Metropolitan Water Reclamation District of Greater Chicago

Protecting Our Water Environment

RESEARCH AND DEVELOPMENT DEPARTMENT

REPORT NO. 2000-6

QUALITY CONTROL ANALYSES OF POLYMERS DELIVERED TO

THE STICKNEY WATER RECLAMATION PLANT

April 2000

100 East Erie Street

Chicago, IL 60611-2803

(312) 751-5600

QUALITY CONTROL ANALYSES OF POLYMERS DELIVERED TO THE STICKNEY WATER RECLAMATION PLANT

By

John Kaschak Research Scientist I

David T. Lordi Research Scientist III

Stanley Soszynski Research Scientist I

Bernard Sawyer Coordinator of Research

Prakasam Tata Assistant Director of Research and Development Environmental Monitoring and Research Division

> Cecil Lue-Hing Director of Research and Development (Retired)

Research and Development Department Richard Lanyon, Director

April 2000

TABLE OF CONTENTS

	Page
LIST OF TABLES	iii
LIST OF FIGURES	iv
ACKNOWLEDGMENTS	vi
DISCLAIMER	vi
SUMMARY AND CONCLUSIONS	vii
INTRODUCTION	1
Background	1
Description of Program	2
METHODS	3
Viscosity	3
Total Solids	3
Active Total Solids	4
FTIR Spectra	5
RESULTS	6
Secodyne 7250	6
Allied Colloids 7952AE - 1997	9
Polydyne LE891	9
Allied Colloids 7952AE - 1998	12
FTIR Spectra	12

i

.

TABLE OF CONTENTS (Continued)

	Page
DISCUSSION	14
Viscosity	14
Active Total Solids	19
Recommendations	26
APPENDICES	
Quality Control Results for Polymers Used Dur- ing June 9, 1997 Through October 29, 1997	AI-1
Quality Control Results for Polymers Used Dur- ing November 5, 1997 Through January 26, 1998	AII-1
Quality Control Results for Polymers Used Dur- ing January 28, 1998 Through May 29, 1998	AIII-1
Quality Control Results for Polymers Used Dur- ing June 7, 1998 Through September 30, 1998	AIV-1

LIST OF TABLES

.

Table No.		Page
4	Polymers Used at Stickney WRP Centri- fuge Facility	7
2	Summer Polymer Secodyne 7250, Results of Quality Control Tests, June 9, 1997 Through October 29, 1997	8
3	Summer Polymer Allied Colloids 7952AE, Results of Quality Control Tests, No- vember 5, 1997 Through January 26, 1998	10
4	Winter Polymer Polydyne LE891, Results of Quality Control Tests, January 28, 1998 Through May 29, 1998	11
5	Summer Polymer Allied Colloids 7952AE, Results of Quality Control Tests, June 1, 1998 Through September 29, 1998	13
AI-1	Quality Control Results for Secodyne 7250 - June 9, 1997 Through October 29, 1997	AI-1
AII-1	Quality Control Results for Allied Col- loids 7952AE - November 5, 1997 Through January 26, 1998	AII-1
AIII-3	Quality Control Results for Polydyne LE891 - January 28, 1998 Through May 29, 1998	AIII-1
AIV-1	Quality Control Results for Allied Col- loids 7952AE - June 7, 1998 Through September 30, 1998	AIV-1

iii

ЗI,

LIST OF FIGURES

Figure No.		Page
1	Relation of Viscosity to Active Total Solids for Secodyne 7250	15
2	Relation of Viscosity to Active Total Solids for Allied Colloids 7952AE (1997)	16
3	Relation of Viscosity to Active Total Solids for Polydyne LE891	17.
4	Relation of Viscosity to Active Total Solids for Allied Colloids 7952AE (1998)	18
5	Relation of Active Total Solids to Total Solids for Secodyne 7250 (6/9/97 - 10/24/97)	20
6	Relation of Active Total Solids to Total Solids for Allied Colloids 7952AE (11/5/97 - 1/26/98)	21
7	Relation of Active Total Solids to Total Solids for Polydyne LE891 (1/28/98 - 5/29/98)	22
8	Relation of Active Total Solids to Total Solids for Allied Colloids 7952AE (6/7/98 - 9/30/98)	23
9	Probability Plot Regression Residuals for Allied Colloids 7952AE (11/5/97-1/26/98)	27
10	Probability Plot Regression Residuals for Allied Colloids 7952AE (6/7/98-9/30/98)	28

iv

...

LIST OF FIGURES (Continued)

- Frequency Distribution of Regression Re-29 11 siduals Allied Colloids 7952AE (11/7/97-1/26/98)
- Frequency Distribution of Regression Re-12 siduals Allied Colloids 7952AE (1/7/98-9/30/98)

*

The authors wish to thank Ms. Laura Franklin and Ms. Karen Vallos for typing this report.

DISCLAIMER

Mention of proprietary equipment and chemicals in this report does not constitute endorsement by the Metropolitan Water Reclamation District of Greater Chicago.

*

SUMMARY AND CONCLUSIONS

The Metropolitan Water Reclamation District of Greater Chicago (District) uses cationic polymers to condition anaerobically digested sludge for dewatering by centrifuges. A full-scale test procedure has been developed and is used for selection of the best performing polymer, at the least cost, based upon percent cake solids and percent solids capture. Cationic Mannich polymers delivered under contract to the Stickney Water Reclamation Plant (WRP), which has the largest centrifuge complex, are routinely sampled and checked for total solids.

In order to further monitor the polymers being delivered to the Stickney WRP centrifuge complex, a more intensive monitoring program was initiated in June 1997. The program consisted of collection of grab samples of the polymer delivered by trucks three times a week. These samples were tested for viscosity, total solids, and active total solids. The active total solids were considered a measure of the actual polymer solids. In addition, Fourier Transform Infrared (FTIR) spectra were determined on each of the samples. The results reported in this study cover the period of June 9, 1997 through September 30, 1998. Different polymers are used depending on

vii

the time of year, summer or winter. The results are divided into four time periods, one for each of the polymers used, as follows:

June 9, 1997 through October 29, 1997: Secodyne 7250 (summer polymer)

November 5, 1997 through January 26, 1998: Allied Colloids 7952AE (winter polymer)

January 28, 1998 through May 29, 1998: Polydyne LE891 (winter polymer)

June 7, 1998 through September 30, 1998: Allied Colloids 7952AE (summer polymer)

The average percent total solids and percent active total solids for Secodyne 7250 were 6.0 and 4.72, respectively. The variability (two standard deviations) in concentration was approximately ±1 percent for both total and active total solids.

The average percent total solids and percent active total solids for Allied Colloids 7952AE delivered during November 5, 1997 through January 26, 1998 were 4.24 and 3.86, respectively. The variability in the concentration was ±0.75 percent for total solids and ±0.45 percent for active solids.

