

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: G. D. Boyd-32

GDB-63-80

August 5, 1980

Laboratory Department Summary

July, 1980

DISTRIBUTION

Chemical

P. E. Hime
W. H. Brough
R. R. Carpenter
J. Chervenka
K. G. Gallimore
B. P. Hanson
J. M. Hodges
R. W. James
B. C. Kerr
C. W. Lee
D. E. Lockwood
R. P. Lowry
A. G. McGehee
D. G. Pausky
G. R. Scholz
D. D. Siemonsma
W. L. Sharp
H. H. Thigpen
W. H. Meyer
Bishop Files

Plastics

A. C. Abshier
R. G. Brandt
R. L. Bunkley
H. L. Davis
E. J. Heinz
H. W. Hill
E. Munoz

CCCTC

G. J. Fisher
W. E. Heinz
W. T. McNair
Central Files (2)

J. N. Gann, Bay City
J. E. Sanborn, Bayport Termini
J. W. Dunn, Clear Lake
T. H. Golson, Dallas
A. J. Bran-
W. H. Tuke
F. B. McAnc
J. Zitomer,

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AUG 13 1980

CELANESE CHEMICAL COMPANY
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Bishop, Texas

To: C. W. Harrison

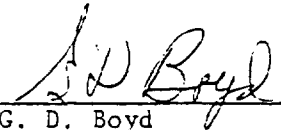
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Laboratory Department Summary
July, 1980

A summary of each Groups' Monthly Report is 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 Groups and Staff Chemist projects by Chemist assignment. A table listing the priorities, justifications, estimated completion dates of current Special Problems projects and a graph of the Laboratory Effort Analysis is also included in Section II.


G. D. Boyd

GDB:jr

Attached Report

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I. ANALYTICAL GROUP

Competitive Evaluations

Six competitive samples of Methyl Alcohol (MeOH) (Du Pont, Tenneco, Borden, Air Products, Monsanto, and Georgia Pacific) and a sample of the Clear Lake Plant MeOH product were analyzed this month. A regular comparative report will be issued. Specially requested Amine determination work on these samples will follow as priorities permit.

Methanol Oxidation

Samples of Technical Center Pilot Plant two stage HCHO product and Chemcel Plant typical HCHO were analyzed for comparative analysis. Data gathered is preliminary for discussions with Derivados Forestales concerning new Paraformaldehyde technology.

Polyol

Hot and cold Sulfuric color determinations were made on a series of PE finished product. Data was furnished for an ongoing study to determine correlation between PE quality and customer end use color problems.

Utilities and BOD

Daily pH determinations were made on all Plant effluent sumps from 5-28-80 to 6-27-80. This was to assist Engineering in design of a pH control system for the BOD unit feed.

Four effluent samples from the Celaya, Mexico Plant were analyzed to provide data for operating conditions for Celaya's new BOD Unit.

Waste oil drum sampling is complete. Coordination of disposal is being handled through Environmental, Utilities and Maintenance Departments.

Analytical Group General

Quality Assurance - Updating and approval, under revised format, of Manufacturing Control Specifications. is complete. Revisions have been forwarded to Dallas Quality Assurance where issuing is scheduled for last quarter 1980.

Revised Quality Notice (to customers) procedures, in which we report against sales specification rather than reporting actual analysis, is progressing well. Since May 1, 1980 start only one customer, Upjohn - 37% HCHO, has requested actual analysis (Certificate of Analysis). Standard Quality Notice forms for Plant products are being prepared. Their use will result in a time savings for clerical personnel.

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Special Analytical Services

F. R. Dow, Chemist, and B. W. Wise, Technician, transferred to the Analytical Group this month. Special analytical services will be performed in the Analytical Group. Plans are to transfer one additional professional from the Special Problems Group as manpower permits. Group rotating technicians will be utilized to help provide special services as the work load allows.

