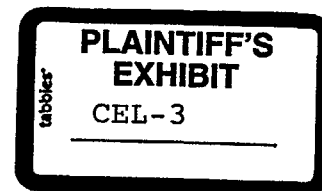


CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

From: G. D. Boyd

GDB-112-81

April 7, 1981

Laboratory Department Summary
March, 1981

DISTRIBUTION

Chemical

J. J. Fritsch
W. H. Brough
J. B. Brown
R. R. Carpenter
M. W. Clifton
R. S. Hahn
R. W. James
B. C. Kerr
C. W. Lee
D. E. Lockwood
R. P. Lowry
A. G. McGehee
W. H. Meyer
D. G. Pausky
O. W. Schnellenberger
G. R. Scholz
D. D. Siemonsma
W. L. Sharp
K. R. Stelts
H. H. Thigpen
W. L. Wallace

Plastics

R. G. Brandt
A. C. Abshier
R. L. Bunkley
H. L. Davis
D. A. Reck
E. J. Heinz

CCCTC

G. J. Fisher
W. E. Heinz
W. T. McNair
Central Files (2)

J. N. Gann, Bay City
J. E. Sanborn, Bayport Terminal
J. L. Paul, Clear Lake
W. H. Tuke, Pampa
T. H. Golson, Dallas

003494

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

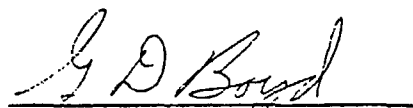
From: G. D. Boyd

GDB-112-81

April 7, 1981

Laboratory Department Summary
March, 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

GDB:jr

003495

HIGHLIGHTS

- The rhodium extraction project was completed; the report is being issued.
- BOD II startup to date compares well with lab simulations.
- The PE formal strong acid-resin catalyzed hydrolysis system passed 239 days (~8 months). No activity loss has been detected since regeneration 120 days (4 months) ago.
- The intra-lot consistency of PE linear formal content of PE lot 81-55-3P was confirmed to be good. Dow Chemical analyzed preshipment samples and has placed an order for PE from this low formal (~0.1 wt.%) lot.
- Results of a short (2 day) Plant trial attempting to produce low U.V. absorbing (220 nm) PE for DuPont by feeding only MO HCHO to the unit were inconclusive. The data will be reanalyzed following studies to improve the reproducibility of the analytical method.
- NPDES Permit renewal sampling was finished 3-25-81. Required analysis is underway.
- BOD outfall during a period of high outfall COD (the first week in March) was shown by analysis not to contain compounds characteristic of Chemcel effluent. It is believed that the COD was the result of bacteria or bacteria "slime".

003496

I. ANALYTICAL GROUP

Competitive Products

No samples received this month.

Methanol Synthesis (MS-MeOH)

Chloride analysis by Ion Exchange Chromatography was provided for samples of CLP and Bayport Terminal MeOH. Levels of 0.01 - 0.02 ppm chlorides indicate that the material meets a special customer specification of 0.05 ppm maximum.

Polyol Area

Pentaerythritol - Analyses are being provided for a study to determine the source of UV absorbance in our Pure PE product. This is in an effort to meet a special customer specification (DuPont). Work continues.

Utilities - BOD

The Analytical Group has assumed the routine determination of nutrient in the BOD I & II systems. Analysis by ion exchange chromatography should provide much better control of this operation.

Special Analytical Assignments

Solar Pond Feed to Aerobic Tanks (F. R. Dow, B. W. Wise) - On March 5, 1981 three blends of solar pond material were prepared to feed to three different aerobic tanks. These blends were analyzed for a 6 day period to monitor components through the aerobic system. The analysis determined that hexamethylenetetramine (HMTA) was being formed in the feed vessel because nutrients were added prior to the aerobic tanks. In one tank, the HMTA buildup was equivalent to ~10% of the outfall COD. The nutrient addition point has been changed.

101 BOD Outfall COD Buildup (F. R. Dow, B. W. Wise) - The COD in the 101 outfall began to increase from ~150 to ~350 during the first week of March, 1981. Unit parameters were normal, so samples were analyzed for organic and nitrogen compounds in all effluent streams and the BOD ponds. Analysis determined that the 101 BOD outfall did not contain any organic or nitrogen compounds characteristic of Chemcel effluent, so it is believed that the high COD in the outfall was the result of bacteria or bacteria "slime".

