

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

From: G. D. Boyd

GDB-105-81

March 6, 1981

Laboratory Department Summary
February, 1981

DISTRIBUTION

Chemical

J. J. Fritsch
W. H. Brough
J. B. Brown
R. R. Carpenter
M. W. Clifton
K. G. Gallimore
R. S. Hahn
J. M. Hodges
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
H. W. Hill
E. Munoz
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

File No: 303.3

003478

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

From: G. D. Boyd

GDB-105-81

March 6, 1981

Laboratory Department Summary
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

GDB:jr

003479

HIGHLIGHTS

- Anaerobic/aerobic solar pond treatability study was initiated.
- Utilization of T-288 lower draw as MO feed was begun and is being followed.
- Shortened service runs on Train II Demineralizer were found to be a result of fines buildup in the weak base resin bed.
- UV absorbance at 220nm PE quality problem appears to be related to impurities in the HCHO from the weak HCHO unit.
- The PE formal strong acid-resin catalyzed hydrolysis system passed 212 days *7.1 months). No activity loss has been detected since the regeneration 92 days ago.
- Analysis required for validation of the new method to monitor CO exposure was completed and will be submitted to the validation committee.
- Analytical support of the Waste Systems Management Task Force effort to comply with the current TDWR Enforcement Compliance Directive is underway and on schedule.

003480

I. ANALYTICAL GROUP

Competitive Products

One product (Iso Butyl Acetate) was received from the Eastman Corp. for evaluation. A report will be issued when analysis is completed.

Methanol Synthesis (MS)

Methanol - Since the conversion of "B" train to single column purification, the lower draw off stream of T-288 (finishing column) has gone to feed the MO units. The Instrument Section is providing sodium analyses on this stream to insure low levels of sodium in the formaldehyde produced in the MO units.

Girdlers - Special analyses for MEA and heat stable salts are being provided on both systems. This information is being used to control the corrosion rates in the systems.

1,3 Butylene Glycol (1,3-BG)

T-225 OH (Crude Ethanol) is again being analyzed to determine it's "typical" composition. Truck load quantities, of this material are being shipped to the Pampa Plant for a trial run to recover the ethanol. This stream is otherwise burned at Chemcel.

Waste Systems Management Task Force (WSMTF) Support

Analytical Group support for the WSMTF effort to comply with the current TDWR Enforcement Compliance Directive is underway and on schedule. All February well and creek samples have been analyzed. In addition, analytical support is being provided for agronomy studies of inactive solar ponds, deep well injection compatibility studies and various Special Problems Group biological oxidation studies.

Special Analytical Assignments

Effluent, BOD and Solar Pond Analysis (F. R. Dow, B. W. Wise) - The BOD test pond, surge pond, equalization basin and outfall streams were monitored for 5 consecutive days by capillary column GC to develop base case component data for BOD II. The GC computer raw area and COD were plotted to determine if a correlation existed between COD and components detected by GC. During the 5 day period, the COD remained relatively constant, but the GC component raw area increased.

003481

Solar Pond Feed to Aerobic Filters (F. R. Dow, B. W. Wise) - Two filters are being fed solar pond material diluted with equalization basin and/or river water. Solar pond/equalization basin and equalization basin feed to the filters is being analyzed by capillary GC. Sufficient data has not been obtained to determine specific conclusions about the individual components. However, the pH of the individual feeds decreases daily. The solar pond/equalization basin/river water feed pH change was from 6.35 to 5.18 during the period 2-13-81 to 2-18-81.

Monitor Well Analysis (F. R. Dow, B. W. Wise) - The capillary column GC is being used to analyze all monitor wells for dibutyl phthalate. To date 11 monitor wells have been scanned, 1 well showed 8 ppm, 1 well 3 ppm, 1 well 2 ppm and 8 wells did not show any. The minimum detectable limit for the dibutyl phthalate is 2 ppm.

Ion Chromatograph (J. E. Russell) - The methods for cation and anion analyses have been written and a precision study run for the cation method. The cation method will be published after the statistics on the precision study are received. The anion method precision study has not been completed.

The Laboratory is currently running aeration basins 1 and 2 by the cation method, specifically for NH_3^+ concentrations, on a routine 5 day per week basis. Phosphate analysis will be included as time permits.

