CELANESE CHEMICAL COMPANY
Chemcel Plant
Bishop, Texas



To: Dr. R. R. Graham

From: G. D. Boyd GDB-196-82 October 7, 1982

LABORATORY DEPARTMENT SUMMARY - SEPTEMBER, 1982

Attached is the Laboratory's monthly summary for September. 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

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. Golson, Dallas

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HIGHLIGHTS

- All BOD pilot studies have been discontinued.

- Sodium sulfate levels in AML have been reduced to meet the VCI specification of less than 0.5 wt% on a contained-salt basis. Na_2SO_4 levels have remained consistently below this specification during the latter part of the month while the unit was producing Tech-grade PE. Efforts are continuing to ensure this specification will be met during Pure-grade PE production.

- Pilot studies indicated that ultrafiltration is not an economically feasible alternative for solids removal from solar pond liquids prior to deep-well injection.

- The Laboratory organization has been restructured. An organization chart is attached.

- A relatively simple test (turbidity of an acetone-paraform mixture) was developed which will allow better control of the Para Prill Unit and allow lab studies of the effects of temperature and additives on the polymerization rate of para liquid.

I. CHEMICAL ANALYTICAL GROUP

Methanol Oxidation (MO) - Formaldehyde (HCHO)

Low-acid, high-assay HCHO shipments to Plastics Mfg. Co., Dallas, Texas were successfully initiated this month. Nine shipments of 50% HCHO at 0.003 wt% acid maximum have been made since September 15.

In conjunction with this Plastics Mfg. Co. account, a sample of Reichhold 50% HCHO to Plastics Mfg. was obtained and competitively analyzed at Chemcel. The major difference, as expected, was high sodium content (70 ppm) in the Reichhold HCHO. The comparison of Reichhold HCHO and Chemcel HCHO is published in AGM-305-82.

We have apparently regained the Dow Chemical Co., Freeport, Texas, Versene™ account with our MO-IV unstabilized 37% HCHO product. 12 tankcar shipments were made in September and 21 cars are currently scheduled for October. The preshipment test for Versene™ "scum" formers continues to be utilized.

Paraform Area

The Chemical Group is continuing to monitor the Para Prilling Pilot operation.

Data on particle size distribution of high assay Para Flake (95% HCHO) has been gathered. This data is to determine our ability to meet Formica specifications.

Customer Complaints

None this month.

Quality Waivers

9-15-82 - 50% HCHO to Plastics Mfg. Co., Dallas, Texas - 46,900 lbs - Assay at 50.78 wt% vs. max. spec. of 50.25 wt% - SQD.

PRODUCT QUALITY WAIVER HISTORY

	% QD[1] Month	% QD YTD	% SQD[2] Month	% SQD YTD
Methylal	0	32.1	0	0
PE-Pure	0	0	0	0.3
n-Propyl Acetate	0	0.3	0	0
Diacetone Alcohol	0	4.8	0	0
PE-Tech	0	0	0	0.4
Sodium Formate	0	0	0	3.4
MEK	0	0	0	7.9
PVB n-BuH	0	0	0	2.1
Formaldehyde	0	0	0.3	<0.1

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

Sample Count	Sept.	Sept. YTD	Previous YTD
Chemical Section	5257	42,792	41,498
Instrument Section	3229	25,257	24,218
Total	8486	68,049	65,716
Technician Overtime			
Chemical Section	48	520	510
Instrument Section	8	456	321
Total	56	976	831
Sample Shipping			
Domestic	60	536	551
Export	4	26	50
Plastics	3	8	10
Others	15	94	89
Total	82	664	700
QA Statistics			
Shipments	549	5134	5874
Waivers (QD)	0	15	19
Waivers (SQD)	1	12	12
Complaints	0	15	11

II. ANALYTICAL GROUP - F. R. Dow, Group Leader

Water Samples from Edmonton (Dow, Frankenhauser) - Five water samples were received from Edmonton for Dionex anion analyses. The samples were numbered but not specifically identified. The five samples were analyzed for sulfate, formate, acetate and butyrate. A report has been issued detailing the results.

