

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

From: G. D. Boyd

GDB-160-82

February 4, 1982

LABORATORY DEPARTMENT SUMMARY - JANUARY 1982

Attached is the Laboratory's monthly summary for January. 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

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HIGHLIGHTS

- Color from MO units has been extracted and concentrated from carbon on unit filters. Attempts to identify the concentrated colored compound(s) are underway.

- Evaluation of Celanese methods for determining formaldehyde exposures is approximately 50% complete.

- The study to evaluate the aerobic treatability of Polyol effluent has been discontinued due to low biomass build, low COD removal, and long detention times.

- Preliminary work indicates that >99% conversion of bis-TMP linear formal can be achieved via methanolysis at a 5:1 molar ratio of MeOH to the TMP heavy ends stream (V-747 residue).

- The pulse test of the anaerobic reactor has been scheduled for March 9.

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I. ANALYTICAL GROUP

Methanol Oxidation (MO)

Blending Unit - The Analytical Group provided analyses for the conversion of the first formaldehyde anion exchanger to computer control. Further studies will be conducted before conversion of additional anions.

Extensive analytical work was required this month in obtaining, blending and loading a quality HCHO product for sales. MO IV operational line-out following startup, and more consistent operation of the other MO units alleviated the problems late in the month. The remaining problem area is in obtaining consistent and correct levels of stabilizer in the preblend inventory. Operational changes and close quality monitoring are continuing in this area.

Methanol Synthesis (MS)

Amine System - The Analytical Group is currently providing support for a Plant trial to determine if sodium metavanadate (NaVO_3) can be used effectively in the shift section as a corrosion inhibitor.

Oxo Unit

Triphenylphosphine (TPP) - The Analytical Group analyzed thirteen samples of M&T TPP and one sample of C. Itoh TPP for all purchase specifications. The purpose was to determine if C. Itoh material was of suitable quality to merit a Plant trial and ultimately qualify as an alternate supplier of TPP. The sample analyzed met our purchase specifications. A Plant trial will be scheduled in the near future.

Customer Complaints

None this month.

Quality Waivers

1-7-82 - Methylal - 10M gal. car to Dow Chemical Co., Freeport, TX - MeOH content at 1.5 wt.% vs. MCS of 1.0 max. Sales Spec. = 2.0 wt.% max. 73,840 lbs.- QD.

<u>Product</u>	<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	100.0	100.0	0	0

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.

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Analytical Statistics

	<u>Jan.</u>	<u>Jan.</u> <u>YTD</u>	<u>Previous</u> <u>YTD</u>
<u>Sample Count</u>			
Chemical Section	4880	4880	4806
Instrument Section	<u>2841</u>	<u>2841</u>	<u>2403</u>
	7721	7721	7209
<u>Technician Overtime</u>			
Chemical Section	78	78	161
Instrument Section	<u>60</u>	<u>60</u>	<u>32</u>
	138	138	193
<u>Sample Shipping</u>			
Domestic	60	60	60
Export	1	1	2
Plastics	2	2	0
Others	<u>0</u>	<u>0</u>	<u>3</u>
<u>QA Statistics</u>			
Shipments	565	565	455
Waivers (QD)	1	1	1
Waivers (SQD)	0	0	0
Complaints	0	0	1

II. STAFF CHEMISTS

Process Technology
B. C. Kerr

Di-PE Recovery Project - Laboratory work on separating di and tri-PE from mono-PE has progressed to a stage where it is necessary to determine a source for di-PE. Production unit test runs have shown that di enriched Tech PE, containing in excess of 12% di-PE, cannot be processed with the filtration system currently available. Since normal Technical Grade PE contains 9% di-PE, an increase of available di-PE of only 2 or 3% is not sufficient to support commercial separation.

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Laboratory work is being concentrated on evaluating techniques for obtaining substantially increased quantities of di-PE without having to materially change PE Unit equipment. Essentially, it appears that there are only two practical routes to obtaining substantial quantities of di-PE:

1. Change unit reaction formulations to produce large quantities of di-PE and devise a method for segregation of the HCHO stripped product for off-site purification.
2. Develop a process for direct conversion of mono-PE to di and tri-PE.

Laboratory work is currently being restricted to the development of a direct conversion process.

