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CEL-1

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: R. R. Graham

From: G. D. Boyd GDB-97-81 February 5, 1981

Laboratory Department Summary
January, 1981

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January, 1981

A summary of each Group's Monthly Report is attached. Section I contains a summary of the Quality Assurance and Analytical Group activities with tables of analytical statistics, shipments, quality waivers, quality complaints and sample shipping statistics. Section II describes the status of Special Problems Groups and Staff Chemist projects by chemist assignment. A table listing the priorities, justifications, and estimated completion dates of current Special Problems projects is also included in Section II.

G D Boyd
G. D. Boyd

GDB:jr

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HIGHLIGHTS

- The retention times for approximately 70 Bishop Plant products and by-products have been determined by capillary GC. This work will make it possible to trace specific effluent components from their specific units to their disappearance through the BOD Unit.

- Evaluation of a new method for reactivating silver catalyst using electrolysis and ultrasonic cleaning has begun.

- Front ditch high COD alarms were caused by chloride interference rather than organic pollutants. A modified high salinity COD method will be used for high chloride samples in future laboratory analyses.

- An improved analytical method for Oxo Catalyst solution has been developed. The new method is more reliable, reduces mantime, and uses only one GC rather than two as with the old method.

- Information was generated to initiate and support process modifications to reduce the dissolved fuel gas concentration in Monsanto Butyraldehyde.

- The PE formal strong acid-resin catalyzed hydrolysis system passed 188 days (6.3 months) of exposure of the resin to reaction conditions. The resin activity is the same as fresh resin and there has been no detectable activity loss since reactivation at day 120.

- Extraction system on PE-AML to VCI demonstrated <3% organics (on a contained salt plus organics basis) in the NaFo stream (raffinate). NaFo levels in the organic extract still higher than target.

- UV absorbance at 220 nm PE quality problem may be related to impurities in HCHO feed. Need more data to confirm.

I. ANALYTICAL GROUP

Competitive Products

Two products were analyzed during January. One was Pentaerythritol Pure grade from an unknown Chinese producer. The other was Diacetone Alcohol from Union Carbide. A report has been issued.

Methanol Synthesis (MS)

Methanol - The Analytical Group is providing special analyses for the MS unit during the conversion of "B" train to single column purification. "A" train is already operating on single column purification and no major problems are anticipated. Support will continue as long as necessary.

Methanol Oxidation (MO) and Formaldehyde (HCHO) Blending

Analytical coverage is being provided for attempts to load and ship unstabilized 50% HCHO prior to installation of required facilities. Customer (Badische) iterim specification equals <2 ppm stabilizer with ultimate specification requirement to be "none by test". First attempt of 1-22-81 failed with stabilizer showing in both tank and trailers. Second attempt of 1-27-81 successful with two trailer loads (approximately 10M gal.) shipping at <2 ppm stabilizer. Permanent facilities of segregated tankage, pumps and liner is scheduled for completion in June of this year.

Polyol

Pentaerythritol (PE) - A new Gas Chromatographic (GC) method has been developed to determine the composition of the PE product. This method has been thoroughly evaluated in the laboratory and provides more accurate data than the previously used GC method. On Monday, 1-26-81, this method was instituted as the routine means for analyzing PE by GC.

Di-Acetone Alcohol Unit - A barge load of Shell Chemical Co., Houston, Texas, acetone was received and evaluated the week of 1-11-81. All quality points were met and Shell has been added to our list of approved vendors. Shell product was purchased following Carbide notification that they could no longer meet our purchase specifications.

Utilities

Trailer load receipts of 93% Sulfuric Acid from Asarco of Corpus Christi were received and evaluated the week of 1-6-81. The acid met all quality points checked and Asarco has been added to our approved Vendor list. The use of 93% H_2SO_4 versus our usual 98% prevents acid freeze-up during winter.

Special Analytical Assignments

Effluent and BOD Component Identification (F. R. Dow, B. W. Wise) - Analyses of effluent and BOD streams with an OV-101 capillary column shows that components identification is possible and the components can be traced from a specific unit to their disappearance in the BOD system. The retention times for approximately 70 compounds, characteristic of Chemcel products and by-products, have been determined with this GC method.

Future work will include monitoring effluent components across BOD II.

Lube Grade PE (F. R. Dow, B. W. Wise) - Samples of Tech PE were provided to Emery and Hatco for comparative analysis. Emery and Hatco both reported approximately 8% more Di-PE than Bishop had determined. Hatco and Bishop agreed on the Tri-PE concentrations, but Hatco reported approximately 25% more tri-PE than Bishop.

