



CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

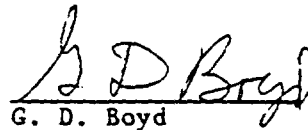
From: G. D. Boyd

GDB-186-82

July 9, 1982

LABORATORY DEPARTMENT SUMMARY - JUNE, 1982

Attached is the Laboratory's monthly summary for June. The summaries are by groups. A table listing priorities, justifications, and estimated completion dates of Special Problems projects is also included.


G. D. Boyd

GDB:erg

Distribution:

Chemical

J. J. Fritsch
D. G. Pausky
W. H. Meyer
A. A. Miller
G. R. Scholz
M. D. Harvey
G. L. Cade
C. W. Lee
D. D. Siemonsma
W. L. Sharp
D. E. Lockwood
L. M. Harvey
H. H. Thigpen
A. G. McGehee
B. C. Kerr
F. R. Dow
W. P. Hightower
R. R. Carpenter
R. S. Hahn
J. C. Rubiano
K. R. Stelts
W. H. Brough
Lab Chemists (20)

Plastics

A. C. Abshier
R. L. Bunkley
D. A. Reck
D. W. Steele
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
J. L. Paul, Clear Lake
D. W. Harris, Pampa
T. H. Golson, Dallas

File " 332 003706

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HIGHLIGHTS

- Both granular carbon and XAD-2 resin will remove Versene™ scum formers from MO-II HCHO. Studies are underway to determine whether beds can be regenerated.

- The effect of additives (sodium hydroxide or oxalic acid) on prill drying at 150°F is almost complete. Severe dusting occurs during fluid bed drying with no additive or with oxalic acid additive. Small amounts of sodium hydroxide (approximately 1 ppm) greatly reduce the dusting problem but reduce the drying rate. Addition of either oxalic acid or sodium hydroxide reduces the drying rate.

- The PE resin bed is on stream as of June 26. The bed is operating well.

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I. ANALYTICAL GROUP

Rhodium - Oxo

Rhodium analyses by AA were provided on numerous Run VI-ash samples to determine assay. This material was sent to Johnson-Matthey Inc. for a trial rhodium recovery.

Polyol Area

Trimethylolpropane (TMP) - The Analytical Group provided sodium analyses on samples from a Special Problems Group sodium formate back wash simulation of the TMP expansion. A report will be published detailing the results of the project.

Pentaerythritol (PE) - Incoming 50% NaOH is being analyzed for chloride content in an effort to determine the cause of corrosion in the Polyol Unit. All material to date has met purchase specifications of < 1000 ppm. The material will continue to be monitored periodically.

Pentaerythritol (PE) - Approximately 150 samples from three tech grade PE lots were analyzed for ash content. This was to segregate any material that was on test and to establish ash levels so that the remaining material could be blended on spec. with current production.

Customer Complaints

6-7-82-Shipley Co.-Alston, Mass.-complained of Para Flake receipts aging to an insoluble state too rapidly. No product returned-Customer will cut orders and inventory by 50% to solve problem.

6-9-82-Plastics Eng.-Sheboygan, Wis.-complained of low assay on HCHO tankcar receipt (51.2% vs Sales Spec. of 52.0 wt%). Original analysis shipped at 52.5%.

Quality Waivers

6-16-82 - Methyl Ethyl Ketone to Cone Solvents - Oklahoma City, Ok - 16470 lbs - H₂O at 0.13 wt.% vs. 0.10 max. Sales Spec. - SQD.

6-17-82 - Methyl Ethyl Ketone to Chem Central - San Antonio, TX - 19790 lbs. - H₂O @ 0.13 vs. 0.10 wt.% Max. sales spec. - SQD.

6-22-82 - Methyl Ethyl Ketone to Delta Solvents - St. Gabriel, La - 19500 lbs - H₂O @ 0.13 vs. 0.10 wt.% max. Sales Spec. - SQD.

6-25-82 - Pentaerythritol Tech. - St. Johns Chem Co. - Lake City, FL - 40,000 lbs - Ash @ 0.02 wt.% vs Max Sales Spec of 0.10 - SQD.

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PRODUCT QUALITY WAIVER HISTORY

	<u>% QD¹</u> <u>Month</u>	<u>% QD</u> <u>YTD</u>	<u>% SQD²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Methylal	0	44.4	0	0
PE-Pure	0	0	0	0.6
n-Propyl Acetate	0	0.3	0	0
Diacetone Alcohol	0	7.2	0	0
PE-Tech	0	0	2.0	0.5
Sodium Formate	0	0	0	5.9
MEK	0	0	80.0	10.1

¹ Lbs. of product shipped out of Manufacturing Control Specification vs. total lbs of that product shipped for month and YTD.

² Lbs. of product shipped out of Sales Specification vs. total lbs. of that product shipped for month and YTD.

Analytical Statistics

<u>Sample Count</u>	<u>May</u>	<u>May YTD</u>	<u>Previous YTD</u>
Chemical Section	5201	28,178	27,845
Instrument Section	2856	17,002	15,858
Total	8057	45,180	43,703

Technician Overtime

Chemical Section	50	329	432
Instrumental Section	60	323	214
Total	110	652	646

Sample Shipping

Domestic	53	371	363
Export	6	18	30
Plastics	0	4	7
Others	13	61	59
Total	72	454	459

QA Statistics

Shipments	609	3528	4007
Waivers (QD)	0	15	10
Waivers (SQD)	4	9	8
Complaints	2	12	5

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II. STAFF CHEMISTS

Process Technology

B. C. Kerr

Dow Scum Problem - A technical service call was made to Dow on June 22, 1982. Gary Boyd, Mr. Ron Edwards and Bob Kerr represented Celanese. A presentation of the work that had been done by Celanese to resolve the scum problem in Bishop formaldehyde was made. The plant agreed to incorporate a Laboratory EDTA preparation in the Special Customer requirements for Dow. A detailed report was issued (See BCK-101-82).

Lumps in Paraform Flake - Some of our customers for Paraform Flake are critical of lumps in Para flake. Laboratory Lump Count data for January - June, 1982 were examined for number distribution and the geometric mean number. The distribution closely approached a log normal distribution with a geometric mean of 8 lumps/lot. 95% of the lots have 46 or fewer lumps.

Formaldehyde Concentration Variability in 3rd Stage Product - Unit assay data on the Paraform 3rd Stage Product for the month of May has been examined to determine its variability. For the month of May, the HCHO assay had a daily average of 88.17% with a standard deviation of 0.45%. During this time period, the flake lots produced had an average assay of 92.04% with a standard deviation of + 0.73%.

Process Technology Manual - Final typing and proofing are in progress for the revised Process Technology Manual.

Special Analytical

F.R. Dow, B.W. Wise

TMP Purity - Analysis of Celanese TMP by capillary column GC continues to show a 98.5% purity. The unknown that elutes immediately after the mono-TMP varies in concentration from 0.2-1.0%. A material balance around V-747 flasher and T-205 finishing column has shown the unknown to be a light end in the TMP finishing system. The material balance samples showed that T-205 OH contained approximately 2% of the unknown compound and T-205 SS (product) contained approximately 0.2%.