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

An intensive analytical effort is being made to determine the source and identity of the "scum" in Dow Versene™.

During January, a technique was developed to evaluate formaldehyde, methanol, antifoams and/or any other component remotely related to formaldehyde that could cause a "scum" in Dow Versene. The procedure did detect differences between formaldehyde samples from Dow that were labeled "good" and "bad". The "good" formaldehyde did not generate "scum", the "bad" formaldehyde did.

On January 13, 1982, a trip was made to Dallas to update Dallas QA and Marketing on the Dow "scum" problem. At this meeting all known information was presented. At that time the only identified "scum" producer was glycol dilaurate, but other streams in the MO III and IV systems also produced a "scum" when evaluated with the new analytical procedure.

Since the Dallas meeting "scum" has been produced by the analytical procedure from the following: 1) MO vaporizer blowdown, 2) crude methanol, 3) T-185 OH (weak formaldehyde) 4) AF-60 5) C₁₀ - C₂₀ hydrocarbons 6) glycol dilaurate and 7) formaldehyde polymers.

A concentrated extract from MO IV product was analyzed at CCCTC by GC/MS. The GC/MS data determined that previously unknown heavy ends were of two classes of compounds, each of which from a homologous series of peaks. One class identified was the cyclic oxygenated compounds such as trioxane and tetraoxane. The other class of compounds was identified as being long chain polyoxymethylene compounds such as methylal and dimethoxymethylal. It is not known if these compounds can or do contribute to the "scum" problem at Dow.

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III. SPECIAL PROBLEMS

Group I - H. H. Thigpen

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

Analytical AMR's (Glenister) - January 4, 1982 - MO III Cold Startup.

	<u>Indicated</u>	<u>Analytical</u>
"A" Train	0.88-0.89	1.43
	0.88-0.89	1.37
	0.88-0.89	1.31
	0.88-0.89	1.37
"B" Train	0.86-0.88	2.70*
	0.86-0.88	2.61*

* Sampling discontinued due to low MeOH feed on startup, and concentration problems. Operations opened one reactor and came up smoothly on the train.

January 5, 1982 - MO III - Rate Up - Operations requested additional AMR's to verify indicated readings:

	<u>Indicated</u>	<u>Analytical</u>
"A" Train	0.88-0.89	0.86
	0.87-0.88	0.86
"B" Train	0.74-0.75	0.86
	0.74-0.75	0.96
	0.74-0.75	0.99
	0.80-0.81	0.99

**The unit was experiencing problems with the instrument monitoring methanol flow. Discontinued sampling until problems resolved.

January 26, 1982 - MO II Rate Up

	<u>Indicated</u>	<u>Analytical</u>
MO II	1.16	1.56
	1.16	1.59
	1.16	1.60

HCHO Stability Study (Day) - Efforts to correlate HCHO stability with organic impurities are still underway.

Attempts to correlate HCHO stability with MO waste streams, such as T-185 OH, T-240 OH, T-288 upper and lower drawoffs, and the MO IV vaporizer blowdown, are also underway.

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Summary of Product Color Trends In The MO Units (Glenister) - MO II (T-297); the average product color was 13 APHA with a range of 5-23 APHA. The product had high color (23 APHA) on January 13, 14. Operations reported the level of trioxane in the T-240 stream to MO II had risen to approximately 9% (D.V. Shattuck). This unit is also continuing to receive the T-185 OH stream from Weak Formaldehyde. (Since December 3, 1981.)

MO III (T-313) the average product was 6 APHA with a range of <5 to 14 APHA. The unit produced formaldehyde of higher color (up to 20 APHA) after startup and after coming up at <5 APHA product color. An increase in product color was observed when MO III received T-185 OH for a short time (approximately 16 hours) during the month. The unit is producing spec color (<5 APHA) formaldehyde at present.

MO IV (T-33) Product color on spec at <5 since December 8, 1981 (<5 APHA).

Residue Coating on Heating Coils in Tank Car CELX 13425 (Glenister)
- The Laboratory analyzed a sample of residue wiped from the heating coils of the tank car. This car had returned with off spec. methyl formcel material due to high color (35 APHA). Analysis of the sample indicated high iron (94 ppm).