003497

Quality Waivers

1. 3-2-81 - DiAcetone Alcohol - 47.4M lbs., to Accron Chemical Co., Houston, Texas - (QD) - Color 12 vs. MCS of 10 maximum - Sales Specification = 25 maximum.
2. 3-16-81 - DiAcetone Alcohol - 15.3M lbs., to Exxon Corp., Houston, Texas - (QD) - Color 17 vs. MCS of 10 maximum - Sales Specification = 25 maximum.
3. 3-16-81 - 50% Unstabilized HCHO - 183.2M lbs., to Badische Corp., Freeport, Texas - (QD) - Color 7 vs. special customer specification of 5 maximum - Sales Specification - 10.

<u>Product</u>	<u>% QD¹</u> <u>Month</u>	<u>% QD</u> <u>YTD</u>	<u>% SDQ²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Para F. Pwd.	0	0	0	3.0
DiAcetone Alcohol	6.7	3.5	0	0
Formaldehyde	0.8	0.3	0	0

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

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

Analytical Statistics

<u>Sample Count</u>	<u>March</u>	<u>March</u>	<u>Previous</u>
	<u>YTD</u>	<u>YTD</u>	<u>YTD</u>
Chemical Section	4090	12872	15024
Instrument Section	2625	7293	7970
Total	6715	20165	22994
<u>Technician Overtime</u>			
Chemical Section	79	262	170
Instrument Section	45	108	148
Total	124	370	318

003498

Shipping and QA Statistics

<u>Sample Shipping</u>	<u>March</u>	<u>March YTD</u>	<u>Previous YTD</u>
Domestic	70	176	221
Export	9	14	6
Plastics	0	4	20
Other	<u>19</u>	<u>34</u>	<u>27</u>
Total	98	228	274

QA Statistics

Shipments (No. of Orders)	802	1898	2881
Waivers QD	3	3	0
SQD	0	2	3
Complaints	0	3	11

Analytical Group - General

Instrument Section Technicians and Special Problems Chemists attended an inhouse seminar on capabilities and operation of the Dionex Ion Exchange Chromatography system.

003499

II. SPECIAL PROBLEMS GROUP

Process Technology B. C. Kerr

Solar Pond Disposal - Information received during the past month indicates a very low probability for sale of solar pond material as a source for sodium carbonate. However, samples of solar pond material have been sent to an independent laboratory for fuel value analysis. When these data are available, a report will be issued giving the pertinent information which has been developed for reference purposes.

Methanol Oxidation, Formaldehyde Blending T. A. Jasek

GC/MO Reactor - Some tests were run on the GC/MO Reactor during the month. Included were tests to confirm reproducible delivery of a standard air-methanol mixture for reaction. Also, preliminary investigations were made into the changes of CO, CO₂ and H₂ with temperature. Currently, the purchase of a new GC has been approved and the reactor will be installed on it when the GC arrives.

Ultrasonically Cleaned Used Catalyst - The Laboratory found the washed, used catalyst which was being charged into reactors still contained a substantial amount of carbon. By ultrasonically cleaning the previously washed catalyst, an additional 0.1 wt.% (based on silver weight) carbon could be removed. The Laboratory is in the process of ultrasonically cleaning 150 oz. of silver catalyst to be used in an experimental run at MO IV. Because of the labor intensive cleaning process using a lab scale ultrasonic cleaner, this silver will be used to top two washed catalyst charges.

MO Unit Support - The Laboratory performed analytical AMR's at MO II following a short shutdown for maintenance. The averaged analytical AMR was 0.66 while the indicated unit AMR was 0.86 - 0.92.

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

BOD II Startup Support (S. G. Smith) - A "pulse" test was performed on the BOD anaerobic filter to determine flow distribution patterns and the void volume of the reactor. A drum of t-butanol was added to the filter; samples of the filter liquid at different points were taken over a 24-hour period and analyzed for t-BuOH. Results of this test are to be published.

Support is being provided in the startup of the anaerobic/aerobic BOD II system. Feed was introduced to the seeded filter on March 20, 1981 at an OL $\approx$ 0.01. After 7 days, feed was discontinued due to increasing recycle COD, HCHO, and MeOH levels, and decreasing pH. Caustic was added to increase the pH from 6.45 to 6.80. Currently, HCHO levels have decreased to <10 ppm, MeOH concentration is at approximately 170 ppm, COD $\approx$ 1100 ppm. Feed to the filter will be resumed once MeOH levels begin to decrease.