A joint analytical effort with CCCTC is in progress to isolate and identify the impurities in current TMP production.

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Hydrocarbon Concentrations in MO-IV "B" Train Vaporizer - Samples of MO-IV "B" Train vaporizer were analyzed for hydrocarbon concentration. This material balance determined that 95% of the C₇-C₂₀ hydrocarbons from the crude methanol feed are entrained in the vaporizer overhead. The C₂₀+ hydrocarbons remain in the vaporizer and/or are removed by the vaporizer blowdown. A report has been issued detailing this data.

III. SPECIAL PROBLEMS

Group I - H.H. Thigpen

Oxo, Butanol T. A. Jasek

Pilot Study - H₂ PO₄ Extraction of Oxo Run VI Catalyst Solution (T.A. Jasek) - No progress. Further work on this study is pending and awaiting the outcome of the Johnson-Matthey trial recovery.

Oxo Catalyst to Reclaimers (Jasek) - The Laboratory conducted moisture determinations of a sample of Oxo catalyst ash from Johnson-Matthey, Inc (JMI). All attempts resulted in weight gains in all samples even when conducted at higher temperatures (130°C as opposed to 110°C), in a nitrogen-purged oven or a vacuum oven. The reason for this weight gain is not known. JMI did report a weight loss, but did not disclose a value.

The decision was made to analyze the sample "as is". The first results indicated a rhodium concentration of 4.57 wt%. This did not check with the 4.09 wt.% obtained by JMI. Investigation showed that the stock solution used for the rhodium standard was low by approximately 7%. Analysis are currently underway to determine a revised value for the rhodium concentration in the ash.

Heavy Ends Build in Oxo Reactor and Purification Train - The study to determine the heavy ends build rate in the butyraldehyde purification train has been completed. The data has been transmitted to Process Engineering and a report is being issued.

Cross-check Rhodium Analysis - The Bishop Laboratory was requested to analyze a sample of Oxo Reactor catalyst solution to cross-check the current method used for rhodium analysis by CCCTC. The data was reported.

Paraformaldehyde, Formcels®, Esters
J. E. Russell

Paraformaldehyde Prills - Prill drying studies continue. Sodium hydroxide reduces dusting attrition in a fluid bed dryer when used as an additive in the 1-10 ppm range. It also seems to decrease the drying rate.

Cloud times were determined for paraliquid that had been spiked with varying levels of NaOH and oxalic acid. The results showed that our 3rd stage product still has approximately the same cloud time that it has had for the past several months, approximately 95 min. Increasing the NaOH level decreases the cloud time. The cloud time reached zero at about 11-12 ppm Na. The cloud time reached a maximum of 106 min. with about 12.5 ppm oxalic acid. From there, the cloud time decreased progressively with increasing oxalic acid. Zero cloud time was reached at approximately 400-600 ppm oxalic acid.

An initial test of drying prills made with oxalic acid as the additive is inconclusive. The dusting appears to be more severe without an increase in drying rate. The lab will attempt to confirm this observation with further drying tests.

A sodium analysis for prills made with NaOH addition has been developed which should be relatively free from contamination from glassware. Likewise, a melting point analysis has also been developed. Both of these methods will be documented for Laboratory use.

OSHA
C. E. Gary, K. V. Parker

Noise Exposure Survey (Gary, Parker) - A noise exposure survey of the Chemical Maintenance and some ABI personnel has been completed. (Report in Prep.)

Formaldehyde Exposure Survey (Parker) - An initial formaldehyde exposure survey (after expansion) of MO-IV personnel is being obtained. Also, the 3M formaldehyde monitor badge will be evaluated (eight samples will be taken).

Formaldehyde Methods Validation (Gary) - The laboratory validation study of the Celanese and NIOSH chromatropic acid formaldehyde methods is 60% complete. Experiments to study the effect of varying sampling time have been completed. (The same results were obtained by both methods at 1, 2, 4, and 8 hour sampling times.) The Metronics Dynacalibrator output of 0.1 ppm was confirmed by analysis employing the CEA-555 instrument specific for formaldehyde.

Plans are underway to evaluate, if possible, the formaldehyde exposure caused by the pilot prilling operation. Initial surveys using Draeger tubes show a background concentration of 1-2 ppm HCHO around the prilling pilot plant area with our prevailing wind direction. Most of this appears to be from the open flaker drum and aging belt. Only the west flaker train was operating at the time of the initial surveys. The background level is likely to be higher when the East train is running. It is closer to and still up wind of the prilling area. The high levels of HCHO from the flakers and aging belts may make it impossible to measure the HCHO contribution of the prilling operation unless the winds are from the North or North East. Additional base case data will be obtained using sampling pumps (our normal personnel monitoring method).

Methanol Oxidation, Formaldehyde Blending
J.F. Glenister, P.W. Young

Summary of Product Color in the MO Units - (Glenister) -MO II and MO IV product colors were good at <5 APHA for entire month. MO III product was good up to the shutdown on 6/12/82. Production Color Quality has been good (<5 APHA) since the end of March, with no extended periods of higher color excursions. The T-185-OH and T-240 streams continue at MO II.

Analysis of Solids from MO IV Vent Blower - (Glenister) The Laboratory received for analysis a sample of a solid scalelike material from the MO IV Vent Blower. Operations had opened the fan on the blower to inspect for the cause of increased vibrations and discovered a coating of scale on the fan blades. A sample of the scale was ashed to determine the percentage volatile. The sample was approximately 44% non-volatile and 56% volatiles. The ash resulting was analyzed and found to be entirely iron oxide. A Total Organic Carbon analysis of the scale resulted in a TOC value of approximately 200 ppm. However, analysis of the sample for paraformaldehyde was very low. Subsequent ion-exchange chromatography analysis indicated high formate and phosphate levels. These results considered with the high iron content indicates the scale may be a mixture of iron formate and iron phosphate. Additional work to verify these results will be completed and a letter issued.

Analysis of Solids in MO IV Vaporizer Blowdown Line - (Glenister) Analysis of a mixture of solids (waxy and red particles) was requested by J. D. Presley to verify the red particles to be iron or copper. The Laboratory confirmed the red particles as iron. A qualitative analysis of the waxes present was done using capillary gas chromatography. This analysis indicated hydrocarbons ranging from C₁₅ through C₃₀. No further work planned.

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Removal of Impurities from Formaldehyde (Young) - The laboratory has tried silica gel, activated carbon and resins (Rohm and Haas IRA 938 and XAD-2) to remove impurities from scum-forming MO II formaldehyde (while the unit was receiving T-185 OH). The two best prospects for impurity removal are activated carbon and XAD-2 resin. The carbon outfall showed scum after 169 bed volumes with a feed of 36.88 wt.% formaldehyde. The XAD-2 outfall showed scum after 135 bedvolumes. The columns were _____ regenerated with cold methanol. The tests are being repeated on the regenerated columns to determine the degree of success of the regeneration.