Treatment of "Off Spec" Formaldehyde in Tank 2588 (Glenister) -
Operation's requested that the Laboratory examine possible treatment of off spec formaldehyde in Tank 2588 to reduce color (18 cloudy). A sample of the formaldehyde was treated using filtration, activated carbon, addition of EDTA and passage through a heated bed of XFS-4048 (The resin used in the blending unit). Filtration through 0.75 μ m glass filter paper reduced the original 18 cloudy to 10 cloudy and the addition of 50 ppm EDTA reduced the color to approximately 6. Passage of approximately 1500 ml of the off spec material through the resin removed some color initially, but exhausted the resin and lost its effectiveness after 1-2 bed volumes. The reduction of color by addition of EDTA indicates that a significant amount of color is due to iron.

Sampling of T-33 Scrubber Vent Stream to Process Analyzer (Glenister) - Process engineering requested Laboratory assistance in attempts to verify the formaldehyde concentration in the scrubber vent. The process analyzer indicated a high vol.% HCHO loss out the vent which results in a higher efficiency loss for the unit. The Laboratory sampled the stream on several days, but the results obtained were inconsistent. Additional work is planned.

Formaldehyde, Methanol Concentrations in T-313; A Column Profile (Glenister) - Process Engineering requested analysis for HCHO and MeOH concentrations in samples collected from T-313. The data will be used to evaluate a program developed for distillation column design work. The Analytical Group completed the analyses and a letter is in writing.

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Isolation and Identification of Color Bodies (Jasek) - The color study of the MO units product filters continued. The study has been directed toward determining if the presence or absence of color in the carbon on the product filters is related to any of the methanol recovery streams which may be present in MO II or MO IV at various times. Initial indications are that least color is extracted from filters from MO III without T-185 OH as opposed to where T-185 OH is being fed to the unit. Additionally, substantially more color was extracted from MO II filters than the amount from MO III with T-185 OH. In this case, both T-240 OH and T-185 OH were being fed to the unit. No other MO II filters have been extracted thus far because the feed streams remain the same.

CCCTC confirmed the UV-visible absorbances reported last month for a color concentrate extracted from carbon on MO unit product filters. The use of LC (liquid chromatography) for trapping the color body(ies) will begin as soon as the new UV cell is installed.

Ultrasonic Cleaning of Used Silver Catalyst (Jasek) - The ultrasonic cleaner was installed in the silver rewash area. Trial runs to determine optimum cleaning parameters are underway.

Determination of Silver in Rewash Drain (Jasek) - Operations discovered that some very fine silver crystals were penetrating the screen trays set up on the water discharge of the silver catalyst rewashing drum. The drain line and sump were cleaned and the material will be sent for reclaiming. The Laboratory determined the approximate silver content of the two samples received. The first sample from the drain line was 83% silver while the sample from the sump was 67%. The remainder of the samples was carbon and sand.

Methanol Synthesis
J. C. Bustabad

Sodium Metavanadate as Corrosion Inhibitor - A Plant trial of sodium metavanadate as a corrosion inhibitor for the Shift MEA system is in progress. Upon the addition of sodium metavanadate to the Shift MEA system, black solids were found dispersed in the MEA. This was an expected occurrence associated with a "cleaning-up" of the system. The major components of the black solids were iron and vanadium.

Paraformaldehyde
J. E. Russell

Paraformaldehyde Prills - Base case data on the cloud time behavior of typical MO IV product is being gathered. The more or less 51% HCHO is concentrated to approximately 88.0% in a laboratory apparatus. The paraliquid is then kept in a 2 oz. bottle in a 100°C bath until it clouds to nearly opaque. Different additives will be tried to define the effect of each on cloud time. These are added when the concentration step is completed.

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The cloud time of CCCTC pilot plant (MOPP) 51% HCHO when concentrated in the Bishop Laboratory apparatus showed that it behaves differently than Bishop MO IV HCHO. A letter documenting (JER-40-82) the results was published. Subsequent cloud time tests on MOPP HCHO showed that it behaved similarly to Bishop HCHO if the absorber water was left out.

Bishop will continue to check MOPP HCHO as long as CCCTC requests cloud time information or until they have a cloud time apparatus of their own. Further studies involving other additives (catalysts) will be continued at Bishop using MO IV product.