The average percent total solids and percent active total solids for Polydyne LE891 were 7.68 and 5.20, respectively, with a variability of ± 1.54 and ± 0.79 , respectively.

viii

The average percent total solids and active total solids for Allied Colloids 7952AE delivered during June 7 through September 30, 1998 were 4.85 and 4.03, respectively, with a variability of ±1.07 and ±0.85, respectively.

The Secodyne and Polydyne polymers had higher percent total solids and active total solids than the Allied Colloids polymer; Polydyne LE891, the winter polymer, had the highest solids concentrations.

All of the polymer samples tested had viscosities less than the upper limit of 60,000 centipoises specified in the polymer contract.

The FTIR spectra for the samples of a given polymer showed that the polymer delivered at different times exhibited similar spectra, and have the same composition as the polymer specified in the contract.

Although a regression analyses of active total solids to total solids showed a significant relationship, the correlation coefficients were so low that the active total solids cannot be predicted from the total solids. In addition, the statistical regression residuals data, that is, the differences between the statistically predicted values and the actual values, was shown to have a uniform distribution, rather than a normal distribution. Thus, neither total nor active

ix

total solids concentration of a single sample from a batch of polymer delivered should be used as the sole basis for its rejection.

The evaluation of the polymer's actual performance at the Stickney WRP centrifuge complex should continue to be used as the criteria for fulfilling the contract.

х

INTRODUCTION

Background

The Metropolitan Water Reclamation District of Greater Chicago (District) spends more than three million dollars annually for polymers used to condition anaerobically digested sludge which is dewatered by centrifuges. A full-scale test procedure has been developed and is used for selection of the best performing polymer, at the least cost, based upon percent cake solids and percent solids capture.

It was observed that the polymer dosage required to dewater the digested sludge at the Stickney WRP fluctuates widely between summer and winter. It was determined that the major factor was the alkalinity of the sludge. Thus, two different polymers are used at the Stickney WRP centrifuge complex, one for the summer period and another for the winter period.

Upon selection of a particular polymer during the course of the polymer contract, delivered polymer was sampled and checked for total solids and viscosity. A viscosity upper limit of 60,000 centipoises is specified in the contract to ensure that the polymer is pumpable. Originally, additional testing for active polymer solids and FTIR spectra were done only if Maintenance and Operations personnel expressed concern

that the polymer was not performing satisfactorily. Because of the large quantity of polymer being purchased for use at the Stickney WRP, it was decided to implement an expanded quality control program to test polymer shipments as received. This report describes the program which was initiated at the Stickney WRP in June 1997, and presents the results obtained for the period of June 1997 through September 1998.

Description of Program

The quality control program which has been carried out by the District since June 1997 involves collecting grab samples of polymer from random truck deliveries as the liquid polymer is pumped into the holding tank at the Stickney WRP centrifuge complex. Three separate truckloads out of approximately 15 deliveries per week are sampled for analysis. The samples are taken to the Dr. Cecil Lue-Hing Research and Development Complex for analysis of total solids, active total solids, and viscosity. In addition, an FTIR spectrum is determined on each sample. The total solids and active total solids are determined by the Analytical Laboratories Division, and the viscosity and FTIR spectra are determined by the Wastewater Treatment Research Laboratory.

METHODS

Viscosity

A Brookfield Digital Viscometer (Brookfield Engineering Laboratory, Inc., Model LVTDV-1) was used to measure the viscosity in centipoises at 25°C. A 400 mL polymer sample was tested, with a no. 4 spindle at a speed of 12 rpm. (Brookfield Engineering Laboratories, Operating Instructions Manuals M/85-150-D and M/85-160-G.)

Total Solids

The determination of total solids was carried out by a conventional oven method. The basic steps in the determination are as follows:

- 1. Set oven temperature at 70°C.
- Dry aluminum dish or watch glass in oven for a minimum time of 60 minutes.
- 3. Measure the net weight of dried aluminum dish or watch glass.
- 4. Spread approximately 5 gms of sample evenly and thinly in the dish or glass.
- 5. Measure the wet sample weight.
- 6. Heat in the oven until constant weight is obtained (approximately 1.5 to 2.5 hours).

7. Percent total solids is calculated as:

total solids = $\frac{\text{wt of dried sample}}{\text{wt of wet sample}} \times 100$

Active Total Solids

There is no one standard method for the determination of the active polymer solids. Thus, for the purpose of testing the mannich-type polymers delivered to the Stickney WRP centrifuge facility, a test procedure used in the polymer industry for measuring active polymer solids has been adopted. This test procedure involves the addition of an organic solvent (acetone) to the polymer, separating and discarding the solvent phase to remove non-polymeric solids from the insoluble residue (the polymer), drying the residue, and weighing it as active polymer content. The basic steps in the procedure are as follows:

- 1: Dry glass beaker in the oven at 70°C. Measure the (net) dried beaker weight.
- 2. Weigh out approximately 5 gms of sample into a 250 mL glass beaker. Add 95 gms of acetone to sample in beaker. Use a spatula or glass rod to spread the sample and bring the sample into full.

contact with the acetone, approximately 15 minutes.

3. Drain out the acetone solution. Dry the polymer solids in the beaker in the oven at 70°C until constant weight is obtained (approximately one to two hours). Calculate the percent polymer active solids.

% polymer active solids = $\frac{\text{wt of dried polymer}}{\text{wt of wet sample}} \times 100$

The percent polymer solids determined as above may be considered as the active polymer solids of the polymer product.

FTIR Spectra

The FTIR spectra of the polymer samples were determined using a Mattson Galaxy 3020 FTIR spectrophotometer. (Mattson Instruments, Galaxy Series 3000, 5000, 7000 FTIR Spectrometer User's Manual and U-First Software User's Manual.)

RESULTS

During the period of June 1997 through September 1998, three different polymers were used at the Stickney centrifuge sludge dewatering facility as shown in Table 1. The Secodyne 7250 and Allied Colloids 7952AE polymers were originally tested and considered as summer polymers, and the Polydyne LE891 was tested as a winter polymer. Three random shipments out of approximately 20 per week were sampled upon delivery and analyzed for viscosity, total solids, and active total solids according to the procedures previously described. The FTIR spectra of these polymers were also determined. The individual results for each of the polymers are presented in Appendix A. These tables show the date of the sample, the bill of lading number for the truck delivery, as well as the results of the viscosity, active total solids, and total solids analyses.

