

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: R. R. Graham

From: G. D. Boyd GDB-85-80 December 5, 1980

Laboratory Department Summary
November, 1980

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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 from Degussa Corp. for evaluation during November: Paraformaldehyde granular, Paraformaldehyde powder, Tech grade Pentaerythritol and Pure grade Pentaerythritol. Reports will be issued as analyses are completed.

Methanol Oxidation (MO)

Analyses for color, Iron, Copper, Zinc, and Calcium were provided around the MO units to help determine the cause of high-color HCHO and Formcels®. No significant information was gained that would explain the color problem.

Methanol Synthesis (MS)

Seven samples around the MEA section of the MS unit were analyzed to provide base case data prior to putting the new Dow corrosion inhibitor into the system.

Oxo-Butyraldehyde (BuH)

A newly developed Potassium Pyrosulfate fusion method for determining the Rhodium content of the Oxo reactor and T-210 residue has been completed and submitted for typing. This method replaced the old digestion method in the Instrument section. The fusion method has been thoroughly tested. It yields comparable results and is much safer than the previously used method.

1,3 Butylene Glycol (1,3-BG)

T-225 OH (Crude Ethanol) is being analyzed in the Instrument section to determine its "typical" composition. This project is to determine if Ethanol in this stream can be recovered in the Pampa Plant.

Polyol

Trimethylolpropane (TMP) - The sales specification for TMP has been revised. The maximum acid content has been raised from 0.002 wt.% to 0.003 wt.%.

Pollution Control

Analytical Group support continued for the monitor well sampling during the month. This work is in support of the current study to determine the extent of solar pond seepage.

Special Analytical Services

Lube Grade PE (F. R. Dow, B. W. Wise) - A meeting with Emery Industries (Cincinnati, Ohio) was attended to discuss the PE analytical procedure and to determine what PE composition Emery needs to produce Lube Esters. The analytical procedure used by Emery and Celanese are comparable and should yield the same analytical results if the same calibration sample is used by both parties. Emery was furnished a sample of the Celanese Tech PE Monitor and 99+ wt.% pure standards of Mono, Di, and Tri-PE.

Emery stated that they require 84-86 wt.% Mono-PE and that the Non-Mono-PE consists of the total Linear Formal, Di and Tri-PE. They did not specify values for any of the Non-Mono-PE components. A report detailing the meeting with Emery is in typing.

Identification of Solar Pond and Monitor Well Components (F. R. Dow, J. E. Russell, B. W. Wise) - As reported in October, a capillary GC is being utilized to separate components in the solar ponds and monitor wells. Expansion of this work included the utilization of the GC/MS at CCCTC (Dr. Cato McDaniel and Lyndle Betz). They were able to identify 70 major peaks in a solar pond composite sample. These 70 peaks account of approximately 95% of the total ion intensity of the sample.

The Chromotropic Acid determination of HCHO is being used to analyze HCHO or HCHO containing compounds in the monitor wells. This may be the best method of determining low level contamination. More wells will have to be analyzed before this method can be used for that determination.

All of the solar pond and monitor well analyses are being performed to satisfy a Texas Department of Water Resources "Enforcement Compliance Directive".

Ion Exchange Chromatograph (J. E. Russell) - The Ion Exchange Chromatograph that was ordered for BOD II will be used to help identify ionic contaminants in the solar ponds and ground water samples until it is needed for BOD nutrient analysis. This instrument should provide a reasonably complete analysis of inorganic constituents in our effluent system.

Special Analytical Projects Pending

Amines in competitive MeOH.

Construction of EPA-approved Hazardous Waste Samplers.

Quality Waivers and Delays

Delays

11-17-80 Bercen Chemical Co., Denham Springs, La. - 55% Methyl Formcel, 5000 gal. tank truck, delayed one day due to high natural color (12 Pt-Co units vs. 10 maximum).

11-19-80 Uniroyal Chemical Co., Geismar, La. and Eneco, Inc., Memphis, Tenn. - 55% Methyl Formcel, 5000 gal. tank trucks delayed one day due to high color (12 Pt-Co units vs. 10 maximum).

11-19-80 Sun Chemical Corp., Chester, S.C. - 55% Methyl Formcel, 20000 gals. delayed due to cloudy product.

