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CEL-248

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: W. H. Meyer WHM-136-78 3/2/78

Laboratory Department Summary

February, 1978

D I S T R I B U T I O N

Chemical

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J. Chervenka
T. N. Dowdy
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K. G. Gallimore
R. S. Hahn
M. D. Harvey
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M. A. John
B. C. Kerr
C. W. Lee
D. E. Lockwood
Dr. C. E. Loeffler
Dr. R. P. Lowry
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A. G. McGehee
J. T. McMahon
R. A. Mickelson
J. C. Rubiano
J. M. Sawyer
G. R. Scholz
W. L. Sharp
R. W. Swanbeck
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T. L. Trumbley
. J. Volpe

Plastics

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A. C. Abshier
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R. L. Bunkley
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K. A. Rose
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CCCTC

G. J. Fisher
W. E. Heinz
Dr. C. C. Hobbs
W. J. Humphrey
W. T. McNair
Dr. D. R. Napier
W. E. Taylor
Central Files (2)

Dr. J. C. Trebellas

J. N. Gann — Bay City
J. W. Dunn — Clear Lake
J. M. Ramey — New York
Dr. T. H. Golson — New York
J. E. Sanborn — Bayport Terminal
W. H. Tuke — Pampa
F. B. McAndrews — Summit
Dr. F. Zitomer — Summit

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The Bishop Plant Laboratory activities during February are attached. Section I contains a summary of the Analytical Group activities with a table of analytical statistics. Section II contains a Quality Assurance Summary and a table listing shipments, quality waivers, quality complaints and sample shipping statistics. Section III describes the status of Special Problems Group projects by Unit Chemist assignment. A table listing the priorities, justifications and estimated completion dates of current Special Problems projects is also included in Section III.

W. H. Meyer

WHM/dls
Attached Report

003194

I. ANALYTICAL GROUP

Competitive Evaluations

Pentaerythritol (PE) - Samples of Japanese and Korean PE (companies unknown) were received and analyzed. The samples were obtained through the AZS Company, a Celanese customer. Main points of variance with Chemcel Tech Grade product are ash content (high), color (high) and formal content (high).

A sample of Perstorp PE was obtained through another customer, the Cincinatti Millacron Co. This material was comparable to our Pure Grade PE.

Complete reports on these competitive samples will be issued by the Quality Assurance Group.

Methanol Oxidation (MO), Formaldehyde (HCHO)

Composite samples of MS Unit Finishing Column side stream draw off (Methanol) are being monitored for a 30 day period. These analysis will establish average purity levels of this current MO unit feed stock. A report will be issued at the end of the 30 day study.

Methanol Synthesis (MS)

Analytical assistance was provided for column profile studies around T-280 and T-288. A report will be issued by F. R. Dow.

Oxo Area

Propylene - Four samples of Arco produced propylene were received and analyzed for the Clear Lake Plant. This is a periodic spot check of the feed stock supplied to Clear Lake via pipeline. Oxygen content of the samples was high ranging from 80-350 ppm. All other parameters met specification.

Paraform

Formcel® - Assistance was provided for analysis of Methyl Formcel® produced in laboratory quantities from Paraformaldehyde Flake.

Methylal (Meal) - Spheroid tank 689 is now being used for Meal inventory. Due to lack of sampling and circulating facilities a representative sample of this tank cannot be obtained. One 5.0m gallon TT shipment (on2/7/78) loaded from the vessel met all specifications. Sampling facilities need to be installed in order to follow the inventory quality.

Polyol

Pentaerythritol (PE) - Extensive analytical assistance was required during the early February product switch from Pure to Technical Grade PE. This extra analytical load has continued throughout the month and appear to be related to our inability to obtain representative samples of the finished product. Numerous resamples, repeatable within themselves, are not reproductible across the days (one lot) production. The automatic composite sampling apparatus recommended to obtain accurate samples is not yet functioning.

