



CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: R. R. Graham

From: G. D. Boyd- 01

GDB-71-80

September 8, 1980

Laboratory Department Summary

August, 1980

Distribution

Chemical

J. J. Fritsch, Jr.
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
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. W. Dunn, Clear Lake
T. H. Golson, Dallas
A. J. Branecky, Edmonton
W. H. Tuke, Pampa
F. B. McAndres, Summit
J. Zitomer, Summit

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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

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I. ANALYTICAL GROUP

Competitive Evaluations

The following competitive Products were received during August; Union Carbide - Normal Butyl alcohol and Iso Butyraldehyde, Texas Eastman - Normal Butyraldehyde and Iso Butyraldehyde, Georgia Pacific and Air Products - Methyl alcohol and Hercules Mono and Tech grade Pentaerythritol. Competitive product reports will be issued as analyses are completed.

Methanol Oxidation (MO)

The MO Unit requested and was provided analytical support to determine the cause of high iron levels in the MO-IV product. The cause was traced to higher-than-normal levels of iron in the methanol feed to MO-IV.

Metalose analyses were provided for the Special Problems Group "Formaldehyde Stabilizer Study." The purpose of the analyses was to determine if polyvinyl alcohol, in the new polyvinyl alcohol/metalose stabilizer system, interferes with the present method of Metalose analysis.

Oxo-Butyraldehyde (BuH)

Propylene-Special analytical coverage was provided for the Oxo Unit startup. The Propylene pipeline was again placed in service. Two samples of the pipeline propylene were received and analyzed by the Instrument Lab. Both samples met our purchase specifications for propylene.

Polyol

Trimethylolpropane (TMP) - Extensive analyses and overtime were provided this month because of TMP acid problems. The acid ranged from 0.001 to 0.007 (spec. 0.002 wt.% max.). Samples of the Butyraldehyde feed and the Formaldehyde feed were monitored for high acid levels. No specific cause of the problem has been established. Work continues.

Pentaerythritol (PE) - Hot and cold Sulfuric color determinations were made on the PE cake in support of the Special Problems project to develop a system to monitor PE reaction conversion.

Safety, Health and Environment (S.H.E.)

Numerous samples were analyzed for the new S.H.E. group. The work included samples of San Fernando Creek, standing water around the Plant and water from old cooling tower basins. Manpower was also furnished for obtaining samples.

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Special Analytical Services

PE-Monitor (F. R. Dow, B. Wise)- In the July Monthly Summary Di-PE content of the PE Monitor was reported as 8.2 wt.%. Since that report was issued, additional comparisons of Di-PE trapped by L.C. and the Canadian Di-PE standard has shown that the PE Monitor contains 8.7 rather than 8.2 wt.% Di-PE. This value is very near the 8.9 wt.% Di-PE that has been reported by Edmonton since the late 1960's.

Comparison of the Canadian standard with L.C. trapped Di-PE shows that the Canadian standard is 95.2 rather than 98.8 wt.% Di-PE.

This project will continue until the PE Monitor Di-PE value is firmly established.

PE-Competitive (F. R. Dow) - Samples of competitive PE from Hercules and Mobil are expected soon. The Di and Tri content of these samples are of special interest.

Quality of Plant Air (F. R. Dow)- Instrumentation to monitor Plant air quality in Boiler house #2 has been installed. Actual operation has not started due to delays caused by the hurricane and Plant problems.

Amines in Competitive MeOH (J. E. Russell)- The instrument used to analyze amines in MEOH is being tuned so that the Competitive MeOH study may be completed. The data on all the samples should be gathered by the first week in September.

Characterization of Solar Ponds (J. E. Russell)- The Lab is embarking on a study to characterize the contaminants in our Solar Ponds. Analytical methods and sampling techniques are being worked on at this time.

Projects Pending

1. Continue evaluation of Liquid Chromatograph and train Laboratory personnel in its use.
2. Use L.C. to check composition of PE standard and upgrade analysis if required.
3. Organize and present an Atomic Absorption course for Laboratory technicians.
4. Install purchased Capillary G.C. and scan Plant products for applicability.

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5. Complete Plant air quality study.
6. Amine level determination in Celanese and competitive methyl alcohol.
7. Characterization of Solar Pond effluent.