Methanol Synthesis
J. F. Glenister

Analysis of Carbon Monoxide Levels Through M.S. Shift Section
(Glenister) - T. Tshatchula of Process Engineering requested analysis of the carbon monoxide levels throughout the M.S. Shift Section. Analysis was completed using Gas Chromatography on the following samples: (1) Before the low temperature shift, (2) After the low temperature shift, (3) After the high temperature shift, and (4) After the guard beds. A letter summarizing the results has been issued.

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Group II - L.M. Harvey

Waste Management

B. K. Huelle, K. R. Kaderlik

Deep well Injection of Solar Pond Liquids (Huelle) - Due to the successful removal of suspended solids from solar pond material using a coagulation treatment, a comparative study of a coagulation/clarification process vs. a precoat filtration process is in progress. Meanwhile, the coagulation/clarification process is being optimized.

A complete investigation of the effect of cationic, nonionic, and anionic polymers on coagulation is underway. Once the optimum polymer combination is found, settling-rate tests will be done at various mixing times. The apparatus has been constructed for these tests.

A lab-scale model of a coagulation/clarification process has been designed and will be used to determine what effect floc recycle and the addition of bentonite clay to the clarifier will have on TSS-removal efficiencies.

At present, the effectiveness of these solids-removal systems (precoat filtration and coagulation/clarification) is determined solely by their ability to reduce TSS levels in solar pond material. This does not address the question as to the suitability of the resulting pond material for deep well disposal. The best known way to determine suitability is through core permeability tests.

Past core tests by Underground Resource Management (URM) were done on a solar pond composite filtered to <0.45 micron which did not contain Pond 7B. Results indicated that plugging problems would occur unless the pond material was preceded by a brine solution. Further tests were conducted at URM this month. Pond 7B filtered to <0.45 micron caused a severe reduction in flow rate and probable core plugging. Since complete regeneration of the core material was never achieved, the following tests were somewhat questionable. Pond 11E filtered to <0.45 micron would not plug the core but would require twice the pressure needed to pump the brine solution. A 50:50 mixture of Equalization Basin:11E filtered to <0.45 micron would also be suitable for injection. A sample of 11E coagulated (floc removed after 1 hour) would plug the core. However, it is not known whether the plugging was due to remaining suspended solids or the coagulant.

Because these core-permeability tests are inconclusive, such tests will be performed in our Laboratory using samples from each of the solids removal processes. Core material has been obtained from the Bay City Plant.

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U.R.M. had also reported precipitation and gas evolution with 50:50 mixtures of solar pond and BOD equalization basin material. A once-a-week, ten-week study is underway using various combinations of BOD and solar pond to observe if gas evolution, pH changes or precipitation occur upon mixing. After 2 weeks none of the above have occurred.

Solar Pond Compatability Study (Kaderlik) - The solar pond compatability study has shown that mixing effluent from pond 7B with samples representing a cross-section of the other ponds resulted in the formation of an unknown precipitate. A report has been issued. (KRK-04-82)

TOD/COD Correlation of VCI Effluent (Kaderlik) - A study of the relationship between TOD and COD of VCI effluent was conducted during the period of April to June. The results allow approximate daily TOD poundage to be determined from VCI's COD analysis of their effluent. VCI will be responsible for controlling their effluent flow to BOD II based on daily TOD poundage values established by Celanese. A report has been issued. (KRK-04-82)

Methanol Synthesis
R.L. Day

Rapid Carbon Deactivation (Day) - A letter has been written documenting the investigation of the rapid deactivation of the carbon guard beds in the MS Unit.

Analyses of four spent carbon samples showed that two of the samples had activities comparable to new carbon, and two samples had activities slightly less than new carbon. Regeneration using solvent-extraction techniques in the Lab were unsuccessful at increasing the sulfur capacity of the carbon.

On May 20, Channel Gas opened up and put on-line several new, apparently "cleaner" wells. The sulfur levels in the methane feed have dropped considerably since that time, and the carbon guard beds have not had to be regenerated as often. The Laboratory will develop a method of analysis for sulfur compounds in incoming fuel gas.

In addition, maintenance on the coalescer April 15 may have reduced the liquid hydrocarbon carryover to the carbon guard beds. Process Engineering will follow the effect of this work on carbon life.

HCHO Stability
R. L. Day

A letter has been written documenting the variability seen in HCHO stability over a 6-month period. The letter will be issued in July.

003716

Trimethylolpropane
J.C. Bustabad

TMP Expansion (Bustabad) - Two-stage sodium formate (NaFo) backwashing from TMP was completed at 25°C using concentrated feed (15% TMP vs. typical 10%). With approximately 25% reduction in wash water rates (water/TMP), NaFo concentrations were the same (2-3 ppm) in the organic phase when using 15% TMP feed and 10% TMP feed. The concentrated feed wash showed a substantial increase in TMP in the aqueous phase.

When using 15% TMP feed at ambient temperature, wash-water reductions of $> 50\%$ in two-stage washes were found to be unfeasible. Wash water reductions of 50% showed (a) no phasing or (b) slow phasing and a substantial increase of NaFo concentration in the 2nd-stage organic phase. Wash water reductions of 65% showed no phasing.

A study of phasing times showed phasing times increased when using higher temperatures, more concentrated feed, and reduced wash water rates.

A report will be issued in July.

CCCTC Samples from Feed Preparation for TMP Melt Crystallization Study (Bustabad) - CCCTC requested analyses by the Bishop Laboratory of two samples from the preparation of feed for a TMP melt crystallization study. Bishop and CCCTC analyses for the residue sample (#30051-23-2) were within 1.9% absolute. CCCTC and Bishop analyses of the overhead sample (#30051-23-1) were significantly different for several components. Using the Bishop method accountability of the sample was increased from 79.3% (CCCTC method) to 90%.

A letter has been issued.

NaFO Analyses on T-204 Residue and V-747 Residue (Bustabad) - The specific-ion electrode method used for NaFo analyses in the unit lab has been found to be inadequate. Samples of T-204 residue and V-747 residue have been analyzed for NaFo successfully using a perchloric-acid potentiometric titration method. This method will be written and issued for use in the Unit Lab to replace the present specific-ion electrode method. In the interim, NaFo analyses are being provided by the Laboratory as needed.

Solids from Reboiler Tubes of T-286 (Bustabad) - Uniform-sized, disc-shaped solids were found in the reboiler tubes of T-286 during a general maintenance check of equipment. A sample was sent to CCCTC for analyses by x-ray diffraction and fluorescence. Results showed the

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solids to be primarily graphite. Since the conditions in T-286 or T-286 reboiler are not severe enough for the production of graphite, it is speculated that the solids are a result of larger pieces of graphite "wearing away" and being molded to the diameter of the reboiler tubes. The origin of the graphite pieces is unknown.

A letter was issued.

