

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: G. D. Boyd - 31

GDB-57-80

July 8, 1980

Laboratory Department Summary

June, 1980

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CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

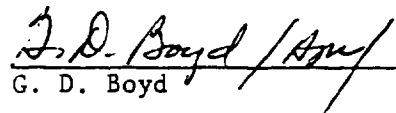
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Laboratory Department Summary
June, 1980

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 GROUPCompetitive Evaluations

None this month

Methanol Synthesis (MS)

Samples of a trailer of MeOH shipped from the San Pedro Terminal to J. T. Baker Chemical Company were sent to the Bishop Lab for analysis. Our analysis confirmed their complaint of acetone contamination at approximately 160 ppm. Retain samples from the trailer to J. T. Baker and another methanol shipment the same day also showed acetone indicating a San Pedro storage tank contamination. Jim Sanborn reports the material has been disposed of.

Oxo Butyraldehyde

BuH - The Analytical Group provided support for a study to increase Oxo unit efficiencies by reducing the residue drawoff on T-210. This study was interrupted by the Oxo shutdown. Support will be available, if needed, when Oxo resumes production.

Triphenylphosphine (TPP) - Nine preshipment samples of TPP were received from M & T for analyses. All the samples meet our purchase specifications and check very closely with M & T's analytical results.

Analytical Group General

C. E. Bull, Instrument Laboratory Supervisor, and J. H. Harris, Wet Chemical Section Supervisor, each attended a one day Batten, Batten, Hudson and Swab, Inc. seminars on June 9, 1980 and June 23, 1980, respectively. Both seminars dealt with improving supervisory skills.

Customer Complaints

June 25, 1980 - San Pedro Terminal, San Pedro, California - n-Butyl Alcohol - 20,000 gal. - shipped June 9, 1980. Material arrived at San Pedro with a natural color of 20 Pt-Co units. Retain analysis indicated a color of 6 (max. spec. 10). Samples of the tank car to be returned to Bishop. An attempt will be made to determine the cause of the high color.

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Analytical StatisticsSample Count

	<u>June</u>	<u>June Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section	4772	29231	29825
Instrument Section	<u>2217</u>	<u>15036</u>	<u>16461</u>
Total	6989	44267	46286

Technician Overtime

Chemical Section	0	521	500
Instrument Section	<u>35</u>	<u>215</u>	<u>307</u>
Total	35	736	807

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Sample Shipping and Quality Assurance Statistics

Statistics For June, 1980

<u>Sample Shipping</u>	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Domestic	75	406	414
Export	3	15	15
Plastics	9	49	6
Other	8	44	59
Total	95	574	494

Quality Assurance Statistics

	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Shipments (NO.of Orders)	642	5016	5612
Delayed	0	3	7
Waviers <u>Internal</u>	3	6	7
<u>External</u>	0	4	14
Complaints (Total)	1	20	32
Complaints (Legitimate)	1	6	10
Complaints (Non-Legitimate)	0	13	15
Complaints - Outstanding (Unresolved)	0	0	7

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II. SPECIAL PROBLEMS GROUP

Methanol Oxidation, Formaldehyde Blending T. A. Jasek, R.L. Day

Studies were begun in preparation for a plant test to add polyvinylalcohol/metolose stabilizer to 50% HCHO shipments. Mixing techniques are being developed. Work is also in progress to develop an analytical method for PVA, and to determine if the PVA interferes with our present analytical method for metolose. Storage stability tests will be set up during the plant test. (Day)

MO Silver Catalyst Screening - No new shipments were received.

Crude Methanol Plant Trial - The plant trial ended on June 23, 1980. Analysis of the metal contaminants around the vaporizer have been completed. Sampling is scheduled to determine the level of metal contaminants around the vaporizer during a non-crude methanol mode of operation.

Laboratory Sinter Test vs. Catalyst Performance Correlation Study. - Weekly sampling of the reactor containing Matthey-Bishop silver catalyst continues. As reported last month, this catalyst charge failed the Laboratory Sinter Test. It is being evaluated to determine if the Laboratory Sinter Test provides useful information on catalyst performance.

G. C. MO Reactor - An MO reactor is being installed in a G. C. If operable, the reactor will be used in various studies, such as catalyst evaluations, efficiency improvements, and effects on catalyst life.

