- R+
- PS - Need to peak?
- which combination will get below 250 ppb 100% of the time?
- summer/winter difference?
- are we ready to say we can meet 250 100% with what we know now?

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5305 RESIDUAL VCM REDUCTION

ABERDEEN PVC PLANT

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RESIDUAL VCM REDUCTION
ABERDEEN PVC PLANT

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- I. Nested Experimental Designs

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RESIDUAL VCM REDUCTION
ABERDEEN PVC PLANT

I. Introduction

A competitor has guaranteed American Mirrex that 80% of its resin will have less than 250 ppb RVCM. Prior work has shown that Vista can produce 250 ppb or less RVCM resin only one third of the time during the winter months. The short term goal of this project is to provide American Mirrex with 250 ppb RVCM 5305 resin.

The longer term goal is to consistently produce 5265 and 5305 resin with RVCM less than 250 ppb. Furthermore, the business area believes that eventually RVCM levels will be targeted down to 10 ppb in 5265 and 5305, and 100 ppb in 5385 and 5415.

Historically, RVCM in Vista's resin is highly variable. There is a seasonal trend for RVCM, with RVCM being the highest during the winter months and lowest during the mid summer. Aberdeen can consistently meet the short term 250 ppb RVCM target during the summer months, but cannot during the winter and spring. To better illustrate, historical RVCM data is shown below.

Table I
Historical RVCM Data

Resin	Avg	Control Limits Upper	Control Limits Lower
Summer 1989			
5265	70 ppb	120 ppb	20 ppb
5305	100 ppb	180 ppb	20 ppb
Winter 1988			
5265	440 ppb	650 ppb	220 ppb
5305	340 ppb	550 ppb	130 ppb

For the last four months, PED and Aberdeen have been conducting extensive research into RVCM reduction. Three major test runs were completed between November 1989 and February 1990. Two experimental designs were done to determine the major factors of 5305 RVCM removal. This report is the culmination of that work.

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II. Recommendations

The following are recommended ways to reduce RVCM in 5305. These recommendations are classified into three categories: A) those with a **major effect** on RVCM, B) those with a **moderate effect** on RVCM, and C) those with an **unknown effect** on RVCM.

A. Major Effect

The following is a list of recommendations which are believed to have a major effect on RVCM removal in 5305. Some of these recommendations, however, do not have an additive RVCM removal effect with others. ~~and discretion should be used in determining what recommendations are used.~~

1. **Plant Air Reliability** - Aberdeen's wet air system reliability needs to be improved. Blend tank and silo aeration account for nearly all of the post reactor RVCM removal in the process. With the current systems, Aberdeen's aeration air is in very tight supply. The blend tanks, silos, and activators do not consistently get the required amount of air needed for their purposes. This results in higher final product RVCM.

2. **Resin Particle Size** - Aberdeen should evaluate the effect of changing the 5305 particle size specification. RVCM has a strong correlation with resin particle size. A 20 micron decrease in targeted particle size (from 140 to 120 micron) will result in a 35% decrease in product RVCM. Customer quality specifications need to be reevaluated to determine if Aberdeen can shift its particle size target finer.

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VLT longterm

3. **Recipe Optimization** - A detailed experimental design should be conducted in Aberdeen to determine optimum conditions for 5305. Variables should include Polivic, F-50, GH-20, initiator and kill charge amounts. These variables should be looked at to optimize CPAs, CoVs, RVCM, color and glassies. Morphology characteristics could also be studied by R&D by using their microbalance.

2.

4. **Heated Silo Air** - An air heater should be installed to heat silo aeration air to 150°F. Heating the silo air helps reduce residual RVCM by up to 25%, and does not have a detrimental not resin color or contamination.

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5. **Blend Tank Heating** - Aberdeen should rinse the 5305 reactors with hot water. Increasing the small blend tank temperature improves RVCM removal in the slurry. By rinsing the reactor with hot water, blend tank temperatures could be raised by 10 to 15°F. Hot water rinsing to the blend tanks can improve slurry RVCM by 20%.

To further reduce blend tank RVCM, Aberdeen should install a blend tank steaming system. Continuously steaming the blend tanks to maintain temperature above 150°F during the 12 hour aeration period also helped reduce slurry RVCM. Steaming the blend tanks reduced slurry RVCM out of the blend tanks by 50% during January test runs.

However, the effects of blend tank steaming and hot water rinsing are not additive. Steaming <u>and</u> hot water rinsing will not reduce RVCM beyond the 50% claimed by steaming alone.

6. **Blend Tank Agitation** - Aberdeen needs to investigate ways to improve small and large blend tank agitation. With the current systems, a large resin heel forms in the tanks which does not get aerated. Improved blend tank agitation and/or aeration would improve slurry blend tank RVCM.

Increased agitation/aeration by increasing air rates by 50% resulted in a 50% reduction in blend tank RVCM. Further improvement should be expected if aeration air flow is increased for the large slurry blend tank T-407 also.

7. **Dryer Controls** - Rotary dryer controls should be improved. The 5305 dryers (dryers 6 and 8) currently have the most difficulty controlling temperature.

Improved dryer controls would reduce temperature swings in the dryers. Tight temperature control would cause RVCM variability out of the dryers to decrease. Reduced RVCM variability out of the dryer would significantly improve resin RVCM variability in the silo and consistency of the final product.

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B. Moderate Effect

The following recommendations are believed to have a moderate effect on reducing product residual VCM.

1. **Reactor Stripping** - Temporary systems have been set up to allow a small flow of cooling water to the condenser during batch stripping. By supplying some cooling to the system, steam flow to the reactor will increase to maintain temperature setpoint. In reactors D-700 and D-745, there has been a 50% reduction in reactor RVCM with the new stripping procedures. However, reactor RVCM has only a moderate effect on product RVCM due to an RVCM dampening effect in the blend tanks.

A permanent system needs to be installed to help reduce slurry RVCM and plant VCM stack emissions.

2. **F-50 Consistency** - Aberdeen should investigate ways to decrease F-50 variability. In order to make consistently low RVCM, tight particle size control is essential. Inconsistent F-50 activity compounds the problem.

3. **Aeration Indication** - Blend tank sparger pluggage must be minimized. Installation of air flow meters on the large and small 5305 blend tanks would give early detection of sparger pluggage. Also, tank air flow meters would reduce RVCM variability by improving air distribution to all of the tanks.

4. **Silo Aeration Improvements** - RVCM can be reduced by improving silo operation. Automatic air flow control is needed on every 5305 silo, and yard operators need to follow 5305 railcar unloading procedures carefully.

Silo air valve indication lights need to be repaired on the dryer control panel also.

5. **Silo Air Piping Changes** - Piping changes to the aeration air lines at the silos would result in more consistent aeration to each silo. Because of the current piping setup to the silos, some silos get better aeration than others. Piping changes would allow a more uniform air distribution to all the silos.

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6. **Silo Air Pressure Control** - Air pressure to the silos is now controlled by a manual valve. The manual valve does a poor job controlling air pressure to the silos. Replacing the existing inoperative pressure control valve would improve silo aeration and lower RVCM.