Analytical Group General

The temporary summer employees, B. Botard and L. McDonald terminated their employment to return to college. A utilization report will be issued.

Three professionals and three technicians from the Analytical Group attended a two day, in-house seminar on G.C. and L.C. maintenance and trouble shooting. The seminar was presented by Varian Instruments personnel and was very well received. A Varian presentation on general chromatography utilization, column selection and methodology is planned for the near future.

Standard Quality Notice forms (as opposed to Certificate of Analysis) have been prepared for all Plant products and are being used on a routine basis. A time savings for clerical staff is being realized.

The Laboratory was shutdown, coordinating with the Plant shutdown for Hurricane Allen, from August 8, 1980 PM to August 11, 1980 AM. Minor damage to hood exhaust vents on the roof has been repaired and all Laboratory systems are functioning.

The decision was made to implement the purchased solutions-project. Ready made reagent solutions for use in both the Laboratory and the Units will be purchased from the La Mar Ka Company of Baton Rouge, Louisiana. Quality, cost and service will be closely monitored and reported upon for a trial period.

J. E. Russell transferred to the Analytical Group on August 20, 1980 from Special Problems.

Customer Complaints

August 7, 1980 - Tenn. Eastman, Kingsport, Tenn. - Para Flake - 132 x 300 lb. cartons. - lumps in product plugged feeding lines. Customer used product. Valid complaint based on returned samples.

August 12, 1980 - 3M Co., Newark, N.J. - Para Flake, 8650 x 50 lb. bags - 1 to 1½ inch lumps would not dissolve in reaction. All material to be returned to Plant. Valid complaint.

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Waivers and Delays

Delays - None this month.

Waivers - August 4, 1980 - Molten TMP to Hatco Chemical - Heyden, N.J.-
20M gal. tankcar - Acid at 0.003 wt.% versus 0.002 max. specification.

August 15, 1980 - 50% HCHO to Reichold Chemical - Kansas City, Kansas-
2 x 20M gal. tankcars - Customer agreed to take off-test HCHO inventory.

August 19, 1980 - Molten TMP to Hatco Chemical - Heyden, N. J. -
20M gal. tankcar - Acid at 0.003 wt.% versus 0.002 max. specification.

ANALYTICAL STATISTICS

Sample Count

	<u>August</u>	<u>August Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section	3748	36871	39165
Instrument Section	<u>1688</u>	<u>18689</u>	<u>21082</u>
Total	5436	55560	60247

Technician Overtime

Chemical Section	7	546	573
Instrument Section	<u>8</u>	<u>247</u>	<u>377</u>
Total	15	793	950

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Sample Shipping and Quality Assurance Statistics

Statistics For August, 19 80

<u>Sample Shipping</u>	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Domestic	57	532	560
Export	4	20	23
Plastics	5	60	13
Other	5	55	64
Total	71	667	660

Quality Assurance Statistics

	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Shipments (NO.of Orders)	448	5991	7313
Delayed	0	3	27
Waviers <u>Internal</u>	0	6	7
<u>External</u>	3	7	23
Complaints (Total)	2	26	40
Complaints (Legitimate)	2	11	15
Complaints (Non-Legitimate)	0	15	24
Complaints - Outstanding (Unresolved)	0	0	1

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Methanol Oxidation, Formaldehyde Blending
T. A. Jasek

MO Silver Catalyst Screening (Jasek) - A test shipment (10 oz.) of silver catalyst needles were received from RefineMet International for evaluation. Laboratory sinter test was "good". A sample was sent to the Tech Center for evaluation in the micro unit. Results indicate that the catalyst is comparable to H & H in terms of efficiency. A letter recommending a Plant trial will be issued.

G.C./M.O. Reactor (Jasek) - No progress due to priorities.

Crude Methanol Plant Trial (Jasek) - Evaluation of the resin from the Crude Methanol to MO-IV Plant Trial was completed. Results, obtained at two different vertical locations in the resin bed, were inconsistent. Therefore, the level of exhaustion of the entire bed could not be determined. However, all samples showed at least 50% of the initial capacity for iron of the new resin.

HCHO Stabilizer Studies (Day) - Attempts to quick-freeze gallon samples of formaldehyde have been discontinued. Samples to customers will be shipped in quart bottles instead. Quart bottles are easier to freeze, easier to thaw, and in general, easier to handle than gallon jugs. Studies are underway to evaluate the stability of quick-frozen HCHO samples after being "thawed".

