PLAINTIFF'S EXHIBIT
tabbies
CEL-13

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

From: G. D. Boyd GDB-170-82 April 12, 1982

LABORATORY DEPARTMENT SUMMARY - MARCH 1982

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

G. D. Boyd

GDB:jr

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
M. W. Clifton
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

File No: 303.3

003664

HIGHLIGHTS

- In the phosphoric acid extraction of amine contaminated Run VI Oxo catalyst solution, a new washing technique gives short phasing time (overnight vs 3 weeks for previous technique). Evaluation of a second pilot batch with two water washes is underway.

- TPP from C. Itoh received for Plant testing, meets purchase specifications. The Plant test will be scheduled.

- Paraform prill fluid bed drying tests at 40°C showed that prill size affects the drying rate and maximum assay attainable in 4-5 hours drying time. The target assay of 91-92% HCHO is reached in 2-4 hours.

- A pulse test of the Celrobic® anaerobic reactor was conducted on March 9. Data from the test will be sent to Dr. Joel Hougen of Austin for interpretation.

- A vertical COD profile of the Celrobic anaerobic reactor was conducted on March 8. Results indicated that biodegradation is occurring primarily in the first 6-7% of the reactor. A report has been written.

- Characterization of vacuum truck contents was resumed after issuance of a "clean truck" policy which included inspection procedures.

- The new boiler which receives liquid waste streams as fuel (Boiler #16) was shutdown due to mechanical problems. Laboratory analyses of ash samples indicated incomplete combustion.

- A one-gallon sample of crotonaldehyde (CrH) was sent to Nepera Chemical Co. CrH is a potential coproduct in the 1,3-BG Unit. The sample was obtained from a unit stream with final processing steps being performed in the Laboratory.

003665

I. ANALYTICAL GROUP

Competitive Products

The Analytical Group analyzed samples of anhydrous sodium formate from Hercules and Perstorp for competitive evaluation. Samples were provided by Virginia Chemical Co. A comparison report with Bishop product will be issued.

Paraform Area

Methylal - The Analytical Group provided around-the-clock coverage for rerunning approximately 35000 gallons of methylal that had been contaminated with propyl acetate.

Utilities - BOD

The Analytical Group provided analyses to determine the COD reduction across the anaerobic reactor. A report will be issued by the Special Problems Group.

The Analytical Group provided extensive analytical support for the recent "Pulse Test" on the BOD anaerobic reactor. The Special Problems Group is gathering and organizing the data. The data will then be forwarded to an outside consultant for evaluation.

Customer Complaints

3-16-82 - Thompson Hayward Co., Chicago, IL and Tab Chemicals, Inc., Chicago, IL - Complained of foaming while drumming 37-7% HCHO from Argo Terminal car Celx 13445. This car shipped from Bishop with higher than normal levels of Metolose™ stabilizer (approximately 35 ppm) which may have contributed to the foaming.

3-25-82 - Dow Chemical Co., Midland, MI - Complained of end product color from PE Pure Lot 82-48-3P. This lot met all sales specs. including Dow's special spec. of 0.15 wt.% max. linear formals. Work is underway to resolve the problem.

Quality Waivers

2-25-82 n-Propyl Acetate - Delta Solvents, Dallas, TX-n-butyl acetate content at 0.21 wt.% vs. max. MCS of 0.20 wt.% - 18,570 lbs - QD

2-26-82 - Methylal - Duolite International, Inc., Redwood City, CA-150,680 lbs-QD

2-26-82 - Grant Chemicals, Irene, PA - 30,620 lbs - QD

3-1-82 - Celanese Bayport Terminal - 3,580 lbs - MeOH content at 1.3 vs. max. MCS of 1.0 wt.%

3-9-82 - Diacetone Alcohol - Celanese Newark Terminal - Color 12 vs. MCS of 10 Pt-Co Units - 158,820 lbs - QD

3-12-82 - 37-0 HCHO - Petro Chemicals Co., Inc. -Ft. Worth, TX-MeOH content at 1.2 wt.% vs. Sales Spec. of 1.0 Max. - 45,060 lbs - SQD

3-16-82 - Diacetone Alcohol - Delta Solvents, Dallas, TX - Color 17 vs. 0 max. MCS - 19,670 lbs - QD

3-23-82 - PE Tech - PPG Industries, East Point, GA-Combined di-PE and tri-PE = 9.85 vs. customer special spec. of 10.0 wt.% min.-32,500 lbs-SQD