7. **Silo Air Distribution** - RVCM levels and variability can be reduced by improving the air distribution through the silos. With the present system, heavy air channeling occurs and can be witnessed by observing the large air "perks" at the top of the silo. The perks are all in the center of the silo. The silo's cone and outer edge do not get aerated as fully as the middle.

C. Unknown Effect

The following recommendations have an unknown effect on RVCM. However, it is believed that they do have merit and good potential for improving residuals.

1. **Bench Scale Silo** - Based on experience from the last series of test runs, it is believed that there is an optimum air flow rate. By testing a bench scale silo, many things can be learned and applied, such as optimum air flow, air temperature, and air distribution.

2. **Increased Aeration of First Batch** - RVCM seems to always be at its highest at the beginning of a campaign. One reason for this may be that the first batch of slurry gets a bare minimum amount of aeration in the large blend tank. It is recommended that if possible, the first batch of 5305 be held in the large blend tank for four to eight hours before the dryers are started.

3. **Ethanol Based Kill** - There is a theory claiming that ethanol, put in the reactor, oxidizes to form an aldehyde which is hard to strip and masks the VCM peak on the gas chromatograph. In December, ethanol was being used in the Isonox kill system for 5305. At this time, Mirrex noted that they saw a second peak on their gas chromatograph which was partially masking VCM.

No evidence was found during the test runs to support or oppose the claim. However, just to be safe, it is not recommended that ethanol be used in the kill system.

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4. **Continuous Silo Baghouse Pulsing** - Some of the 5305 silos had baghouse problems resulting in continuous air backflow to the filter. When the silo hatch was opened, it was noted that these silos were under a small amount of pressure. These silos also could not get obtain adequate aeration flow rates and consequently had higher RVCM values.

5. **Blend Tank Fume Exhausters** - One observation during the RVCM test runs was that the fume exhauster was pulling air out of some blend tanks better than others. The west row of tanks seemed to have a higher vacuum on their exhaust line. It was also noted that the west row of tanks cooled off quicker than the east side tanks. These temperature differences could contribute to blend tank RVCM variability.

Details about some of these recommendations can be found in the discussion portion of this text.

III. Observations/Conclusions

Most of the major conclusions were addressed in the recommendations section of this report. Listed below are some further conclusions and observations pertinent to RVCM removal.

1. Reactor residuals have a positive effect on small blend tank pumpover residuals. For adequate results, reactor RVCM should **consistently** be below 120 ppm.

2. Reactor particle size and RVCM do not vary during the reactor dump cycle.

IMPRESSIVE!

3. RVCM removal in the blend tanks fits a non-Fickean diffusion curve. R&D microbalance results also show non-Fickean behavior for 5305. 5385 and 5415 show Fickean diffusion behavior.

4. For most efficient RVCM removal in the blend tanks, the small blend tanks should be aerated for **at least** 12 hours. Any further aeration beyond 16 hours in a cold blend tank does not significantly reduce RVCM.

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5. Of the total RVCM variation in the blend tanks, one third of it is attributable to sampling and lab testing. The remaining two thirds of the overall variation comes from the process itself.

6. Blend tank residuals have a positive effect on dryer RVCM. Lower blend tank RVCM reduces dryer and product RVCM. Large blend tank RVCM should be targeted at close to one ppm as it is fed to the centrifuge.

7. Small blend tank RVCM varied during pumpover to the large blend tank. Pumpover RVCM was at its highest during the very start and end of the pumpover. There was no particle size variation during pumpover.

8. Large blend tank T-407 has only four spargers in it. This allows for dead spots on the bottom of the tank. If a sparger were to plug, the dead spot would become very large.

9. All the blend tanks use only a fraction of the recommended amount of air needed for complete agitation.

10. CPAs were run on dryer particle size screen cuts to see if there were any relationship. The results gave a parabolic curve with a minimum CPA at 100 mesh. The overall composite, however, had an even lower CPA than the 100 mesh cut. ?

11. Average particle size seems to be related to the coefficient of variance. However, the correlation is not always in the same direction. During August 1989 and February 1990 there was a positive correlation. During July 1989, however, there was a negative correlation. All correlations had greater than 95% confidence limits.

12. During the test runs, there was a positive correlation between dryer moistures and dryer RVCM. Improved dryer controls giving more consistent moistures would reduce RVCM variability.

13. RVCM removal in the silos does not seem to follow a smooth removal curve. This may be explained by

    a. severe air channeling in the silos
    b. high dryer variability and mixing in the silos
    c. frontal chromatography

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14. There is a significant amount of drying for 5305 in the silos. After 24 hours aeration, the resin moistures are reduced by over 50%.

15. Silo aeration removes at least 30% of the remaining RVCM out of the dryers. Silos should aerate at least 24 hours. However, no additional RVCM removal is seen with cold air after 30 hours aeration.

16. RVCM measured by the Aberdeen lab often matches the lab result reported by American Mirrex. Differences in Aberdeen's and Mirrex RVCM numbers is due to weathering and sample location. Aberdeen samples from the bottom of the car, while Mirrex often samples from the top of the car.

17. Lab RVCM testing for dry resin samples had very little variability. Slurry lab RVCM testing had a very large variability.

IV. Discussion

A. Plant Air Reliability

With the current systems, Aberdeen's aeration air is in very tight supply. Frequently, the air demand is high enough that air flow to the silos, blend tanks, and activators is insufficient. When this occurs, RVCM greatly increases. Silos and blend tanks get minimal aeration resulting in higher RVCM.

High air demand results in lower system air pressure. Low air pressure limits the efficiency of the dryer activators. Low air pressure can also lead to slower resin transfer, plugged lines, and increased sifter overflow. Furthermore, with low air pressure, the blend tank air spargers often plug.

In order to adequately aerate the silos, air pressure to the silos must be maintained above 25 psig. Currently, silo aeration air pressure is below 25 psig over 40% of the time. Low air pressure causes insufficient silo aeration and high residual VCM.

Aeration in the blend tanks is also inadequate. Blend tank air flow rates are up to 50% lower than recommended for adequate agitation in RQIP. Furthermore, air flow to each of the blend tanks is variable. The blend tank air flow is controlled by a throttling a manual plug valve at each individual sparger. Air distribution to each sparger is unequal, causing RVCM variability and possible sparger pluggage.

Because the agitation is insufficient, a resin heel forms in the blend tank (especially in the large blend tank T-407). The resin in the heel does not get aerated and tends to yellow and degrade. Improved blend tank aeration as was provided for in the Blend Tank Mixing Improvement portion of RQIP would help reduce 5305 RVCM, b-color and contamination.

A recently completed wet air survey for Aberdeen is attached in Appendix A.

B. **Resin Particle Size**

The current target for average particle size of Vista's 5305 resin is 140 micron. However, the average 5305 railcar particle size ranges between 120 and 165 micron. Particle size distributions typically tend to get wider with coarser resin produced.

Tight particle size control is important in making low RVCM 5305 resin. Lab results show a strong correlation between RVCM and average particle size in dryer and railcar samples for both summer and winter. In all cases, coarse resin tends to have higher RVCM.

