

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

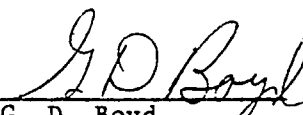
From: G. D. Boyd

GDB-172-82

May 13, 1982

LABORATORY DEPARTMENT SUMMARY - APRIL, 1982

Attached is the Laboratory's monthly summary for April. 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
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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1782 No: 303.3

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HIGHLIGHTS

- In the phosphoric acid washing of amine-contaminated Run VI Oxo catalyst, a third water wash was completed. The pH continued to increase with each wash. Activity and rousing remained satisfactory. Aldol tests are underway.
- Reflux and efficiency loss requirements for removing propyl propionate from propyl acetate to meet our 100 ppm spec while using 250 ppm propyl propionate feed to the heavy-ends column were defined. Process Engineering will calculate the economic penalty.

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I. ANALYTICAL GROUP

Paraform Area

The Analytical Group provided extensive analytical support for Special Problems study of paraform prill production. Preparations are underway for Laboratory support of paraform prill piloting early in August.

Polyol Area

Pentaerythritol (PE) - A variability study is underway on Pure-grade PE lots 82-108-1P and one-half of lot 82-107-3P. The purpose is to determine if the quality throughout the lots will meet Dow Chemical Company's special linear formal spec. (0.15 wt.% Max.) The Instrument Section is providing GC analyses on samples taken from each pallet in these lots. The Special Problems section will issue a report upon completion of the analytical work.

Analytical Group General

Charles Bull attended a Hewlett-Packard laboratory data systems users group meeting 4-14-82 in Houston. This meeting is held twice each year in conjunction with the meeting of the Gulf Coast Instrumental Analysis Group. Approximately 50 users from throughout Texas, Oklahoma and Louisiana were in attendance including representatives from CCCTC, Pampa and the Clear Lake Plant.

Customer Complaints

4-5-82-Cyanamid, Venezuela complained of finished product turbidity problems using Para Flake Lots 323-81 and 324-81. Shipping and retain analysis reveal nothing that could be identified as the cause.

4-15-82-Mobil Chem. Co., Richmond, Va. complained of reaction problems when using Celanese TMP as compared to Perstorp material. Comparative analysis continues to show Perstorp TMP to be a higher purity than Celanese product. AGM-288-82.

4-16-82-B.F. Goodrich, Henry, Ill. complained of 1/2" lumps in Para Flake receipts - Lots 321-81 and 036-82. Shipping and retain analysis do not confirm a lumpy product. Returned sample contained 2x3/8" lumps in a quarter lb. package.

Quality Waivers

3-31-82-Diacetone Alcohol - Houston Solvents, Houston, TX- Mesityl oxide at 1.0% vs. MCS of 0.5 max. 12.420 lbs. - QD

4-14-82-Sodium Formate-Virginia Chemicals, Leeds, SC. Wt.% NaFo at 58.8% vs. Sales Spec. of 59.0 min. 2x20 M gal. cars - 272,530 lbs - SQD

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<u>Product</u>	<u>% QD¹</u> <u>Month</u>	<u>% QD¹</u> <u>YTD</u>	<u>% SQD²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Methylal	0	71.9	0	0
PE-Pure	0	0	0	0.8
n-Propyl Acetate	0	0.4	0	0
Diacetone Alcohol	2.4	11.3	0	0
Formaldehyde-Uninh.	0	0	0	<0.1
PE-Tech	0	0	0	0.3
Sodium Formate	0	0	37.1	9.1

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

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

Analytical Statistics

<u>Sample Count</u>	<u>Apr.</u>	<u>Apr. YTD</u>	<u>Previous YTD</u>
Chemical Section	4618	18,664	18,082
Instrument Section	2477	11,655	10,143
	7095	30,319	28,225

Technician Overtime

Chemical Section	46	241	331
Instrumental Section	34	224	168
	80	465	499

Sample Shipping

Domestic	87	271	240
Export	2	10	14
Plastics	1	4	4
Others	10	39	44
	100	324	302

QA Statistics

Shipments	635	2412	2557
Waivers (QD)	1	15	4
Waivers (SQD)	2	5	4
Complaints	3	8	3

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II. STAFF CHEMISTS

Process Technology B. C. Kerr

Dow Scum Problem - The Laboratory Versene™ Synthesis has been refined. The synthesis has been used to evaluate stream samples, Dow shipments and samples containing known impurities. The test, although it is not quantitative, is useful for laboratory testing. A report describing the Dow scum problem has been written.

