

CELANESE ENGINEERING RESINS
Bishop, Texas

TO: D. A. Reck

FROM: E. J. Heinz

EJH-15-83

February 8, 1983

LABORATORY DEPARTMENT MONTHLY REPORT

JANUARY, 1983

DISTRIBUTION

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Mr. G. Artripe
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Mr. R. G. Brandt
Mr. R. L. Bunkley
Mr. S. Carlile
Ms. N. A. Flaten
Mr. B. R. Garza
Mr. L. J. Gilliam
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Dr. R. M. Gronner
Mr. C. B. Halness
Mr. R. G. Hilbrich
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Ms. P. S. Maryan
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Mr. M. A. Purcell
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Mr. D. L. Wallen
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Chatham

Mr. T. C. Bohrer
Dr. M. J. Hannon

Summit

Dr. R. Dix
Mr. J. Chen

Central Files (3) - CCCTC

009694

MONTHLY SUMMARY - JANUARY

I. SAFETY

There were no accidents reported during January.

A small fire resulted when a heater bar became dislodged from a one inch extruder allowing molten Celcon purge to fall on the heater. The polymer caught fire and was quickly extinguished with a CO₂ extinguisher. No damage was sustained and measures have been taken to secure the heaters.

January EMS crew training centered around a review of caring for the trauma patient.

II. ROUTINE ANALYTICAL GROUP

- A. Physical property testing on miscellaneous Summit generated samples continues with 78 sets completed in January. There are 174 sets backlogged.

Samples compounded in Summit as part of the CAO-5 stabilizer replacement study continue to be analyzed in the Bishop Laboratory. Both routine and physical property analyses are being conducted.

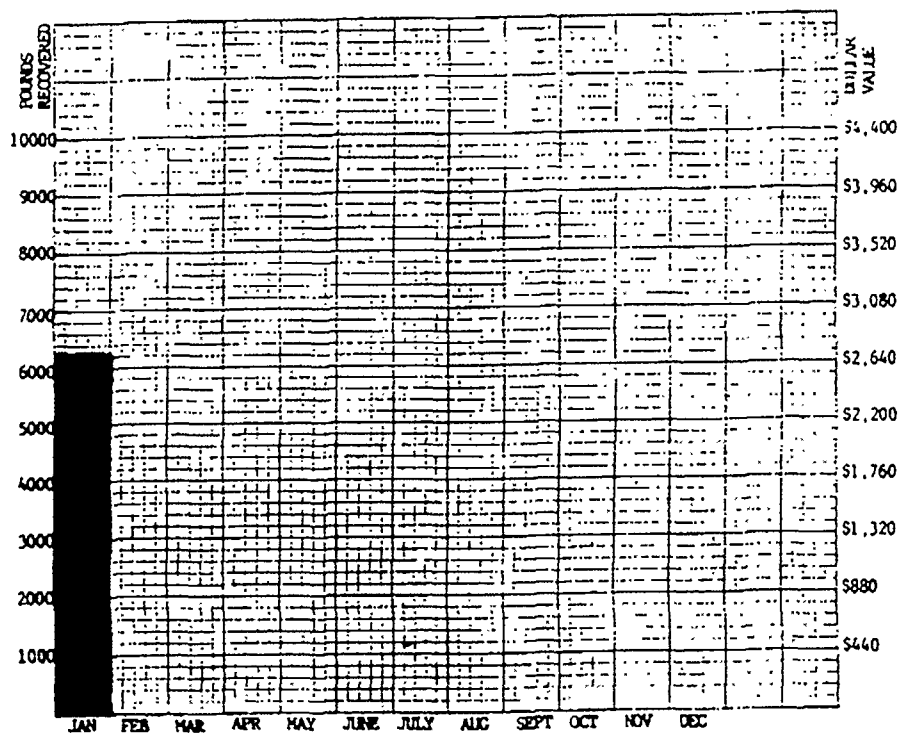
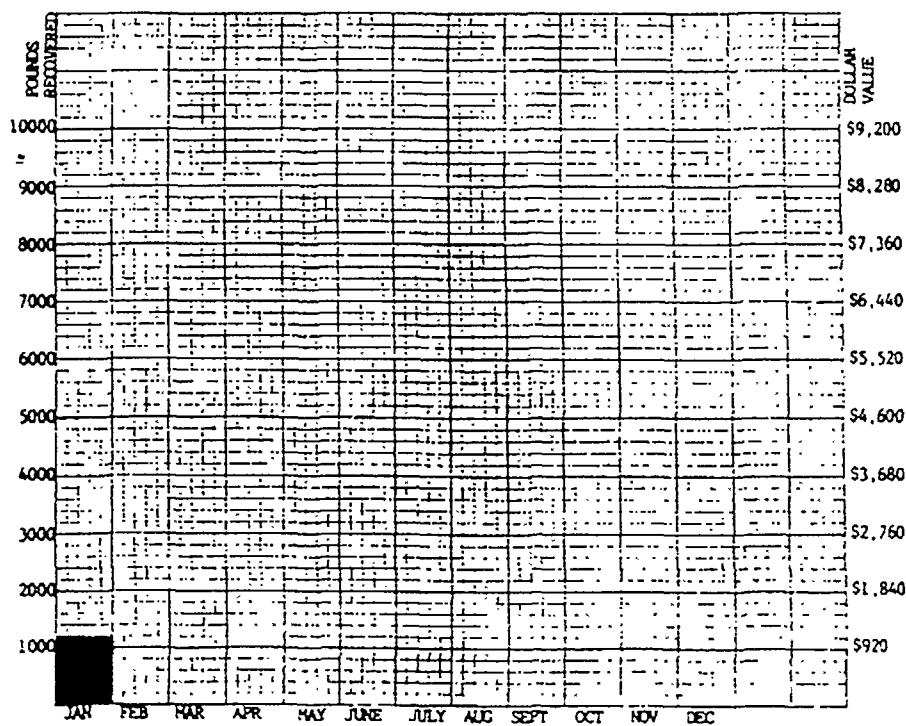
- B. The instrument lab began analyzing the fresh formaldehyde feed tanks to TO-2 daily for sodium content. High sodium levels were suspected of causing reactor conversion problems in early January. Sampling will continue until a good data base has been developed.