A letter will be written to report the details of this sample exchange.

Amines in Competitive MeOH (J. E. Russell) - The levels of amines in MeOH were determined by ion chromatography for various MeOH producers including Celanese Bishop and Clear Lake. A letter has been submitted documenting the results.

TDWR, ECD, Government-Required Analytical (J. E. Russell) - A letter was submitted documenting the results of the tests for the four characteristics of hazardous waste for VCI Centrate and Pond 13E soil. Both proved non-harzadous.

Methods and sampling routines are being prepared to implement a study of inorganics in the groundwater as part of the TDWR ECD. The data that is gathered, in conjunction with data on organics, may determine if biological activity is degrading the contaminants presently in the groundwater. This is a long term study and will require entensive use of the ion chromatograph.

Quality Waivers

None This month.

Note: As per Company Quality Assurance request, to better measure the effectiveness of Plant quality control systems, a new method of reporting quality waivers will be instituted this year. The new method will key on deviation from Manufacturing Control Specifications (quality deviation - QD) and deviation from Sales Specifications (significant quality deviation - SQD). As a product is shipped on quality waiver, either QD or SQD, the pounds of that product shipped will be expressed as a percentage of the total pounds of that product shipped for the month and for the year to date. For example:

QD - Quality deviation - from Manufacturing Control Specifications (MCS) =

$$\frac{\text{lbs. product shipped outside MCS}}{\text{Total lbs. product shipped}} \times 100$$

SQD - Significant quality deviation - from Sales Specifications (SS) =

$$\frac{\text{lbs. product shipped outside SS}}{\text{Total lbs. product shipped}} \times 100$$

There were no products shipped under waiver this month. The proper calculated percentage will be added to this report for each product as the occurrence arises. Additionally, each waiver will continue to be individually delineated in the monthly report.

Customer Complaints

1-19-81 - 3M Co. - Newark, N.J. - Complained of lumpy Paraform Flake in Lot 80-318 received from the Newark Terminal. 3M also complained of Newark replacement Lot 80-334. Original and retain sample analysis show that a lump free product was shipped. Company QA investigation revealed that the customer desires a product that totally passes a 1/4" screen and was not referring only to lumps. Our typical Para Flake does not meet this requirement and the customer was referred to our 95% Para Flake (a smaller flake) or to our 92-96% Para Coarse Powder product.

ANALYTICAL STATISTICS

	Jan.	Jan. YTD	Previous YTD
Sample Count			
Chemical Section	4806	4806	5704
Instrument Section	2403	2403	3000
Total	7209	7209	8704
Technician Overtime			
Chemical Section	161	161	26
Instrument Section	32	32	62
Total	193	193	88

Sample Shipping and Quality Assurance Statistics

Statistics For January 1981

Sample Shipping	Month	YTD	Previous YTD
Domestic	60	60	56
Export	2	2	4
Plastics	0	0	1
Other	3	3	10
Total	65	65	71

Quality Assurance Statistics

	Month	YTD	Previous YTD
Shipments (NO.of Orders)	455	455	874
Delayed			
Waviers Internal	0	0	2
External			
Complaints (Total)	1	1	1
Complaints (Legitimate)			
Complaints (Non-Legitimate)			
Complaints - Outstanding (Unresolved)			

Analytical Group General

SNE employee Rey Garcia transferred to the Analytical Group 1-5-81.

C. E. Bull attended the four day, Plant sponsored, Emergency Care Attendant school and received State certification.

II. SPECIAL PROBLEMS GROUP

Process Technology
B. C. Kerr

Maintenance of Technology - A letter has been written recommending that the Plant adopt the Document Retrieval Program, "INQUIRE®", which has been put in service by the Technical Center.

Solar Pond Disposal Project - The ultimate disposal of water from solar ponds may require a substantial reduction in salt content. Laboratory work indicates that one possible method for salt reduction combines a solvent "strike" and extraction with acetone. A laboratory study is underway to establish required operating parameters for this process.

Methanol Oxidation, Formaldehyde Blending
T. A. Jasek

GC/MO Reactor - a newly designed reactor was installed. Some preliminary runs were made to test the reactor. Work is continuing.

MO Silver Catalyst Modifiers - Awaiting Tech Center evaluation of previously produced silver crystals (crystals grown in $HgNO_3$ solution containing 1000 ppm Bi). Once these results have been received, the future course of action will be developed.

