

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: W. H. Meyer WHM-119-78 January 11, 1978

Laboratory Department Summary
December, 1977

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The Bishop Plant Laboratory activities during December are attached. Section I contains a summary of the Analytical Group activities with a table of analytical statistics. Section II contains a Quality Assurance Summary and a table listing shipments, quality waivers, quality complaints and sample shipping statistics. Section III 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 III.

W. H. Meyer

WHM/jm
Attached Report

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I. ANALYTICAL GROUP

Competitive Evaluation

None this month.

Methanol Oxidation (MO), Formaldehyde (HCHO)

MO-II - Analytical assistance was provided for several studies involving the feeding of Plastics Company recovered stream T-240 overhead to the MO-II Unit.

MO-IV - Continued increased analytical monitoring of the Metolose stabilizer in sales grade HCHO indicates an improvement achieved in consistent addition. Increased monitoring frequency will continue.

Oxo, Butyraldehyde (BuH), Butanol (BuOH)

Special analysis of crude BuH samples were run to assist in a BuH material balance. The purpose of the balance was to generate data on the reaction system, and to tune the math model.

Paraform

Special COD and TOC determinations were run on T-103 residue (alcohol recovery column) for several days in an effort to locate the source of high COD content of Paraform-Polyol effluent to the BOD Unit. This stream was proven to not be the source of the problem.

Polyol

Diacetone Alcohol (DAA) - Analytical assistance was provided in an attempt to quanitfy and control carbon loss out of T-295 residue. Work is continuing and when complete, a report will be issued.

Trimethylolpropane (TMP) - Vent and vent drain samples of V-759 were analyzed for weight percent HCHO for the Plant air emission survey.

1,3-Butylene Glycol (1,3-BG) - Samples were received representing a customer (Conap, Inc.) complaint on Newark Terminal drummed 1,3-BG. Odor panal and G.C. identification work established that Lot 381 sample contained methyl acrylate (< 0.05wt.%) and that Lot 394 sample contained trace amounts (< 200 ppm) of both an unidentified acrylate and n-BuOH. Lot 394 sample also exhibited a very slight HCHO odor. No further work is planned.

II. QUALITY ASSURANCE

The New York Office reported a complaint from Amoco Chemical Corp. (Wood River, Illinois), regarding 44% HCHO arriving in a cloudy condition. The customer has rejected CELX 1235 shipped to them on December 1, 1977. Amoco also reported the arrival of three previous cloudy shipments. All retain samples were analyzed for regular sales specifications, and checked out OK. The Metolose stabilizer was analyzed, and levels from 3.3 to 10.0ppm were found. The tank car rejected by the customer showed the highest stabilizer (10ppm). No information on the status of this car is available. The car was equipped with a heating coil, but we are not sure if the customer attempted to use it. Information has been requested regarding the temperature on arrival to the customer. Loading temperatures here were in the normal range (135-140°F). Winter loading temperatures are now in effect for loading 44% HCHO in the range of 140-145°F.

Paraformaldehyde Flake - An export shipment to Amcel Europe, September 15, 1977, contained 700 fifty-pound bags of Paraform Flake, Lot 256-77. The quality of this lot was low in purity (89.5%). A quality waiver was obtained from New York Quality Assurance (NYQA) to ship this export to Amcel. Recently, we received a letter from Mr. Peter Schmidt, Brussels, Amcel Europe S.A., noting the low assay, and pointing out that their customer noted the low assay. He indicated that they were not rejecting the material, but would like assurance that future shipments will be within sales specs. Every effort will be made to prevent the low assay problem from happening again.

During the month, Paraform Flake, Lot 340-77, was produced at an assay of 93.4%. A quality waiver was obtained to ship all of this material to the Traylek warehouse. There were 5,250 bags of this material shipped.

During December there were seven occasions when the assay of Paraform Flake was higher than 93.0wt.% maximum. Resamples were within specification range of 91.0-93.0wt.%. Operations and the Lab have not been able to determine why original samples all showed high assay.

Trimethylolpropane - Last month it was reported that the TMP purity was borderline at 98.0%. Since the middle of December, all TMP lots produced were 99.0%, or better. Feed nozzle location on T-202 seems to correlate with product assay.

