

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

From: G. D. Boyd

GDB-116-81

May 12, 1981

Laboratory Department Summary
April, 1981

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CELANESE CHEMICAL COMPANY
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Bishop, Texas

To: Dr. R. R. Graham

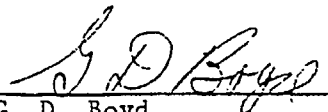
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A summary of each group's monthly report is attached. Section I contains a summary of the Quality Assurance and Analytical Group activities with tables of analytical statistics, shipments, quality waivers, quality complaints and sample shipping statistics. 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

GDB:jr

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HIGHLIGHTS

- BOD II startup is continuing to progress smoothly.
- Testing of powdered activated carbon to enhance aerobic treatment has begun.
- A plan has been formulated to develop more laboratory data for propylene extraction, do cursory design, and request microunit studies by CCCTC.
- The PE formal strong acid/resin catalyzed hydrolysis system run time passed 254 days (~8.5 months). No activity loss has been detected after the equivalent of 135 days (~4.5 months) of exposure since regeneration.

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I. ANALYTICAL GROUP

Competitive Product Analysis

A sample of Derivados Forestales prilled formaldehyde was obtained from a distributor in Australia. Analysis of this material showed it to be comparable to a previous sample, i.e., dust-free, uniform particle size, and free-flowing.

Utilities

A study of the pH of the lime pit discharge is under way. This is to assist Engineering in an effort to better control the pH of material discharged to the Plant front ditch.

Special Analytical Assignments

Capillary Column GC Investigations (B. W. Wise) - Routine Instrument Laboratory GC procedures are being evaluated and compared to Capillary Column scans. The main objective is to determine the applicability of Capillary Chromatography to routine analysis. An additional segment of this study is to obtain "Base Case" scans of Chemcel products and by-products and retain the scans for future comparisons.

Solar Pond and Retrieval Well Feed to Aerobic Tank (F. R. Dow, B. W. Wise) Several basic analytical tests were made on the five Laboratory aerobic filters (3 with solar pond feed and 2 with retrieval well feed). The investigation was primarily concerned with potential interferences to COD analyses. It was determined that the Cl^- content was not a problem with the COD analysis, but an unusual Cl^- distribution between the feed and the filters was found. Three filters (2 retrieval well and 1 solar pond) had Cl^- values the same as the feed, but the two filters containing solar pond feed showed a Cl^- increase from 250 ppm Cl^- (feed) to 420 ppm in the filter. The reason for this increase has not been determined.

Quality Waivers

1. 4-6-81 - Para Fine Powder - 20M lbs. to Ethyl Corp., Orangeburg, S.C. - (SQD) - Iron 2.9 ppm vs. spec. of 2.0 ppm max.
2. 4-9-81 - n-Butyl Acetate - 599,270 lbs. to Celanese Bayport Terminal - (SQD) - Assay 99.43 vs. spec. of 99.5 min.
3. 4-16-81 - 50% Unstabilized HCHO - 186,670 lbs. to Badische Corp., Freeport, Texas - Color = 7 vs. max. spec. of 5 (QD).

<u>Product</u>	<u>% QD¹</u> <u>Month</u>	<u>% QD</u> <u>YTD</u>	<u>% SQD²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Para F. Pwd.	0	0	4.8	3.5
Diacetone Alcohol	0	2.8	0	0
Formaldehyde	0.7	0.4	0	0
n-Butyl Acetate	0	0	10.8	3.8

¹ = Lbs. of product shipped out of Manufacturing Control Specification vs. total lbs. of that product shipped for month and YTD.

² = Lbs. of product shipped out of Sales Specification vs. total lbs. of that product shipped for month and YTD.