Catalyst Revitalization Study - Preliminary work has begun to evaluate process of revitalizing old catalysts without complete regrowing of the crystals. Via electrolysis, the surface of the old catalyst crystals is removed. The remaining catalyst is then cleaned in an ultrasonic cleaner. The cleaned catalyst is returned to the electrolysis cell, but this time, new silver is plated on the catalyst.

MO Unit Support - Laboratory analytical AMR's were performed at MO-III and MO-IV. The results are illustrated in the following table:

Date	MO Unit/Train	AMR Indicated	AMR Analytical
21 Jan 81	III/A	0.78-0.80	1.02
27 Jan 81	III/B	0.96-0.98	1.25
	III/B	0.89-0.91	1.22
28 Jan 81	IV/B	0.96-0.98	0.95

November MO Color Problems - Awaiting analysis of methanol sampled during the color problems in November by GC/MS at CCCTC.

Utilities, BOD

S. G. Smith, P. G. Young, G. R. Stasney

Anaerobic-Aerobic Pilot Studies

Since void volume measurements in December, the filters are now back up to organic loadings of 0.4 lbs COD/ft^3/day with COD removal efficiencies at ~85%. Following aerobic polishing at 3.5 days detention, final outfall COD's are approximately 150 ppm. These systems will be used for studies to develop a treatment system for solar pond organics and for further Plant effluent studies.

Anaerobic Filter Feed COD Variability (Smith) - In order to determine the need for a feed COD shock study, the variability of feed COD fed to the pilot units was analyzed. Analysis showed feed COD day to day variations of 1-10% to be normal, variations of 10-20% not unusual, and a few COD changes of 30-35%. The COD changes of 30-35% usually occurred at constant OL levels; no unusual effects due to these COD changes were observed. It is felt that studying drastic changes in feed components would give more useful information.

Solar Pond Treatability (Young, Stasney)

Experimental tanks #5 (Solar pond diluted with equalization basin only) and #6 (Solar pond diluted with both equalization basin and river water) are now treating solar pond material diluted 1:47. Outfall COD's of the control and experimental tanks increased dramatically during the month to levels of 450 (#4-Control), 900 (#5), and 800 ppm (#6). This increase is felt to be a result of excessively high COD equalization feed, at approximately 10,000 ppm. Since that time feed COD has decreased. Outfall COD's of all 3 tanks have also decreased to 290 (Control), 775 (#5), and 600 ppm (#6).

Elimination, Reduction of Solar Pond Odor (Smith, Jasek)

A report on this project is being written.

Utilities Support (Smith, Harris)

Recent front ditch alarms, due to high COD via the on stream COD analyzer, were not confirmed by high ditch TOC. The problem is believed to have been caused by unusually high Cl^- levels in the front ditch. High Cl^- interferes with the on stream COD analysis. The high Cl^- was apparently from brine regeneration of the zeolite beds in the water softner system combined with low flow in the front ditch. The low flow was because BOD Unit outfall was going to the solar ponds during the problem period.

The ditch COD analyzer Cl^- interference was confirmed in the Laboratory with a high saline COD method.

Methanol Synthesis
J. C. Bustabad

MS-II Unit Support - The breakdown of any residual thiocyanate from the presence of Dow's corrosion inhibitor in the MEA systems is negligible ≤ 1.4 ppm/50 days (12/3/80 - 1/22/81). The thiocyanate levels on 1/22 determined by UV-Visible absorption employing a standard additions method were 1.3 ppm in both the 5MM and shift MEA systems.

Dräger tube monitoring for H_2S in CO_2 from the MEA system of the Girdler reformers continued through the middle of the month, when the function was undertaken by Operations. The H_2S ranges for the 5MM and 2MM systems through mid-January were 2.5 - 3.5 ppm and 1 - 2 ppm, respectively.

Two components (sodium nitrite and sodium molybdate) of the Celanese developed MEA corrosion inhibitor system were charged to the shift and 5MM Girdler MEA systems aon 1/16 and 1/26th, respectively. Laboratory methods being used for determining nitrite and molybdate are satisfactory. The nitrite is determined by UV-Visible absorption employing a standard additions method. The molybdenum is determined by AA.