The ion chromatography system is being extensively used for pollution control water analysis.

Quality Waivers

1. 1-30-81 - Para Fine Powder - 30M lbs., to Ethyl Corp., Orangeburg, South Carolina - (SQD) - Iron content at 2.6 ppm vs. maximum sales spec. of 2.0 ppm.

2. 2-9-81 - Para Fine Powder - 10M lbs., to Koppers Co., Petrolia, PA. (SQD) - Iron content at 5.0 ppm vs. maximum sales spec. of 2.0 ppm.

<u>Product</u>	¹		²	
	% QD <u>Month</u>	%QD <u>YTD</u>	%SQD <u>Month</u>	%SQD <u>YTD</u>
Para Fine Pwd.	-	-	10.2	5.0

¹
= Lbs. of product shipped out of Mfg. Control Specs. vs. total lbs. of that product shipped for month and year to date.

²
= Lbs. of product shipped out of Sales Specs. vs. total lbs. of that product shipped for month and year to date.

003482

Customer Complaints

2-11-81 - Para Flake - 50M lbs. - Lots 80-283 and 80-357. Customer complained of ½ inch lumps screened from several bags of each Lot. Returned samples of screened lumps confirm ½ inch size.

ANALYTICAL STATISTICS

<u>Sample Count</u>	<u>Feb.</u>	<u>Feb. YTD</u>	<u>Previous YTD</u>
Chemical Section	3976	8782	10337
Instrument Section	<u>2265</u>	<u>4668</u>	<u>5519</u>
Total	6241	13450	15856

Technician Overtime

	<u>Feb.</u>	<u>Feb. YTD</u>	<u>Previous YTD</u>
Chemical Section	22	183	60
Instrument Section	<u>31</u>	<u>63</u>	<u>104</u>
Total	53	246	164

Shipping & QA Statistics

<u>Sample Shipping</u>	<u>Feb.</u>	<u>Feb. YTD</u>	<u>Previous YTD</u>
Domestic	46	106	145
Export	3	5	4
Plastics	4	4	9
Other	<u>12</u>	<u>15</u>	<u>16</u>
Total	65	130	174

QA Statistics

Shipments (No. of orders)	641	1096	1845
Waivers - QD ¹	0	0	0
SQD ²	2	2	3
Complaints	1	3	9

¹ Quality Deviation from Manufacturing Control Specifications.

² Significant Quality Deviation from Sales Specifications.

003483

Analytical Group General

Don Skrovan transferred to the Special Analytical Group from the wet chemical section on 2-2-81. Mr. Skrovan will assist in the coordination and analysis of the monitor wells, solar ponds and soil samples.

Analytical Group supervision attended a Time Management Seminar given in-house on February 9 and 10.

The Instrument Section Analytical Group is being provided with intensive extra training in basic gas chromatography and in ion-exchange chromatography.

The decision was made to withdraw from the past 6 month trial of purchasing standard solutions. By the end of the 1st Quarter the Laboratory will again be preparing and providing all chemical reagents and solutions for use throughout the Plant. The trial was not contributory to service and cost effectiveness.

003484

II. SPECIAL PROBLEMS GROUP

Process Technology

B. C. Kerr

Solar Pond Disposal - Experiments with continuous extraction equipment to reduce the salt content of solar pond liquid have not been mechanically successful due to salt precipitation within the extractor. The continuous extraction system does reduce the amount of acetone required to obtain phasing and crystal strike from a solvent to feed ratio of 5:1 for a batch process to approximately 2:1 in the continuous process. However, the salt removal efficiency drops from approximately 50% to approximately 20% in the continuous vs. batch process. Work on this approach is being set aside in order to pursue the more attractive studies for recovery of the solar pond sodium in black liquor furnace type operations.

A sample of Solar Pond 10 & 11E, taken on 9-25-80, was ashed and fused at bright red heat to produce a virtually colorless salt which contained 45.4% Na_2CO_3 and 7.6% NaHCO_3 based on the ashed sample. Assuming that the solar pond material may be concentrated by a combination of solar evaporation and a final forced evaporation to contain sufficient fuel value to evaporate the remaining water, it appears possible to dispose of the solar pond salts through conversion to Na_2CO_3 in black liquor furnace operations. This work is continuing.