Waivers

PE Pure - Color 7 Pt-Co units vs. maximum specification of 5. Shipments were made in stated amounts to the following customers:

11-7-80 Imc Chemical Corp. - Gomex, Utah - 800 x 50
11-10-80 Imc Chemical Corp. - Gomex, Utah - 800 x 50
11-11-80 Jones Blair, Dallas, Texas - 200 x 50
11-11-80 C. Itoh, Taiwan - 500 x 50
11-17-80 Spencer Kellog, Pensacola, Fla. - 150 x 50
11-21-80 Imc Chemical Corp. - Gomex, Utah - 800 x 50
11-24-80 Pinturas Optimus, SA - 300 x 50
11-24-80 Polurequima, SA - 300 x 50
11-11-80 PE Tech - Jones Blair - Dallas Texas - 600 x 50 - Water content 0.35 wt.% vs. 0.30 maximum.
11-13-80 Gliddens Coatings - Columbus, Ga - 150 x 50 PE Tech, Water content 0.35 wt.% vs. 0.30 maximum.

ANALYTICAL STATISTICS

Sample Count	November	November Y-T-D	Previous Y-T-D
Chemical Section	4160	49764	55978
Instrument Section	2486	25457	29829
TOTAL	6646	75221	85807
Technician Overtime			
Chemical Section	40	667	880
Instrument Section	73	398	467
TOTAL	113	1065	1347

Sample Shipping and Quality Assurance Statistics

Statistics For November 1980

Sample Shipping	Month	YTD	Previous YTD
Domestic	40	695	744
Export	3	30	28
Plastics	0	77	18
Other	5	83	76
Total	48	885	866

Quality Assurance Statistics

	Month	YTD	Previous YTD
Shipments (NO.of Orders)	679	7967	10288
Delayed	4	7	31
Waviers Internal	8	23	9
External	2	25	40
Complaints (Total)	0	29	46
Complaints (Legitimate)	0	13	18
Complaints (Non-Legitimate)	0	16	28
Complaints - Outstanding (Unresolved)	0	0	0

II. SPECIAL PROBLEMS GROUP

Process Technology
B. C. Kerr

Solar Pond Disposal Study - Laboratory work has continued on this project. General schemes for organic concentration and salt removal that have been examined are as follows:

1. Fractional crystallization to remove water.
2. Solvent extraction of untreated and acidified solar pond material.
3. Salt removal by acetone addition.
4. Activated carbon absorption.
5. Membrane separation with regenerated cellulose dialysis tubing.
6. Flocculents for settling and filtration.

Dialysis, using dry acetone, shows some promise of making a useable separation. It is planned to continue this work. However, in all cases examined, raw material, energy and equipment costs appear to be formidable.

Enforcement Compliance Directive Report - Work has begun on assembling data for the ECD report which is due in Austin on January 2, 1981.

Methanol Oxidation, Formaldehyde Blending
T. A. Jasek

GC/MO Reactor - No progress, awaiting the arrival of some reactor heaters.

MO Silver Catalyst Modifiers - A test sample of silver crystals was grown from a silver nitrate solution containing 100 ppm Bi (bismuth). These were sent to the Tech Center to determine the concentration of Bi. This value will be used to determine the amount of of Bi which needs to be added to the electrolytic solution so that silver crystals with a desired amount of Bi can be grown.

MO Unit Support - Formaldehyde Color Problem

MO-III - Developed high color in T-313 Residue (50% HCHO) beginning November 7th and continued several days thereafter. The color decreased to 12 on November 12th and ranged from 10 to 12 thereafter until shutdown on November 23rd. As suspected, several cracks were discovered in the T-313 false bottom. This allowed for the formation of color bodies (probably formose sugar type compounds) in the area of the false bottom. These color bodies then apparently returned via the cracks to the 50% HCHO.

Analytical AMR's were conducted at MO-III by the Laboratory on November 24th and 25th following the repair of the cracks in the T-313 false bottom. The following table illustrates the data obtained:

MO Unit/Train	No. of Samples	AMR Indicated	AMR Analytical
MO-III/A	3	0.98-1.02	1.64-1.73
MO-III/A	2	0.84-.86	1.18
MO-III/B	8	0.90-.92	1.78-3.49

The differences between the indicated and analytical AMR's appear to result from a very low flow rate as well as the sampling system, especially at B train. As the AMR's were being conducted, the sample tap was exposed to a cold wind as well as rain. This apparently caused condensation of the methanol, raising the AMR. Although the sample line from the sample tap was steam traced, this did not remedy the situation as the actual AMR had already been changed by the sample tap.