Exposure Testing - Chevron Products - Accelerated exposure testing of potential Plant use coatings items is underway. A potential ditch lining material, Chevron Industrial Membrane, and a potential concrete slab coating material, Chevron Sucoat are being tested against selected Plant products. Completion of testing is expected in mid-August. A report will be issued to Plant Engineering.

Assistance to CCPC - Analytical assistance in the determination of the purity of start up Propylene product was given to the neighboring Corpus Christi Petrochemical Co. during the PM of 7-18-80. A Laboratory instrumentation failure at CCPC necessitated the call for assistance. The product was good at 99.9% purity and no further assistance was requested.

PE - Monitor - F. R. Dow

The Laboratory Liquid Chromatograph is being used to isolate Di-PE from crude Di-PE crystals. Enough of the high purity Di-PE has been separated to prepare several synthetic blends for comparison with the Laboratory PE-Monitor. Based on limited data the Di-PE blends support the 8.2 wt.% Di-PE content of the Laboratory Monitor.

Quality of Plant Air - F. R. Dow

A F.I.D. G.C. was installed in Boiler House #1 to monitor Plant air just prior to entry into the main loader system.

Previously reported results were confirmed as G.C. peaks were observed when the Boiler House pick-up truck was started or stopped near the compressor air intake. Boiler House operators also labeled peaks associated with the diesel switch engine passage.

Previous reports have described the impurity level as determined in the Laboratory, but more impurities for longer periods of time were recorded at the Boiler House location.

Special Analytical Services Projects Pending

1. Continue evaluation of Liquid Chromatograph and train Laboratory personnel in its use.
2. Use Liquid Chromatograph to check composition of PE standard and upgrade analysis if required.

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3. Arrange for and present a Varian Basic G.C. Course for Laboratory personnel.
4. Organize and present an Atomic Absorption Course for Laboratory technicians.
5. Install purchased Capillary G.C. and scan Plant products for applicability.

Customer Complaints

July 7, 1980 - Delta Solvents, Shreveport, La. - HCHO 37-12/15 Inh. - Shipment made 11-14-79 - Their customer (Cooper Rubber) complained of 10% acidity in a receipt. Sample of "similar material" returned to us showed 0.004% acidity. Complaint classed as non-valid.

July 14, 1980 - Ciba Giegy, McIntosh, Ala. - PE Pure - Customer found combined Linear Formal and Di PE content to be 1.2% vs. his maximum allowable of 1.0%. Original and Retain analysis confirm that 0.9% combined content was shipped. Complaint classed as unresolved pending receipt of returned sample and customer methodology.

July 2, 1980 - Southwest Solvents and Chemicals, Dallas, Tx. - Compartmented trailer of Ethyl Acetate and Propyl Acetate arrived with reversed compartment seals. Unloading based on seals resulted in inventory contamination. Customer kept products at distressed price. Complaint classed as valid.

July 24, 1980 - Arco Chemical, Sand Springs, Ok. - Para Flake - Customer complained of lumps in product plugging feed orifice. 27,300 lbs. to be returned as customer service for "non typical product." No specification on particle size. Complaint classed as valid.

Waivers and Delays

None this month.

Analytical Statistics

Sample Count

	<u>July</u>	<u>July YTD</u>	<u>Previous YTD</u>
Chemical Section	3892	33123	34527
Instrument Section	<u>1965</u>	<u>17001</u>	<u>16461</u>
Total	5857	50124	50988

Technician Overtime

Chemical Section	18	539	522
Instrument Section	<u>24</u>	<u>239</u>	<u>363</u>
Total	42	778	885

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Sample Shipping and Quality Assurance Statistics

Statistics For July 1980

<u>Sample Shipping</u>	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Domestic	69	475	460
Export	1	16	23
Plastics	6	55	30
Other	6	50	103
Total	82	596	616

Quality Assurance Statistics

	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Shipments (NO.of Orders)	527	5543	6377
Delayed	0	3	18
Waviers <u>Internal</u>	0	6	7
<u>External</u>	0	4	17
Complaints (Total)	4	24	38
Complaints (Legitimate)	2	9	14
Complaints (Non-Legitimate)	1	14	18
Complaints - Outstanding (Unresolved)	1	1	6

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II. SPECIAL PROBLEMS GROUP

Methanol Oxidation, Formaldehyde Blending T. A. Jasek

MO Silver Catalyst Screening - Shipments of both Handy and Harman and Engelhard Catalyst were received. Both shipments were acceptable. No further work is planned at this time.

