

CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: P. J. Volpe

From: W. H. Meyer

WHM-86-76

October 13, 1976

Laboratory Department Summary
September, 1976

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Laboratory Department Summary
September, 1976

The status of Chemical Company-related projects active during September are described below. Analytical statistics, current project priorities, and a list of reports in progress are attached.

PROCESS

MO Efficiency Improvement with
Trace Amounts of Halogenated Hydrocarbons - Branecky

Experimental work with boron trifluoride (BF_3) indicates it has potential for increasing reaction efficiency in MO. A test in which BF_3 gas was injected directly into the feed system of the micro unit revealed efficiency increase of 6.5% (83 to 89.5) and conversion increase of 5% (58 to 63). Due to difficulties in handling the injection on the extremely small micro unit scale, the concentration of BF_3 fed is not known accurately, but is estimated to be between 50 and 100 ppm. Several new effects were observed in this test. The addition of BF_3 was found to: (1) lower bed temperature significantly, (2) increase hydrogen make, (3) decrease CO_2 and CO make.

The data, when applied to the CCCTC MO REDOX Balance computer program, indicates an increase in dehydrogenation of methanol. The observed temperature drop agrees favorably with computer calculations which are based on reactor product distributions.

In further testing, the BF_3 was added in the methanol as the BF_3 -butyl ether adduct. At 50 ppm BF_3 , 3% efficiency and 5% conversion increase was observed. At 200 ppm BF_3 , the reactor product distribution was altered drastically, but the overall effect was constant efficiency (CO and CO_2 decrease, MeFo increase). This

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indicates a great potential for higher conversions through higher AMR's at a constant efficiency.

Testing will continue as time permits.

MO New Catalyst Screening - Branecky

Samples of silver foam metal have been received and evaluations are planned in the near future.

A sample of Matthey-Bishop silver is being obtained in order to complete lab evaluations started last year. Matthey-Bishop silver is now lower priced than Engelhard. Efforts will be made to approve Matthey-Bishop as a supplier of catalyst for MO.

MO Catalyst Reuse - Branecky

No progress due to priorities.

MO Analytical Manual - Branecky

Work was started this month on preparing a comprehensive analytical manual for MO unit labs. Emphasis is on basic explanation of methods to improve overall understanding.

PE Color - Thigpen, Stasney

PE product color has been relatively good since August 26 when the acid digester (hydrolysis reactor) was cleaned out and the ozonator was cleaned and repaired. Since that time (through September 30), daily lots of Pure PE have met our color spec (5 or less Pt-Co units for a 5% solution) 21% of the time (4 of 19 days). This contrasts with only 8% of the daily lots (10 of 120) on spec for the January 1 to August 26 period. Tech PE daily lots were on spec (10 or less Pt-Co units, 5% solution) 100% of the time (15 of 15 days) during August 26 to September 30. This contrasts with only 43% of the time (48 of 111 days) on spec during January 1 to August 26.

It appears that a "clean" acid digester is one of the many critical requirements for minimizing color formation in the PE unit. The digester should be cleaned periodically after the carbon bed (for color removal) is installed in the unit. This should extend the life (minimize costs) of the carbon.

Even with the "improved" color experienced recently in the unit, we still need the fixed bed carbon treatment to regain BASF-Wyandotte sales and meet competition.

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When our Pure PE is "on spec" at 5 Pt-Co units of color (5% solution), it contains approximately twice the amount of color bodies as typical Hercules Pure PE, and still does not meet BASF-Wyandotte requirements.

The final draft of the RFA for the fixed bed carbon treatment of digester product ("B" crystallizer feed) to remove color is in preparation, and should be submitted for approval in early October.

PE Efficiency - Thigpen

No laboratory work due to priorities.

PE "A" Magma, Effects of Increased Cooling - Thigpen

No laboratory work due to priorities.

Low Sulfate PE AML Sodium Formate - Thigpen

No laboratory work. Another plant trial of higher digester temperature will be scheduled when consistently low colored Pure PE product is produced in the unit. This is necessary to determine the effect on color. A previous plant trial (HHT-12-76, DLS-24-76) indicated that the target of <0.5 wt. % sodium sulfate in the AML sodium formate (dry basis) can be met with a digester temperature of about 250° F versus typical 215° F (results in less sulfuric acid needed for hydrolysis of linear formals), but effect on color was inconclusive. The next plant trial may have to wait until after the carbon bed is installed in the unit.