Tech-PE Intra-Lot Variation (Wise) - An investigation is underway to determine the Tech PE product variation. Two lots (229-3T and 263-1T) have been analyzed and analytical data shows that the mono-PE content varies more than 2 wt% between lot samples and samples from pallets. This project is continuing.

TMP Crystallization Process Samples from CCCTC (Dow) - A feed and 2 hydrogenated crude TMP samples were received from Dr. C. D. Murphy for analysis. The hydrogenated crude TMP contained approximately 4 wt% monocyclic formal. This compares with a 1 wt% monocyclic formal content of the crude TMP. The monobis-TMP linear monoformal was reduced from 3.6 wt% to approximately 0.4 wt%. The major unknown was reduced from 0.5 to <0.1 wt% by hydrogenation.

Pampa Well Water (Glenister) - A sample of Pampa well water was received from D. Harris of the Pampa Plant for analysis of weak organic acids. Analysis was by the Dionex Chromatography Exclusion Column (I.C.E.), calibrated with a standard containing sulfate, formate, acetate, proprionate and butyrate. The level of acetate was of primary interest because of the volume of "acetate" production at the Pampa Plant. An I.C.E. scan of the filtered (0.45 m) sample of the well water did not indicate the presence of acetates when compared to the same solution spiked with acetate. Analysis of a concentrated solution of the water, prepared by reducing a 1000-ml sample to a volume of 20 ml, also did not show the presence of acetate compared to a similar acetate spike. Additional analysis to confirm formate will be complated and the results documented.

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

Formaldehyde Exposure (Parker) - The effect of the Prilling Pilot Unit on Paraform area formaldehyde concentrations is being determined.

Follow-up Formaldehyde Exposure (Parker) - Sampling is continuing to determine the effect of the Phase I formaldehyde reduction program.

Formaldehyde Methods Validation (Gary) - The Laboratory methods validation work has been completed and the report is in progress.

Monsanto Research Formaldehyde Facility Study (Gary) - Preparations have been made to sample formaldehyde and methanol exposures during the Monsanto Research visit to the Plant on October 11-15, 1982.

III. STAFF CHEMISTS

Process Technology
B. C. Kerr

Pond Soil Recovery - Soil samples and flood wash water from Pond 4E were analyzed to obtain a measure of the effectiveness of river water flooding for removing salts and COD from dried solar ponds. A report was issued with suggestions for obtaining better utilization of the wash water.

Methylal Unit Support - Assistance was provided to Paraform to determine the cause for low methylal efficiency. The problem was traced to a faulty analysis.

Regression Equation for Formaldehyde Acidity Build - Arrival acidity data on formaldehyde shipments to Argo, Ill., were used to develop a regression equation which predicts arrival acidity on shipments based upon loading temperature, assay, initial acidity and transit time.

Paraform Unit Efficiency Problem - Yield Accounting data indicate that the Paraform Unit is operating about 1.5% below standard for the year. It has not been possible to determine, from Y. A. data, any particular period of operations which would account for the problem. Work is continuing on this project.

Paraform Prill Pilot Plant Support
H. H. Thigpen

A test was developed to indicate the degree of polymerization of fresh para prills. This should allow us to know the relative degree of polymerization of fresh para prills at any given time and to measure the amount of change which occurs when we make changes in the unit variables. It should also allow the pilot unit to respond to changes in the stability of the para liquid from the paraform unit.

The test is a measurement of the turbidity of an acetone-paraform solution. Increasing amounts of higher molecular weight polymers increase the turbidity. The test is simple enough to be used as a control method in the unit lab. One gram of paraform is whipped in a food blender with 500-ml of acetone for 3 minutes. The turbidity of the acetone is then read using a Hach Turbidimeter.

The acetone-paraform ratio has been set to give zero turbidity on para liquid samples taken directly from the pan. Turbidity of para liquid samples held at 100°C for increasing amounts of time gives a linear plot vs. time out to the time of clouding at 100°C (cloud time). Turbidity readings are 80-90 NTU at the cloud point. Thus, there is ample sensitivity to measure small changes in the degree of polymerization.