BOD, Utilities

D.R. Akin, K.R. Kaderlik, G.R. Stasney

Polyol Treatability (Akin, Kaderlik, Stasney) - This last month a study was begun on the anaerobic-aerobic system to determine if the anaerobic reactor could be seeded with facultative bacteria from the activated sludge system at BOD II. If these facultative bacteria were acclimated to Polyol effluent, theoretically they could convert to anaerobic bacteria which could degrade Polyol effluent. The anaerobic reactor's COD-removal efficiency and kinetic efficiency were observed closely for any increase due to an increase in the degradation of polyol effluent. The feed to the system consists of 27 wt% polyol effluent TOD and 73 wt% equalization-basin-liquid TOD with an organic loading of 0.29 lb COD/ft³/day. The feed also contained about 40 ppm of aerobic biomass. The kinetic efficiency has dropped slightly from about 60 to 50%, but the anaerobic removal efficiency has held constant at approximately 80%. The aerobic outfall COD ranges from 250-350 ppm COD with a 2.9-day detention time. This study will continue.

BOD II Unit Support (Akin) - The anaerobic reactor was operated at an average organic loading of 0.16 lb COD/ft³/day during June. Anaerobic COD removal efficiency ranged from 70-80%. The aerobic outfall COD averaged 193 ppm with an average detention time of 4.2 days. Both aeration basins were increased in capacity during June. Flows to the Unit during June were reduced during this construction. During June the unit also switched from sodium formate buffer to sodium hydroxide buffer for pH control. The Laboratory is providing support in the optimization of the amount of buffer added to the reactor.

Vacuum Truck Content Characterization (Akin) - This characterization has been completed and documented (DRA-026-82).

Chemical Oxygen Demand (COD), pH, and Volatile Fatty Acid (VFA) Profile of the Anaerobic Reactor (Akin) - On June 9, samples were collected from sample taps at varying heights on the anaerobic reactor for a COD, pH, and VFA profile. The COD profile of the reactor was basically the same as a previous profile (PWY-10-82). A report has been issued (DRA-27-82).

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VCI/Polyol Treatability (Akin, Kaderlik, Stasney)- One of the objectives of this study was to increase the Polyol effluent concentration in the feed stream to the highest level possible and still meet outfall specifications at normal operating rates. The polyol TOD contribution of the feed was increased to 28 wt.% (16650 lbs TOD/day - 4800 lbs TOD/day from equalization basin liquid, 11850 lbs TOD/day added). The remainder of the feed consisted of 5 wt.% VCI effluent TOD and 67 wt.% equalization basin liquid TOD. The organic loading remained at 0.32 lb COD/ft³/day throughout the study. The kinetic efficiency ranged from 45-50% with an anaerobic outfall COD of 2000-2500 ppm. The aerobic outfall COD for the study ranged from 450-600 ppm at a 2.7-day detention time. These anaerobic and aerobic outfall COD concentrations remained stable throughout the study. At present the components of the feed stream have been adjusted to 22 wt.% polyol effluent TOD (equates to 12,000 lbs TOD/day), 5.4 wt.% VCI effluent TOD (equates to 3000 lbs TOD/day), and 72.6 wt.% equalization basin (equals to 40,000 lbs TOD/day). As soon as the aerobic outfall stabilizes in the 200-400 ppm range the VCI effluent contribution to the feed stream will be increased.

Aerobic Digestion of Ultrasonically Ruptured Cells (Akin, Kaderlik) - A study has begun to determine if bacteria which have had their cell walls ruptured can be digested by aerobic bacteria at such a rate as to be feasible for full-scale operation. This study consists of a control tank which is fed a controlled amount of ruptured biomass each day. Both the control and the other tank are fed equalization basin liquid at a 2.7-day detention time. This study will continue.

Pentaerythritol
R. L. Day, K. R. Kaderlik

PE Resin Bed (Day)- The PE Unit resin bed for hydrolysis of formals came on-line June 26. Conversion of PE linear formal during Tech-grade production has been excellent despite initial problems with the acid-addition system. The Laboratory will follow the performance of the resin bed for the next several months. Future plans are to establish the relationship between pH of the feed to the resin bed and linear formal content of the resin bed product.

Separation of UV Absorber from PE (Kaderlik) -Samples of high UV absorbing PE were eluted through an XAD-2 resin to separate out the UV absorbing contaminate. Efforts will now be made to isolate and characterize the contaminate.

Yellow lumps in Pure PE (Day) - Inolex Co., (Philadelphia, PA) complained of yellow particles in 3 lots of Pure PE. Investigation of approximately 30 retain samples, dating back to April, 1981, showed very few samples with yellow particles but a slight increase in the yellow particles in recent months.

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Yellow particles examined under a microscope were much smaller than PE crystals. The yellow particles are thought to be made in the dryer. Work will continue as more yellow particles are isolated.

General

- Jim Russell attended a para prill meeting at the Tech Center on June 25.
- Hub Thigpen attended a para prill meeting at the Tech Center on June 11.
- Belinda Huelle performed core-permeability tests on solar pond liquids at Underground Resource Management in Austin, Texas on June 9, 10, and 11.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Glenister	2	To Establish Alternate Catalyst	Continuing
GC Methanol Oxidation Reactor Optimization		2	Catalyst Evaluation for Possible Efficiency Improvements	November, 1982
Formaldehyde Color Problem		1	MOB	Continuing
Confirm MO Unit Efficiency Losses Via Vent Sampling and Anal.		1	MOB, CR	August, 1982
Stability of 37% Unstabilized HCHO (for Dow Chemical Co.)		1	MOB	September, 1982
Oxo Run VI Cleanup (H_3PO_4 extraction-wash)	Jasek	1	Cost Reduction	July, 1982
Rhodium Accounting		1	MOB, CR	Continuing
Rhodium Reclaiming by Johnson-Matthey		1	CR	August, 1982
Support Paraform Prill Pilot Operation at Bishop	Russell	1	CR, MOB	September, 1982
Support Flocculant Plant Trial	Kaderlik	2	MOB	October, 1982
TMP Expansion	Bustabad	1	MOB	December, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
TMP Extractor Optimization	Bustabad	1	Raw Materials Savings	July, 1982
Condensing Polyol Vents		2	Cost Reduction	July, 1982
Update TMP Unit Methods Manual		2	MOB	August, 1982
Optimize Solar Pond Filtration Prior to Deep-Well Injection	Huelle	1	WMSTF Support	July, 1982
Recover TMP from Formals in TMP Heavy Ends (V-747 Residue)		1	Raw Materials Savings	July, 1982
Evaluate Alternate Filter Cloths for PE filters		2	MOB	October, 1982
Improve PE Quality - UV Absorbance and Formal Content	Huelle/ Kaderlik	1	MOB	July, 1982
TPP P ₂ O ₅ Emissions	Kaderlik	1	Emissions Quantification	July, 1982
Evaluate "Unsteady-state" Polyol VCI and other effluents	Akin/ Kaderlik	1	MOB	September, 1982
Aerobic Degradation of Ruptured Sludge	Akin/ Kaderlik	1	MOB	August, 1982
BOD II Aerobic Optimization	Akin	1	MOB	August, 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Evaluate ATP, OUR and Dehydrogenase Methods	Akin	2	MOB	July, 1982
Methanol Personal Exposure Monitoring	Gary	1	MOB (Environmental Health)	September, 1982
Area Surveys for Misc. Organics		2	MOB (Environmental Health)	February, 1983
Industrial Health Formaldehyde Methods Validation		1	MOB (Environmental Health)	July, 1982
Maintenance Noise Exposure Monitoring		1	MOB (Environmental Health)	June, 1982
Recover PE from VCI Waste Streams	Day	1	Cost Reduction	September, 1982
Determine Cause of MS Carbon Bed Deactivation		1	MOB	June, 1982
Formaldehyde Stability		2	MOB	June, 1982
Follow Performance of PE Resin Bed		1	MOB	Continuing
Update PE Unit Methods Manual		2	MOB	September, 1982
Removal of Impurities from Formaldehyde	Young	1		July, 1982

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CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: Dr. R. R. Graham

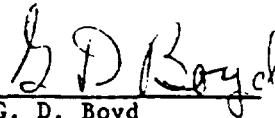
From: G. D. Boyd

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August 6, 1982

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Attached is the Laboratory's monthly summary for July. The summaries are by groups. A table listing priorities, justifications, and estimated completion dates of Special Problems projects is also included.


