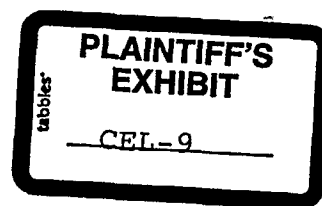


CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

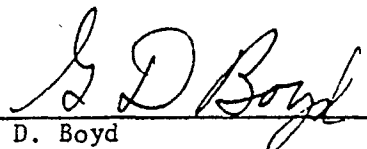
From: G. D. Boyd

GDB-149-81

November 6, 1981

Laboratory Department Summary - October 1981

Attached is the Laboratory's monthly summary for October. 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
W. H. Brough
J. B. Brown
G. L. Cade
R. R. Carpenter
M. W. Clifton
R. S. Hahn
W. A. Heenan
A. A. Miller
B. C. Kerr
C. W. Lee
D. E. Lockwood
A. G. McGehee
W. H. Meyer
D. G. Pausky
J. C. Rubiano
O. W. Schnellenberger
G. R. Scholz
D. D. Siemonsma
W. L. Sharp
K. R. Stelts
H. H. Thigpen
F. R. Dow

Plastics

R. G. Brandt
A. C. Abshier
R. L. Bunkley
H. L. Davis
D. A. Reck
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

003600

NOV 13 81

Highlights

- Treatment of steady state polyol effluent at low BOD unit loading has been demonstrated in a laboratory unit.
- Successful treatment of variable strength VCI waste continues in a laboratory unit.
- Preliminary vacuum truck waste characterization indicates that much of this waste is treatable in BOD II.
- A Plant trial for use of phosphoric extracted Oxo catalyst has been planned for December.
- A Plant trial for use of C. Itoh TPP has also been scheduled for December.
- Studies indicate that some quantity of 2-ethylhexylamine (2EHA) will have to be left in the VCI wiped-film evaporator (WFE) residue before our proposed PE recovery process will be effective on that stream. Studies are underway to determine the amount of 2EHA required.
- The discrepancy between the Celanese and Acme Resins HCHO assay methods was resolved. The Celanese method is the more accurate of the two.

003601

I. ANALYTICAL GROUP

Methanol Oxidation (MO)

Unstabilized 37% HCHO produced with a minimum level of antifoam is being shipped to Dow Chemical, Freeport, Texas. This is an attempt to alleviate "solids scum" formation problem in the customers process. Approximately 92 gallons of product on hand in Dow's tanks will be returned to the Plant for claiming.

Polyol Area

Pentaerythritol (PE) - The Instrument Section is providing analytical support for a project to evaluate potential processes for separating di and tri-PE from PE unit product.

Utilities and BOD

BOD analysis were provided for the CCCTC pilot study of the Celrobic[®] system. The CCCTC study is to evaluate prospective customers effluent in the system. The results were forwarded to Susan Smith for evaluation.

Chemical Oxygen Demand (COD) analyses are being run daily on the Polyol effluent sump. This is to establish base case data prior to feeding this material to the BOD system.

Customer Complaints

9-29-81 - Hooker Chemical, N. Tonawanda, N.Y. - 50% HCHO - 20 M Gal. ca
Complained of arrival with 20 color.

10-1-81 - American Cyanamid, Kalamazoo, Michigan - 52% HCHO - 20 M Gal.
Complained of arrival with assay of 51.7% HCHO.

10-1-81 - Dow Chemical Co., Freeport, Texas - 37% HCHO - 92 M Gal. in cu
inventory - Complained of formation of "scum" in process when using this proc

10-27-81 - Hooker Chemical, N. Tonawanda, N.Y. - 50% HCHO - 20 M Gal. ca
Arrived with 15 color.

Quality Waivers

9-30-81 - iso-BuAc to Celanese, Argo Terminal - iso-butyl propionate at
wt.% vs. max. of 0.01 wt.% - 192,770 lbs. - QD.

10-1-81 - iso-BuAc to Celanese, Celriver Terminal - iso-butyl propionate
0.02 wt.% vs. max. of 0.01 wt.% - 192,150 lbs. - QD.

