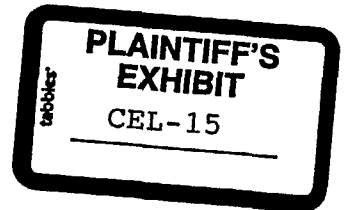


CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

From: G. D. Boyd

A
GDB-172-82
GDB-0172A-82

June 4, 1982

LABORATORY DEPARTMENT SUMMARY - MAY 1982

Attached is the Laboratory's monthly summary for ~~April~~ *May*. The summaries are by groups. A table listing priorities, justifications, and estimated completion dates of Special Problems projects is also included.

G D Boyd
G. D. Boyd

GDB:erg

Distribution:

Chemical

J. J. Fritsch
D. G. Pausky
W. H. Meyer
A. A. Miller
G. R. Scholz
M. D. Harvey
G. L. Cade
C. W. Lee
D. D. Siemonsma
W. L. Sharp
D. E. Lockwood
L. M. Harvey
H. H. Thigpen
A. G. McGehee
B. C. Kerr
F. R. Dow
W. P. Hightower
R. R. Carpenter
R. S. Hahn
J. C. Rubiano
K. R. Stelts
W. H. Brough

Plastics

A. C. Abshier
R. L. Bunkley
D. A. Reck
D. W. Steele
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
J. L. Paul, Clear Lake
D. W. Harris, Pampa
T. H. Golson, Dallas

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HIGHLIGHTS

- Addition of ~5 ppm high molecular-weight cationic polymer to solar-pond liquid produces a heavy, dense floc which settles rapidly. The amount of solids not removed by coagulation and settling does not appear to be reduced using the proposed pressure precoat filtration system.
- A pulse test of the anaerobic reactor was conducted on May 17 using a total-recycle tracer method. The void volume of the reactor was determined to be 74.3% below the overflow weirs or 66.8% in the packed region below the weirs.
- Paraform prills containing sodium hydroxide (1-10 ppm) have been dried to 95+% in 5-6 hours at 150°F. About 10-12% of the HCHO went with the drying air. This would be recovered in the scrubber.
- Silica gel has shown some promise as an absorbent for Versene scum formers in MO-2 HCHO produced while the unit was receiving T-185 O.H. from the weak HCHO unit.

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I. ANALYTICAL GROUP

Paraform Area

Ester Unit - The Analytical Group analyzed samples of fifteen tank cars of acetic acid from the Congrejera, Mexico Plant to be used in ester production. The acid was very high purity. Current production of Butyl Acetate utilizing this material meets manufacturing control specs.

Methanol Oxidation (MO)

MO-IV - The Analytical Group has developed a GC method for quantifying the impurities in MO IV middle recycle formaldehyde. A comparison study of the middle recycle stream and our recent 37% HCHO shipments indicates that the impurity levels in the recycle stream are less than, or comparable to those in our 37% HCHO shipments with the exception of methyl formate. A report detailing the results of this study has been published.

Utilities & BOD

BOD Reactor - Several samples of the BOD anaerobic reactor were analyzed for ppm COD in conjunction with the BOD reactor pulse test. A report will be published by the Special Problems Group.

Customer Complaints

5-6-82-Acme Resins-Oregon, Ill.-complained of a flange leak at the bottom outlet valve on a tank car receipt of 50% HCHO. The car will be inspected upon return to the plant.

5-11-82-Inolex-Philadelphia, PA-complained of yellow particles in 3 lots of PE-Pure (1700 x50# bogs). The material was returned to the Newark Terminal for sampling inspection and confirmation. Replacement shipment will be made from Newark pending checks on returned and inventoried products. Yellow particles are not present in our retained samples of these lots and were not noted on original analysis particle inspection. If present, they are likely produced in our PE drying operation and were not picked up during the bagging composite sample preparation. Production department personnel have been notified.

Quality Waivers

None this month.