Secodyne 7250

A summary of the quality control tests for the samples of Secodyne 7250 which were analyzed over the period of June 9, 1997 through October 29, 1997, is presented in <u>Table 2</u>. All of the samples had viscosities less than the allowable maximum contract specified limit of 60,000 centipoises, with an

TABLE 1

POLYMERS USED AT STICKNEY WRP CENTRIFUGE FACILITY

Polymer	Period of Usage
Secodyne 7250	June 6, 1997 - October 29, 1997
Allied Colloids 7952AE	November 5, 1997 - January 26, 1998 and June 1, 1998 - September 30, 1998
Polydyne LE891	January 28, 1998 - May 29, 1998

TABLE 2

* SUMMER POLYMER SECODYNE 7250 RESULTS OF QUALITY CONTROL TESTS JUNE 9, 1997 THROUGH OCTOBER 29, 1997

Parameter	Minimum	Maximum	Average	Median Value	Standard Deviation
Viscosity, centipoises	19,850	48,500	33,141	32,500	6,222
Total Solids, percent	5.17	7.11	6.0	5.86	0.51
Active Total Solids, percent	3.85	5.74	4.72	4.70	0.45
Ratio Active Solids/Total Solids	0.609	0.896	0.789	0.797	0.051

Number of samples = 58.

2:

 ∞

average value of 33,141 centipoises. The total solids and active solids averaged 6.0 and 4.72 percent, respectively, with the average ratio of active solids to total solids being 0.789, or 78.9 percent of the total solids were active solids. The average and median values for each of the measured parameters were very close, indicating that the data were generally normally distributed.

Allied Colloids 7952AE - 1997

<u>Table 3</u> presents a summary of the results for the Allied Colloids polymer which was used from November 5, 1997 through January 26, 1998. All samples had acceptable viscosities and were less than the maximum contract specified limit of 60,000 centipoises. The average and median values of each of the measured parameters were approximately the same. The average percent total solids was 4.24 and the average percent active solids was 3.86 percent. The average ratio of active to total solids was 0.914, or 91.4 percent of the total solids.

Polydyne LE891

Table 4 presents the quality control test results for the Polydyne LE891 polymer which was used during the winter period of January 28, 1998 through May 29, 1998. All of the samples had acceptable viscosities within the maximum contract

TABLE 3

*SUMMER POLYMER ALLIED COLLOIDS 7952AE RESULTS OF QUALITY CONTROL TESTS NOVEMBER 5, 1997 THROUGH JANUARY 26, 1998

Parameter	Minimum	Maximum	Average	Median Value	Standard Deviation
Viscosity, centipoises	14,900	43,000	23,211	22,525	7,164
Total Solids, percent	3.45	5.07	4.24	4.21	0.375
Active Total Solids, percent	3.38	4.34	3.86	3.86	0.229
Ratio Active Solids/Total Solids	0.757	1.003	0.914	0.935	0.070

Number of samples = 32.

TABLE 4

WINTER POLYMER POLYDYNE LE891 RESULTS OF QUALITY CONTROL TESTS JANUARY 28, 1998 THROUGH MAY 29, 1998

Parameter	Minimum	Maximum	Average	Median Value	Standard Deviation
Viscosity, centipoises	28,250	55,000	44,372	44,800	5,118
Total Solids, percent	6.45	9.81	7.68	7.59	0.773
Active Total Solids, percent	4.35	6.07	5.20	5.16	0.394
Ratio Active Solids/Total Solids	0.560	0.833	0.680	0.681	0.053

Number of samples = 49.

specified limit of 60,000 centipoises specified in the contract, with an average of 44,372 centipoises. The average percent total solids was 7.68 and the average percent active solids was 5.20 percent, with an average ratio of active to total solids of 0.680 or 68 percent. The average and median values of each of the parameters were almost identical.

Allied Colloids 7952AE - 1998

<u>Table 5</u> presents a summary of the Allied Colloids 7952AE polymer which was used during the summer period of June 1, 1998 through September 29, 1998. As with the other polymers, the viscosity of all samples tested was less than the maximum contract specified limit of 60,000 centipoises. The average and median values of each of the parameters measured were approximately the same. The average percent total solids was 4.85 and the average percent active solids was 4.03. The ratio of active to total solids averaged 0.833, or 83.3 percent.

FTIR Spectra

The FTIR spectra for the samples are stored in the data system. The system compares the spectra with the initial sample spectrum. This comparison showed no major differences be-

TABLE 5

*SUMMER POLYMER ALLIED COLLOIDS 7952AE RESULTS OF QUALITY CONTROL TESTS JUNE 1, 1998 THROUGH SEPTEMBER 29, 1998

Parameter	Minimum	Maximum	Average	Median Value	Standard Deviation
Viscosity, centipoises	16,350	33,000	22,422	22,500	2,952
Total Solids, percent	3.72	5.88	4.85	4.93	0.537
Active Total Solids, percent	3.12	5.16	4.03	4.10	0.424
Ratio Active Solids/Total Solids	0.718	0.987	0.833	0.824	0.071

Number of samples = 51.

à:

μ ω

DISCUSSION

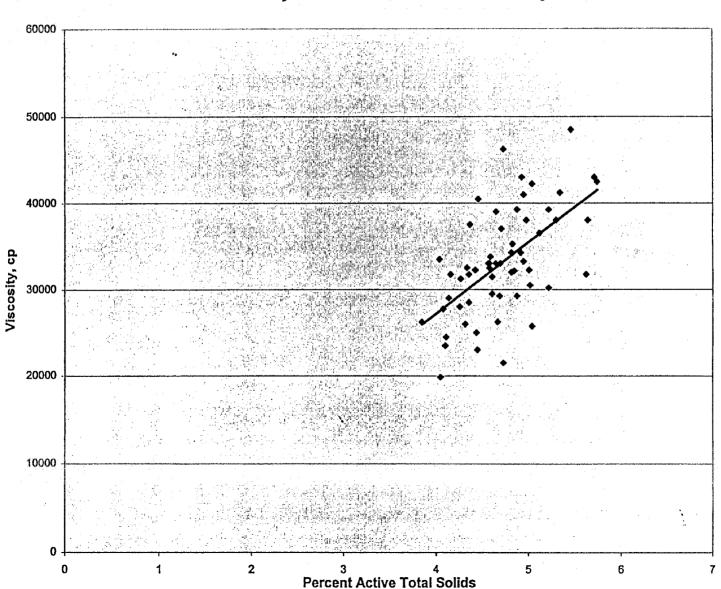
Viscosity

While the primary purpose of measuring the viscosity of the different polymers being supplied was to ensure the pumpability of the polymer (viscosity lower than 60,000 centipoises), an effort was also made to determine whether there is a relationship between the viscosity and active total solids content of the polymer. A plot of the viscosity versus the percent active total solids was made for each of the polymers sampled during this study period.

Figures 1 through 4 are plots of viscosity versus the percent active total solids for Secodyne 7250, Allied Colloids 7952AE (1997), Polydyne LE891, and Allied Colloids 7952AE (1998), respectively. In general, the viscosity rises with the increase in polymer solids. However, there is a wide scatter in the individual sets of data, so that viscosity could not be used to determine the actual percent active total solids content, or vice versa.

Similar trends were also observed when the viscosity was plotted against the percent total solids.