Plans are to use the turbidity measurements on fresh falling prills to te indicate the degree of polymerization. Attempts will be made to correlate the turbidity reading with melting points and unit operability. Lab studies on the effect of temperature and additives on the polymerization rate (turbidity r and increase) will be conducted as time permits.

Attempts to indicate the degree of polymerization by pouring para liquid down an incline met with only limited success. Several surfaces were tested. There was too much scatter in the data and it was very technique sensitive.

Measurement of the degree of polymerization by silylation-GC analysis also met with limited success. Although some shift toward higher molecular weight polymers was indicated as para liquid was held at 100°C, there was evidence that significant amounts of depolymerization occurred during the dissolving and silylating steps. Studies of the GC procedure will continue with efforts to minimize the depolymerization and provide useful data.

IV. SPECIAL PROBLEMS - L. M. Harvey, Group Leader

Waste Management
B. K. Huelle

Solar Pond Solids-Removal Studies - In an attempt to improve the permeate (product) flow rate through the ultrafiltration (UF) module, Pond 11E was flocced before UF treatment. It was hoped that removing solids by flocculation would improve the flow rate. While floc was generated using 10 ppm polymer and 200 ppm bentonite clay, no change in turbidity occurred. Laboratory experiments indicate 40 minutes is the optimum fast-mixing time. General Filter, a clarifier manufacturer, also found that a 30-minute induction period is necessary before turbidity is decreased. Fast-mixing time in the UF pilot unit was 10 minutes. Because of equipment limitations, additional mixing time is not possible.

003763

Although guidelines have not been established for the degree of solids removal and particle size limits necessary for deep well injection. The Laboratory is investigating particle count and size using a blood cell counting chamber called a hemacytometer. The data indicates that particles are present as high as 10 micron with the majority of particles in the 1 to 3 micron range. 225,000 particles were found in 1 ml of solar pond 11E. After flocculation, approximately 50% of the particles are removed, however, the particle size distribution is similar to pond material before treatment.

General Filter has shown that over 30% of solids over 1 micron are still present after flocculation and these particles appear to be microbes. The Laboratory has tried to measure the effect of killing these microbes and determine how this would affect flocculation. Biocides, ultrasound, HCHO, and heat were used individually and in combination as a means of killing the microbes. No change in flocculation efficiency occurred. Turbidity increased after biocide treatment. General Filter has agreed to pursue this aspect of the problem.

In addition to particle counting, present Laboratory studies are concerned with the evaluation of current Plant waste streams as to their suitability for deep-well injection in the event of a BOD upset. This involves core testing of equalization basin liquid, test pond liquid, AML, and VCI Centrate. Both equalization basin and test pond liquids passed core tests; AML also passed the core test but had a flow rate four times slower than a brine solution. A 50% increase in flow rate was observed when a 50:50 mixture of AML and equalization basin liquid was tested. VCI centrate has not yet been evaluated.

Finally, the viability of sending filtrate, from the planned belt filter press, which will contain polymer to the test ponds or equalization basin is being studied. A 1 ppm polymer solution fails core testing. Since polymers are known to decompose with time and heat it was hoped that residual polymer would not be of any risk to the injection well. However, analysis by liquid chromatography shows that polymer is still present after two weeks of standing at room temperature.

BOD, Utilities

D. R. Akin, G. R. Stasney

BOD II Unit Support (Akin) - The anaerobic reactor was operated at an average organic loading of 39,000 lbs. COD per day during the month of September. Anaerobic COD-removal efficiency ranged from 52-67%. The aerobic outfall COD averaged 258 ppm with an average detention time of 4.2 days. A total-recycle pulse test has tentatively been scheduled for November 9, 1982.

VCI/Polyol Treatability (Akin, Stasney) - This study has been discontinued due to priorities. Before being discontinued the anaerobic recycle COD ranged from 2000-4000 ppm COD with an OL of 0.04 - 0.08 lb COD/ft^3/day. The feed to the unit was 100% equalization basin liquid.

