MEMORANDUM

To: T. C. Noll
Date: 04 April 1984
From: B. L. Rose
Copies: C. J. Agee
B. D. Bazzell
G. N. Boyd
C. Riley
C. J. Smith
M. H. Stermon
Re: LABORATORY HIGHLIGHTS
MARCH, 1984

I. INDUSTRIAL HYGIENE

A. Emulsion - Noise Monitoring (G. Boyd, C. Riley)

A complaint of excessive noise levels in the emulsions truck loading alley was investigated. Although the noise from the 4 chiller compressors is annoying, it does not exceed the OSHA action level of TWA 85 dB.

B. PVC - Personnel Monitoring for Vinyl Chloride (J. Joyce)

PVC operating personnel were monitored for VCM exposure with each shift being sampled for 15 minute and 8 hour TWA.

II. PROCESS ASSISTANCE

A. Emulsion

1. DTA Instrument Installation (C. Riley, J. Joyce, C. Agee, Emulsion Technicians)

Manufacturer service came in for repair of the DTA instrument recently purchased, finding a corroded wire to be at fault. Equivalency of this new instrument with the unit presently in service was established with one analyst in the main laboratory. The new unit has now been moved to the emulsion laboratory and equivalency between the emulsion lab with four technicians and the main lab with only one analyst is being established. A problem with recorder zero drift was experienced at one point, leading to greater variation in results than desired. This recorder has since been returned to the manufacturer for repair. The replacement recorder with which instrument equivalency was previously established has been installed for use. DTA results will continue to be reported from the main lab until equivalency is shown.

2. Constant Temperature Bath (C. Riley)

A new constant temperature bath was purchased to comply with the emulsion quality assurance program. The model selected is equivalent to the unit in service at the other emulsion plant laboratories.

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B. Polyvinyl Alcohol

1. Vinol 203 Test Trial (G. Boyd, PA Group, Shift Technicians)

Laboratory support to PCT was provided March 16-27 during the plant trial to produce Vinol 203. Round the clock shift coverage was required for extra poly kettle, paste stripper, dryer discharge, and screw press samples. The process assistance group analyzed dryer condensate and filtrate samples for percent solids, and dryer discharge samples for the special "NAPP" tests. The NAPP tests include 20% solids, 10% viscosity and color, gels through 20 and 35 mesh screens, and 4% UV transmittance measurement at 320 nm. The analyses of many of the dryer discharge samples were near the targeted values for the test trial. As of this writing final lot analyses are incomplete. Over 500 extra analyses were required for the test trial.

2. Wedco Testing (G. Boyd, Shift Technicians)

As requested by S. Crowley, 15 PSD and volatile analyses were conducted on various samples from the PVOH grinding area. The work is part of an on-going project to debottleneck this area of the plant.

3. Analysis of Turbo Dryer Housing for Rustguard (C. Agee)

A piece of metal from the 5200 turbo dryer housing was analyzed to determine if the housing was coated, as specified, with the rust-proofing agent, Rustguard 101. The metal sample was allowed to soak in acetone (a suitable solvent for Rustguard 101) overnight. The acetone extract was analyzed via infrared spectroscopy for the characteristic absorbances of the rust-proofing agent. The spectrum showed no signs of the Rustguard coating on the metal. Results were reported in a memo to Ron Scott.

4. Pure Acid Column Corrosion (M. Sullivan)

Corrosion of the 5304 pure acid column was again a problem with chloride levels reaching nearly 50 ppm in Tray 27 samples. A small rise in Cl⁻ concentrations was seen across the 5304 condenser. Purges of the column were carried out until the new condenser was installed near the end of the month. Chloride levels then dropped to <5 ppm in Tray 27. Replacement of the old condenser should eliminate the primary source of Cl⁻ contamination.

C. Acetylenics

1. Determination of Specific Gravity and Viscosity of KOH/IPE Slurries
(C. Agee)

M. McHale requested that the specific gravity and viscosity of 30-40% KOH in ether be determined to assist in the acetylenics

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expansion planning. (Previous work had only made these determinations for up to 26% caustic.) A slurry tank sample (~22% KOH) was used as the base material and ether decanted from it to increase the KOH concentration to the desired range. Two slurries, one of ~32% KOH and the other of ~39% KOH, were produced and specific gravity, viscosity, % KOH and % K_2CO_3 determined. McHale reported that this laboratory experimental data is comparable to the data derived by extrapolating the previous data for lower KOH concentrations.

2. Distillation of High Color Karbochem Methyl Butynol (D. Lawson)

A sample of MB shipment purchased from Karbochem was analyzed and found to be very high in color and water. The laboratory was requested to determine if distillation was an effective procedure for removing the color and water. The distillations was carried out using the Oldershaw apparatus with 20 theoretical plates. Results as listed below show the distillation to be very effective in removing color and water.