FIGURE 1



Relation of Viscosity to Active Total Solids for Secodyne 7250

FIGURE 2

Relation of Viscosity to Active Total Solids for Allied Colloids 7952AE (1997)

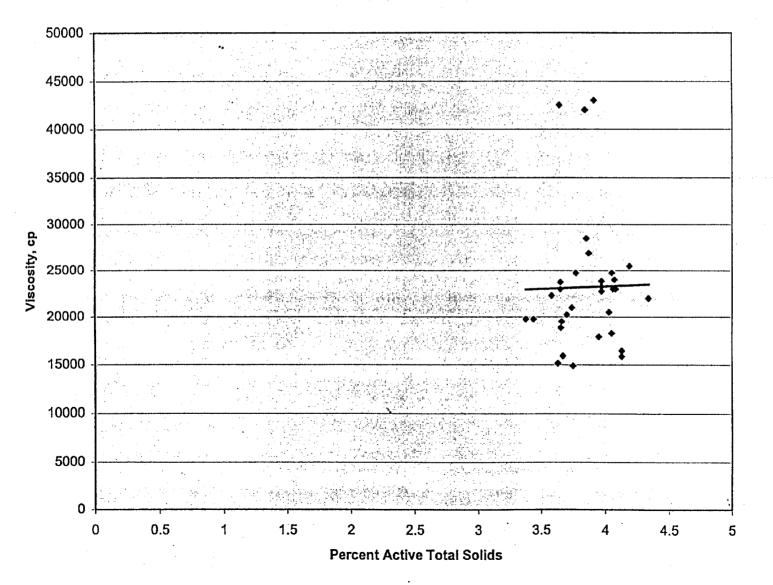


FIGURE 3

Relation of Viscosity to Active Total Solids for Polydyne LE891

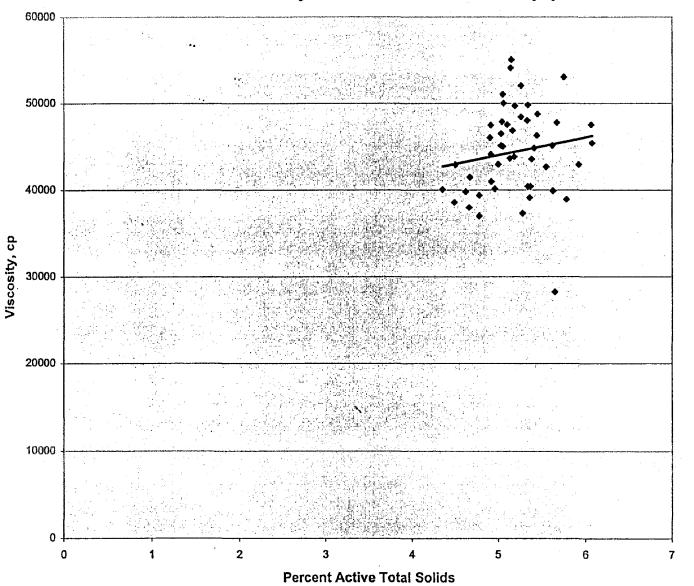
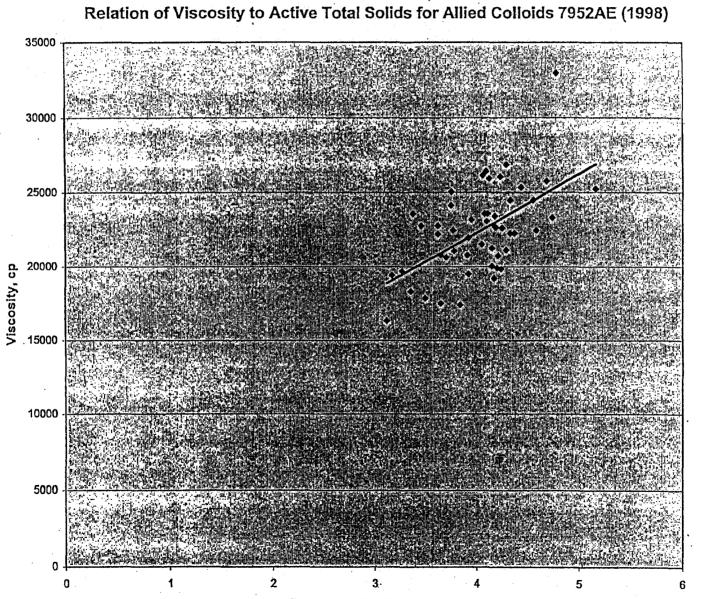


FIGURE 4



Percent Active Total Solids

Active Total Solids

Plots of the active total solids versus total solids are presented in Figures 5 through 8.

All four graphs show the relationship of <u>active</u> total solids as a linear function of total solids. Each graph shows this relationship for a particular polymer during a specific time period. Each of the four relationships is highly significant statistically, as shown by the following F, P, and R² values:

Polymer	F(DF1, DF2)	P	<u>R</u> ²
7250	F(1,56) = 21.92	1.8E-5	.281350
7952AE	F(1,30) = 8.15	7.7E-3	.213619
LE891	F(1,47) = 28.35	2.8E-6	.376138
7952AE	F(1, 49) = 48.18	8.2E-9	.495800

P is the probability of the regression line being nonsignificant, DF1 and DF2 are the degrees of freedom for the F distribution, and R^2 is the coefficient of determination which is the square of the correlation coefficient. Even though the regressions are highly significant, the correlation coefficients are low. This can be seen visually by the large variation about the regression lines in each of the plots. This means that the relationships produce poor predictions.

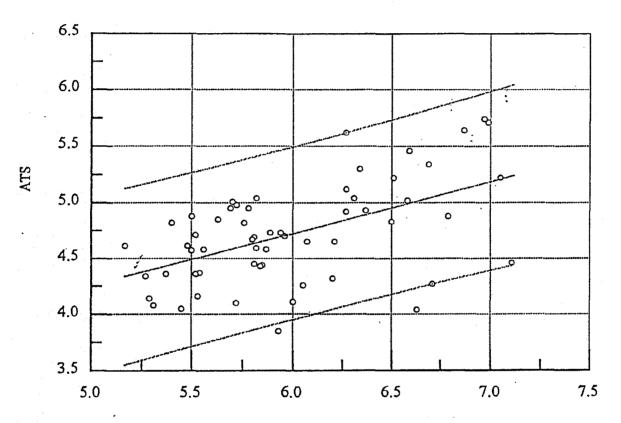
Each regression line has upper and lower 95% confidence bands. The confidence intervals are ranges between two

FIGURE 5

RELATION OF ACTIVE TOTAL SOLIDS TO TOTAL SOLIDS FOR SECODYNE 7250 (6/9/97-10/29/97)

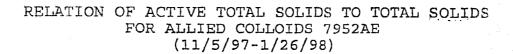
1

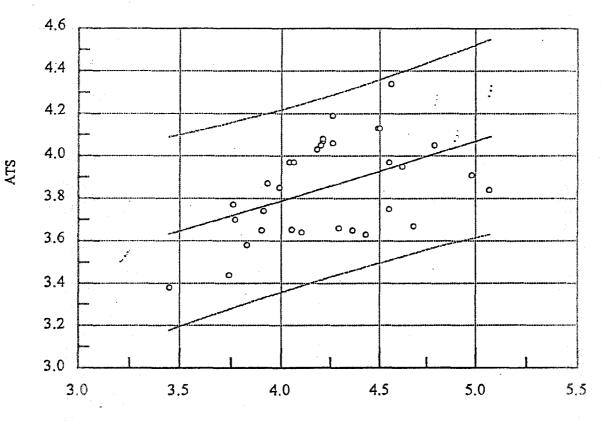
.