Crude Methanol Plant Trial - The resin bed used during the Crude Methanol Plant trial was opened and sampled. Evaluation of the resin capacity for iron at various levels in the resin bed is in progress.

Laboratory Sinter Test Vs. Catalyst Performance Correlation Study - Sampling of the Matthey-Bishop catalyst charge to determine if the Laboratory Sinter Test provides useful information on catalyst performance has been completed. The catalyst, when removed because of low efficiency, was completely sintered. A report describing this study will be issued.

G. C./MO Reactor - An MO reactor has been installed in a Gas Chromatograph to study Methanol Oxidation parameters. Preliminary studies will be directed to catalyst evaluation, efficiency improvements, and the effects of various contaminants on catalyst life. Initial data, generated with this equipment, indicates that useful information will be obtained. Mr. Keith Arthur has provided valuable assistance in modifying G. C. equipment for this work.

Boiler Vent Analysis - At the request of M. Lyons, who is conducting a Boiler efficiency study, the Laboratory evaluated several Boiler vent samples. Work is continuing in this area.

HCHO Stabilizer Studies - (R. L. Day) - Work continues in preparation for a Plant test to add polyvinyl alcohol/metolose stabilizer to 50% HCHO shipments. Mixing techniques have been developed, and samples have been stored at 40°C to repeat/reproduce original stability tests. Results are not yet available. Tests are continuing.

Work thus far indicates that Polyvinyl Alcohol at the 3 ppm level will not interfere with our current method for analyzing metolose.

Attempts are being made at the present time to quick-freeze gallon samples of HCHO, to be able to send customers large samples of HCHO with the new stabilizer mix.

MO Unit Support - The Laboratory ran analytical AMR's at MO-IV on July 25, 1980. The table below illustrates the analytical and indicated Control Room AMR's for A and B trains. Also illustrated are the AMR's observed on rechecking on July 28, 1980.

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<u>Date</u>	<u>Train</u>	<u>Analytical</u>	<u>Indicated</u>
25.7.80	A	0.60	0.89-0.90
25.7.80	A	0.72	0.98-1.00
25.7.80	B	0.68	0.85-0.92
25.7.80	B	0.77	0.98-1.02
28.7.80	A	0.80	1.14-1.18
28.7.80	B	0.98	1.00-1.02

Utilities, BOD
S. G. Smith, P. G. Welling, G. R. Stasney, B. W. Wise

Anaerobic-Aerobic Pilot Studies

Evaluation of TOD Analyzer - The COD/TOD correlation study has been completed. A report detailing this work has been issued, SGS-32-80. (Smith)

Anaerobic-Aerobic Pilot Unit #1: Effects of NaOH Addition to Anaerobic Recycle - Caustic (1.0 N) was added in-line to the recycle at a constant flowrate, while organic loading to the filter was increased from 0.10 to 0.6 lb. COD/ft³/day. This corresponds to adding 600 to 200 ppm Na⁺ in the feed. During this time, filter pH remained in the proper range of 6.6 - 7.6.

The pH of the recycle at the point of caustic addition was also investigated. Adding caustic to the recycle in the amounts corresponding to levels of 350 to 700 ppm Na⁺ on the feed resulted in pH increases of 0.3 to 0.6 pH units at the point of addition (Recycle to feed ratio = 10:1), and in some cases, caused the recycle pH to rise to unacceptable levels, i.e., greater than 7.6.

