



CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas

To: C. W. Harrison

From: W. H. Meyer

WHM-187-78

August 1, 1978

Laboratory Department Summary

July, 1978

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The Bishop Plant Laboratory activities during July 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/as
Attached Report

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I. ANALYTICAL GROUPCOMPETITIVE EVALUATION

None this month.

Methanol Oxidation (MO) - Formaldehyde (HCHO)

Special analysis were provided for Material Balance studies around MO Units II, III and IV this month. Good coordination of the studies by Mr. D. V. Shattuck (Engineering) eased the additional sample load on our Section.

The possibility of an analytical bias between Newark Terminal and Chemcel Laboratories is being investigated. Newark consistently reports low assay values (wt.% HCHO) on HCHO receipts from Bishop. Extensive checking of methodology, technique, equipment and reagents has not found significant differences. Initial sample exchanges comparisons are within acceptable limits. Newark will begin the return of analyzed samples taken from HCHO receipts for continued cross checking and to permit an evaluation of shipped versus received product.

Paraform - Esters

Analytical support was provided for the identification and quantification of the incidence of higher than normal heavy end impurities in the last run of iso-BuAc. It was determined that the iso BuOH feed stock, purchased from Union Carbide, was of low quality causing the heavy ends in the ester. Support was provided throughout the rerun, clean up operation.

Polyol Area

Extra analytical coverage is being provided for an apparent "sweating" (H₂O) problem in Diacetone Alcohol storage. Product water level build occurring in inventory is apparently associated with inventory levels, ambient temperature and tank breathing. DAA production remains normal at 0.02 - 0.04 wt % H₂O (max spec. = 0.10 wt %) and shipping requirements are being met via blending. Operations is investigating installing dry blanket gas on the tank.

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Utilities - BOD

The cooperative study undertaken for evaluation of the dependability of the Plant Total Carbon Analyzer alarm system continues. Good routine agreement between Main Laboratory and Unit analyzers is being achieved. The comprehensive unit method of analysis for Total Organic Carbon has been written and is awaiting approval and typing prior to issuance and incorporation into unit operation.

Plant effluent streams fed to the BOD Unit are still being monitored for ppm Sodium. This is to determine sources of Inorganic Carbon in the Unit.

The expanded Quality Control Program composed of a schedule of duplicate and spiked analysis on EPA and TDWR regulated outfall samples is 90% complete and is being implemented this week. A report detailing the procedures will be issued. The self report forms have been updated to allow reporting of the increased analysis.

Texas Department of Water Resources representatives were in the Plant Tuesday, 7-25-78 for a routine sampling of our outfalls. A portion of the samples taken were reserved by the Laboratory for comparative analysis. A report will be issued upon receipt of the TDWR results.

General

Hewlett Packard (Computer Division) personnel have presented the HP GC Data Systems for our consideration and replacement of the current obsolete Laboratory EAI Computer. Investigation of the alternative configurations continues.

A new chemist employee, Jennifer White, has completed 3 weeks of orientation in the Analytical Group work areas.

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August 1, 1978

Analytical Statistics

<u>Sample Count</u>	<u>July</u>	<u>July Y-T-D</u>	<u>Previous Y-T-D</u>
Chemical Section	3,611	31,523	35,207
Instrument Section	<u>2,625</u>	<u>18,125</u>	<u>17,141</u>
Total	6,236	49,648	52,348

Technician Overtime

Chemical Section	36	694	535
Instrument Section	<u>16</u>	<u>143</u>	<u>494</u>
Total	52	837	1,029

II. QUALITY ASSURANCE

Customer Complaints Due to Quality - On 6-30-78 N.Y. Quality Assurance reported Cargill, Inc. at Carpentersville, Ill. received ACFX 61366, a hopper car with PE Pure, and complained about the "High" fines. Customer had problems unloading, and decided to return the car to Celanese. The car arrived at Bishop 7-27-78 and the quantity and quality of the product returned will be evaluated. The retain sample analysis indicated specification product and a screen analysis did not show an abnormal high fines content.

