PLAINTIFF'S EXHIBIT
CEL-255

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: W. H. Meyer WHM-207-78 October 3, 1978

Laboratory Department Summary

September, 1978

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September, 1978

The Bishop Plant Laboratory activities during September are attached. Section I contains a summary of the Quality Assurance and Analytical Group activities with tables of analytical statistics, shipments, quality waivers, quality complaints and sample shipping statistics. Section II describes the status of Special Problems Group projects by Unit Chemist assignment. A table listing the priorities, justifications and estimated completion dates of current Special Problems projects is also included in Section II.

W. H. Meyer

WHM/as
Attached Report

003315

## I. QUALITY ASSURANCE AND ANALYTICAL GROUP

### Competitive Evaluation

None this month.

### Methanol Oxidation (MO) - Weak Formaldehyde Recovery

Special analytical assistance was provided for determination of Methanol in the T-185 OH stream (47 to 56 wt. %) to update efficiency calculations.

### Polyol Unit

Pentaerythritol (PE) - Continued routine inspection and quantification of lumps in the daily packaged Lots indicates that current production contains many (20+/lb.) small, soft lumps greater than 12 mesh (0.066 inch) in size. Planning continues for the installation of 12 mesh screens at unit screening locations.

### Utilities - BOD

The data gathering portion of the evaluation of the dependability of the Plant Total Carbon Analyzer Alarm system has been completed. Preliminary evaluation indicates a very good correlation between reflux COD (Lab) and analyzer (Unit) TOC (Total Organic Carbon). A complete report summerizing the data along with recommendations is being prepared.

A Laboratory reagent problem resulted in an erroneous high COD value being reported on the front ditch outfall for 9-10-78. Correction of the reagent problem and recheck analysis show no reportable excursion of our permit. SOP's have been instituted to prevent a reoccurance.

## Customer Complaints

Mobay Chem. Co., Natrium, W. Va. rejected a trailer load of molten TMP (order # 60581, shipped 9-05-78). Their complaint was for high color, high water content and low freezing point. The returned product was sampled, met all specification, and was off loaded to production flaking tanks. The replacement shipment, made 9-13-78, was apparently accepted without incident. This was not a valid complaint.

## Quality Waivers and Shipment Delays

Quality waivers were obtained to ship a 20 M gal car of n-Butyl Formcel® to Celanese Canada and a 5 M gal trailer of 55% Methyl Formcel to Enenco, Inc. at Memphis, Tenn. These products were considered atypical due to low Sodium values (9 ppm each) when compared to our now normal Caustic treated product. Apparently the Caustic addition treatment step was omitted from the production of these Formcel batches.

## Quality General

The Methanol specifications for General Electric, Waterford, N.Y. were reviewed against Chemcel product and rejected due to a low level maximum Ethanol content of 100 ppm. The proposed Methanol purification scheme is expected to result in a product well within our regular grade Ethanol maximum of 750 ppm but above the 100 ppm level.

At the request of the St. Louis Sales Office, a sample of iso-Butyl Acetate, a scan and our G.C. Method for purity determination were forwarded to Superior Solvents & Chemicals at Indianapolis, Ind. We understand that this product is to be evaluated by the Eli Lilly Co. in that area, a customer that has rejected us as a supplier in the past.

## Personnel and Group General

The Quality Assurance Group has employed a permanent full-time secretary after several years of part-time and temporary clerical help. The timing of reports from the Group should be improved.

The Instrument Section of the Analytical Group has experienced a 50% turn-over in personnel (2 of 4) in the last month. One was a scheduled rotation of untrained personnel and one was the result of replacing a resignation vacancy.

The vacancy created by the resignation is being covered in the Chemical Analytical Section via overtime. The position will be filled soon.

## Analytical Statistics

| | Sept. | Sept. Y-T-D | Previous Y-T-D |
|---|---|---|---|
| **Sample Count** | | | |
| Chemical Section | 4,633 | 40,606 | 44,773 |
| Instrument Section | 2,925 | 23,937 | 21,356 |
| Total | 7,558 | 64,543 | 66,129 |
| **Technician Overtime** | | | |
| Chemical Section | 46 | 768 | 633 |
| Instrument Section | 13 | 165 | 604 |
| Total | 59 | 933 | 1,237 |

## Sample Shipping and Quality Assurance Statistics

Statistics For Sept. 1978

| Sample Shipping | Month | YTD | Previous YTD |
|---|---|---|---|
| Domestic | 53 | 603 | 824 |
| Export | 2 | 25 | 79 |
| Plastics | 3 | 37 | 63 |
| Other | 17 | 138 | 709 |
| Total | 75 | 803 | 1,675 |