1.3 Butylene Gylcol (1.3 B. G.) - A customer (Union Camp) provided procedure for Heat Stability Test was run on 4 samples of 1,3 B.G. representative of 1977 production. Our results were relayed to the customer. Quality Assurance is attempting to obtain customer test result and specification requirement.

Trimethylolpropane (TMP) - Three samples of TMP (1= Perstorp, 2=Celanese) were analyzed for CCCTC, Dr. J. A. Broussard) in support of a color improvement in TMP-tri-acrylate project. Results were published in JHH-17-78.

BOD Unit,Utilities

Ditch Outfall - Analytical assistance was provided in support of efforts to isolate the source of inorganic carbon buildup in the ditch outfall and in the BOD Unit. Equialization Basin, Sodium analysis were run on all Plastics Co. streams to BOD. Results were inconclusive and assistance will continue as needed. COD results on the outfall remain normal.

Analytical Statistics

Sample Count	February	February YTD	Previous YTD
Chemical Section	4385	8843	9506
Instrument Section	2220	4652	4485
Total	6,605	13,495	13,991
Technican Overtime			
Chemical Section	151	242	133
Instrument Section	0	58	290
Total	151	300	423

I. Quality Assurance

Formaldehyde - During the month there were nine formaldehyde blend problems due to problems with the in-line blender. This rate is not as high as in previous months but problems still exist in the blender operation.

Two customer complaints were received from Gulf Adhesive at Alexandria, La. The first was on a Tank Car of 50% HCHO in Celx 13208 which they had partially unloaded, stored the balance for a while and upon inspection the product had become cloudy. They claimed credit for this balance and returned the car. A second car of 50% HCHO in Celx 13232 shipped on 2/8/78, arrived at a temperature of 85°F. Product was cloudy. Tank car Celx 13232 was the subject of an earlier complaint from Acme Resin. This tank car along with Celx 1295 which has been involved in two or more complaints in high formaldehyde service, are being considered for other service until tank car supply will permit a detail inspection of the car insulation.

A quality waiver was granted, by New York, Q. A., to ship an inhibited blend of HCHO (37%-12-15% Methanol) to the Rinchem Co., Phoenix, Arizona. The customer trailer (CPO) was loaded with product that was misblended (34.85% HCHO, 15.74% Methanol).

Methanol - A proposed set of manufacturing control specification for ACS Grade Methanol has been received. The specifications will be reviewed by production and the laboratory and comments will be forwarded to New York when complete.

A train derailment near Abilene in early February caused a number of trailers of Methanol to be trucked to Bishop, sampled and analyzed. Only one trailer load, apparently contaminated with diesel fuel and water had to be burned. All of the other material was shipped back to Pampa as spec product.

Formcel - Regular grade Methyl Formcel continues to be made for all customers except for American Cyanamid. No major quality problems have been noted this month.

On February 16th a test load of the new grade of Methyl Formcel was made and shipped via tank truck to Bayport. The product was then loaded on board a deck tank for export shipment to Rotterdam. The quality was comparable to what we ship to American Cyanamid.

Iso Butyl Acetate - Tank 636, with 27,000 gallons, inventory tank of Iso Butyl Acetate, was found to be wet (0.16% water). Part of this inventory was blended with tank 635 and the balance will be used to start up the Iso Butyl Acetate run which is now in operation. The new product will be used to blend down the residual inventory to below spec @ 0.10% water. Source of the water is unknown at present.

003197

Paraform - Paraform bagging has implemented the sampling procedure recommended in Hub Thigpen's letter (HHT-30-78). The results of this bagging composite to date are encouraging. The assay appears to be at a uniform level and within the specification range of 91.0 to 93.0%.

1, 3 Butylene Glycol - The Butylene Glycol unit started operation during this past week. Low purity during the start up is related to adol reaction problems.

