

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

From: G. D. Boyd

GDB-190-82

September 8, 1982

LABORATORY DEPARTMENT SUMMARY - AUGUST, 1982

Attached is the Laboratory's monthly summary for August. 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:erg

Distribution:

Chemical

J. J. Fritsch
D. G. Pausky
W. H. Meyer
A. A. Miller
G. R. Scholz
M. D. Harvey
G. L. Cade
C. W. Lee
D. D. Siemonsma
W. L. Sharp
D. E. Lockwood
L. M. Harvey
H. H. Thigpen
A. G. McGehee
B. C. Kerr
F. R. Dow
W. P. Hightower
R. R. Carpenter
R. S. Hahn
J. C. Rubiano
K. R. Stelts
W. H. Brough

Plastics

A. C. Abshier
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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No: 303.3
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HIGHLIGHTS

- A total recycle pulse test conducted on the anaerobic reactor revealed a void volume of 62% in the packed region of the reactor. This is a 4.8% decrease of void volume in a 3-month period.
- A letter documenting the Laboratory evaluation (to date) of solids-removal systems for solar ponds prior to deep well injection has been published (BKH-26-82, DLS-02-82).
- Dow Versene "scum" formers can be removed from HCHO using a carbon bed or XAD-2 resin bed. Both can be regenerated with methanol. The carbon appears to lose about 25% of it's capacity and the XAD-2 loses about 10% of it's capacity after each exhaustion and regeneration. A report will be issued and an economic evaluation will be recommended.
- The laboratory phase of the study to validate our HCHO personnel monitoring method will be complete in September. Field validation studies must now be completed before the protocol is completely satisfied.

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I. ANALYTICAL GROUP

Methanol Oxidation (MO)

A study is underway to determine the acid build rate of 50% formaldehyde. One tank car contains product which has been anion exchanged and one car contains unit production acid level. A report will be issued upon completion of the study.

Paraform Area

The Analytical Group is presently monitoring the quality of the Paraform prilling pilot unit. Desired particle size (70% between 0.3 MM and 0.9 MM plus <1% through 100 mesh) appears to be routinely available.

Utilities, BOD

The Plant BOD Unit started receiving VCI effluent for treatment on August 24, 1982. Both Chemcel and VCI laboratories are monitoring the COD level during the initial transfers for comparative analyses.

Analytical General

Tom Robinson, Analytical Group Technician was transferred to Plastics Co. Laboratory on August 24, 1982. Yvonne Trevino is to be transferred to the Plastics Co. Laboratory on September 7, 1982.

Customer Complaints

8-5-82 - Union Camp Co. Valdosta, Ga. - complained of dark particles in receipts of Paraflake. Returned sample verified the complaint although shipping and retain samples were clear.

8-9-82 - Hoechst Chem. Co. - Brazil and Mexico - complained of 6 month old Para F-Powder failing a heretofore unknown solubility test. Testing of the customer method indicated failure of all age Para (0 - 12 months). We have contacted the customer for better delineation of his method and with suggestions for method revision.

Quality Waivers

None this month.

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PRODUCT QUALITY WAIVER HISTORY

	<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	0	35.1	0	0
PE-Pure	0	0	0	0.4
n-Propyl Acetate	0	0.3	0	0
Diacetone Alcohol	0	5.5	0	0
PE-Tech	0	0	0	0.4
Sodium Formate	0	0	0	3.9
MEK	0	0	0	8.4
PVB n-BuH	0	0	0	2.2

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

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

Analytical Statistics

<u>Sample Count</u>	<u>Aug.</u>	<u>Aug. YTD</u>	<u>Previous YTD</u>
Chemical Section	4240	37,535	36,121
Instrument Section	2309	22,028	22,815
Total	6549	59,563	58,936

Technician Overtime

Chemical Section	64	472	481
Instrumental Section	64	448	310
Total	128	920	791

Sample Shipping

Domestic	63	476	496
Export	2	22	45
Plastics	0	5	9
Others	9	79	79
Total	74	582	629

QA Statistics

Shipments	552	4585	5181
Waivers (QD)	0	15	15
Waivers (SQD)	0	8	8
Complaints	2	13	11

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I. STAFF CHEMISTS

Process Technology

B. C. Kerr

Paraform Unit Efficiency - Yield Accounting data indicates that the year to date efficiency is 1.5% below plan. Further, there appears to be a trend toward lower efficiency. The trend to lower efficiency predates the start-up of the experimental Prilling Operation. The Laboratory plans to begin investigating this problem.