G. D. Boyd

GDB:erg

Distribution:

Chemical

J. J. Fritsch
D. G. Pausky
W. H. Meyer
A. A. Miller
G. R. Scholz
M. D. Harvey
G. L. Cade
C. W. Lee
D. D. Siemonsma
W. L. Sharp
D. E. Lockwood
L. M. Harvey
H. H. Thigpen
A. G. McGehee
B. C. Kerr
F. R. Dow
W. P. Hightower
R. R. Carpenter
R. S. Hahn
J. C. Rubiano
K. R. Stelts
W. H. Brough
Lab Chemists (20)

Plastics

A. C. Abshier
D. A. Reck
D. W. Steele
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
J. L. Paul, Clear Lake
D. W. Harris, Pampa
T. H. Golsom, Dallas

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HIGHLIGHTS

- "Scum" formers in HCHO from a MO Unit with T-185 OH in the feed can be removed by either a carbon bed or XAD-2 resin bed. Either bed can be regenerated with methanol.
- Seeding the anaerobic reactor with "polyol-acclimated" aerobic bacteria did not result in an increase in the kinetic efficiency of the anaerobic reactor.
- Aerobic digestion appears feasible for disposal of sludge after the waste bacteria have had their cell walls ruptured ultrasonically.
- The performance of the PE resin bed has been inhibited by high sodium formate levels (>1000 ppm) in the feed to the bed. Unsatisfactory filtering on "A" belt (due to problems with the vacuum system) and erratic pH control are the major problems.

003725

I. ANALYTICAL GROUP

Methanol Oxidation (MO) Formaldehyde (HCHO)

Extensive analytical assistance was provided to efforts to prepare and ship two 52% HCHO loads. Unit product concentration, high acid and resin bed color throw problems resulted in delayed shipping. Shipments were made after filtering to remove final color.

In-line blender problems resulted in missed 37% HCHO blends in 10 of 17 trailers (60%) and 7 of 35 tankcars (20%) this month. No high assay blends were missed. Each missed blend results in extensive resampling, reblending and shipment delay. Reblended shipments are made based solely on calculated quality as no shipping vessel mixing facilities are available to facilitate final blend sampling. This leaves us vulnerable to quality complaints. In-line blender problems have increased the HCHO-shipping analytical work load by approximately 30%.

MO-IV production exhibited cloudy product with high particle concentration throughout the month. All HCHO inventory tanks, preblend tanks and Formcel® production have been affected. Product filtering is required to make shipment.

Polyol Area - Pentaerythritol (PE)

Analytical and overtime coverage was provided for start-up of the PE Unit resin bed hydrolysis system.

Customer Complaints

7-12-82 - Mobay Chemical Company - Natrium, W. Va. complained of receipt of a molten TMP trailer shipment at 0.10 wt.% water. Before and after tank analysis and trailer shipment retain sample show 0.03 wt.%. The load was replaced with same level product and accepted. This is the first complaint from this customer after switching his critical end-use from flake to molten TMP in late 1979. We suspect a temporary analytical problem at the customer end.

Quality Waivers

7-20-82 - PVB Grade n-Butyraldehyde - to Monsanto Chemical Company at Indian Orchard, Mass. - 192,540 lbs. - H₂O at 0.14 wt.% vs. max sales spec. of 0.10 wt.% - SQD.

7-21-82 - PVB Grade n-Butyraldehyde - to Monsanto Chemical Company at Trenton, Mich. - 189,670 lbs. - H₂O at 0.14 wt.% vs. max sales spec. of 0.10 wt.% - SQD.

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PRODUCT QUALITY WAIVER HISTORY

	<u>% QD¹</u> <u>Month</u>	<u>% QD</u> <u>YTD</u>	<u>% SQD²</u> <u>Month</u>	<u>% SQD</u> <u>YTD</u>
Methylal	0	40.5	0	0
PE-Pure	0	0	0	0.5
n-Propyl Acetate	0	0.3	0	0
Diacetone Alcohol	0	6.4	0	0
PE-Tech	0	0	0.	0.4
Sodium Formate	0	0	0	4.5
MEK	0	0	0	8.9
PVB n-BuH	0	0	25.5	2.4

- ¹ Lbs. of product shipped out of Manufacturing Control Specification vs. total lbs of that product shipped for month and YTD.
- ² Lbs. of product shipped out of Sales Specification vs. total lbs. of that product shipped for month and YTD.

Analytical Statistics

<u>Sample Count</u>	<u>July</u>	<u>July YTD</u>	<u>Previous YTD</u>
Chemical Section	5117	33,295	31,811
Instrument Section	2717	19,719	18,876
Total	7834	53,014	50,687

Technician Overtime

Chemical Section	79	408	460
Instrumental Section	61	384	260
Total	140	792	720

Sample Shipping

Domestic	42	413	438
Export	2	20	36
Plastics	1	5	9
Others	9	70	66
Total	54	508	549

QA Statistics

Shipments	505	4033	4604
Waivers (QD)	0	15	15
Waivers (SQD)	2	11	8
Complaints	1	13	9

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II. STAFF CHEMISTS

Special Analytical F.R. Dow, B.W. Wise

Hydrocarbon Concentration in MO-IV "B" Train Vapor (F. R. Dow, B. W. Wise) - A second set of samples from MO-IV "B" Train Vaporizer was obtained to confirm C₆-C₂₀ hydrocarbon entrainment in the vaporizer overhead as reported in June, 1982.

The second profile included a cross-section profile of the vaporizer that showed a heavy wax concentration on both ends of the vaporizer, but little or no floating waxes were present on samples from the middle of the vaporizer.

Analyses again showed that the C₆-C₁₅ hydrocarbons (there were no C₁₆-C₂₀ hydrocarbons in the feed) were removed in the vaporizer overhead and all others remained in the vaporizer liquid. The C₁-C₅ alcohols were also removed in the vaporizer overhead.

A report will be issued detailing this data.

MO-III Color Problem (F. R. Dow, J. A. Glenister) - During mid-July, product color problems were experienced by MO-III. Analysis of T-313 (formaldehyde distillation column) has shown that the top half of the column contains more than 100 different compounds, most of which are foreign to the MO process. During the period of maximum product color, T-185 OH (weak formaldehyde recovery column) was being fed to MO-III. T-185 OH was removed from MO-III feed and after several days the MO product color returned to a normal APHA color of approximately 5.