Butanol Production in the 1,3-BG Unit - Thigpen, Salinas

The effect of temperature and pH on the rate of acetaldehyde dehydration to crotonaldehyde (butanol precursor) was determined. The data will allow estimates of the effect of residence time on crotonaldehyde production where acetaldehyde is handled in the unit. This can provide the basis for determining whether additional cooling or pH adjustment to reduce butanol production can be justified. A report was completed and should issue in October.

PrOH Material Balance - Bacon

Work postponed until October. Piping now available to tank hydrogenation product. A letter with Process Engineering covering the June 29 to July 3 balance is in final stages.

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BuOH Material Balance - Bacon

Analytical methods were set up for a BuOH material balance. The run was carried out from September 23, 12:00 midnight through September 28, 12:00 midnight. Initial calculations for mass balance was 101%. Analytical workup is continuing.

Oxo Buffer Gas Measurements - Bacon

Oxo compressor buffer gas was measured to determine methane efficiency losses in the low pressure vent. To do this low pressure vent samples were taken with buffer gas in and out of the low pressure vent system. Calculations by H. W. Bryan showed that losses were somewhat less than plan (7M SCFH).

TMP Adipic Acid Ester Color - Jackson

No progress due to priorities.

DAA Catalyst Evaluation - Jackson

Report being prepared.

DAA Finished Product Color Stability - Jackson

Report being prepared.

DAA Parallel Reactors Plant Trial - Jackson

A test run was conducted in the DAA unit to determine if two reactors in parallel would increase DAA conversion per pass by increasing catalyst bed residence time. The unit is usually operated on one reactor. Results were increased unit capacity (as high as 19 MM lbs./yr.), but a decrease in unit efficiency (~7%) due to increased mesityl oxide make in the reactors. More work is planned to minimize mesityl oxide make. A report will be issued.

TMP Bag Evaluation - Jackson

Bags from all vendors have been set up for evaluation under normal warehouse conditions. The test will cover 90 days storage.

Elimination of Acetate Finishing Column - Hilbrich

The study for elimination of T-160 during propyl acetate production is completed. A report is in progress.

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Improvement of Formcel Blending Operations - Hilbrich

A study of the methyl Formcel reactor blending procedure is complete. The inability to develop an accurate blending procedure was traced to a significant loss of methanol through the vent system. A report is in progress.

The procedure for blending Formcel rundown tanks to specification has been observed. Assistance was given to Process Engineering in developing an alternate procedure for determining necessary addition volumes. Process Engineering will issue a report.

Ester Unit Catalyst Evaluation - Hilbrich

Samples of the present catalyst (MSA) and a proposed replacement have been sent to the Technical Center for corrosion evaluation. Laboratory evaluation of the alternate will begin following notification of results.

Paraform Unit Material Balance - Hilbrich

Samples were taken around the recovery evaporator. Analytical results were inconclusive due to inability to obtain mass closure around the system. Work will be repeated as soon as a flow meter on the recovery evaporator residue is installed.

Front Ditch Contamination Studies - Brady

The precision study on the carbon furnace A.A. was completed by the CCCTC. Considerable trouble was encountered in maintaining the high atomization temperatures required for chromium and nickel, consequently, poor precision was obtained for these two metals. CCCTC has contacted Instrumentation Laboratories about this problem. No further action is recommended by the Bishop Laboratory, until this problem is resolved.

Monitoring of heavy metals and COD in the front ditch continues.

Purchase Specifications Update - Brady

Purchase specifications have been written and/or updated for all but two of the chemicals purchased by Chemcel. Work continues.

Demineralizer Startup Support - Brady

The Laboratory was represented in training on the new demineralizer that was conducted from August 30 to September 3, 1976.

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Analytical backup for the demineralizer process analyzers was provided in the Laboratory during startup. The analyzers were determined to be functioning properly.