Seven soil samples were submitted to the Laboratory by URM in support of work by a consulting agronomist. The acetone soluble organics have been extracted from those samples for a qualitative organic analysis. Portions of these samples are being analyzed in the analytical laboratory.

Methanol Oxidation, Formaldehyde Blending

T. A. Jasek, J. D. Presley

GC/MO Reactor (T. A. Jasek) - Continuing tests as workload allows.

MO Silver Catalyst Modifiers (T. A. Jasek) - No progress, awaiting micro unit results from CCCTC.

MO Unit Support (T. A. Jasek, J. D. Presley) - Laboratory analytical AMR's were performed at three MO units. The results are illustrated in the following table:

<u>Date</u>	<u>MO Unit/Train</u>	<u>AMR</u>	
		<u>Indicated</u>	<u>Analytical</u>
31 Jan 81	IV/A	0.83-0.87	0.90
3 Feb	II	0.87-1.00	1.04
6 Feb	II	0.98-1.00	1.13
18 Feb	I	0.86-0.94	0.97

003485

Utilities

J. D. Presley, T. A. Jasek

Demineralizer Train II Problems - Channeling in resin bed V-1409 (weak base anionic resin) was found to be a result of resin fines buildup. Laboratory support consisted of identification of the source of the fines, analytical support during regeneration, and evaluation of stored resin for replacing existing resin. A report detailing Laboratory support is forthcoming.

BOD

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

Solar Pond Treatability Studies

Solar Pond Characterization (S. G. Smith) - Samples of Solar Ponds 7A, 7B, 8A, 8B, 8C, 8D, 9, 10/11, and a composite of the ponds are undergoing extensive analytical characterization by the Analytical Section. Results, thus far indicate wide variations in pond COD, TOC, and Na content:

	<u>Composite</u>	<u>Pond Range</u>
COD	90,000 ppm	24,000-240,000 ppm
TOC	32,200 ppm	8,500- 84,600 ppm
Na	22,000 ppm	2,200- 62,000 ppm

Anaerobic-Aerobic Treatment of Solar Pond Inventory (S. G. Smith) - Began acclimating a mature anaerobic filter/activated sludge system to solar pond material during the second week of February. The current target is to replace BOD II feed with a mixture of solar pond and river water. Anaerobic/aerobic unit #3 is currently being operated as a control, being fed only BOD II feed. Both systems are operated at an anaerobic OL = 0.4 lbs. COD/ft³/day and an aerobic detention of 3.5 days. Little difference has been observed in anaerobic filter activities thus far, the final outfall of filter #2 contains 280-300 ppm COD compared to 175-200 ppm in #3 outfall.

Aerobic Treatment of Solar Ponds (P. W. Young) - Experimental tank #4 is currently being acclimated to solar pond material in preparation for a study to determine treatability of contaminated groundwater. Tank #5 is being converted to a feedstock containing only river water and solar pond material. Tank #6 is now treating a 1:15 mixture of solar pond in river water (no equalization basin). Feed detention is now at approximately 9 days, outfall COD = 1250 ppm, removal efficiency = 80%. A new aerobic tank, #7, was setup as a second stage treatment step and is being fed outfall from the tank. Second stage COD removal is only 5-10%; overall 2-stage removal efficiency $\cong$ 85%.

Anaerobic Feed COD Variability (S. G. Smith) - A report is to be co-authored with Process Engineering discussing results of this study.

003486

Elimination, Reduction of Solar Pond Odor (S. G. Smith, T. A. Jasek) - A report on this project has been published

BOD Unit Support (S. G. Smith) - Guidelines for nutrient addition levels in the anaerobic and aerobic system are being documented.

BOD II Startup Support - Assistance is being provided in preparation for a filter "pulse" test, which measures the distribution characteristics of the flow through the anaerobic filter.

Methanol Synthesis

J. C. Bustabad, J. D. Presley

MS-II Unit Support - There was interfacing between the Analytical Group and Special Problems Group as the responsibility of testing for sodium molybdate and sodium nitrite (components of the Celanese developed corrosion inhibitor) in MEA was transferred to the Analytical Group.

