



CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: R. R. Graham

From: G. D. Boyd ⁶¹

CDB-82-80

November 6, 1980

Laboratory Department Summary
October, 1980

Distribution

Chemical

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

Plastics

A. C. Abshier
R. G. Brandt
R. L. Bunkley
H. L. Davis
H. W. Hill
E. Munoz
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 Terminal
J. L. Paul, Clear Lake
T. H. Golson, Dallas
W. H. Tuke, Pampa

File: 303.3

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CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: R. R. Graham

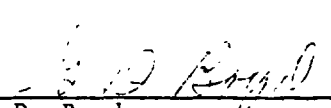
From: G. D. Boyd

GDB-82-80

November 6, 1980

Laboratory Department Summary
October, 1980

A summary of each Group's 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

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I. ANALYTICAL GROUP

Competitive Products

The following products were received for evaluation in October: Union Carbide- n-Butyraldehyde and n-Propyl Acetate, DuPont - Methyl Alcohol, Perstrop- pure and tech grade Pentaerythritol and American Hoechst Corp.- n-Butyl Alcohol. Reports will be issued as analyses are completed.

Methanol Synthesis (MS)

A barge of Methanol (SC-1700) was received from the Bayport Terminal on 10-1-80. One compartment(#2 Starboard) contained a high water content 0.48 wt.% (spec. 0.10) and a chloride level of 30 ppm (spec. < 1). It was determined that sea water had leaked into the compartment during transport. The material was reshipped to a non-critical customer.

Special analytical support was provided for the recent unit start-up.

Oxo Butyraldehyde (BuH)

Triphenylphosphine - The Analytical Group analyzed a sample of Ihara Corp. (Japanese) triphenylphosphine by our routine analytical methods to determine if the material meets Celanese purchase specifications. This is part of an effort to qualify Ihara Corp. as a TPP supplier. A report will be issued upon complete evaluation.

Paraform

Screen analyses are being run on Paraform flake production and shipping lots. This is base case data to establish lump specifications on Paraform flake production. Excessive lumps continued in the Paraflake production until the unit shutdown for maintenance on October 20, 1980. Only the west production train has operated since the start-up. It has been relatively lump free.

Polyol

Trimethylolpropane (TMP) - Extensive analyses continued to be provided this month in search of the TMP acid problem. The acid level has diminished to 0.001 to 0.002 wt.% (Spec. 0.002 max.) Work continues to establish control of this problem.

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Pollution Control

Samples taken from test wells from various locations on Celanese property and from the solar ponds were analyzed for total organic carbon. This work is in support of the current study to characterize the solar ponds and to determine the extent of solar pond seepage. This study is continuing and Analytical Group support is being provided on a high priority basis.

Analytical Group General

Mr. Jim Vargas transferred to the Instrument section from the Wet Chemical Section and Mr. Jerry Foster to the Wet Chemical Section from the Instrument Section on September 29, 1980.

Special Analytical Services

PE - Monitor (F. R. Dow, B. W. Wise) - Confirmation of the correct values for the Tech PE Monitor has been completed. Final values were obtained with a Laboratory prepared sample of 99+ wt.% Tri-PE. With pure samples of Di and Tri PE the Tech PE - Monitor was found to have 8.8 wt.% Di-PE and 1.6 wt.% Tri-PE rather than 8.2 wt.% Di-PE and 2.2 wt.% Tri-PE now being used for calibration.

A report has been issued detailing the results.

Identification of Solar Pond and Monitor Well Components (F. R. Dow, J. E. Russell, B. W. Wise) - A capillary G.C. is being utilized in an attempt to identify the organic components in the solar ponds and monitor wells. Current Plant streams are also being analyzed so that solar pond components can be correlated with specific units.

Another phase of this investigation is to determine a "tracer" component to monitor solar pond seepage.