In order to test the theory that RVCM is higher in larger particles, several dryer samples were taken and screened into particle size cuts. Each screen cut was then tested for RVCM. In all cases, resin on the 60 and 80 mesh screens had RVCM over **ten** times higher than 200 mesh and pan resin. Results were duplicated using lab dried reactor dump samples. Table II shows the results of the lab tests for each particle size cut.

Table II
Dryer RVCM Particle Size Relationship

Particle Size Cut	RVCM
Composite	1.1
60 Mesh	4.7
80 Mesh	2.5
100 Mesh	1.4
120 Mesh	0.6
140 Mesh	0.4
200 Mesh	0.3
Pan	0.1

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Using the information on RVCM content for each particle size cut, the improvement in RVCM can be predicted for any particle size distribution. Table III below shows a the expected improvement for different particle size distributions. Since product RVCM is so variable, it is extremely difficult to assign absolute numbers to each particle size cut. Therefore, for the calculated RVCM, each particle size distribution will be compared with the target particle size. The target particle size (140 micron) will be assigned an arbitrary RVCM value of 1.

Table III
Particle Size Effects on RVCM

Avg Micron	CoV	% on 60/ 80 mesh	Calculated ppm RVCM	Actual ppm RVCM
120	0.288	3.6%	0.64	0.366
130	0.272	6.8%	0.84	0.505
140	0.189	5.5%	0.90	
Target-140	0.284	11.4%	1.00	0.608
150	0.281	17.1%	1.20	0.811
160	0.298	23.7%	1.41	1.487
180	0.357	31.4%	1.87	1.721

Attachments [redacted] in appendix B also illustrate the particle size effect on RVCM.

Table III shows that if the target particle size is shifted to the finer side, a significant reduction in RVCM can be expected. The table also shows that RVCM can be reduced by reducing the particle size distribution (measured by the coefficient of variance - CoV). Both lowering the target particle size and tightening the size distribution are in effect reducing the number of large, high RVCM particles.

C. Recipe Optimization

Since the RQIP temporary systems have been installed, there has been a lot of work done to determine how the recipe effects resin properties. However, the work done up to now concentrated on what was required to make an acceptable new technology performance resin as soon as possible. For example, Polivic charge amounts have not been changed or investigated since Aberdeen started making acceptable 5305.

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Now that Aberdeen has established a baseline quality for its performance resins, Vista has the opportunity to improve resin quality by optimizing reactor charge recipes.

Recipe optimization probably would consist of a detailed experimental design to determine the effects of suspending agents, initiators, and kill agents.

The 5305 resin recipe optimization should focus on improving resin color, CPA, RVCM, glassies, and CoV. Morphology characteristics should also be investigated by R&D by using their microbalance. Once 5305 recipe is optimized, Aberdeen should focus on optimizing 5415.

D. Silo Air Heating

Back in 1986, when Aberdeen was trying to reduce RVCM to below 2 ppm for Nibco, R&D built a bench scale model of a resin silo and performed aeration tests. One test showed that heating silo aeration air helped reduce RVCM in old technology 5265. However, since the increased reduction was minor (10%), no further work was done.

One major conclusion to the bench scale tests was that the rate of diffusion was dependent on the resin temperature. Cold aeration air would cool the resin and slow the rate of diffusion. Results of the bench scale test are attached in Appendix C.

During the February test runs, a 2^3 experimental design was completed which studied the effects of 1) silo aeration air temperature, 2) aeration time, and 3) Isonox kill amount. Results showed that air temperature and aeration time significantly reduced resin RVCM. Isonox kill amount (varied from 75 to 125 lb) had no effect on RVCM.

In early January 1990, Aberdeen installed a small electric heater ~~that was found in the warehouse.~~ The heater was used to heat the silo aeration air up to 100°F. It was found that RVCM could be reduced by up to 25% in the silos by heating the air.

To ensure 5305 quality, during the heated air tests, the lab monitored resin color, contamination, and moisture. Heated air had no effect on resin color or contamination using heated air up to 130°F. Resin moisture levels in the silo, however, were reduced.

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A larger air heater should be installed to heat all the silo aeration air to 150°F. The existing heater is too small and at full aeration rates can only heat the air to 80°F in the winter. A larger heater with consistent temperature control would be able to reduce RVCM by at least 25% and also reduce final product variability.

Another finding during the silo aeration tests is that silo aeration time has a significant effect on RVCM removal. For best RVCM removal, the silos should be aerated at least 24 hours. No significant RVCM removal is seen after 30 hours aerating using cold aeration air.

Results of the experimental design, ~~the silo sampling program,~~ and aeration studies done by R&D are attached in Appendix C. OK

E. **Small Blend Tank Heating**

One of the experimental designs to lower product RVCM was heating the small blend tanks during the 12 hour aeration period. The 2^3 experimental design was done using blend tank steam heating, hot water reactor rinsing, and blend tank aeration time as the three variables for study.

Steam heating the small blend tanks reduced small blend tank RVCM by 50%. Rinsing the reactors with hot water instead of cold water reduced the blend tank RVCM by 20%. As should be expected, hot water rinsing in combination with blend tank steam heating did not reduce blend tank RVCM beyond the 50% realized with steam heating alone. The benefits of steam heating and hot water rinsing are not additive.

Currently, reactor D-700 and D-745 are dumped and rinsed with cold water to the small blend tanks. The slurry is then aerated for 12 hours prior to being pumped over to the large blend tank T-407 to be dried. The small blend tank temperature is about 150°F after rinsing. In the winter, after 12 hours aeration, the tanks can cool down to as low as 100°F (typically 135°F). The cooling of the small blend tanks slows the RVCM removal.

During the test runs, blend tank heating was not supposed to actually heat the blend tanks, but to keep them from cooling off. By rinsing the reactor with hot water instead of cold water, the blend tank temperature after rinsing was 165°F and the tank cooled down to 145°F after 12 hours aeration. Steam heating the blend tanks kept the temperature of the blend tank temperature constant throughout the 12 hour aeration period. Steam heating kept the temperature above 160°F when

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rinsing with hot water, and 150°F when rinsing with cold water.

During the blend tanks heating tests, resin color and contamination were closely monitored. No increase in contamination or b-color were seen during the test.

Due to practicality, only one set of blend tanks could be used for the test. Because only one set of tanks were used, and the slurry was blended, no attempt was made to determine the effect steam heating had on dryer RVCM.

The small blend tank steam heating test also studied the effect of aeration time on blend tank RVCM removal. It was discovered that with no blend tank steam heating, RVCM variability was high. It was also discovered that there was effective RVCM removal in the blend tank for up to 12 hours aeration. Further aeration beyond 16 hours gave very little RVCM removal.

The steam heated blend tanks had a much lower RVCM variability and a somewhat different RVCM removal curve. When the blend tanks were steamed, RVCM removal followed a removal curve very similar to a non-Fickean diffusion curve. R&D microbalance studies have shown that diffusion 5305 is non-Fickean in nature.

All this information implies that when the blend tanks are steamed, the rate limiting factor for RVCM removal in the blend tanks is VCM diffusion out of the PVC particle. When the blend tanks are not heated in any way, VCM stripping out of the water may be limiting the rate of RVCM removal.