TS

FIGURE 6



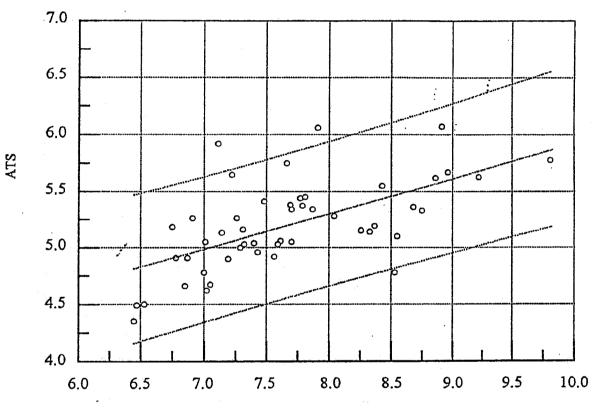


TS

21

FIGURE 7

RELATION OF ACTIVE TOTAL SOLIDS TO TOTAL SOLIDS FOR POLYDYNE LE891 (1/28/98-5/29/98)

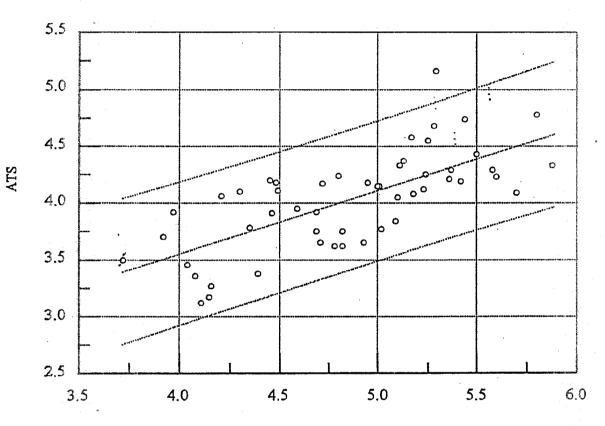


ТS

22

FIGURE 8

RELATION OF ACTIVE TOTAL SOLIDS TO TOTAL SOLIDS FOR ALLIED COLLOIDS 7952AE (6/7/98-9/30/98)



TS

23

.

limits, within which the true value of the predicted active polymer solids may be expected to be found. The two limits are called upper and lower confidence limits. The term "confidence" implies that it can be asserted with a given degree of confidence (i.e., a certain probability) that the interval includes the true value of the predicted active polymer solids at a specific value of total solids.

It is typical to use 95% as the confidence probability. Confidence intervals above 95% will be wider, while those below 95% will be narrower. The 95% confidence intervals for the four relationships are relatively wide which is consistent with the low correlation coefficients mentioned previously.

These are the general concepts behind regression confidence intervals for predicted values, but in order to make valid inferences from such confidence intervals, the underlying distributions must really be normal or at least approximately so. This is particularly important if the confidence intervals are to be used as a mechanism for rejection of active total solids for purposes of indicating atypical or outof-specification polymer batches.

It is unfortunate, but the fact that the samples of polymer are from different trucks all but guarantees that these distributions will not be normal. Quality control

professionals cope with such problems by taking more than one sample and applying the central limit theorem in their data analysis. This theorem states that regardless of the shape of the distribution being sampled, the distribution of averages of random samples taken from this distribution will be essentially normal (exactly normal as the number of samples approaches infinity).

If three or four samples of polymer were taken from each truck (instead of just one), the <u>average</u> of the total solids values from those three or four samples would, for all practical purposes, have normal distributions. Then these averages can be applied to control charts or confidence intervals, which can then legitimately be used for rejection of atypical or out-of-specification polymer batches.

The regression residuals, which are the differences between the regression equation model values and the actual data points, were calculated for Allied Colloids polymer 7952AE (in two different time periods). To demonstrate the fact that the polymer samples are not normally distributed, the regression residuals from polymer are analyzed for their distribution characteristics. The regression residuals must have normal distributions if the 95% confidence intervals are to be valid.

In both periods, the regression residuals are analyzed by probability plots and by histogram plots. The probability plots, <u>Figures 9</u> and <u>10</u>, show the telling signals of uniform distributions: instead of plotting in a straight line, the regression residuals come down at the left end (deviate from the straight line) and come up at the right end (again deviating from the straight line). Both histograms <u>Figures 11</u> and <u>12</u> visually show that the regression residuals have uniform distributions rather than normal (bell-shaped) distributions.

Thus, the 95% confidence bands should not be drawn for these linear regression equations, nor are they to be used for making inferences regarding rejection of polymer batches.

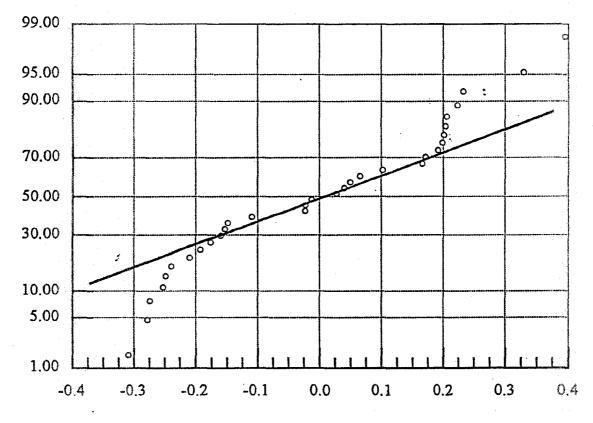
Recommendations

The contract documents for furnishing and delivering polymer gives the District the option to test polymer samples, but does not set specific numerical limits on any parameter except viscosity. Based on the results of this study, it is recommended that:

 The present procedure for terminating a contract based upon performance with regard to dosage, cake solids, and percent capture be continued.