Analytical Statistics

<u>Sample Count</u>	<u>Apr.</u>	<u>Apr.</u> <u>YTD</u>	<u>Previous</u> <u>YTD</u>
Chemical Section	5210	18082	20704
Instrument Section	2850	10143	10776
Total	8060	28225	31480

Technician Overtime

Chemical Section	69	331	355
Instrument Section	60	168	190
Total	129	499	545

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<u>Sample Shipping</u>	<u>Apr.</u>	<u>Apr.</u> <u>YTD</u>	<u>Previous</u> <u>YTD</u>
Domestic	64	240	275
Export	0	14	10
Plastics	0	4	32
Others	<u>10</u>	<u>44</u>	<u>29</u>
Total	74	302	346

QA Statistics

Shipments	659	2557	3663
Waivers (QD)	1	4	0
Waivers (SQD)	2	4	4
Complaints	0	3	16

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II. SPECIAL PROBLEMS GROUP

Process Technology B. C. Kerr

Solar Pond Disposal - A report has been written that gives physical and chemical data on Chemcel solar pond liquids.

Injection Well Compatability - URM, Inc., has expressed concern that Chemcel Solar Pond liquids may not be suitable for injection well disposal without prior chemical treatment. URM, Inc., has tentatively suggested the use of hydrochloric acid to adjust the pH to 6.0. The primary goal appears to be conversion of carbonates to chloride and off-gassing of CO₂. Laboratory work is planned to evaluate this procedure. Other alternatives, i.e., treatment with lime or calcium chloride, are also being investigated.

Methanol Oxidation, Formaldehyde Blending T. A. Jasek, S. G. Smith

Ultrasonically Cleaned Used Catalyst - Two used catalyst beds were topped (10%) with ultrasonically cleaned catalyst in MO IV on April 8th. One charge was not brought up due to a cracked reactor and the thermocouple malfunctioned on the second, so no temperature data could be obtained. Initially, the bed ran approximately 0.5% lower efficiency than the control (water washed catalyst). The % CO has started to build in the test charge and it will be taken off line shortly. Additional tests of ultrasonically cleaned silver catalyst will be conducted.

Color Problems - MO III experienced a product color problem early in the month. This increase in color appeared to correlate with the transfer of T-240 OH to MO III during the MO II turnaround. The color, although it could be reduced by the addition of EDTA (an iron complexing agent), seemed to be the result of some species present in the T-240 OH. The color appeared to be the cumulative result of the presence of a color species in the T-240 OH and the iron present in the system.

A second color problem in all the units occurred later in the month. This problem was traced to the MeOH feed. Operations took the necessary corrective action.

MO Unit Support - The Laboratory performed analytical AMR's at the following units during the month of April:

<u>Date</u>	<u>Unit</u>	<u>Indicated</u>	<u>AMR</u>	<u>Analytical</u>
10 Apr '81	MO II	0.88-0.93		0.77
15 Apr '81	MO I	0.86-0.90		0.86

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Utilities, BOD

S. G. Smith, P. W. Young, D. R. Akin, G. R. Stasney

BOD II (S.G. Smith) - Support continues to be provided for startup of the anaerobic/aerobic BOD II system. Following daily examination of operating parameters, recommendations are being made for loading increases and other process parameter changes. Currently, the organic loading to the filter is approximately 0.065 (feed rate = 170 gpm), anaerobic outfall COD $\approx$ 1100 ppm. The filter pH is holding at $\sim$ 7.0; only sodium formate is being added for supplemental Na^+ (no 20% caustic). The aerobic section, being fed both equalization basin and anaerobic outfall, has an outfall COD of $\sim$ 100 ppm.

Input has been provided for a bi-weekly status report covering BOD II startup published by the Technical Startup Team.

A report covering the results of the filter "pulse" test is being written.

Waste Treatment (P. W. Young)

Retrieval Well Aerobic Treatability - Acclimation of laboratory aerobic unit to retrieval well feed is continuing. Well material makes up 10% of the volume and 100% of the COD of the current feed.

Solar Inventory Aerobic Treatability - Solar inventory diluted with water (1 part to 15 parts) make up is the current feed for an aerobic lab unit. Current efficiency is 88% with an outfall COD of 900 ppm (calculated based on zero evaporation).

Carbon Polishing - The current efficiency of an activated carbon column treating aerobic lab unit effluent (treated solar inventory) is 70%. A carbon bed exhaustion rate study is underway.

Solar Inventory Treatability-PAC - 2880 ppm powdered activated carbon (PAC) was added to an operating aerobic unit. Outfall COD decreased from 800 ppm to 650 ppm (89% to 91% removal) during the first full detention period. Plans include varying the carbon concentration to improve removal efficiency.