### Quality Assurance Statistics

| | Month | YTD | Previous YTD |
|---|---|---|---|
| Shipments (NO.of Orders) | 758 | 6,750 | 7,748 |
| Delayed | 1 | 23 | 24 |
| Waviers Internal | | 2 | 38 (Total) |
| External | 2 | 17 | |
| Complaints (Total) | 1 | 30 | 46 |
| Complaints (Legitimate) | | 12 | 14 |
| Complaints (Non-Legitimate) | 1 | 10 | 32 |
| Complaints - Outstanding (Unresolved) | 0 | 8 | -- |

## II. SPECIAL PROBLEMS GROUP

### Methanol Oxidation, Formaldehyde Blending
J. D. Presley, T. A. Jasek

MO Efficiency Improvements with
Trace Amounts of Halogenated Hydrocarbons - No progress due to priorities.

Iron in the MO Air Supply - Work is scheduled to resume in October to determine the extent of MO catalyst contamination due to Iron in the air supply.

Reduce Methylal Formation in MO Distillation Columns - A letter is in progress recommending a Plant trial to attempt to reduce Methylal formation in the MO distillation Columns by the addition of Sodium Hydroxide. Recommendations will be based on potential raw material savings, and the success achieved at Celanese, Alberta, Canada in a similar Plant trial.

MO Unit Support - Efforts to determine the reasons for poor sintering of Englehard Silver Catalyst have been iniated. It has been speculated that variation in particle size distribution (within our spec.) may be a contributing factor to the problem. Free fall packing densities, determined on various samples of Englehard and Handy and Harmon catalyst, consistently showed Englehard as having the lower of the two. Further testing is planned, e.g., particle size distribution vs sintering of Englehard catatlyst. Work continues.

Reacter analysis and vent analysis in MO-IV have shown discrepancy in $CO_2$ formation. It is suspected that product degradation in the Reacter outlet header is contributing to this. Lab tests are being conducted to substantiate this and to determine other possible sources of the discrepancy.

A letter (MEH-13-78, JDP-37-78) has been issued recommending an alternate recovery of MeOH from MO-IV Methanol Vaporizer Blowdown. This should provide reduced metal contaminates in Mo-IV product thus assuring more stable Paraform Unit operation, improved Formaldehyde quality, and decreased chances for high metals to Paraform in a second Crude Methanol to MO Plant Trial.

Complaints of Formaldehyde color were received from the Celanese terminals. The problem was traced to color build in tankcars. A letter (JDP-40-78) has been issued summarizing work.

Utilities, Raw Materials, BOD
S. G. Smith, J. R. White, G. R. Stasney

BOD Unit Analytical Methods Manual - No progress due to priorities.

Anaerobic Testing of Chemical Wastes - 1. General Studies.

A simulated nylon waste effluent was included in the anaerobic feed on 8-27-78. Following addition of this effluent, organic loading was held at 0.54 lb COD/$ft^3$/day for several days. Kinetic loading varied between 0.36 - 0.43 lb COD/$ft^3$/day, chemical removal between 80-87%.

The organic loading to the filter was increased to 0.6 lb COD/$ft^3$/day by 9-06-78 and held at this loading throughout September. During this time, kinetic loading has varied between 0.4 - 0.5 lb COD/$ft^3$/day. Chemical removal has averaged at approximatley 80%, kinetic removal varying between 70-80%.

Samples of the anaerobic vent gas have been submitted to CCCTC to be analyzed for $H_2S$ content. The results of these analyses have not been received.

All of the anaerobic outfall is being fed directly to the aerobic tank, so that detention time in the tank is still low, ranging from 0.68 to 1.39 days during September. The food to biomass ratio has varied between 0.39 and 0.59, outfall COD between 216 - 484 ppm. Volatile suspended solids in the tank has been between 3000 - 4000 ppm. Plans are to split the anaerobic outfall flow to the tank in order to increase detention time in the tank.

COD removal over the entire anaerobic-aerobic system has been between 93 and 98% during September.

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2. Startup Studies.

The construction of a 20-liter, bench scale anaerobic filter, to be used for developing startup procedures, is nearly completed. Initial use of this filter is scheduled for the beginning of October.

Aerobic Treatment of Nylon Wastewater - Nylon wastewater, 10% by volume in equalization basin, and normal BOD unit feed (eq. basin) were fed to two laboratory aeration tanks at varying food to biomass ratios up to 0.55. No adverse effects were observed at this loading of nylon effluent.