MO-IV Product Acidity - A study of a 50 day run of MO-IV Product (T-33R) acidity shows a skewed distribution of analytical values. Based upon the 50 day data base, acidity values of 0.010 or less will occur only 15% of the time.

Special Analytical

F. R. Dow

Arsenic in Cotton Gin Trash - Cotton gin trash is a potential soil conditioner for solar pond reclamation. The trash contains arsenic compounds as the result of chemical defoliates used for cotton leaf removal.

A sample of cotton gin trash was submitted to the laboratory for arsenic analysis prior to use in a solar pond reclamation test plot. Three portions of the sample showed the following arsenic concentrations:

	Arsenic, ppm
#1	380
#2	360
#3	500

A report has been issued detailing these results.

Another portion of the gin trash sample was water extracted by the EPA procedure and 14 ppm arsenic was found.

Arsenic concentrations were determined at CCCTC.

TMP Quality - Chemical hydroxyl analyses have been run on three samples of TMP (2 Celanese lots and one Perstorp® good sample). All three samples show a chemical hydroxyl value approximately 0.7-0.9wt% less than the calculated hydroxyl value from GC analysis. This discrepancy is being investigated.

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TMP Crystallization Process Samples from CCCTC - Twenty-six TMP samples from the CCCTC crystallization Process Studies were received from Dr. L. O. Wheeler (CCCTC) for analysis. The analyses are complete and the data will be sent to Dr. Wheeler for incorporation into the final analytical report.

Natural Gas Feed to the MS Unit - A sample of natural gas feed to the MS Unit was analyzed on 8-25-82. This spot sample was very similar to natural gas samples analyzed in May and June 1982. Briefly, it contained 1052 BTU/Ft³ and 93.6 mole% methane.

III. SPECIAL PROBLEMS

Group I - H.H. Thigpen

Oxo, Butanol
T. A. Jasek

Oxo Catalyst to Reclaimers - A sample of the composite from the shipment of Oxo Catalyst solution to Johnson-Matthey, Inc. has been sent to JMI for additional, comparative analysis as requested by Dallas (W.W. Wilkison). Analysis showed the assay to be 0.176 wt.% rhodium versus the 0.179 wt.% rhodium previously reported. This change may be due to the settling out of a precipitate which was not completely soluble when heated to 80°C. This sample will be used in an attempt to rectify the difference in rhodium concentrations between JMI (0.16 wt.%) and the Bishop analysis (0.179 Wt.%).

Improved Rhodium Recovery from the Oxo Catalyst Solution - As reported last month, an apparently improved reduction of the Oxo catalyst solution can be accomplished by the addition of fine, powdered aluminum. Use of hydrochloric acid or sodium hydroxide to dissolve the aluminum resulted in rhodium recoveries of 80 and 80%, respectively. However, during the removal of the aluminum, hydrogen is generated. In order to avoid the hydrogen generation and improve the rhodium recovery, the approach of using water soluble salts, namely sodium chloride was tried. Adding finely powdered sodium chloride or potassium chloride resulted in a reduction of the Oxo catalyst solution to a fine ash containing approximately 2.0 wt% rhodium. Recovery of the rhodium was greater than 96% based on the starting rhodium concentration.

Miscellaneous - Isolation of TMP Unknown - A preliminary evaluation of extracting the unknown present in TMP was made. Using methylene chloride as the initial extractant, an increase in the unknown to approximately 20% was observed. However, other identified impurities also extracted into the methylene chloride. Recrystallization will be evaluated shortly, as will the use of adsorbing resins for the isolation of the unknown.