Sample Shipping and Quality Assurance Statistics

Statistics For February 197

Sample Shipping	Month	YTD	Previous YTD
Domestic	42	79	214
Export	3	7	23
Plastics	2	4	17
Other	12	20	263
Total	59	110	517

Quality Assurance Statistics

	Month	YTD	Previous YTD
Shipments (NO.of Orders)	663	1308	1673
Delayed	1	6	5
Waviers Internal		1	4(T) *
External	3	5	
Complaints (Total)	2	6	8
Complaints (Legitimate)	1	2	4
Complaints (Non-Legitimate)		2	4
Complaints - Outstanding (Unresolved)	1	2	

* Total Waviers

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III. SPECIAL PROBLEMS GROUP

Methanol Oxidation, Formaldehyde Blending
J. D. Presley

MO Efficiency Increase with
Trace Amounts of Halogenated Hydrocarbons

Recent review of data has suggested that the 1977 Plant test was conducted at too low an air-to-methanol ratio (AMR) to realize the predicted efficiency gain. Additional laboratory work with the bench scale MO Unit is planned to better determine the relationship of AMR to efficiency during halide injection. The Celanese R. F. P. A. for Halide Addition to MO is currently under review by the U. S. Patent Office.

MO Catalyst Reuse -No progress due to priorities.

MS II Crude to MO - Status Unchanged.

Reduce Methylal Formation in MO Distillation Columns - No progress due to priorities.

Nepera Blend - CCCTC has requested a one gallon sample of the Nepera Blend. The material will be used to determine the effects on tank linings.

T-240 Overhead Effects on MO II - A report of the Plant test is in progress.

MO Unit Support - A method has been devised to determine iron in the MO Units air supply. Work will begin on arrival of sampling equipment. Sampling taps are to be installed in MOIV to accomodate the sampling procedure.

During the 2-22-78 MO IV shutdown, the air check valves ahead of the air MeOH mixer were replaced with new spring loaded valves. The old valves (a different type) showed excessive wear. The "flapper" shaft alone had lost an equivalent of 900 ppm Fe on 12 catalyst bed surfaces (top 1/8 in.) over the six month period the valve was in service. This equates to 5 ppm Fe per bed per day based on 12 reactors and top 1/8 in. of catalyst. Other parts of the old check valve also showed excessive wear but an accurate determine of Fe lost was not possible. This is about 1 oz of iron over 6 months.

Since late January, MS finishing columns upper and lower sidestreams have been blended with MO grade Methanol. No adverse affects have been observed in the MO production units due to the sidestreams.

Work is underway in MO IV to obtain AMR vs efficiency data. The object of this work is to determine if the MO units can be operated more efficiently by optmizing AMR on a daily basis.

Utilities, Raw Materials, BOD
S. G. Smith, G. R. Stasney

Purchase Specification Update - No progress due to priorities.

BOD Unit Analytical Methods Manual - Operations is testing the feasibility of substituting TOC for COD in the Unit Analysis of the equilization basin, aeration basins, and outfall. Until these changes are tested we will not finalize the methods manual.

Anaerobic Testing of Chemical Wastes - The Laboratory Pilot Unit came up on start-up solution on 2/8/78. Gas production was noted on 2/10/78.

Plans are to introduce Plant effluent during the week ending 3/4/78/

Demineralizer Expansion Support - A report is in progress on the effects of increased demineralizer capacity on the Lime Pits outfall and the neutralizer tank.

Compatibility of T-240 Overhead with the Gasoline Tank - A report is in progress.

800 GPM Demineralizer Unit Support, Marvin Harris - The weak base resin (IRA-94) was found to be organically fouled. (60% capacity). The 60% capacity was confirmed by analysis of the resin in the laboratory, and by determining the acid removal capacity of V-2409. Laboratory studies have also shown that the resin can be restored to approximately 80% capacity by NaOH/Brine treatment. This treatment will be attempted in the Unit during March.

Stability of 50 and 52% Formaldehyde - Marvin Harris - Lab work is complete A report is in progress.

MO Unit Support - Marvin Harris - Due to apparent external chloride stress corrosion in the MO II distillation column T-296, the laboratory was asked to determine the chloride content of the material used to insulate the column. Results showed 720 ppm water soluble chlorides. A report is being written.