FIGURE 9

PROBABILITY PLOT REGRESSION RESIDUALS FOR ALLIED COLLOIDS 7952AE (11/5/97-1/26/98)



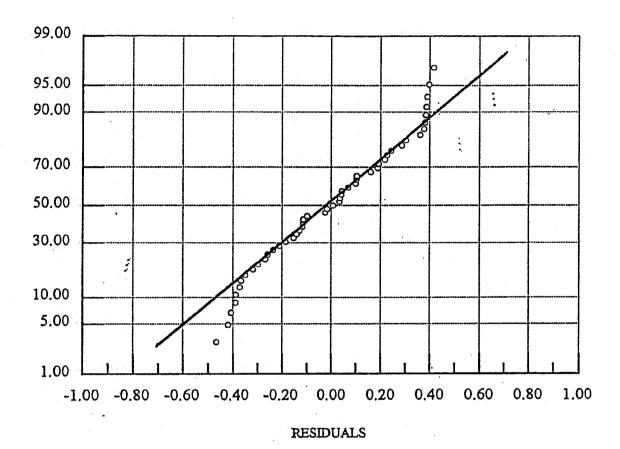
RESIDUALS

PROBABILITY

. . . *

FIGURE 10

PROBABILITY PLOT REGRESSION RESIDUALS FOR ALLIED COLLOIDS 7952AE (6/7/98-9/30/98)

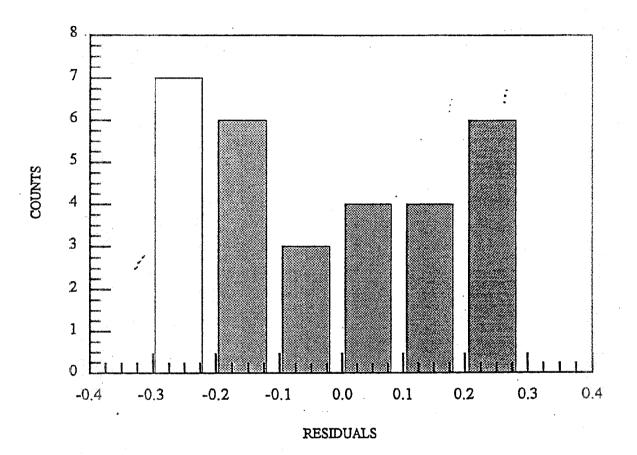


PROBABILITY

.

FIGURE 11

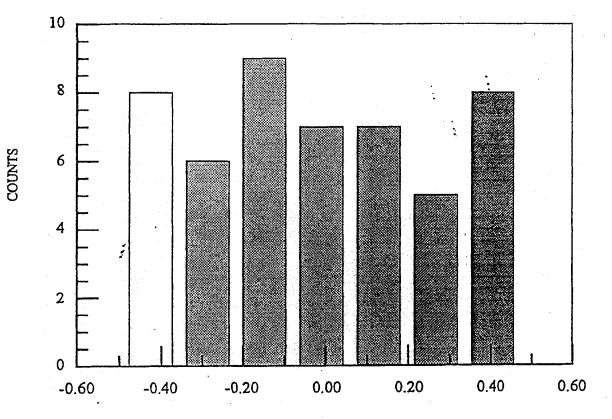
FREQUENCY DISTRIBUTION OF REGRESSION RESIDUALS FOR ALLIED COLLOIDS 7952AE (11/7/97-1/26/98)



• •

FIGURE 12

FREQUENCY DISTRIBUTION OF REGRESSION RESIDUALS FOR ALLIED COLLOIDS 7952AE (1/7/98-9/30/98)



RESIDUALS

30

. .

- 2. The determination of viscosity and FTIR spectra of samples collected at random three times a week be continued.
- 3. The present testing of each polymer delivery for total solids be continued.
- 4. These data can be used as ancillary information in support of any questions regarding performance of the polymer.

APPENDIX AI

QUALITY CONTROL RESULTS FOR POLYMERS USED DURING JUNE 9, 1997 THROUGH OCTOBER 29, 1997

TABLE AI-1

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
6/9/97	NR	1322944	38000	5.64	6.87	0.821
6/11/97	NR	1322949	42500	5.74	6.97	0.824
6/13/97	NR	1322950	43000	5.71	6.99	0.817
6/16/97	NR	1326862	41250	5.34	6.69	0.798
6/18/97	NR	1326863	39250	5.22	7.05	0.740
6/20/97	NR	1326864	39250	4.88	6.79	0.719
6/23/97	NR	1336679	43000	4.93	6.37	0.774
6/25/97	NR	1336680	48500	5.46	6.59	0.829
6/27/97	NR	1336681	38000	5.3	6.34	0.836
7/2/97	1227	1351654	32500	4.34	5.27	0.824
7/7/97	NR	1351664	33750	4.59	5.82	0.789
7/9/97	NR	1351665	32500	4.58	5.87	0.780
7/11/97	1255	1364515	33500	4.04	6.63	0 .609
7/14/97	1271	1364516	34250	4.92	6.27	0.785
7/16/97	1279	1364517	33000	4.58	5.56	0.824
7/18/97	1289	1374390	30500	5.02	6.58	0.763
7/21/97	1302	1374392	26000	4.32	6.2	0.697
7/23/97	1317	1374394	25000	4.44	5.85	0.759
7/25/97	1324	1374565	33000	4.65	6.21	0.749
7/28/97	1336	1380886	40500	4.46	7.11	0.627
7/30/97	1348	1381182	32000	4.82	5.76	0.837
8/1/97	1358	1394890	30200	5.22	6.51	0.802
8/4/97	1369	1394892	27750	4.08	5.31	0.768
8/6/97	1376	1394894	21500	4.73	5.89	0.803
8/8/97	1383	1394895	32250	4.43	5.84	0.759
8/11/97	1400	1402573	19850	4.05	5.45	0.743
8/13/97	1408	1402574	28000	4.26	6.05	0.704
8/15/97	1410	1417192	29000	4.14	5.29	0.783
8/18/97	1423	1417193	24500	4.11	6	0.685
8/20/97	1433	1417194	31750	5.62	6.27	0.896
8/22/97	1442	1417195	42250	5.04	6.31	0.799
9/25/97	1456	1423977	31250	4.27	6.71	0.636
8/27/97	1459	1423979	29250	4.69	5.81	0.807
8/29 /97	469	1423980	31750	4.16	5.53	0.752
9/3/97	1476	1431684	33250	4.95	5.78	0.856

QUALITY CONTROL RESULTS FOR SECODYNE 7250 JUNE 9, 1997 THROUGH OCTOBER 29, 1997

AI-1

TABLE AI-1 (Continued)