Utilities, Waste Treatment (D. R. Akin)

Anaerobic-Aerobic Treatment of Solar Pond Inventory and Retrieval Well Effluent - Anaerobic-aerobic filter #2 continues to be fed solar pond inventory. Currently, the #2 filter feed contains 4.2 volume % solar pond material with the remaining volume being river water and BOD II feed. 63% of the total feed COD is from solar pond material. The recycle COD of filter #2 has gradually risen during the month of April from $\sim$ 2800 ppm to 3500 ppm. Due to this rise in COD the organic loading was reduced from 0.4 to 0.3 lb COD/ft³/day. Aerobic detention time is set at 3.5 days. The outfall COD from the aerobic section has risen from $\sim$ 600 ppm to $\sim$ 750 ppm during the month of April. The current target is to replace BOD II feed with a mixture of solar pond material and river water.

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Anaerobic-aerobic filter #3 continues to be fed retrieval well effluent. The current feed dilution is 1.7 volume % retrieval well material with the remaining volume being river water and BOD II feed. 20% of the total feed COD is from retrieval well material. The recycle COD of filter #3 has been in the range of 1200-1500 ppm during April. The organic loading is set at 0.4 lb. COD/ft³/days. The aerobic detention time is set at 3.5 days with final outfall COD in the range of 150-350 ppm. The current target for filter #3 is to replace BOD II feed with a mixture of retrieval well and river water.

Dialysis of Aerobic #2 Outfall and Aerobic #6 Outfall - Dialysis was performed on the outfall from Aerobic Tank #2 and also on Aerobic Tank #6 to determine if the higher than expected COD could be attributed to refractory compound(s) or high molecular weight compounds of biological origin. Matter with a molecular weight greater than 12,000 would be retained by the dialysis tubing. After 72 hours 70% of the original COD of both outfalls had passed through the tubing. This would tend to indicate that the higher than normal COD was caused by relatively low molecular weight refractory compound(s).

Methanol Synthesis
J. C. Bustabad

MS Unit Support - During the catalyst change-out of the 5MM and 2MM Girdlers at the end of last month, solids were found in the reboiler of T-25. There was a concern that sulfur compounds might be present in these solids. CCCTC analyses by x-ray fluorescence showed that if sulfur were present, it was only present as a trace component.

Undetectable levels of H₂S (4/8 and 4/27) in recycled CO₂ were observed (Dräger tube) after the turnaround of the Girdlers.

On April 23rd the Ni(CO)₄ level determined in the nitrogen purge gas from the South Bank of the 5MM Girdler reformer was <.001 ppm/v. The reformer had been brought down for quench pot maintenance work.

The Laboratory's screening of Sharp Chemical Company's Inhibitor 105 without borax [sodium metavanadate (NaVO₃) in water] was completed. CCCTC analysis by x-ray fluorescence showed 0.2% (±20% accuracy) sulfur in the solids isolated from Inhibitor 105 by evaporation (Inhibitor 105 was 4.3% solids). H₂S formation from the sulfur contaminants in Inhibitor 105 should not be ruled out, according to L. E. Wade (CCCTC). Thus, some degree of poisoning of the catalyst in the Girdlers via H₂S in the recycled CO₂ should not be ruled out. A letter detailing the Laboratory's screening of Inhibitor 105 is being reviewed.

(J. D. Presley, J. C. Bustabad) - Analyses of the rhodium solution returned from Johnson-Matthey, Inc. showed .350% rhodium (6.49 troy ounces of rhodium). This information was presented in a joint letter.

(F. R. Dow) - Laboratory analyses of contaminated MeOH in storage (23.5M gal) showed 3416 ppm and 6579 ppm MEA in MeOH in V-645 and V-650 respectively. There is a possibility of further laboratory work to support reclaiming this contaminated MeOH.

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Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - The reactor has been upset several times this month due to maintenance shutdowns. Composition has been fairly constant with TPP at 25-27 wt.%. Average reactor accountability for April is 85+%.

Rhodium Accountability - Analysis of samples taken during catalyst storage in the dump vessel indicate 101% accountability. Total rhodium losses in T-210 Res. are about three pounds for this run.