Appendix D contains the results of the experimental design, as well as results from the extensive sampling program done to determine blend tank RVCM removal efficiency.

F. Blend Tank Agitation/Aeration

With the present blend tank aeration system, air distribution is such that a large resin heel forms in the tanks. This heel does not get aerated and therefore contains high RVCM levels. The heel can degrade and cause high b-color and contamination. Improved blend tank agitation/aeration would potentially improve blend tank RVCM, b-color, and contamination.

Testing in November 1989 showed that increased blend tank air flow improves RVCM removal in the slurry. By increasing air flow rates to the blend tank by 50% , a 50% reduction in blend tank RVCM can be expected. Even further slurry RVCM

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improvement can be expected if air flow is increased for the large blend tank T-407 also.

Aeration in all the blend tanks is inadequate. Blend tank air flow rates currently are up to 50% lower than recommended for adequate agitation in RQIP. Furthermore, air flow to each of the blend tanks is variable. The blend tank air flow is controlled by a throttling a manual plug valve at each individual sparger. Air distribution to each sparger is unequal, causing RVCM variability and possible sparger pluggage.

Blend tank agitation is the worst in the large blend tank T-407. T-407 only has four air spargers, although according to the RQIP designs, it is supposed to have eight. Air flow rates to T-407 are well below the recommended rate for complete agitation. Improved blend tank aeration as was provided for in the Blend Tank Mixing Improvement portion of RQIP would help reduce 5305 RVCM and b-color.

One of the biggest causes of high RVCM is blend tank sparger pluggage. Blend tank air spargers plug fairly frequently. However, there is no way to readily know when a air sparger is plugged. Air flow meters on the blend tank aeration line would not only show when a sparger is plugged, but would also help provide consistent air flow to all the tanks.

Further information about blend tank agitation/aeration and its effect on RVCM is attached in Appendix E.

G. Dryer Controls

During the months of November 1989, and February 1990 extensive sampling was done to determine the how the process affects RVCM. A major finding was that the magnitude of the process variability of the dryers was one of the largest contributors to overall product variability.

It was always known that dryer RVCM from day to day was highly variable. What was discovered was that dryer RVCM was highly variable from hour to hour. In fact, dryer RVCM could change up to 25% within one dryer activator cycle. Since the test variability, as determined from the silo nested experimental designs, is small, a great deal of process variability is being introduced in the dryers.

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There are some other factors that are believed to have a major effect on dryer RVCM, but have not been proven in the field. These include:

a. Large Blend Tank Level
b. Drying Efficiency
c. Individual Dryer Performance
d. Dryer Temperature

It was shown during previous test runs that the slurry solids concentration changes significantly with blend tank level. Maintaining a consistent blend tank level or improving blend tank agitation would ensure a more consistent feed rate to the dryers. In the advent of an external stripping column for 5305, the continuous nature of the process would help keep a consistent blend tank level. In the meantime, efforts should be focused on improving blend tank aeration.

It is also believed that individual dryers have different VCM removal efficiencies. This theory could not be verified because of the high variability of the process and limited number of data points. Presently, dryers 6 and 8 are used to dry 5305. From looking at the dryer temperature strip charts, it can be concluded that dryers 6 and 8 are least tightly controlled dryers.

The outlet temperature for both dryer 6 and 8 have a cyclic pattern while dryers 4, 5, and 7 have much tighter temperature control. Figures 1 through 4 in Appendix F show the temperature strip charts for each of these dryers. Oscillating dryer temperature may explain why dryer RVCM is highly variable between each activator cycle.

There are many differences between Aberdeen's rotary dryers. Dryer 4 seems to contaminate resin more rapidly than the other dryers and therefore cannot be used for 5305. Dryer 6 is the slowest dryer in Aberdeen. Often dryer 6 runs with somewhat higher moistures than the other dryers. This is probably because operators try to push as much resin through the dryer as possible to keep up with production.

During the February test run, a correlation between dryer resin moistures and RVCM was revealed. Dryer resin had less RVCM than moist resin. This is not a surprise, since water removal and VCM removal in the dryers follow the same mechanism.

The attached figures in Appendix F help illustrate the dryer controls situation.

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H. Silo Operation Improvements

Silos need to always be aerated properly in order to make 250 ppb 5305. Most of the operational problems which result in high RVCM happen at the silos. There is potential RVCM improvement by improving silo operations. Suggested operating improvements are listed below.

1. Automatic air flow control is needed at the silos. This can be achieved by installing a 1" bypass line around the silo air control valve (identical to the set-up on silos 678-680, and 682). The yard operators need to be aware of what these lines are for.

2. Silo aeration valve indication is needed in the control room so that the dryer operator can tell if the air is on. Currently, practically none of the lights on the air valve on the dryer control panel are operable.

3. In order to ensure adequate 24 hour aeration, yard operators must unload the silos properly. When a silo is unloaded into a railcar, that silo needs to be completely empty before the railcar is topped off with another silo.

4. Improved communication between the dryer panel operator and the yard operator is needed. The yard operator needs to call the dryer panel operator to turn the air on or off when unloading a silo. After the yard operator is through, he needs to inform the dryer panel operator so that the air valve can be turn back on or off.

Complete operating instructions are attached appendix G.

I. Silo Aeration Improvements

Silo aeration can be improved in a number of ways including:

1. Silo Air Pressure Control
2. Improved Air Distribution among the silos
3. Improved Air Distribution in the silos

The items above are ranked in order of best effect on silo RVCM removal. Silo air pressure control valve would keep a constant air pressure out to the silos. Keeping a constant silo air pressure helps ensure that every silo gets adequately aerated.

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Currently, there is a problem with air distribution among the 5305 silos. The silos which are the farthest down on the air header (silos 678, 679, and 680) do not get as much air flow as the silos farthest upstream on the header (683 & 684). Consequently, silos 683 and 684 remove RVCM better than silos 678, 679 and 680.

All silos experience severe air channeling during aeration. The center of the silo aerates much more than the outer edges or the cone. Improved air distribution would give a more uniform RVCM resin.

Appendix G further contains some charts which further illustrate the silo aeration problems.

J. Bench Scale Silo

Silo aeration currently is highly variable. Large pressure swings on the air header cause silo air flows to fluctuate widely. Because of this, it is practically impossible to conduct aeration tests in the plant. In 1986, R&D built a small bench scale silo to test aeration on old technology 5265. It is felt that a similar apparatus would be helpful in determining optimum air flow rates and temperatures for 5305 RVCM removal.

Results of the bench scale studies in 1986 are attached in Appendix C. During the move to Austin, R&D dismantled the silo and threw most of it away. However, according to R&D it would be very inexpensive to build another bench model.

K. Reactor Stripping

In early November, it was decided to try to improve reactor stripping for 5305 in an effort to reduce product RVCM. To do this, a system was set up that would open the condenser cooling water valve 10% when a) the reactor was in the recovery mode, and b) the slurry temperature was at least 175°F. By supplying some cooling during stripping, steam flow through the slurry would increase, resulting in lower reactor dump RVCM.