Ethyl Acetate - The current inventory of ethyl acetate in Tank 1274 is off-test on acidity at a level of 0.020% as acetic. This acidity is due to CO_2 which has been absorbed by the ethyl acetate during the unloading of incoming tank cars from Pampa. The quality as received, is within specifications (.005 maximum). The Plant nitrogen (high in CO_2) is causing the acidity problem.

Sample Shipping and Shipping Statistics

Sample Shipping

	December	December Y-T-D	Previous Y-T-D
Domestic	56	1,017	1,178
Export	5	92	181
Plastics	6	80	118
Other	8	742	287
Total	75	1,931	1,764

Shipping Statistics

Shipments (# of Orders)	962	10,288	9,351
Delayed Due to Quality	2	28	33
Quality Waivers	3	42	13
Quality Complaints	4	52	27
Quality Compalints (Accepted)	1	16	11

Quality Waivers and Shipments Delayed Due to Quality
December, 1977

Date	Product	Customer	Destination	Tank Car Tank Trailer Solids	Delay or Waiver	Comments
12-01-77	45% Formaldehyde	Celanese Chemical	Argo, Illinois	20M - CELX 1215	Delay	Missed blend. Low assay. Mechanical problems.
12/02/77	37% Formaldehyde	American Cyanamid	South River, Missouri	10M - GATX 77438	Delay	Low assay. Had to be adjusted. Three days delay.
10/04/77	Paraform Flake	Amcel Europe	Belgium	Export 700 x 50 Lot 256-77	Waiver	Assay low, 89.6%. Approved by J. C. Scott. Only 700 x 50# bags shipped. (This item not reported in October.)
12/13/77	Paraform Flake	Traylek Warehouse (Celanese)	Chicago, Illinois	Lot 340-77 5250 bags	Waiver	Assay high, 93.4% formaldehyde. All material to be shipped to Traylek warehouse. Approved by T.H. Golson, NYO.
12/15/77	Ethyl Acetate	Chemcel Inventory TK 1274	-	39,000 gallons	Waiver	Acidity high due to CO_2. Level, 0.020 Spec, 0.005. Without CO_2= 0.002

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

Purchase Specification Update - No progress due to priorities.

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

Anaerobic Testing of Chemical Wastes - A letter co-authored with Process Engineering was issued outlining the objectives of the laboratory anaerobic-aerobic pilot unit. Design information concerning the feed distribution and the gas/liquid take-off for the anaerobic reactor was received from Process Engineering, and construction of these components is in progress. Also, calculations of design parameters concerning the aerobic section and clarification section have been received from Process Engineering. The reactor vessels arrived in early January.

BOD Unit Support - A minor upset occurred early in the month when outfall COD's peaked at 904ppm on December 5, 1977. Feed flow rates were reduced and diluted with river water, resulting in a relatively quick recovery of the unit. The cause of the upset was not determined. No indications of an overload (high equalization basin COD) or poisoning of the bugs (high metals, methanol or formaldehyde in the feed) was evident. Upsets of this kind may be the result of the presence of nonbiodegradables such as PE, TMP, or TOX in the feed. The Laboratory does not have a method to determine these components in waste streams, except for trioxane (TO). TO levels in outfall samples during this period were negligible. Thirty-day retainment of test pond and equalization basin samples has been put into effect in an attempt to be able to explain such upsets.

800gpm Demineralizer Unit Support - Data is being collected to monitor the acid solution out of the strong acid cation bed (V-2408) during regenerations with and without blocking flow. Information from this study along with results of the previous blocking flow study (sodium leakage of the SAC) will be published in a letter to be co-authored with Process Engineering.

Demineralizer Expansion Support - Currently, the highly acidic neutralization tank (pH $\simeq$2.0) is transferred to the lime pits where the pH is raised to within the acceptable outfall limits of 6-9. A study was initiated to determine the effect of increased demineralizer capacity on the lime pits outfall. Data is also being generated to aid in establishing pH control methods for the lime pits outfall. A report will be written.

OSHA Monitoring
J. M. Jackson, L. M. Pena

A report on personal 8-hour TWA exposure of Operator Helpers to benzene, methyl alcohol and ehtylene oxide in TO-II and SH-II is in progress. A follow-up sampling program will be scheduled.