3-25-82 - Diacetone Alcohol - Cone Solvents, Memphis, TN-Color 23 vs. CS of 10 max. - 7,830 lbs-QD

Product	% QD[1] Month	% QD[1] YTD	% SQD[2] Month	% SQD YTD
Methylal	83.7	99.4	0	0
PE-Pure	0	0	0	1.2
n-Propyl Acetate	2.9	0.6	0	0
Diacetone Alcohol	70.0	15.3	0	0
Formaldehyde-Uninh.	0	0	0.3	0.1
PE-Tech	0	0	1.8	0.5

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

	Mar.	Mar. YTD	Previous YTD
Sample Count			
Chemical Section	5151	14046	12872
Instrument Section	3832	9178	7293
	8983	23224	20165
Technician Overtime			
Chemical Section	46	195	262
Instrument Section	95	190	108
	141	285	370
Sample Shipping			
Domestic	64	184	176
Export	3	8	14
Plastics	1	3	4
Others	15	29	34
	83	224	228
QA Statistics			
Shipments	589	1787	1898
Waivers (QD)	7	14	3
Waivers (SQD)	2	3	2
Complaints	2	5	3

II. STAFF CHEMISTS

Process Technology
B. C. Kerr

Laboratory Synthesis of Versene™ (EDTA) - A "performance" type synthesis for Versene has been developed. The test procedure has been used to evaluate various types of impurities which are present in Celanese formaldehyde solutions. The following compounds have been shown to be "scum" formers:

1. Metolose™ stabilizer @ 20 ppm
2. Diglycol dilaurate antifoam @ 10 ppm
3. General Electric AF-60 antifoam
4. Waxes from crude methanol
5. High molecular weight formaldehyde polymers
6. Natrosol 250-LR stabilizer @ 100 ppm

Routine testing of unstabilized MO IV product indicates that this material is consistently free of "scum" in the Dow process.

Work is continuing on this project.

Control of Stabilizer Addition to Formaldehyde - Mr. A. G. McGehee has gathered stabilizer analyses on formaldehyde blend tanks for the period 9-14-81 through 3-19-82. These data show that a very serious problem exists in manufacturing control of stabilizer addition. Preliminary analysis of the analytical data show that stabilizer control deteriorated from bad to worse about the 8th of January, 1982.

A letter is being written proposing significant modifications in the stabilizer addition system.

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

Distillation and Analysis of Methanol Recovery Streams (Dow, Wise, Jasek) - Samples of methanol recovery streams that are ultimately used for MO feed were blended and distilled for component identification and distribution. The samples were blended to simulate two operational modes of the MS purification system with constant flows from MO III and IV vaporizer blowdowns and weak formaldehyde. This work is the result of several informal meetings that have discussed formaldehyde quality problems such as high color and the Dow "scum" problem. The formaldehyde quality problems may be the result of impurities in the methanol feed to the MO Units. The laboratory work is almost complete and a report will be issued.

MO IV "A" Train Vaporizer Material Balance - A material balance is being made and the MO IV "A" Train Vaporizer in an attempt to determine the hydrocarbon (waxes) and/or other high boiler carryover. High molecular weight hydrocarbons are being found on the MO IV product filters confirming that the vaporizers do have heavy end carryover. The analytical is not complete, but preliminary data is showing that there is little or no difference between the component composition of the vaporizer feed or overhead. A report will be issued detailing a material balance around the "A" train vaporizer.

Dow "Scum" Problems (Dow) - The Laboratory methylene chloride "scum" test was used to evaluate the "scum" formation potential of crude methanol and the upper and lower draw offs from the MS purification system. The experiments used formaldehyde from the shipment to Dow Chemical Co. (2-2-82) as the base case sample and 25 ml of each of the streams listed above. As reported previously, the Dow shipment failed the lab "scum" test because it was not free and clear of surface film, so this sample was selected as the base case because it would amplify the effects of any potential "scum" producing component.

The Dow shipment plus the impurities gave the following methylene chloride "scum" test results:

Dow + crude methanol - 100% coverage w/light scum
Dow + T-288 OH - 100% coverage w/medium film
Dow + T-288 Upper Draw Off - 100% coverage w/light film
Dow + T-288 Lower Draw Off - 50% coverage w/light scum

III. SPECIAL PROBLEMS

Group I - H. H. Thigpen

Methanol Oxidation, Formaldehyde Blending
J. F. Glenister, T. A. Jasek

AMR's (Glenister) - MO II Startup March 1, 1982

Indicated	Analytical
0.965	0.782*
0.965	0.907
0.965-0.968	1.04
0.99 -1.00	0.984

* Adjusted flow rate to sample system.