A similar manual setup was tested on reactor D-744 with 5415. The results of that test showed a 42% reduction in slurry RVCM to 68 ppm. Testing lasted two months, and condenser fouling on D-744 was comparable to the other reactors.

VAB.0001097528

An automatic system was setup for reactors D-700 and D-745 in mid November, 1989. 5305 reactor residuals were reduced by 50% in late November through January 1990. 5305 reactor RVCM was reduced from 185 ppm to 92 ppm. The improved stripping procedure also significantly reduced slurry RVCM. Appendix H has some figures that better illustrate the RVCM improvement during the test.

During the test, product quality was closely monitored. No resin quality deterioration was found during the test. Resin b-color did not change, and contamination was low.

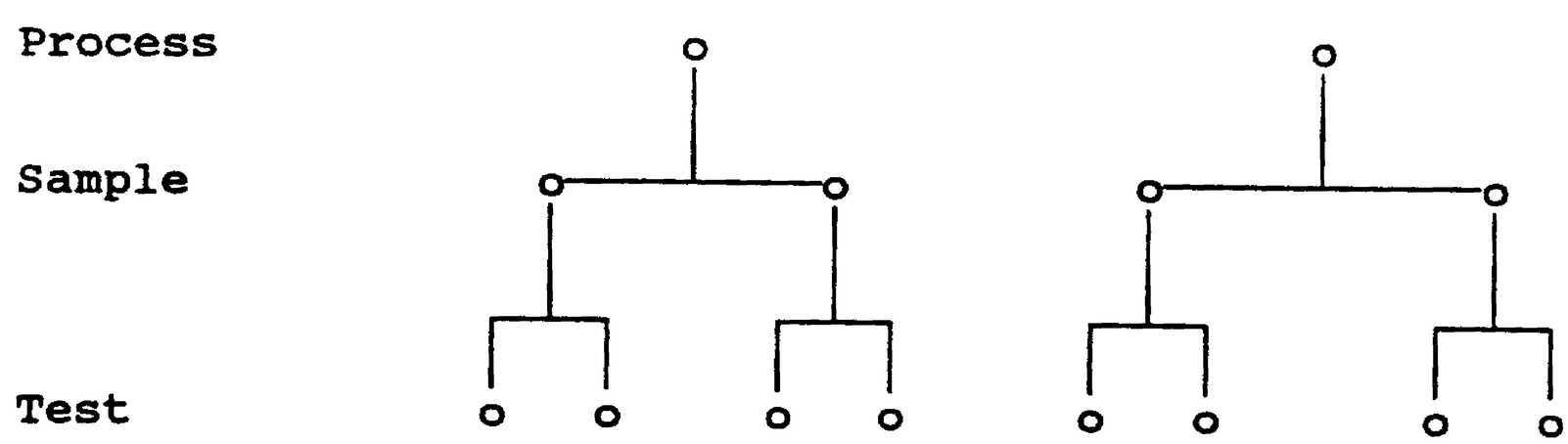
L. Nested Experimental Designs

A nested design attempts to assign a percent of the total variability of a process to different areas. The area with the highest percentage of the total variability would be the area worked on the improve. The actual percentage is not as important as the relative value in comparison to the other areas.

Two nested designs were completed in January. Both designs determined the percent variability due to the process, sampling and testing. Process variability refers to the actual variability in the process including equipment and operating variation. Sampling refers to the variation between samples and from sampler to sampler. Testing includes variation of the lab test and its technicians.

In order to nest a process, samples are taken in "layers". Each time a data point is needed, the sampler takes two samples. Each of these samples is divided so that each sample is tested in the lab twice. This forms three "layers" of which the variabilities are calculated. Figure 1 shown below further illustrates this layering.

Figure 1
Nested Design Layers



VAB.0001097529

1. Blend Tank Nesting

Based on the blend tank nested design it was determined that the process must be improved before the test and sampling method are improved.

The blend tanks were nested to discover the process, sampling, and testing variability. Two blend tank samples were taken and these samples were split into two. Four data points are obtain per sample so that the three layers can be formed.

Eleven sets of blend tanks were sampled for this nesting design. The sets of two were divided into individual tanks for evaluation. The twelve hour aeration average RVCM for both modules was 1.54 ppm for all the tanks. Old module blend tanks averaged 2.16 ppm while the new module averaged 0.8 ppm. Listed below in Table IV are the assigned percent variations due to the process, sampling, and testing. Average RVCM and overall standard deviations are also included in Table IV

Table IV
Blend Tank Nested Design

	Overall	Old Module	New Module
% Variance - Process	66.9	35.4	71.3
% Variance - Sampling	13.9	28.7	5.6
% Variance - Testing	19.2	35.9	23.1
Average RVCM, ppm	1.54	2.16	0.80
Standard Deviation	1.04	0.97	0.51

In the overall and new module cases the process is the best area to concentrate the efforts to improve. The old module percentages are practically equal, therefore, process concerns are as important as the other two areas. The process area becomes a larger contributor to the total variance as the RVCM value falls. The importance of the process is therefore magnified. The process should be improved first.

There is a definite difference between old and new module blend tanks. The differences are probably caused by variations in the new reactor stripping procedures discussed earlier in this report. During the nesting experiment, the improved reactor stripping system was operating on D-745, but not on D-700.

The detailed results of the blend tank nested experimental design are included in Appendix I.

VAB.0001097530

A

2. Silo Nesting

Based on the silo nested design, the process needs to be improved before the lab testing and sampling methods are improved.

The silos were nested to discover the process, sampling, and testing variability. Two silo samples were taken and the samples were split into two. Four data points are obtained per sample taken so that the three layers can be evaluated.

Ten silos were chosen for the nested design data. The silos were sampled from the top. The average RVCM value was 0.37 ppm. Listed below in Table V are the assigned percent variations due to the process, sampling, and lab testing. Also included in Table V is the average RVCM value, and the overall standard deviation.

Table V
Silo Nested Design

Item	Percent
% Variance - Process	86.6
% Variance - Sampling	1.8
% Variance - Test	11.6
Average RVCM, ppm	0.37
Standard Deviation	0.30

The percent variance in the process is the obvious area to concentrate efforts to improve. This data verifies past lab studies which showed testing to be only 10% of the overall variability of dried resin samples.

The detailed results of the silo nested experimental design are included in Appendix I.

V. Summary

This report is the culmination of four months of intensive test run work at Aberdeen in an effort to reduce RVCM. The recommendations presented in this report should allow Vista to produce 5305 resin with less than 250 ppb consistently.

To get to the long term target of 10 ppb RVCM, Vista will probably have to make some fundamental changes in the way it produces PVC. An experimental design for recipe optimization and a potential future external steam stripping columns are some fundamental changes that may contribute towards the long term target.

VAB.0001097531

APPENDICES

Appendix A

Plant Wet Air Survey

VAB.0001097534

TO: Eric Meyer

FROM: Rich Tebo
DATE: January 8, 1990

Interoffice Communication

VISTA

SUBJECT: Plant Wet Air Survey

Attached is a detailed account of wet air users at Aberdeen. The wet air is supplied by the East Joy centrifugal and Joy reciprocating compressors. These two compressors send air to 1) all the blend tanks, 2) all the dryer activators, and 3) all the 600 series silos.