MO-IV and MO-II - Both MO-IV and MO-II experienced color problems during the month of November. Colors of 5 to 7 were observed in both units until operational changes were made. This caused the color to drop to <5.

The feed methanol to the units at this time was slightly colored (5 to 7). Some color was caused by particulate iron and was filtered out (only at MO-IV). The remaining color was affected by an iron chelating agent. Attempts were made to extract color species from the MO-IV vaporizer blowdown, but were not successful. Work will continue in an attempt to isolate a color species in the methanol which may have been responsible for the color problems at MO-IV and MO-II.

Lauric Acid - A sample of white material was removed from the quench water filters at MO-III. Analysis of the material showed it to be lauric acid. The source appears to be the polyol area which uses lauric acid esters as antifoam agents. The common link betwen polyol and MO-III is the methanol received from the Weak Formaldehyde Unit via T-185 OH.

Utilities, BOD

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

Anaerobic-Aerobic Pilot Studies

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

Start-Up Study/Upflow Velocity Study (Welling, Smith, Stasney) - Two anaerobic-aerobic units are being run to study 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 ft/hr (to simulate the upflow expected in the commercial unit). Waste from the Oxo unit sump was added to the feed to the filters on 11-1-80 at levels projected for 1981 (about 3.3% of filter loading). This increased the organic loading (O_2) to 0.36 lbs COD/ft^3/day and decreased the detention on the aeration units from 3.5 days to 3.4 days as is expected for 1981. No detrimental

effects were observed following this addition. Polyol unit waste was started to the units on 11-19-80 as a level of 0.5% of anaerobic feed volume with an OL of 0.38 lbs COD/ft^3/day and aerobic detention time of 3.4 days. The concentration of polyol in the feed will be increased gradually to the 1981 projection of 3% of feed volume, with an OL of 0.45 lbs COD/ft^3/day, and aerobic detention time of 3.0 days. The only effect that has been seen thus far on filter operation since addition of polyol is the need to add more Na^+ for buffering (pH dropped to 6.7). Filter recycle COD's have averaged 835 ppm for #2 and 617 ppm (90% COD removal) for #3 this month. The kinetic efficiencies have averaged 67% and 70%, respectively. After aerobic polishing the outfall CODs have averaged 121 ppm for #2 and 109 for #3.

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 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 159 parts equalization or equalization + water to one part pond material. The outfall CODs at this dilution were 340 ppm for #5 (95% COD removal) and 330 to 340 ppm for #6 (95% COD removal) with a control COD of about 190 ppm (97% removal). On 11-14-80 the concentration of solar pond material was increased (151:1). The outfall CODs at this dilution were about 380 ppm (94.5% COD removal) for #2 and 340 ppm (95% COD removal) for #3 with a control COD of 230 ppm (96.5% COD removal). On 11-24-80 the concerntration of pond material was again increased (135:1). It is too early to determine the results of this increase.

Elimination, Reduction of Solar Pond Odor (Smith, Jasek) - Investigations concerning the elimination of bacterial activity of odor producing BOD sludge blowdown have been in progress. Applications of commercial biocides, H_2O_2, calcium hypochlorite and heat have been tried. The following additions to 100 ml sludge blowdown samples look promising for reducing anaerobic activity and odor of the sludge: 4-5 ml Oxysol (made by Ind-Sol Chemicals), 5-10 ml 30% H_2O_2, and 0.1-0.3 gm calcium hypochlorite. Increasing the pH of a sludge sample to pH 12 (NaOH), initially produced an offensive odor, but after several days, the odor became more acceptable, even sweet smelling. Boiling the sludge at 100°C for up to 20 minutes did not reduce anaerobic activity or odor of the sludge. An odor masking agent, comprised of in-Plant acetates was prepared, consisting of equal parts of n-Propyl acetate, n-butyl acetate, and i-butyl acetate. This combination appeared effective in the 0.010-0.015% (volume) range.

Methanol Synthesis
J. C. Bustabad

MS-II Unit Support (Bustabad, Presley, Russell) - Base case data were obtained on the MEA systems prior to the charging of Dow's corrosion inhibitor, ammonium thiocyanate. Concentrations of corrosion inhibitor in the MEA systems has been monitored since the initial charging of the inhibitor on November 6th.