As analytical methods are developed, the Laboratory will determine the organic components of each individual solar pond. This will be supplemented by an inorganic analysis of each solar pond and monitor well as time permits. The total effort should result in a complete chemical analysis of each solar pond and monitor well so that future options on how to eliminate the solar ponds may be more clearly defined.

Projects Pending

- (1) Amines in competitive MeOH.
- (2) Sampler material acquisition and construction.

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Customer Complaints

September 30, 1980 - Rhom and Haas - Port Richmond, PA - Methyl Formcel-20M gal. tankcar - Customer reports receipt at 100 ppm Sodium Formate - Maximum specification = 50 ppm. Original and Retain analysis confirm low sodium shipment at 2 ppm Na. Customer apparently used material. Complaint classed as non-legitimate.

Waivers and Delays

Delays - None this month

Waivers - TMP - Flaked product at 0.003 wt.% acid. Maximum Specification = 0.002%. Bagged shipments were made in stated amounts to the following customers:

10-2-80	Celanese - Gen. Bonded Whse. - Charlotte, N. C. - 850 x 50
10-2-80	Celanese - Newark, N.J. - 3200 x 50
10-3-80	Celanese - Newark, N.J. - 1400 x 50
10-7-80	Mobay Chem., - Natrium, W.V. - 800 x 50
10-8-80	Owens Corning - Newark, OH - 800 x 50
10-9-80	Celanese - Rexdale, Ont. - 3200 x 50
10-9-80	Celanese - Newark, N.J. - 3200 x 50

10-3-80 - n-Butyl Formcel - 4900 gal. TT - Celanese Polymer Co., Louisville, KY.
- Color at 15 vs. Maximum Spec. of 10.
10-13-80 - Methyl Alcohol - 1500 gal. TT - Celanese Tech Center, Corpus Christi, TX - H₂O at 0.03 wt.% vs. Maximum Spec. of 0.01.
10-23-80 - n-Butyl Formcel - 4900 gal. TT - Celanese Polymer Co., Louisville, KY.
- H₂O at 7.6 wt.% vs. Maximum Spec. of 7.5%.

ANALYTICAL STATISTICS

Sample Count

	<u>October</u>	<u>October Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section	3748	45586	50096
Instrument Section	<u>1929</u>	<u>22971</u>	<u>26906</u>
TOTAL	5677	68557	77002

Technician Overtime

Chemical Section	30	627	813
Instrument Section	<u>35</u>	<u>325</u>	<u>406</u>
TOTAL	65	952	1219

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Sample Shipping and Quality Assurance Statistics

Statistics For October 1980

<u>Sample Shipping</u>	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Domestic	63	655	684
Export	2	27	26
Plastics	4	77	18
Other	11	77	69
Total	80	836	797

Quality Assurance Statistics

	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Shipments (NO.of Orders)	598	7288	9169
Delayed	0	3	31
Waviers <u>Internal</u>	8	15	9
External	2	23	38
Complaints (Total)	1	29	44
Complaints (Legitimate)	0	13	17
Complaints (Non-Legitimate)	1	16	27
Complaints - Outstanding (Unresolved)	0	0	0

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II. SPECIAL PROBLEMS GROUP

Process Technology B. C. Kerr

Solar Pond Disposal Study - Work has been started on ways in which high strength wastes stored in Chemcel solar evaporation ponds may be treated to facilitate disposal. Ponds 10 and 11E have been chosen as a representative source for samples and treatment studies.

The current solar pond water inventory is approximately 200 MM gallons. In addition, there is reason to believe that there will be a thick layer of highly contaminated sludge in the bottom of each pond. With such large quantities of material requiring treatment, even a few cents per gallon will have a significant effect on Plant costs.

Work will continue on this project.

Technical Service Call to Badische at Freeport - At the request of Dallas Quality Assurance, I accompanied Marvin Harris on a technical service call to Badische at Freeport on October 23rd. The purpose of the trip was to provide Badische personnel with information on the storage and handling of 50% HCHO solutions. In addition, data on the Celanese HCHO stabilizer system was presented. Badische's neo-pentyl glycol process appears to require a non-stabilized HCHO feed. A further requirement is a maximum chloride content of 1 ppm.