A complaint was indicated from Sherwin-Williams at Garland, Texas related to lumps found in a recent shipment of PE Pure. This complaint was noted in a letter from P. C. Doughty to T. Golson in New York. We have not received a return sample so validity of complaint is unknown. The shipment information is also unknown.

On 7-11-78, Pampa Plant reported that Methanol from Bishop has an Amine like odor. The odor was strong enough to cause a rejection from I.C.C. at Arvada, Colo. The "amine like" odor has been present in varying degrees in Chemcel Methanol for quite a long time based on retain samples and it is apparently more pronounced in the summer. Future evaluation of this problem will depend on the quality of the Methanol produced from the new catalyst in MS II.

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August 1, 1978

Jim Stafford at Newark has reported two tank cars, with 45% HCHO, arrived at the Argo Terminal with high color. CELX 13213, shipped 6-27-78, had a color of 25 and CELX 13228, shipped 6-29-78, had a color of 20 with particles present. The sample retains of these shipments were analyzed and found to be specification material. The product was filtered and blended off at the Argo Terminal.

Two tank cars of Inhibited (6.5% - 7.5%) Formaldehyde arrived at the Argo Terminal with high color (35). Jim Sanborn reported this complaint 7-17-78. CELX 4217 was shipped 6-23-78 and CELX 3251 was shipped 7-7-78. Once again a reanalysis of the retain samples of these cars confirm that both tank cars were loaded with specification product. Jim Sanborn stated that the product will be filtered and blended off for use in the Argo area. A return sample from CELX 3251 received 7-27-78 confirms the high color (35). All other points meet our sales specification.

Jim Sanborn on 7-19-78 reported a third tank car of 45% Formaldehyde in CELX 2257 arrived at Argo on 7-18-78 with high color at 20. The retain sample analysis of this car shows color <5 and Iron <0.1 ppm. Sanborn reported that this material will be treated as the previous cars.

Quality Waivers and Shipments Delayed - A shipment of 50% Formaldehyde to Acme Resins at Forest Park, Ill. in CELX 13204 was delayed three days due to high color (15) and loading temperature (161°F). Acme Resins require loading temperature to be 150 - 160°F.

CELX 1320 with 50% Formaldehyde for Acme Resins at Oregon, Ill. was delayed 2 days because of color (12) and a low minimum Formaldehyde content (50.10). Acme requires 50.2 minimum.

Two tank cars of Iso Butyl Acetate required a quality waiver from T. H. Golson, New York Quality Assurance, because of 0.3% heavy ends, later identified as Iso and n-Amyl Acetate. The waiver was granted for CELX 5265 for San Pedro, Ca. and PTLX 120339 for Celriver, S.C. The waiver was granted to ship these cars on 7-14-78.

Production Quality

Formaldehyde - Due to the large number of color complaints on Formaldehyde loadings, an inspection was made on a tank car scheduled for Formaldehyde. This car was CELX 4244 which has a Herisite liner. The car was cleaned very thoroughly with Electrosal and jet washed and then hand washed and dried. The car was loaded with 37% Formaldehyde inhibited (7.0% Methanol) and shipped to Argo on 7-25-78. The analysis of the product showed a color <5 with all other specs on test. We have asked for follow up analysis on this car after it arrives at Argo. The Special Problems Section of this report contains further problem solving information on the Formaldehyde color problem.

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Sample Shipping and Quality Assurance Statistics

Statistics For July 1978

<u>Sample Shipping</u>	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Domestic	77	460	724
Export	3	23	69
Plastics	3	30	55
Other	17	103	692
Total	100	616	1,540

Quality Assurance Statistics

	<u>Month</u>	<u>YTD</u>	<u>Previous YTD</u>
Shipments (NO.of Orders)	682	5,141	6,019
Delayed	2	18	22
Waviers <u>Internal</u>		2	(29)
<u>External</u>	1	10	Total
Complaints (Total)	6	27	37
Complaints (Legitimate)	1	11	12
Complaints (Non-Legitimate)	0	5	25
Complaints - Outstanding (Unresolved)	5	11	---

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III. SPECIAL PROBLEMS GROUP

Methanol Oxidation, Formaldehyde Blending
J. D. Presley

MO Efficiency Increase with
Trace Amounts of Halogenated Hydrocarbons - No progress due to priorities.