The colorimetric method provided by Dow has been used for these analyses. Charging of the corrosion inhibitor was halted on November 12th when it was suspected that the 4.2% and 2.5% methane slippages of the 2MM and 5MM Girdler reformers, respectively, were related to the corrosion inhibitor. Laboratory tests (Tracor instrument and Draeger tubes) confirmed that the ammonium thiocyanate was breaking down forming H_2S, a catalyst poison for the 2MM and 5MM Girdler reformers. The H_2S was reaching the reformers via the recycled CO_2 from the MEA systems. The Laboratory recommended venting as much of the contaminated CO_2 as possible to purge the system of the H_2S. This recommendation was implemented in the Plant. A further Laboratory recommendation, to eliminate H_2S from CO_2 fed to the reformers was to put a slip stream of the contaminated CO_2 through a copper treated carbon bed. This recommendation, based on Laboratory tests conducted by Jim Presley, was also implemented successfully in the Plant.

H_2S levels were determined to be non-detectable or neglibible (<15 ppb) for syn gas to Oxo (2MM and 5MM systems), hydrogen before and after methanator, and purchased CO_2.

Oxo, Butanol
J. D. Presley

Reactor Stability/Accountability - The reactor has maintained stable operation for most of November, and accountability has averaged better than 85%.

A cross check standard was prepared and submitted for evaluation. Results indicate reported TPP values to be about 2% low and BuH values to be about 4% high. Work will continue to correct these discrepancies.

Activity tests have been run on BASF and Ihara TPP samples as a comparison. Both samples were deemed suitable. A letter will be written

Rhodium Accountability - Rhodium accountability is not available at this writing. We are currently in the process of changing to the new fusion method of analysis.

Improved Rhodium Recovery - Activity tests of the third phase extractions are complete. Additionally, phase four extractions have been started. These will test oxygen reactivation of extracted material. Work continues.

Oxo Unit Support - A check of methane slip across the 2MM and 5MM Girdlers was made. A letter has been written.

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

A personal noise exposure survey in the Paraform/Acetate area showed that all unit personnel should be included in the hearing conservation program. Only the Guager and Grinder need to wear hearing protection (8 hr TWA > 85 dBA). (CEG-35-80, KVP-05-80)

Personal noise exposure data is being obtained in the Utilities and MO areas. (KVP)

Monitoring for CO exposure has been completed in the Oxo area and is being continued in the MS area. (CEG)

Arthur Bros. personnel engaged in loading Paraform flake were monitored for formaldehyde exposure in conjunction with monitoring by Ms. Sharon Ratliff of OSHA. The eight hour time weighted average exposure was 1.4 ppm and the highest short-term exposure level for 30 minutes was 1.8 ppm. (CEG, KVP)

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

Formcels® Unit Support (Akin, Day) - Studies were conducted to measure the amount of color removed from Formcels® by various methods. Carbon bed treatment proved effective in removing about 60% of the color. Another method which proved effective in removing color was "cooking" the samples at 93°C. After 12 hours 40% of the color was removed from the Formcel samples. (Akin)

Methods which proved ineffective in removing color were treatment with EDTA, H_2O_2, and Citric acid.

Low colored Butyl Formcel was made in the Laboratory using paraliquid from the 3rd stage evaporator. (Day)

Paraform Lumps (Day) - It appears that there has been an improvement on both the East and West train flake since the October shutdown. The West train is currently producing better quality flake (lower wt.% lumps) than the East train. A letter with recommendations will be co-authored with Process.

Pentaerythritol
D. R. Akin, P. J. Buras

Eliminate VCI Centrate Waste Stream (Buras) - Evaluation of alternatives to the previously identified extraction process continues. The initial objective is to non-selectively 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.

Several solvents were evaluated for removal of organics from AML and concentrated AML. Preliminary data obtained from one-stage shake outs indicated that the best solvents for extraction are t-butanol and n-butyraldehyde. Adding methanol to butyraldehyde improves the ability of butyraldehyde to extract "PE-type organics" from aqueous sodium formate. This mixed solvent system (MeOH + n-BuH) appears to be superior to all others previously evaluated. Work continues to identify improved mixed solvent systems. Evaluation of the MeOH/BuH mixed solvent in a continuous laboratory extraction is in progress.