This study has been completed. A report is being written covering Na⁺ addition to the anaerobic filter. (Smith, Stasney, Wise)

Start Up Studies/Upflow Velocity Study - Two anaerobic-aerobic units were started up to further study the switch from the present aerobic waste treatment system to the future anaerobic-aerobic treatment system. Also to be investigated are the effects of upflow velocity on filter startup and gradual solids accumulation in the filter bed. The aerobic units were started on July 12th, with equalization basis feed. Acclimatization to the feed with a 3.5 day detention and outfall CODs < 200 was attained on July 18th. Anaerobic units 2 and 3 (void volumes = 20 liters) were started on July 23rd. The units were seeded with 4.5

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volume percent San Antonio Rilling Road secondary anaerobic digester sludge and 0.5 vol.% aerobic blowdown from the BOD unit. Filter #2 is run at an upflow velocity of 1 to 2 ft/hr (to maintain the 10:1 recycle to feed ratio), while #3 is run at an upflow velocity of 10 ft/hr (to simulate the upflow expected in the commercial unit). The feed (containing equalization basin, T-312 residue for Na^+ and nutrients N and P) was started to the filters at an organic loading of 0.015 lbs/ft³/day. Formaldehyde levels are being monitored closely. Gas-make was first detected in filter #3 after 7 days of operation. As of July 30th, gas-make has not been detected from filter #2. Anaerobic outfall CODs have run 400 to 600 ppm. After aerobic polishing, the outfall CODs have been <150 ppm. (Welling, Smith, Stasney).

Operations Manual - No assistance provided this month. (Smith)

BOD Unit Support - The unit method for analysis of nitrate nitrogen in aeration basin samples is being checked by three methods: (1) HACH kit, (2) Specifiction electrode, (3) Chromatiopic acid method. Analytical interferences are hindering confirmation of one method by another. The HACH kit is definitely limited in its reliable range and is greatly affected by the presence of carbonate and bicarbonate ions. Work continues. (Wise)

Oxo Butanol
J. D. Presley, N. Elizalde

Reactor Stability/Accountability - The Oxo reaction system has been down the entire month of July.

Based on Laboratory data, the decision has been made to bring the unit up with new catalyst. However, effort continues to reactivate Run VI contaminated catalyst. If it can be reactivated, the Run VI catalyst could possibly be used for Rh make-up in Run VII. The probability for economical reactivation is low.

Attempts to determine levels of amine in the contaminated catalyst have been unsuccessful. The BuH by-product high-boilers mentioned last month have been identified as primarily 2-ethyl-2-hexenal and 2-ethylhexanal.

Rhodium Accountability - Contaminated Run VI Oxo catalyst was transferred from the catalyst dump vessel to the catalyst recovery tower, T-168. There, the catalyst was thoroughly mixed and sampled. Rhodium analysis of these samples and known rhodium losses in T-210 Residue sum to essentially 95% accountability.

A letter was issued (JDP-66-80) showing minimal Rh losses in water drained from the dump vessel.

Improved Rhodium Recovery - The effects of ambient temperature and BuH concentration (of the catalyst solution) are being studied in the hydrocarbon extraction study. Completion of this phase of the study has been delayed pending repairs to the Laboratory walk-in refrigerator. In the meantime, the third

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phase, activity testing of extracted catalyst fractions, has been started.

Oxo Unit Support - A letter (JDP-67-80) with detailed results of Bishop Laboratory tests with Bay City T-4 Residue concentrate has been issued.

Methanol Synthesis
J. E. Russell

MS-II Unit Support - Another Chemist, Dan Akin, was familiarized with the Ni(CO)_4 analysis of purge gas. Mr. Akin subsequently performed the Ni(CO)_4 analysis when the Selas was shut down for maintenance on July 13.

Butyraldehyde Hydrogenation Improvement - Equipment is being gathered for testing modified Raney Nickel as an improved hydrogenation catalyst. Due to priorities, these samples have not been tested yet. Further work will proceed as time permits.

Identification of Components in San Fernando Creek - Samples of soil around a small organic outcrop seeping into San Fernando Creek are being analyzed for characterization of the seepage. Samples of creek water upstream and downstream of the seepage point are also being analyzed to determine the effect of the seepage on water quality. Work is continuing on this project.