POLLUTION CONTROL

Numerous samples of rain water from recent rain are being analyzed for TOC and COD. The data is being gathered for the Rain Water Task Force group to reestablish the maximum TOC levels that can be released to the Plant front ditch.

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PRODUCT QUALITY WAIVER HISTORY

	<u>% QD¹</u> <u>Month</u>	<u>% QD</u> <u>YTD</u>	<u>% SQD²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Methylal	0	56.8	0	0
PE-Pure	0	0	0	0.7
n-Propyl Acetate	0	0.3	0	0
Diacetone Alcohol	0	10.4	0	0
PE-Tech	0	0	0	0.3
Sodium Formate	0	0	0	7.3

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>May</u>	<u>May YTD</u>	<u>Previous YTD</u>
Chemical Section	4313	22,977	22,477
Instrument Section	2491	14,146	12,495
Total	6804	37,123	34,972

Technician Overtime

Chemical Section	38	279	381
Instrumental Section	39	263	207
Total	77	542	588

Sample Shipping

Domestic	47	318	298
Export	2	12	21
Plastics	0	4	6
Others	9	48	51
Total	58	382	376

QA Statistics

Shipments	507	2919	3238
Waivers (QD)	0	15	5
Waivers (SQD)	0	5	6
Complaints	2	10	3

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II. STAFF CHEMISTS

Process Technology
B. C. Kerr

Dow Scum Problem - A report has been issued which describes the causes and cures for the Dow Versene™ scum problem. Dow has started using Celanese Formaldehyde again.

High Concentration Formaldehyde For Sales - Marketing requested that the plant evaluate the potential to manufacture and sell 56% HCHO solutions. A joint letter, with R. Clos, was issued in which a production route was outlined. The letter outlined some of the more important questions which must be answered.

The production and marketing of HCHO solutions above 50% concentration is dependent upon obtaining an economically attractive system for producing the product and upon developing the necessary technology for storing and shipping the product. We believe that the problems associated with storage shipping and customer facilities must be solved prior to evaluation of production modifications. There is no question that we can produce the higher HCHO concentrations (at a cost); however, there is serious doubt concerning our ability to handle the product. The Laboratory is beginning a study of techniques for handling HCHO concentrations in excess of 52%.

Special Analytical
F.R. Dow, B.W. Wise

TMP Purity - Analysis of Celanese, Japanese and Perstorp TMP has shown that Celanese TMP has a lower assay. Based on capillary column GC analysis (instead of the SMA procedure) Celanese TMP averages ~ 98.5% Mono TMP while Perstorp TMP consistently analyzes above 99.3 wt.%.

The main difference between Celanese and its competitors is an unknown component that elutes immediately after the mono-TMP. A coordinated investigation with CCCTC will attempt to identify this impurity.

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MS Methane Feed - During the week of May 19-28, 1982, five different samples of MS methane feed were taken for analysis. A summary of the component concentrations shows the following:

	<u>5-20-82</u>	Male %	<u>5-28-82</u>
Methane	92.2	increased to	94.7
Ethane	4.3	decreased to	3.1
Propane	1.4	decreased to	.7
BTU Value	1073.		1046.

The changes in the methane, ethane and propane concentrations were gradual during this period. After the analysis had been completed, it was learned that the volume of gas from 2 of our suppliers had been altered.

Additional data is being obtained and will be reported when the investigation is complete.

III. SPECIAL PROBLEMS

Group I - H.H. Thigpen

Pilot Study - H_3PO_4 Extraction of Oxo Run VI Catalyst Solution
(T.A. Jasek) - Aldol studies have been completed on the Oxo catalyst solution from Run VI after one, two, and three washes with water. The butyraldehyde conversion was followed to determine the effects due to the "pH" (measured in a methanol-water solution) change noted after each washing. The "pH" increased from 2.55 to 4.05 from the first wash through the third. This compares with a "pH" of 4.2 of the current Run VII. The butyraldehyde conversion rate decreased with an increase in "pH". The catalyst washed for a third time exhibited a butyraldehyde conversion rate of approximately twice that of the Run VII catalyst solution.