The nylon portion of the feedstock was then increased to 20% by volume. Food to biomass ratios have varied between 0.45 and 0.55. No adverse effects have been observed.

Evaluation of Alternate Weak Base Resins for the Demineralizer - Reported higher capacity weak base resins, Rohm and Haas IRA-68 and Dow WGR-2, are being evaluated as alternates for the presently used Rohm and Haas IRA-94. Work is being done to establish the relative organic and acid capacities of these resins. Both IRA-68 and Dow WGR-2 have demonstrated approximately twice the acid capacity of IRA-94. Because the organic removal efficiency of all the resins apparently increases with subsequent service cycles, more work is necessary to establish the relative organic capacities of the resins. Work continues as time permits.

Formaldehyde Stability at 160°F - Due to Acme Resins complaint of low assay of Formaldehyde shipments, the stability of 50% Formaldehyde at 160°F has been investigated. The Formaldehyde (both stabilized and unstabilized) showed an average acid increase of .004%/day and exceeded acid specifications at day 12. Formaldehyde assay data showed a loss/day greater than could be accounted for by Formaldehyde acid build via the Cannizzaro reaction. A letter is in progress summarizing the results of the study.

Utilities Support - $SO_2$ in 93% $H_2SO_4$ received in the Plant was found to be about 220 ppm. The Plant was switched back to 98% $H_2SO_4$, which contained <25 ppm $SO_2$, until Corrosion evaluates the effects of high $SO_2$ ($H_2SO_4$) on present Plant equipment.

## OSHA Monitoring
J. M. Jackson

A review was made of the Hydrazine addition system in the Methanol Synthesis Area. Recommendation was made to Mr. Ille that the exhaust from the surge pot be extended above and out of the immediate area to prevent possible Hydrazine exposure.

A monitoring schedule has been set up with the Oxo Unit Supervisor to obtain additional TPP exposure data (samples will be taken Oct. 2 thru Oct. 6).

A study is in progress to evaluate Teflon sample bags for potential use in personnel Formaldehyde monitoring via the CEA 555. Samples of Formaldehyde will be stored in the Teflon bags to determine if there is a concentration decrease due to adsorption to the walls of the bag.

Chemical Plant tours were conducted during September for Mr. M. R. Stenzel, Chemical Company Industrial Hygienist, and Dr. John Clary, Corporate Director of Toxicology.

Attended the Company Health Meeting in Dallas, September 14 and 15, 1978.

## Oxo, BuOH, PrOH
J. D. Presley

Rhodium Accounting - Work has been initiated to look for sources of Rhodium loss.

Reactor Stability/Accountability - Reactor solution accountability this month has been varible reaching a low of about 81%. As of 9-27 accountability is greater than 86%.

Questions have been raised about the accuracy of the reactor TPP analysis by G.C. Work is underway to resolve any problems by cross-checking samples with the titration method.

Oxo, BuOH, PrOH unit Support - A Plant Trial to inject Hercules CR 408 into T-229 as an aldol inhibitor is scheduled for the week ending October 7.

## Methanol Synthesis
F. R. Dow

Fuel Gas Quality - No samples were analyzed due to other priorities.

MS-II Analytical Support - A letter has been published detailing the "fishy odor" in Spec Methanol. It was determined that Nitrogen in the finished Methanol is introduced into the MS-II Unit via the natural gas feed, and that Nitrogen in the steam is being recycled to the reformers. A recommendation was made for the Laboratory to evaluate the potential use of a strong acid ion exchange resin to remove the odor causing impuities (if justified by Marketing).

Limited work has been done to determine if a significant concentration of paraffins are being made in the Lurgi Convertors. An extraction of a segment from one of the Crude Methanol filters (9-19-78) did not contain a detectable concentration of paraffins. However, visual inspection has indicated the presence of paraffins in retain samples from the lower trays of both finishing columns.

A sample from Tray 7, T-280, was concentrated and then extracted with Chloroform. The Chloroform extract was analyzed by IR at CCCTC and identified as a paraffin. The paraffin concentration was ∿0.01 wt %.

## Trimethylolpropane
L. Mayerle

TMP Unit Support - The compatibility study of the TMP and 1,3-BG Vacuum Flashers residue streams with other waste streams currently burned in a boiler has been completed. Recommendations have been made to conduct a plant trial to determine the suitability of the heavy-ends streams as boiler fuel. A scheme has been proposed for facilitating the transferring and burning of V-747 Residue and V-778 Residue based on limitations identified in the study. A letter has been issued with details (LM-47-78).