	<u>Initial Sample</u>	<u>Distilled MB Cut</u>
MB, %	99.15	99.71
H ₂ O, %	0.55	0.16
Unknown, %	0.30	0.13
Color, APHA	610	14

3. SVS Monomer Analysis (C. Agee)

Calvert City has recently assumed the role of drumming location for bulk sodium vinyl sulfonate monomer. With this comes responsibility for all QC testing of this material. A preshipment sample of SVS complete with Paulsboro analyses and another sample of an actual bulk trailer shipment to Calvert have been analyzed for all specification properties via Paulsboro methods. Initially some variation was seen between Paulsboro and Calvert City analyses. Since talking to Renee Stack, Paulsboro, these differences have been resolved and good agreement between the two labs achieved.

4. Acetylenics Special Products/Blend Analyses (D. Lawson, J. Joyce)

Numerous special products and blends, e.g. Ancor 50-TT, Surfynol 104GA, Agent AT-548 (raw material), MB/H₂O azeotrope, etc., have required the support of the PA group in analysis. These samples require use of an internal standard for GC analysis or other special testing not routinely performed.

5. Surfynol 104 Intermediates Distillation (D. Lawson)

As requested by the plant an attempt was made to distill 104 intermediates in the Oldershaw column. Plant personnel suggested adding 10% water to the intermediates mixture in the pot to more effectively distill the Surfynol 61. No effective separation could be achieved as MIBK was produced throughout the distillation and was present in each cut in substantial amounts.

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D. Polyvinyl Chloride

1. Particulate Testing of Niro Dryer Emission Stacks (G. Boyd, C. Agee)

As requested by T. Garrisi, a particulate emissions test was conducted 02 March 1984 on the flasher dryer of the Niro system. The test procedure was EPA Method 5, "Determination of Particulate Emissions From Stationary Sources". The air flow was 32,000 standard cubic feet per minute and the particulate emission was 1.6 lbs/hr., well below the level which our permit allows. The work is necessary to assure state compliance at increased rates.

2. Iron Analysis of PVC Caustic Tank Samples (C. Agee)

PVC production personnel are concerned that the caustic rinse solution is picking up iron from the transfer line. As a result, five samples of the PVC caustic tank solutions were taken on different days and analyzed for iron. The iron content ranged from 7 ppm to 43 ppm by weight. A sample of the incoming 50% NaOH at PVOH was also analyzed and found to contain 1 ppm iron. Results were reported in a memo to K. Feather.

E. Acetic Acid

1. Eastman Acetic Acid Testing (M. Sullivan)

Ten shipment samples of Eastman acetic acid contracted by APCI for resale have been analyzed for the full spectrum of APCI merchant sales specifications. All samples were within spec with the exception of one which was low on assay. This discrepancy was also detected by Eastman and this shipment was halted. From this point on, we will adopt skip-lot testing of 1 in 10 samples as directed by T. Noll.

2. Analysis of 85% Acetic Acid Trailer (C. Agee)

Some difficulty has been experienced in the past with trailer shipments of dilute acetic acid due to the lack of a mixing tank or agitator. To verify sufficient mixing of acid and water in the trailer during transit, two samples were taken from a trailer of 85% acid after it reached its destination in Ironton, Ohio. The two samples analyzed 85.86% and 85.15% HAc, probably indicating sufficient agitation. Results were reported to J. Outland.

3. Acetic Anhydride Analysis (C. Agee)

E. Kuchar felt that chlorides present in our acetic acid might react to produce some positive interference in the acetic anhydride test. An experiment suggested by Kuchar indicated that their presence created no interference in analysis.

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F. Utilities

1. Waste Treatment

a. State Compliance Evaluation Inspection (C. Riley, V. Cates)

The annual state compliance evaluation inspection by the environmental engineer with the Division of Water was conducted. The laboratory was in compliance with all state regulations. The state engineer was impressed especially with our Quality Assurance Program and expressed interest in the Analytical Products Group environmental proficiency program in which we participate. The number of standards, spikes, and duplicates being analyzed had increased from 1% to 10% from the last audit.

b. Dilution Water (C. Riley)

A carbon filter was installed in the air line for purging dilution water. This appears to help control the oxygen depletion in the dilution water. Also new discharge tubing was installed on the glass still and a drain hose was placed in the vent line in order to eliminate possible contamination from the condensate. A study indicated that the oxygen probe is not a significant source of contamination.

c. Warburg Studies (C. Riley)

A Warburg study of a sample from the emulsion No. 2 earthen pit was completed. This study was extended to 28 days in order to study the long-range effects. The data is being calculated.

2. Fluorescent Dye Analysis (C. Agee)

F. C. Rehberg has requested that a method be developed for detecting and quantitating very low concentrations of fluorescent dyes, Rhodamine B and WT. He plans to use these dyes to calibrate flow meters for distribution of costs and to determine points of cooling water losses within the plant. Samples of the dye have been diluted and a detection level of 0.1-0.2 ppm set with the UV-Vis spectrophotometer presently available. The use of our Koll-Morgen Color Eye for analysis of lower concentrations is being studied. Also, information on fluorometers is being obtained from various vendors.

