



Air Products and Chemicals
INC.

CALVERT CITY PLANT
INTEROFFICE MEMORANDUM

To: T. C. Noll

Date: 04 January 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
DECEMBER, 1983

I. INDUSTRIAL HYGIENE

A. PVC - Vinyl Chloride Monitoring (J. Joyce)

Personnel monitoring of PVC operators and foremen for vinyl chloride exposures over 8 hour periods was conducted with each shift being monitored. Pump samples to measure 15 minute exposures have also been taken.

II. PROCESS ASSISTANCE

A. Emulsions

1. Bioresistance Testing of FL-325 (C. Riley)

F. H. Fernengel submitted 3 lots of FL-325 for bioresistance testing. All lots had been treated with the biocide Giv-Gard at the plant. The biocide was highly effective in 2 lots but not effective in lot #K123. An aliquot of the original sample of lot #K123 was treated with an additional 1000 ppm Giv-Gard. This also failed to control the challenges. The recommended concentration is 1500 ppm. This lower concentration apparently was not high enough to control the challenges. This study will be completed soon.

2. Check for Cross-Mixing of Igepal 630 and 730 (C. Agee)

There was some question as to whether shipments of Igepal 630 and 730 had been mixed together. As a result, samples were taken of the top of the tank and the line out of the bottom of the tank for each Igepal storage facility for identification.

The infrared spectra yielded little conclusive information as to the identity of each since the spectra of both Igepals are so similar. Therefore, a cloud point determination which varies between the two Igepals was made on all samples. The cloud points were all within spec for the Igepal as sampled, leading to the conclusion that no appreciable contamination of one Igepal with the other had occurred. As a double check, cloud point determination was made for several ratios of 630 in 730, and a visible difference was seen with 10% Igepal 630 in 730.

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B. Polyvinyl Alcohol

1. Volatiles Study on Dryer Discharge Samples (G. Boyd)

As requested by J. R. O'Leary, a study was conducted on dryer discharge samples to determine the effect of drying time on % volatiles. V-325, V-523, and V-540 dryer discharge at 0800 11/28/83 were analyzed for solids at 1/2, 1, 2, 3 1/2, and 5 hours at 105°C. The results as displayed in Figure 1 (attached) indicate that after one hour the volatiles increase is minimal and one hour is the recommended time for volatiles determination in any future tests involving the PVOH grinding area.

2. Air Purging of Polyvinyl Alcohol (G. N. Boyd)

As requested by R. E. Varilek, laboratory tests were conducted to simulate air purging the PVOH silos as addressed in the recent PHR. The test work was needed to determine the air flow required to maintain the organic vapor concentration below 25% of the LEL. Due to the hygroscopic nature of PVOH, preliminary tests were conducted to determine the feasibility of purging the final product silos with air at a controlled relative humidity. In doing this, a positive impact on yield could be achieved rather than a negative impact which would be experienced if the silos were purged with dry air. These tests indicate that air at approximately 20% relative humidity would decrease solids, yet stay above the 95% specification, and therefore, effect a positive impact on yield.

3. Polymerization Catalyst Test (G. Boyd, PA Group, Shift Technicians)

During the week of December 12, 1983, lab support was provided for T. E. Boland/PCT to conduct a plant test using t-Butyl Peroxy-pivalate as the initiator in the production of medium viscosity paste. Three days of round the clock coverage were provided to analyze extra catalyst, paste stripper, poly kettle, and vinyl acetate samples.

During this run using the pivalate catalyst as opposed to the neodecanoate catalyst, the plant was experiencing polymerization difficulties - more catalyst was having to be used even though the pivalate catalyst has a higher active oxygen content than the neodecanoate catalyst. As a result of this, the active oxygen content of the pivalate catalyst in use was checked and found to be 7.13%, above the spec of $6.90 \pm 0.09\%$ and therefore obviously not low in activity. The vinyl acetate in use was then checked for the level of inhibitor (hydroquinone) and found to contain 1.7 ppm HQ, 0.5 ppm BQ. This level of inhibitor would be expected and would not interfere with polymerization.

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C. Acetylenics

1. Surfynol 440 Analyses (M. Sullivan)

Three Surfynol 440 samples were received from Emery Ind. and final product tests run. One lot was out of spec on both pH and color while one other lot was also out of spec on color. All other properties tested were within the specifications with one lot passing all tests. Two of these lots have been sent to Allentown for additional testing.

2. Testing of MB for European Specifications (J. Joyce)

Additional testing of MB for benzene, dry residue, and free acid has been necessary to ensure that these export specifications are being met. These specifications have again been met for lots tested this month.

D. Acetic Acid

1. Acetic Anhydride Content of Acetic Acid (M. Sullivan, C. Agee)

Bimonthly analyses of product acetic acid for acetic anhydride is being conducted in order to establish the level typically present. Analyses for the months of November and December average 0.028% acetic anhydride in the acid, above the E. M. Science spec of 0.01%. Further testing will continue.