~~Work is in progress to determine the level of Polyvinyl alcohol (PVOH) in-~~
terference with Metalose analytical methods. Analysis of PVOH/Metalose mix-
tures in water show no interference, yet PVOH/metalose mixtures in formaldehyde
show some interference. Work continues.

Formaldehyde stability samples in a 40°C oven are being monitored daily to repeat/reproduce previous work.

MO Unit Support (Jasek, Presley, Russell) - The Laboratory conducted several analytical AMR's during the last month. These are illustrated in the Table below:

Date	MO Unit/Train	AMR	
		Analytical	Indicated
12 Aug 80	II	0.60	1.10
11 Aug 80	III/A	1.59	0.98-0.99
		1.24	0.90-0.91
		0.73-0.76	0.80-0.81
		0.60	0.88-0.92
13 Aug 80	IV/A	0.69	1.10-1.12
	IV/B	0.77	0.95-0.98
	IV/B	0.97	1.05-1.10
	III/B	0.94	0.78-0.83
18 Aug 80	IV/A	0.74	0.94-0.96
29 Aug 80	IV/A	0.84	1.06-1.10
	IV/B	0.88	1.00-1.06

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

Anaerobic-Aerobic Pilot Studies

Effects of NaOH Addition to Anaerobic Recycle(Smith) - A report detailing this work has been issued, SGS-33-80.

Operations Manual (Smith) - Assistance has been provided to Process Engineering concerning the process variable section of the BOD II Operations Manual. Input has also been provided concerning startup and shutdown procedures for the unit.

Effluent Component Analysis (Smith) - The study to analytically define major components in Bishop effluent streams and across the waste treatment unit has begun. Current work includes a preliminary look at polyol sump components. No conclusive results to report as yet.

Start Up Studies/Upflow Velocity Study (Smith, Welling, Stasney) - Two anaerobic-aerobic units are being run to further study the switch from the present aerobic waste treatment system to the future anaerobic-aerobic treatment system. Also being investigated are the effects of upflow velocity on filter startup and gradual solids accumulation in the filter bed. Filter #2 is being run at an upflow velocity of 1-2 ft/hr (to maintain the 10:1 recycle to feed ratio), while filter #3 is being held at an upflow velocity of 10 ft/hr (to simulate the upflow expected in the commercial unit). Organic loading to both filters has been gradually increased to 0.075 lbs/ft³/day. Both filters were shutdown on August 8th due to Hurricane Allen and started back up on August 12th at a slightly reduced organic loading. Filter #3 has shown gas-make since the 7th day of startup, working gradually up to a kinetic efficiency of 40% as of August 28th. Filter #2 has been plagued by mechanical difficulties (recycle pump and wet test meter malfunctions) so that gas-make was not detected until August 26th. As of August 28th the kinetic efficiency was 28%. #3 anaerobic outfalls ran from 500 to 700 ppm and COD removal efficiencies of 80 to 85% were recorded. #2 has had CODs of 200 to 800 ppm and removal efficiencies of 79 to 94%. After aerobic polishing, at a detention time of 3.5 days, the outfall CODs have run <160 on both filters.

Solar Pond Treatability Study (Welling, Stasney) - Work has begun to determine the extent to which Solar Pond "organics" can be reduced by biological oxidation.

~~----- Utilities Support (Presley, Bustabad, Frankenhauser) - August 28, 001 -----~~
outfall showed Total Suspended Solids (TSS) >60 mg/l. Analysis of the solids showed them to be primarily Calcium Carbonate (CaCO₃) and silica (SiO₂). A probable cause of recent high TSS was traced to a leak in the caustic tank used to raise the pH of the front ditch. ~~TSS immediately after the caustic~~
leak was 332 mg/l at a pH ~11. No solids were noted above the caustic leak. At pH 11 CaCO₃ originating from the lime pits would precipitate.

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Sulfuric acid is added downstream of the caustic to lower the pH of the front ditch when necessary. Acid was being added during the problem period to bring the pH of the ditch to ~7. This is not low enough pH, however, to put CaCO_3 back into solution. The caustic leak has since been repaired and TSS in 001 outfall is currently <60 mg/l.