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

Paraformaldehyde Prills (Russell) - The pilot prill unit was completed and started for the first time on July 29, 1982. Nearly the entire month of August was spent on pilot plant unit support. A summary of the pilot prill unit operations for the month can be found in the minutes of the weekly Para Prill Status Meeting and will not be repeated here.

Laboratory studies have continued to a limited extent only. A new method for determining Na in solid HCHO is being tested. It involves dissolving enough solid HCHO (flake, powder, prills) in a 1% triethylamine solution to make a 10% HCHO solution. The solution is performed in a polymethylpentene flask so as to avoid Na contamination from glass. The 10% HCHO solution is then analyzed for Na by AA.

Comparative solubility studies of prills are also being continued. The specific procedure for determining solubility is contained in laboratory notebook #28113. It consists of agitating solid HCHO in water and following amount dissolved with time.

Ester Unit Support (Young) - Support was given to operations during the rerun of contaminated propyl acetate and butyl acetate. This support consisted of unit lab gas chromatography optimization in order to monitor the progress of the purification.

Formcel Unit Support (Young) - Support was given to operations during a time of cloudy formaldehyde feed. The cloud was filtered out by 2 μ m filter paper. The cloud is believed to have been paraform.

OSHA

C. E. Gary, K. V. Parker

Formaldehyde Exposure (KVP) - The area formaldehyde levels in the Paraform Unit is being to obtained data with both vent blowers in operation. Then, we will select times for additional sampling of the paraform prill area when the back-ground from the flaker pan is being blown away from the prilling unit.

Follow-up Formaldehyde Exposure (KVP) - Sampling is continuing to determine the effect of the Phase 1 formaldehyde reduction program.

Formaldehyde Methods Validation (GEG) - The Laboratory methods validation work continues and will be completed by September 3.

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Methanol Oxidation, Formaldehyde Blending

J. F. Glenister, P. W. Young

Summary of Product Color in MO Units (Glenister) - MO-II (T-297)
production showed "moderate" levels of color at 7-9 APHA. This color was due to the presence of higher levels of iron in the product. Atomic Absorption analysis showed 0.18-0.31 ppm iron and treatment of a 7 APHA colored solution with EDTA reduced the color to <5 supporting iron as the cause of the color. Analysis of the methanol feed before and after the cation guard bed showed no change in the resin's capacity for removal of iron from the feed. The source of iron in the product was discovered to be MeOH feed entering T-297 through the anion bypass system. At full rates, it is difficult to provide enough feed to the vaporizer, while feeding methanol solely through the guard bed. For this reason a portion of the MeOH feed was being fed T-297 through the bypass line, without resin treatment. The iron in this MeOH was moving down the distillation column and into the residue. Operations closed the bypass and reduced rates, reducing the iron and color of the product to <.1 ppm Fe and <5 APHA respectively.

MO-III (T-313) production was good at <5 APHA before its shutdown on August 14th. MO-IV production remains very good at <5 APHA.

Laboratory Support for Study of Tray Efficiency in T-315 (MO-(V) (Glenister) - The Routine Analytical Group provided support in a study of the tray efficiency in T-315. D. M. Estes, of Process Engineering is examining the efficiency of the tray section in T-315 at various unit rates. The study is continuing and the analytical results will be documented on completion.

Laboratory Support for MO-IV Unit Stream Bypass of Acid Resin Beds (Glenister) - The Laboratory provided analytical support during a trial bypass of the Anion beds with MO-IV formaldehyde. The MO-IV product stream typically contains a low acid. Operations installed a new transfer line which allows the MO-IV stream to bypass the resin beds and go directly into a blend tank. A trial was conducted to examine the variation in wt% HCHO and HCOOH which might occur during a bypass of the beds. The Routine Analytical Group provided support during the recent high blend and direct transfer trial. The unit stream during the run averaged approximately 53.10 wt% HCHO, 0.008-0.010 wt% HCOOH, indicating a change of only 0.34 wt% in assay during the transfer.