2. Conformance of Acetic Acid to USP Specifications (M. Sullivan)

Product acetic acid has been checked for conformance to USP specifications and found to pass all tests.

E. Utilities

1. Waste Treatment - Emulsions Pretreatment Bypass Project (C. Riley)

A meeting to discuss the emulsions pretreatment bypass test and analytical manpower support was held with K. B. Synder from ICT. A list of analyses that would be required was compiled. All test work except BOD measurements will be performed by ICT. Suspended solids and TOC analysis will be done in the main lab on the after-noon shift. This will prevent interferences with routine analyses. This project is scheduled to begin 09 January 1984 and run for 6 weeks. The waste treatment lab personnel will work closely with the ICT group on this project.

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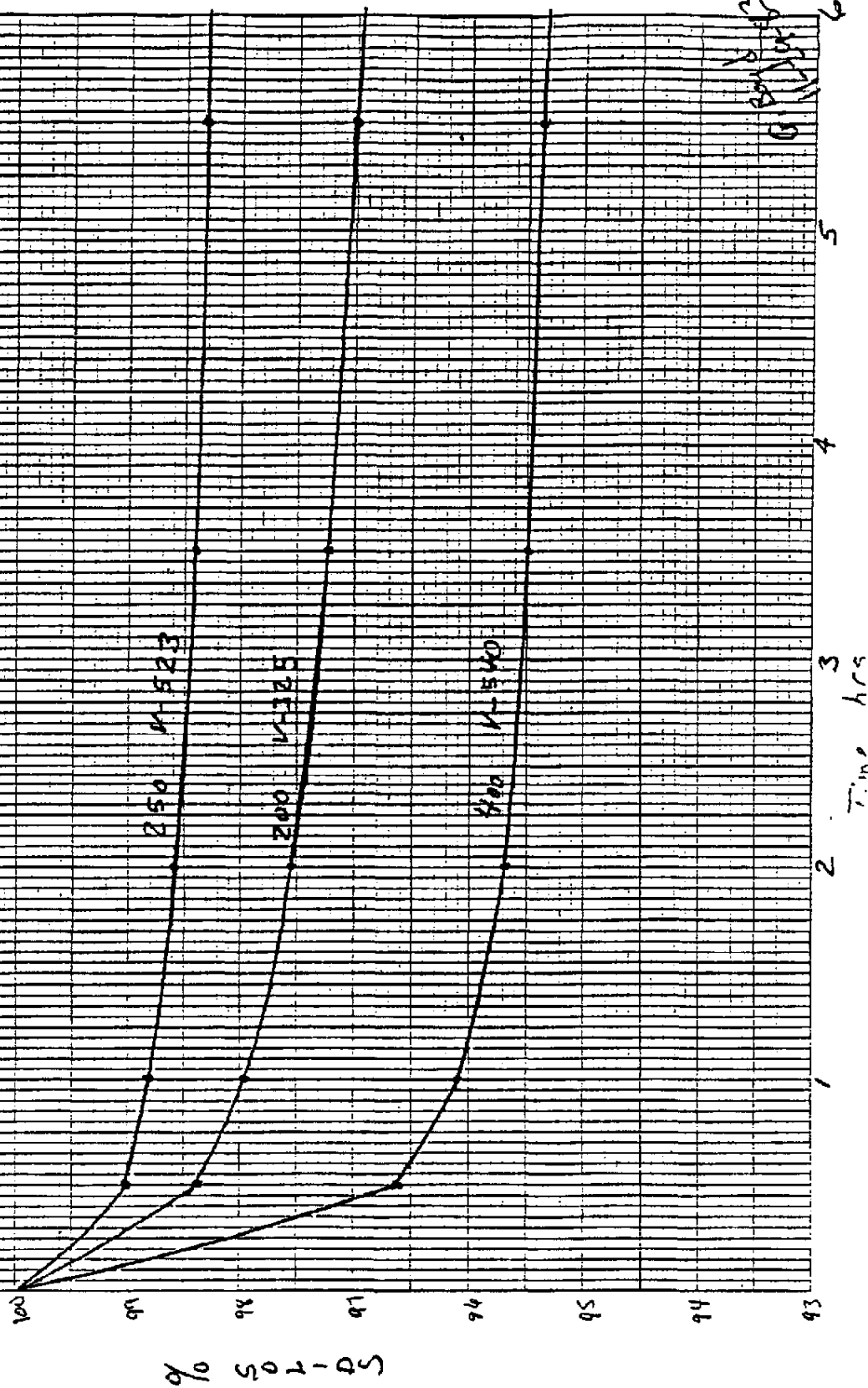
% Solids vs Time on Dryer Discharge Samples

8800 - 11-28-83

Analysed 11-28-83

Dried at 105°C

Figure 1



8800
11-28-83

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III. QUALITY CONTROL LABORATORY

A. Acetylenics

1. Surfynol 104A lots 13103 and 13233 retain samples were analyzed for specific gravity at the request of B. S. Melnick. The specific gravity values were within the 0.866-0.872 range that was previously published by Performance Chemical as typical.
2. Calvert analyses of Surfynol 440 lots I-939-L and I-950-L pre-shipment samples received from Emery failed to comply with the product specifications. Emery's certificates of analysis showed the material to comply to the product specification.