At least one more wash is planned on the H_3PO_4 extracted Run VI solution. Additional washes will be used to determine a "pH" to which an operational wash should be carried.

Heavy Ends Build in Oxo Reactor and Purification Train - The Laboratory has begun a study to determine the heavy ends build rate in the Oxo reactor and the butyraldehyde purification train. This work was requested by Process Engineering to calculate the economics of using poly alpha olefins in the Oxo Reactor as makeup on startup. Only very preliminary results have been obtained to date.

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Oxo Catalyst to Reclaimers - The Laboratory completed the analysis of a composite of ten drums of Run VI catalyst solution sent to Johnson-Matthey, Inc. for a trial recovery.

Also the Laboratory received two samples of ash from JMI for moisture analysis and assay. These analysis are currently underway.

Analytical Method for the Oxo Reactor Catalyst Solution - A modified method, based on an internal standard, has been developed for analyzing the Oxo Reactor solution. This method has been implemented in the Instrument Section in the Laboratory.

Paraformaldehyde, Formcels®, Esters
P. W. Young, J. E. Russell

An attempt to relate prill drying rate to prill bed temperature was not successful. Each drying curve was mathematically fitted to an equation of the form $y = mt^2 + b$, where y = assay, t^2 = time, minutes, m = slope of the line and b is the intercept. Each m was then regressed against temperature. There seemed to be a good correlation with temperature and with starting assay.

NaOH has been used as an additive in the 1-10 ppm range. There are definitely some physical property changes that have allowed drying to 95% with lower losses (10-12% vs ~ 50% with no additive).

At 100°F, the drying rate does not seem to be significantly affected by the NaOH. However, some prills have been dried to ~96% in 6 hours at 150°F with only 12% losses. Those prills had been aged for about 4 days. Fresh prills were dried to 95% in less than 5 hours with no aging and only a 10% loss. The bed temperature was approximately 145°F. No further conclusions can be drawn until the NaOH addition study is completed.

Plans are to determine if the additive effect is simply a pH effect and to determine the optimum level of additive.

OSHA
C. E. Gary, K. V. Parker

1. Additional noise exposures were obtained on Operations personnel to assist in determining the need of engineering controls. (Report CEG-63-82, KVP-18-82).

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2. The initial noise exposure survey of Maintenance personnel should be complete by June 15, 1982 (KVP).
3. The Laboratory validation of chromotropic acid methods for determining formaldehyde in the atmosphere is approximately 50% complete (CEG).

Methanol Oxidation, Formaldehyde Blending

J.F. Glenister, P.W. Young

Summary of Product Color Trends in the MO Units Glenister) - MO II product color was good at <5 APHA with the exception of 4 days sporadic high color. MO III and MO IV product color was very good at <5 APHA.

Development of Method for Analysis of Formaldehyde in MO IV Absorber VENT STREAM - A method for the determination of formaldehyde in MO IV absorber vent is being developed utilizing the reaction converting formaldehyde to hexamine (hexaminethylenetetramine HMTA) by reacting it with Ammonia. The resultant HMTA may be readily analyzed using Gas Chromatography. Previous studies related to the BOD system show this reaction of HCHO and ammonia to form HMTA is rapid and quantitative at ordinary temperatures. Current objectives include developing a GC method and verifying the reaction can be monitored quantitatively. The GC method has been developed and work is being completed to verify the accountability of the reaction to insure the reaction proceeds to completion and the method is reliable in quantifying various concentrations of formaldehyde.

Analysis of Additional Samples from MO IV Flame Arrestor Blowdown (Glenister) - D.M. Estes requested wt.% HCHO assays on samples from the blowdown of the Flame Arrestor at MO IV, at two different blowdown rates. The analyses were completed by the Routine Analytical section and results showed essentially no difference in the formaldehyde concentration at these different rates. The formaldehyde concentration of samples at a rate of 1.7 gpm showed 2.09 wt.% with 2.5 wt.% at a rate of 2.60 gpm.