MO Catalyst Reuse - No progress due to priorities.

MS-II Crude to MO - Additional lab work has been done to determine effects of filtering Crude Methanol. Also, MeOH samples have been analyzed by CCCTC for Nitrogen. They report less than 2 ppm. A report is in progress.

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

MO Unit Support - The Lab provided analytical support for material balances on MO II, MO III and MO IV. Results will be published by Process Engineering.

MO IV showed an indicated AMR drop from 1.01 to 0.93 when the Methanol feed water compensator was disconnected. An analytical AMR check by the Lab showed an AMR of 1.0. Work continues.

Feed Methanol to MO II and MO III was contaminated by water early this month. It is believed that, during the MS shutdown, a slug of water got through the MS finishing system and contaminated V-1695 and V-1688.

Recently, there have been critical problems (sintering and acid make) with new MO Silver Catalyst received from Engelhard. Samples have been sent to CCCTC for surface analysis. In a meeting of S and D with Engelhard the problem was discussed. Engelhard agreed to send pertinent information requested by the Bishop Lab.

A short time Paraform color problem was noted during July. The feed HCHO was checked for contaminants by M. E. Harris (See Paraform). A source of possible contamination was thought to be MO IV Vaporizer

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Blowdown which is fed to the Absorber for MeOH recovery. Both the MeOH feed to the Vaporizer and the Vaporizer Blowdown were checked for Iron levels, feed showed 0.13 ppm and blowdown showed 0.12 ppm. This amount of Iron in the HCHO product would be only <10 ppb. This (10 ppb) level should not be sufficient to cause the color problem seen in Paraform.

Complaints of HCHO color problems have been received from the Celanese Terminals. Retain tank car samples were checked and no color build was seen. The acid level of the retain from a car reported to be highly colored was treated with Formic Acid to try a forced color build. None was noted. Some of the tank cars used to ship the HCHO were inspected and found to have a rusty film on the interior surface. From surface samples analyzed, it is believed that sufficient Iron was present to cause the color problem. It was recommended that the old tank car cleaning procedure (scrubbing with soap) be reinstituted in addition to the normal steam cleaning.

A check was made for possible cooling water leaks in the MO IV Absorber Residue (T-33 Res.) and Distillation Column Overhead (T-2720H). Results showed <0.3 ppm chlorides in T-33 Res and <1 ppm chlorides in T-2720H, indicating no leaks.

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

BOD Unit Analytical Methods Manual - No progress due to priorities.

Anaerobic Testing of Chemical Wastes - During July chemical loading to the anaerobic filter has been gradually increased from 0.24 to 0.45 lb COD/ft³/day. Kinetic loading has increased from approximately 0.15 to 0.3 lb COD/ft³/day. Chemical removal has remained at 75 - 80%, kinetic removal at 65 - 70%. Formaldehyde concentration in the equalization basin feed has ranged from 1800 ppm to 3800 ppm. It was determined that the Schiff's Test for HCHO used on the anaerobic filter outfall samples was giving erroneously high results (50 - 100 ppm HCHO) possibly due to interfering compounds in the samples. CCCTC analysis of outfall samples (by polarograph) have shown HCHO levels to be 2 - 14 ppm. Loading will continue to be increased. Oxo waste stream will be added to the feed after the filter has stabilized at 0.5 loading.

Most of the mechanical aspects of the aerobic polishing section have been worked out. A new 5 l clarifier was built and installed. The aeration tank was recharged with BOD aeration basin bacteria on 7-25-78.