Methanol Oxidation, Formaldehyde Blending T. A. Jasek

G. C./MO Reactor - No progress due to priorities.

MO Silver Catalyst Modifiers - Recent studies at the Tech Center indicate that bismuth (Bi) added to silver increases efficiency of the silver catalyst needles. In support of the Tech Center project, the Laboratory has set up an electrodeposition cell to grow silver catalyst needles. Pure silver needles from the cell have been sent to the Tech Center to establish base case data. Silver needles containing Bi will be produced for the Tech Center following the establishment of base case data.

MO Unit Support - Laboratory analytical AMR's were conducted at MO-III for start up of "A" train. Analytical AMR's were also conducted at MO-II following instrument work. The table below illustrates the data obtained.

<u>Date</u>	<u>MO Unit/Train</u>	<u>AMR</u>	
		<u>Indicated</u>	<u>Analytical</u>
10-13-80	II/A	0.86-0.88	1.32
10-15-80	III/A	0.85-0.87	0.87
10-22-80	II	1.14-1.16	1.28
10-24-80	II	1.03-1.06	1.01

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New HCHO Stabilizer (Day) - Four samples of formaldehyde containing the new stabilizer combination of Metalose/Polyvinyl alcohol were shipped to Plastics Manufacturing Co. in Dallas for customer evaluation. The Bishop Laboratory is prepared to send more samples as requests are received.

Utilities, BOD
S. G. Smith, P. G. Welling, G. R. Stasney

Anaerobic Aerobic Pilot Studies

Operations Manual (Smith) - Assistance has been provided to Process Engineering by reviewing the final draft of the BOD II Operations Manual and providing information when necessary.

Effluent Component Analysis (Smith) - No progress due to priorities.

Start-Up Study/Upflow Velocity Study (Welling, Stasney) - Two anaerobic-aerobic units are being run to further study the switch from the present aerobic system to the future anaerobic-aerobic system. Also being investigated are the effects of upflow velocity on start-up and solids accumulation in the filter bed. Filter #2 is being operated at an upflow velocity of 1-2 ft/hr (to maintain the 10:1 recycle to feed ratio), while filter #3 is being operated at an upflow velocity of 10 ft³/hr (to simulate the upflow expected in the commercial unit). The organic loading to both filters was raised to 0.35 lbs COD/ft²/day. The kinetic efficiency on the filters averaged 66% and 65% respectively. The percent anaerobic COD removal on the filters ranged from 85% to 94%. Recycle CODs on filter #2 ran 500 to 700 ppm averaging 564 ppm, while filter #3 had recycle CODs of 600 to 800 ppm with an average of 618 ppm. Aerobic outfall CODs rose to 260 ppm and 275 ppm during the first part of the month due to reduced sludge activity at low temperatures. With a temperature increase the outfall CODs after aerobic polishing were 80 ppm to 115 ppm.

A report detailing start-up work is being written.

Elimination, Reduction of Solar Pond Odor (Smith, Jasek)

The major sources of solar pond odor have been identified to be (1) a strong chemical odor originating from surface oil on several of the ponds, and (2) a septic odor originating from Pond 7B. The major sources of oil to the ponds were identified as the Oxo sump and the "gasoline" tank. A report discussing the solar pond oil and recommending evaluation of methods of oil elimination by Process Engineering has been issued (SGS-34-80). The source of septic odor in Pond 7B is the BOD sludge blowdown stream which is discharged into this pond. Once in the pond, the sludge turns anaerobic and begins to produce CH₄, CO₂, and H₂S. Possible solutions under consideration for elimination of anaerobic related odors in Pond 7B are: (1) pond aeration, (2)

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increase of pond pH, (3) modification of method or point of sludge addition, (4) elimination of the sludge itself or the bacterial activity of the sludge. Tests have shown that anaerobic activity will probably occur in any of the ponds provided that a high enough concentration of sludge is present. Current work concerns methods of elimination of bacterial activity by application of heat, commercial biocides, calcium hypochlorite, and hydrogen peroxide to the sludge blowdown. Elimination of sludge blowdown will probably involve a long-range investigation into methods of sludge disposal (provided sludge digestion in the anaerobic filter is not a viable solution).