003500

Anaerobic-Aerobic Treatment of Solar Pond Inventory (D. R. Akin) - Anaerobic/aerobic filter #2 continues to be fed solar pond inventory at increasing concentrations. Currently, the #2 filter feed contains 3 volume % solar pond material with the remaining volume being river water and BOD II feed. 48% of the total feed COD is from solar pond material, currently the final outfall of filter #2 contains 430 - 530 ppm COD. The current target is to replace BOD II feed with a mixture of solar pond material and river water.

Two retrieval well samples were received during the second week of March. These were McKinney Farm (~55,000 ppm COD) and Collins Farm (~1400 ppm COD). The Laboratory began acclimating anaerobic/aerobic filter #3 to McKinney Farm retrieval well material during the last week in March. The current feed dilution is 1 volume % retrieval well material with the remaining volume being river water and BOD II feed. 16% of the total feed COD is from retrieval well material. The current target for filter #3 is to replace BOD II feed with a mixture of retrieval well and river water. The final outfall of filter #2 contains 110-140 ppm COD. Both systems are operated at an anaerobic OL = 0.4 lbs COD/ft³/day and an aerobic detention time of 3.5 days.

Aerobic Treatment of Solar Inventory and Retrieval Well Effluent (P. W. Young)-
Tank #4 is being acclimated to contaminated groundwater from the McKinney Farm retrieval well. Tanks #5 and #6 are being fed 15:1 mixtures of river water and the solar pond composite collected in February (COD = 90,000 ppm). At a detention time of 9 days, outfall COD = 1900 and 1600 ppm respectively, removal efficiency = 76 and 78%. System parameters are being changed to improve removal efficiency. Activated carbon is being studied for use as a polisher and as a detention aid in the activated sludge aeration tanks.

Methanol Synthesis
J. C. Bustabad

MS Unit Support - A letter that documents the Laboratory support provided for the Dow corrosion inhibitor trial is being reviewed.

Water that is sewered from the 5MM Girdler CO₂ stream contained 2.7 ppm sulfur (sampled 3/3 and 3/9) (microcoulometry). Water that is recycled from the 5MM Girdler CO₂ stream contained <1 ppm sulfur (microcoulometry). Sulfur levels in these streams will continue to be monitored after the turnaround.

With the catalyst change-out of the 5MM and 2MM Girdlers scheduled for the week of March 23rd, there was concern about the sulfur containing inhibitor that deposited on the metal in the MEA systems during the use of Dow's corrosion inhibitor. Thus, a precipitate from the 5MM Girdler MEA and solids from the Shift MEA filter were submitted to the Tech Center for sulfur analysis by x-ray. Sulfur was detected in trace amounts in both samples.

Nickel tetracarbonyl levels determined on the nitrogen purge gas from the 5MM and 2MM Girdlers prior to catalyst change-out were <.001 ppm/v (within OSHA guidelines).

003501

Solids found in the 2MM Girdler quench pot during the turnaround were approximately 95% iron and approximately 5% nickel.

The routine lab is running weekly samples for MEA loading. The average monthly lean loadings of .17 moles CO₂/mole amine in both 5MM Girdler and Shift MEA systems are close to the upper range of the recommended levels of .08 - .15. The average monthly rich loadings were .50 and .39 moles CO₂/mole amine in the 5MM Girdler and Shift systems respectively. The recommended level for rich loadings is $\leq$.45 moles CO₂/mole MEA; thus, the Shift amine is within range, while the 5MM Girdler amine is high.

Based on the results of foam tests, Chem Central's (new MEA vendor) MEA is comparable to McKesson's (former MEA vendor) MEA in terms of foaming.

Sodium metavanadate vendors were contacted to confirm sodium metavanadate's use as a corrosion inhibitor in syn gas MEA systems.

Solids concentration was determined by evaporation to be 4.3% in the sodium metavanadate solution from Sharp Chemical Company (Inhibitor 105 without borox). These solids were sent to the Tech Center for sulfur analysis by x-ray. Results are pending.

Foam tests conducted with the Sharp inhibitor showed it to have a negligible effect on foaming.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - For the first part of the month the TPP concentration has remained 27-29 wt.%. The reactor has been down since March 21 for minor maintenance while the catalyst is being changed in the 2MM and 5MM Girdler. Plans are to restart March 31 with the same catalyst. The Oxo catalyst still has a good chemical and physical appearance. Hence, no startup problems are anticipated.

Rhodium Accountability - Through March 13, reactor rhodium accountability has remained high, 95+%. During the shutdown, the catalyst was stored in the dump vessel. A sample and accurate level reading were taken prior to returning the catalyst to the reactor. The analysis will be noted in the March Unit Rhodium Accounting.