Tower profile samples of T-313 were taken during and after the product excursion. During the color problem period, tower profile samples of T-313 showed a yellow color in samples from the top half of the column, however after T-185 OH was removed from the feed the top profile samples were clear.

Analysis is continuing in an attempt to identify the yellow colored compound(s).

003728

III. SPECIAL PROBLEMS

Group I - H.H. Thigpen

Oxo, Butanol
T. A. Jasek

Pilot Study - H_3PO_4 Extraction of Oxo Run VI Catalyst Solution
(T.A. Jasek) - Further work has been curtailed awaiting a decision on the possible reclaimer of Run VI. All the catalyst solution used in the lab experiments has been drummed and will be returned to the Oxo Unit. The extraction pilot unit will remain in place should additional pilot work be required.

Oxo Catalyst to Reclaimers (Jasek) - The Laboratory completed the reanalysis of the Oxo catalyst ash from Johnson-Matthey, Inc (JMI). The ash was analyzed in an "as is" condition. Reanalysis with the new, corrected standards indicated a rhodium concentration of 3.88 wt.%. This compares with the initial JMI concentration of 4.09 wt.%. The new analytical results were transmitted to Dallas.

Improved Rhodium Recovery from the Oxo Catalyst Solution (Jasek) - A preliminary study is underway to determine if a different treatment of the Oxo catalyst solution can increase the return of rhodium from the catalyst solution sent to reclaimers. Initial results indicate that if the catalyst solution is mixed with fine aluminum powder and then ashed, a more complete ashing occurs with apparent reduction in the physical loss of the catalyst solution. Studies have not been completed to determine the best way to remove the aluminum or if any of the rhodium was lost during ashing.

i-Butyraldehyde in Crude Butyraldehyde from Bay City (Jasek) - The Laboratory checked some of the incoming tank trailers of crude butyraldehyde from Bay City for i-butyraldehyde (i-BuH) content. Also, Tk 379, containing several shipments, was analyzed. The Bishop analysis indicated an i-BuH content of 8.72-8.76 wt.%. This compares with Bay City's analytical results of 8.4 wt.% i-BuH.

Paraformaldehyde, Formcels®, Esters
J. E. Russell

Paraformaldehyde Prills - Prill drying studies in the Laboratory are nearly complete. The drying rate increased slightly with oxalic acid addition, but dusting and losses to the drying air (attrition) increased to about 70% in the fluid bed dryer. Under gentler conditions in the roto-vap, attrition losses were as high as 28% with oxalic addition.

A test to simulate bagging, transportation, and handling attrition is being developed. The screening apparatus for particle size distribution tests is being used. Hopefully, the new test will allow an attrition comparison between Celanese prills and competitor's prills. A more severe test may be required to get meaningful data.

The free-flowing bulk density of Laboratory-produced prills was measured. The results show that a wide variation in prill bulk density exists, ranging from 31 lb/ft³ to 54 lb/ft³. Out of 8 samples, six fell within the range 46 to 54 lb/ft³. The variability in bulk density is similar to Canadian experience. Hopefully, Unit-produced prills will show more uniformity in bulk density.

OSHA
C. E. Gary, K. V. Parker

Noise Exposure Survey (Parker, Gary) - The completion of the personal noise exposure of Maintenance personnel finished the noise exposure sampling for 1982. (CEG-64-82, KVP-19-82)

Formaldehyde Exposure (Parker, Gary) - The 3M Formaldehyde diffusion type monitor badge was evaluated against the Celanese CA-HCHO method at 0.1 - 0.3 ppm formaldehyde personal exposure. The results show the badge to be acceptable in this range. (CEG-66-82, KVP-20-82)

Preliminary data on area formaldehyde exposure levels have been obtained in the Paraform Unit prior to the Prilling Unit start-up.

Follow-up Formaldehyde Exposure (Parker) - Sampling has begun to determine the effect of the Phase I formaldehyde reduction program in the areas where the work is complete.

Formaldehyde Methods Validation (Gary) - The three new permeation tubes for generating dynamic formaldehyde gas standards were found to be accurate. Work has resumed on this project which is approximately 60% complete.

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Asbestos Exposure (Gary) - A report has been issued indicating no asbestos exposure problems occur when ABI personnel are working at the asbestos removal pit. (CEG-65-82)

Methanol Oxidation, Formaldehyde Blending
J. F. Glenister, P. W. Young

Air to Methanol Ratios (Glenister) -

MO-III Start-up "A" Train June 30th

<u>Indicated</u>	<u>Analytical</u>
0.880 - 0.900	0.850
0.880 - 0.900	0.864
0.880 - 0.900	0.853

MO-II Start-up July 20th

<u>Indicated</u>	<u>Analytical</u>
0.903 - 0.905	0.780
0.903 - 0.905	0.770
0.903 - 0.905	0.770

Summary of Product Color in MO Units (Glenister) - MO-II (T-297)
production showed a period of "moderate" color early in July with colors trending at 6-8 APHA. The Unit production improved and the HCHO color was <5 when the Unit shut down on July 13th. (The T-185 WFRU waste stream was transferred to MO-III on MO-II shutdown.) MO-II was returned to production on July 21st with an initial product color of <5 APHA. However, MO-II product color increased from a <5 APHA to a 18 APHA when T-185 was fed to the absorber following the high blend production on July 24th.

MO-III (T-313) product color on startup was moderate with 2 days of 6-7 APHA color. Product color was good at <5 APHA until July 12th when the product color increased to 20-25 APHA. This sudden increase in product color correlates with the transfer of the WFRU T-185 OH stream from MO-II to MO-III during the MO-II shutdown. The unit also experienced higher formic acid levels (0.08 - 0.10 wt.%) in the product coincidentally with increased color. The T-185 OH stream was removed from MO-III on July 14th to prepare the Unit for high blend production. Product color improved to <5 APHA by July 18, 19th, but the product acid levels remained above normal at 0.08 - 0.09 wt.%.

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MO-IV product color continued good at <5 APHA.

High Color in MO-III Product July 12-18th (Glenister) - MO-III product color increased significantly to 20-25 APHA following the transfer of the T-185 OH stream to T-314 on July 11th. The Laboratory monitored the product color and attempts were initiated to isolate the colored compound(s). A yellow colored mixture was extracted from a product filter taken from MO-III during the period T-185 OH was being fed to the Unit. The mixture was extracted in chloroform and is being analyzed by F. R. Dow using Capillary Column Gas Chromatography and Liquid Chromatography. A series of samples of the distillation tower, showed the presence of color at Trays 28 and 45 with the T-185 OH stream and the absence of color without the T-185 OH stream.

Color Removal by Carbon or XAD-2 Resin (Young) - Colored formaldehyde (Color = 17 to 24 APHA) from MO-III (while T-185OH was being fed to the unit) was fed to an activated carbon bed and to a resin bed (Rohm and Haas XAD-2) in an attempt to reduce the color. The carbon bed reduced the color from 17 - 24 to 5 - 7. There was a color breakthrough (color = 13 - 17) after 240 bed volumes. Regeneration with methanol was unsuccessful (Outfall color = 13 - 15).