Stability Studies of 37 wt.% Unstabilized, Uninhibited Formaldehyde at $\sim 81^{\circ}\text{F}$ and $\sim 90^{\circ}\text{F}$ (Glenister) - The stability of unstabilized, uninhibited 37 wt.% formaldehyde is being examined to recommend an optimum storage temperature for typical product shipped to Dow, Freeport. MO IV product from storage tank 2587 is blended to ~ 37 wt.% and placed in an oven at the appropriate temperature. Several different production runs will be examined to determine the optimum storage temperature.

Group II - L.M. Harvey

Waste Management
B. K. Huelle

Removal of Suspended Solids from Solar Ponds Prior to Deep-Well Injection (Huelle) - During the past several months, Process Engineering has been developing a process to remove suspended solids from the solar ponds which will be injected into deep wells. Their investigation has led to a precoat pressure filtration system using diatomaceous earth (DE) as the precoat and filter aid. This system however, requires large amounts of DE, and handling facility costs are high. The Laboratory was asked to look into alternative filtration processes, or alternative filter precoats and filter aids for the present system, and/or pretreatment of the solar ponds.

As alternative filter precoats and aids, Kaolin clay and "Fibra-cel" (cellulose fiber) were investigated. The Kaolin clay broke through the precoat and is not suitable for the system. "Fibra-cel" was just as effective as DE but is five times the cost of DE. Other tests will be conducted with fly ash from the Pampa Plant as a filter aid.

More novel ways of removing suspended solids from the ponds such as ultrasonic vibration and electrolytic treatment indicated no significant change in suspended solids concentration. Lower pH and higher temperatures also had no effect on the filtration process.

Pretreatment of waste water before filtration is common practice according to the literature. Chemical coagulation and flocculation, with the use of aluminum or iron salts and/or cationic polymers, are often used to help lower suspended solids concentration. Aluminum sulfate (alum) at 500 ppm level produced a fine floc which took 2 to 3 hours to settle. A high molecular-weight cationic polymer at 5 ppm level produced a heavy, dense floc, most of which settled in under 5 minutes.

Samples of the ponds left standing for long periods will eventually show solids accumulation. The rate of accumulation depends on the initial suspended solids concentration. A sample was left standing to completely settle for several days. The TSS (total suspended solids) of the decanted liquid was unchanged after precoat filtration treatment. Preliminary data indicates that suspended solids left after settling or flocculation will not be removed by the precoat system.

Work is underway to find the best cationic polymer to achieve coagulation and flocculation. Also, further removal of the suspended solids left in solution after coagulation will be attempted.

Total-Recycle Pulse Test (Akin, Kaderlik) - A total-recycle pulse test was conducted May 17 on the anaerobic reactor to determine the liquid void volume. The liquid void volume determined was 0.988 MM gal. This corresponds to a 66.8% void volume in the packed region of the reactor (or 74.2% void volume in the entire region below the weirs).

Vacuum Truck Content Characterization (Akin) - This characterization is complete and will be documented shortly.

VCI/Polyol Treatability (Akin, Kaderlik, Stasney) - An anaerobic-aerobic system is being fed a 18.5 wt.% Polyol Effluent COD, 4.5 wt.% VCI Effluent COD, with the balance being equalization-basin liquid (equates to 10,250 lbs Polyol COD/day, 2500 lbs VCI Cod/day). The organic loading is 0.32 lb COD/ft³/day with a kinetic efficiency of ~ 50% and an anaerobic outfall COD of ~2200 ppm. The aerobic outfall COD for May ranged from 250 to 600 ppm at a detention time of 2.7 days. The plan for this unit is to continue to increase the polyol COD contribution until the unit no longer operates at good stability or a target Polyol concentration of 50 wt.% COD is reached.