In addition, the Laboratory is evaluating several masking and oxidizing agents as short term solutions to solar pond odor. A commercially available citrus masking agent as well as a current Plant stream (which for the most part is burned) were evaluated as masks for the odor. The citrus odor, while more pleasant, is more expensive and more subject to wave action. The Plant stream, strip butanol highboilers (mainly butyl butyrate), is inexpensive and the amount required is less, but is an acceptable masking agent over a very narrow concentration range. At too high a concentration, it becomes more unacceptable than the solar pond odor itself.

Solar Pond Treatability (Welling, Stasney) - The extent to which solar pond "organics" can be removed by aerobic biological oxidation is under investigation. Three eight-liter aeration tanks are being used in the study. Tank #4 (the control tank) is being fed only equalization basin material. Tank #5 is being fed solar pond material diluted with equalization basin. Tank #6 is being fed pond material diluted with equalization basin and river water. During the first part of the month the pond material was diluted 175 parts equalization or equalization + water to 1 part pond material. The outfall CODs at this dilution were 200 ppm for #4 (95.7% COD removal, 200-220 ppm (95.5% COD removed) for #5, and about 280 ppm (93.7% COD removal) for #6. The concentration of pond material was increased during the month (dilution 159:1). The outfall CODs increased to 500 ppm (90.8% COD removal) for #5 and 440 ppm (91.9% removal) for #6 with a control COD of 240 ppm (95.2% removal). These low COD removals were probably at least partially caused by an upset unrelated to the increase in solar pond concentration. In an effort to reduce the outfall COD, the detention time was increased from 6 days to 7 days, but no reduction was seen. A partial replacement of bacteria in the Laboratory units with bacteria from BOD I unit resulted in a reduction of outfall CODs to 188 ppm (96.6% COD removal) in #5, 190-200 ppm (96.3% removal) for #6 with a control COD of 120 ppm (97.4% removal). These CODs were obtained with a detention time of 6 days.

Methanol Synthesis
J. C. Bustabad

MS-II Unit Support - Laboratory support continued for the MS turnaround. A report will be issued in November detailing the support provided.

Sodium from the boiler feed water was identified as the major component of solid deposits found in HE-1985 during the turnaround.

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A leak in a feed line of the 5 MM Girdler reformer resulted in the reformer being brought down for maintenance. Testing showed nickel tetracarbonyl levels to be within OSHA regulations.

Levels of iron in process condensate from the DPG reformed gas have been determined to be lower than they were prior to the turnaround. These lower iron levels are a result of some CS piping being changed to SS during the turnaround. This piping change was necessitated because of previous problems with the system (compressors, etc.) being fouled.

MEA and CO₂ levels for the 5 MM Girdler reformer and the hydrogen shift MEA systems have been determined for use as partial base data when Dow's MEA corrosion inhibitor is changed in early November.

Approximate levels of major components of the feed to the MS II flare have been determined. This work is the beginning of a study to determine fugitive leaks from the MS unit to the flare.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - Oxo Reactor accountability has averaged 87% for October, however, the BuH concentration is suspect (41.9%). Work continues.

During the September shutdown the Tischenko Ester concentration built to about 8% in the static reactor solution. Heavy stripping since start up has reduced this to 5-6%.

Work has been initiated to confirm Ihara Chemical as an alternate supplier of TPP.

Rhodium Accountability - Run VII rhodium accountability remained high for October (approximately 112%). Reactor samples taken before start up in early October indicate a more believable 95% accountability. This confirms the problem with the level reading when the reactor is in a dynamic mode.