Waste water from the demineralizer was monitored for its effects on the front ditch. The waste water has a high acid content which is partially neutralized in the lime pits. The sludge recycle pump is not functioning because it cannot be primed. This sludge must be mixed with the waste water for optimum neutralization. Work is continuing on this problem.

The lime pits outfall was also checked for levels of Cu, Ni and Cr. The levels of these metals in the lime pits outfall has increased since the demineralizer startup. Work continues to determine the lasting effects.

Utilities Support - Brady

Filters in the condensate lines became plugged with a fine, black solid. Thought to be carbon, it was analyzed to be iron oxide.

Evaluation of Rohm and Haas IRA-94 for Formaldehyde Service - Brady

No progress due to priorities.

BOD Unit Analytical Methods Manual - Brady

No progress due to priorities.

1, 3-BG Daily Carbon Accountability - Hermann

No further work was conducted due to priorities. Procedure will be checked and costs calculated when unit operation resumes in November.

1, 3-BG Unit Support - Hermann

Laboratory measurements and calculations were begun to determine the effect of operating the finishing system without the Wiped-Film Evaporator, MS-96. R. S. Hahn, of Operations, has proposed that the vacuum flasher, V-778 operate alone to improve the operation of the unit. Initial lab results indicate that the effect on BG efficiency would be very small. Further work will finalize these results and also determine the present effect of the vacuum flasher on BG efficiency.

Laboratory measurements and calculations were made of the density of the crude butanol in the BG unit. An average density of 6.95 lbs./gal. at 90°F was found. Daily accounting of the crude BuOH has been based on a density of 6.622 lbs./gal.

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A letter was submitted recommending a change in the accounting, and future work to improve the BuOH accounting is planned. The change will cause the BG unit efficiency to BuOH to increase approximately .5%.

1,3-BG Unit Efficiency - Hermann

Work was conducted in the unit and the Laboratory to determine the effect of changes in the top temperature of T-219, AcH stripper, on the by-product production. Initial results indicate that present operating conditions are optimum. Future work will finalize these results and will determine the effects of the condensate which is added to the steam fed to T-219.

TMP Unit Support - Hermann

Work was conducted in the Laboratory to isolate the acidic compound in TMP product. Work was not completed, but will continue as time permits.

TMP Daily Carbon Accountability - Hermann

Report on procedure will be completed and submitted during October. All necessary analyses have been determined and the joint report with Process Engineering (J. R. Kirk) will be prepared.

ANALYTICAL

Fuel Gas Quality - Dow

The Tracor sulfur analyzer has been installed and several samples of MS fuel gas were scanned. Quantitative values could not be assigned because optimum operating conditions have not been established.

MS fuel gas analysis shows it to be 97+ vol. % methane with -0.1 vol. % C₆+

MS-II Analytical Support - Dow

Crude methanol was analyzed routinely for specific gravity, water and DME. The DME concentration decreased from about 2 wt. % to 0.2 wt. % as unit rates were increased.

Three material balances were analyzed around the Lugi converters. These balances were obtained to determine stream composition and to cross check the process analyzers.

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Oil Content of MS Carbon Beds - Dow

No work due to priorities.

Iron in TMP Sodium Formate - Hermann

The Instrument Laboratory checked the iron content of the residue of the NaFo concentrator, V-1331, during the week of September 10 through September 17, 1976. Iron levels were compared with levels in spot samples of the NaFo holding tank, V-161, and of NaFo shipments. No discrepancies were found which might explain higher than normal levels of iron which have been observed in NaFo shipments during July and August. Data and results will be reported when data on iron levels in customer samples has been obtained.

BG Hydrogen Vent Return Condensate - Hermann

A sample of the liquid which collects in the BG hydrogen return stream to Boiler House number 1 was analyzed by the Laboratory. Analysis was needed to determine cause of unusual amounts of liquid collected. The sample contained approximately 55% H₂O, 30% BuOH, 15% EtOH and a trace of BG. A report has been submitted with M. F. Brady and A. J. Bacon.