TOD/COD Correlation of VCI Effluent (Kaderlik) - A study of the relationship between TOD and COD of VCI effluent began in April and continued through May. This study will allow approximate daily TOD poundage to be determined from VCI's COD analysis of their effluent. VCI will be responsible for controlling their effluentflow to BOD based on daily TOD poundage values established by Celanese. A report will be issued before installation of the pipeline to carry VCI effluent is complete.

Utilities Unit Support (Kaderlik) - The Demineralizer II weak-base resin has been exhibiting short service runs. Lab analysis of the resin revealed incomplete regeneration by the Demineralizer II train. The extent of regeneration is approximately 59%. A report has been issued (KRK-02-82).

Methanol Synthesis
R.L. Day

Rapid Carbon Deactivation (Day) - The rapid deactivation of the carbon guard beds in the MS Unit continues to be investigated. Analyses on four spent-carbon samples obtained from the unit show sulfur capacities and bulk densities typical of good carbon samples. Solvent extractions of these four carbon samples did not increase the sulfur capacity.

Attempts to identify the sulfur compounds in the methane feed have been hampered by instrument problems.

Carbon Monoxide Analysis through "Reformer and Shift Sections"
(Glenister) - Process Engineering requested carbon monoxide analysis after the reformer, high temperature converter, sulfur beds and the low temperature shift. Standards were available for carbon monoxide analysis of the stream after the high temperature converter, but it was necessary to blend and verify a primary standard for the analysis of CO after the reformer (~15%). Two standard blends were prepared and checked against each other. Agreement was within 3%, but the accuracy of the results requires much closer agreement. Additional standards will be prepared and analyzed in attempts to obtain the best primary CO standard (1% relative) at the higher CO levels.

Trimethylolpropane
J.C. Bustabad, B.K. Huelle

TMP Expansion (Bustabad) - Sodium Formate (NaFo) backwashing from TMP has been completed at various temperatures and wash-water rates using ethyl acetate-trimethylolpropane (EtAc-TMP) that was 10% TMP. The studies confirm earlier findings that the two-stage backwash at lower temperatures (in this case 77°F vs 109°F) resulted in a significantly lower concentration of NaFo (<1 ppm) in the EtAc-TMP organic phase. This was accompanied by an increase of TMP in the aqueous phase. Reducing wash-water rates decreased TMP in the aqueous phase, but this was accompanied by an increase of NaFo concentration in the EtAc-TMP organic phase.

Laboratory work is now in progress using a TMP feed that is 15% TMP (vs. 10% used in previous studies) for the NaFo backwashes.

Recover TMP From V-747 Residue (Huelle) - No progress due to priorities.

Diacetone Alcohol
R.L. Day

DAA Color Build (Day) - A cryogenic nitrogen (N₂) blanket was placed on the DAA inventory tank at the end of April. Previous lab work had shown a 70% reduction in color build with a N₂ blanket vs. the plant air blanket normally used.

The color build of the N₂ blanketed DAA in the inventory tank at the end of the production run in late April was compared to the color build of the plant air blanketed DAA in the inventory tank at the end of the production run in mid-February. The production rates and level in the inventory tank were similar for both runs. Results showed a 37% reduction in the rate of color build per day with the N₂ blanketed DAA.

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The inventory tank is being checked for leaks, and the method used for flushing the tank with N_2 has been changed. The Lab will investigate the DAA color build during the next production run (the DAA Unit started up May 27).

BOD, UTILITIES

D.R. Akin, K.R. Kaderlik, G.R. Stasney

Polyol Treatability (Akin, Kaderlik, Stasney) - A Laboratory anaerobic-aerobic system is being fed 16.7 wt.% Polyol and 83.3 wt.% equalization-basin liquid (equates to 8500 pounds of Polyol effluent COD/day). The organic loading is 0.29 lb. COD/ft³/day with a kinetic efficiency of ~65% and an anaerobic outfall COD of ~2000 ppm. The aerobic detention time for the waste is 2.9 days with May outfall CODs ranging from 220 to 300 ppm. The plan for this unit is to investigate the feasibility of digesting aerobic sludge blowdown in the anaerobic reactor.