Summary of Product Color Trends in the MO Units (Glenister) - MO II product color was very good. It ranged from <5 to 5 for the entire month. MO III product color experiences an upward trend with fluctuating colors. The Weak HCHO methanol stream (T-185 OH) continued into MO III on MO II startup with the T-240 stream being returned to MO II. Product color at MO III ranged from 5 to 12 APHA, with average colors approximately 6-7 APHA. Operations switched T-185 OH to MO II on March 23 and the MO III product color decreased to <5 by March 27. Presently the unit is on spec. product of <5 APHA.

Sampling of T-33 Scrubber Vent Stream (Glenister) - The sampling procedure has been improved and an analytical method has been developed and evaluated. Sampling has started. Plans include completing sampling and analysis during the next month.

Evaluation of T-33 Packing Ring (Glenister) - As a result of the plugging of the MO IV absorber, Operations requested that the Laboratory check to determine if the packing in MO IV T-33 absorber can withstand 40# steam temperatures in the event steam would be used to clean the absorber after any future plug ups. The Laboratory examined the ring material by 1) placing it in the steam bath for 2 hours, 2) placing it in a 106°C oven for 4 days and 3) heating a section to 130°C in a butylene glycol oil bath and monitoring for any softening or deformation. The packing was unaffected under these conditions and retained its original shape and texture. No further work is planned.

Methanol Loss in Absorber Vent at MO III (Glenister) - Operations requested additional vent analysis to monitor methanol loss in the absorber vent. The vent sample of March 22 showed 6.3 mol.% MeOH. Operations (D. V. Shattuck) noted the absorber was experiencing cooling problems due to some fouled heat exchangers and adjusted the top absorber temperature to 66°F from approximately 80°F in attempts to reduce the MeOH loss. Additional vent analysis, following these operational changes, showed a decrease in MeOH loss to a value of 2.3 mol.%.

"Scum" in Dow Versene™ (Jasek) - Infrared spectroscopy (IR) examination of the original Dow Versene with scum and numerous other "scums" generated was continued. Several sources of scum were determined. Evaluation of these sources in the lab prep of EDTA were conducted by B. C. Kerr. The scums, if any, were analyzed by IR. Scums similar to the Dow scum were observed with the following materials added to the lab prep of EDTA: T-185 OH, Metolose, and bis-ethylene glycol dilaurate.

Oxo, Butanol

J. C. Bustabad, T. A. Jasek

Rhodium From Boiler House I Clean-Out (Bustabad) - Analyses of solid samples from the clean-out of Boiler House I showed rhodium to be 0.07%. Nickel and iron averaged 3.4% and 3.0% respectively. The concentration of rhodium was considered insufficient to pursue reclaiming alternatives.

Pilot Study - H_3PO_4 Extraction of Oxo Run VI Catalyst Solution (Bustabad, Jasek) - Pilot Batch II is in progress. This batch was water washed two times. The water washes were done by recirculating the water only and spraying it over the surface of the catalyst. Phasing time of water and catalyst solution is acceptable (overnight). Activity, rousing and aldol tests are in progress on catalyst samples after the first and second water washes.

C. Itoh TPP (Bustabad) - Seven drums of C. Itoh TPP have been received for a Plant trial. A composite sample taken of this material (Lot #110072) showed acceptable quality in purchase specifications. A letter detailing this work has been submitted for review.

Oxo Catalyst Solution to Reclaimer (Jasek) - The Laboratory is determining the Rh concentration in three samples of Oxo reactor catalyst. When the Rh analysis are completed these samples will be sent to reclaimers for recovery evaluation.

Paraformaldehyde

J. E. Russell

Paraformaldehyde Prills - Preliminary drying tests on paraformaldehyde prills produced in the Laboratory indicate that particle size may have a significant effect on final assay. Under similar

conditions, initial tests show that prills with an average diameter of ≅600 μm (microns) dry to a higher assay than prills with an average diameter of 1200 μm. The difference is on the order of 1 wt.% in the 91-93 wt.% HCHO range. As a result of this finding, two more fluidized bed dryers were constructed so that three particle sizes may be evaluated simultaneously.

Plans are to continue drying tests with and without additives before studying the effects of additives on solubility, hardness, and ageing.