Oxo, BuOH Analytical - Bacon

Samples of rhodium nitrate solutions from Matthey-Bishop's final workup of 300 troy ounces of rhodium were analyzed in the Lab. All spec points were met with the exception of free HNO₃ (7% versus 3.5%). On the basis of Tech Center work on the effect of excess acid, the Lab has recommended accepting this material.

A report was written on the optimization of the microcoulometer analysis of Oxo raw materials for chloride.

Samples of T-67 liquid sidestream (spec BuOH) are still being analyzed in the Spec Lab to determine the effects of BG rerun on liquid sidestream quality.

Work continues on PrOH purification streams analysis to determine lbs./gal. for better daily accounting.

Analytical Group Support - StaffCompetitive Evaluations - None this month.MO Units

MO-III - The Instrument Laboratory provided analytical coverage (sodium by AA)

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for the optimization study of the Waste Heat Boiler. Recent modifications to this system have alleviated the sodium carryover problem previously experienced. Sodium carryover has dropped from the 2-5 ppm level to <0.1 ppm.

Formaldehyde Blending - In-line blender: Continued, intermittent excursions of the formaldehyde in-line blending apparatus are resulting in an increased sample and analysis load for the Laboratory. Reblending, reloading and re-sampling to insure quality of shipments are occurring at rates of 1-6 per week with overtime requirements of 3-9 hours per week. Late and/or delayed shipment count is up. Batch blending in available tankage has been reinstituted for time-critical customers (Plastics Mfg. Co.).

Paraform Area

Weak Formaldehyde Recovery - Methanol in T-185 overhead feed to MO-II was monitored for a twelve-day period. Higher than expected levels of MeOH (- 80-98%) were found. Since this value has an effect on unit efficiencies, the need for an analytical and/or accounting program is being investigated.

Methyl Formcel - A customer (American Cyanamid) complained of methylal in his recent receipts of methyl Formcel. Methylal (not a point of specification) was determined on shipment retain samples and found to be at usual and expected levels of 0.3-0.7%. Shipments to American Cyanamid will be monitored for methylal content for purposes of history. Retains will be analyzed after one to two months to determine if methylal builds up in storage.

PE - Cargill complained of fines in their bulk receipts of PE Pure. Car number SHPX60193 was held up at their location. Analysis of the car in question (3.9% fines) and of all bulk loadings for the past year (3.5-7.5% fines) determined that our product has not changed. Cargill agreed to accept recent shipments and to examine their unloading procedures for any changes. Extensive sieve testing was required for this parameter which again is not a point of specification.

1,3-BG - Incoming shipments of acetaldehyde (AcH) continue to be monitored for the Clear Lake Plant study of carbon steel tank car suitability. Analysis data reports are submitted monthly.

Samples of 1,3-BG jet condensate were added to the routine sample schedule for the loss accountability program. Organic losses will be followed via Total Organic Carbon Analyzer. Initial jet condensate samples show 1-3% TOC.

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Plant Rainfall Runoff Program - Source sampling for the plant rainfall runoff program has been expanded as per JMS-789-76 on September 14, 1976. Current sources described in the letter include points A, B, C, E, F, G, H and J. Analysis and chart plot reports will continue to be issued weekly from the Laboratory.

Corpus Christi Terminal - Analytical assistance is being provided for the barge loading of MS-MeOH on callout basis. Equipment, reagent and standard requirements for the Terminal Lab setup are nearly complete. It is anticipated that the setup will be completed in early October. Two liftings have been made with good product quality and minimal delay.

ENVIRONMENTAL

OSHA Compliance Measurements - Retherford

The paraform unit lab was monitored for formaldehyde vapor as part of a biweekly monitoring program. This monitoring is to insure employee exposure to formaldehyde vapor does not increase. The monitoring program will continue biweekly until engineering controls have been evaluated and implemented (AER-47-76).

An Industrial Health meeting at the Pampa Plant was attended, September 23, 1976. J. M. Ramey, New York, headed the meeting. Progress and organization of the Industrial Health Program were discussed.

TO-I was monitored for benzene on an 8-hour TWA basis. Area samples, as well as personnel samples, were taken. Monitoring results will be published.