Improved Rhodium Recovery - A letter has been written detailing the results of the hydrocarbon extraction study. Using catalyst from Bishop Oxo Run 6 as an example, we were able to extract 59% of the rhodium as active catalyst and 90% of the TPP. This would represent a possible total savings of \$365M over sending the catalyst to the reclaimer. A 25% foaming reduction was also achieved. Finally, we were able to partially reactivate the low activity phase of the extraction.

Plans are to continue with earlier work to attempt forced total deactivation of Oxo catalyst so that the rhodium can be concentrated and the TPP level reduced. Thereby, we could achieve better rhodium recovery for a total reactor charge at the reclaimer.

003502

Oxo Unit Support - In the past, serious Oxo compressor (C-322) seal leaks allowed butyraldehyde to get into the lubrication oil and cause problems. Before and during the shutdown, samples of T-32 lubricating oil were taken from C-322. The samples were analyzed and found to contain about 400 ppm butyraldehyde. This level is considered normal and should present no problems.

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

The initial noise exposure survey has been completed. Also, previously reported job classifications with high (>85dBA) exposures were resampled. (KVP)

The formaldehyde exposure (<1.0 ppm 8 hr TWA) in the temporary building housing the MO IV project team has been determined. Further sampling will be done on a warmer day. The temporary building housing the SHE Department will be sampled. (KVP)

Routine formaldehyde exposure resampling has begun. (KVP)

Two samples suspected of containing asbestos were analyzed and gave positive results. (KVP)

The validation of the proposed method for determining the carbon monoxide 8 hr TWA using long term Dräger CO 50/a-1 tubes was completed and reported. (CEG-44-81)

The evaluation of the Monsanto method for determining formaldehyde exposure has begun. (CEG)

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

Unit Support - The study of methanol losses in methyl formcel was completed and documented in a joint report with Process Engineering. Adjusting the back pressure regulator on the reactor vent appears to have minimized the losses.

Trimethylolpropane
R. L. Day

TMP Efficiency - Due to efficiency problems in TMP, Operations requested that some lb TMP/gal factors used in Yield Accounting be verified. Pounds TMP/gallon factors for three WIP tanks and for the finished product were checked. Process Engineering will use the data in their examination of yield accounting procedures.

T-312 Extractor Optimization Project - Documentation of base case data gathered in July and December, 1980, is about 75% complete. Evaluations of some of the data will be included. Plans include a meeting with Production and Process to discuss future work.

003503

Diacetone Alcohol
B. K. Huelle

Unit Support - None provided.

Pentaerythritol
D. R. Akin, P. J. Buras, B. K. Huelle, J. E. Russell,
F. R. Dow, H. A. Ducote, B. W. Wise

Lubricant Grade PE Plant Trial (P. J. Buras, F. R. Dow, H. A. Ducote, B. W. Wise) - Laboratory analytical support for the Plant trial was completed. The proposed product specifications for ash, linear formal, cyclic formal, and tri-PE were met during the Plant trial. An extra operator was required to maintain equipment operation necessary to meet the proposed moisture specification. It was not possible to control the di-PE content of the finished product within the proposed 11-12 wt.% range.

Based on AML analysis, unit efficiency decreased approximately 1.5% during lube grade PE production. This does not include any efficiency losses due to shut downs and start ups. No increase in sulfates in AML was observed.

Samples of PE Dryer Baghouse dust were collected and analyzed. Work is in progress to define the composition of this dust for possible use in formulating lube grade PE.

Minimizing U.V. Absorbance at 220 nm (B. K. Huelle) - PE reactions using HCHO from feed streams to the weak formaldehyde unit yielded good product with absorbances comparable to absorbances from product made from MO HCHO. A Plant trial was conducted on March 23 to March 25 using only HCHO from MO IV. Also, the caustic excess levels were kept in the desired range between .01 to .02 wt.% in the reactor product.

Samples taken from B belt during the trial had absorbances ranging between .040 A and .053 A. The specification is .050 A. The lowest absorbance attained in the laboratory work was .045 A. It was recently discovered that heating of the 2% PE solution before u.v. analysis decreases the u.v. absorbance at 220 nm. The method used in the instrument laboratory in routine analysis of PE pure product has always called for heating the solution before u.v. analysis. Some variability may also be attributed to instrument problems. We are attempting to improve the reproducibility of the analytical method.

Dow High Formal/Color Complaint (P. J. Buras) - Dow believes that occasional variations in the PE linear formal content of Celanese PE have an adverse effect on the color and performance of their product (dibromoneopentyl glycol). We select lots and attempt to ship them PE containing <0.2 wt.% linear formal.