III. QUALITY CONTROL LABORATORY

A. Acetylenics

At the request of B. S. Melnick, a sample from a Karochem methyl butynol container was shipped to Calvert via Paulsboro for evaluation. The sample analyses failed to comply with the Air Products methyl butynol manufacturing specifications for color and water concentrations.

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B. Polyvinyl Alcohol

1. Four samples of Air Products polyvinyl alcohol were returned from City of Industry for grade(s) identification, reference R19961. The four samples were identified as Vinol 205 material.
2. Particle size analyses were determined for the "S" grade samples received from Tanner. Composite samples that failed to meet the products PSD specifications are Vinol 205S lot 010244-T, 010494-T, and 0100438-T; Vinol 523S lot 4-82037-T.

C. Overtime

The quality control and process assistance analysts worked 757.5 hours of overtime during March, 1984. The overtime represents 15.6 percent of the total hours worked by these analysts. Tabulated below is a summary of the overtime:

<u>Reasons for Overtime</u>	<u>Quality Control</u>	<u>Process Assistance</u>	<u>Total (Hrs.)</u>
Illness	20	-	20
Excused Absence	12	-	12
Safety/Training	20	-	20
Vacation	200	-	200
Acetic Acid	-	8	8
Acetylenics	-	13	13
Catalyst	13	-	13
Emulsion	-	15	15
PVC	122	44	166
PVOH	198	30	228
Waste Treatment	-	62.5	62.5
	581	172.5	757.5

IV. SAFETY

There was one (1) first aid case recorded during March which is the first - first aid case recorded in the laboratories during the last two years. There were no OSHA recordables or time away from work cases for FY84 to date. The laboratory has operated 14 years, 7 months without a time away from work case.

The first aid case No. 88-84 that was recorded in the catalyst laboratory on 02 March 1984 was reviewed in detail with the laboratory personnel. The use of the required personnel protective equipment for each job was strongly emphasized.

An incident report was filed on 13 March 1984 when a portable liquid oxygen sample vessel pressure relief device opened resulting in an uncontrolled release of high pressure oxygen into the laboratory area. There were no injuries or property damage. The sampling procedure has been revised to bring a low pressure gaseous oxygen sample to the laboratory.

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Two safety meetings and a safety committee meeting were held as scheduled. Make-up meetings will be scheduled for all those employees that were absent during the the regularly scheduled March, 1984 safety meetings.

Updated safe work practices for Inserting Thermometers into Cork or Rubber Stoppers - No. 2 and Connecting Rubber Tubing with Glass - No. 1 were approved and distributed.

V. SAMPLE DEPARTMENT

An audit was performed by Chemicals Group Customer Service to determine the elapse time for processing sample orders during February, 1984. The audit shows the average elapse time from entry in Trexlertown to shipment from Calvert is 1.3 days for Specialty Additives samples and 1.5 days for Polymers samples. The standard is an average of 4 days maximum.

The Calvert Sample Department completed 530 sample orders during March, 1984. A total of 1,823 separate samples were processed for shipment. The number of samples per storeroom manhour for March was 6.6. The number and percentage of samples shipped by product line are listed below:

<u>Product Line</u>	<u>Number of Samples</u>	<u>Percent of Total Samples</u>
Acetic Acid	3	0.2
Acetylenics	412	22.6
Airflex Emulsion	327	17.9
Atmospheric Emulsion	206	11.3
Automotive Catalyst	30	1.6
Beads	38	2.1
Polyvinyl Alcohol	326	17.9
Polyvinyl Chloride	51	2.8
Miscellaneous	430	23.6

The number of samples shipped by sample sizes for each product line is as follows:

<u>Acetic Acid</u>	<u>Acetylenics</u>	<u>Airflex Emulsion</u>
8 oz.-----1	8 oz.-----409	qts.-----11
qts.-----1	qts.-----1	gals.-----311
gals.-----1	gals.-----2	5 gals.-----5
<u>Atmos. Emulsion</u>	<u>Automotive Catalyst</u>	<u>Beads</u>
4 oz.-----6	4 oz.-----12	1bs.-----35
qts.-----11	qts.-----6	5 lbs.-----2
gals.-----185	gals.-----6	10 lbs.-----1
5 gals.-----4	300cc-----6	

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<u>Polyvinyl Alcohol</u>	<u>Polyvinyl Chloride</u>	<u>Miscellaneous</u>
1bs.-----284	4 oz.-----46	430 Samples
5 lbs.-----25	8 oz.-----4	
10 lbs.-----8	5 lbs.-----1	
50 lbs.-----9		

B. L. Rose
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