C. Product Recovery Program

	<u>POUNDS RECOVERED JANUARY</u>	<u>POUNDS RECOVERED YTD</u>	<u>ESTIMATED SAVINGS, YTD</u>
Celcon	6,300	6,300	2,772
Nylon	1,200	1,200	1,104
Celanex	3,300	3,300	3,201

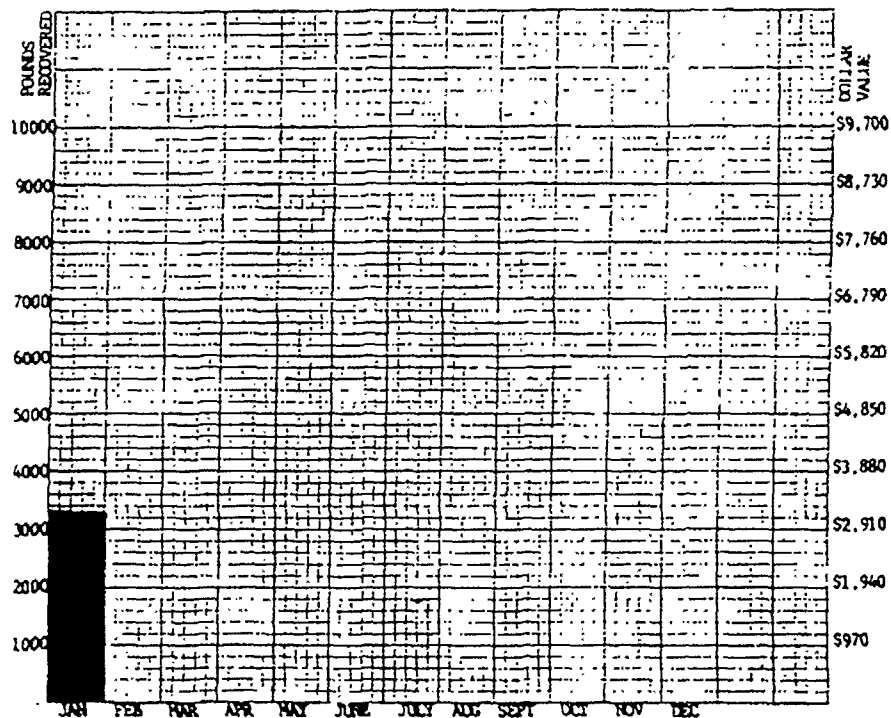
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CELCON RECOVERYNYLON RECOVERY