10-1-81 - PE-Tech (Lube grade) to Reichhold Chemical Co., Oakdale, La.-
15,000 lbs. - SQD - Note: This Lube grade PE shipped as Tech grade as per
manufacturing agreement.

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10-2-81 - iso-BuAc to Celanese, San Pedro Terminal - n-BuOH content @ 0.08 wt.% vs. max. of 0.05 wt.% - 147,460 lbs. - QD.

10-8-81 - iso-BuAc to Celanese, Newark Terminal - iso-butyl propionate @ 0.02 wt.% vs. max. of 0.01 wt.% - 194,220 lbs. - QD.

10-28-81 - iso-BuAc to Celanese, Argo Terminal - iso-butyl propionate @ 0.02 vs. max. of 0.01 wt.% - 192,720 lbs. - QD.

Product	% QD ¹ Month	% QD YTD	% SQD ² Month	% SQD YTD
Para F-Powder	0	0.9	0	2.3
Para C-Powder	0	0	0	5.5
Diacetone Alcohol	0	1.2	0	0
Formaldehyde	0	0.2	0	0
<u>n</u> -Butyl Acetate	0	4.2	0	1.4
Sodium Formate	0	0	0	1.1
Pentaerythritol-Tech* *(Lube as Tech)	0	0	0.6	1.4
<u>iso</u> -Butyl Acetate	100.0	10.9	0	0
<u>n</u> -Butyraldehyde	0	0	0	0.5
Para 95% Flake	0	0	0	7.3

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>Oct.</u>	<u>Oct.</u> <u>YTD</u>	<u>Previous</u> <u>YTD</u>
<u>Sample Count</u>			
Chemical Section	4416	45914	45586
Instrument Section	<u>2757</u>	<u>26975</u>	<u>22971</u>
Total	7173	72889	68557

Technician Overtime

Chemical Section	28	538	627
Instrument Section	<u>9</u>	<u>330</u>	<u>325</u>
Total	37	868	952

Sample Shipping

Domestic	42	593	655
Export	2	52	27
Plastics	1	11	77
Others	<u>13</u>	<u>102</u>	<u>77</u>
Total	58	758	836

QA Statistics

Shipments	605	6479	7288
Waivers (QD)	5	24	15
Waivers (SQD)	1	13	23
Complaints	4	15	29

003604

II. STAFF CHEMISTS

Process Technology B. C. Kerr

Technology Manual Revision - The revision of the Chemcel Plant Technology Manual is approximately 80% complete. Text sections for the new MO IV conversion and expansion and some parts of Utilities remain to be completed. Several Process Flowsheets are still needed from Process Engineering. First draft typing for the manual is approximately 60% complete.

Groundwater Contamination - URM, Inc., has supplied the Plant with copies of their analyses of groundwater quality. These analyses are being reviewed in preparation for the 18 month review required for the Texas Water Quality Control Board. A review meeting with URM, Inc. is planned for November 2, 1981.

Di-PE Recovery Project - A study has been started of potential methods for separating commercial quantities of di-PE from the existing PE Unit operation. At present, all effort is being devoted to an investigation of separation techniques. If a commercially applicable method is found, then it will be necessary to determine how to produce the additional di-PE. Currently, Laboratory work is centered on trying to utilize the nearly 20-fold difference in solubilities between mono-PE and di-PE. Although it is possible to obtain a di-PE enrichment by the addition of water to Tech grade PE, the resulting slurry is difficult to filter and the enrichment is less than theoretical solubilities would indicate. Another technique which is being studied is to slurry PE in a solvent which has a low solubility in water and then emulsify water into the slurry with an intensive blender. This procedure appears of giving di-PE enrichment much closer to the theoretical solubility limits. Work with both n-butyl acetate and methylal have shown that Tech grade PE with an initial di-PE content of about 11% can be enriched in excess of 85% by sequential emulsion extractions. It should be noted that there is a concurrent concentration of tri-PE in these procedures. Combined di- and tri-PE concentrations of approximately 95% have been obtained in this manner. So far attempts at using elevated temperatures to reduce the volume of water required to remove the mono-PE have not been very successful. At 70°C, it has been difficult to maintain the emulsion characteristics which lessen the filtration problems at lower temperatures. Work is continuing on this project.