Several jet condensate samples were collected to determine TMP losses. A sample of the TMP Hotwell effluent and jet condensate samples from T-205 OH, T-205 SS and V-747 OH were analyzed to support Process Engineering in a project to recycle the condensate for recovery of TMP.

Potential for Increased Reactor Cooling - Methanol production across the TMP reactors has ranged between 0.2 and 0.3 Δ wt % MeOH. These data indicate that an excessive amount of HCHO is still being lost to the straight Cannizzaro reaction. Data will continue to be collected every two weeks under normal operating conditions. Further evaluation requires a shutdown of the TMP Unit for the purpose of cleaning the reactors (JRK-50, LM-44-78).

TMP Alternate Purification Scheme - Cursory studies of alternate jet-tray extractor designs are being conducted at CCCTC. Feed for the extractor and analysis of samples is being provided by the Bishop Laboratory. Initial work has been done using Oldershaw columns. Analyses of organic overhead and aqueous TMP samples were documented (LM-45-78).

## Pentaerythritol
R. G. Hilbrich

Recovery of Energy from PE Unit "A" Evaporator Vent - Samples condensed from the steam vent were collected on three days. Analysis showed the level of PE in the stream to be insignificant (1 - 10 ppm). Total Carbon analysis indicates the presence of an unknown contaminant(s). Only about half of the total Carbon (1000 ppm) can be accounted for. Work continues.

PE Unit Analytical Methods Manual - Laboratory work is essentially complete on the Sodium ion electrode method for determining Sodium Formate concentration in the dissolve tank. Completed manual will be submitted this month.

Seeding the PE "A" Crystallizer - A Laboratory study was conducted to determine the volume of "A" magma which must be left so that all of the crystals do not dissolve when the "A" crystallizer is filled with fresh feed. It was recommended that during the planned plant test, the volume of magma left be 15% of the crystallizer volume when it is filled with fresh feed. A report was issued (RGH-19-78).

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## Pentaerythritol
L. Mayerle

Removal of Crystallization Inhibitors in PE "A" Crystallizer Feed - A study is being conducted to investigate the removal of crystallization inhibitors in PE "A" Crystallizer feed. Initial studies were performed by passing T-286 Residue over XAD-2 resin or Carbon, evaporating water to a typical "A" evaporator composition and crystallizing using a typical "A" crystallizer cooling curve. No significant increase in Pe crystallization was seen after treatment with XAD-2 resin, however an increase was seen after treatment with Carbon. This is a tentative conclusion. Each run will be repeated to verify the initial data. Some questions about the analytical results will also be resolved before any final conclusions are reported.

## Esters, Methylal, Paraform, Formcel® Products
M. E. Harris

Acetate Unit Support - Start up of T-343 Laboratory simulation has been accomplished. Ten experimental runs have been made. Several problems have been encountered which were not previously thought to be serious: 1. At various concentrations and boil up rates, foaming has been found to be a problem. 2. The lower boiling (89.4°C) ternary azeotrope competes significantly with the binary (90.2°C) azeotrope in the top 20 to 25 trays. As a result, Alcohol is concentrated to 18 - 20 wt % in the overhead distillate at water/organic ratios greater than 0.41 in the reflux with a total reflux ratio of 0.97 (reflux/take off).

The foaming problem could have contributed to the unusual profiles observed during the T-343 start-up. Additional studies are underway to further determine what types of conditions and compositions may cause operating problems in the unit.

Formcel® Products Unit Support - No progress.

Paraform Unit Support - A report has been issued (JDP-37-78/MEH-13-78) which recommended an alternate recovery scheme for MO-IV MeOH Vaporizor Blowdown which should significantly improve the quality of Formaldehyde being received by the Paraform Unit. This should significantly reduce operating problems in the Paraform Unit.

003326

## 1,3-Butylene Glycol
P. J. Buras

BG Unit Support - Removal of the demister in V-778 has not caused serious problems to date as BG efficiency has been high for a 17 day period between Sept. 4 and Sept. 20 (78.4% verses 72% plan) and no significant problmes have been experienced with color or odor of the finished product. Lab analysis of V-778 residue has shown that BG losses have been minimized. The flowrate is down to 0.5 gpm, representing about 2.5% efficiency loss as BG (10 - 12% in previous runs) and about 2.5% as heavy ends. A letter was written documenting the status of the current BG startup.

Hydrogenation pressure was decreased for a 6 day period 9-19 - 9-26 to increase the catalyst recycel pump seal life. Due to increased Butanol production, the test was discontuined. Another plant test with more extensive analysis will be conducted in the future.

T-227 Residue Product verses Vapor Side Stream - In order to build inventory of spec. product, T-227 was returned to the vapor side stream mode of operation. A plant test will be made in October to determine if BG specification (color in particular) can be met using T-227 residue product.