Conclusions

Wet air supply is insufficient to adequately supply all the users. Because of the limited air supply, air flow rates are well below recommended levels for proper blend tank and silo aeration. The recommended air flows are based on the RQIP design basis and are needed to a) adequately agitate the blend tanks, and b) adequately strip RVCM from the resin in the blend tanks and silos.

Recommendations

A. Short Term

Two additional reciprocating compressors should be started up to supply additional air to the silos and blend tanks.

B. Long Term

PED or Aberdeen should investigate the alternatives for increasing plant air capacity. These alternatives may include:

1. Installing a low pressure air system to supply air to the silos and/or blend tanks,

2. Optimizing aeration demands at a level below what was recommended by RQIP,

3. Installing mechanical agitation on blend tanks,

4. Installing a dilute phase V-11 dryer transfer system, or

5. Installing additional air compressors.

A

The attached survey shows that Aberdeen's air supply to the silos and blend tanks is well below what is needed. Also, with the start-up of Low Mole Expansion, air demand will further increase (by over 500 SCFM). Additional air compressors need to be started up until a long term solution can be implemented.

Rich Tebo

Rich Tebo
Process Engineer
Engineering Department

Attachments

cc: RWS \ PJK \ RBN \ CJMc \ SCH \ RDM \ PCS \ KWB \ DAMo

File ABD.0.900108.1

ABERDEEN WET AIR SURVEY

Basis

The attached tables show how wet plant air is being used and supplied in Aberdeen. The basis for these tables is as follows:

Silos

1. Each 5305 or 5265 silo should consume 375 to 400 SCFM for 40 hours while it is filling and aerating (based on work done by LZK and RJL in 1986).

2. During a 5305 run, up to 7 silos can be aerating at once. Five of the silos will be for 5305, one for 5265, and one for 5305 start-up.

3. The empty 5305 silos use 50 SCFM.

4. All the other silos use 50 SCFM when the silos are being unloaded in order to keep the resin fluidized.

5. When Low Mole Expansion starts up, the process will change such that three rotary dryers will dry 5305 to one silo.

6. One 5265 silo will be held for trucks when 5305 is run in the extra large reactors.

7. A separate silo will be used for 5305/5265 start-up in order to minimize debox. This silo will need to be aerated.

Blend Tanks

1. The existing blend tank restriction orifices are designed for 75 SCFM air flow per tank.

2. RQIP recommended air superficial velocity for adequate agitation is 1.3 ft/min (SCFM/sq ft). RQIP recommends 1.3 ft/min for T-407 and 1 ft/min for the small tanks. This translates to 105 SCFM for the small tanks and 330 to 375 SCFM for the large tanks.

3. Blend tanks T-654, 655, and the V-10 blend tanks will not need any further increase in agitation. Air flow to these tanks will remain at 75 SCFM.

Activators

1. The five activators in the V-11 dryer building use 458 SCFM when transferring. The V-11 dryer acitvator's have a transfer frequency of 3.71 minutes in a 5.54 minute cycle. Assuming that the transfer frequency and air rates are the same for all five activators and transfer at a random binomial distribution gives the results listed in the table below.

Table I
Activator Frequency of Occurance
Plant Wet Air Survey

Number of Activators Transferring Simultaneously	Frequency of Occurance, %	Air Usage SCFM
1	4.4	458
2	16.2	916
3	32.8	1374
4	33.1	1832
5	13.5	2290

Most frequently, four activators are transferring at one time using 1832 SCFM. This is the listed average rate. Maximum air demand is five activators transferring which happens 13.5% of the time. This information was supplied by V.L. Thornhill in a telephone conversation in February 1988.

PLANT PROCESS AIR SURVEY

East (Wet) Air System Users

Silos	<------ Air Usage (SCFM) ------> Actual	Recommend	LMW Exp
675	0	50	50
676	50	50	50
677	0	0	0
678	50	50	50
679	80	375	375
680	80	375	375
681	0	0	0
682	110	375	375
683	110	375	375
684	110	375	375
685	0	0	375
686	0	0	50
687	110	375	375
688	0	0	0
689	110	375	375
690	50	50	50
691	50	50	50
Total	910	2875	3300

12/89 Estimated Air Flow Rate	910
Targeted Recommended Flow Rate	2875
LMW Expansion Absolute Maximum	3300

A

Activator Usage

Activator	Avg SCFM Consumed
4	366
5	366
6	366
7	368
8	366
Total	1832

Blend Tank Usage

	<---------- SCFM Consumed ---------->		
Blend Tank	Actual	Recommend	LMW Exp
T-646	60	105	105
T-647	60	105	105
T-648	60	105	105
T-649	60	105	105
T-650	60	105	105
T-651	60	105	105
T-652	60	105	105
T-653	60	105	105
T-654	50	50	50
T-655	50	50	50
T-407	220	330	330
T-501	220	330	330
T-747	220	375	375
T-502	220	375	375
4 Pond Resin Tanks	200	200	200
New Low Mole Silo	0	0	105
Total	1660	2550	2655

A

Total Usage
SCFM

User	Normal	Recommend	LMW Exp
Silos	910	2875	3300
Activators	1832	1832	1832
Blend Tanks	1660	2550	2655
Total	4402	7257	7787