Vendor quotes from purchasing showed sodium molybdate from other vendors (Alfa Products and Climax Molybdenum Company) to be 1.5 and 4.9 times cheaper respectively than that obtained from the present vendor (J. T. Baker). The laboratory testing of the sodium molybdate from these different vendors is in progress.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - An improved method of analysis for Bishop Oxo Catalyst solution has been developed. The new method is a single analysis for reactor components and has shown good repeatability and accurancy. Advantages include reduction of sample handling (sample contains TPP) and elimination of benzene (Used in the present internal standard method). A letter has been written and work has been started to incorporate the new method as soon as possible. Some results with the new method indicate stable reactor composition for the month n-BuH is 40-41 wt.%, Esters are about 9 wt.%, TPP is 34-35 wt.% and TPPO is 2.5 - 3.0 wt.% overall, accountability is about 95%.

Rhodium Accountability - January rhodium accountability has remained favorable 95+%. Rhodium lost via T-210 residue is about 2 pounds RTD for Run VII.

Improved Rhodium Recovery - TPP and TPPO analysis of extraction are nearly complete. Final rhodium analyses are pending arrival of AA replacement parts. Work Continues.

Oxo Unit Support - Dissolved fuel gas in our BuH has continued to pose unloading problems for Monsanto at Indian Orchard, Massachusetts. At a meeting with the Laboratory, Process Engineering, and Operations it was decided that minimization of fuel gas contact with the BuH was the best solution. A hot butyraldehyde vapor pad on the distillation column OH Receiver and cryogenic nitrogen for inert padding on the rundown tanks were the two suggestions to alleviate the major problems. A letter has been written detailing laboratory work to define sources of fuel gas absorption.

Environmental Health
C. E. Gary, K. V. Parker

Two samples of insulation were tested for asbestos - one positive test. (KVP)

The Plant noise survey is >80% complete. Overall, more of the operating personnel will be included in the hearing conservation program (8 hr TWA ≥ 80 dBA) and required to wear hearing protection (8 hr TWA ≥ 85 dBA) than was expected. (KVP)

The portable building that houses the MO-IV construction team contained 1-1.5 ppm formaldehyde when analyzed by Dräger tubes. (CEG)

The validation of long term Dräger tubes method to determine the carbon monoxide 8 hr TWA is approximately 50% complete. (Completion by 3-1-81. CEG)

Acetates, Paraform, Formcel® Products
D. R. Akin

Unit Support - None provided

Trimethylolpropane
R. L. Day, B. K. Huelle

Instrument Laboratory Standards (Day) - 2-Ethyl-1,3 propanediol is being synthesized in the laboratory to be used as an Instrument Laboratory standard in the analyses of routine TMP samples by GC. The compound is prepared by reacting 2-ethyl diethyl malonate with lithium aluminum hydride in the presence of ether.

Extractor Optimization (Day) - Minor progress was made on the documentation of the T-312 extractor base case data due to priorities.

Effect of Sodium Formate on TMP Decomposition (Huelle) - Decomposition rates have been established for V-747. In addition, studies are now being conducted to establish decomposition rates for V-760 and V-759, first and second stage concentrators.

Diacetone Alcohol

B. K. Huelle

Unit Support (Huelle) - None provided.

Pentaerythritol

D. R. Akin, P. J. Buras, B. K. Huelle

PE Formal Hydrolysis (Akin) - Studies continue on the resin-strong acid hydrolysis system. The system utilizes an ion exchange resin (Rohm and Haas Amberlyst 15) and a strong acid (HFo or H_2SO_4) as co-catalysts of the hydrolysis of PE mono linear formal. The resins exposure time to date is 188 days. No activity loss has been detected since reactivation of the resin after 120 days. In the past, studies were conducted with continuously fed strong acid. An attempt was made to determine if pulse feeding the acid would reduce the quantity of acid required for conversion at a constant residence time and sodium level. Thus far, the conversion of mono linear formals by pulse feeding the acid has been comparable to conversion with continuously fed acid. Work will continue in this area.

Eliminate VCI Centrate Waste Stream (Buras) - Several solvent systems were evaluated in a continuous laboratory extractor with the objective of removing all organics from AML. Using t-butanol or a mixture of methanol in butyraldehyde as solvent (solvent:feed ratio $\sim$ 3:1) an aqueous raffinate containing less than 3% organics (on a contained salt basis) was obtained. However, it was not possible to obtain an organic extract with less than 10% sodium formate (on a contained salt basis) without increasing the organics in the aqueous raffinate.

The extractor will be converted to a dual solvent countercurrent extractor. Lowering the AML feed point to the middle of the extractor and feeding water on the top tray should decrease the amount of salts left in the organic extract. Short range plans are to evaluate t-BuOH as solvent in this countercurrent extractor.