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Aerobic Treatment of Nylon Wastewater - Nylon wastewater and normal BOD Unit feed are being fed to two laboratory aeration tanks at food to biomass (F/M) ratios of 0.5 - 0.7. Both tanks are showing adverse effects in terms of high outfall COD's (400 - 600 ppm) and poor settling characteristics since the increase in F/M ratio. The study is continuing; effects of increasing the nylon waste load at varying F/M ratios will be studied.

BOD Unit Support - Daily analyses of BOD outfall samples showed decreasing Hexamethylenetetramine (HMTA) levels accompanied by decreasing COD values. A report coauthored with L. Mayerle and K. Parker, has been submitted.

An increase in outfall COD for several days (7-17-78 through 7-23-78) was investigated. No PE, TMP (analysis by L. Mayerle), or TO was detected in outfall samples.

OSHA Monitoring
J. M. Jackson

The Formaldehyde Survey in the Plastics Company SH I/TO I area is complete. Personnel eight-hour time weighted average were taken to document exposure. The Operator Helper job description (duties performed) showed a significant level of exposure. Area samples were taken to determine high exposure areas. A report is in progress.

Formaldehyde follow-up monitoring for employees in SH I/TO I areas during July was completed. Seven action forms were issued in the SH I/TO I areas during July. Follow-up monitoring for these samples will be done in August.

Nickel Tetracarbonyl monitoring was conducted during MS shutdown. Outlet nitrogen purge gas was monitored from the following; 5 MM and 2 MM Girdlers, Methanator and Selas and Davey Powergas Reformers. No detectable Nickel Tetracarbonyl was found. A report will be issued.

An observation of the Paraformaldehyde bulk loading operation was made during hopper car loading. Excessive powder was observed escaping around the loading hatch. Concern was given to 1,3 BG tank car loading down-wind of this area. Work will be scheduled to determine particulate exposure down-wind of the Para bulk loading facilities.

003287

Esters, Methylal, Paraform, Formcel® Products
M. E. Harris

Acetate Unit Support - The VLE data needed to complete both Lab and Process Engineering studies for T-343 start up is essentially complete. Recent data indicate that acetic acid has a large effect on vapor phase composition at high concentrations (60 - 70 wt %) in the liquid phase. However, the data does not entirely explain the T-343 profiles taken during start up trials. A report is planned for early August.

The Lab plans to simulate T-343 using a 45 tray column to study a number of operating parameters. This study will begin in early August.

Contaminated iso-Butyl Alcohol - Five tank cars of iso-Butanol received July 7 - 10 from Union Carbide were considerably below the quality of previous shipments. Acetate quality problems were encountered due to the higher than normal concentrations of both light and heavy alcohols (Amyl Alcohols). Union Carbide has provided an average analysis of these shipments which indicates that our sales spec. for maximum n-Butanol was also not met (sales spec <0.2% vs Carbide average analysis of 0.27%). The iso-Butyl Acetate was purified by stripping heavy ends out in T-160 to meet our sales spec's.

Paraform Unit Support - Paraform recently was upset due to an iron contamination problem which resulted in poor flaker performance, color problems, and possibly was the cause of contaminated n-Butyl Formcel® solutions produced from this material. Iron concentrations of 4 ppm were found in the flaker pans. The initial indication is that the iron contamination came from V-1745 (HCHO storage tank), where a sample was found to contain 1.2 ppm iron. Evidently, this was a "slug" of contaminated material, because earlier in the morning, no iron was seen in the V-1745 sample in the wet lab. A report is forth coming.

Formcel® Products Unit Support
R. G. Hilbrich

A sample of "fibrous" material reported by Enenco to have been filtered from our Methyl Formcel® product was identified as cotton string from our loading line filter. Operations is issuing an AVO for installation of a strainer in the loading line after the filter.

003288

Butyl Formcel® Color Problems
M. E. Harris

This month, 24,000 gallons of Butyl Formcel had to be burned due to extremely high color which prevented blending to spec. The color was caused by high concentrations of iron (1 - 3 ppm). Zinc (.5 - 1.00 ppm) may also have caused some color formation. Intense investigation is underway to identify the source of the contaminants. A report is planned in August.