Production of Crotonaldehyde in the BG Unit - The Plant has received inquiries concerning the availability of crotonaldehyde for sale. While this product is not currently available, the potential exists in the BG Unit to produce some quantity of a relatively crude grade of crotonaldehyde concurrently with BG production. This potential has been discussed with the Technical Center and Process Engineering. A letter outlining the operational equipment requirements for co-producing BG and crotonaldehyde is being planned by H. H. Thigpen.

Special Analytical F. R. Dow

Polyol Feed to BOD II (Dow, Wise) - Analytical procedures have been finalized and documented for analysis of Polyol components in the BOD System. Routine analysis will be transferred to the Instrument Laboratory.

003605

Rainwater Runoff (Dow, Wise) - Daily samples from Chemical and Plastics Co. rainfall runoff were composited and analyzed for COD and identifiable components. During the period from 10-5-81 to 10-29-81 the Chemical Co. COD range was from 15,680 to 55,760 ppm and the Plastics Co. COD range was 322 to 1317 ppm. The major component contributors from the Chemical Co. were sodium formate, 1,3-BG and PE.

Analysis of GE-60 (Antifoam) in Formaldehyde (Dow) - A complaint was received from Dow Chemical, (Freeport) stating that a "scum" formed on their versene when it was made with Celanese formaldehyde, but the "scum" did not form when Monsanto formaldehyde was used. Dow also stated that analysis by mass spectrometer showed the "scum" contained a high concentration of silicone.

With this information it was concluded that the potential problem may be with the addition of GE-60 antifoam in the MO distillation columns or the addition of GE-60 with metolose stabilizer.

The Chemcel Plant Method B-1-30 to analyze GE-60 [poly(dimethylsiloxane)] was evaluated and found to be unacceptable for the analysis of Si in formaldehyde. Further work determined that any ashing procedure would volatilize the GE-60 before it could be properly ashed. An alternate procedure is being investigated to extract the GE-60 and to analyze the extract for Si by AA.

III. SPECIAL PROBLEMS - H. H. Thigpen, O. W. Schnellenberger

Methanol Oxidation, Formaldehyde Blending

T. A. Jasek, J. F. Glenister, R. L. Day, B. K. Huelle, J. E. Russell

Analytical AMR's (Glenister)

MO III Start-Up "A" Train September 30, 1981

<u>Indicated</u>	<u>Analytical*</u>
0.80-0.83	1.45
0.80	1.60
	1.32
	1.37
	1.28

MO I Start-Up October 20, 1981

0.990-1.0	0.993
	0.954
0.999	0.952

* Unit was starting up a low methanol feed, therefore, throughput for analysis was low causing higher than expected analytical AMR's.

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Specific Gravity Using Westphal Balance (Jasek) - The Laboratory provided Operations with a backup method for determining the specific gravity of the methanol-water solution in the vaporizers at MO IV. The backup method uses a Westphal specific gravity balance. A formula was developed which will calculate the percent water (wt.%) in a range of 35-55% and 10-30°C. Technicians at the Laboratory and Operations personnel were trained in the use of the Westphal balance.

GC/MO Reactor (Jasek) - The new Varian chromatograph was set-up and plumbed for the GC/MO reactor. The reactor will be installed shortly. Studies utilizing the GC/MO reactor will then begin.

Precipitate in Unstabilized 37% Formaldehyde, Tank 2603 (Glenister) - The Analytical group noted the presence of a precipitate from the check for methocel in 37% unstabilized HCHO from Tank 2603. The precipitate varied in appearance from samples of a white, red mixture of particulates to presence of a red colored precipitate. An increase in this precipitate occurred around October 12, after MO IV shutdown and when MO III product was started into Tank 2603. Analysis of limited quantities suggest the presence of Dacalite, a filter aid used in the Blending Unit. Tank 2603 was cleaned the week of 10/26-10/30. The Laboratory will follow-up on results following the cleanout.