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

Dow Scum Problem (Dow, Wise) - A capillary column GC procedure has been developed to analyze hydrocarbons in formaldehyde. This procedure was developed specifically to monitor Dow formaldehyde shipments. Even though a limited number of samples have been analyzed, hydrocarbon concentrations for the 3 MO units are showing <5 ppm for MO III and IV and <5 to 14 ppm for MO II. A 4-23-82 Sx of MS crude MeOH (V-2360) showed 36 ppm hydrocarbon (C₇-C₂₀).

Current investigations (on a high priority basis) are to determine the GC precision and minimum detectable limits of the hydrocarbon procedure. It is known that formaldehyde covers the C₅-C₉ hydrocarbons, however this same GC procedure will detect all of the hydrocarbons in crude MeOH.

III. SPECIAL PROBLEMS

Group I - H. H. Thigpen

Methanol Oxidation, Formaldehyde Blending J. F. Glenister, P. W. Young

AMR's (Glenister) - MO III Startup "B" Train, April 19th

<u>Indicated</u>	<u>Analytical</u>
.88-.90	.772
.88-.90	.773
.88-.90	.874

MO III AMR Check at Rate

<u>Indicated</u>	<u>Analytical</u> -
.99-1.00	1.24
"	1.31
"	1.01
"	1.07

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Summary of Product Color Trends in the MO Units (Glenister) - MO II product color was good with only 3 days of slight color (6 APHA). MO II continues to receive both the T-240 and T-185 OH streams. MO III and MO IV product colors were very good at <5 APHA.

White Particles in 50% HCHO from CELX 13401 (Glenister) - The Laboratory analyzed a sample of white particles filtered from 50% HCHO (CELX 13401). The particles were suspected of being paraform or the dicalite filteraid. IR spectroscopy confirmed that the white solids were paraform. No further work planned.

Samples from MO IV Vent Incinerator (Glenister) - The Laboratory analyzed solid samples collected from the preheater and burner of the MO IV incinerator to determine if the solids were salts from the water to flame arrestor or high molecular weight paraform. Analysis was completed for sodium, calcium, iron, sulfates and chlorides. The results of ion exchange and AA analysis confirmed the solids as salts of sodium, calcium with high levels of chlorides, sulfates and iron (burner opening residue). Results to be published.

Removal of Waxes From MO IV Formaldehyde (Young) - Equipment has been set up to investigate the removal of waxes from Dow formaldehyde. At the present time, the investigation of the effects of different carbon and resin beds is planned.

Oxo, Butanol
T. A. Jasek

Pilot Study - H₃PO₄ Extraction of Oxo Run VI Catalyst Solution - Three water washes of Batch II were completed. An increase in "pH" measured in a methanol-water solution from 3.65 to 4.05 was noted after the third wash vs. the second wash. Rousing studies conducted by CCCTC indicate the rousing characteristics to be well below that of the current Run VII (1.15 for Run VI-all washes and 3.0 for Run VII). Activity studies also indicate approximately 10-40% increase in activity over the untreated Run VI. Aldol tests are currently in progress for catalyst solution of each of the three washes.

Oxo Catalyst to Reclaimers - Analyses were completed on three samples of Oxo catalyst solution to; Metz Metallurgical Corp., PGP Industries, and Platina Labs. The samples were then sent to the reclaimers.

The Laboratory sampled ten drums of Oxo catalyst solution which was being sent to Johnson Matthey, Inc. for trial recovery evaluation. The samples were composited and divided with JMI. Analyses of the composite are being conducted by both the Instrument and Special Problems Lab at Bishop.

Analytical Method for the Oxo Reactor Catalyst Solution - The Laboratory is developing a modified method for analyzing the Oxo Reactor solution. This method is based on an internal standard. Conversion to this method is underway.

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

Propanoic Acid Specification Increase in Clear Lake Acetic Acid (Young) - Ester unit efficiency losses and increased energy usage which would result from an increase in propanoic acid in Clear Lake acetic acid from 100 ppm to 250 ppm were investigated. A 20-tray column was run to simulate the heavy-ends column (T-160) in the ester unit to determine what conditions would be required to decrease the propyl propionate concentration from 250 ppm to the Bishop spec level of 100 ppm. Four different reflux-to-takeoff ratios were run. Process Engineering will calculate the economic penalty resulting from the spec change using the data obtained.

Paraformaldehyde Prills (Russell) - An attempt to relate drying rate to prill bed temperature is underway. The data from each run (7 hours of drying) is being fitted mathematically to an exponential form. Since each run is at a different temperature, the slope of the logarithmic form varies. The slope is then related to the bed temperature. In this way, drying rate will be related to temperature.

Plans are to relate NaOH-additive levels to drying rate after the temperature relationship is determined. Other variables to be related to drying rate include particle size and humidity of the fluidizing air. After the effects of temperature, NaOH level, particle size, and humidity on drying rate are established, the effects of other additives will be checked.