Analysis of Flame Arrestor Blowdown at MO-IV (Glenister) - Process Engineering continues to monitor the amount of carry over of formaldehyde in the Flame Arrestor. The continuous antifoam addition is in service and progress has been made reducing the amount of carry over with its installation. The Routine Analytical Group analyzed samples for wt% HCHO of the Flame Arrestor Blowdown at various blowdown rates. Analysis was also completed for wt% Formic and iron for an indication of any problems in possibly recycling the blowdown stream to the absorber system.

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Removal of Formaldehyde Impurities (Young) - Scum-forming impurities were removed from 37 wt% MO-II formaldehyde by activated carbon and Rohm and Haas XAD-2 resin beds. Each bed was exhausted three times and regenerated 2 times. The carbon bed had an original capacity of approximately 200 bed volumes. An average of 25% of this capacity was lost with each subsequent exhaustion. The XAD-2 bed had an original capacity of approximately 180 bed volumes. An average of 10% of this capacity was lost with each subsequent exhaustion. The formaldehyde feeds to these beds varied in the amount of scum formers present. All of the above data is based on a very scummy formaldehyde feed. This is worst data case.

Methanol Synthesis
J. E. Russell

Ni (CO)₄ Monitor - A new method to insure that Ni(CO)₄ is limited to the OSHA Threshold limit Value (TLV) is being investigated. It has been determined, using literature data (JER-46-82), that Ni(CO)₄ will remain below .001 ppm/v (TLV) if the CO level in the purge gas is below 5 ppm. A Drager tube analysis should easily determine if CO is present in that amount. Analyzer development personnel are being consulted to determine if a more accurate and easier method is available.

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Group II - L. M. Harvey

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

Anaerobic Reactor Pulse Test (Akin, Kaderlik) - A total-recycle pulse test was conducted on the anaerobic reactor August 10. The liquid void volume determined by the test was 0.932 MM gallons. This indicates a loss of 4.8% (from 66.8% to 62%) of the void volume of the packed region of the reactor. Thus far it appears that the void volume decline of the Bishop reactor has been linear since startup. Results of this test are documented in letter DRA-28-82, KRK-07-82, and JM-21.

BOD II Unit Support (Akin) - The anaerobic reactor was operated at an average organic loading of 0.236 lb COD/ft³/day during August. Anaerobic COD-removal efficiency ranged from 61-78%. The aerobic outfall COD averaged 230 ppm with an average detention time of 4.5 days.

VCI/Polyol Treatability (Akin, Kaderlik, Stasney) - The Laboratory anaerobic unit is being fed 100% equalization-basin liquid at a low (0.04-0.08 lb COD/ft³/day) organic loading in an effort to slowly bring the unit back up to loading (0.30 lb COD/ft³/day). The aerobic unit is being fed supplemental anaerobic recycle liquid from BOD II to maintain a 2.7-3.0 day feed detention time. The anaerobic outfall has ranged from 2000-4000 ppm COD. Once anaerobic activity improves and loading can be increased, the polyol and VCI effluent contributions to the feed will be reintroduced.

Aerobic Digestion of Ultrasonically Ruptured Cells (Akin, Kaderlik, Stasney) - This study was initiated to determine if bacteria whose cell walls had been ruptured could be digested by aerobic bacteria at such a rate as to be feasible for full-scale operation. The second stage of the study began in early August. In this stage both the control and the experimental tanks are fed equal amounts of anaerobic recycle liquid in addition to the ruptured biomass fed to the experimental tank. Both tanks have exhibited virtually the same COD removal efficiency with the same 3 day feed detention time. The experimental tank still maintains a higher VSS (about 15% greater than the control). There is very little difference in the oxygen uptake rates for both tanks. As expected the experimental tank exhibits a higher outfall VSS than the control. More data is required before conclusions can be drawn to the feasibility of this type of operation.