GROUP II - L. M. Harvey

OSHA

C. E. Gary, K. V. Parker

Noise Exposure Survey (Parker) - The monitoring of Maintenance personnel for noise exposure is approximately 85% complete. This work was temporarily suspended to obtain more data on Operations personnel to pinpoint the need for engineering solutions to noise problems.

Exposure Data Files (Parker) - Computer print-outs of exposure data are being checked and filed.

Formaldehyde Methods Validation (Gary, Parker) - The evaluation of variations of Celanese chromotropic acid-formaldehyde methods is complete and the report has been issued (CEG-61-82). Work has begun on validation and comparision of the recommended Celanese method and the NIOSH method.

BOD - Utilities

D. R. Akin, R. K. Kaderlik, P. W. Young, G. R. Stasney

BOD II Support (Young, Akin) - Continued support was provided to BOD II operations. Polyol addition is up to 5000 lbs. polyol COD/day. A vertical COD profile was obtained on the anaerobic reactor. A report is to be published. A pulse test was performed on the anaerobic reactor. Samples are being analyzed for the tracer. A report will be published upon completion of the analyses.

VCI/Polyol Treatability (Young, Akin, Stasney) - An anaerobic-aerobic system is being fed a feed of 7 vol.% polyol (COD = 20,000 - 40,000 ppm; this equates to 7800 - 16,700 lb/day), 5.5 vol.% VCI (COD = 4000 - 8000 ppm, this equates to 1100 - 3100 lbs/day and 87.5 vol.% equalization basin and nutrients (COD = 6000 - 8000 ppm, or 32,400 - 43,200 lbs/day). The organic loading (OL) is 0.28 lb. COD/ft^3/day with a stable kinetic efficiency of 50-55% and anaerobic outfall COD of 1900 - 2500 ppm. The aerobic outfall COD is 260 - 350 ppm at a detention time of 2.7 days.

Polyol Treatability (Young, Akin, Stasney) - An anaerobic-aerobic system is being fed of 7 vol.% polyol (COD = 20,000 - 40,000 ppm, or 8,300 - 17,000 lb/day) and 93 vol.% equalization basin and nutrients (COD = 6000 - 8000 ppm, or 32,400 - 43,200 lb/day). The OL is 0.30 lbs.COD/ft^3/day with a stable kinetic efficiency of 61-67% and anaerobic outfall COD of 1900 - 2500 ppm. The aerobic outfall COD is 280 - 300 ppm at a detention time of 2.75 days.

Vacuum Truck Content Characterization (Akin) - The study resumed on March 22 following the issuance of an inspection procedure for cleanliness. One truck has been rejected since the beginning of this procedure. The procedure and study are proceeding smoothly.

Biodegradation of Branched Polyols (Akin) - The sample of solar pond 11E which contained bacteria shows signs of microbe proprogation (small flocs can be seen in the aerated liquid). Thus far growth has been slow. The only feed source to this Lab aerobic unit has been a dilute solution of PE and TMP in water. Another Lab aerobic unit containing bacteria from a soil sample is also being fed PE and TMP. Growth in this unit is slower than the growth in the unit containing solar pond 11E. Work will continue.

Analysis of Material Found in Boiler #16 Firebox (Akin) - The new liquid incinerator was shutdown on March 20 due to a mechanical problem. After cool down the incinerator's firebox was entered and samples were removed from the wall, floor and boiler tubes. Analyses of these samples indicated varying amounts of the following compounds: sodium (1-2 wt.%), nickel (0.1 wt.%), phosphates (5-20 wt.%), carbon (1-2 wt.%), chromium (trace), nickel (trace), and silica (major portion). These analyses indicate incomplete combustion within the firebox.

1,3-Butylene Glycol

R. L. Day

Crotonaldehyde Sample for Nepera - Nepera Chemical Co. is currently evaluating crotonaldehyde (CrH) as a substitute raw material for acetaldehyde (AcH) in their production of α-picoline. Nepera requested a 1-5 gallon sample of our potential CrH product for laboratory evaluation in their process.

Initial plans were to obtain the CrH sample from the residue of the acetaldehyde recovery column (T-135). Previous samples of T-135 res, after cooling, showed CrH levels high enough to surpass the solubility limits in water and cause phasing of the CrH in a "oil" layer. However, due to the current reduced rates and high efficiencies in the 1,3-BG Unit, sufficient CrH to promote phasing was not present in T-135 residue. Considerable effort was required to obtain the minimum 1 gallon sample requested, as it was necessary to distill 25 gallons of T-135 res in order to obtain 1-1/2 gallons of CrH. A report has been issued (RLD-31-82). The sample was sent March 19 (LMH-106-82).