Effect of Temperature on the BOD Units - McReynolds, Thigpen, Stasney

Four tanks were started up in the laboratory on September 7 and run at 75° F for 7 days. Temperatures were then adjusted on three of the tanks and the tests continued with each tank at a different temperature (60° F, 70° F, 75° F and 90° F. Feed to the units is typical plant BOD unit feed containing 8,250 mg COD per liter. Retention time is 4.5 days. VSS is controlled at about 2,000 mg per liter by daily removal of a portion of the tank contents.

Some definite effects of temperature on the performance of the tanks are evident from examination of the data through September 30. In general, performance is poor at 60° F, and is progressively better as temperature increases to 90° F. Some of the trends which will be quantified upon completion of the studies (about October 7) are as follows:

- Settling velocity is poor at 60° F and improves dramatically as temperature increases.

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- COD in the outfall is significantly higher at 60°F (about 470 mg/liter) and progressively decreases as temperature increases (about 90 mg/liter at 90°F).
- Production of volatile suspended solids (VSS) is high at 60°F and decreases as temperature increases.
- Oxygen uptake rates measured on samples of the tank contents (with and without feed added) are lower at 60°F and increase as temperature increases.

A report will be issued upon completion of the studies. No additional studies are planned.

TMP T-206 Overhead Receiver Vent - Hermann

The vent of T-206 overhead receiver was sampled and the organic carbon emission was determined. A report will be issued with Process Engineering (J. R. Kirk).

MISCELLANEOUS AND PERSONNEL

Analytical Supervisors C. E. Bull and J. H. Harris attended a one-day PMA Seminar entitled "Motivation and Discipline" on September 9 in Corpus Christi.

All supervisors of exempt personnel attended the seminar on "Performance and Career Planning" at Texas A and I.

During September the Chemical Section started its complete four man rotational work schedule. The procurement and training of the necessary manpower and the implementation of this work schedule has finally relieved the Wet Chemical Section Supervisor of the routine bench aspects of his work.

Ann Retherford attended the Company Industrial Health and Monitoring meeting at Pampa on September 23, 1976.

The Bishop Laboratory and Chemical Company lost one of its top scientists as a result of the death of Principal Chemist, Mr. W. O. McReynolds on September 17, 1976.


W. H. Meyer

WHM/jm
Attachments

002902

Current Project Priorities

PROJECT	Priority	Chemist
MO Efficiency Improvement with Trace Amounts of Halogenated Hydrocarbons	1	Branesky
MO Catalyst Screening	2	Branesky
MO Catalyst Reuse	2	Branesky
MO Analytical Manual	2	Branesky
PE Color	1	Thigpen
PE "A" Magma, Effects of Increased Cooling	2	Thigpen
PE Efficiency	1	Thigpen
Low Sulfate PE AML Sodium Formate	1	Thigpen
TMP Adipic Acid Ester Color	3	Jackson
DAA Finished Product Color Stability	1	Jackson
DAA Catalyst, Comparative Evaluation	1	Jackson
TMP Bag Evaluation	1	Jackson
Oxo Reactor Accountability	2	Bacon
PrOH Material Balance	1	Bacon
PrOH - Pounds/Gallon for Daily Accounting Improvement	1	Bacon
T-67 Liquid Sidestream	1	Bacon
T-101 Residue to Butyl Acetate	1	Bacon
BuOH Material Balance	1	Bacon
Removal of the Acetate Finishing Column	2	Hilbrich
Improvement of the Formcel Blending Operation	1	Hilbrich
Ester Unit Catalyst Evaluation	2	Hilbrich
Paraform Unit Material Balance	1	Hilbrich
Front Ditch Contaminants Studies	1	Brady
Purchase Specifications Update	1	Brady
Demineralizer Startup Support	1	Brady
Utilities Support	1	Brady
Evaluation of Rohm and Haas IRA-94 for Formaldehyde Service	2	Brady
BOD Unit Analytical Methods Manual	2	Brady
1,3-BG Unit Support	1	Hermann
1,3-BG Daily Carbon Accountability	2	Hermann
TMP Unit Support	1	Hermann
TMP Daily Carbon Accountability	2	Hermann
1,3-BG Efficiency	1	Hermann
<u>ANALYTICAL</u>		
Oxo Analytical Support	1	Bacon
Fuel Gas Quality	1	Dow
MS-II Analytical Support	1	Dow
Oil Content of MS Carbon Beds	3	Dow
Iron in TMP Sodium Formate	2	Hermann
<u>ENVIRONMENTAL</u>		
OSHA Compliance Measurements	1	Retherford
BOD Unit Operation, Temperature Effects	1	McReynolds, Thigpen, Stasney