In addition, 1980 001 outfall TSS values have been plotted vs. corresponding 101 outfall COD values. Results of this work shows that TSS is independent of COD i.e., not related to VSS carryover from the BOD unit clarifier.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability (Presley) - The Oxo reactor was started up with fresh catalyst in mid-August. During the catalyst formation the water concentration in the reactor was monitored as a precaution against rhodium precipitation. The water was stripped from the reactor each time it approached 0.5 wt.%. The catalyst charge took approximately 16 hours to prepare. The BuH and TPP concentrations were also monitored. The levels ranged 28-31 wt.% and 30-34 wt.%, respectively.

The Dowtherm used in the catalyst start-up mixture is now down to about 10 wt.%. Reactor composition since start-up has been erratic. This is believed due to control difficulties at the present low production rates.

Rhodium Accountability (Presley) - Rhodium accountability for this Run (Run VII) on August 19, 1980 was 99.4%. To date, rhodium losses in T-210 residue have not been calculated.

Improved Rhodium Recovery (Presley) - Lack of a "fresh" source of old catalyst has prompted preparation of a standard solution for the third phase of the extraction project, autoclave testing. The standard solution is a composite of the last few months of reactor samples from Run VI. Work continues.

Oxo Unit Support (Presley) - Analytical support was provided for the Oxo Unit start-up. Reactor composition was monitored closely during the catalyst formation for Run VII.

~~The "hard-soft" acid-base theory is being employed to attempt extraction of the amine contamination from Run VI catalyst. It is hoped that removal of the amine will return the catalyst to its pre-contamination activity. Aqueous silver, Ag^+ (as the nitrate) and sodium, Na^+ (as the nitrite) have been tested for amine removal ability. Results indicate the "hardness" of the ions tested was insufficient to effect amine removal. Aluminum, Al^{+3} (as the nitrate) and others will be tested when more catalyst sample is available.~~

003389

Methanol Synthesis
J. C. Bustabad

MS-II Unit Support (Bustabad) - Levels of MEA contaminant in approximately 100M gallons of crude and approximately 230M gallons of finished product methanol were determined by titration. These levels ranged from 0.2 - 300 ppm by weight. Samples of T-288 from various trays were titrated to determine MEA levels. Results showed that T-288 was effective in separating MeOH and MEA.

MS-III Unit Support (Russell, Presley) - The Laboratory provided analytical support during startup of the 2MM and 5MM Girdlers. The MEA systems for both units suffered residual contamination from an earlier foaming agent incident. Upon initial startup, the MEA showed excessive foaming, necessitating a MeOH wash of the system. After extensive washing and steaming, the units came up with no problems.

An intermediate solution to the problem was attempted and was unsuccessful. The addition of GE-60 Antifoam proved successful at reducing the foaming for a short period of time only. A letter will be issued documenting those results.

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

The results of monitoring ABI personnel for soluble compounds as nickel during Selas reformer catalyst loading showed no exposure exceeded the present ACGIH TLV's (8 hr TWA-0.1 mg/M³ and STEL-0.3 mg/M³). The actual range was 0.000 -0.021 mg/M³. (CEG-24-80, KVP-02-80)

Analysis by Draeger tubes around the perimeter of a lime pit during clean-out by Steve's Oilfield Services showed a range of 1.0-1.5 ppm H₂S (ACGIH STEL/ 10 min-15 ppm). (CEG-27-80, KVP-03-80).

Sampling continues on the formaldehyde exposure survey. (estimated completion date, 9-15-80) (KVP)

Experimental work using long-term Draeger tubes to determine carbon monoxide 8 hr.TWA's was suspended 8-13-80 waiting on supplies. The equipment was received 8-28-80. (CEG)

The determination of respirable dust exposures has begun. Equipment checkout and technique refinement continues. (CEG)

The evaluation of a proprietary formaldehyde-oxidizing solution (Montell Co.'s Hyde-Away) was completed. Repeated treatments of an atmosphere containing approximately 30 ppm HCHO reduced the level to approximately 13 ppm after 40 minutes. Treatments of an atmosphere containing 6.5-7 ppm HCHO reduced the level to 2-4 ppm after 20 minutes. (CEG)

A survey of the Laboratory showed two areas of high formaldehyde exposure. Corrective measures have been recommended. (CEG)

003390

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

Unit Support (Day) - None provided due to higher priority work.