Diacetone Alcohol
R. G. Hilbrich

DAA Unit Support - The Laboratory evaluation of the Goodyear Acetone run in the DAA Unit is complete. Results indicate that Goodyear Acetone has not improved in quality and still causes rapid catalyst bed deactivation. Report in writing. It is recommended that we terminate our Acetone Purchase contract with Goodyear.

An accelerated DAA storage stability study was started in Laboratory to determine if extended storage or storage at non-refrigerated temperatures can result in failure of the miscibility test. This was initiated by recent DAA quality problems involving failure of the miscability test.

Pentaerythritol
R. G. Hilbrich

PE Unit Analytical Methods Manual - Laboratory work has started on developing the Sodium ion electrode method for determining Sodium Formate concentration in the dissolve tank.

Trimethylolpropane
L. Mayerle

TMP Unit Support - A compatibility study has begun to determine the feasibility of transporting TMP Vacuum Flasher (V-747) residue, TMP Finishing Column (T-205) overhead, 1,3-BG Vacuum Flasher (V-778) residue, and 1,3-BG Alcohol Column (T-225) overhead through a common line. This would provide an economical way to collect these streams for use as boiler fuel.

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Potential for Increased Reactor Cooling - Methanol production across the TMP reactors has increased from 0.15 to 0.30 Δ weight-percent MeOH. (Methanol production of 0.30 Δ weight-percent corresponds to a Formaldehyde efficiency loss of $\sim$ 8% in the TMP Unit.) This increase may correspond to a rate increase (5 GPM BuH to 7.25 GPM BuH) that was initiated the same day as sampling. Data will continue to be collected every two weeks at normal operating conditions. If Methanol production remains at or above 0.30 Δ wt. %, reactor temperatures and reactor fouling will be investigated for possible correlations with the increase in MeOH.

Alternate TMP Purification Scheme - Increased stirring rates failed to show a significant improvement in separations. However, recent shake-out data continues to show favorable distribution coefficients for separation. The phase separation characteristics of this system prevent satisfactory operation in the current Lab equipment (York-Scheible Extractor). No further Bishop Laboratory work is planned using this equipment. A meeting will be scheduled with CCCTC personnel to review the status of the project. Further extractor development studies will probably be performed at CCCTC. Final documentation of the Bishop study should issue by September 1.

TMP Unit Analytical Methods Manual - No progress due to priorities.

Pentaerythritol
L. Mayerle

PE Unit Air Emission Survey - An attempt to sample the Drier Baghouse Vent in the PE Unit for particulates was unsuccessful. Possible reasons for the inability to sample this vent include the following:

- (a) The velocity of the emissions through the stack exceeds the capability of the instrument used for sampling.
- (b) Atmospheric conditions were not favorable for sampling.
- (c) The moisture content of the vent stream varied too greatly during sampling.

The Drier Baghouse Vent will be resampled for particulates when this problem is resolved. Determination of particulate emissions from this vent and the Hopper Baghouse Vent will complete the air-emission survey in the PE Unit.

003290

Methanol Synthesis
F. R. Dow

Fuel Gas Quality - No samples were analyzed due to other priorities.

MS-II Analytical Support - Support was provided to monitor oxygen for the Lurgi Catalyst oxidation. The Teledyne Oxygen Analyzer was ideally suited for this analysis due to its rapid response to oxygen.

The instrument was placed in the MS-II Analyzer House and "Walkie Talkie" radios were used to provide optimum communication during the start up and line out period. One problem that should be noted is that during transmission periods the radio nearest the oxygen analyzer caused positive needle deflections. This was easily overcome by reading the meter prior to any communication transmission.

Oxo, ProH, BuOH Unit Support
A. J. Bacon

Assistance was provided and samples were taken during the catalyst drumming following the July 7 Oxo shutdown. Most analytical has been completed. We are tentatively showing that ~90 lbs Rh were returned to Engelhard for recovery. A report will be issued in early August.