Presence of Substances in 37% Stabilized HCHO Causing "Scum" Formation in Dow Versene 100 (Glenister) - Dow Chemical Co. reported the formation of a "scum-like" layer in their versene (50% water solution of EDTA) when made using Celanese 37% inhibited formaldehyde. A sample of this versene was received and the scum isolated using a procedure suggested by Dow. An IR spectra of the unknown material was obtained and compared to the IR's of MeCl_2 extractions of some components of the Celanese formaldehyde. Various extractions of GE-60, stabilizer and versene scum were analyzed by capillary gas chromatography by B. Wise. The Laboratory also received a sample of Celanese 37% inhibited formaldehyde used by Dow, it was extracted with MeCl_2 and the separate layers analyzed by capillary gas chromatography. These results were compared to those obtained for the MeCl_2 extractions of GE-60, stabilizer mix, and the versene "scum". No conclusions can be drawn to date. The study continues. Dow conducted a Plant trial using unstabilized 37% formaldehyde successfully and are currently using the unstabilized 37% in their process.

The Laboratory is checking the stability of the 37% unstabilized formaldehyde at lower temperatures, to examine the effect of lower temperatures on storage.

MO Catalyst Sinter Test (Glenister, Jasek) - Operations has placed a charge of Engelhard-198, fair sintering silver in MO II and MO III respectively. The Laboratory will monitor its performance.

Formaldehyde Assay Discrepancy Between Acme and Celanese (Huelle, Russell) - Work has been completed and results reported in a letter published in late October (BKH-08-81, JER-35-81). The study showed the pH adjustment of the sodium sulfite to be the key difference between the methods. Acme's adjustment of the sulfite solution before the addition of HCHO causes approximately .24 wt.% error in the assay.

003607

A small (~0.1 wt.%) error in the Celanese HCHO assay method caused by excessive indicator was found and corrected.

Using caustic as a "substitute" standard, the Celanese method proved to be more accurate than the Acme method.

The reproducibility of Acme's method was adversely affected by exposure of the calomel electrode to the sulfite solution.

HCHO Stability Study (Day) - The HCHO stability study was full-time this month. Samples were set up from MO I, MO II, MO III, MO IV, the blending unit, and unit storage tanks. Each sample was analyzed for assay, acid, color, sodium, and iron. Age of the HCHO was also noted. Twelve ppm metolose stabilizer was added in the Laboratory.

The samples in the 49°C oven have lasted longer than our spec. of 30 days.

In the samples set up in the 40°C oven a large range was seen in stability. Some samples lasted as few as 3 days, and some lasted as long as 24 days.

A study is underway to attempt to correlate the presence of specific impurities with stability. An extraction of the HCHO followed by capillary GC analysis of the extract is used to indicate the presence of specific impurities. Several solvents were screened for use in the analytical procedure. Methylene chloride, chloroform, a butyl ester, a butyl ether, and 1,4-dioxolane were evaluated. Based solely on one data point, there may be several initial impurities that correlate with stability. Before any specific conclusions can be drawn, however, additional data must be collected.

Monsanto HCHO (Day) - We are currently receiving Monsanto HCHO for resale and internal use while MO IV is down. Four quarts of this Monsanto HCHO were quick-frozen for future evaluation.

The Monsanto HCHO was extracted with methylene chloride, and the extract was analyzed by capillary GC. There are differences in the GC scans of Monsanto and Celanese HCHO.

Some of the Monsanto HCHO is being evaporated down to isolate their stabilizer. This stabilizer will then be put back in our own HCHO, and its effect on HCHO stability determined.

Trimethylolpropane
R. L. Day

Unit Support - None provided

747 Residue Methanolysis - No progress, full time on HCHO stability.

Extractor Optimization - No progress, full on on HCHO stability.

