



Air Products and Chemicals
INC.

CALVERT CITY PLANT
INTEROFFICE MEMORANDUM

To: C. L. Cook

Date: 08 November 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
OCTOBER 1984

I. INDUSTRIAL HYGIENE/ENVIRONMENTAL

A. Industrial Hygiene

1. Emulsion - Industrial Hygiene Program (C. Riley)

R. DeBernardi and C. Riley met with J. F. Marschhauser to discuss the industrial hygiene program for the emulsion plant. Areas of excessive noise levels were identified. The most effective means of posting the problem areas were discussed and signs are to be ordered. A schedule for monitoring vinyl chloride was approved. Marschhauser suggested that it may be wise to increase the amount of vinyl acetate monitoring. He is reviewing the FY84 monitoring schedule. A meeting to draw up the FY85 schedule will be held in the near future.

2. Emulsion, General - Acrylamide Contamination of Instrument Air (C. Agee, G. Boyd, C. Riley)

As a result of the October 16 power outage, instrument air at the Emulsion plant was contaminated with acrylamide. The instrument air at Acetylenics, Emulsion, and PVOH was analyzed and a trace of acrylamide was found at Emulsion. It was undetected at Acetylenics and PVOH. Presently, another source of breathing air is being used at the Emulsion plant.

3. PVOH - Industrial Hygiene Program (C. Riley)

R. DeBernardi and C. Riley met with S. Portner to discuss the industrial hygiene program for PVOH. Areas with excessive noise levels were identified and designated to be posted. Signs have been ordered. Also a schedule for monitoring methanol and methyl acetate was approved and the tests have been completed. Of the four operators monitored, two were exposed to excessive concentrations of both methanol and methyl acetate.

4. PVC - Noise Survey (C. Riley)

The noise survey for the PVC plant has been completed except for the silo area. Several areas of excessive noise levels were located. Those employees who are exposed to excessive noise levels will be identified when the survey is completed.

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

PVC operating personnel were monitored for vinyl chloride monomer exposure with each shift being sampled for 15 minute and 8 hour TWA exposures.

6. Catalyst - Industrial Hygiene Program (C. Riley)

J. Santa Cruz submitted the Catalyst Plant Industrial Hygiene Program for FY85. It was reviewed and will soon be completed.

B. Environmental

1. APG Environmental Testing Proficiency Program (V. Cates, C. Riley)

All parameters for the September APG environmental testing proficiency program were within one standard deviation of the mean.

II. SAFETY

There were no first aid, OSHA recordables or time away from work cases during October 1984. The laboratory has operated 15 years, 1 month without a time away from work case.

Two safety meetings and a safety committee meeting were held as scheduled. Make-up meetings will be scheduled for those employees that were absent during the regularly scheduled October 1984 safety meetings. Laboratory safety meeting attendance for FY84 was 100 percent.

Updated laboratory safe work practices for Determination of Acetic Acid in Vinyl Acetate - No. 34 and Preparation of 10 Percent Silver Nitrate Solution - No. 37.

III. PERSONNEL & TRAINING

Critical Skills Development Program "Communicating with Confidence" was attended by B. L. Rose, Carol Smith, B. D. Bazzell, and M. H. Stermon.

Service anniversary dinners were held to honor Joe Peavler and Charles Hallmark for twenty-five years of continuous service.

The quality control and process assistance analysts worked 763 hours of overtime during October 1984. The overtime represents 13.8 percent of the total hours worked by these analysts. Tabulated below is a summary of the overtime:

<u>Reason for Overtime</u>	<u>Quality Control</u>	<u>Process Assistance</u>	<u>Total (Hrs.)</u>
Illness	144	0	144
Vacation	312	0	312
Safety/Training	21	0	21
Floating Holiday	40	0	40

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<u>Reason for Overtime</u>	<u>Quality Control</u>	<u>Process Assistance</u>	<u>Total (Hrs.)</u>
Acetic Acid	0	4	4
Acetylenics	0	0	0
Emulsion	8	8	16
PVC	96	5	101
PVOH	41	8	49
Waste Treatment	0	76	76
	662	101	763

IV. PROCESS ASSISTANCE

A. Emulsion

1. Standardizing Laboratory Methods for Emulsion (C. Riley)

A response to the CRSD Emulsion methods manual has been issued. A task force to resolve the problems has been formed and will meet on 09 November 1984.