Oxo, Butanol
J. C. Bustabad

C. Itoh TPP - Analyses of C. Itoh TPP Lot #110072 (representative of lot to be sent for Plant trial) were very close to analyses completed on an earlier C. Itoh TPP sample (Q-010018). Based on analyses of the earlier sample, a Plant trial of C. Itoh TPP was recommended. Dallas Purchasing was informed of results of laboratory analyses; C. Itoh TPP has been ordered for a Plant trial.

Phosphoric Acid Extraction of Oxo Run VI Catalyst Solution - The phosphoric acid extraction of 275 gallons of Oxo Run VI catalyst solution is in progress.

GENERAL

- Bob Kerr and Hub Thigpen (along with Jack Christenson and Jeff Kirk of CCCTC) visited Hatco Chemical Co. in Fords, NJ on January 20 to discuss di-PE production methods.

- Jim Russell and Hub Thigpen attended meetings on paraform prill production on January 12 and January 29.

Group II - L. M. Harvey

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

Noise Exposure Survey (Parker) - The noise exposure survey of Maintenance personnel continues. (Approximately 75% complete)

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Asbestos Sampling (Parker) - A sample of insulation was tested for asbestos content by the screening method and was positive. The sample was submitted to CCCTC for X-ray analysis and was negative.

Formaldehyde Methods Validation (Gary) - The evaluation of the five internal Celanese methods for determining formaldehyde exposures is approximately 50% complete.

BOD

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

BOD II Support (Young) - Continued support was provided to BOD II operations. A pulse test of the anaerobic reactor is scheduled for March 9.

VCI/Polyol Treatability (Young, Stasney) - An anaerobic-aerobic system is acclimated to a feed of 5.5 vol.% VCI effluent, 4 vol.% polyol effluent and 90.5 vol.% equalization basin and nutrients. The organic loading (OL) is 0.27 lbs.COD/ft³/day and the aerobic detention time is approximately 2.9 days. The anaerobic and aerobic outfall CODs are 1200 ppm and 200 ppm respectively with the kinetic efficiency stable at approximately 55%. The concentration of polyol effluent will be increased to 7 vol.%.

Polyol Treatability (Young, Stasney) - An anaerobic-aerobic system is being fed a feed consisting of 7 vol.% polyol effluent and 93 vol.% equalization basin and nutrients. The OL is 0.08 lbs.COD/ft³/day. The anaerobic reactor is recovering from a setback earlier in the month. The OL will be raised to 0.28 and the detention time lowered to 2.9 days upon reactor recovery.

Aerobic Treatment of Polyol Effluent (Akin, Stasney) - This study has been concluded. Due to low biomass build, low COD removal, and high detention times it has been concluded that aerobic treatment of polyol effluent is not feasible. Documentation of this study is forthcoming.

Vacuum Truck Content Characterization (Akin) - This study has been delayed since the first week in November due to a problem with the cleanliness of the trucks. S.H.E. has developed a procedure for inspecting the trucks for cleanliness upon entering the Plant. This study will resume when the inspection procedure is working.

Evaluation of Powdered Activated Carbon in Aerobic Optimization (Akin, Stasney) - A study has begun to determine if powdered activated carbon mixed with aeration liquid will expand the capacity of the aerobic system, and also increase sludge settling velocities. This experiment consists of a control and a system which has powdered activated carbon dispersed in the liquid (PAC system).

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BOD II Aerobic Optimization (Akin) - The purpose of this study is to determine the optimum operating parameters for the aerobic system (during and after polyol acclimation). Parameters currently being investigated are oxygen uptake rates, ATP levels, dehydrogenase levels, and mixed liquor volatile suspended solids. Later in the study the adequacy of the clarifier at future loadings and the optimum sludge age with polyol effluent will be investigated.

Pentaerythritol
R. L. Day, B. K. Huelle

Recover PE From VCI Waste Stream (Day) - Analytical has been completed on shake-out experiments to determine the minimum amount of 2-ethylhexylamine required in VCI's wiped film evaporator residue to facilitate PE recovery. A report will be issued in February.