The foam tests of sodium molybdate from Climax Molybdenum Company and J. T. Baker were comparable. Further laboratory evaluation of cheaper sodium molybdate from Climax Molybdenum Company and Alfa Products was stopped when the decision was made to discontinue use of the Celanese developed corrosion inhibitor.

In an effort to prevent corrosion rates in the MEA system from becoming excessive, testing of the MEA (% MEA, loadings, and heat stable salts) on a weekly basis has begun. As these responsibilities were undertaken by the Analytical Group, there was interfacing with the Special Problems Group.

M.S. Unit Support (J. D. Presley) - Work is underway to qualify T-288 lower draw off (LDO) for mixing with MO feed methanol. No problems are expected. A joint letter from the Laboratory and Process Engineering has been written detailing analyses and economic evaluation. A \$201M/year savings could be realized over present burn value of T-288 LDO.

The Shift Loop MEA system experience severe corrosion problems in early February. Acid gas loading determinations were made and found to be excessive, approximately 1.1 (corrosion is considered severe at 0.5). The MEA concentration was determined to be 4 to 5% high. When the MEA was reduced the corrosion problem subsided. A letter with complete analysis is in preparation.

Oxo, Butanol

J. D. Presley

Reactor Stability/Accountability - Overall reactor accountability is 90+% for February. Esters and other heavy ends have increased slightly for the month, approximately 0.5wt.%. TPP concentration has fallen to 29-30%. However, the TPP decrease was planned in compliance with increased isobutyraldehyde production.

003487

Rhodium Accountability - February rhodium accountability remains high at 95+%. Total rhodium losses out T-210 Residue are 2.24 pounds (as of February 20, 1981) for Run VII.

Improved Rhodium Recovery - A TI-59 program has been developed to speed intrinsic activity data calculation. The new nebulizer for AA has arrived and has been installed so that pending rhodium analysis can be completed.

Oxo Unit Support - Rhodium analyses of test coalescer samples have been completed and a letter has been written. Complete analyses shows butyraldehyde condensation was occurring. Hence, data was inconclusive and no recommendations could be made on the possibility of coalescer product return to the reactor.

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

The noise survey continued through the month, approximately 90% complete. (KVP)

The use of long term Dräger CO 50/a-L tubes for carbon monoxide 8 hr TW exposures is recommended. The analyses for validation are complete and the method is being written for submission to the Company validation Committee. (CEG)

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

Methyl Formcel MeOH Losses - A Laboratory and Process Engineering study to determine the methanol losses in the Methyl Formcel Unit is in progress. Two separate studies in recent years, one by the Laboratory and the other by Process, presented different conclusions. In the present study, both the Laboratory and Process agree to the methods of analysis. Results of the study will be published soon.

Trimethylolpropane
R. L. Day

T-312 Extractor Optimization - Documentation of the T-312 extractor base case data is still in progress. Previous work indicates that the incoming ethyl acetate temperature most strongly influences column temperature. Some of the future work will involve changing the temperature of the feeds to reduce losses. Data gathered also indicates that an increase in the ethyl acetate temperature or the base temperature will decrease the ethyl acetate and TMP losses out the residue. If the ethyl acetate and TMP losses out T-312 residue could be eliminated entirely, roughly \$58M per year could be saved with

003488

little or no capital investment. Other incentives to pursue this project include knowledge gained on the extractor for future expansions, smoothing out T-312 operations, possible increased capacity, and possible energy savings.

TMP Efficiency - A meeting was held Thursday, February 27, to discuss low TMP efficiencies and possible causes. The Laboratory will conduct a unit data surveillance to see if there are any correlations to the low efficiency. The Lab will also check some density factors of the WIP tanks and molten TMP.

Diacetone Alcohol
B. K. Huelle

Unit Support - None provided.

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

Minimizing U.V. Absorbance at 220 nm (Huelle) - Attempts were made to lower the absorbance of formaldehyde before its use in the PE reaction. Methylene chloride extraction lowered the absorbance; however, use of this lower absorbing HCHO (from either the PE feed tank or MO blending) in PE reactions did not lower the absorbance of the PE product.