Aerobic Digestion of Ultrasonically Ruptured Cells (Akin, Stasney) - This study has also been discontinued due to priorities. More data is required before conclusions can be drawn as to the feasibility of this type of operation. This study will resume when time permits.

Methanol Oxidation

J. F. Glenister, T. A. Jasek, P. W. Young

Unit Support (Glenister, Young) - Support was provided at MO-II to resolve a problem with the unit lab GC analysis. A new column was installed, and the electronics of the gas chromatograph were checked by instrument maintenance personnel. The problem has been resolved.

Vent analyses were furnished to MO-II and MO-IV Operations personnel for their efficiency calculations.

Summary of Product Color in MO Units (Young) - MO-IV production had good color all month at <5 APHA.

MO-II (T-297) production showed a few days of "moderate" levels of color at 6-9 APHA. There was one day of a 12 color. A spot resample gave a color of 5. The iron in the samples, as analyzed by atomic absorption, was <0.1 ppm.

Loss of Reaction in Reactors at MO-II (Jasek, Young) - There have been three incidents during the latter half of the month in which partial, temporary deactivation of silver catalyst has occurred in 2-7 reactors at MO-II. In all cases, the same reactors were involved. All catalyst beds were of the same age and had just been installed when the first incident occurred. This "early" problem with the "new" beds may have made these catalysts more susceptible to the later deactivations.

T-185 OH was the prime suspect in the first incident. Samples of T-185 OH were collected along with samples from the decanter. A solid, scummy layer was observed on the decanter samples. The solid is suspected to be lauric acid - from bis-ethylene glycol dilaurate, the antifoam used in the polyol units. Other analyses of these samples were inconclusive. T-202 OH from the polyol unit was also analyzed for 2-ethyl-2-hexenal. The levels of the hexenal were normal (approximately 0.04 wt%).

The other two incidents occurred while T-185 OH was not being fed to the unit. Samples were taken around the system (including T-240 OH) shortly after the latest incident. Analyses of the samples are incomplete and thus far are inconclusive.

The reason(s) for the partial deactivation is unknown. Samples of the silver catalyst which deactivated were sent to CCCTC for surface analysis. Additionally, a sample of catalyst of approximately the same age which did not deactivate was also sent to CCCTC for surface analysis. This reactor had been brought down because of a reactor crack.

The surface analysis did not indicate any substantial difference between the catalyst that did not deactivate and one of the catalysts which deactivated. The other catalyst which deactivated did show a difference, but within acceptable limits for the analysis (based on previous surface analysis). However, these differences did not indicate any sort of surface contamination.

Sodium in MO-II, T-297 Residue (Glenister, Young) - An increase in the sodium (Na) level in T-297 residue occurred beginning September 1st. Normal sodium levels are <0.1 ppm as determined by Atomic Absorption Spectroscopy. The initial increase to approximately 4 ppm suggested a possible cooling water leak, but analysis for Ca indicated <0.1 ppm (an increased level of Ca is typically used to indicate such a leak). Analysis of the lower, upper and middle recycles of T-296 Absorber showed Na levels <0.1 ppm indicating the source was fed directly to the distillation column. The day following this work the Na level in the residue increased to 102 ppm. It was found that the vaporizer blowdown in MO-IV, which is fed to MO-II, contained 600-800 ppm Na. The crude MeOH feed to MO-IV had a Na concentration of 12-15 ppm.

Some problems were found with the caustic addition in MS. Since these have been corrected, the sodium levels in T-297 residue have decreased from a high of 123 ppm to 2 ppm. The crude MeOH feed to MO-IV is down from a high of 21 ppm to 1-2 ppm Na. The vaporizer blowdown from MO-IV is down from a high of 700-800 ppm to approximately 150 ppm.

Methanol Synthesis
J. C. Bustabad, P. W. Young

Monoethanolamine (MEA) Chemcel Methods (Bustabad) - Methods for MEA concentration, loading, and heat stable salts were revised and issued as Chemcel Plant methods. (JCB-15-82).

Unit Support (Young) - Support was provided during dew-point determinations of the natural gas before and after the guard beds by CCCTC personnel on September 16.

