



CALVERT CITY PLANT
INTEROFFICE MEMORANDUM

To: T. C. Noll
Date: 07 May 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
APRIL, 1984

I. INDUSTRIAL HYGIENE

A. Catalyst Plant - Acid Monitoring (G. Boyd)

Air sampling was conducted at the catalyst plant while operators were charging hydrochloric acid to a solution tank. The results indicate that during normal working conditions, operator exposure is well below the ceiling limit of 5 ppm for HCl. An exhaust system at the tank has helped reduce exposure while charging the acid.

B. Utilities - Noise Monitoring (C. Riley)

Annual noise monitoring in the utilities area has been completed. From the data accumulated a sign will be posted in the air compressor building stating that ear protection is required if one works over three hours in that building. A detailed report will be issued at a later date.

C. PVC - Vinyl Chloride Personnel Monitoring (J. Joyce)

Personnel monitoring of PVC operators for vinyl chloride was conducted with each shift being tested during the month. Both 8 hour and 15 minute TWA exposure measurements were taken.

II. PROCESS ASSISTANCE

A. Emulsion

I. DTA Equivalency Established (J. Joyce, C. Agee, C. Riley)

Sufficient data has been collected to allow for the establishment of equivalency between DTA results determined by the main lab with Series 990 unit and only one analyst vs. those results from the emulsion lab with Series 99 instrument and four analysts. The interlab standard deviation of 0.83 fell within the guideline of 1.0°C given by ASTM procedure D-3418, "Transition Temperatures of Polymers by Thermal Analysis". Emulsion DTA results are now being reported from the emulsion laboratory with out-of-spec results being confirmed by the main lab.

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2. Raw Materials Quality Assurance Program (C. Riley)

The Emulsions Plant Raw Materials Quality Assurance Program was approved and distributed. The program was implemented 01 May 1984.

3. Control Charts (C. Riley)

Control charts for solids, viscosity and grits for A-400, A-400H, and A-300 were initiated. The control charts will be maintained by the laboratory chemist until emulsion plant personnel can assume the responsibilities.

4. SVS Monomer Analysis (C. Agee)

A shipment of SVS monomer was received by the emulsion plant with no lot number or accompanying certificate of analysis. A sample of this material was analyzed for all specification properties. The SVS assay was slightly low for this sample while methanol insolubles were ~2 times the maximum allowed by specifications.

B. Polyvinyl Alcohol

1. Comparison of Old and New Amberlite IR 118 Resin (G. Boyd)

A series of tests were conducted to compare the particle size and catalytic activity of the Amberlite IR 118 ion exchange resin used presently by PVOH plant versus a new ion exchange resin. The results indicate that the old resin is slightly smaller in particle size and is slightly lower in catalytic activity. At room temperature, the reaction rate of $\text{MeAc} + \text{H}_2\text{O} \rightarrow \text{HAc} + \text{MeOH}$ was higher with the new resin. At increased temperature, there was no difference in reaction rate using the old vs. the new resin. Therefore, replacing the old resin with new would not effect an increase in conversion at the plant.

2. Revision of Acetic Acid's Permanganate Test to Reduce Analysis Time (G. Boyd)

A revised method has been proposed for running the acetic acid permanganate time test for detection of oxidizable substances in the acid. This method has been developed to reduce the present two hour test to 30 minutes. This method will be instituted in June for process run-down tanks and will enable quicker transfer of acid to the storage tanks.

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

As requested by S. Crowley, 13 particle size distribution and percent solids analyses were performed on various samples from the grinding area. The work is required to define the grinder and screening efficiencies as part of an on-going project to de-bottleneck the area.

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4. Pure Acid Column Corrosion Problems (M. Sullivan, C. Agee)

Corrosion problems once again surfaced in the 5304 pure acid column. Various samples - the 5304 condenser outlet, 5308 acid splitter reflux and btms, the high color rework acid, and DM water used in making dilute acid shipments - were analyzed for chloride in an attempt to find the problem. The rework acid itself was found to contain 0.2-0.5 ppm Cl^- and was considered suspect. Rework of the acid was finished by month's end with Tray 27 Cl^- concentrations and btms iron concentrations decreasing.

C. Acetylenics

1. Flash Point of Hexynol Containing Isopropyl Ether (J. Joyce)

As requested by T. Noll, isopropyl ether was added in small increments to Hexynol lot #23320 until a flash point of 100°F was reached. While the original sample contained no IPE and had a flash point of 125°F (closed cup), addition of 2.1% IPE yielded material with a flash point of 100°F.

2. Color Stability of Dimethyl Hexynediol with Nitrogen Blanket
(C. Agee, J. Joyce)

As suggested by J. Pervier, an experiment was conducted to determine if nitrogen blanketing of molten DH would prevent color formation in the product. A relatively pure nitrogen blanket was maintained during the study as DH was held in the molten state at 110°C for ~6 hours. For comparison purposes an air blanket was maintained over a second control set of samples which were likewise heated. It was found, contrary to prior belief, that the nitrogen atmosphere seemed to have a detrimental effect on color compared to an air atmosphere (Color N_2 :Air = 1800:1000 APHA units).

3. Acetylenics Raw Materials Quality Assurance Program (C. Agee)

The Acetylenics Raw Materials Quality Assurance Program was completed and is circulating for approval. This program will be initiated next month pending approval. Most laboratory procedures outlined in the program are presently in effect.

4. Acetylenics Special Products/Blend Analyses (M. Sullivan, J. Joyce)

Numerous special products and blends, e.g. Surfynol D-204, Ancor 771, SVS monomer, Surfynol 465/485, etc., have required the support of the PA group in analysis. These samples require special testing not routinely performed.