Following examination of preshipment qualification samples of Lot 81-55-3P by Dow, they placed an order for 40M lbs. Extensive analysis of this lot at Bishop showed there is normally little pallet-to-pallet variation (± 0.01 wt.%) in linear formal content during periods of smooth operation. We should be able to supply Dow with low formal PE of consistent quality.

003504

Analysis of identical samples by Dow and Celanese showed that Dow consistently gets about 0.1 wt.% higher results for linear formal than we do. Dow continues to use the old Celanese SMA which utilizes a different silyation reagent. They plan to evaluate our revised SMA. We will continue our efforts to resolve the analytical discrepancy.

Eliminate VCI Centrate Waste Stream (P. J. Buras) - A meeting with VCI was held on March 19, 1981. VCI revealed that they have also been working on a system to extract all of the organics from AML. The solvent used in their studies (2-ethylhexylamine) appears to be superior to those being studied by Celanese (t-BuOH, BuH). We will evaluate amine-alcohol mixed solvents. We will also provide some analytical support to VCI for their extraction studies.

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 H₂SO₄) as co-catalysts. The resin bed has been used with no loss of activity for the equivalent of 119 days since reactivation and a total of 239 days.

In order to study the conversion with respect to increased residence time, a larger resin bed is needed due to the inability of existing pumps to feed the minimum flow rates required by the existing system. A new resin bed has been installed so that data may be gathered to re-check activity of new resin in addition to lengthening residence time up to 2 hours. By checking the new resin separately, we can compare the activity of new resin to the activity of the original resin bed. Studies will continue at longer (>1.0 hr) residence times with the two beds in series.

1,3 Butylene Glycol
B. K. Huelle, P. J. Buras

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

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

Miscellaneous
R. L. Day

Formaldehyde Stabilizers - The HCHO stabilizer study is almost complete. A letter will be issued in early April. Some conclusions from this study are as follows:

1. The polyvinyl alcohol/metolose stabilizer mix is as good as the metolose stabilizer alone, but not better (at the concentrations tested).

003505

2. The stability times differ with the source of HCHO used. The two sources used were HCHO directly off the finishing column (T-272) in MO IV, and HCHO which has passed through the ion exchange resin bed. All HCHO shipped out of the Plant passes through the resin bed. The material from the finishing column had longer stability times than the resin treated material.
3. Data indicate that the current average level of 12.7 ppm metolose in HCHO shipments may not be the optimum.
4. There may have been a difference in the stability times by the two techniques used. However, the differences observed could also be due to differences in the concentration of stabilizer in the HCHO samples.

Future work will involve determining the reason for the different stability times when using different sources of HCHO. The optimum concentration of metolose stabilizer in HCHO shipments will also be determined. In addition, further testing of higher concentrations of the PVOH/metolose mix in HCHO will be performed.

Special Problems Group, General

- ° Dan Akin transferred from Hub Thigpen's to Otts Schnellenberger's group.
- ° Jim Russell transferred from Bud McGehee's to Hub Thigpen's group.
- ° Hub Thigpen and Otts Schnellenberger attended the "Achieving Your Potential" inhouse seminar.

003506

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Solar Pond Disposal	Kerr	1	Government Regulation	June 1, 1981
MO Catalyst Screening	Jasek	1	To Establish Alternate Catalyst	Continuing
Formaldehyde Product Color		1	MOB	April, 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
Solar Pond Retrieval Well Treatability Studies	Young Akin	1	Environmental Mandatory	July 1, 1981
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
Ultrasonically Cleaned Used Silver Catalyst	Jasek	1	Catalyst Efficiency Improvements	June, 1981
TMP Extractor Optimization	Day	1	Raw Material Savings	Undetermined

003507

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
New Formaldehyde Stabilizer System	Day	2	MOB	June, 1981
Develop a Low Energy-High Efficiency AcH-Aldol Separation Process for the BG Unit	Buras ↓	2	Raw Materials and Steam Savings	Undetermined
Extract All Organics From AML Prior to VCI Processing	↓	1	Solve Environmental Problem	June 1, 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.	↓	2	Solve Environmental Problem 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	June, 1981
Determine the Effect of Sodium Formate on TMP Decomposition	↓	2	MOB (Quality) and Raw Materials Savings	July, 1981
Determine the Potential of PE Dust for Use as Lube Grade PE	Will be assigned by mid-April	1	Supply Growing Market for PE	June 1, 1981

003508