Samples of natural gas at the inlet and at the outlet of the sulfur guard beds for the DPG reformer were sent to CCCTC for analysis of sulfur components. Although on-line analyzers are not indicating sulfur breakthrough, there appears to be some poisoning of the reformer catalyst. Undetected sulfur compounds are suspected.

Oxo, Butanol
T. A. Jasek

Low Pressure Vent Compressor Gas Analysis - The Laboratory analyzed the Low Pressure Vent compressor gas stream as requested by Process Engineering. A leak in the seals on the compressor was suspected and confirmed. This results in a loss of propene efficiency. The analytical results were transmitted to Process Engineering.

Improved Rhodium Recovery from the Oxo Catalyst Solution - Work was completed on the comparison of using sodium chloride and potassium chloride in the ashing of catalyst solution. Results indicate an ash concentration of approximately 2.0 wt% Rh from an initial solution of 0.18 wt% Rh. These studies were conducted at 700°F. It is believed high concentrations could be achieved if higher temperatures for the ashing could be used. However, the laboratory study was limited by the Vycor crucibles used in the study because of reaction with the sodium or potassium chloride. Metal crucibles cannot be used because of reaction with TPP or rhodium.

1,3-Butylene Glycol

J. C. Bustabad

Alternate Catalysts - Equipment was assembled for hydrogenation studies using Degussa's BK 113W and Grace's 3100 Raney Nickel. A capillary-column GC method was developed for analyzing the hydrogenation reactor mixture. The studies will begin as soon as feed is available from the 1,3-BG Unit.

Pentaerythritol

D. R. Akin, J. C. Bustabad

PE Hydrolysis Resin Bed Support (Akin) - The sodium sulfate (Na_2SO_4) levels in the AML have gradually declined to less than 0.5 wt% on a contained-salt basis during the month of September (0.5 wt% is the VCI Spec.). The Laboratory has been working with VCI lab personnel in standardizing the Na_2SO_4 levels in the AML (DRA-31-82).

Na_2SO_4 levels have been declining because the sodium in the resin-bed feed has been consistently low (100-250 ppm Na^+) during September. This is due to good 'A' belt operation. Operations has also minimized the amount of H_2SO_4 used in the hydrolysis of PE linear monoformal (LF). Conversion of LF has been good throughout the month. Half of the month was spent on a pure run and the latter half was spent on a tech run. The unit is due to start-up on pure the second week of October.

Update Unit Lab Methods for Sodium (Bustabad) - Unit methods for sodium in the dissolve tank and neutralizer tank have been modified. These methods still include use of the Orion 901 Ionalyzer in the concentration mode. A unit method for sodium in 'B' cake is being developed to give an indication of ash in the final product. These methods will be issued in October.

Trimethylolpropane

J. C. Bustabad, T. A. Jasek

Isolation of TMP Unknowns (Jasek) - A bag from a TMP lot containing approximately 1.75 wt% unknowns (4 major unknowns) was obtained from the TMP warehouse. The unknowns were concentrated by serial recrystallization of 1.5 kg of TMP from propyl acetate. Two unknowns in the extract constitute approximately 60% of the total unknowns (based on silylation). One of the unknowns was recovered as a solid and purified (97.6% pure). This unknown was sent to the Tech Center for identification. The second unknown has evaded concentration to greater than 25%. A sample containing 25% of the second unknown was also sent to CCCTC for possible identification.

Update Unit Lab Method for Sodium (Bustabad) - Methods of analysis for sodium in T-204 residue and V-747 residue have been modified. Sodium in T-204 residue should be determined using the Orion 901 Ionalyzer in the millivolt mode; sodium in V-747 R should be determined by perchloric acid titration. These methods will be issued in October.

Miscellaneous

T. A. Jasek, P. W. Young

Fire-fighting Foam Analyses - The Laboratory received six samples of 3M's 3%-6% ATC from concentrate, representing the Plant's supply. The Laboratory conducted foam height characterization, film formation and sealability tests on each sample according to 3M's standard test. All samples met or exceeded the minimum required for an active agent. (Jasek)

Two solutions of foaming agent were received for verification of their concentration. Solutions of 3 vol.% and 6 vol.% were made using the mixing device on the fire truck. The assays were verified by refractive index comparison. (Young)

General

J. E. Russell has been on sick leave following surgery to repair a herniated disc. He is expected to return to work in early October.