Air Supply

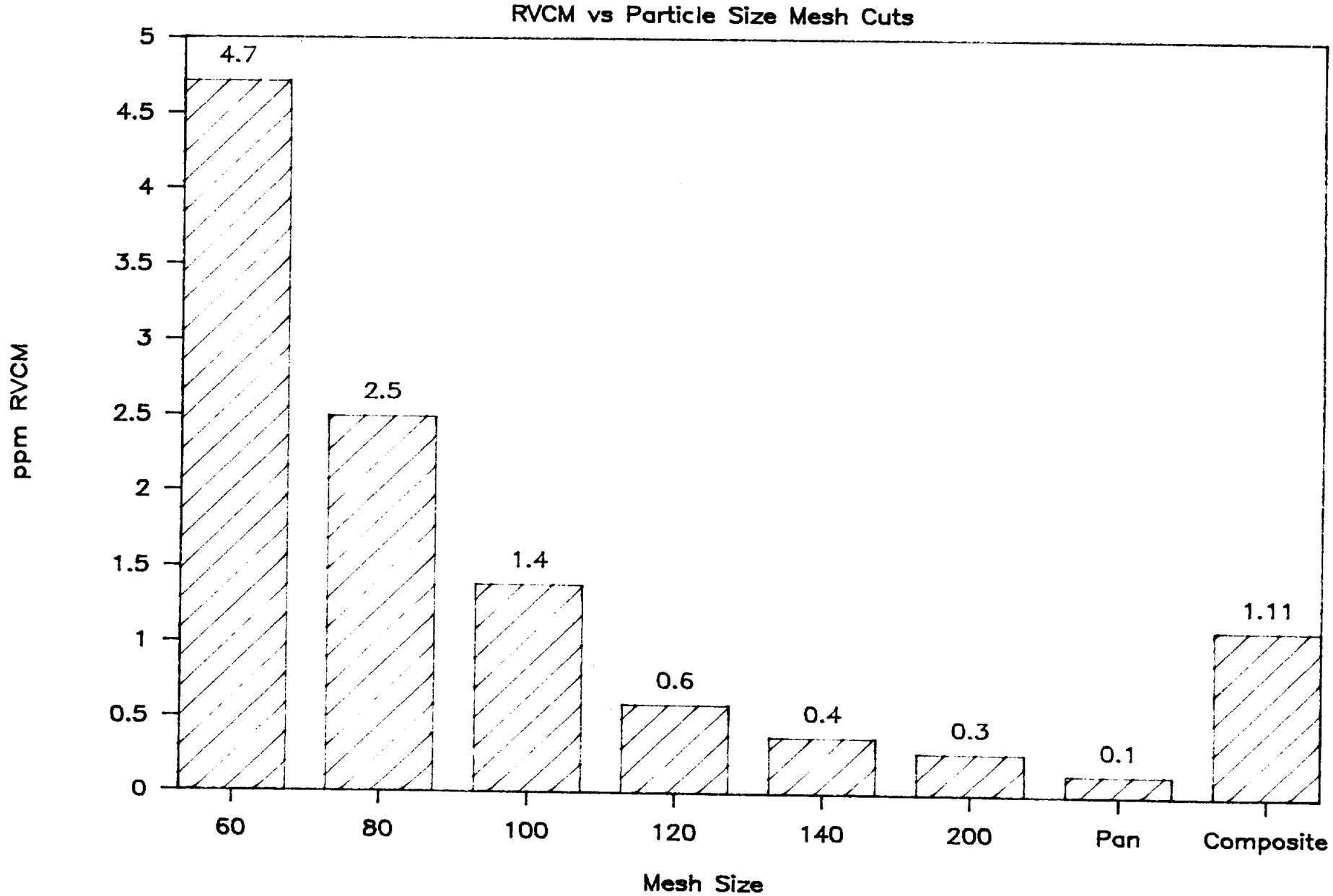
Source	Normal	Max
East Joy	3600	3600
Joy Recip	500	500
Ingersoll Rand Recip #1	0	500
Ingersoll Rand Recip #2	0	500
Ingersoll Rand Recip #3	0	450
Ingersoll Rand Recip #4	0	450
North Joy (emergency generator)	0	500
South Joy (emergency generator)	0	500
Total	4100	7000

Particle Size

Appendix B

Resin Particle Size and Its Effect on 5305 RVCM

5305 Dryer RVCM Distribution



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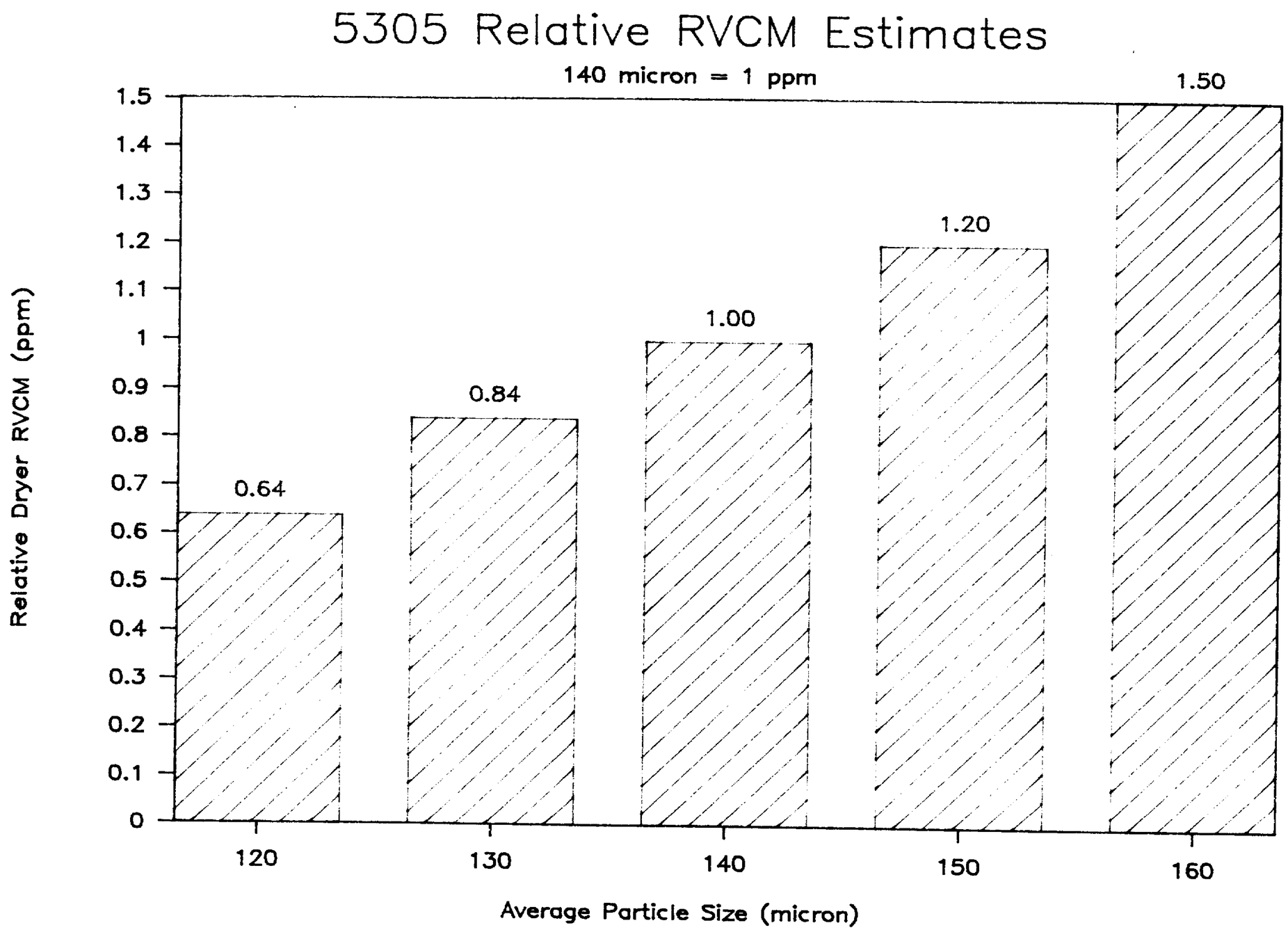