Diacetone Alcohol
B. K. Huelle

Unit Support (Huelle) - None provided.

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

Recovery of PE from VCI Centrate (Harvey) - The proposed alternative of extracting a mixture of VCI Centrate and PE-WIP recycle with n-Hexanol appears to be as efficient or better for all major PE impurities than the extraction of PE AML with n-Hexanol. This conclusion is based on comparisons of shake-out studies on both proposed processes. Two continuous runs will be made for the Centrate-WIP extraction using n-Hexanol solvent ratios of 1:1 and 1:3 (n-Hexanol : Centrate-WIP) in early September to verify this conclusion.

Analytical support was provided to CCCTC personnel involved in the laboratory demonstration of the final design for the AML extractor and back extractors. Analyses were done to supply material balance data for laboratory runs.

Hazardous Waste Characteristics of VCI Centrate (Buras) - Analysis of a sample of centrate for heavy metals was completed. The concentration of specified metals was below the maximum allowed for the characteristic of EP Toxicity. A letter was written (PJB-32-80) stating that in our judgement, VCI Centrate does not exhibit the hazardous waste characteristics of ignitability, corrosivity, reactivity, or EP toxicity as defined by EPA.

Entrainment in HCHO Stripping Column (Buras) - Modifications to T-286 including tray replacements and conversion from a 19 to a 26 tray stripper were completed in July. Operations found indications that PE was being entrained overhead in T-286 following startup on August 4, 1980, and requested that the Laboratory determine the actual entrainment rate. No significant entrainment was found with the feed nozzle on tray 23.

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PE Formal Hydrolysis (Akin) - Studies continued using ion exchange resins as co-catalysts with a continuously fed strong acid. Amberlyst 15 has a resin bed-life of 83 days to present. The resin still appears to have good activity and further activity-life tests will continue through September. The effects of varying sodium concentration, acid concentration, and residence time are being studied for Amberlyst 15. Mechanical difficulties accounted for the low exposure time for the month.

Development of a System to Monitor PE Reaction Conversion (Huelle) - Batch reaction studies were conducted to simulate the PE reaction system at various mole ratios of NaOH to AcH (1.0, 1.025, 1.05, 1.08, 1.1, 1.12, 1.15). Analyses were performed on samples of the reaction mixtures, AML, and "A" filter cake. Infrared spectra and G.C. studies were completed on each set of samples. Ultraviolet spectra of the AML indicate an inverse relationship between absorbance at 220 nanometers and the mole ratio. U.V. data was also taken on the PE cake. Hot melt and hot sulfuric color tests did not yield meaningful data. These tests will be rerun after the PE cakes have been hydrolyzed. Further batch reactions and analyses are planned. Work has begun on monitoring the progress of PE reactions through the continuous measurement of redox potentials.

Trimethylolpropane
R. L. Day, P. J. Buras

Unit Support (Day) (Buras) - ~~High acid problems occurred in the TMP unit~~ almost the entire month of August. A weak base ion exchange resin bed was set up in the Laboratory to isolate the acid(s) in TMP finished product. The objective is to concentrate and identify the acid(s) which are contributing to the problem. Work is in progress.

In addition, the two phases of the extractor 312 OH stream are being analyzed for acid. ~~The distribution coefficients (wt.% acid in organic layer / wt.% acid in H₂O layer)~~ during the high acid run are being compared to those during a normal run.

Batch distillation of T-205 feed and sidestream product showed that acids in the finished product are light ends to TMP. These light ends were further purified by distillation and a sample sent to CCCTC for identification.

Acid levels in TMP unit process streams were continuously monitored during August. Data collected thus far has been inconclusive.

Work continues to identify and correct the cause of acid problems.

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Special Problems Group - General

S. G. Smith attended a meeting at CCCTC on August 26, 1980 and gave a presentation of Bishop Laboratory work concerning anaerobic filter technology to representatives of Kimberly-Clark Co., interested in licensing Celanese technology for waste treatment. Following the meeting, she and Danna Sharpe conducted a tour of the Bishop facility now under construction.

Leanne Harvey attended the CCC Equality Committee meeting in Pampa on August 20, 1980.