Hub Thigpen attended a paraform prill meeting at the Technical Center on Friday, September 17.

SPECIAL PROBLEMS PROJECTS

Unit/Project	Chemist	Priority	Estimated Completion Date
Paraform			
Determine efficiency losses	Young	1	October 1982
Support paraform prilling unit	Thigpen	1	December 1982
Methanol Oxidation			
Improve energy efficiency with vacuum distillation of absorber product	Kerr	1	December 1982
Determine stability of unstabilized 37% HCHO at 80° or 90°F	Young	1	December 1982
Improve Unit lab method for H_2O in MeOH	Young	1	December 1982
Determine absorber vent losses	Russell	1	December 1982
Clean-up recycle streams	Russell	1	Continuing
Support Plant trial using MO-IV sidedraw in low-assay blending	Russell	1	December 1982
Develop improved washing technique for catalysts	Russell	2	February 1983
Monitor product color	Russell	2	Continuing
Monitor catalyst start-up performance	Russell	2	Continuing
Develop method for catalyst evaluation	Jasek	2	February 1983
Methanol Synthesis			
Support Plant trial of corrosion inhibitor in MEA system	Young	1	March 1983
Recover contaminated carbon using ultrasonic cleaning	Jasek	1	January 1983

003769

Unit/Project	Chemist	Priority	Estimated Completion Date
Oxo/BuOH			
Continue Rhodium Accounting	Huelle	1	Continuing
Support Plant trial using poly alpha olefin as reactor solvent	Huelle	1	March 1983
Recover Rhodium at Bishop	Huelle	2	June 1983
BOD II			
Support Operations during polyol and VCI acclimations	Akin	1	December 1982
Conduct pulse test of anaerobic reactor	Akin	1	November 1982
Evaluate aerobic digestion of sludge after ultrasonic treatment	Jasek	1	March 1983
Utilities			
Evaluate DuPont Sulfuric Acid 80%	Bustabad	1	December 1982
Determine TPP, TPPO emissions from Boiler No. 16	Akin	1	December 1982
Support deep well project	Huelle	1	December 1982
Reclaim Pond 4E	Kerr	1	December 1982
Eliminate zeolite beds	Bustabad	2	March 1983
1,3-Butylene Glycol			
Evaluate alternate catalysts	Bustabad	1	March 1983
Minimize V-778 wash-outs	Bustabad	2	Continuing
Trimethylolpropane			
Update unit lab analyses for sodium	Bustabad	1	October 1982
Identify major unknowns in product	Jasek	1	Continuing
Conduct Plant trials to optimize extractor	Bustabad	1	July 1983

Unit/Project	Chemist	Priority	Estimated Completion Date
Pentaerythritol			
Support resin-bed start-up	Akin	1	November 1982
Update unit lab analyses for sodium	Bustabad	1	October 1982
Identify UV-absorbing compound(s)	Jasek	1	Continuing
Monitor low-formal shipments to Dow	Akin	1	Continuing
Evaluate alternate filter cloths	Huelle	2	December 1982
Recover PE from VCI extract	Jasek	2	July 1984
Industrial Health			
Validate HCHO methods	Gary	1	October 1982
Assist Monsanto research team in facility study	Gary/Glenister	1	October 1982
Monitor methanol exposure	Gary	2	January 1983
Monitor HCHO around Paraform Prilling Unit	Gary	2	Continuing
HCHO exposure follow-up	Gary	2	Continuing
Monitor nuisance dust in PE	Gary	3	March 1983
Monitor silica in additional areas	Gary	3	March 1983
Survey areas for miscellaneous organics	Gary	3	February 1983
Miscellaneous			
Update Analytical Group sample schedule	Glenister	1	October 1982

LABORATORY DEPARTMENT
September 16, 1982

003772