Rhodium lost out T-210 residue for Run VII is now about 0.8 pounds.

Improved Rhodium Recovery - Extractions of the catalyst for the third phase of the program have been completed. Also, most of the activity tests have been completed. Work continues.

Oxo Unit Support - The Tischenko Esters synthesized in September have been purified. A letter will be issued when analysis is complete.

Analysis of initial samples from the test coalescer showed high BuH levels. Condensation in the coalescer is suspected. More insulation will be added. Work continues.

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High propylene in the recycle was resulting in low efficiencies in mid-October. A high H₂ to CO ratio (1.2) of the syn gas was believed to be the major problems. Lowering the ratio to 1.1 helped markedly. However, operational problems have caused the ratio to vary somewhat. Work continues to define other sources of efficiency loss. Addition of Rh has helped somewhat.

Problems have also been encountered with the unit sulfur analyzer. Apparent high (10+ ppm) carbonyl sulfide (COS) in the feed propylene caused some concern. Communications with CCPC revealed that they run total sulfur on a daily basis; and, they had not seen sulfur (<1.0 ppm) in propylene leaving their facility. The unit feed was sampled and sent to CCCTC for confirmation of CCPC's numbers. Less than 1 ppm total sulfur was seen. The problem appears to be with the column system in the analyzer. Suggestions to alleviate the problem have been given to instrument maintenance. Work continues.

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

The hearing conservation and noise survey continues. (CEG, KVP) (CEG-32-80)

A report recommending that long-term Drager CO 50/a-1 tubes be used to obtain 8 hour time weighted averages (8 hr TWA) for carbon monoxide was issued. (CEG-31-80).

The ABI personnel are adequately protected from respirable silica dust while working in the sandblasting yard. (CEG-34-80)

Personal exposure samples for 8 hr TWA-carbon monoxide are being obtained using the long-term Drager CO tubes.

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

Unit Support (Huelle) - None provided.

Improved Aldol Condensation and AcH Recovery (Buras) - No progress due to priorities.

Acetates, Paraform, Formcel[®] Products
R. L. Day, D. R. Akin

Acetates Unit Support (Akin) - Analytical support was provided when the MSA catalyst in the Propyl Acetate unit lost activity. The unit was shutdown for one day. After start up the problem did not reoccur. The reason for lost activity was not determined.

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Corrosion Inhibitor (Day) - Copper (II) phosphate corrosion inhibitor was added once again to the acetate reactor kettle 1408. The corrosion coupons will be checked during the next shutdown. An evaluation of the corrosion inhibitor in this system will be made at that time.

Paraform Lumps (Day) - The Paraform Unit shutdown recently to fix the chilled water nozzles on the flaker drums of the West train and to adjust a blade holder on a flaker drum on the East train. Hopefully, these corrections will eliminate the lump problem. The Laboratory will closely monitor flake product after start up to ensure that characteristic flake is once again being produced.

Diacetone Alcohol
B. K. Huelle

Unit Support (Huelle) - None provided.

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

Recovery of PE from AML (Buras) - Evaluation of alternative extraction schemes continues. The initial objective is to nonselectively extract all organics from AML. This would improve recovery of sodium formate by VCI and hopefully eliminate the centrate waste stream. The organics removed from AML could be burned or PE could be selectively recovered for recycle to the PE unit.

Extraction shake out studies were conducted to screen a range of solvents for removal of organics from AML and concentrated AML. The best solvents appear to be t-BuOH and n-BuH. Continued extraction studies using the solvents will be conducted to determine the removal efficiency.

Development of a System to Monitor PE Reaction Conversion (Huelle) - PE batch reactions at six different caustic to acetaldehyde ratios were repeated. An efficiency curve comparable to the curve obtained by the Technical Center was observed. The L.C. work continues; however, several mechanical failures have hampered progress. L.C.'s of the reaction mixtures have a common peak which diminishes with increasing mole ratio. This is a repetition of last month's results. At 1.15 mole ratio, no peak appears at the 220 nm U.V. range.