The possibility that sources of PE HCHO feed besides MO cause high u.v. absorbance were explored. An extremely high u.v. absorbance was obtained on PE produced from a reaction using only HCHO from the weak HCHO unit. This result would imply that the increased use of HCHO from weak HCHO as feed in the PE reaction gives rise to a PE product with higher absorbance. Interestingly, tech grade runs which use more of this stream than in pure runs has consistently made higher absorbing PE product. Weak HCHO feed was also found to be the main contributor of color in "A" cake and AML from laboratory experiments. The reaction yield in the laboratory experiments dropped 20% when using strictly HCHO from the weak HCHO unit.

Studies are now underway to identify the components in the HCHO from the weak HCHO unit which cause the high u.v. absorbance (and low yield).

Lubricant Grade PE Plant Trial (P. J. Buras, F. R. Dow, H. A. Ducote, B. W. Wise) - Approximately 1.3MM lbs. of "lubricant grade PE" were produced during the Plant trial 2-6-81 through 2-17-81. Operating problems limited production rates to approximately 80% of capacity. The Laboratory provided analytical support which helped to monitor unit operations during the trial. A report documenting these results will be prepared following completion of sample analysis.

PE Formal Hydrolysis (D. R. Akin) - Work continues 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 H₂SO₄) as co-catalysts of the

003489

hydrolysis of PE mono linear formal. The resins exposure time to date is 212 days. No activity loss has been detected since reactivation of the resin after 120 days. The conversion of mono linear formals by pulse feeding the acid into the system proved to be no better than the conversion with continuously fed acid. Variable studies to determine the effect of acid concentration, sodium concentration, and residence time on conversion of mono linear formals will continue.

Dow High Formal/Color Complaint (P. J. Buras) - A meeting was held in Bishop on 2/19/81 to discuss PE quality and analytical procedures. Representatives of Dow Chemical (Michigan Division), Dallas Marketing and Quality Assurance, and the Southfield (Michigan) Sales Office attended. Dow believes that high PE linear formal in Celanese PE has an adverse effect on the color and performance of their product (dibromoneopentylglycol). They also said that pallet-to-pallet quality variation within the same lot had caused processing problems due to the small size of their reactors.

We suspect that unknown peaks in their GC scans, comparable in size to those of normally observed impurities, are causing the analytical discrepancy between Dow and Celanese. We do not see these extraneous peaks in our analysis. We again recommended that they use the revised Celanese SMA for PE which utilizes a different silylating reagent. A step-by-step demonstration of the Celanese procedure was presented.

A series of sample exchanges to resolve the analytical discrepancy was agreed to. Samples of competitive product which performs satisfactorily in their unit will be sent by Dow to us. Preshipment qualification samples of three lots from current low formal production will be sent to Dow. They agreed to order a truckload of PE for Plant trial if this material meets their approval.

We agreed to monitor unit operations during low formal production. A study will be made to determine the lot variation during periods of normal operation. We stated that it may be difficult to control lot variation within their specifications.

Identification of Material Plugging WIP/T-287 Heat Exchanger (P. J. Buras) - The brown material plugging the tubes was identified as rust. A dried sample contained 77 wt.% ash. The source and/or cause of the corrosion products in the WIP stream is unknown.

Eliminate VCI Centrate Waste Stream (P. J. Buras) - No progress due to priorities.

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.

003490

GDB-105-81
Page Thirteen
March 6, 1981

Miscellaneous
R. L. Day

Formaldehyde Stabilizers - 52% formaldehyde samples were set up in January to determine whether or not the polyvinyl alcohol/metolose stabilizer mix has synergistic effects.

The tests are as yet incomplete, and results will probably be available for the March monthly report.

003491

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	March, 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, 1981
Solar Pond Treatability Studies	Young Smith	1	Environmental Mandatory	July 1, 1981
Anaerobic Testing of Chemical Wastes	Smith	1	Support Unit Expansion	April 1, 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	March, 1981
EMP Extractor Optimization	Day	2	Raw Material Savings	Undetermined

003492

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
New Formaldehyde Stabilizer System	Day	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	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	Akin	1	Stream, Acid and Capital Savings	June, 1981
Develop a Way to Monitor Conversion in the PE Reaction System	Huelle ↓	1	MOB (Quality) and Raw Materials Savings	April, 1981
Determine the Effect of Sodium Formate on TMP Decomposition		2	MOB (Quality) and Raw Materials Savings	June, 1981

003493