MOB - The Laboratory checked the AMR at MO-IV "A" train after the train experienced a decrease in catalyst bed temperatures with no change in AMR. The unit was at low rates at that time. The averaged lab AMR was 0.93 while the indicated Control Room AMR was 1.05 - 1.08.

The Laboratory conducted analytical AMR's at MO-III "A" train after its annual turnaround. The averaged analytical AMR was 1.15 while the indicated Control Room AMR was 0.86 - .87. The problem appeared to be due to the very low flow rates at the time of start-up. Recheck of the AMR's on the following day showed very close agreement. The indicated Control Room AMR was 0.90 - .92 while the averaged analytical AMR was 0.94.

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

Anaerobic-Aerobic Pilot Studies

Anaerobic-Aerobic Pilot #1 - A study to determine the effects of NaOH addition to the anaerobic recycle was begun. Sodium (Na^+) is added to the recycle to help maintain proper filter pH of 6.6 - 7.6. Feed to the filter contains equalization basin, oxo sump, and nutrients N and P (NH_4OH and H_3PO_4). During the study, organic loading to the filter has varied between 0.10 and 0.40. Caustic has been added in-line to the recycle in amounts equivalent to adding 220-600 ppm Na in the feed; filter pH has remained between 7.2 - 7.6 during this time. Filter efficiency has been 70-80%. On one occasion, filter pH rose to 8.9 when the feed was discontinued due to pump problems while NaOH addition was maintained. Following restoration of proper pH by acid (HCl) addition, feed was reintroduced to the filter. Filter performance was unaffected by this relatively short (several hours) exposure to high pH.

The Caustic Addition Study is being continued at higher filter organic loadings. Also, the pH of the recycle at the point of addition is to be investigated.

Aerobic section performance has remained good during June. Outfall $\text{COD} \leq 200$ ppm at a 3 day feed detention time.

Anaerobic Aerobic Bench Scale Unit #2 - The Laboratory simulation of the anaerobic-aerobic startup has been completed. The simulation entailed the complete switch over of our present aerobic system to the 2-stage BOD II System. Oxo and polyol sumps were also included in the feed at organic loadings up to 0.45 lb. $\text{COD}/\text{ft}^3/\text{day}$ to the anaerobic filter and at aerobic detention times down to 3 days (design projections for 1981). Outfall COD's remained ≤ 230 ppm during the entire simulation. A report detailing this work is to be written.

Ammonia Nutrient Addition - A report detailing effects of NH_3 addition to the recycle has been submitted.

Evaluation of TOD Analyzer - Development of a TOD/COD correlation on anaerobic feed and recycle samples continues. A report is to be written.

Anaerobic Filter Upflow Velocity Study - Preparations are underway to begin a study to investigate effects of upflow velocity on filter startup and gradual solids accumulation in the filter bed.

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Operations Manual - Assistance in writing the BOD II Operations Manual is being provided.

BOD Unit Support - The unit method for nitrate nitrogen is being checked by three methods: (1) HACH kit (2) Specific ion electrode (3) Chromatropic acid method. No conclusive results to report yet.

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

Measurement of the Task Oriented and STEL/30 min. formaldehyde exposures requested by the Formaldehyde Task Force have been completed and reported. (CEG-20-80, KVP-01-80). (Gary, Parker)

Measurement of the formaldehyde exposures of several job classifications needed to complete the overall plant survey continues. (Parker)

The initial computer input data forms for the industrial hygiene-medical surveillance module have been filled out for key punching. (Gary)

The long term Draeger CO 50/a-L tubes are being evaluated for obtaining the 8 hr. TWA personal exposure to carbon monoxide. Initial results are encouraging. Evaluation should be completed in July.

Oxo, Butanol
J. D. Presley, N. Elizalde

Reactor Stability/Accountability - Composition of the Oxo reactor was fairly stable during June. TPP was maintained at 27-30 wt.%. Overall accountability is staying at 70%.

A severe upset in the Girdler Unit Amine System caused contamination and consequently, a shutdown of the Oxo catalyst solution. Monoethanol amine was carried into the Oxo reactor via the syn gas system. The contamination occurred just several hours before the scheduled Oxo turnaround.