2. Cloud Point Procedure for Igepal CO-730 (M. Sullivan, C. Agee)

GAF, our supplier of Igepals, recently advised that they are changing the test procedure, and therefore the specification, for cloud point of Igepal CO-730. To determine that this revised procedure and specification would be equivalent, the cloud point of a sample of CO-730 was determined via both procedures. The material was found to be within both the old and new specifications. More data showing equivalency is being requested from GAF prior to changing our specifications to incorporate this new method.

3. Fisheye Procedure (Shift Technicians, C. Riley)

The #24 wound wire for the fisheye count was received and is being utilized. The tentative fisheye procedure is being followed.

4. Cenco Solids (C. Riley)

Analysis of the quality assurance results for the Cenco solids confirms that the analysts are as precise and accurate as the instrument design allows. The standard deviation for balance #1 and #2 is $\pm 0.2\%$ while the standard deviation for balance #3 is $\pm 0.3\%$.

5. Quality Assurance Program (Shift Technicians, C. Riley)

The downtime was utilized to analyze the QA samples - quarterly final lot sample, quarterly Cenco solids sample, monthly viscosity standards. The results on the viscosity standards were within acceptable limits. The Cenco solids results were slightly more erratic than normal because the sample was old. Enough data points have not been collected to establish upper and lower limits for the final product.

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B. Polyvinyl Alcohol

1. Organic Emissions from Dryer Scrubber Vent Gas (G. Boyd, J. Joyce, D. Lawson)

Laboratory support was provided for the 23 October 1984 state audit of the dryer scrubber vent gas discharge to the atmosphere. This support included triplicate sampling and analysis of the 200, 250, and 400 lines. A report containing the analytical procedure, sampling procedure, results, and GC scans was issued to L. C. Toney. The purpose of the audit was to demonstrate compliance after construction of the turbo dryers.

2. Color Analysis of V-325 Dryer Feed and Dryer Discharge (G. Boyd)

As requested by S. G. Eason, 4% solutions of V-325 dryer feed and V-325 dryer discharge were analyzed for color. The results are:

- Dryer feed, 10/2/84, 1400 - Duplicate analyses, 8.2, 8.4
- Dryer discharge, 10/2/84, 0001 - 11.0
- Dryer discharge, 10/2/84, 0800 - 12.4
- Dryer discharge, 10/3/84, 0001 - 11.5

The purpose of the work was to determine the turbo dryer's impact on color.

3. Volatiles Composition of V-325 (G. Boyd)

As requested by S. W. Portner, volatiles composition analyses were performed on two V-325 lots: one produced with "normal" slurry tank water, and one produced with "high" slurry tank water. The % water of the lot produced with "high" slurry tank water was slightly higher than that of the lot produced during "normal" slurry tank water conditions.

4. V-523 Grinding Trial (G. Boyd, QC Technicians)

Laboratory support was provided to PCT the week of October 08 for a V-523 grinding trial. The objective of the trial was to determine the effect of screening before grinding on the particle size distribution. The analyses required were 168 PSDs, 42 % solids, and 21 bulk densities. During the test trial % fines through the 100 mesh screen were effectively reduced.

5. Pure Acid Column Corrosion Monitoring (M. Sullivan, C. Agee)

Chloride concentrations in 5304 pure acid column tray 27 samples remained low (<10 ppm) much of the month with purging of the column in effect. The 5304 btms samples were heavily fouled with iron, etc. (black in color) until the sludge still was cleaned out mid-month. Since this the btms samples have shown much less

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corrosion. Several acid/water splitter condenser samples were also analyzed for chloride, but no condenser leak was indicated. Inspection and testing of this condenser during downtime proved this to be the case.