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

The lime pit area was sampled using Draeger tubes for hydrogen sulfide during vacuum truck removal of wastes. The results ranged from 1.0 - 1.5 ppm HgS well below the 15 ppm short term exposure level. (KVP)

Additional personal exposure samples to formaldehyde were taken. (The initial Plant-wide survey is nearly complete). (KVP)

Numerous samples for nickel exposure were obtained and analyzed while Selas catalyst was being loaded into the reformer tubes. All results were below the permissible exposure level of 0.1 mg/meter^3 . (KVP)

The results of formaldehyde analysis using the B & L Spectronic 20 and the new B & L 2000 spectrophotometer were the same. Therefore, the Spectronic 20 will continue to be used to save time. (KVP)

The evaluation of long term Draeger tubes to determine the 8 hour TWA for carbon monoxide continues. Sampling pump problems have caused erratic results. A pump designed especially for long-term Draeger tubes, has been ordered from Draeger. (CEG)

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1,3-Butylene Glycol
B. K. Huelle, P. J. Buras

Unit Support - None provided in July. (Huelle)

Improved Aldol Condensation and AcH Recovery - A new low energy/high efficiency processing scheme is being studied whereby a mixture of AcH and Pentane is passed over a solid catalyst bed. An AcH/Pentane mixture is then separated from aldol in a distillation column. Separation of Aldol from Pentane is accomplished by decantation and/or extraction into a Butanol or Ethanol solvent for hydrogenation.

It was not possible to prevent rapid deactivation previously observed using macroreticular ion exchange resin catalysts. The calcium hydroxide catalyst used in the DAA reactor showed higher initial conversions and extended catalytic activity compared with the ion exchange resins previously investigated. It was possible to reactivate the DAA type catalyst by washing with DI water, whereas the ion exchange resins could not be reactivated.

The bench scale reactor was rebuilt with twice as long a catalyst bed in order to improve mixing by increasing velocity through the bed while maintaining the same residence time. Different reactor conditions (e.g., temperature, solvent, residence time) are being investigated to minimize by-product formation and extended catalyst life. (Buras)

DiAcetone Alcohol
B. K. Huelle

Unit Support - None provided.

Acetates
R. L. Day

Reactor Corrosion Control - Copper (II) Phosphate corrosion inhibitor is being added to the reactor kettle, 1408. The Laboratory is monitoring copper levels to determine when more corrosion inhibitor is needed.

The first batch of copper (II) phosphate was added in April. The corrosion coupons will be checked around October, to determine how well the corrosion inhibitor works in this system.

Paraform, Formcel Products
R. L. Day

Unit Support - None provided due to higher priority work and the absence of serious problems in this area.

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Trimethylolpropane
R. L. Day

TMP Extractor Optimization Studies - Base case data is being taken on the overhead and residue streams of the 312 extractor. Studies continue.

Low Butyraldehyde Efficiency - Butyraldehyde efficiencies for this month have been 77%; plan is 85%. Samples of T-202 overhead were analyzed for BuH by-products. If insufficient caustic levels exist, BuH by-products will form and can be detected in 202 OH. Calculations of efficiency losses are not yet complete. Work continues

Pentaerythritol
L. M. Harvey, D. R. Akin, B. K. Huelle, P. J. Buras

Recovery of PE from VCI Centrate - Shake-out studies were conducted on the extraction of a mixture of VCI Centrate and PE WIP recycle with n-Hexanol. This extraction has been proposed as an alternate to the extraction of PE AML with n-Hexanol (LMH-74-80). A report has been written and will be issued in August (LMH-82-80).

Physical property data on streams to be exchanged between Celanese and VCI continues to be generated to assist Process Engineering in the preparation of the preliminary process design and material balance. (Harvey)

PE Formal Hydrolysis - Work was continued using ion exchange resins as co-catalysts in conjunction with continuously fed strong acid. Amberlyst 15 exposure time to date is 67 days. Activity on this resin still appears to be good. Activity-life studies will continue. Variable studies of the effects of sodium concentration and acid concentration (with a constant residence time) on conversion are underway on Amberlyst 15.