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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 Treatability Study	Welling	1	Compliance With Federal Regulations	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
Develop a Low Energy-High Efficiency ACH-Aldol Separation Process for the BG Unit	Buras	2	Raw Materials and Steam Savings	November, 1980
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	November, 1980
Develop a Way to Monitor Conversion in the PE Reaction System	Huelle	1	MOB (Quality) and Raw Material	November, 1980

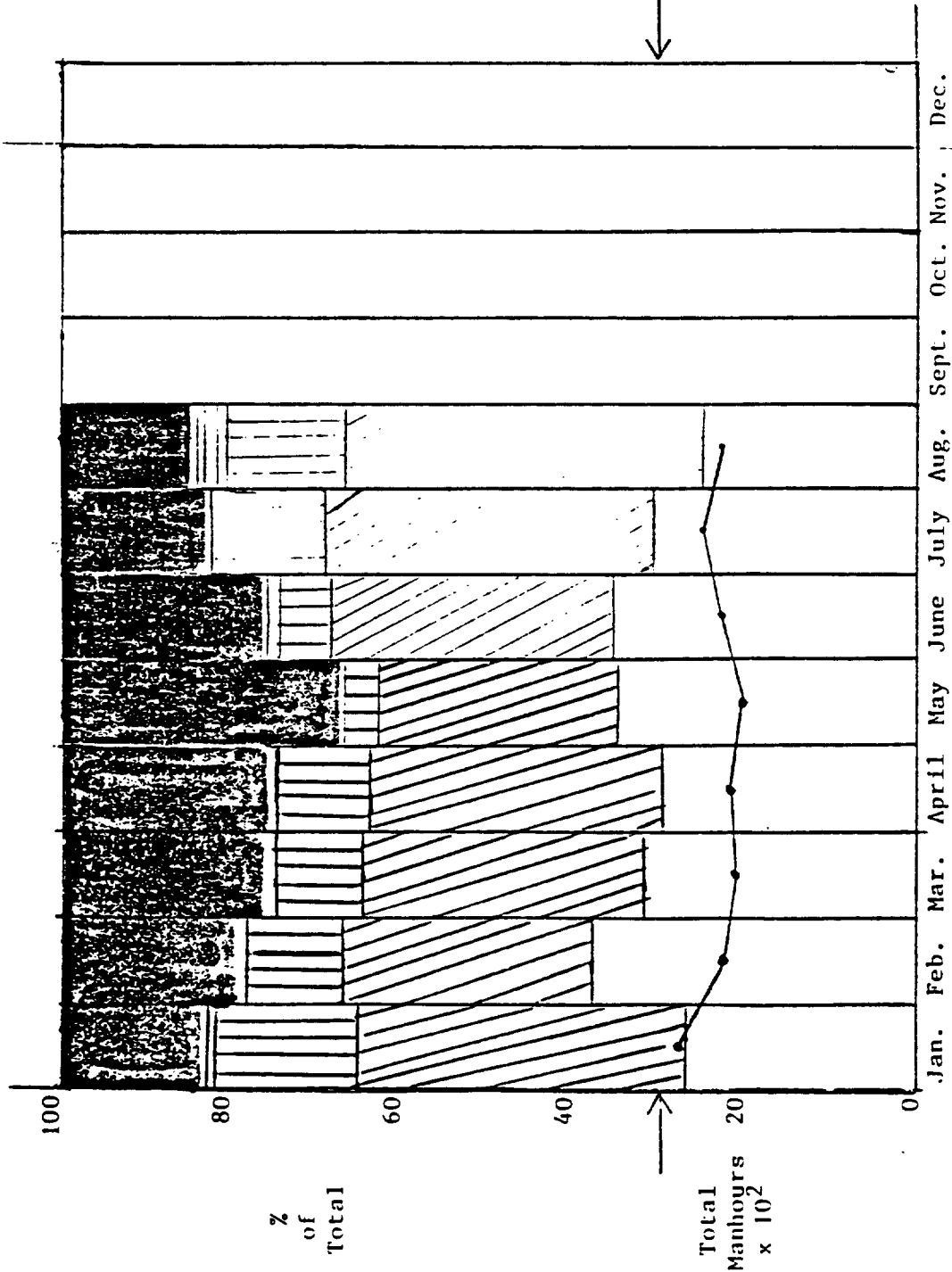
Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
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	November, 1980

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Effort Analysis - Laboratory 1980

Special Problems Groups



1980 Cpst Reduction Objective

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