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Pentaerythritol

L. M. Harvey, B. K. Huelle, J. E. Russell

Recover PE From VCI Waste Stream (Harvey) - The process recently proposed by the Laboratory for the simultaneous recovery of PE and 2-ethylhexylamine (2EHA) from VCI extract does not appear to be economically attractive due to its energy-intensive nature. VCI is proceeding with plans to recover the 2EHA using a wiped-film evaporator (WFE). Efforts to recover PE from the proposed WFE residue indicate that some amount of 2EHA must remain in the WFE residue to obtain a PE of suitable quality for recycle. The 2EHA in the WFE residue would function in the same way as in the proposed process using VCI extract (to extract impurities from the aqueous PE stream). Since an azeotropic distillation step similar to the original proposal would be necessary in any PE recovery scheme (to insure complete removal of all 2EHA from the PE recycle stream), studies are in progress to determine the minimum amount of 2EHA which must be left in VCI's WFE residue in order to obtain a PE stream of suitable quality for recycle to the PE Unit.

Efforts to simulate VCI's proposed WFE met with mechanical difficulties. As 2EHA was flashed from VCI extract material, PE precipitated on the walls and the blades of the laboratory-model WFE making steady-state conditions impossible to achieve. Mr. Joe L. Bush of VCI said they had no evidence of a similar problem in their pilot-scale WFE; however, frequent washings of their WFE with water did improve their 2EHA-recovery efficiency. A simple batch flashing procedure is being used instead of the laboratory-model WFE in experiments to determine the minimum amount of 2EHA required in the WFE residue for PE recovery.

Evaluation of Filter Cloths for the A and B Belts (Huelle) - An evaluation of filter cloths for the A and B belts is near completion. Eight filter cloths were evaluated; three cloths with the best flow rates were evaluated further at various cake thicknesses. None of the cloths had porosities great enough to allow fines to break through. The cloth currently in use is among the top three candidates.

PE Hydrolysis (Russell) - The final Laboratory report was issued.

1,3-Butylene Glycol

J. E. Russell

V-778 Residue Burning - The analyses of September samples of V-778 Residue were completed. The results were published.

Acetaldol for Nepera - A one-gallon sample of T-219 Residue was collected and sent to Nepera's Harriman, New York facility at the request of Dallas Quality Assurance. The sample was not assayed, but is expected to contain 30-40% acetaldol in water with small amounts of crotonaldehyde and acetaldehyde.

T-219 Analytical Method - A method is being developed to analyze T-219 overhead and T-219 residue for acetaldehyde, crotonaldehyde, and acetaldol. A good

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analytical method will enable the Laboratory, Operations and Process Engineering, to optimize T-219 operating conditions. At optimum conditions T-219 should strip enough acetaldehyde from the reactor product to minimize efficiency losses to ethanol, but conditions should not be severe enough to dehydrate the acetaldehyde to crotonaldehyde, which is an efficiency loss to BuOH. Currently, an analytical method is being developed which uses the Laboratory's liquid chromatograph.

BOD - Utilities

P. W. Young, D. R. Akin, J. C. Bustabad, G. R. Stasney

BOD II Support (Young) - Continued support was provided to BOD II operations. Input was given on the procedure to add polyol effluent to BOD II. Technical support will be given during this addition.

Polyol Treatability In BOD II (Young, Stasney) - A Laboratory anaerobic-aerobic system (No. 3) was acclimated to 7-8% steady state polyol effluent (COD=31,000) (~25-30% COD). Operated at an organic loading (OL) of 0.2 lbs COD/ft³/day and an aerobic detention time of 3.5 days. The aerobic outfall COD=160-180 ppm. At this time, 5.5% VCI effluent was added to the above feed (OL=0.28 lbs COD/ft³/day, aerobic detention time = 2.9 days). The aerobic outfall rose to 300-500 ppm. Feed was lowered (OL~0.2, det. time ~3.5 days). This upset probably occurred due to the rapid increase in F/M in the aerobic section. This shows that acclimation time and time for increase in biomass concentration is needed to include 5.5% (25 gpm) VCI in BOD II feed. Upon recovery, the OL of 0.28 and detention time of 2.9 days will be attained slowly, thus giving the bacteria acclimation time. Live polyol samples are being collected and analyzed for future laboratory pilot unit feed.