PE Formal Hydrolysis (Akin) - Work was continued using Amberlyst 15, an ion exchange resin, as a co-catalyst in conjunction with a continuously fed strong acid. This resin's exposure time to date is 134 days. The conversion rate of linear formals increased after a 10% sulphuric acid wash followed by a backwash with methanol. The sulphuric acid wash removed measurable amounts of Nickel and Copper from the bed. These metals are believed to have come from connecting fittings in the laboratory hydrolysis system. The metal fittings were replaced with glass fittings. The methanol wash removed a brown film from the resin. This brown film is believed to be high boiling material from the PE feed. A larger, glass wool filter was installed to remove the film. The conversion rates of Linear Formals after reactivation of the bed were comparable to the rates of conversion in July. Variable studies of the effects of sodium concentration and acid concentration (with a constant residence time) on conversion of Linear Formals were conducted on this resin.

High Na_2SO_4 Levels in AML (Akin) - On November 21st, VCI reported high levels of Na_2SO_4 in the AML. The AML on this date was sent to the solar ponds rather than to storage. When Na_2SO_4 levels on a contained salt basis exceed 3.0 wt.% VCI cannot take the AML. VCI was having problems with their analytical method. The Bishop Laboratory provided analytical support to VCI. High Na_2SO_4 levels were confirmed by the Laboratory on samples taken on November 22nd and 23rd. On November 25th, Na_2SO_4 levels were low enough that the AML could be stored. VCI began taking the stored AML on November 26th. On November 25th the level of Na_2SO_4 on a contained salt basis in storage was at 2.3 wt.%. On November 26th the level of Na_2SO_4 in the AML tank was about 2.0 wt.%. By December 1st the level of Na_2SO_4 in the AML tank was at 1.0 wt.%.

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.

Diacetone Alcohol
B. K. Huelle

Unit Support (Huelle) - None provided.

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

Unit Support (Huelle) - The GC analysis of 747 OH jet exhaust contains several peaks with retention times coinciding with decomposition products and 202 OH retention times. A letter concerning this analysis and jet exhaust efficiency was co-authored with Raymond Yu.

Decomposition Study (Huelle) - A study of the effect of NaFo on TMP decomposition has been started. Three experiments were done using 100, 500, or 1,000 ppm NaFo charged to a vessel containing TMP and decane. The vessel was heated for three hours at 200°C, collecting volatiles overhead during the last hour. Analysis of the overhead collection indicate a significant production of methanol, water, and organic decomposition products at 500 and 1,000 ppm NaFo. GC analysis of the TMP layer in the reaction vessel indicate the presence of non-volatile heavy ends. Further study varying either temperature or reaction time will be done in December.

Acid Problem (Day) - A letter was written (RLD-17-80, PJB-37-80) on the TMP high acid problem. The extended Oxo shutdowns, coupled with butyric acid as the major acidic component in TMP 205 SS, makes the butyraldehyde feed a possible suspect as to the cause of the problem.

Extractor Optimization (Day) - The T-312 extractor optimization project is still in progress; one set of base case data has been gathered, and another set will be collected in the near future.

Special Problems Group - General

Kathryn Parker, Chuck Gary and Hub Thigpen attended a three day Industrial Hygiene Course in Dallas, November 11-13.

Susan Smith and Pam Welling attended meetings with Pampa Celanese Chemical Co. and Vernon CP & S Company personnel, November 17-19. The meetings provided an exchange of information between the Plants concerning the design, startup, and operation of the Celanese anaerobic filter.