PE Formal Hydrolysis (Akin) - Work was continued using ion exchange resins as co-catalysts in conjunction with continuously fed strong acid. Amberlyst 15 exposure time to date is 115 days. The conversion rate of linear formals by this resin has dropped during the latter part of October. The strong acid feed was switched from formic to sulphuric in an attempt to reactivate the resin. This technique proved ineffective. Another approach to be tried in November is the flushing of the bed with a 10% solution of sulphuric acid.

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The studies being conducted on Amberlyst XN-1010, another Rohm and Haas ion exchange resin, were discontinued. This resins activity proved to be comparable to the Amberlyst 15 activity, but at a much higher cost.

Variable studies of the effects of sodium concentration and acid concentration (with a constant residence time) on conversion of linear formals were conducted on both resins.

AML Carbon Usage (Akin) - Studies were completed on the removal of color from AML by carbon bed treatment. This study will be documented in November.

Trimethylolpropane
R. L. Day, B. K. Huelle

Unit Support (Huelle) - On the 7th, 8th and 9th of October samples were taken of 7470 OH vacuum jet exhaust, 747 OH, and 747 residue. The NaFo levels are still high in comparison to past data for the 747 residue. Efficiency losses through 747 jet exhaust are approximately .3% for the 3 day period. Two years ago the exhaust accounted for .06% efficiency loss.

On the 20th of October, Raymond Yu, Process Engineering, and I sampled 747 OH, 205 OH, and 205 sidestream jet exhausts. The combined efficiency loss (almost all from 747 Oh exhaust) was approximately .6%. The G.C. analysis of 747 exhaust organics proved to 95% one compound. The retention time of this compound is also seen as a very small component in 202 OH samples. A TMP solution will be heated and the overhead collection analyzed by G. C. to provide evidence that the organics out 747 OH exhaust are indeed decomposition products of TMP.

Acid Problem (Day) - The resin bed study to isolate the acids in TMP 205 SS was completed this month. The elution from the resin bed was concentrated by use of a rotavap, then distilled on a spinning band distillation column. Two of the fractions and the residue were sent to CCCTC for identification by G.C./Mass Spec. The major component of the two fractions was butyric acid. Work is currently in progress to determine the source of the butyric acid.

Special Problems Group - General

1. Jim Presley attended ExpoChem '80 in Houston.
2. Tom Jasek attended the DuPont "Strategy of Experimentation" seminar in Houston.

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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
Anaerobic Testing of Chemical Wastes	Smith Welling	1	Support Unit Expansion	Continuing
Solar Pond BOD Treatability Study	Welling	1	Environmental Mandatory	Continuing
Elimination/Reduction of Solar Pond Odor	Smith Jasek	1	Environmental Mandatory	December 1, 1980
Oxo Reactor Accountability/Stability	Presley ↓	1	Improve Reactor Control	Continuing
Rhodium Accounting		1	MOB, Catalyst Savings	Continuing
Improved Rhodium Recovery		1	Raw Materials Saving	December, 1980
Develop a way to Monitor Conversion in the PE Reaction System	Huelle	1	MOB (Quality) and Raw Material	January, 1981
TMP Extractor Optimization	Day ↓	2	Raw Material Savings	Undetermined
Ester Reactor Corrosion Control		2	Extended Equipment Life	December, 1980