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Extent of Mixing and Aeration in Aeration Basin No. 2 (Kaderlik) - A
study was conducted to determine the extent of aeration and mixing in Aeration Basin No. 2 under two different conditions: (1) operating the aeration basin with two aerators and (2) operating the aeration basin with four aerators. A report has been issued. (KRK-08-82)

Waste Management
B. K. Huelle

Solar Pond Solids-Removal Studies (Huelle) - A letter documenting the laboratory evaluation (to date) of solids-removal systems for solar ponds prior to deep-well injection has been published (BKH-26-82, DLS-02-82).

Core-permeability tests to determine the chemical compatibility of each solids-removal process's liquid with the core matrix has been done. (Samples are filtered through .45 micron filters to remove solids prior to testing.) The tests show that precoat and ultrafiltration (UF) product are chemically compatible. Flocculation product using polymer also passed the test. However, it was discovered that 1 ppm polymer in brine solution, not filtered through .45 filters, failed the test. Apparently polymer is being filtered out onto the .45 micron filters. Core-permeability tests will be repeated on more advanced equipment in the future.

The risk of plugging the wells associated with a flocculation/clarification process using polymer could be reduced by (1) ensuring that there is no residual polymer in the clarified product, (2) ensuring that polymer is removed by 5 micron cartridge polishing filters, or (3) flocculating with an agent other than polymer.

An analytical method was developed to determine polymer concentration. Flocculation product was shown to contain 1 ppm polymer after a 10 ppm addition for flocculation. Polymer solution filtered through .45 micron filters indicated that no polymer was present. Polymer in flocculation product is still present after filtration through 5 micron millipore filters, and therefore, 5 micron cartridge polishing filters will not be useful in removing polymer. As a result of these findings, there is a definite risk of plugging the wells if flocculation product containing polymer is injected.

The third alternative, flocculating with other types of agents, has been studied. Bentonite clay is more effective than aluminum sulfate (alum) in solids-removal. Flocculation using 500 ppm clay is just as

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effective as flocculation with 10 ppm polymer and 200 ppm clay, and more effective than using the polymer alone. Bentonite clay levels above 500 ppm indicated no improvement in solids removal based on turbidity measurements. A major drawback of a flocculation process using the clay is the slow settling rate of the floc in comparison to floc formed with polymer. Flocculated solar pond samples using bentonite clay are being centrifuged before evaluation by turbidity.

General Filter Co., a clarifier manufacturer, has indicated that approximately 70% of the solids over one micron are removed by flocculation using 10 ppm polymer and 200 ppm bentonite. The remaining 30% appear to be microbes. Laboratory microscopic evaluation of solar pond material have confirmed their findings. The Laboratory is currently determining if these microbes can be removed by some process or at least reduced in size. Microbes appear to range from .5 to 3 microns in diameter, and are seen individually and in clusters. The possibility that killing the microbes will reduce their size (thus, decreasing turbidity) and improve the suitability of solar pond flocculation product for injection does exist.

Ultrafiltration product has been shown to be virtually free of microbes and to yield a 99% decrease in turbidity as compared to UF Pond 11E feed. However, the UF pilot study has not shown promising results, to date. The problems with the UF pilot are being addressed by Process Engineering.

Pentaerythritol

D. R. Akin, R. L. Day, J. C. Bustabad, K. R. Kaderlik

PE Hydrolysis Resin Bed Support (Akin, Day, Bustabad) - The Laboratory continues to support Operations with the operation of the PE hydrolysis resin bed. For the major portion of August the unit was on a tech run. The unit came up on a pure run on August 19.

Sodium formate (NaFo) levels of the dissolve tank liquid have on the average been less than 1000 ppm. This improvement in the NaFo levels can be traced to improved 'A' belt operation during the month of August. For efficient conversion of linear formals, the PE stream to the resin bed must have NaFo levels less than 1000 ppm. The pH control of sulfuric acid addition to the process stream improved significantly with the removal of steam injection from the PE stream. Currently the acid addition is being optimized by gradual increases in pH. The Laboratory continues to monitor the conversion of linear formals across the resin bed with these increases in pH. Consequently, with reduced acid addition

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rates the Na_2SO_4 levels in the AML will also decrease. Thus far this month the Na_2SO_4 levels have not been consistently less than 0.5 wt% (formate base). Laboratory support will continue in this area.