Current Special Problems Projects

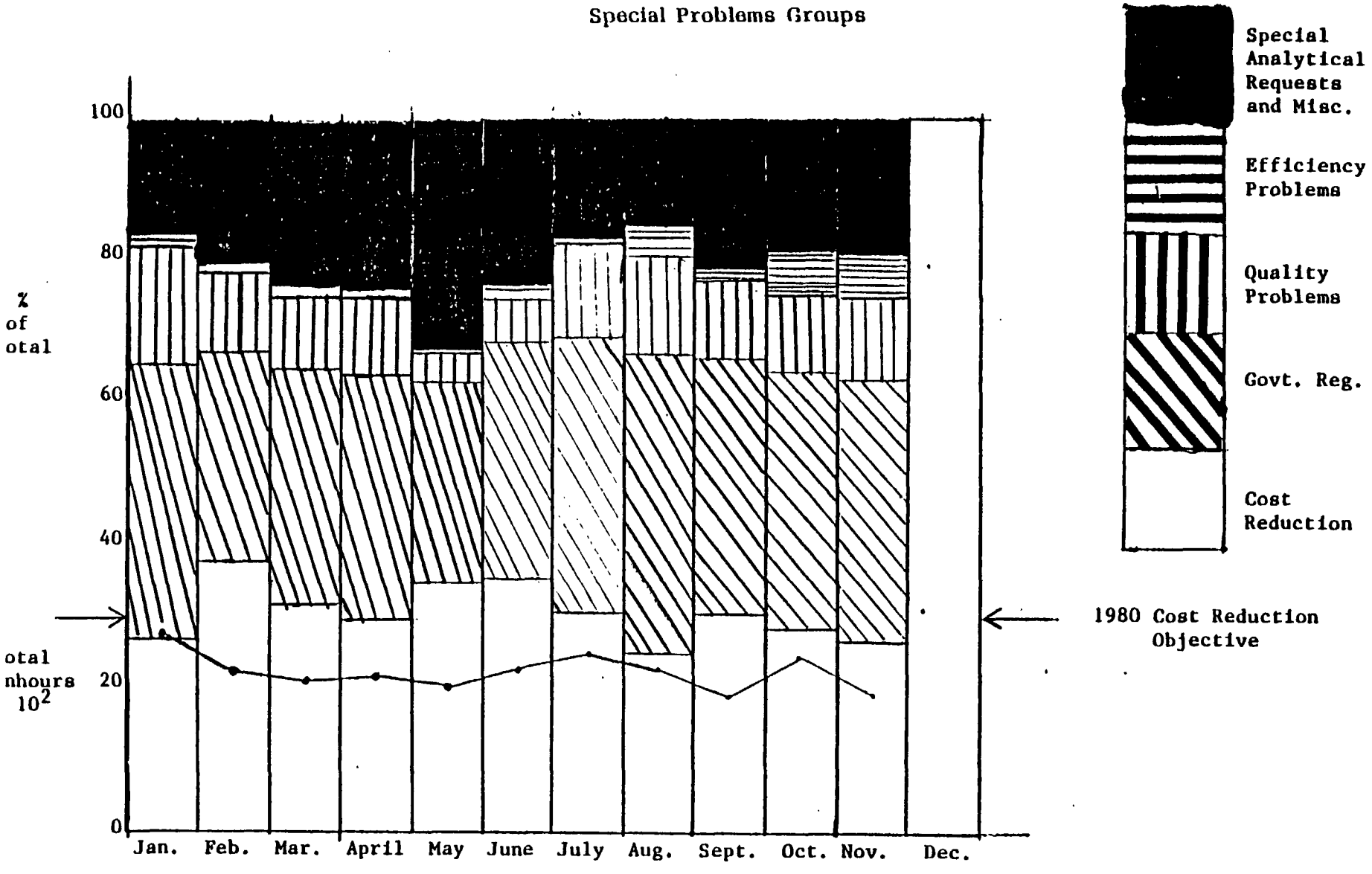
Project	Chemist	Priority	Justification	Estimated Completion Date
Solar Pond Disposal	Kerr	1	Government Mandatory	June 1, 1982
MO Catalyst Screening	Jasek	1	To Establish Alternate Catalyst Suppliers	Continuing
Formaldehyde Product Color		1	MOB	January, 1981
GC Methanol Oxidation Reactor Studies		2	Catalyst Evaluation for Possible Efficiency Improvements	Continuing
Modified Silver Catalyst		2	Catalyst Efficiency Improvements	Continuing
Anaerobic Testing of Chemical Wastes	Smith Welling	1	Support Unit Expansion	Continuing
Solar Pond Treatability Study	Welling	1	Environmental Mandatory	Continuing
Elimination/Reduction of Solar Pond Odor	Smith Jasek	1	Environmental Mandatory	January, 1981
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

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Develop a Way to Monitor Conversion in the PE Reaction System	Huelle	1	MOB (Quality) and Raw Material	February, 1981
TMP Extractor Optimization	Day	2	Raw Material Savings	Undetermined
Ester Reactor Corrosion Control		2	Extended Equipment Life	February, 1981
New Formaldehyde Stabilizer System		1	MOB	January, 1981
Develop a Low Energy-High Efficiency AcH-Aldol Separation Process for the BG Unit	Buras	2	Raw Materials and Steam Savings	June, 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	March, 1981

Effort Analysis - Laboratory 1980

Special Problems Groups



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