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Reports in Preparation

Color Removal from PE Streams,
Laboratory Status Report - - - - - Thigpen

DAA Material Balance- - - - - Jackson

MO Efficiency Improvement by Addition of
Trace Amounts of Halogenated Hydrocarbons - - - - - Branecky

Justification for Charcoal Tube Thermal Desorber - - - Retherford

High Acidity in Formaldehyde Blends- - - - - Brady

MO Absorber Vent Analysis- - - - - Branecky

MO Analytical Manual - - - - - Branecky

DAA Catalyst Evaluation - - - - - Jackson

DAA Finished Product Color Stability - - - - - Jackson

DAA Parallel Reactors Plant Trial - - - - - Jackson

Condensate in H₂ Vent from BG Unit - - - - - Hermann, Brady, Bacon

Density of Crude BuOH in BG Unit - - - - - Hermann

TMP Daily Carbon Accountability Procedure- - - - - Hermann, Kirk

TMP T-206 Overhead Receiver Vent Emissions - - - - - Hermann, Kirk

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STATISTICS

	<u>September</u>	<u>September Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section Samples	3,649	35,859	33,795
Instrument Section Samples	<u>1,953</u>	<u>18,908</u>	<u>17,551</u>
TOTAL	5,602	54,767	51,346
Sample Shipping - Domestic	76	872	1,083
- Export	11	140	46
- Plastic	5	106	63
- Other	<u>35</u>	<u>206</u>	<u>209</u>
TOTAL	127	1,324	1,401
Technician Overtime - Hours			
Chemical	41	568	438
Instrument	24	447	249
Special Problems	<u>18</u>	<u>46</u>	<u>32</u>
TOTAL	83	1,061	719

Shipments (No. of Orders)	735	6,821	4,587
Delayed Shipments	4	29	15
Quality Waivers	1	12	4
Quality Complaints	3	18	6
Quality Complaints Accepted	0	9	6

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QUALITY SUMMARYFormaldehyde Blending

A delay in truck deliveries of formaldehyde to Plastics Mfg., Dallas, was caused by sulfuric acid breakthrough from the Graver demineralizer. Their CPU had to be sent back empty (at their request) because of their need for the CPU trailer in other service. Plastics Mfg. has a special acid spec at a maximum of 0.003 wt. % in 44% formaldehyde. At the present time, the blending unit is batch tank blending for loading trailers for Plastics to minimize trailer delays. The condensate problem was also resolved.

Chemagro had complained that their receipts of 37% uninhibited formaldehyde were low in assay. Retain samples of previous shipments to them were reanalyzed and show that the material shipped to them was on spec. Complaint not valid.

Paraform

Had one complaint during the month from Core-Luke concerning the presence of yellow lumps. On occasion small yellow lumps are present in our production. No sample has been received from them to date to verify their complaint.

BG

Three 8,000-gallon cars are in inventory with a 1.2 odor. This material is being held awaiting orders from less critical accounts. The odor cannot be blended.

Methylal

Three thousand gallons of methylal was returned to Chemcel from Vulcan (September 15, 1976). This material is of good quality, very dry, and was returned after 3 years. The material was added to current inventory after testing and approval.

Diacetone Alcohol

Two tank cars sent to Newark were found to have a color buildup. One car shipped August 16, 1976, Celx 3281, arrived at 35 color. Retain confirmed color. Celx 9206 shipped July 23, 1976 and held in inventory, had a color greater than 60. This car has been sold to a customer at a distressed price.

TMP

The acid content has been borderline (spec 0.002%) to high (0.003%) most of the month. The bulk of the material has been shipped export. Control of the acid and water level on TMP (0.05%) has been very difficult all month.

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