Separation of UV Absorber from PE (Kaderlik) - Samples of high UV absorbing PE were eluted through an XAD-2 resin to separate out the UV absorbing contaminate. Efforts are being made to isolate and characterize the contaminate.

Trimethylolpropane
J. C. Bustabad

NaFo Analysis on T-204 Residue - T-204 residue analysis for NaFo can be completed successfully using the Orion 901 ionalyzer in the relative millivolt mode instead of the concentration mode that is now used. This method will be issued in September for use in the Unit Lab.

1,3 Butylene Glycol
J. C. Bustabad

Alternate Catalysts - Equipment is being assembled for hydrogenation studies using Degussa's BK113W and Grace's 3100 Raney Nickel.

General

- J. E. Russell and H. H. Thigpen attended a Paraform Prill meeting at the Tech Center on August 13.
- J. C. Bustabad attended a meeting at the Bay City Plant on August 25 to discuss the results of BCP testing of Degussa Raney Nickel catalyst.
- R. L. Day resigned effective September 1.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Industrial Health Formaldehyde Methods Validation	Gary	1	MOB (Environmental Health)	September 1982
Methanol Personal Exposure Monitoring		1	MOB (Environmental Health)	January 1983
Area Surveys for Misc. Organics		2	MOB (Environmental Health)	February 1983
Confirm MO Unit Efficiency Losses Via Vent Sampling and Analysis	Glenister	1	MOB, CR	October 1982
Stability of 37% Unstabilized HCHO (for Dow Chemical Co.)		1	MOB	November 1982
GC Methanol Oxidation Reactor Optimization		2	Catalyst Evaluation for Possible Efficiency Improvement	November 1982
Formaldehyde Color Problems		1	MOB	Continuing
MO Catalyst Screening		2	To Establish Alternate Catalyst	Continuing
Improved Rhodium Recovery From Oxo Catalyst Solution	Jasek	1	CR	October 1982
Rhodium Accounting		1	MOB, CR	Continuing
Isolate Impurities From TMP Product for Identification		2	MOB	September 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Support Paraform Prill Pilot Operation at Bishop	Russell	1	CR, MOB	December 1982
Removal of Scum forming impurities from Formaldehyde	Young	1		September 1982
Evaluate Alternate Catalyst for 1,3-BG Hydrogenation	Bustabad	1	MOB	September 1982
TMP Expansion - Purification Train Simulation		1	MOB	December 1982
Update TMP Unit Methods Manual		2	MOB	September 1982
Condensing Polyol Vents		2	Cost Reduction	October 1982
Optimize Solar Pond Filtration Prior to Deep-Well Injection	Huelle	1	WMSTF Support	September 1982
Evaluate Alternate Filter Cloths for PE Filters		2	MOB	October 1982
Improve PE Quality - UV Absorbance and Formal Content	Huelle/ Kaderlik	1	MOB	September 1982
TPP, P ₂ O ₅ Emissions	Kaderlik	1	Emissions Quantification	October 1982
Evaluate "Unsteady-state" Polyol, VCI and Other Effluents	Akin/ Kaderlik	1	MOB	September 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Aerobic Degradation of Ruptured Sludge	Akin/ Kaderlik	1	MOB	October 1982
BOD II Aerobic Optimization	Akin	1	MOB	October 1982
Evaluate ATP, OUR and Dehydrogenase Methods		2	MOB	October 1982
Follow Performance of PE Resin Bed	Akin/Day	1	MOB	Continuing
Determine Cause of MS Carbon Bed Deactivation	Day	1	MOB	June 1982
Formaldehyde Stability		2	MOB	June 1982
Recover PE from VCI Waste Stream		1	Cost Reduction	September 1982
Update PE Unit Methods Manual		2	MOB	September 1982

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