Studies to Eliminate OXO - T-210 - Marvin Harris - No progress due to priorities

OSHA Monitoring
J. M. Jackson, L. M. Pena

Samples of airborne formaldehyde and respirable paraformaldehyde dust were taken in the Paraform Warehouse powder bagging area. Results were obtained and sent to the Safety Supervisor (Personnel Department) for distribution.

Area and personnel 8 hour TWA samples for formaldehyde have been taken in the MO IV area. Analytical results are complete. Data has been sent to the Safety Supervisor (Personnel Department) for distribution.

OSHA Monitoring Cont'd

All results of personnel monitoring in the Plastic Company TO II, SH I and SH II areas have been received and sent to the Safety Supervisor (Personnel Department) for distribution.

Personnel 8 hour TWA samples for benzene and formaldehyde are being taken in the MO II area. Benzene exposure is being determined because Celcon T-240 overhead (which contains benzene) is being fed to the MO II scrubber (T-296).

Esters, Methylal, Paraform, Formcel Products
R. G. Briell, R. G. Hilbrich

Alternate n-Butyl Acetate Process - Progress has been hindered by reoccuring pump failure due to corrosion. Modifications to the liquid metering system are being made to work around this problem. Work continues on attempting to reduce the acid to an acceptable level in the finished product.

Paraform, Formcel Products and Ester Unit Analytical Manual - No progress due to priorities.

Acetate Unit Support - Work is complete on determining the composition of standards for use with the acetate reactor vapor analyses by on-line G. C. Standards that are stable from further reaction and phasing have been prepared for three acetates produced. A report has been written..

Paraform, Formcel Products Unit Support - Caustic treated Methyl Formcel was produced in the laboratory using 92% paraflake as the source of formaldehyde. A stable product with 80 ppm sodium was obtained after a two hour "cook time". Eighteen hours was required when no caustic was added. Producing Formcel products in this manner is a good laboratory method for estimating relative catalytic effects of varying amounts of caustic addition. It is planned to evaluate the catalytic effects of caustic addition while making Butyl Formcel by this method. Possible incentives for producing Formcel products from Paraflake will be considered.

Control Reflux on the Ester Unit Reactor Column - M. E. Harris
This project was begun by R. G. Hilbrich and some preliminary data was reported under acetate process unit support. There has been no progress in recent months because of other high priority projects. M. E. Harris will complete this project to correlate the acid content of the organic layer in the overhead receiver to conductivity in the water layer. Automatic monitoring of conductivity would allow the microprocessor to control reflux so than only the amount needed is used. This will save energy.

PENTAERYTHRITOL
R. G. Briell

PE Unit Air Emissions - Sampling has been completed on all air emission sources in the PE unit. All analysis of the condensable protions of the samples have been completed. We are waiting for the mass spectrometer analysis being done by the CCCTC on the non condensable portions of the samples to complete the analysis. The particulate sampling from dust collection stacks has been delayed and will be completed when weather permits. A joint letter with Process Engineering will be issued upon completion of analysis and particulate sampling.

003202

Update of PE Unit Methods

The new sodium specific ion electrode needed for one of the new methods arrived damaged. has been returned and we are now waiting for still another electrode. The manual will be completed and issued after the new electrode arrives.

Reducing Sulfates in PE AML - A comparison was made between the amount of sodium sulfate detected by the Bishop lab, the amount detected by Virginia Chemical Industries (VCI), and sulfuric acid consumption as reported by yield accounting. The purpose was to determine whether analyses of AML for sodium sulfate accounted for the sulfuric acid fed to the unit. Results indicate that about 30-40% of the sulfuric acid fed is not accounted for by the analyses. Our sodium sulfate analyses account for less of the sulfuric acid than the VCI analyses. We have checked and rechecked our method and it appears to be accurate. We will continue efforts to resolve the differences between our analytical method, the VCI analytical method and yield accounting. A status report will be issued in March with recommendations which should reduce the sodium sulfate content of AML.