The XAD-2 resin gave a color breakthrough (Color = 13) after only 48 bed volumes.

Removal of Formaldehyde Impurities (Young) - Scum-forming impurities have been removed from 37 wt.% MO-II formaldehyde (while T-185OH was being fed to the Unit) by beds of activated carbon and Rohm and Haas XAD-2 resin. A feed that formed heavy scum during the EDTA-sodium salt lab preparation was fed to each bed. No scum was visible in the outfall from either bed for 120-150 bed volumes. Upon regeneration with methanol, both beds appeared to maintain the 120-150 bed volume capacity.

Another carbon bed was fed 50 wt.% MO-III colored formaldehyde (while T-185 OH was being fed to the Unit). This feed made moderate scum in the EDTA-sodium salt lab preparation. No scum was visible in the outfall for 130-160 bed volumes. The bed was regenerated with methanol. The success of this regeneration is still being investigated.

003732

Color in High Blend Tank 2484 (Glenister) - The high blend formaldehyde in Tank 2484 exhibited a color building trend during storage. Attempts to make a high blend from MO-III had shown color in the product downstream of the anion resin beds. The MO-III-HCHO had a high acid content and it was believed the acid was removing the iron absorbed on the resin beds and causing a color build in storage. Operations made two attempts to make the high blend at MO-III, but experienced poor quality downstream of the anions due to color. A high blend was made from MO-II during July 21st and 22nd. The MO-II product had a much lower acid content (0.046 - 0.050 wt.%). The routine laboratory provided analytical support in monitoring the assay and color of Unit production and also the resin-treated product. Product color remained <5 following resin treatment compared with the color present in the MO-III high blend product. However, an increase in product color occurred in 2484 following a 24-hour storage time. The color could be completely removed by filtering through a 0.45 μ m disc filter. A known amount of HCHO was filtered to remove the color and the filters were washed clean with concentrated hydrochloric acid. Analysis of the washings showed 0.2 ppm iron was present in the colored formaldehyde. Additional studies will be conducted to determine the cause of the iron contamination through the resin beds.

Filtration tests showed the color in 2484 could be removed by filtration through a filter paper precoated with the dicalite filter aid used to filter the product on loading. The Laboratory recommended increasing the amount of filter aid used to coat the loading filter and this method was successful in reducing the 17 APHA in 2484 to 5 APHA on loading of a high blend shipment.

Group II - L. M. Harvey

BOD, Utilities

D. R. Akin, K. R. Kaderlik, G. R. Stasney

BOD II Unit Support (Akin) - The anaerobic reactor was operated at an average organic loading of 0.17 lb COD/ft³/day during July. Anaerobic COD-removal efficiency ranged from 65-72%. The aerobic outfall COD averaged 165 ppm with an average detention time of 5 days.

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Polyol Treatability (Akin, Kaderlik, Stasney) - The purpose of the final stage of this study was to determine if aerobic biomass (Facultative bacteria) acclimated to polyol effluent could seed an anaerobic reactor. There was no increase in kinetic efficiency or anaerobic COD-removal efficiency during the course of the study. Kinetic efficiency dropped from approximately 60% to approximately 50%, but the anaerobic COD-removal efficiency remained about 80%. This study has been discontinued and the reactor has been shutdown.

VCI/Polyol Treatability (Akin, Kaderlik, Stasney) - In an effort to stabilize aerobic outfall COD concentrations to within the 200-400 ppm range, the addition of polyol and VCI effluents to the anaerobic feed have been discontinued. Thus the anaerobic unit is being fed 100% equalization-basin liquid at an organic loading of approximately 0.115 lb COD/ft³/day. The kinetic efficiency has ranged from 36-44% with an anaerobic outfall COD of 1100-2000 ppm. The aerobic outfall COD has ranged from 500-1000 ppm with a 5-day detention time. Plans are to conduct a pulse test on the reactor to determine liquid void volume. Additional polyol and VCI effluent will be reintroduced as soon as aerobic outfall COD stabilizes in the 200-400 ppm range.

Aerobic Digestion of Ultrasonically Ruptured Cells (Akin, Kaderlik, Stasney) - This study was initiated to determine if bacteria whose cell walls had been ruptured could be digested by aerobic bacteria at such a rate as to be feasible for full-scale operation. The first stage of the study consists of a control tank containing biomass which are in a state of endogenous respiration and another tank containing biomass which are being fed a regulated amount of ruptured bacteria. Results thus far indicate a higher VSS and higher OUR/MLVSS for the experimental tank which would indicate a higher activity level there than in the control tank. These results indicate that the ruptured bacteria are being digested by the aerobic bacteria. To determine the feasibility for full-scale operation, a second stage of the study will soon be initiated. In this stage both the control and the experimental tanks will be fed equal amounts of anaerobic recycle in addition to the ruptured biomass fed to the experimental tank.

Elimination of Zeolite Softening from the Treated Water Process (Kaderlik) - A laboratory study has been undertaken to investigate the feasibility of eliminating the sodium zeolite softeners from the treated water area.

Waste Management
B. K. Huelle

Solar Pond Solids Removal Studies (Huelle) - Laboratory data indicates that the gravimetric analysis for total suspended solids (TSS) at the 1 μ m level is not accurate. In addition, the colloidal nature of the suspended solids will tend to stop flow through the pores in 1 μ m glass fiber filter paper (colloidal particles are 0.001 to 0.01 μ m in size). Because of this analytical problem, data from long-tube settling tests was not meaningful.

In order to overcome the problem, alternative methods of comparing three feasible solids-removal systems [precoat filtration, flocculation/clarification, and ultrafiltration (UF)] were pursued. After discussions with manufacturing representatives for clarifiers, ultrafiltration equipment and cartridge filters, only two alternatives appear feasible.

Measuring the ease of filtration of the product from each system through 0.45 μ m filter paper will give some idea of solids content. While precoat and flocculation products plug a 0.45 μ m filter after 6 and 5.5 mls respectively, over 400 mls of UF product pass through without plugging.

Another possible comparative method is differential turbidimeter readings. Turbidity had previously been abandoned as an analytical tool since the 90°-reflectance turbidimeter used was not accurate for colored samples.

Turbidity, not TSS, is the analysis used by the clarifier manufacturer in determining settling rates. Whether the differential turbidimeter provides an accurate measurement of solids removal has not yet been determined.

Analysis of the UF product by the TSS method shows 30-60 ppm in the product at the 1 μ m level. This is further evidence of this analytical method since UF efficiently removes particles much smaller than 1 μ m. The UF product is clear and slightly colored while precoat and flocculation products are turbid and highly colored. COD and NaFo levels were not significantly changed in comparison to the feed, indicating the improvement in product appearance is due to removal of colloidal particles.