Improved Rhodium Recovery - Operations, Process Engineering, and the Laboratory met to discuss the direction of the rhodium extraction project. Specific tasks were assigned for development of cursory economics and design. Details of the meeting are reported in RRC-63 (R. R. Carpenter).

A short project has been initiated to determine the feasibility of removing rhodium from T-210 Residue by activated carbon treatment. In initial tests 91% of the rhodium was removed. Temperature variation will be tried to see if the carbon removal capacity can be extended.

Analytical Support - Samples of vinyl acetate (VA) were received from Clear Lake and Bay City for "round-robin" analysis of copper. Riechold discontinued the purchase of Celanese VA because of high copper levels. The samples of VA received here show no detectable copper.

A leased tank car (MOBX 52534) was received at Bishop which contained unknown material. A sample of the unknown was analyzed by Infrared Spectroscopy and found to be polyethylene.

Underground Resource Management, Inc. (URM) has been testing various combinations of Bishop liquid waste streams for possible deep well injection. When the streams were combined and heated a precipitate was formed. Various tests have been conducted on the precipitate, but total identification is still incomplete. The sample was found to contain ~30% iron by weight. No other metals (nickel, copper, chromium, sodium) were detected in significant quantities. The presence of organics is indicated by the vigorous reaction with aqua regia. However, the Infrared Spectrum does not indicate organic composition. Work continues.

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

The Plant-wide personal noise exposure survey has been completed and the report is in preparation.

Several insulation samples were tested for asbestos. Three samples from the Boiler area were positive. (KVP)

A Plant-wide follow-up formaldehyde personal exposure survey is being conducted. (KVP)

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The feasibility of the Monsanto formaldehyde monitoring method is being determined. (CEG)

Acetates, Paraform, Formcel[®] Products
J. E. Russell

Unit Support - None provided.

Trimethylolpropane
R. L. Day

TMP Efficiency - The 1b TMP/gal factors used in yield accounting were verified. No recommendations for change are necessary. However, it was recommended that yield accounting incorporate temperature corrections for butyraldehyde transfers into the TMP unit. This work has been documented and co-authored with Process Engineering.

T-312 Extractor Optimization - Documentation of this work is still underway. Further investigation has revealed that the TMP losses out the base are inversely proportional to the ratio of base temperature/sodium formate concentration in the residue. It appears that the base temperature is most strongly influenced by the sodium formate concentration plus the incoming ethyl acetate temperature. Work is underway to determine the factors influencing the ethyl acetate losses. It appears that there are three or four factors simultaneously affecting these losses. In addition, there appears to be significant differences between the relationships established with the ethyl acetate losses for July and December. The reason for these discrepancies will be investigated.

Pentaerythritol
P. J. Buras, B. K. Huelle, J. E. Russell

Minimizing UV Absorbance at 220 nm (B. K. Huelle) - In order to achieve better agreement between Bishop and Du Pont UV analytical results, the Bishop method will be modified. The new method will be implemented in the next PE Pure run.

This new method should decrease variability in results. However, some variance is inevitable because a truly homogenous lot sample can not practically be obtained. The routine analytical lab has shown that smaller PE crystals retained on 200 mesh screen has higher absorbance than the total sample. PE dust is added in "slugs" to the PE hoppers and is possibly the cause of the variance. Du Pont has had a history of UV sample variations within one bag of PE.

The possibility that antifoam and age of HCHO caused high UV absorbance was eliminated. The possibility that impurities in formic acid or conditions around the HCHO stripper column cause high absorbance will be explored. Another

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Plant trial using only MO HCHO is tentatively planned during the next PE Pure run, depending on weak HCHO inventory. After the second Plant trial, final conclusions and recommendations will be published.

Dow High Formal/Color Complaint (P. J. Buras) - Low formal PE produced during February performed well in Dow's end use. The discrepancy in formal analysis between Dow and Celanese has not been resolved. We are awaiting results of Dow's evaluation of our revised SMA.

Eliminate VCI Centrate Waste Stream (P. J. Buras) - Several samples of the raffinate from VCI's bench scale extractor were analyzed. PE-type organics averaged 1.3 wt.% (range 0.7-2.1 wt.%) in the raffinate.