Trimethylolpropane

R. L. Day, B. K. Huelle

Condensing Polyol Vents to Send To BOD (Day) - Progress on this project was interrupted by priorities this month, but work will resume in early April.

TMP Expansion (Day) - A Laboratory recommendation for the future expansion of the TMP Unit calls for modifying the purification train to this sequence: a preflasher (to remove light ends), a heavy-ends removal column, and a light-ends column where the finished product is removed as a liquid sidestream. Future Laboratory work will center on evaluating these modifications. A Laboratory apparatus modeling a part of this purification train has been built.

Methanolysis of Hydrolysis of V-747 Residue (Huelle) - Work continues to evaluate different processes for converting bis-TMP linear formal in V-747 Residue to TMP. Past work using T-205 OH as a diluent to V-747 Res. was done at a 1:1 wt. ratio; experiments using a 6:1 ratio of V-747 Residue to T-205 OH shows no decrease in yield. Many experiments using V-747 feed in a neat hydrolysis process were completed. However, these experiments are questionable because of analytical problems associated with storage of V-747 feed that were later discovered. Apparently, V-747 feed component concentrations change with time. To reslve this problem, the feed to the resin bed will be analyzed immediately before its use.

Pentaerythritol

R. L. Day, B. K. Huelle

Minimizing High UV Absorbance in Pure PE (Huelle) - Work to isolate and identify the UV absorbing compound(s) in pure PE continues. A sample of a LC trapped component from 5% PE solutions with high UV absorbance ws sent to the Tech Center. The sample was not concentrated enough for effective NMR and GC/MS data.

In order to provide a more concentrated sample for identification, 20 liters of 5% PE solutions with high UV absorbance were ran through an XAD-2 resin bed. Past work had shown that this procedure had removed the UV absorbing material fromt he PE solutions. The LC chromatograms of the feed and the product from the resin bed indicate the disappearance of 2 components previously correlated to UV absorbance. The compounds were eluted off the resin with methanol and three fractions were collected. The second fraction had a yellow color and an extremely high UV absorbance at 220 nm. Analysis of the fraction by LC showed two peaks which were individually trapped. One of these trapped components had a high UV absorbance. THis material will be concentrated and submitted to the Summit Laboratory for identification using FTNMR, FT-IR, and/or GC-MS techniques.

Viscosity Data For VCI (Huelle) - Viscosity data was determined on samples of the "First Effects" and "Centrate Return" streams from the VCI sodium formate unit during a recent rate run using TMP sodium formate.

GENERAL

Jane Bustabad attened an Oxo Technology Exchange meeting in Bay City, March 9.

Hub Thigpen attended paraform prill meetings at the Technical Center on March 19 and 26. Jim Russell attended on March 26.

Mr. R. Keith Kaderlik reported to work on March 15. Keith will be working in the BOD-Utilities area.

Frank Dow, Joann Glenister and Blaine Wise attened a presentation at CCCTC on capillary column gas chromatography (optimization and maintenance) conducted by Perkin-Elmer representatives.

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
BOD II Aerobic Optimization	Akin	1	MOB	April, 1982
Vacuum Truck Characterization	↓	1	WMSTF Support	May, 1982
Anaerobic Pulse Test II		1	MOB	May, 1982
TPP, P_2O_5 Emissions		1	Emissions Quantification	May, 1982
Evaluate ATP, OUR, and Dehydrogenase Methods		2	MOB	May, 1982
Support Flocculant Plant Trial	Kaderlik	2	MOB	October, 1982
Evaluate "Unsteady-state" Polyol, VCI and other effluents	Akin/ Kaderlik	1	MOB	September, 1982
Recover PE from VCI Waste Stream	Day	1	Cost Reduction	July, 1982
Formaldehyde Stability	↓	1	MOB	April, 1982
TMP Expansion	Bustabad	1	Expansion	July, 1982
TMP Extractor Optimization	↓	2	Raw Materials Savings	July, 1982

003676

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Recover TMP From Formals in TMP Heavy Ends (V-7474 Residue) Via Alcoholysis	Huelle	1	Raw Materials Savings	May, 1982
Improve PE Quality - UV Absorbance and Formal Content	↓	1	MOB	July, 1982
Monitor Conversion in PE Reaction System	↓	2	MOB	July, 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)	June, 1982
Maintenance Noise Exposure Monitoring	↓	1	MOB (Environmental Health)	April, 1982

003677