Activity testing of the catalyst solution (using the stirred autoclave) showed a decline to about 60% of the pre-contamination catalyst. Laboratory attempts to reactivate the catalyst, using the hydrocarbon extraction (propylene), have shown some success. The catalyst activity after extraction is about 85% of the original. However, the extraction is very successful in reducing the foaming of the catalyst. Samples of catalyst before and after contamination have been sent to CCCTC for a total nitrogen analysis.

G. C. analysis shows that roughly 95% of the BuH originally present in the catalyst has gone to high boilers. Lab study is underway to see if additional BuH will be consumed in extracted and unextracted catalyst. Work continues.

An alternate G.C. method for TPP analysis has been developed. Initial results are promising.

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Rhodium Accountability - In June, rhodium accountability was erratic, ranging from 92% - 111%. As in May, this is believed due to the reactor foaming problems. A sample and accurate level reading taken from the catalyst dump vessel during the June turnaround yielded 95.4% accountability.

Total rhodium losses out T-210 residue are now at 16.92 pounds.

Assays of two drums of used Oxo Catalyst sent to Engelhard have been completed. A letter has been published JDP-65-80.

Improved Rhodium Recovery - Thirteen solvents have been tested in the hydrocarbon extraction study on Oxo catalyst. All solvents have shown a tendency toward one best catalyst to solvent ratio. Three of the solvents (pentane, decane, and propylene) have been chosen for the second phase of the study. This will include testing the effects of temperature pressure and BuH concentration.

Oxo Unit Support - Process Engineering has suggested the possibility of installing a coalescer downstream of the Oxo reactor knock-out pot to reduce TPP (and possibly rhodium) losses. A test coalescer designed by Process Engineering was brought to the lab for packing with glass wool (density is $\sim 4.4 \text{ g/cm}^3$). The test unit, when installed, should provide data to determine the need for a coalescer. The lab will provide technical and analytical assistance.

An analytical method for determining BuH in Oxo compressor oil has been developed. Work to date shows BuH level in the compressor oil to be in the range of 0.05 to 0.07 wt.%. Another contaminant has also been found in the compressor oil. At present it is believed to be propylene and is about 0.2 wt.%. Work continues.

Special Analytical Assignments
F. R. Dow

PE Monitor - Separation of Di-PE from Tech-PE has been found to be most effective by a series of water crystallizations. Tri-PE is removed by crystallization in an excess of water, then the water concentration is decreased incrementally (usually 100 ml) and the crystals from each water concentration are removed and analyzed. This procedure is used twice, once on the original Tech-PE and again when the crystals containing the highest concentrations of Di-PE are composited for the second crystallation run. To date the highest Di-PE concentration obtained by this procedure has been 89-90%.

The high Di-PE concentration sample will be used for sample trapping with the L.C.

Methanol Synthesis
J. E. Russell

MS-II Unit Support - The Laboratory analyzed the Selas purge gas on June 2 for $\text{Ni}(\text{CO})_4$ due to a maintenance shutdown. On June 21, the 5 MM and 2 MM

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Girdlers here analyzed for Ni (CO)₄ in the purge gas due to maintenance requirements during the Oxo shutdown. Letters were published for both events.

MS-II Unit Support - Caking of an unknown material on an interior wall of the Selas furnace prompted an investigation of the identity of a tar-looking substance in the fuel gas line to the Selas burners. The remaining ingredient after ashing and fusing the material was silica. The spun-glass filters in the fuel gas coalescer are suspected as the source of silica. The filters were subsequently changed.

The extent of caking will be checked periodically to determine if the problem has been solved.

Ms-II Unit Support - The MEA system for the 2 MM - 5 MM syn gas unit was contaminated with a foaming agent on June 20. Subsequent analysis showed that the activated charcoal used in the PE unit will restore the MEA to fresh MEA condition. The quantity of charcoal required will be less than 20 ft³. The exhaustion point has not been determined yet, so a smaller quantity may suffice. Analysis shows that the approximately 5500 gallons contains 10.0% MEA by weight. This equates to a value of ~\$2700.

Recovery of Methanol From Purge Gas - The Laboratory performed some preliminary work in an effort to recover the MeOH from the high pressure separator purge gas. Initial results yielded ~95% removal using a 1,3 Butylene Glycol scrub of the purge gas. Operating parameters consisted of 100-155 psig purge gas, ~25°C, and space velocity of 2.4 hr⁻¹. Further work will be considered as time permits.