Dow High Formal/Color Complaint (Buras) - The Laboratory and Dallas Quality Assurance are cooperating with Dow (Midland, Michigan) to improve the quality of PE used in their production of dibromoneopentylglycol (DBNPG). Dow believes that high PE linear formal in Celanese PE has an adverse effect on the color of DBNPB and the performance in their customers' end use. They have discontinued purchase of Celanese PE pending resolution of differences in the GC analysis for PE linear formal.

Five samples of pure grade and a sample of our monitor were sent to Dow for comparative analysis and we received copies of their GC results. The indicated that there was about twice as much linear formal as our analysis shows. The analytical discrepancy does not appear to result from errors in calibration or method of calculation. We suspect that differences in the GC scan itself result from their use of a different silylation reagent in the sample workup. We will attempt to duplicate this effect.

A meeting in Bishop to discuss the analytical discrepancy with representatives from Dallas Quality Assurance and Dow has been scheduled in February.

Lubricant Grade PE Plant Trial (Buras) - An action plan for the upcoming Plant trial was developed at a meeting held on 1/8/81. Changes in unit operations, expected problems, and contingency plans were discussed. A schedule of sample analyses was developed to monitor unit performance (PJB-38-81).

Collection of base case data began on 1/28/81 and will continue through the Plant trial scheduled to start on 2/9/81.

Minimizing U.V. Absorbance at 220 nm (Huelle) - After several experiments, results indicate that additional caustic in PE reaction mixture does not lower U.V. absorbance in the "A" cake. It was observed that absorbances of PE "A" cakes from present experiments are much lower than previous results. The only difference between previous and present experiments is the formaldehyde used. During the last week of the Pure PE run, absorbances were low. Correspondingly, formaldehyde used during the run also had low absorbance. No further work will be done on this study until the U.V. absorbance of formaldehyde increases. The abosorbance will be monitored periodically.

1,3-Butylene Glycol

B. K. Huelle, P. J. Buras

Unit Support (Huelle) - None provided.

Improved Aldol Condensation and AcH Recovery (Buras) - No progress due to priorities.

Methanol Oxidation, Formaldehyde Blending

T. A. Jasek, R. L. Day

Formaldehyde Stabilizers (Day) - Recent studies of the polyvinyl alcohol/metalose stabilizer have failed to reproduce the synergistic effect of the combination which was reported in the original work. There are some differences in the techniques and source of HCHO used in recent studies and those used in the original study.

Tests were set up at 40-41°C with 52% HCHO which are designed to indicate whether the differences are important. Some results of these tests should be available for the February monthly report.

Current Specia. Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Jasek	1	To Establish Alternate Catalyst	Continuing
Formaldehyde Product Color		1	MOB	February, 1981
GC Methanol Oxidation Reactor Studies		2	Catalyst Evaluation for Possible Efficiency Improvements	Continuing
Modified Silver Catalyst		2	Catalyst Efficiency Improvements	Continuing
Anaerobic Testing of Chemical Wastes	Young Smith	1	Support Unit Expansion	Continuing
Solar Pond Treatability Study	Young	1	Environmental Mandatory	Continuing
Elimination/Reduction of Solar Pond Odors	Smith	1	Environmental Mandatory	January, 1981
Oxo Reactor Accountability/ Stability	Presley	1	Improve Reactor Control	Continuing
Rhodium Accounting		1	MOB, Catalyst Savings	Continuing
Improved Rhodium Recovery		1	Raw Materials Saving	February, 1981
Develop a Way to Monitor Conversion in the PE Reaction System	Huelle	1	MOB (Quality) and Raw Material	February, 1981

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TMP Extractor Optimization	Day	2	Raw Material Savings	Undetermined
Ester Reactor Corrosion Control	↓	2	Extended Equipment Life	February, 1981
New Formaldehyde Stabilizer System	↓	1	MOB	March, 1981
Develop a Low Energy-High Efficiency AcH-Aldol Separation Process for the BG Unit	Buras	2	Raw Materials and Steam Savings	June, 1981
Eliminate VCI Centrate	↓	1	Provide Waste Disposal Alternatives	Continuing
Extract All Organics From AML Prior	Buras	1	Solve Environmental Problem	Continuing
OSHA Monitoring	Gary	1	Insure Employee Health and Safety - OSHA Compliance	Continuing
Develop an Improved Hydrolysis System for PE Linear Formal	Akin	1	Steam, Acid and Capital Savings	March, 1981

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DOCUMENTS PRODUCED TO PLAINTIFFS

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