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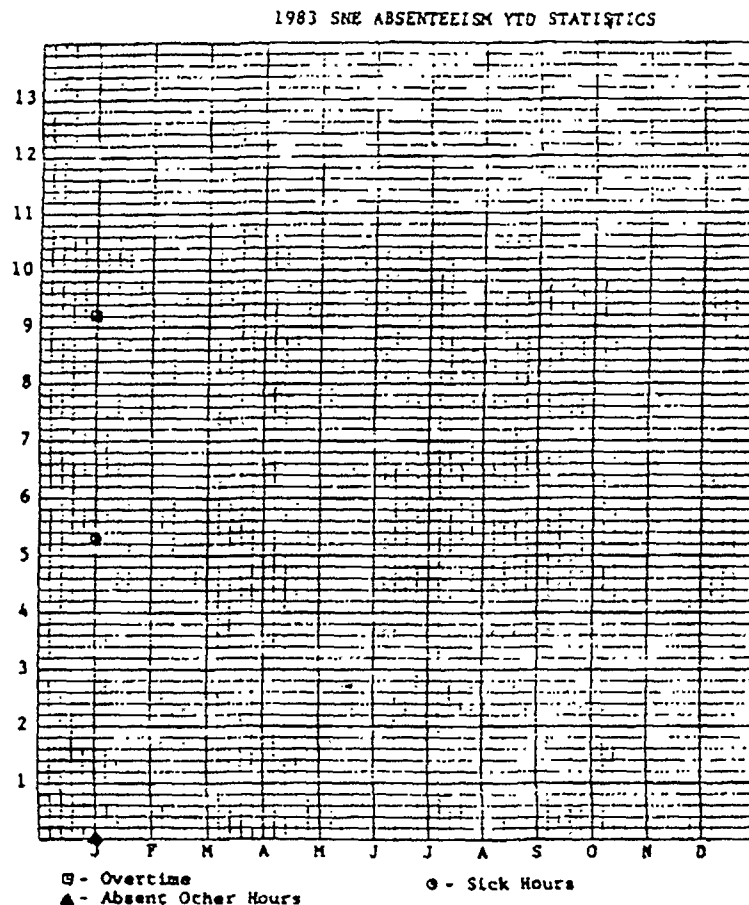
CELANEX RECOVERYIII. SNE ABSENTEE STATISTICS (As of 1-23-83)Head Count

26

Straight Time Hours3656
3656Jan.
YTDSick Hours192
192Jan.
YTD% of Straight Time5.3
5.3Jan.
YTDAbsent Other Hours0
0Jan
YTD% of Straight Time0
0Jan.
YTDOvertime Hours331
331Jan.
YTD% of Straight Time9.1
9.1Jan.
YTD

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IV. SAMPLE COUNT AND OVERTIME DISTRIBUTION - Salinas

The sample count for both the Specification and Color Laboratories for January is summarized on Tables I and II. The total laboratory sample count, Specification Laboratory sample count and the Color Laboratory sample count are graphed on Figures I, II and III respectively. The overtime distribution for this month and the Calcium Fluoride (CaF_2) inventory are shown below:

January Overtime Distribution

ECA Training	10
Safety Meeting	8
Vacation	64
Sick Leave	106
Instron	104
Evening Relief	88
Work Load	17
	397

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NYLON NUCLEANT PRODUCTION INVENTORYRaw Materials

Potassium Fluoride	64 drums (32,000 lbs.)
Calcium Acetate	65 drums (33,735 lbs.)
Nucleant (CaF_2)	13 drums
In Process (CaF_2)	17 drums

V. PERSONNEL

- A. Mr. R. de Vaudrecourt returned to work from sick leave January 20, 1983.
- B. Mr. B. Garza, Laboratory Supervisor received his 15 year service award.
- C. Ms. Lucy Lopez and Ms. Sarah Perez were hired as Laboratory Technicians effective January 3, 1983.

VI. SPECIAL PROBLEMS GROUP - R. G. Hilbrich1. Process Analyzers - Allen, Cunningham

Specifications for the analyzers will be completed in early February. Analyzer house and sample system specifications will follow and should be ready in early March.

2. Analysis of Nylon Salt in Effluent - Allen

A new analysis for nylon salt levels in the effluent is being investigated. This procedure combines a non aqueous and an aqueous titration to determine HMDA and salt concentration. Work continues.