At the direction of B. S. Melnick, a portion of the Emery Surfynol 440 lots preshipment samples were returned to Emery for re-analyses.

B. Polyvinyl Alcohol

1. At the request of the PVOH Business Area, Vinol 523S, lot 090023-T was analyzed for particle size by skid. All of the skid samples particle size analyses complied to the product manufacturing specifications, except for skid 10.
2. Particle size analyses were determined for the "S" grade samples received from Tanner. Vinol 523-S, lot 100733-T samples failed to meet the product manufacturing particle size specifications.

C. Polyvinyl Chloride

A meeting was held with the PVC production personnel to discuss analytical test load. The results of the meeting were to utilize overtime to minimize final product testing backlog and minimal testing reduction.

D. Miscellaneous

1. ORI-SA was conducted for the consolidated Main Laboratory Building. The action items identified will be completed by 13 January 1984.
2. An introductory meeting was held with ICT - Environmental Personnel concerning the Calvert City Emulsion Pretreatment Bypass test. All of the analytical tests except BOD measurements will be performed by ICT personnel. Suspended solids and TOC analyses will be performed by ICT personnel in Calvert laboratory on the afternoon - night shifts. The ICT personnel will be located in the old WT laboratory.

E. Overtime

The quality control and process assistance analysts worked 690.5 hours of overtime during December, 1983. The overtime represents 16.2 percent of the total hours worked by these analysts. Tabulated below is a summary of the overtime.

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<u>Reasons for Overtime</u>	<u>Quality Control</u>	<u>Process Assistance</u>	<u>Totals (hrs)</u>
Illness	28	-	28
Safety/Training	11	-	11
Vacation	372	-	372
Acetylenics	-	11	11
Catalyst	12	-	12
Emulsion	-	6	6
PVC	43	38	81
PVOH	36	46	82
Waste Treatment	-	87.5	87.5
	<u>502</u>	<u>188.5</u>	<u>690.5</u>

IV. SAFETY

There were no first aid cases, OSHA recordables, or time away from work cases for FY84 to date. The laboratory has operated 14 years, 4 months without a time away from work case.

The minimum housekeeping standards for the laboratories were issued to all laboratory employees. Written inspection report results will be issued with copies being sent to the individual laboratory employee responsible for the work area.

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 regularly scheduled December, 1983 safety meetings.

Updated safe work practices for Operation of Wiley Mill - No. 35 and Handling Hot and Cold Equipment - No. 40 were approved and distributed.

V. SAMPLE DEPARTMENT

An audit was performed by Chemicals Customer Service to determine the elapse time for processing sample orders during November, 1983. The audit shows the average time for Polymers Chemicals and Performance Chemicals samples from entry in Trexlertown to shipment from Calvert was 3 days. The average elapse time standard for the Calvert Sample Department is four days maximum.

The Calvert Sample Department completed 448 samples orders during December, 1983. A total of 1,193 separate samples were processed for shipment. The number of samples per storeroom manhour for December, 1983 was 5.7.

The number and percent 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	0.3
Acetylenics	415	34.8

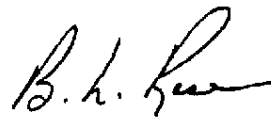
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<u>Product Line</u>	<u>Number of Samples</u>	<u>Percent of Total Samples</u>
Airflex Emulsion	219	18.4
Atmos. Emulsion	107	8.9
Automotive Catalyst	62	5.2
Beads	48	4.0
Polyvinyl Alcohol	179	15.0
Polyvinyl Chloride	62	5.2
Miscellaneous	98	8.2

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>
qts.-----3	4 oz.-----2	qts.-----26
	8 oz.-----403	gals.-----187
	qts.-----9	5 gals.-----6
	gals.-----1	
<u>Atmos. Emulsion</u>	<u>Auto Catalyst</u>	<u>Beads</u>
qts.-----6	4 oz.-----20	lbs.-----48
gals.-----94	8 oz.-----4	
5 gals.-----7	qts.-----19	
	gals.-----9	
	300cc-----10	
<u>Polyvinyl Alcohol</u>	<u>Polyvinyl Chloride</u>	<u>Miscellaneous</u>
1/2 lb.-----14	1bs.-----57	98 samples
1bs.-----143	3 lbs.-----4	
5 lbs.-----17	1000 lbs.-----1	
50 lbs.-----5		


B. L. Rose

BLR:d11

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