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D. Polyvinyl Chloride

1. Sigma 2000 Analyzer Set-Up (D. Lawson)

Set-up of the new PE Sigma 2000 analyzer for residual vinyl chloride analysis of PVC slurries, resin, and water samples has been accomplished. EPA method 107 has been followed as the guideline and the appropriate instrument conditions necessary to conform with the standard procedure determined. Effective next month RVCN analyses of all PVC samples will be conducted with this instrument rather than the manual procedure.

E. Acetic Acid

1. Acetic Acid Investigation - Renal Systems (C. Agee, M. Sullivan)

Renal Systems, a drum customer of Aspen Chemical, complained of iron, aldehydes, and non-volatile matter in a recent acetic acid shipment. The retain sample from the date of shipment to Aspen and a sample from Renal were analyzed for these properties. While the retain sample was in-spec for these properties, the sample from Renal was out-of-spec on iron and high in non-volatiles. This would tend to support the belief that the contamination problem occurred at Aspen Chemical.

F. Utilities

1. Waste Treatment

a. Quarterly Independent Laboratory Testing (V. Cates, C. Riley)

The results of the quarterly independent laboratory check with McCoy & McCoy, Madisonville, Kentucky were extremely close to the APCI test results. A standard from Alpha-Trol was included with the samples to McCoy & McCoy. Also the APG environmental proficiency testing results were within the standard deviation for each test.

b. Warburg Studies (C. Riley)

Warburg studies were conducted on 1192 polymer used in waste treatment and a rinse water solution from the catalyst produced by Degussa. Neither were toxic to the system nor readily biodegradable. Extremely high concentrations of polymer (400 ppm) did show a definite increase in oxygen uptake after 172 hours.

2. Water Insolubles in Salt (M. Sullivan)

Sodium chloride from utilities, used in making up brine solution, was analyzed for solids which would not dissolve in an excess of water. Water insolubles constituted approximately 0.77% of the salt.

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G. Miscellaneous

1. Statistical Quality Control Course (PA Chemists)

Process assistance chemists attended a 16-hour in-house course on statistical quality control conducted by MIS and QA personnel. The basic types of control charts, their use, interpretation, and benefits were presented as well as other statistical methods of analysis. $\bar{x}$ and R charts will be maintained by PA chemists for the laboratory PVOH, PVC, acetylenics, and emulsion standards analyses.

III. QUALITY CONTROL LABORATORY

A. Acetic Acid

At the request of the acetic acid quality board, area commercial analytical laboratories were surveyed to determine their trace metals analyses capabilities. McCoy & McCoy and Aware laboratories will submit written cost and timing estimates for determining the 21 trace elements as indicated on the SEMI acetic acid specifications.

B. Miscellaneous

1. Laboratory analyst, Vernon Cates, was nominated for the Air Products Community Service Award. Mr. Cates' participation in and contribution to the Sedalia, Kentucky Volunteer Fire Department was the basis for the nomination.
2. ORI was conducted for the laboratory sidewalk construction project. There were no hazards or action items identified.

C. Overtime

The quality control and process assistance analysts worked 515.5 hours of overtime during April, 1984. The overtime represents 11.4 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>Totals (Hrs.)</u>
Illness	28	0	28
Excused Absence	9	0	9
Safety/Training	8	0	8
Vacation	148	0	148
Floating Holiday	8	0	8
Acetylenics	6	2	8
Catalyst	2	3	5
Emulsion	1	0	1
PVC	81.5	49.5	131
PVOH	91	23.5	114.5
Waste Treatment	0	55	55
	382.5	133.0	515.0

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IV. SAFETY

There were no first aid cases in April, 1984; one first aid case in FY84. There were no OSHA recordables or time away from work cases for FY84 to date. The laboratory has operated 14 years, 8 months without a time away from work case.

Laboratory policy was established which states there must be an approved safe work procedure and employee training before any hazardous sampling can be performed by laboratory personnel. Hazardous sampling is determined by the nature of the sample material and/or the location of the sample point.

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 April, 1984 safety meeting.

Updated safe work practices for Preparation of Reagents Which Require Strong Acids or Alkalies - No. 14A and Using Vacuum Pumps - No. 18 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 March, 1984. The audit shows the average elapse time from entry in Trexlertown to shipment from Calvert is 1.2 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 575 sample orders during April, 1984. A total of 1,283 separate samples were processed for shipment. The number of samples per storeroom manhour for April was 4.9. 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	6	0.5
Acetylenics	454	35.4
Airflex Emulsion	274	21.3
Atmos. Emulsion	138	10.7
Automotive Catalyst	10	0.8
Beads	141	11.0
Polyvinyl Alcohol	187	14.6
Polyvinyl Chloride	1	0.1
Miscellaneous	72	5.6

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

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<u>Acetic Acid</u>	<u>Acetylenics</u>	<u>Airflex Emulsion</u>
pts.-----4	4 oz.-----1	qts.-----6
qts.-----2	8 oz.-----452	gals.-----266
	qts.-----1	5 gals.-----2
<u>Atmos. Emulsion</u>	<u>Automotive Catalyst</u>	<u>Beads</u>
qts.-----7	4 oz.-----4	lbs.-----140
gals.-----124	qts.-----2	10 lbs.-----1
5 gals.-----7	gals.-----2	
	300cc-----2	
<u>Polyvinyl Alcohol</u>	<u>Polyvinyl Chloride</u>	<u>Miscellaneous</u>
lbs.-----186	5 lbs.-----1	72 samples
50 lbs.-----1		

B. L. Rose

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