3. Trioxane Reactor Monitoring - Gronner

The first TO-2 reactor laboratory stripout following the mid-December start-up was done after four weeks of operation. Tar level was predictably low, at <0.1 wt.%. A second stripout is currently underway.

Reactor monitoring this month also included Na analyses of the reactor contents and HCHO feed tanks. High Na was the suspected cause for reactor conversion problems. Analysis showed 2300 ppm Na in the reactor on January 4 and 3700 ppm Na on January 28. Analysis of HCHO feed tanks showed 5-10 ppm Na early in the month. After CCC replaced their ion exchange resin bed for MOIV on January 18, the level dropped to 1-5 ppm, and when the MOII resin bed was replaced by CCC on January 25, Na levels dropped to <0.1 ppm. The reduced Na in the feed will greatly reduce the rate of Na accumulation in the reactor.

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4. Finished Trioxane Stability - Gronner,

A project was initiated to determine the necessity for adding 0.5% water to finished Tox, destined for sales, as a polymerization inhibitor. A literature search turned up a 1965 CCC lab report (LD-7089-JMR) which showed that anhydrous Tox built up $\geq 2.5\%$ polymer in 24 hours, while Tox stabilized with water showed < 100 ppm polymer. These experiments were repeated in the lab to determine polymer build-up rates in Tox which has undergone two-stage finishing. After 24 hours, the "stabilized" sample had 107 ppm polymer, while the anhydrous sample had 1120 ppm. In addition, a $3\frac{1}{2}$ month old anhydrous sample had 2460 ppm, while an 11 month old "stabilized" sample had only 260 ppm. While the two-stage finished Tox appears to polymerize less, water addition still improves stability. A report will be issued.

5. Analysis of KoKneader Contaminants - Gronner, Halmess

Natural Finishing developed a problem with off-color pellets in M90-04 product from the KoKneader. Examination of the unit found light brown fines in the pellet water system, and dark brown material in the pellet dryer. These materials were analyzed in the lab. The light brown material was identified as Celcon fines, by IR and HCHO determinations. The dark brown material was found to be mostly Acrawax C. A report is being written.

6. Analysis of Irg-259 in Celcon - Gronner

Samples were collected around the KoKneader Crosshead extruder, during a C244 production run, for an Irg-259 material balance. Three samples each of product, C-244 rerun, and stabilizer blend were taken. The average Irg-259 contents found were 0.48% in product, 0.49% in the rerun, and 16.3% in the stabilizer blend. This data, along with the measured flow rates, yields 96% accountability of the Irg-259 across the extruder. Because the percent Irg-259 in the stabilizer blend was low (20% was expected), current work involves confirming the accuracy of this number.

7. CAO-5 Contamination in Irganox 259 Based M90-04 - Purcell

The products produced in Special Products, to study the effects of CAO-5 cross contamination in Irganox 259 based M90-04 products, have been analyzed and the results compiled. Mold deposit levels increase as the CAO-5 contamination concentration increases. Thermal analyses results show Irganox based polymer thermal stability decreases as CAO-5 contamination increases. GPC results show smaller Mw increases after Kd as the CAO-5 level increases. It was recommended that 0.1 wt.% CAO-5 be set as a limit in Irganox 259 based products.

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8. Irganox 259 Replacement for CAO-5, Rerun - Regrind Studies
- Halmess

Twenty-four rerun and regrind study samples prepared in Summit were analyzed for molecular weight, color, Kd, extractable HCHO, and melt index. The study was to compare rerun and regrind stability of CAO-5 and Irganox 259 based M90-04 products. The results did not indicate any problem in rerun or regrind stability due to the use of Irganox 259. A report was issued (CBH-1-83).

9. MH/SH Ratio - Maryan

A study is underway to determine the maximum acceptable level of MH polymer in -04 products. Data from previous studies was compiled and evaluated for trends between routine analytical, mold deposit, color stability, special analytical and the level of MH polymer. Current laboratory work involves determining the effects of MH level on initial color, color stability and mold deposit.

10. Head Space Analysis - Maryan

Samples of M90 polymer from the polymer units are being analyzed by headspace analysis for Trioxane and Tetroxane levels. The initial results confirm that MH polymer has a higher level of Tetroxane and a lower level of Trioxane than SH polymer. Work continues.