1,3 Butylene Glycol, Diacetone Alcohol
A. R. Hancock

1,3 Butylene Glycol Unit Support - The BG unit started up in late February with no Laboratory support needed to date.

DAA Unit Support - The study continues in the DAA unit to determine if the Goodyear acetone is the cause for the rapid deactivation of the catalyst beds that has been seen in the past months. A run with W-10 acetone only (no Goodyear acetone) was completed. Preliminary analyses of reactor product samples showed a longer than normal catalyst bed life. The DAA unit started up again (on the same catalyst bed) with a 2/3 Goodyear acetone: 1/3 W-10 acetone feedstock. Reactor product samples are being collected once per shift and will be analyzed by the Laboratory. T-295 res composite samples are being collected, but will not be analyzed unless data are needed to explain some unusual results. At least one more run with the Goodyear acetone will be made. The laboratory will continue to follow the studies.

Monitoring T-295 Residue for Acetone- A study on the acetone losses in T-295 residue has been completed. It will be recommended for the DAA unit to use the initial boiling point of T-295 residue as a control for acetone losses.

DAA Laboratory Reactor Study - Progress has been slowed on the report of the effects of water in the acetone feed. Higher priority has been given to coverage of the study that is currently underway in the unit to indicate whether Goodyear acetone causes reduced catalyst activity.

1,3 BG and DAA Air Emissions Survey - A joint report with Process Engineering has been completed and will be issued in early March.

Trimethylolpropane
L. Mayerle

The Laboratory extractor continued to experience phasing problems despite an increased concentration of 2-ethyl hexanol in the feed. Laboratory shake out experiments will be directed toward determining temperatures and solvent concentrations which result in satisfactory phasing.

Adjust Reaction Temperatures to Minimize Formaldehyde Loss - A plant trial to determine the relationship between MeOH production and reactor temperature was begun late in February. A letter will be issued with Process Engineering upon completion of the trial.

TMP Unit Air Emission Survey - Three unit vents were resampled in February, T-202 OH Receiver Vent, V-759 Vent (8" stack), and V-759/T206 OH vent (10" stack). Analyses on condensable materials are complete. Final calculations will be completed when mass spectrometry analyses of non-condensable materials are received from CCCTC.

TMP Unit Analytical Methods Manual - Completion of the methods manual continues to be delayed by the lack of a sodium ion electrode.

Methanol Synthesis
F. R. Dow

Fuel Gas Quality

On 2/13/78 a sample of MS-II methane feed was analyzed for component composition. A report is being issued.

MS-II Analytical Support - Back up analysis for H_2S and COS in natural gas feed to the Selas and DPG was provided by the Laboratory. Rapid sulfur break through in the Selas carbon guard bed without a corresponding deterioration in the DPG carbon guard bed initiated the request for Laboratory support. A report is being issued.

Samples of the Selas,DPG and Combined Reformer gas were analyzed in the Laboratory to verify values reported by the process analyzers. For CO at the 15 vol% level a maximum difference of 0.7 vol% was determined. Methane at the 1.5 and 3.0 vol% levels had a maximum difference of 0.09 vol% and CO_2 at this 7-8 vol % level showed a maximum difference of 0.25 vol%.

Four vent streams were analyzed to update MS-II computer data used for calculating BTU values. The vent streams analyzed were T-187 OH vent, T-2790H vent, V-2360 OH Vent (expansion gas) and the Excess Hydrogen Vent.

PROCESS TECHNOLOGY
B. C. Kerr

Technical Report Abstracting - Preparation of technical report abstracts is current. Typing abstract cards has been temporarily delayed.

Operations Contribution to Process Technology Documentation - A simplified report form has been prepared for distribution to Operations. Forms should be distributed during the first week of March.

Statistical Data for PE Unit Operations - Statistical data are being prepared for Pure Grade PE production. Operations data from January 6-28, 1978, are being used for this study.

Purchase Specifications Recommended for T-240 O.H. - A report has been written, in cooperation with Mr. F. R. Dow, recommending specifications and special analytical procedures for use of T-240 O. H. in MO Unit operations. The report is being reviewed by Plastics Co. Laboratory for their input prior to publication.