BOD II Unit Support (Akin) - The anaerobic reactor was operated at an average organic loading of 0.24 lb COD/ft³/day during May. Anaerobic COD removal efficiency ranged from 65-73%. The outfall from the BOD II unit averaged 285 ppm COD. On May 17th a pulse test was conducted on the anaerobic reactor (operated in total-recycle mode of operation) to determine the liquid void volume. After 23 hours of total-recycle operation the feed was started back in at full rates. There have been no ill effects to date from this startup.

GENERAL

L.M. Harvey attended a meeting on May 4 at Bay City Laboratory which reviewed results of BCP evaluation of Degussa Raney-Nickel catalyst. Degussa technical representatives were present.

G.D. Boyd, F.R. Dow, L.M. Harvey and H.H. Thigpen gave presentations at the Bishop MMT meeting on May 11.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TMP Expansion	Bustabad	1	MOB	December, 1982
TMP Extractor Optimization	↓	1	Raw Materials Savings	July, 1982
Condensing Polyol Vents		2	Cost Reduction	July, 1982
Optimize Solar Pond Filtration Prior to Deep-Well Injection		1	WMSF Support	July, 1982
Recover TMP from Formals in TMP Heavy Ends (V-747 Residue)	Huelle	1	Raw Materials Savings	July, 1982
Improve PE Quality - UV Absorbance and Formal Content	↓	1	MOB	July, 1982
Evaluate Alternate Filter Cloths for PE filters		2	MOB	October, 1982
Methanol Personal Exposure Monitoring	Gary	1	MOB (Environmental Health)	September, 1982
Area Surveys for Misc. Organics	↓	2	MOB (Environmental Health)	February, 1983
Industrial Health Formaldehyde Methods Validation		1	MOB (Environmental Health)	July, 1982
Maintenance Noise Exposure Monitoring		1	MOB (Environmental Health)	June, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Glenister	2	To Establish Alternate Catalyst	Continuing
GC Methanol Oxidation Reactor Optimization		2	Catalyst Evaluation for Possible Efficiency Improvements	November, 1982
Modified Silver Catalyst		1	Catalyst Efficiency Improvements	May, 1982
Formaldehyde Color Problem		1	MOB	Continuing
Confirm MO Unit Efficiency Losses Via Vent Sampling and Anal.		1	MOB, CR	June 1, 1982
Stability of 37% Unstabilized HCHO (for Dow Chemical Co.)		1	MOB	July, 1982
Oxo Run VI Cleanup (H ₃ PO ₄ extraction-wash)	Jasek	1	Cost Reduction	July, 1982
Rhodium Accounting		1	MOB, CR	Continuing
Determine Oxo Unit Losses to Heavy Ends		1	CR	September, 1982
Support Paraform Prill Pilot Operation at Bishop	Russell	1	CR, MOB	July, 1982
Support Flocculant Plant Trial	Kaderlik	2	MOB	October, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TPP, P ₂ O ₅ Emissions	Kaderlik	1	Emissions Quantification	July, 1982
Evaluate "Unsteady-state" Polyol, VCI and other effluents	Kaderlik/ Akin	1	MOB	September, 1982
BOD II Aerobic Optimization	Akin	1	MOB	June, 1982
Vacuum Truck Characterization	↓	1	WMSTF Support	June, 1982
Evaluate ATP, OUR and Dehydrogenase Methods	↘	2	MOB	July, 1982
Recover PE from VCI Waste Stream	Day	1	Cost Reduction	September, 1982
Determine Cause of MS Carbon Bed Deactivation	↓	1	MOB	June, 1982
Formaldehyde Stability	↘	2	MOB	June, 1982
Removal of Impurities from Formaldehyde	Young	1		July, 1982

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