11. Thermal Analysis - Purcell

Isothermal TGA is being used to evaluate Irganox 259 based Celcon products. Present work involves analyses of various stabilizer packages for Color Services. Current work is also under way to study oxidative stabilities using a reduced purge gas flow rate. Work continues.

12. Thermal Analysis Instrumentation Evaluation - Purcell

Several manufacturers of thermal analysis instrumentation were evaluated in the Bishop Lab. Our present Mettler TA 3000 system was designed primarily to perform routine analyses which require minimum data manipulation and reduction. The Perkin-Elmer and Dupont systems offer increased operational capabilities and flexibility, automated data analysis, data storage, and the ability to optimize data presentation. Numerous applications have been identified for polymer monitoring and additive analyses. Purchase of the Perkin Elmer TGS-2 TGA module along with the TADS Data Station will be recommended.

009701

13. Effect of Rerun on Celanex 3300 Natural - Halmess

A trial was run on Phase I on January 3-4 while producing Celanex 3300 natural. The objective of the trial was to determine what effect the addition of dried start-up material had on the physical properties of Celanex. The results of the trial indicate that feeding 20% dried (<0.1 wt. % volatiles) rerun does not decrease physical properties. Throughout the trial the tensile values for the Celanex 3300 remained above 19,200 psi, which is ~1,400 psi higher than the 1981 yearly average. A report will be issued.

14. Celanex 3300 Black for Delco Electronics - Halmess

During January a production run of Celanex 3300 black was made on Phase I for Delco Electronics. The melt rheometer results of the two railcars produced were above the specification of 3,000 poise minimum. Since there has been some difficulty meeting the melt rheometer specification, the conditions during this production run were compared to previous production runs. Two differences noted were that during the current production run a slower screw speed was used (70 vs ~78ppm) and a lower feed rate was used (3,200 vs. ~3,600 lbs/hr). A trend in the analytical results was also noted. Railcars that had a tensile value above 18,500 psi typically passed the melt rheometer test while railcars with a tensile value below 18,500 psi failed the melt rheometer. A report will be issued.

15. EPA Toxicity Testing of Pigments - Flaten

The results of the toxicity testing were received from Core Laboratories. The results of the testing show that when the pigments are encapsulated in polymer they are no longer classified as hazardous waste. A report discussing these results is being reviewed.

16. Nucleant Contamination in Non-nucleated Nylon Analysis
- Flaten

A method for screening nucleant contamination in non-nucleated natural nylon formulations has been developed. Significant interferences were found to occur in black formulations. The method is an AA determination of Calcium levels. Several attempts to overcome these interferences were studied. None gave satisfactory results so the method was deemed unsuitable for black formulations. Alternative methods for determining nucleation contamination in black formulations will be investigated.

009702

17. Raw Material Program - Flaten

A program is being implemented to review each raw material used at CER. Required specifications, testing procedures and testing frequency will be determined for each raw material.

VII. INDUSTRIAL HYGIENE - Pritchard1. CER Monitoring Program

The CER Industrial Hygiene Monitoring Program has been revised for 1983. Quarterly surveys will be conducted in each area for each contaminant found in those areas. This will result in better documentation of exposures due to variables in operational procedures as well as environmental conditions (changes in wind direction, temperature, etc.). Previously "Block" sampling was used to document exposures in particular units over shorter periods of time. First quarter emphasis will be on formaldehyde monitoring as part of the Phase I formaldehyde reduction program.

2. Formaldehyde Monitoring

Personnel in the SH-1/TO-1, SH-2/TO-2, and MH-2 units have been monitored for formaldehyde exposure. Additional monitoring will be conducted in these as well as other units with a potential formaldehyde exposure problem.

3. Asbestos Monitoring in Filled Resins Area

Asbestos monitoring has begun in the Filled Resins Area in order to determine potential areas or tasks requiring the use of protective equipment and/or administrative controls. Additional monitoring will be conducted in this and other areas where asbestos are used.