VCI Waste Treatability (Young, Stasney) - Many different samples from VCI waste are being fed to the Laboratory anaerobic-aerobic system (No. 2) to see effects of variability in VCI effluent. No adverse effects are being seen so far.

Plastics Laboratory Effluent (Young, Stasney) - Eighteen samples were collected from CP & SC Laboratory. The results are as follows: flow rate: 6000-12000 gal/day, mean COD: 501 ppm, metals: Na: 145-300 ppm, Ni + Cu + Cr: 0.04-0.26 ppm.

BOD II Aerobic Optimization-Sludge Age Study (Akin, Stasney) - This study was completed during October. The 25 and 35 day sludge ages both had similar COD removal efficiencies, but by utilization of the 35 day sludge age, sludge waste age would be minimized. Settling velocities observed at both sludge ages were much higher than is required in the clarifier. The recommended sludge age for the BOD II system with its present feed is 35 days. This study will be documented and another study begun to determine the optimum sludge age for the BOD II system after the addition of polyol effluent.

Aerobic Treatment of Polyol Sump (Akin, Stasney) - A study is underway to determine if polyol effluent can be treated by aerobic methods alone. A Laboratory unit is removing 70% of COD from a feed with a COD concentration of 8800 ppm.

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This feed is 27% by volume polyol effluent and 63% effluent from another laboratory unit simulating BOD II. The COD contribution from polyol effluent is 90%. The feed detention time is 12 days. Further work will attempt to optimize the system.

Oxygen Uptake Rates in Aerobic Systems (Akin, Stasney) - A study has begun to follow trends in oxygen uptake rates (OUR) versus mixed liquor volatile suspended solids (MLVSS) in aerobic systems. Three laboratory units and BOD II aeration basins have been monitored for changes in OUR/MLVSS. The OUR/MLVSS increases in proportion to increases in feed to biomass (F/M) to the aerobic systems. Since the OUR/MLVSS increases in proportion to F/M the COD removal efficiency remains relatively constant. This trend only holds true up to a level where the excess feed becomes toxic to the biomass. At this point OUR/MLVSS begins to drop. When the loading is reduced to a level that can be tolerated by the biomass, OUR/MLVSS resumes its normal level. A drop in OUR/MLVSS is followed by a drop in COD removal efficiency. This was observed in anaerobic/aerobic system No. 3 treating polyol when VCI waste was added at too high an initial loading. As loading increased so did the OUR/MLVSS. COD removal efficiency was relatively constant as the VSS respiration rate kept up with the loading increase, but once the loading became too high, the OUR/MLVSS dropped drastically indicating a toxic affect from the excess feed. Two days later the COD removal efficiency began a steady decline. This would indicate that the OUR/MLVSS could given an advance warning of COD removal problems before they occur. This would allow the loading to be lowered based on OUR/MLVSS rather than COD removal efficiency, which could eliminate a larger upset than normally would occur. Published reports on this subject recommend an OUR/MLVSS of $<10 \text{ mg O}_2/\text{g VSS} \times \text{Hr}$. The BOD II system currently operates at a satisfactory level of $\sim 7.5 \text{ mg O}_2/\text{g VSS} \times \text{Hr}$. This study will continue indefinitely. Preliminary information will be documented. Operations has been contacted to try to get the on line respirometer in service to aid in this work.

Clarifier Operation (Akin) - The Laboratory has initiated a study into clarifier operation to determine the optimum operating controls. A report published by Environmental Dynamics, Inc. indicates in order to optimize clarifier operation (lower outfall TSS, settle solids, recycle solids) one must reach a balance of hydraulic loading to the clarifier and of solids recycle back to the aeration basins. The Laboratory will work with Operations to determine this balance.