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DAA Laboratory Reactor Study - A report on the effects of water in the acetone feed to the DAA reactors is in progress. Water in the acetone feed suppresses the mesityl oxide make which will increase unit efficiency, and may result in a more stable product with respect to color build.

Monitoring T-295 Residue for Acetone - Samples of T-295 residue are being analyzed in the Laboratory for acetone. Acetone sewered in the residue is a direct efficiency loss to the DAA Unit. Initial results of the study show 1.0-2.0wt.% acetone in T-295 residue samples. This represents about a 0.3-0.7% efficiency loss.

Presently, T-295 residue is monitored in the unit lab by total carbon analysis. The total carbon analysis can sometimes be misrepresentative of how much acetone is being sewered in the residue. The Laboratory is working on a different method for monitoring the T-295 residue. A complete report will be issued.

Evaluation of Pampa
Acetone as a DAA Feed - It has been suggested that acetone from the proposed methyl formate and acetone recovery system in Pampa be used as feed to the DAA Unit. The Bishop Laboratory has concluded that the proposed Pampa acetone could not be fed directly to the DAA reactors. The methyl formate and formic acid in the Pampa acetone would neutralize the alkaline DAA catalyst beds. The damage would probably be irreversible. It is estimated that one catalyst bed would last about 60 days if only Pampa acetone were used as feed. However, caustic treating the Pampa acetone (either in the proposed methyl formate and acetone recovery system, Pampa; or in the acetone recovery column, T-295, at Bishop) might reduce the methyl formate and formic acid in the acetone to acceptable levels. A report will be published in January detailing the evaluation. No laboratory work will be done unless sufficient economic incentive is defined at Pampa.

Pentaerythritol
R. G. Briell

PE Unit Air Emissions Survey - Sampling of vent emissions in the PE Unit was continued in accordance with the air emission survey. T-286 vent, T-287 vent, "A" evaporator vent and the particulate sampling from dust collector stacks have not yet been sampled. All condensable material has been analyzed by the Lab, while results from CCCTC on the non-condensables have not been received. Upon completion of the sampling and Tech Center analysis, a joint report with Process Engineering will be issued.

Update of PE Unit Methods - All items needed for one of the new methods have been received, and completion of the revised analytical methods manual should be early next month. After completion, the manual will be issued to the unit lab.

MS-II Analytical Support - Ethanol in crude methanol (V-2360) has increased from approximately 120ppm (June, 1977) to approximately 170ppm (December, 1977).

There are also indications that DME has increased from approximately 1.1wt.% to approximately 1.4wt.% after the Lurgi converter temperatures were increased to 260°C (December 9, 1977). The DME increase was first observed approximately two weeks after the temperature increase.

T-240 Overhead to MO-II - Analytical procedures were developed to monitor components in T-240 overhead and critical streams in MO-II. The T-240 overhead MeFo concentration varied from approximately 7wt.% to approximately 29wt.% during the unit trial run.

Oxo, Butanol (BuOH), Propanol (PrOH)
A. J. Bacon

- The air emissions survey for the area was completed and a report was issued.

- A material balance was carried out around the Oxo reaction area December 15, 1977. The objective is to provide "snapshot" data for Process Engineering's math model. Data is still being evaluated.

- Several samples were sent to the Tech Center for x-ray analysis. Among these was a sample of T-210 residue solids. This is to support M.E. Harris' studies to eliminate T-210. As was found approximately one year ago, the solids contain Ca, Cl, $SO_4^{=}$ and some Zn, apparently from cooling water. Evidence points to its entry somewhere around T-266, but the exact place has not yet been found.

- The Lab continues to work on the assay of Engelhard's last rhodium nitrate shipment. Composite samples have been taken of all lots. Assays will be done here and again by Ledoux. Our objective is to again establish Ledoux as our official analyst for rhodium nitrate.

- A meeting is planned with Engelhard, January 12, 1977. The disposition of Run #3 ash will be discussed.

- Three pre-shipment samples of rhodium nitrate were received from Matthey-Bishop. All of them contained large amounts of precipitate. Ledoux and Matthey-Bishop were both questioned about this, and they reported finding no residue in any of their samples, even at 35-40°F. Gentle heating dissolved the precipitate which we suspect was a form of rhodium oxide. The cause of its formation in only our samples is not known. However, we do not expect any problems with the material in our process.