4. Number of Monitoring Samples by Area

	<u>Month</u>	<u>YTD</u>
SH-1/TO-1	4	4
SH-2/TO-2	4	4
MH-2	3	3
Special Pproducts	--	--
Black Concentrate Unit	--	--
Natural Finishing	--	--
Filled Resins	--	--
Laboratory	--	--
ABI	--	--
TOTAL	14	14

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VIII. LABORATORY PROJECT ASSIGNMENTS

<u>Project Title</u>	<u>Chemist</u>	<u>Estimated Project Completion Date</u>
TO Process Analyzers	Allen, Cunningham	6/1/84
Nylon Salt Effluent Analysis	Allen	4/1/83
Trioxane Reactor Monitoring	Gronner	As required
Trioxane Stabilization	Gronner	4/1/83
Extractor Reactor Process Evaluation	Gronner	1/1/84
Analysis of Irganox 259 in Celcon	Gronner	4/1/83
CAO-5 Contamination in M90-04	Purcell	2/1/83
Thermal Analysis Method Development	Purcell	9/1/83
New Instrument Evaluation	Purcell	1/1/84
MH/SH Ratio Study	Maryan	4/1/83
Irganox 259 Replacement of CAO-5	Maryan	4/1/83
Headspace Analysis of Celcon Polymer	Maryan	6/1/83
Celanex Physical Property Decline	Halmess	9/1/83
Gel Permeation Chromatograph	Halmess	6/1/83
Raw Material Program	Flaten	1/1/83
Nucleation Contamination in Nylon	Flaten	7/1/83
1983 Environmental Monitoring	Pritchard	1/1/84

E J Heinz

E J. Heinz

EJH/lat
Attachments

009704

CER SPECIFICA LABORATORY
SAMPLE COUNT

MONTH January, 1983

Area	No. Sples. Celcom®			No. Sples. Celanex®			No. Sples. Nylon			Total (All Types)	% of Total (All Types)
	Routine	Non Routine	Total	Routine	Non Routine	Total	Routine	Non Routine	Total		
TO-1	542	47	589						589	589	13.69
TO-2	564	71	635						635	635	14.76
SH-1	178	0	178						178	178	4.14
SH-2	200	28	228						228	228	5.30
MH-2	257	32	289						289	289	6.73
Natural Finishing	553	73	626						626	626	14.55
Special Products	586	7	593						593	593	13.78
Celanex				391	64	455			455	455	10.58
Pilot Plant	0	0	0						0	0	
Raw Materials	251	0	251						251	251	5.83
QC	29	0	29						29	29	0.67
Nylon							423	6	429	429	9.97
Others									0	0	0
TOTAL MONTH	3160	258	3418	391	64	455	423	6	429	4302	100.00
TOTAL YTD			3418			455			429	4302	

Total Samples
(All Types) 4302

Routine	3974	92.38
Non-Routine	328	7.62
Total Sples YTD	4302	

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CER COLO. ORATORY
SAMPLE COUNT

MONTH January, 1983

Area	No. Sples. Celcon®			No. Sples. Celanex®			No. Sples. Nylon			% of Total (All Types)
	Routine	Finished Lots	Total	Routine	Finished Lots	Total	Routine	Finished Lots	Total (All Types)	
SH-1	118	0	118						118	3.03
SH-2	94	0	94						94	2.42
MH-2	96	0	96						96	2.47
Natural Finishing	120	634	754						754	19.38
Special Products	276	406	682						682	17.53
Celanex				395	707	1102			1102	28.32
Nylon							183	389	572	14.70
Specials	77	0	77	117	0	117	93	0	287	7.38
Summit Samples	67	0	67	11	0	11			78	2.00
Monitors	24	0	24	84	0	84			108	2.77
TOTAL (MONTH)	872	1040	1912	607	707	1314	276	389	3891	100.00
TOTAL (YTD)			1912			1314			665	

Total Samples
(All Types) 3891

Rout ne 1755 45.10
Finished Lots 2136 54.90
Total Sples YTD 3891

Reg. T.M.

009706

CER LABORATORY TOTAL SAMPLES
1983

Figure I

13,000

12,000

11,000

10,000

9,000

8,000

7,000

6,000

5,000

4,000

1982 AVERAGE

009707

J F M A M J J A S O N D

CER SPECIFICATION LABORATORY TOTAL SAMPLES
1983

Figure 11

9,000

8,000

7,000

6,000

5,000

4,000

3,000

2,000

1,000

0

1982 AVERAGE

009708

J F M A M J J A S O N D

CER COLOR LABORATORY TOTAL SAMPLES
1983

Figure III

9,000

8,000

7,000

6,000

5,000

4,000

3,000

2,000

1,000

0

1982 AVERAGE

009709

J F M A M J J A S O N D