C. Acetylenics

1. 104 Intermediates Processing - Reilly Tar (C. Agee, J. Joyce)

Various steps have been taken in preparation for bulk shipments of 104 intermediates to Reilly Tar & Chemical for processing:

- 1) A standard method for analysis of peroxide content of 104 intermediates samples has been agreed upon by Reilly Tar, CRSO, and Calvert labs with round robin testing performed on one sample.
- 2) Various storage tanks of intermediates are being washed in the plant with a solution of sodium metabisulfite, a strong reducing agent, to reduce peroxide content to a level acceptable by Reilly Tar. Testing of samples to date has shown all peroxides to be reduced. A titration to determine excess reducing agent in the washed samples is being investigated.
- 3) Prior to wide-scale use, hydroquinone, an inhibitor, was added to an intermediate sample and its GC composition determined before and after to assess any effect that the HQ addition might have.
- 4) Future plans are to assure manufacturing's compliance with APCI's Toller QA Guideline for Reilly Tar, certifying their lab for analysis of these samples.

2. Proposed Surfynol PC Manufacturing Procedure (C. Agee)

The analytical section of a proposed manufacturing procedure and specs revision for Surfynol PC was evaluated. The procedure proposed dictates much more analytical testing of raw materials than is presently in effect, in addition to in-process testing. These comments were made and the impact roughly quantified in a memo to C. L. Cook.

3. Off-Spec Surfynol 465/485 Preshipment Samples (M. Sullivan, C. Agee)

Our quality control tests showed two Emery Surfynol preshipment samples (one lot of Surfynol 465, lot #1X-12364-2844 and one lot of Surfynol 485, lot #1X-12354-2854) to be out-of-specification for various properties (namely pH, color, and cloud point for 465 and color for 485). Portions of our preshipment samples were sent back to Emery for their analysis and they also showed the 465 sample to be out-of-spec. Emery plans to blend and treat the material to meet specifications.

4. Cloud Point Procedure for Igepal CO-730 (M. Sullivan, C. Agee)

See Section IV, A.2.

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D. Polyvinyl Chloride

1. Support During DMF Reactor Cleaning (D. Lawson, J. Joyce)

Numerous samples - reactor rinse water, condensate, caustic tank, blowdown tank, others - were analyzed for DMF content during recent DMF cleanings of reactors. This was necessary in order to determine if reactors had been sufficiently rinsed following cleaning.

2. Assay of PVC Catalysts (D. Lawson)

As requested by B. Gebbia, two samples of PVC catalyst, one of EHP 333 (2-ethyl hexyl peroxydicarbonate) and one of Lupersol 188 (cumyl peroxyneodecanoate), were analyzed for active oxygen/assay. Both catalysts were found to be within specifications for this property.

3. BHT Solubility Testing (J. Joyce, C. Agee)

As requested by T. Garrisi, an experiment was conducted to determine solubility of BHT in various liquids, namely water, methanol, methyl ethyl ketone, and acetone. The material was found to be insoluble in water, somewhat soluble in methanol, and freely soluble in methyl ethyl ketone and acetone. This work was required to determine which solvent would be best suited as a medium for BHT addition in a liquid state.

4. Glycomul L Test (J. Joyce)

C. Grey asked that we screen the effect, if any, that addition of Glycomul L (a potential anti-stat ingredient) to PVC resin might have on the resin's heat stability. Glycomul L was sprayed onto PVC wet cake at a 1% level. The resin was then dried and thermal stability judged via the pressout procedure. No significant differences were discernible between the Glycomul treated resin and the control resin containing no Glycomul.

E. Acetic Acid

1. Publicker Acetic Acid Customer Complaint (M. Sullivan, C. Agee)

Three Publicker acetic acid samples which were the subject of a customer complaint were analyzed for non-volatile residue and assay. The 56% HAc samples (order J8D-28167 Matlack trailer #BL-468102) were found to be 57.7% acetic acid but contained 400-500 ppm residue. Non-volatile residue from a third sample (#28 dated 9/24/84) was isolated and an infrared spectrum made in an attempt to identify the contaminant. This material had an IR scan indicative of polyvinyl acetate, e.g. an Airflex 105 or 728 emulsion. Results were reported in a memo to W. Smith.