Rhodium Accounting - As of December 15, 1977, all rhodium and phosphorus had been accounted for within experimental errors. An estimate of expected rhodium credit from Run #3 was given to Accounting. The Lab recommended using a 50% recovery factor for Run #4 rhodium.

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Efficiency Improvements With Trace Amounts of Halogenated Hydrocarbons	Presley	2	Raw Material Savings	August, 1978
MO Catalyst Screening		2	To Establish Alternate Suppliers And Improve Catalyst Quality	Continuing
MO Catalyst Reuse		2	Reduce Silver Inventory	March 1, 1978
MS-II Crude to MO		1	Steam Savings	Plant Testing Complete by March 31, 1978
Reduce Methylal Formation In MO Distillation Columns		1	Raw Material Savings	Plant Testing By March 1, 1978
T-240 Overhead/MO-II Acid and Methyl Formate Balance		1	Raw Material Savings	Lab Work Complete By December 30, 1978
Long-Term Formaldehyde Stability	Harris	1	Improved Quality Control	March, 1978
Improved Method For Determining Metolose		1	Improved Quality Control	March, 1978
BOD Analytical Methods Manual	Smith	2	Maintenance of Business	March 31, 1978
Purchase Specification Update		2	Maintenance of Business	May 1, 1978
Anaerobic Pilot Testing of Chemical Waste Streams		1	To Test Anaerobic Process As Alternate BOD Expansion	January 1, 1979

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Demineralizer Expansion Support	Smith	2	Support Unit Expansion	March 1, 1978
OSHA Monitoring	Jackson Pena	1	Ensure Employee Health and Safety OSHA Compliance	Continuing
1,3-BG Unit Emissions Survey	Hancock	1	Ensure Compliance With Standards	February 1, 1978
1,3-BG Unit Support	↓	1	Maintenance of Business	Continuing
1,3-BG Cost Reduction	↓	1	Cost Reduction	Feburary 1, 1978
DAA Unit Support	↓	1	Maintenance of Business	Continuing
DAA Unit Emissions Survey	↓	1	Ensure Compliance With Standards	February 1, 1978
DAA Laboratory Reactor Study	↓	2	Increase Unit Capacity and Steam Savings	February 1, 1978
Monitor T-295 Residue Acetone Loss	↓	2	Minimize Efficiency Loss	February 1, 1978
PE Unit Emissions Survey	Briell	1	Ensure Compliance With Standards	February 1, 1978
Reduce Sulfate in PE AML	↓	2	Comply with VCI Request	February 1, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Update PE Unit Lab Methods	Briell	1	Maintenance of Business	February 1, 1978
Acetate Unit Emissions Survey	↓	1	Ensure Compliance With Standards	February 1, 1978
PE Unit Support	Briell,Mayerle	1	Maintenance of Business	Continuing
Paraform-Formcel Unit Support	Hilbrich,Briell	1	Maintenance of Business	Continuing
Acetate Unit Support	↓	1	Maintenance of Business	Continuing
Alternate n-BuAc Process	Hilbrich	1	Steam Savings	March 1, 1978
Paraform, Formcel Products, Esters Unit Analytical Methods	↓	2	Maintenance of Business	April, 1978
TMP Unit Support	Mayerle	1	Maintenance of Business	Continuing
TMP Unit Emissions Survey	↓	1	Ensure Compliance With Standards	February 1, 1978
Alternate TMP Purification Scheme	↓	1	Energy and Raw Material Savings	March, 1978
TMP Unit Analytical Methods Manual	↓	2	Maintenance of Business	February 1, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Fuel Gas Quality	Dow	3	Improve Fuel Gas Accountability	Continuing
MS-II Analytical Support		2	Maintenance of Business	Continuing
T-240 Overhead to MO-II		1	Cost Savings	February 1, 1978
Crude Methanol to MO		2	Cost Savings	March, 1978
DME in Crude Methanol		2	Lurgi Catalyst Conversion	Continuing
Oxo, Butanol, Propanol Support	Bacon	1	Maintenance of Business	Continuing.
Oxo Reactor Accountabiltiy/Stability		1	Improve Reactor Control	Continuing
Rhodium Accounting		1	Maintenance of Business Catalyst Savings	Continuing
T-210 Removal from Oxo Unit	Harris	1	Steam Savings	March, 1978

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