A suitable solubility test has not yet been established for the Laboratory prill drying studies. The effect of the different variables on other properties, such as reactivity, melting point range, attrition, and molecular weight distribution, will not be available until suitable tests and equipment are available.

The Laboratory will set up a fluidized bed using a sintered metal distribution plate. The Laboratory and Process Engineering will jointly observe and judge the results.

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

Noise Exposure Survey (Parker) - The monitoring of Operations personnel to obtain data to "pinpoint" the need for engineering solutions to noise problems is approximately 75% complete.

Formaldehyde Methods Validation (Gary, Parker) - The Laboratory validation of the Celanese and NIOSH chromotropic acid-formaldehyde methods is approximately 50% complete. The effect of sampling time at low formaldehyde concentrations is being studied while waiting delivery of equipment (on order).

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Miscellaneous

T. A. Jasek

Document Retrieval System - The Document Retrieval System has been set up in the Laboratory. The system is functional.

Methanol Synthesis

R. L. Day

Rapid Carbon Deactivation (Day) - A Lab study of the rapid deactivation of the carbon guard beds in the MS unit is underway. Solvent extractions of spent carbon (to determine a means for regeneration) and GC analysis of the sulfur compounds present in the feed are in progress.

Trimethylolpropane

J. C. Bustabad, R. L. Day, B. K. Huelle, J. E. Russell

TMP Expansion (Bustabad) - The cursory design for the TMP expansion includes washing sodium formate from the TMP external to the extractor. Laboratory work is in progress to determine the optimal temperature and wash-water rate.

Hydrolysis/Methanolysis of V-747 Residue (Huelle) - Preliminary evaluations of different processes for converting bis-TMP linear formal and some unknowns in V-747 Residue to TMP have been completed. Six cases have been established: 1) methanolysis of V-747 Residue using a 5:1 molar ratio of MeOH to bis-TMP linear formal, 2) hydrolysis of V-747 Residue using T-205 OH as a diluent in a 1:6 volumetric ratio, 3) hydrolysis of 747 Residue using water as a diluent, 4) treatment of 747 feed with a HCHO scavenger, 2 wt.% hydroxylamine sulfate, and 5 and 6) a combination of case 4 with case 2 or 3. Should any of these cases prove to be economically feasible, a study of resin bed life must be done. Case 1 will require further work in the extraction process. Should case 4 prove to be economical, a study of reaction by-products and their effect on the purification system will be determined at a later date.

Condensing Polyol Vents (Day) - The TMP Concentrator Overheads (V-760 OH and V-759 OH) and the PE "A" Evaporator OH are currently vented to the atmosphere. Economic feasibility of utilizing the latent heat from condensing these vents initiated Laboratory development of vapor-liquid equilibrium (VLE) data. This data will be used to determine efficiency losses out the vents and, as a result of condensing these vents, to determine increases in hydraulic and COD loading to the BOD unit.

A modified Othmer VLE still was used to gather the necessary information. Since there were indications of entrainment in the lab-generated VLE data and because of the large discrepancy between analyses of previous live overhead vent samples and overhead vent samples generated in the Laboratory, these experiments will be repeated using a different apparatus.

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Perstorp TMP Analyses (Russell) - A sample of Perstorp TMP was analyzed for amines on the Dionex IC. The analysis showed 3.3 ppm Na, 0.6 ppm NH₃ and 0.9 ppm K.

Pentaerythritol
B. K. Huelle

Intralot Variation Study During Bagging of PE for Dow Chemical Co. (Huelle) - An intralot variation study on part of Pure-grade PE Lot 82-107-3P and the entire Lot 82-108-3P has been completed. The study was conducted in response to a request from Dallas to resolve questions of analytical differences between Dow and Celanese. Dow recently returned Lot 82-48-3P because of problems in their end-product which they attribute to high bis-PE linear formal. The Dow specification is <0.15 wt.% bis-PE linear formal. Shipping analysis was 0.11 wt.%; Dow's analysis was 0.28 wt%. The intralot variation study showed Dow's specification to be met with no significant variation in the recent lots. These results will be published in early May.

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

BOD II Support (Akin) - Polyol effluent addition to BOD II is at 6000 pounds of COD per day. The organic loading to the anaerobic reactor averaged 0.30 lb COD/ft³/day, with an average outfall of 2100 ppm COD. The average aerobic outfall COD was 270 ppm, with an average detention time of 3.0 days.