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V. QUALITY CONTROL LABORATORY

A. Polyvinyl Alcohol

1. Briggs Manufacturing Company complained that when Vinol 523, lot 02080014 was mixed with CMC, the resulting mixture was very thick, and different from other lots of Vinol 523. Hydrolysis, viscosity and pH were determined for the sample received from Briggs and the analyses met the Vinol 523 manufacturing specification.
2. At the request of D. M. Rogers, the Vinol 325, lot 04050264 retain sample was retested for hydrolysis and viscosity to assist in evaluating the Ajax Adhesives' customer complaint. The retest analyses, as the original analyses, complied to the product manufacturing specifications.
3. A sample of Vinol 205, lot 01100433 was returned from General Electric to evaluate the customer's complaint. The returned sample met all the product manufacturing specifications except for hydrolysis and viscosity.
4. Vinol MM-510G, lot 0110073A batch samples 1, 2, and 3 were returned from ABCO for compliance testing to the product manufacturing specifications. Each of the batch samples failed to meet the product manufacturing specifications for one or more parameters.
5. A sample of Vinol 425, lot 02010724 was returned from West Point Pepperell to evaluate the customer's complaint. The returned sample met all the product manufacturing specifications except for viscosity.

B. Polyvinyl Chloride

Heat stability test data were generated for the 1230P resin shipment samples returned by A. J. Mason from J-M Manufacturing, Wilton, Iowa. The returned samples test data show the two (2) Calvert railcar shipments to comply with the proposed heat stability specifications as measured by the neat resin pressout procedure. The data also showed each of the four (4) Pace shipments to fail to comply with the proposed heat stability specifications.

VI. SAMPLE DEPARTMENT

Statistical summary for FY84 sample shipments distributed by Customer Services shows the Calvert Sample Department processed 5,268 sample orders in average 2.1 days turnaround. The Customer Services computer entry system only accounts for approximately 80 percent of the Calvert sample shipments. An additional 1,217 sample orders were processed by the Calvert Sample Department in FY84 that were not processed through the Customer Services sample system.

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The audit performed by Chemicals Group Customer Service to determine the elapse time for processing sample orders during September 1984 shows the average elapse time from entry in Trexlertown to shipment from Calvert is 1.9 days for Specialty Additives samples, 2.3 days for Polymers samples and 3.3 days for Industrial Chemicals samples.

The Calvert Sample Department completed 655 sample orders during October, 1984. A total of 1705 separate samples were processed for shipment. The number of samples per storeroom manhour for October is 5.1. The number and percentage of samples shipped by product line are listed below:

<u>Product Line</u>	<u>No. of Samples</u>	<u>Percent of Total Samples</u>
Acetic Acid	6	0.4
Acetylenics	590	34.6
Airflex Emulsion	307	18.0
Atmos. Emulsion	272	15.9
Automotive Catalyst	11	0.6
Beads and Powders	71	4.2
Polyvinyl Alcohol	371	21.8
Polyvinyl Chloride	7	0.4
Miscellaneous	70	4.1

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	4 oz.-----1	16 oz.-----36
qts.-----5	8 oz.-----583	qts.-----18
	qts.-----6	gals.-----251
		5 gals.-----2
<u>Atmos. Emulsion</u>	<u>Automotive Catalyst</u>	<u>Beads & Powders</u>
16 oz.-----84	4 oz.-----4	1bs.-----62
qts.-----4	qts.-----2	5 lbs.-----9
gals.-----180	gals.-----2	
5 gals.-----4		
<u>Polyvinyl Alcohol</u>	<u>Polyvinyl Chloride</u>	<u>Miscellaneous</u>
1bs.-----350	1bs.-----2	70 samples
5 lbs.-----18	5 lbs.-----3	
10 lbs.-----3	10 lbs.-----2	

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