Butraldehyde Hydrogenation Improvement - Two samples of Raney Nickel were modified in an effort to improve their resistance to CO and TPP poisoning. Due to priorities, these samples have not been tested yet. Further work will proceed as time permits.

1,3-Butylene Glycol
R. L. Day, P. J. Buras

Unit Support - None provided. The unit was not running. (Day)

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.

Investigation of catalysis by macroreticular resins continues. The bench scale reactor was rebuilt in order to accurately control the reactor temperature, pressure, and residence time. Using a strong base resin, initial conversion was ~20%. Conversion decreased rapidly to ~2% after ~20 bed volumes of AcH was passed through the bed. Different reactor conditions

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and/or catalysts will be evaluated in hopes of extending catalyst life. (Buras)

DiAcetone Alcohol
R. L. Day

Unit Support - None provided. Unit has been down.

Acetates
R. L. Day

A transesterification process for the acetate unit was considered, whereby butyl acetate is made from propyl acetate. However, using azeotropic data, this process would not be feasible in our current system. No further work is planned.

Paraform, Formcel Products
R. L. Day

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

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

Recovery of PE from VCI Centrate - A one quart sample of centrate was received from VCI in Portsmouth, Virginia. The sample was generated from "modified AML" (AML extracted with n-Hexanol) samples sent from Bishop and was to be representative of the centrate to be returned to Celanese in the proposed process to recover PE from VCI Centrate. Analysis of the centrate samples showed 47% Sodium Formate (NaFo), 27% water and 26% PE-type organics (17% PE) indicating ~54% NaFo recovery efficiency. This compares to current VCI efficiencies of 65±5% and current centrate of 30-35% NaFo, 15-20% water, and 45-55% PE-type organics (25-30% PE).

Stability studies of the centrate sample will be initiated upon completion of physical property data gathering. (Harvey)

Shake-out studies on the extraction of current VCI centrate and PE WIP mixtures with n-Hexanol will begin in July. (Harvey)

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

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PE Formal Hydrolysis - Studies were continued using ion exchange resins as co-catalysts with a continuously fed strong acid. Amberlyst 15 exposure to reaction conditions was continued. Total exposure time to date is 41 days. This resin still appears to have good activity, and further activity-life tests will continue. Variable studies of the effects of sodium concentration, acid concentration, and residence time on conversion are underway on Amberlyst 15.

Another reaction system was set up to screen other resins recommended by Rohm & Haas. These ion exchange resins are Amberlite JR-118(H) and Amberlyst XN-1010. (Akin)

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). We are attempting to determine if unit operating data (e.g., carbon bed life) or analytical procedures (e.g., GC, sulfuric acid color) can be used to select lots for shipment to Dow. Work continues. (Buras)

Trimethylolpropane

R. L. Day

Work is in progress to improve the operation of the T-312 extractor in the TMP unit. Lowering the temperature of the feed to 312 may decrease the ethyl acetate losses out the base. Methods were developed to analyze for each component in the overhead and residue streams. A plant trial will ensue after the base case data is taken.

General

1. Dan Akin reported to work June 2 as a chemist (special problems). Dan is from Southwest Texas State at San Marcos.
2. Belinda Huelle reported to work on June 16 as a chemist (special problems). Belinda is from the University of Texas at San Antonio.
3. Pam Welling reported to work on June 30 as a chemist (special problems). Pam is from Texas Tech. at Lubbock.

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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
Laboratory Sinter Test vs. Catalyst Performance Correlation Study		1	Validity of Laboratory Sinter Test in Relation to Catalyst Performance	Undetermined
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	1	Support Unit Expansion	Continuing
Formic Acid Removal From Formaldehyde	Russell	2	Cost Reduction	August, 1980
Butyraldehyde Hydrogenation Improvement	↓	1	Cost Reduction	September, 1980
Recovery of Methanol From Purge Gas		1	Incremental MeOH Expansion	July, 1980
Identify and Develop an Analytical Method for Nitrogen Containing Compounds in HClO	Dow	2	MOB	August, 1980