The screening of Amberlite IR-118(H) and Amberlyst XN-1010 will begin in August. Delay in screening was due to PE unit being down most of July. (Akin)

Development of a System to Monitor PE Reaction Conversion - A series of batch reactions with varying caustic to acetaldehyde mole ratios were performed. Efforts are being made to duplicate unit conditions and AML composition. AML composition is important since recovered efficiencies will be calculated from the AML. Reaction samples taken after the neutralization step will be analyzed by redox potential and other methods in an effort to correlate these measurements to conversion. (Huelle)

Dow High Formal/Color Complaint - The Laboratory is cooperating with Dow (Midland, Michigan) to improve the quality of PE used in their production of dibromoneopentylglycol (DBNPG). Retain samples of PE normally show a hot melt color of 2 to 6 Gardner units. Lot 80-21-1P, which produced high color DBNPG, had a hot melt color of 15. It may be possible to use this test to

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select lots for shipment to Dow. As data from Dow on the performance of different lots of PE becomes available, we will determine the best method of preventing this quality problem. Five samples of PE made during successive stages of the carbon bed's life were sent to Dow for their examination. (Buras)

Hazardous Waste Characteristics of VCI Centrate - Waste centrate currently dumped by Virginia Chemical, Inc. into Celanese solar ponds does not exhibit the hazardous waste characteristics of ignitability, corrosivity, or reactivity as defined by EPA. This judgement is based upon previous handling experience and characteristics of major components (NaFo, PE organics). Analysis of a sample of centrate for heavy metals required for EP toxicity determination is in progress. It is expected that centrate will not be classified as a hazardous waste. (Buras)

General

1. Becky Day attended a Gas Chromatography training course in Houston, Texas on July 16-18.
2. Becky Day, Dan Akin, Tom Jasek, and Susan Smith began an in-house seminar on effective oral communication on July 29. This will continue through August 1.
3. S. G. Smith attended a meeting at CCCTC on July 15, 1980 and gave a presentation of Bishop Laboratory work concerning anaerobic filter technology to representatives of Ralston Purina Co., interested in licensing Celanese technology for waste treatment. She also conducted a tour of the Bishop facility now under construction.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Jasek	1	To Establish Alternate Catalyst Suppliers	Continuing
G.C. Methanol Oxidation Reactor Studies		2	Catalyst Evaluation for Possible Efficiency Improvements	Continuing
Oxo Reactor Accountability/Stability	Presley	1	Improve Reactor Control	Continuing
Rhodium Accounting		1	MOB, Catalyst Savings	Continuing
Improved Rhodium Recovery		1	Raw Materials Savings	December, 1980
Anaerobic Testing of Chemical Wastes	Smith Welling	1	Support Unit Expansion	Continuing
Formic Acid Removal From Formaldehyde	Russell	2	Cost Reduction	September, 1980
Butyraldehyde Hydrogenation Improvement		1	Cost Reduction	September, 1980
Develop A Low Energy-High Efficiency Acil-Aldol Separation Process For The BG Unit	Buras	2	Raw Material and Steam Savings	September, 1980
OSHA Monitoring	Gary	1	Insure Employee Health and Safety, OSHA Compliance	Continuing
Recover PE from Virginia Chemical Company Waste Stream	Harvey	1	Raw Material Savings	Undetermined

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Develop an Improved Hydrolysis System for PE Linear Formal	Akin	1	Steam, Acid and Capital Savings	September, 1980
Develop a Way to Monitor Conversion in the PE Reaction System	Huelle	1	MOB (Quality) and Raw Material	November, 1980
TMP Extractor Optimization	Day	2	Raw Material Savings	Undetermined
Ester Reactor Corrosion Control		2	Extended Equipment Life	December, 1980
New Formaldehyde Stabilizer System		1	MOB	September, 1980