Miscellaneous

Mr. John F. Sherman is retiring effective 3/1/78.

Mr. Filberto Garcia will transfer from the Plastics Co. to the Chemical Company Laboratory Analytical Group.

1977 short term cross section training efforts are paying dividends as evidenced by the absence of training overtime cost, during recent job rotations. The training program will continue this year.

Mr. Keith Arthur (Laboratory Specialist) is attending an in-Plant electronics training course provided by the Process Analyzer Group.

Mr. Bobby Briell resigned effective February 24, 1978. Marvin Harris transferred to the Area III Group to fill the vacancy on unit chemist assignments.

Mrs. Jeri McIntyre resigned effective February 10, 1978.

We are currently trying to fill a Chemist and Laboratory Secretary vacancy.

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Efficiency Improvements with Trace Amounts of Halogenated Hydrocarbons	Presley	2	Raw Material Savings	August, 1978
MO Catalyst Screening		2	To Establish Alternate Suppliers and Improve Catalyst Quality	Continuing
MO Catalyst Reuse		2	Reduce Silver Inventory	Undetermined
MS-II Crude to MO		1	Steam Savings	Undetermined
Reduce Methylal Formation in MO Distillation Columns		1	Raw Material Savings	Plant testing by April 31, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Purchase Specification Update	Smith	2	Maintenance of Business	May 1, 1978
BOD Analytical Methods Manual	Smith	2	Maintenance of Business	June 1, 1978
Anaerobic Pilot Testing of Chemical Waste Streams	Smith	1	To Test Anaerobic Process As Alternate BOD Expansion	January, 1, 1979

003207

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
OSHA monitoring	Jackson Pena	1	Ensure Employee Health and Safety OSHA Compliance	continuing

003208

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Studies to Remove Oxo T-210	Harris	2	Steam Savings	March, 1978
Long Term Formaldehyde Stability	Harris	1	Quality Control	March, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TMP Unit Emission	Mayerle	1	Ensure Compliance with Standard	April 1, 1978
Alternate TMP Purification Scheme	Mayerle	1	Energy and Raw Material Savings	June, 1978
TMP Unit Analytical Methods Manual	Mayerle	2	Maintenance of Business	May 1, 1978
Reduce Sulfates in PE AML	Mayerle	2	Comply with VCI Request	May 1, 1978
PE Unit Analytical Methods Manual	Mayerle	2	Maintenance of Business	May 1, 1978
Alternate N-butyl Acetate Process	Hilbrich	1	Steam Savings	May 1, 1978
Paraform, Formcel Products, Esters Unit Analytical Methods Manuals	Hilbrich Harris	2	Maintenance of Business	April, 1978
Control Reflux on Ester Unit Reactor Column	Harris	1	Steam Savings	May 1, 1978
1,3 - BG Unit Emissions Survey	Hancock	1	Ensure Compliance with Standard	March 15, 1978
DAA Unit Emissions Survey	Hancock	1	Ensure Compliance with Standards	March 15, 1978
DAA Laboratory Reactor Study	Hancock	2	Increase Unit Capacity and steam savings	April 1, 1978
Monitor T-295 Residue Acetone Loss	Hancock	2	Minimize Efficiency Loss	March 15, 1978

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Acetate Unit Emissions Survey	Unassigned (a)	1	Ensure Compliance with Standards	April 1, 1978
PE Unit Emissions Survey	Unassigned (a)	1	Ensure Compliance with Standard	April 1, 1978

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(a) This project was essentially completed by R. G. Briell prior to his resignation effective February 24. Final calculations and reports should be issued by April 1.

Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Fuel Gas Quality	Dow	3	Improve fuel Gas Accountability	Continuing
MS II Analytical Support	Dow	1	Maintenance of Business	Continuing
Crude Methanol to MO	Dow	2	Cost savings	March 1978
DME in Crude Methanol	Dow	2	Lurgi Catalyst Conversion	Continuing

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