Vacuum Truck Content Characterization (Akin, Gonzalez) - In order to develop a system for disposal of vacuum truck wastes, a study was begun in late September to characterize vacuum truck contents. Analyses of these contents include pH, viscosity, specific gravity, specific filtration resistance, % solids, % oil layer, and COD. October composite COD concentration results of each unit that used vacuum trucks are as follows: 1) Paraform - 45,000 ppm, 2) MO IV - 580,000 ppm, 3) Polyol - 29,000 ppm, 4) Landfill - 77,100 ppm, 5) Oxo - 27,610 ppm. 6) MS - 3,110 ppm, 7) MO III - 11,292 ppm, 8) Tank Car Cleaning - 23,400 ppm. Complete analytical results will be published when characterization has been completed.

BOD II Sludge Hazardous Waste Determination (Akin) - Sludge from the BOD II aeration basins has been determined not to be a hazardous waste as determined

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by EPA methodology. EP toxicity result levels were well below maximum permissible levels. Corrosivity of the sludge was also well below the maximum allowable levels. Results of this determination will be published.

Methanol Synthesis
J. C. Bustabad, J. E. Russell

Evaluation of Methods for Determining Microbial Biomass (Bustabad) - A cursory evaluation of the ATP method showed good reproducibility on duplicate samples. Evaluation of this method will continue when more of the required reagents are received.

Familiarization of the literature on the dehydrogenase method of biomass determination has begun.

Monitoring of H₂S in Recycled CO₂ (Bustabad) - An October 26th check of H₂S and COS in recycled CO₂ from the 5 MM Girdler amine system and the Shift amine system showed nondetectable levels (<35 ppb) by Tracor.

Plant Trial of Sodium Metavanadate (NaVO₃) as a MEA Corrosion Inhibitor - Coordination of Laboratory support for a NaVO₃ Plant trial was provided.

Monoethanolamine (MEA) in Storage - A further check of the MEA in storage in V-345 and V-346 showed the sulfur compounds detectable to be relatively stable sulfates (121 ppm SO₄ and 41 ppm SO₄ in V-346 and V-345 respectively). Sulfates in the MEA system are not suspect of creating process problems. However, further Laboratory work was completed to determine the effect of reclaiming on sulfate concentration. A laboratory distillation of MEA from V-345 showed that sulfates, as expected, concentrate in the bottoms. (Overheads were 4.3 ppm SO₄; bottoms were 67 ppm SO₄.) A letter will be issued shortly on this study.

Syn Gas to Oxo - Atypical analyses in the Oxo Unit prompted a check on October 26th of syn gas going to Oxo. An effort was coordinated with CCCTC to determine the concentrations of major components in the syn gas sample. Results showed no abnormalities.

Amines in Clear Lake MeOH (Russell) - Three samples of MeOH were received from Mallinckrodt, one of which was rejected for high odor. Mallinckrodt had purchased the MeOH from Clear Lake through Transchem. The other two samples were free of odor and one also came from Clear Lake while the other was Georgia-Pacific MeOH. An analysis of the high odor MeOH on the ion chromatograph showed approximately 10 ppm monomethylamine. The odor-free MeOH samples showed less than 1 ppm amines. The results were published.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - We continue to have analytical problems providing consistent TPP concentrations in Oxo catalyst. The reactor is operating well.

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Graphing percent of design capacity versus time for Oxo Runs I through VII indicates no cumulative relationship between the percent of time the unit was run above 90% rates and the length of the catalyst life. However, based on Runs I through IV it is believed that after the catalyst is older (e.g. six months), a rate versus time relationship is evident. Based on these runs, older catalyst should ideally not be run below 95% rates for more than 10 consecutive weeks. Longer, low-rate, run times are believed to cause excessive build-up of "foamers" in the catalyst which will necessitate catalyst change out. Work continues to better define these relationships.

Rhodium Accountability - Reactor rhodium accountability for October is about 89%. As with last month, higher than normal reactor rousing causes inaccurate level readings. Using an approximate correction factor the accountability is about 95%.

Losses from T-210 Residue RTD are about 8.2 pounds of rhodium for the run-to-date.

Improved Rhodium Recovery - In a meeting with Operations, Process Engineering, and the Laboratory it was decided to go ahead with a Plant trial to phosphoric acid extract 250 gallons of Run VI catalyst. Work continues. An RFPA for phosphoric acid extraction has been submitted.