A study of the recovery of mono-PE from the organic extract was begun. Following removal of the 2-ethyl hexylamine solvent by VCI, the remaining organics contained ~50 wt.% mono-PE. When these organics were dissolved in hot water, a dark colored oil layer separated. Yellow colored PE crystallized out of the aqueous layer on cooling. Analysis of the dried crystals showed ~85 wt.% mono-PE with the major impurities being PE cyclic formal, di-PE, and tri-PE. Further work will define the recovery efficiency and crystal purity.

PE Formal Hydrolysis (J. E. Russell, D. R. Akin) - Work continues on the hydrolysis system. The system promotes the hydrolysis of PE mono linear formal with a strong acid ion exchange resin (Rohm and Haas Amberlyst 15) and a strong acid (HFO or H_2SO_4) as co-catalysts. The resin bed has been used with no loss of activity for the equivalent of 134 days (~4.5 months) since reactivation and a total of 254 days (~8.5 months).

A second resin bed was installed in series with the original bed so that the residence time could be increased to 2 hours. The available pumps would not pump the low rates required for two hours in the single bed. Sufficient data has been gathered at that residence time so that a return to the original conditions (1 bed only) will be made. The life of the resin bed is the only data now being gathered.

1,3 Butylene Glycol
B. K. Huelle, J. E. Russell

Unit Support (B. K. Huelle) - None provided.

High Caustic Usage (J. E. Russell) - Increased caustic usage in the unit has prompted a search for sources of increased acid in the feed. Suspects are acetic acid and CO_2 in the feed AcH. Nothing has been determined yet. Work will continue.

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Miscellaneous
R. L. Day

HCHO Stabilizers - A letter (RLD-21-81) has been written. Future experiments have been designed and will be implemented shortly.

Special Problems Group, General

- Hub Thigpen visited the University of Texas at San Antonio on April 6th to recruit chemists.
- Paul Buras will transfer to the Clear Lake Plant effective May 15th.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TMP	Day	1	Raw Material Savings	August, 1981
New Formaldehyde Stabilizer System		1	MOB	June, 1981
Develop a Low Energy-High Efficiency AcH-Aldol Separation Process for the BG Unit-Report Work to Date.	Buras	2	Raw Materials and Steam Savings	May 15, 1981
Extract All Organics From AML Prior to VCI Processing - Report Work to Date		1	Solve Environmental Problem	May 15, 1981
OSHA Monitoring	Gary	1	Insure Employee Health and Safety - OSHA Compliance	Continuing
Develop an Improved Hydrolysis System for PE Linear Formal	Russell	1	Steam, Acid and Capital Savings	September, 1981
Eliminate or Reduce the Salt Content on V-778 (1,3-BG heavy ends) to Allow it to be Burned Without Excessive Particulate Emissions.		1	Solve Environmental Problems and Recover Energy by Burning Stream	July, 1981
Develop a Way to Monitor Conversion in the PE Reaction System.	Huelle	1	MOB (Quality) and Raw Materials Savings	July, 1981
Determine the Potential of PE Dust for Use as Lube Grade PE - Assist Production Personnel as Needed.	Harvey	1	Supply Growing Market for PE	July, 1981
Recover PE from VCI Extraction Process Organic Stream		1	Raw Materials Savings	
Solar Pond Disposal	Kerr	1	Government Regulation	June 1, 1981

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Oxo Reactor Accountability/ Stability	Presley	1	Improve Reactor Control	Continuing
Rhodium Accounting		1	MOB, Catalyst Savings	Continuing
Improved Rhodium Recovery II		1	Raw Materials Savings	June, 1981
Solar Pond Retrieval Well Treatability Studies	Young Akin	1	Environmental Mandatory	July 1, 1981
MO Catalyst Screening	Jasek	1	To Establish Alternate Catalyst	Continuing
Formaldehyde Product Color		1	MOB	June, 1981
GC Methanol Oxidation Reactor Optimization		2	Catalyst Evaluation for Possible Efficiency Improvements.	September 1, 1981
Modified Silver Catalyst		2	Catalyst Efficiency Improvements	January 1, 1982

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