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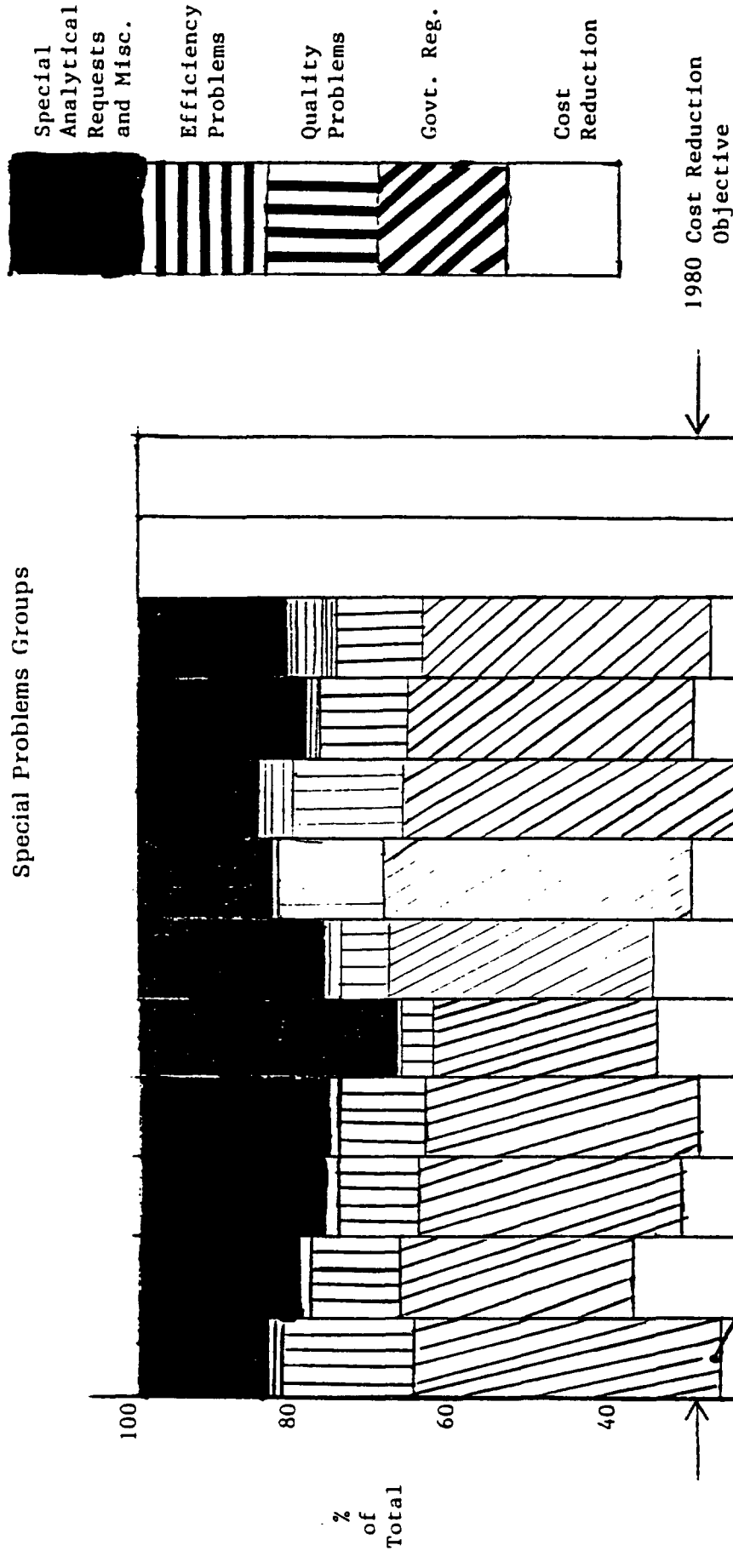
Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
New Formaldehyde Stabilizer System	Day	1	MOB	December, 1980
Avoid Putting VCI Concentrate in Solar Ponds	Buras ↓	1	MOB	Undetermined
Develop a Low Energy-High Efficiency ACH-Aldol Separation Process for the BG Unit		2	Raw Materials and Steam Savings	March, 1981
OSHA Monitoring	Gary	1	Insure Employee Health and Safety OSHA Compliance	Continuing
Develop an Improved Hydrolysis System for PE Linear Formal	Akin	1	Steam, Acid and Capital Savings	February, 1981

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Effort Analysis - Laboratory 1980

Special Problems Groups



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