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Core-permeability tests show that products from the flocculation or precoat filtration of pond 11E are chemically compatible with the core matrix. (These products are filtered through 0.45 μ m filter paper before being introduced into the core.) Tests on UF product and Pond 7B are planned. More reliable core-testing equipment will arrive in early August. The Lab-scale clarifier is operating to test the effects of a Lamella and/or BOD-sludge blanket. Overflow from this clarifier will be sent to a cartridge-filter manufacturer for testing.

Pentaerythritol

D. R. Akin, J. C. Bustabad, R. L. Day, K. R. Kaderlik

PE Hydrolysis Resin Bed Support (Akin, Day) - The Laboratory continues to support Operations with the operation of the PE hydrolysis resin bed. Numerous problems have been encountered which are inhibiting the efficient conversion of linear formals across the resin bed.

For efficient conversion of linear formals, the PE stream to the resin bed must have sodium formate (NaFo) levels less than 1000 ppm. NaFo levels have consistently run higher than 1000 ppm since the resin bed came on-line June 26. The sodium problem has been traced to the PE 'A' belt operation. Work will continue on ways to optimize the 'A' belt vacuum system and crystallizer drop temperature. Another problem is the erratic operation of the pH control which dictates the amount of acid fed to PE stream before the resin bed. The erratic operation of the pH control causes the acid flow to vary to the PE stream. The H₂SO₄ stinger has also plugged on a few occasions allowing PE feed to enter the resin bed with no acid.

With relatively low sodium levels in the feed to the bed, the linear formal conversion has exceeded 90% at low H₂SO₄-usage levels. The Laboratory will continue to identify operating conditions necessary for the production of Dow-grade product with linear formal concentrations less than 0.15 wt.%.

VCI analysis has shown Na₂SO₄ levels in the AML to be less than 0.6% on an absolute basis (less than 2% on a contained-salt basis) since the resin bed has been on-line. The sulfate levels are less than 0.5 wt.% on a contained-salt basis whenever NaFo levels of the feed to the resin bed remain below 1000 ppm.

Trapped Material on PE Resin Screen (Kaderlik) - Material consisting of solids and fibers was found trapped on the screens located at the base of the PE resin bed. Efforts are being made to identify these substances and determine their origin.

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Separation of UV Absorber from PE (Kaderlik) - Solutions of high UV-absorbing PE were passed through an XAD-2 resin bed to separate out the UV-absorbing contaminate. Efforts are being made to isolate and characterize the contaminate.

Sodium Sulfate Turbidimetric Analyses (Bustabad) - A standard curve was established for Na_2SO_4 analyses in AML samples. The Na_2SO_4 method is being rewritten.

Trimethylolpropane
J. C. Bustabad

TMP Expansion - A report was issued on the laboratory two-stage NaFo backwashing from TMP.

Process Engineering requested data to determine if it would be necessary to include static mixers in the heat exchanger upstream from the reactor (T-207) in the TMP expansion design. Laboratory work consisted of mixing 25% formaldehyde, BuH, and 20% caustic (79.9%, 4.9%, 15.2%, respectively) at 40°C. Phasing, if any, was minuscule. Thus, the static mixers are unnecessary.

NaFo Analyses on T-204 Residue and V-747 Residue - The specific-ion electrode method used in the Unit Lab for analyzing NaFo in T-204 and V-747 residues is giving erroneous NaFo concentrations. V-747 residue was previously analyzed adequately in the Laboratory by a perchloric-acid titration method. The perchloric-acid titration method, though thought to be adequate for T-204 residue, has been shown to be inaccurate. Atomic absorption (AA) and Dionex Ion-chromatography methods showed the concentration of NaFo in T-204 residue to be 52-175 ppm NaFo. Work is in progress to find a reliable method that can be used in the Unit Lab. Efforts will then be made to adapt the method for use in the PE Unit Lab analysis of the Dissolve Tank (feed to the resin bed).

General

- J. E. Russell and H. H. Thigpen attended a Paraform Prill meeting at the Tech Center on July 24.
- L. M. Harvey and B. K. Huelle visited Membrane Systems, Inc. in Corpus Christi on July 21 to observe a demonstration of ultra-filtration of Chemcel solar-pond liquid.

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
MO Catalyst Screening	Glenister	2	To Establish Alternate Catalyst	Continuing
GC Methanol Oxidation Reactor Optimization	↓	2	Catalyst Evaluation for Possible Efficiency Improvements	November 1982
Formaldehyde Color Problem	↓	1	MOB	Continuing
Confirm MO Unit Efficiency Losses Via Vent Sampling and Analysis	↓	1	MOB, CR	August 1982
Stability of 37% Unstabilized HCHO (for Dow Chemical Co.)	↗	1	MOB	September 1982
Oxo Run VI Cleanup (H_3PO_4 extraction-wash)	Jasek	1	Cost Reduction	July 1982
Rhodium Accounting	↓	1	MOB, CR	Continuing
Rhodium Reclaiming by Johnson-Matthey Inc.	↗	1	CR	August 1982
Support Paraform Prill Pilot Operation At Bishop	Russell	1	CR, MOB	September 1982
TMP Expansion - Purification Train Simulation	Bustabad	1	MOB	December 1982
Evaluate Alternate Catalyst for 1,3-BG Hydrogenation	↗	1	MOB	September 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Condensing Polyol Vents	Bustabad	2	Cost Reduction	October 1982
Update TMP Unit Methods Manual	↓	2	MOB	August 1982
Optimize Solar Pond Filtration Prior to Deep-Well Injection	Huelle	1	WMSIF Support	July 1982
Recover TMP from Formals in TMP Heavy Ends (V-747 Residue)	↓	2	Raw Materials Savings	July 1982
Evaluate Alternate Filter Cloths for PE filters	↓	2	MOB	October 1982
Improve PE Quality - UV Absorbance and Formal Content	Huelle/ Kaderlik	1	MOB	July 1982
TPP, P ₂ O ₅ Emissions	Kaderlik	1	Emissions Quantification	July 1982
Evaluate "Unsteady-state" Polyol, VCI and other effluents	Akin/ Kaderlik	1	MOB	September 1982
Aerobic Degradation of Ruptured Sludge.	Akin/ Kaderlik	1	MOB	August 1982
BOD II Aerobic Optimization	Akin	1	MOB	October 1982
Evaluate ATP, OUR and Dehydrogenase Methods	↓	2	MOB	August 1982

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Current Special Problems Projects

Project	Chemist	Priority	Justification	Estimated Completion Date
Follow Performance of PE Resin Bed	Akin/Day	1	MOB	Continuing
Recover PE from VCI Waste Stream	Day	1	Cost Reduction	September 1982
Determine Cause of MS Carbon Bed Deactivation		1	MOB	June 1982
Formaldehyde Stability		2	MOB	June 1982
Update PE Unit Methods Manual	↘	2	MOB	September 1982
Methanol Personal Exposure Monitoring	Gary	1	MOB (Environmental Health)	September 1982
Area Surveys for Misc. Organics		2	MOB (Environmental Health)	February 1983
Industrial Health Formaldehyde Methods Validation	↘	1	MOB (Environmental Health)	July 1982
Maintenance Noise Exposure Monitoring	↘	1	MOB (Environmental Health)	June, 1982
Removal of Impurities from Formaldehyde	Young	1		July 1982

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