## DiAcetone Alcohol
P. J. Buras

Identification of DAA unknown - The heavy end unknown was concentrated by vacuum distillation from T-285 residue and samples collected for further analyzia. The unknown decomposes under high GC temperatures. A report will be issued when positive identification has been made.

## Process Technology
B. C. Kerr

Abstracting of Technical Reports - Abstracting of technical reports is current. Typing for Abstract Cards is in progress.

003327

Revision of Purchase Specification Manual - Comments and suggested corrections have been received on the draft copies sent out for review. Work will begin the first week in October to incorporate the required changes.

Preliminary Studies of Plant Sapling and Analytical Procedures - Work on this project has been delayed due to higher priority projects.

M-M-T meeting minutes - The meeting minutes for the Bishop M-M-T meeting held August 29 -30 are being issued with the assistance of R. L. Carpenter.

## Current Special Problems Projects

| Project | Chemist | Priority | Justification | Estimated Completion Date |
|---|---|---|---|---|
| MO Efficiency Improvements with Trace Amounts of Halogenated Hydrocarbons | Presley Jasek | 2 | Raw Material Savings | Undetermined |
| MO Catalyst Screening | | 2 | To Establish Alternate Suppliers and Improve Catalyst Quality | Continuing |
| MS-II Crude To MO | | 1 | Steam Savings | April 1, 1979 |
| Reduce Methylal Formation In MO Distillation Columns | | 1 | Raw Material Savings | April 1, 1979 |
| Iron in MO Air Supply | ↓ | 1 | Raw Material Savings | Jan. 1, 1979 |
| BOD Analytical Methods Manual | Smith | 2 | Maintenance of Business | Undetermined |
| Anaerobic Testing of Chemical Waste Streams | White | 1 | To Test Anaerobic Process as Alternate BOD Expansion | Jan. 1, 1979 |
| Aerobic Treatment of Nylon Waste Water Effluent | | 1 | To Test Treatability of Nylon Waste in Bishop BOD Unit | Oct. 1, 1978 |
| Evaluation of Alternate Weak Base Resins for the Demineralizer | ↓ | 1 | To Increase the Demineralizer Unit Capacity | Dec. 1, 1978 |
| OSHA Monitoring | Jackson | 1 | Ensure Employee Health and Safety. OSHA Compliance | Continuing |
| OXO Reactor Accountability/ Stability | Presley | 1 | Improve Reactor Control | Continuing |





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## Current Special Problems Projects

| Project | Chemist | Priority | Justification | Estimated Completion Date |
|---|---|---|---|---|
| PE Unit Analytical Methods Manual | Hilbrich | 2 | Mainenance of Business | Undetermined |
| Energy Recovery from PE "A" Evaporator Steam Jet | ↓ | 1 | Steam Savings | Jan. 1, 1979 |
| Seeding the PE "A" Crystallize | ↓ | 1 | Raw Material and Steam Savings | Dec. 31, 1978 |
| Paraform, Formcel® Products, Esters Unit Analytical Methods Manual | Hilbrich Harris | 2 | Maintenance of Business | Undetermined |
| New Ester Unit Reactor Column Start Up | Harris | 1 | Steam Savings | Oct. 15, 1978 |
| Establish Potential of Increased TMP Reactor Cooling | Mayerle | 2 | Raw Material Savings | Continuing |
| Remove Crystallization Inhibitors From PE "A" Crystallizer Feed | ↓ | 1 | Raw Material Savings | March, 1978 |
| TMP Unit Analytical Methods Manual | ↓ | 2 | Maintenance of Business | Undetermined |
| PE Unit Air Emissions Survey | ↓ | 1 | Provide Emissions Data | Undetermined |
| 1,3-BG Unit T-227 Residue Stream Instead of Vapor Side Stream | Buras | 1 | Steam Savings | Nov. 1, 1978 |
| Identify Unknown in DAA Product | ↓ | 2 | Maintenance of Business | Nov. 1, 1978 |

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## Current Special Problems Projects

| Project | Chemist | Priority | Justification | Estimated Completion Date |
|---|---|---|---|---|
| Union Carbide Acetone, Help Evaluate Plant Test | Buras | 1 | Maintenance of Business | Nov. 15, 1978 |
| MS-II Analytical Support | Dow | 1 | Maintenance of Business | Continuing |
| Fuel Gas Quality | ↓ | 1 | Improve Fuel Gas Accountability | Continuing |
| One Column Purification | ↓ | 1 | Energy Saving | Continuing |
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