VCI/Polyol Treatability (Akin, Kaderlik, Stasney) - An anaerobic/aerobic system is being fed a blend of polyol effluent, VCI effluent, and equalization basin liquid. The quantity of polyol effluent in the blend is slowly being increased while the quantity of VCI effluent and equalization basin liquid is held constant. At this point the weight-percent polyol effluent in the blend is 16.7 (equates to ~9200 lbs polyol COD to BOD/day at an anaerobic loading of 0.31 lb COD/ft³/day), the weight-percent VCI effluent is 4.7 (equates to ~2600 lbs VCI COD to BOD/day at an anaerobic loading of 0.31 lb COD/ft³/day), and the weight-percent equalization basin liquid is 78.6 (equates to ~43000 lbs equalization-basin-liquid COD to BOD/day at a loading of 0.31 lb COD/ft³/day). Anaerobic outfall COD is at ~2000 ppm. Aerobic outfall COD is at ~250 ppm COD with a 2.7-day detention.

Polyol Treatability (Akin, Kaderlik, Stasney) - An anaerobic/aerobic system is being fed a blend of polyol effluent and equalization basin liquid. The polyol effluent being fed presently (16.6 wt.% which equates to ~8560 lbs of COD to BOD/day at an organic loading of 0.29 lb COD/ft³/day) contains no TMP effluent. This blend has been fed for 3 weeks. The feed is now being changed to 16.6 wt% polyol effluent with no PE effluent present. The purpose is to determine if the differences in

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substrates between TMP effluent and PE effluent will adversely affect the performance of the system. Anaerobic outfall COD is at ~2000 ppm. Aerobic outfall COD is at ~280 ppm with a 2.9-day detention.

Vacuum Truck Content Characterization (Akin, Gonzalez) - No trucks were rejected during the month of April due to uncleanness. The average number of truck loads per week has decreased from 21.7 (2/10/81-6/27/81) and 20.2 truck loads per week (9/28/81-10/23-81) to 10.2 truck loads per week (3/22/82-4/25/82). The three major users of vacuum trucks in the past (2/10/81-6/27/81) were Paraform, MO IV, and Utilities. The major users at present are TO-1/SH-1, MO III, and the Tank Car Cleaning Rack. Solids in the trucks, as a whole, have decreased since the introduction of the clean-truck policy in March. 50% or more of the truck loads dumped in the solar ponds presently can be treated in the BOD system. The findings of the entire study will be documented shortly.

Biodegradation of Branched Polyols (Akin) - The amount of COD removal from Polyol effluent is minor in the lab aerobic system which contains bacteria from solar pond 11E. Propagation of bacteria is slow. Varying amounts of feed are being added in conjunction with variations in the amount of nutrients to determine if this will affect a more rapid propagation.

Investigation of Sludge Stabilization by Refrigeration (Kaderlik) - An investigation of aeration basin sludge stabilization by refrigeration was made. Experimental results show that refrigeration at 2°C can stabilize sludge for a period of 16 hours. A report has been issued. (RKK-01-82)

TOD/COD Correlation of VCI Effluent (Kaderlik) - A study of the relationship between TOD and COD of VCI effluent began in early April and will continue through May. This study will allow approximate daily TOD poundage to be determined from VCI's COD analysis of their effluent since VCI will be responsible for controlling their effluent flow to BOD II based on daily TOD poundage values established by Celanese.

Utilities Support-Analysis of Ash Found in Boiler #16 Firebox (Kaderlik) - Ash samples obtained from the walls, burner and superheater tube of the incinerator firebox of boiler #16 were analyzed for heavy metals and ion content. Analysis revealed varying amounts of the following substances: sodium (24-36 wt.%), phosphate (24-26 wt.%), sulfate (0.9-2.4 wt.%), nitrate (0.4-2.9 wt.%), nickel (0.3-1.3 wt.%), iron (0.82-1.53 wt.%) and traces of copper, chromium and rhodium.

General

R. L. Day, P. W. Young, and C. E. Bull attended a half-day seminar at CCCTC to review capillary-column gas-chromatography techniques and introduce new equipment presented by Hewlett-Packard technical representatives on April 20, 1982.

B. K. Huelle attended the Chemical Processing Table-Top Show in Corpus Christi on April 29, 1982.

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Current Spec. 1 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	June, 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
Formaldehyde Stability	Day	2	MOB	June, 1982
TMP Expansion	Bustabad	1	MOB	July, 1982
TMP Extractor Optimization		1	Raw Materials Savings	July, 1982
Condensing Polyol Vents	↓	2	Cost Reduction	July, 1982
Recover TMP from Formals in TMP Heavy Ends (V-747 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

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
BOD II Aerobic Optimization	Akin	1	MOB	June, 1982
Vacuum Truck Characterization		1	WMSTF Support	May, 1982
Anaerobic Pulse Test II		1	MOB	May, 1982
TPP, P ₂ O ₅ Emissions		1	Emissions Quantification	May, 1982
Evaluate ATP, OUR and Dehydrogenase Methods	↓	2	MOB	July, 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
Determine Cause of MS Carbon Bed Deactivation		1	MOB	May, 1982

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