Many unit lines and vessels were inspected during the shutdown. Though small amounts of "rhodium" dust were found throughout the reaction area, no deposits or signs of plating were found that might explain our ~20 lb Rh loss for the year. Small deposits at the reactor "foam line" contained ~11% Rh and high phosphorus similar to material found in the dump vessel following run #2.

A fine grey powder was found in lines and vessels from the reactor overhead to T-210 reboilers. This was essentially nickel with only small amounts of iron. The base of T-163 contained another material with a composition very close to 304 stainlers. Evidence so far is that $\text{Ni}(\text{CO})_4$ may be formed somewhere in the reactor overhead. The effects and implications of this are under study.

The Oxo reactor was brought up early July 22 on ~34 lbs of Rh. Special care was taken to keep the system dry and to keep syn gas thoroughly dispersed through the solution. A great deal of water was brought in with the 5 MM girdler feed, but was kept below 0.5% in solution by stripping. The result was a clear, active catalyst free of Rhodium fines.

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Analytical work is continuing throughout the reaction area and purification as the unit lines out.

No progress was made on M&T TPP spec tests.

A discrepancy was found in Unit Lab and Spec Lab results for iso-BuH in T-210 O.H. Information was needed for the trade with Union Carbide.

Rhodium Accounting - See above.

Reactor Accountability/Stability - A phone report from CRC related that L.C. work is about to begin and ³¹P NMR data is just now being evaluated. Additional samples were sent at their request.

An opening in the Tech Center Pilot Plant schedule allowed us to do some experiments with Run 4 Bishop catalyst in the Oxo Pilot Plant. Studies were planned to test foaming behavior and foam elimination.

Unfortunately, no foam was observed with our material in the pilot plant reactor. Speculation is that in depressuring the samples for transport, the material was "defoamed". However, Lab tests did not confirm this. Another possibility is that the reactor configuration made the catalyst behave differently. A report is being written.

Process Technology
B. C. Kerr

Statistical Data for Oxo Butanol Unit Operations - Operations data from the Oxo Butanol Unit have been collected and analyzed. These data will be issued in a letter, as time permits.

Purchase Specification Manual Up-date - A comprehensive review of the Plant Purchase Specifications for raw materials and chemicals is in progress. This project has been given a high priority. Draft specifications should be available for review by August 21st.

Improved Reliability of Total Carbon Analyzer for Plant Effluent - Varying amounts of Sodium Bicarbonate and Sodium Carbonate add a degree of uncertainty to the total carbon analysis on Plant Effluent. Cursory

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work is in progress to find a workable system for compensating for the inorganic carbon interference in the total carbon analysis.

Specification Modification for Normal and iso-Butyl Acetates - The possibility for obtaining slightly broader limits for iso-Butyl Acetate in n-Butyl Acetate and n-Butyl Acetate in iso-Butyl Acetate is being investigated. Relaxation of the present specifications would be of assistance in better utilization of available alcohol feeds.

Review of the Mechanics of Data Retrieval from Abstract Cards - A review of the mechanics for retrieving data from the Process Technology Abstract Card system was given to Mr. Chervenka's Process Engineering Group.

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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	Dept. 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 Sept., 1978
BOD Analytical Methods Manual	Smith	2	Maintenance of Business	Undetermined
Anaerobic Testing of Chemical Waste Streams		1	To Test Anaerobic Process As Alternate BOD Expansion	Jan. 1, 1979
Aerobic Treatment of Nylon Wastewater Effluent		1	To Test Treatability of Nylon Waste in Bishop BOD Unit	Sept. 15, 1978
OSHA Monitoring	J. M. Jackson	1	Ensure Employee Health and Safety. OSHA Compliance	Continuing
PE Unit Analytical Methods Manual	Hilbrich	1	Maintenance of Business	September 1, 1978
Alternate n-Butyl Acetate Process		1	Steam Savings	Aug. 15, 1978

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