PE Unit Support (Day) - A high linear formal make in the PE finished product was reported on January 14. A sample of the feed to the acid digester was analyzed for sodium formate (NaFo). A high NaFo level in this sample (0.16 wt.% vs. 0.03 -0.08 wt.% normally) indicated that the problem was due to poor filtration on a spare belt in use while the "A" belt was being repaired. The belts were switched within hours, and the linear formal make was back to normal levels on January 15.

TMP
R. L. Day, B. K. Huelle

Methanolysis of V-747 Residue (Huelle) - Laboratory work to improve the economics of the methanolysis process continues. Data indicates the optimal MeOH:bis-TMP linear formal molar ratio is 5:1 (Previous cursory economics were based on a 25:1 molar ratio of MeOH:linear formal). At 5:1 ratio, 99.6% of the linear formal appears to be converted to TMP, TMP cyclic monoformal, or an intermediate. Changes in the concentrations of other unidentified compounds of V-747 residue is also evident under these conditions. Experiments conducted using V-747 residue with the TMP light ends stream, T-205 OH, (no MeOH added) yield as much TMP as the 5:1 (MeOH:V-747 residue) molar ratio experiments when the temperature of the resin bed is raised from 60°C to 85°C. In addition, the data shows that formic acid is not required for the reaction to occur; TMP cyclic monoformal formation was not suppressed by the presence of formic acid as previously reported (MEH-38-79). Preliminary data on sizing the resin bed indicates a much smaller resin bed than that used in the latest economic evaluation is possible.

TMP Expansion (Day) - Process Engineering requested that the Laboratory determine how many water backwash stages are necessary to remove sodium formate (NaFo) from TMP in V-759 residue (extractor feed). This information will be used in designs of the TMP extraction system for a future expansion.

003643

A two-stage batch simulation of the water backwash in the upper portion of the T-312 extractor was performed. One-stage water backwash at 140°F reduced the NaFo level in the extract from 520 ppm to 70 ppm. Two stages at 140°F reduced the NaFo level in the extract from 520 ppm to 10 ppm. Adding water to the feed sample at 140°F was also tried, but this was allowed to cool to ambient temperature before separating the layers. The NaFo level in the extract was reduced from 520 ppm to 3 ppm. A letter documenting this data will be published in February.

pH Control of Excess Caustic in TMP Reactor Product (Huelle) - At the request of Systems Engineering, the titration curves for the TMP reaction mixture using formic acid and acetic acid were developed. The Laboratory suggests that the unit laboratory collect data on reactor product pH so that a correlation between pH and excess caustic levels can be made. The Laboratory also recommends a reinvestigation into controlling caustic addition to the TMP reactors based on the MeOH make rate across the reactors should the pH control not be feasible. A letter on this work will be published this month.

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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	March, 1982
TPP, P ₂ O ₅ Emissions		1	Emissions Quantification	March, 1982
Evaluate ATP, OUR, and Dehydrogenase Methods		2	MOB	April, 1982
Support Flocculant Plant Trial		2	MOB	October, 1982
Recover PE From VCI Waste Stream	Day	1	Cost Reduction	February, 1982
Formaldehyde Stability		1	MOB	March, 1982
TMP Extractor Optimization		2	Raw Materials Savings	March, 1982
Recover TMP From Formals in TMP Heavy Ends (V-7474 Residue) Via Alcoholysis	Huelle	1	Raw Materials Savings	March, 1982
Monitor Conversion in PE Reaction System		1	MOB	March, 1982
Methanol Personal Exposure Monitoring	Gary	1	MOB (Environmental Health)	September, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Jasek	1	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	Glenister Jasek	1	MOB	March, 1982
Stability of 37% Unstabilized HCHO (For Dow Chemical Co.)	Glenister	1	MOB	March, 1982
Ni(CO) ₄ Method Evaluation	Bustabad	1	Evaluate Current Shutdown and Analytical Procedure	April, 1982
Run VI Oxo Trial Extraction		1	Cost Reduction	March, 1982
Extend Run VII Catalyst Life		1	Raw Materials Savings	March, 1982
Support Paraform Prill Pilot Operation at Bishop	Russell	1	Cost Reduction and MOB	June, 1982
Polyol/VCI Treatability	Young	1	MOB	March, 1982
Anaerobic Pulse Test		1	MOB	March, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Area Surveys for Misc. Organics	Gary	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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