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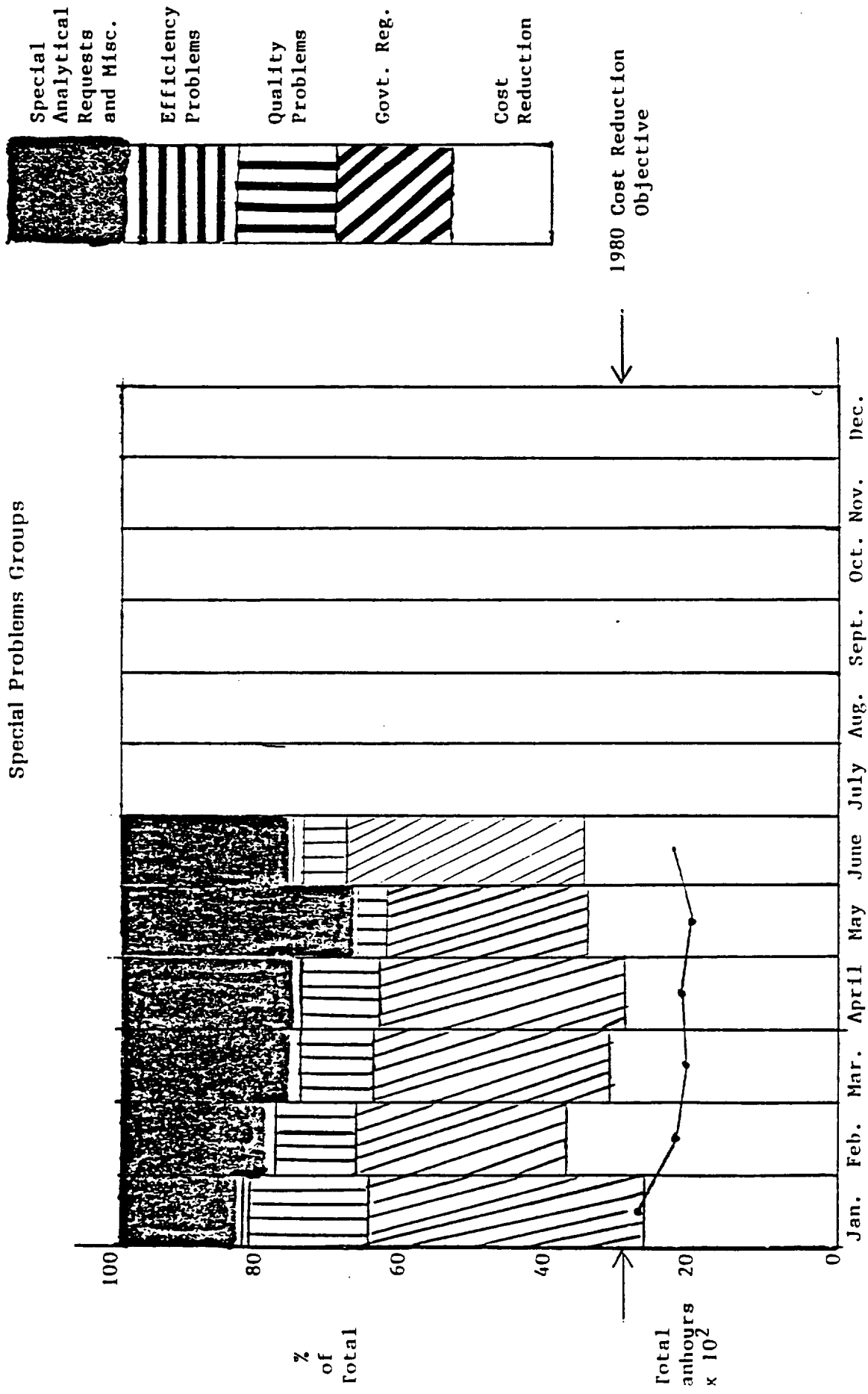
Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Evaluate New L.C., Train Special Problems Chemists in its Use	Dow	2	MOB	August, 1980
Use L.C. to Check the Composition of our PE Standards and Upgrade our PE Analysis, if Required		1	MOB	July, 1980
Develop a Low Energy-High Efficiency AcH-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 Buras	1	Raw Material Savings	Undetermined
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 Savings	November, 1980

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Effort Analysis - Laboratory 1980

Special Problems Groups



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