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
Date	Laung		VISCOSITY	Solius	001103	itatio
9/5/97	1490	1431685	29250	4.88	5.5	0.887
9/8/97	1495	1441745	37500	4.37	5.54	0.789
9/10/97	5120	1441746	46250	4.73	5.94	0.796
9/12/97	1521	1441747	23500	4.1	5.72	0.717
9/15/97	1532	1451355	36500	5.12	6.27	0.817
9/17/97	1539	1451356	32250	5.01	5.7	0.879
9/19/97	1547	1458691	38000	4.98	5.72	0.871
9/22/97	1557	1458692	29500	4.61	5.17	0.892
9/24/97	R001563	1458693	26250	4.67	5.8	0.805
9/26/97	5120	1470116	34250	4.82	5.4	0.893
9/29/97	5120-5140	1470117	28500	4.36	5.52	0.790
10/3/97	2170	1470121	26250	3.85	5.93	0.649
10/6/97	1616	1479680	31750	4.36	5.37	0.812
10/8/97	1627	1479681	33000	4.57	5.5	0.831
10/10/97	NR	1479688	32150	4.85	5.63	0.861
10/13/97	1646	1489335	23000	4.45	5.81	0.766
10/15/97	NR	1489336	25750	5.04	5.82	0.866
10/17/97	1665	1489347	39000	4.65	6.07	0.766
10/20/97	1676	1498674	31500	4.61	5.48	0.841
10/22/97	1681	1498675	37000	4.71	5.52	0.853
10/24/97	1689	1498677	35250	4.83	6.5	0.743
10/27/97	1702	1508408	41000	4.95	5.69	0.870
10/29/97	1708	1508409	33000	4.7	5.96	0.789
Average			33141	4.72	6.00	0.789
Max			48500	5.74	7.11	0.896
Min	•		19850	3.85	5.17	0.609
Std. Dev.			6222	0.45	0.51	0.067
Median			32500	4.70	5.86	0.797
Count			58	58	58	58

QUALITY CONTROL RESULTS FOR SECODYNE 7250 JUNE 9, 1997 THROUGH OCTOBER 29, 1997

NR = Not reported.

APPENDIX AII

QUALITY CONTROL RESULTS FOR POLYMERS USED DURING NOVEMBER 5, 1997 THROUGH JANUARY 26, 1998

*

TABLE AII-1

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
11/5/97	14534	1524509	22000	4.34	4.56	0.952
11/7/97	14541	1524509	25500	4.19	4.36	0.932
11/10/97	14556	1524510	24000	4.07	4.20	0.967
11/12/97	14562	1524512	22750	3.97	4.04	0.983
11/14/97	14564	1526253	23250	3.97	4.06	0.978
11/17/97	14575	1537012	23000	3.65	3.9	0.936
11/19/97	14597	1537013	21000	3.74	3.91	0.957
11/20/97	14605	1537014	20500	4.03	4.18	0.964
11/24/97	14633	1542654	23000	4.08	4.21	0.969
11/26/97	14640	1542655	23000	4.06	4.26	0.953
11/28/97	14645	1542656	24750	4.05	4.2	0.964
12/1/97	14670	1551538	24750	3.77	3.76	1.003
12/3/97	14679	1551548	20250	3.7	3.77	0.981
12/5/97	14689	1551574	19750	3.44	3.74	0.920
12/8/97	14692	1561256	26900	3.87	3.93	0.985
12/10/97	14702	1561257	28500	3.85	3.99	0.965
12/19/97	14719	1573979	22300	3.58	3.83	0.935
12/22/97	14715	1573986	19750	3.38	3.45	0.980
12/24/97	14755	1581688	18850	3.653	4.05	0.902
12/26/97	14756	1581694	16400	4.13	4.49	0.920
12/29/97	14763	1581695	15850	4.13	4.5	0.918
12/31/97	14768	1581700	15900	3,67	4.68	0.784
1/5/98	14771	1591140	17900	3.95	4.62	0.855
1/7/98	14790	1591142	19500	3.66	4.29	0.853
1/9/98	14801	1598012	18250	4.05	4.79	0.846
1/12/98	1480	1598022	15150	3.63	4.43	0.819
1/14/98	14822	1598023	14900	3.75	4.55	0.824
1/16/98	14830	1606001	23750	3.65	4.36	0.837
1/19/98	14856	1606003	23850	3.97	4.55	0.873
1/21/98	14864	1606004	42000	3.84	5.07	0.757
1/23/98	14870	1614831	43000	3.91	4.98	0.785
1/26/98	14895	1614832	42500	3.64	4.1	0.888

QUALITY CONTROL RESULTS FOR ALLIED COLLOIDS 7952AE NOVEMBER 5, 1997 THROUGH JANUARY 26, 1998

TABLE AII-1 (Continued)

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
Average			23211	3.86	4.24	0.914
Max			43000	4.34	5.07	1.003
Min			14900	3.38	3.45	0.757
Std. Dev.	•		7164	0.229	0.375	0.070
Median			22525	3.86	4.21	0.935
Count of Samples			32	32	32	32

QUALITY CONTROL RESULTS FOR ALLIED COLLOIDS 7952AE NOVEMBER 5, 1997 THROUGH JANUARY 26, 1998

AII-2

.12

÷

APPENDIX AIII

QUALITY CONTROL RESULTS FOR POLYMERS USED DURING JANUARY 28, 1998 THROUGH MAY 29, 1998

. :

TABLE AIII-1

Date	Bill of Lading	۱D	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
1/28/98	395	1614833	28250	5.65	7.22	0.783
2/2/98	422	1624963	39800	4.62	7.02	0.658
2/4/98	431	1624964	55000	5.15	8.26	0.623
2/6/98	443	1632517	54050	5.14	8.33	0.617
2/9/98	456	1632518	47500	5.1	8.55	0.596
2/11/98	464	1632519	51000	5.05	7.7	0.656
2/13/98	476	1636610	49700	5.19	8.37	0.620
2/16/98	477	1936614	42850	5.92	7.11	0.833
2/18/98	483	1636617	39400	4.78	8.53	0.560
2/20/98	494	1647657	50000	5.06	7.61	0.665
2/23/98	514	1647664	40050	4.35	6.45	0.674
2/25/98	521	1647665	47750	5.67	8.96	0.633
2/27/98	531	1655576	45400	6.07	8.91	0.681
3/3/98	542	1655598	39900	5.63	9.22	0.611
3/4/98	553	1655519	39100	5.36	8.68	0.618
3/6/98	568	1667344	45100	5.03	7.59	0.663
3/11/98	581	1667351	44800	5.41	7.48	0.723
3/13/98	589	1667352	43800	5.18	6.75 ·	0.767
3/16/98	601	1681643	52000	5.26	7.26	0.725
3/18/98	608	1681646	53000	5.75	7.66	0.751
3/20/98	625	1681647	47500	6.06	7.91	0.766
3/23/98	640	1686407	38900	5.78	9.81	0.589
3/25/98	653	1686417	47850	5.04	7.4	0.681
3/27/98	665	1686418	40400	5.37	7.79	0.689
3/30/98	676	1696285	42600	5.55	8.43	0.658
4/1/98	686	1696286	40150	4.96	7.43	0.668
4/3/98	698	1696287	46250	5.44	7.77	0.700
4/6/98	707	1706505	46000	4.9	7.19	0.682
4/8/98	712	1706506	48750	5.45	7.81	0.698
4/10/98	727	1706507	37950	4.66	6.85	0.680
4/13/98	741	1718863	40400	5.34	7.7	0.694
4/15/98	735	1718864	44050	4.91	6.78	0.724
4/17/98	770	1718865	37250	5.28	8.04	0.657
4/20/98	772	1728252	43500	5.38	7.69	0.700
4/22/98	780	1728257	40950	4.92	7.56	0.651