Various solutions are being used to extract Run VII catalyst to reduce foaming. No results have been obtained. Foaming tests will be run at CCCTC when all samples are ready. Work continues.

Two stage propylene extractions have been used to isolate a catalyst solution fraction. The fraction is believed to contain high concentrations of foamers. Work continues to further concentrate and identify the foaming agent(s).

An effort was made to prove that Run VI catalyst activity will improve as the 2-ethyl-2-hexenal is stripped as it is added to Run VII. A sample of the Run VI catalyst was maintained in a hydrogen atmosphere at reactor temperature and pressure to hydrogenate the 2-ethyl-2-hexenal. After about 3 hours the 2-ethyl-2-hexenal was dropped from about 40% to about 4%. Catalyst activity before hydrogenation was about 0.85 liters/mole-sec. Afterwards, the catalyst displayed an activity of about 0.92 liters/mole-sec.

Industrial Health
C. E. Gary, K. V. Parker

Personal noise exposures were taken to obtain a better statistical base. Report in preparation.

Additional personal formaldehyde exposures sampling was done to provide a better statistical base. Report in preparation.

A personal noise exposure survey of Maintenance personnel was begun. (KVP)

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The Chemical Laboratory CEA-555 instrument was check-out to determine formaldehyde exposure concentrations. The unit pump failed and the instrument was sent to the factory for repairs. The CP & SC instrument has been borrowed and will be replumbed and checked out. (CEG)

The Metronics Dynacalibrator has been installed and one familiarization run completed. A modified sampling system is being fabricated. (CEG)

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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	January 1, 1981
Formaldehyde Color Problem	Jasek Glenister	1	MOB	Continuing
Burn BG V-778 Residue	Russell	1	Recover Energy and Avoid Disposal Problems	December, 1981
Monitor Conversion in the PE Reaction System	Huelle	2	MOB	December, 1981
Evaluate Filter Cloths For PE Unit		1	Cost Reduction	November, 1981
Recover PE From VCI Waste Stream	Harvey	1	Cost Reduction	March, 1982
Dow Chemical Complaint of our HCHO Causing Slime in their Product EDTA	Glenister Dow	1	MOB	December, 1981
TMP - Extractor Optimization	Day ↑	2	Raw Material Savings	January, 1982
Formaldehyde Stability		1	MOB	December, 1981

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Recover TMP From Formals in TMP Heavy Ends(V-778 residue) Via Alcoholysis	Day	2	Raw Material Savings	March, 1982
Methanol Personal Exposure Monitoring	Gary	1	MOB (Environmental Health)	September, 1982
Area Surveys for Misc. Organics		2	" "	February, 1983
Industrial Health Formaldehyde Methods Validation		1	" "	June 1, 1982
Maintenance Noise Exposure Monitoring	↕	1	" "	January, 1982
Support Polyol Waste Treatment Trial	Young	1	MOB	February, 1982
Conduct Anaerobic Reactor Pulse Test		1	MOB	December, 1981
Polyol/VCI Treatability	↕	1	MOB	February, 1982
Ni(CO) ₄ Method Evaluation	Bustabad	1	Evaluate Current Shutdown and Analytical Procedure.	December 1, 1981
Run VI Oxo Trial Extraction	Presley	1	Cost Reduction	December, 1981
Extend Run VII Catalyst Life	↕	1	Raw Materials Savings	December 31, 1981

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Vacuum Truck Characterization	Akin	1	WMSTF Support	December, 1981
TPP, P ₂ O ₅ Emissions		2	Emissions Quantification	April 1, 1982
BOD II Aerobic Optimization (Sludge Age Study)		1	MOB	October, 1982
Evaluate PAC for Aerobic Optimization		2	MOB/Cost Reduction	February, 1982
Document Solar Pond Treatability Study	Akin Young	1	WMSTF Support	December, 1981
Evaluate OUR, ATP, Dehydrogenase Methods	Bustabad	1	MOB	December, 1981
Support Flocculant Plant Trial		1	MOB	December, 1981

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