QUALITY CONTROL RESULTS FOR POLYDYNE LE891 JANUARY 28, 1998 THROUGH MAY 29, 1998

TABLE AIII-1 (Continued)

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
4/24/98	797	1728261	46800	5.16	7.31	0.706
4/27/98	806	1728266	47450	4.91	6.87	0.715
4/29/98	820	1737930	36950	4.78	7	0.683
5/1/98	841	1737931	41450	4.67	7.05	0.662
5/4/98	846	1737932	49800	5.34	7.87	0.679
5/6/98	861	1747735	45100	5.62	8.86	0.634
5/8/98	873	1747747	45000	5.05	7.01	0.720
5/11/98	881	1747797	42900	5	7.29	0.686
5/13/98	895	1759446	46450	5.03	7.32	0.687
5/15/98	904	1759447	43600	5.13	7.14	0.718
5/18/98	920	1759448	48000	5.33	8.75	0.609
5/20/98	926	1773053	48400	5.26	6.91	0.761
5/27/98	959	1773054	42850	4.5	6.53	0.689
5/29/98	972	1773055	38550	4.49	6.47	0.694
Average			44372	5.20	7.68	0.680
Max			55000	6.07	9.81	0.833
Min			28250	4.35	6.45	0.560
Std. Dev.			5118	0.394	0.773	0.053
Median			44800	5.16	7.59	0.681
Count of Samples			49	49	49	49

QUALITY CONTROL RESULTS FOR POLYDYNE LE891 JANUARY 28, 1998 THROUGH MAY 29, 1998

APPENDIX AIV

QUALITY CONTROL RESULTS FOR POLYMERS USED DURING JUNE 7, 1998 THROUGH SEPTEMBER 30, 1998

.4

TABLE AIV-1

Date	Bill of Lading	ID	Viscosity	Percent Active Total Solids	Percent Total Solids	Ratio
6/1/98	18927	1782339	25250	5.16	5.29	0.975
6/2/98	381011	1782340	23350	4.74	5.44	0.871
6/5/98	18948	1782341	24500	4.55	5.25	0.867
6/8/98	18958	1794786	25350	4.43	5.5	0.805
6/10/98	18976	1794791	26400	4.09	5.7	0.718
6/12/98	18983	1794792	26050	4.23	5.6	0.755
6/15/98	18995	1801524	21300	4.15	5.01	0.828
6/17/98	19043	1801528	17850	3.5	3.72	0.941
6/19/98	19050	1801529	19500	3.92	4.69	0.836
6/22/98	19066	1810938	20650	3.7	3.92	0.944
6/24/98	19074	1810937	21550	4.05	5.1	0.794
6/26/98	19102	1810939	20800	3.65	4.93	0.740
6/29/98	19111	1821563	18300	3.36	4.08	0.824
7/1/98	19144	1821569	17500	3.65	4.71	0.775
7/6/98	19155	1821570	20750	4.21	5.36	0.785
7/8/98	19164	1838048	24500	4.33	5.88	0.736
7/13/98	NR	1838049	23600	4.11	4.49	0.915
7/14/98	NR	1838050	22650	4.25	5.24	0.811
7/15/98	19216	1838051	22700	4.19	5.42	0.773
+7/17/98	19223	1838052	21150	4.29	5.58	0.769
7/20/98	19308	1854686	25950	4.12	5.23	0.788
7/22/98	19312	1854701	26850	4.29	5.37	0.799
7/24/98	19324	1854705	23600	4.08	5.18	0.788
7/27/98	19363	1854706	23200	3.95	4.59	0.861
7/29/98	19372	1862569	20800	3.91	4.46	0.877
7/31/98	19378	1862570	33000	4.78	5.8	0.824
8/1/98	19383	1862571	22300	4.37	5.13	0.852
8/2/98	19426	1862576	16350	3.12	4.11	0.759
8/3/98	19428	1862577	19700	3.27	4.16	0.786
8/5/98	19437	1872532	19400	3.17	4.15	0.764
8/7/98	19444	1872536	22250	3.62	4.78	0.757
8/10/98	19482	1872542	23600	3.38	4.39	0.770
8/14/98	19500	1884310	25050	3.75	4.69	0.800
8/17/98	19509	1884311	22500	3.77	5.02	0.751
8/19/98	19541	1884312	22800	3.62	4.82	0.751

QUALITY CONTROL RESULTS FOR ALLIED COLLOIDS 7952AE JUNE 7, 1998 THROUGH SEPTEMBER 30, 1998

TABLE AIV-1 (Continued)

	Bill of		1. ¹ . 1.	Percent Active Total	Percent Total	ta an
Date	Lading	ID	Viscosity	Solids	Solids	Ratio
8/21/98	19549	1896324	22750	4.18	4.95	0.844
8/24/98	19558	1896325	19900	4.2	4,45	0.944
8/26/98	19602	1896326	21100	3.78	4.35	0.869
8/28/98	19613	1907964	24150	3.75	4.82	0.778
8/31/98	19622	1907965	19200	4.17	4.72	0.883
9/4/98	19652	1907966	17400	3.84	5.09	0.754
9/9/98	19667	1918788	22500	4.58	5.17	0.886
9/11/98	19734	1918789	22800	3.46	4.04	0.856
9/14/98	19742	1918790	19850	4.24	4.8	0.883
9/16/98	19751	1930838	23100	4.1	4.3	0.953
9/18/98	19791	1930841	23450	4.18	4.48	0.933
9/21/98	19802	1930846	20050	4.15	5	0.830
9/23/98	19812	1942024	25750	4.68	5.28	0.886
9/25/98	19854	1942052	22300	4.33	5.11	0.847
9/28/98	19863	1942053	26150	4.06	4.21	0.964
9/30/98	19871	1942054	22000	3.92	3.97	0.987
Average			22422	4.03	4.85	0.833
Max			33000	5.16	5.88	0.987
Min			16350	3.12	3.72	0.718
Std. Dev.			2952	0.424	0.537	0.071
Median			22500	4.10	4.93	0.824
Count of Samples			51	51	51	51

QUALITY CONTROL RESULTS FOR ALLIED COLLOIDS 7952AE JUNE 7, 1998 THROUGH SEPTEMBER 30, 1998

NR = Not reported.

AIV-2

÷2