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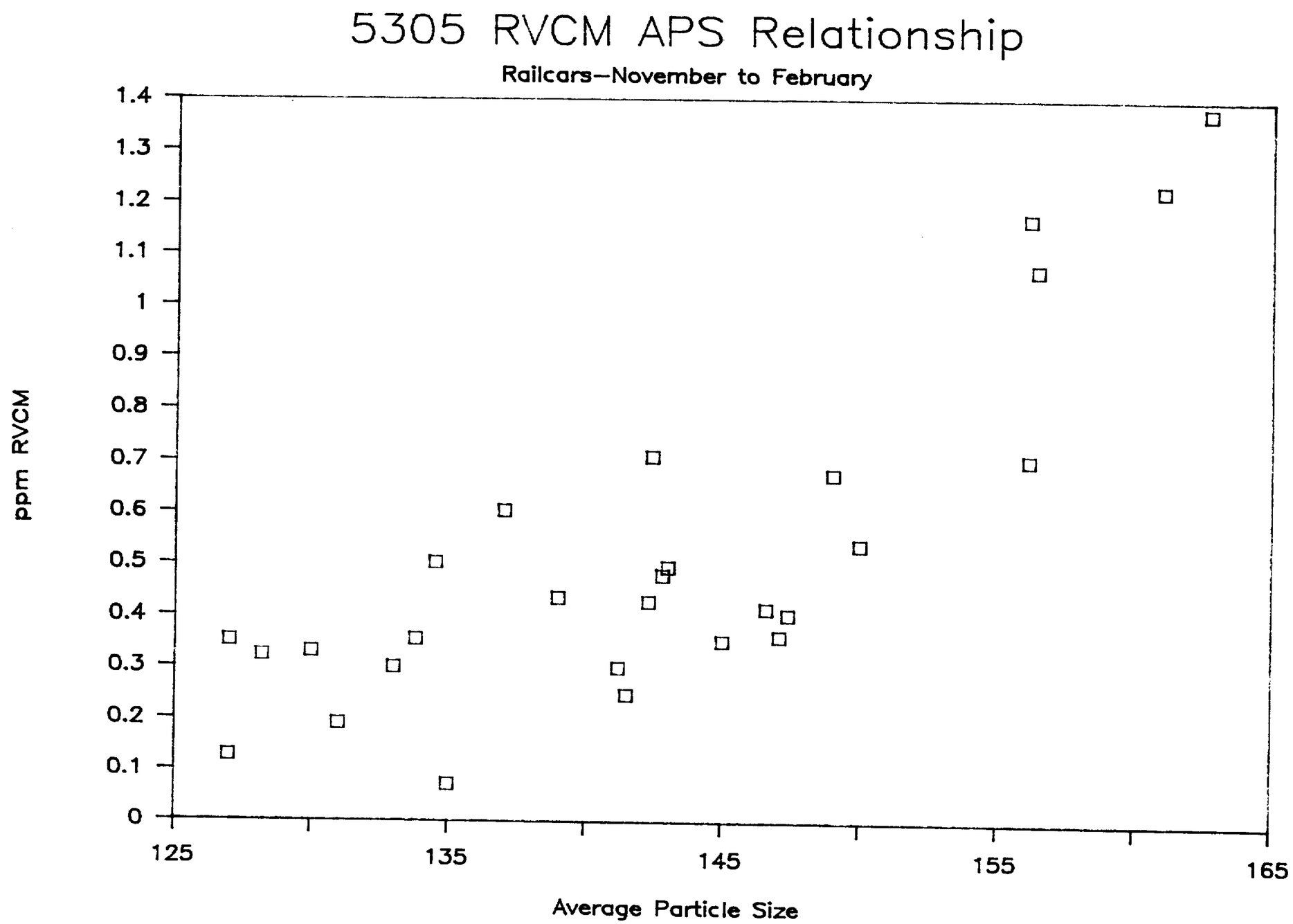

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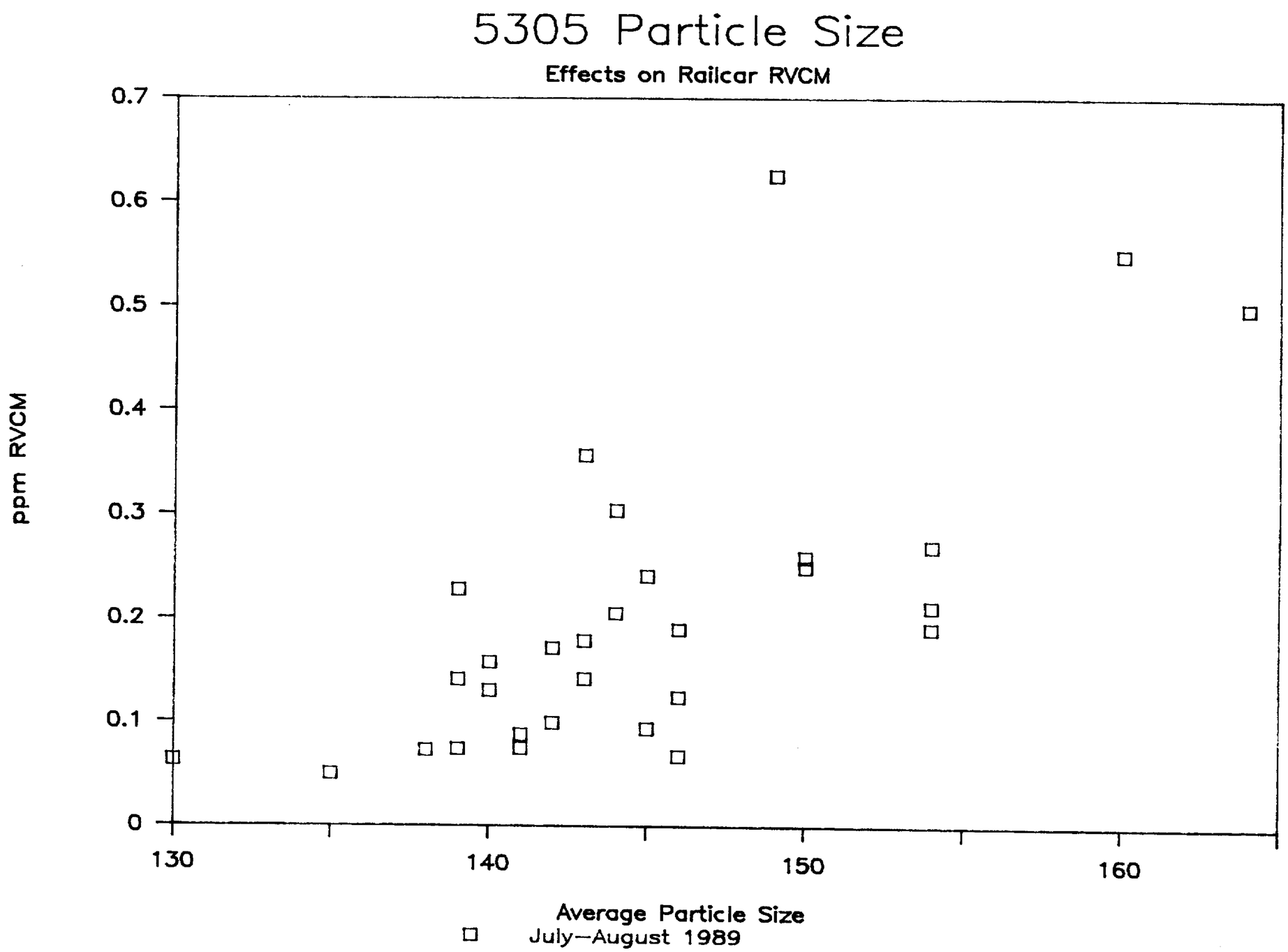

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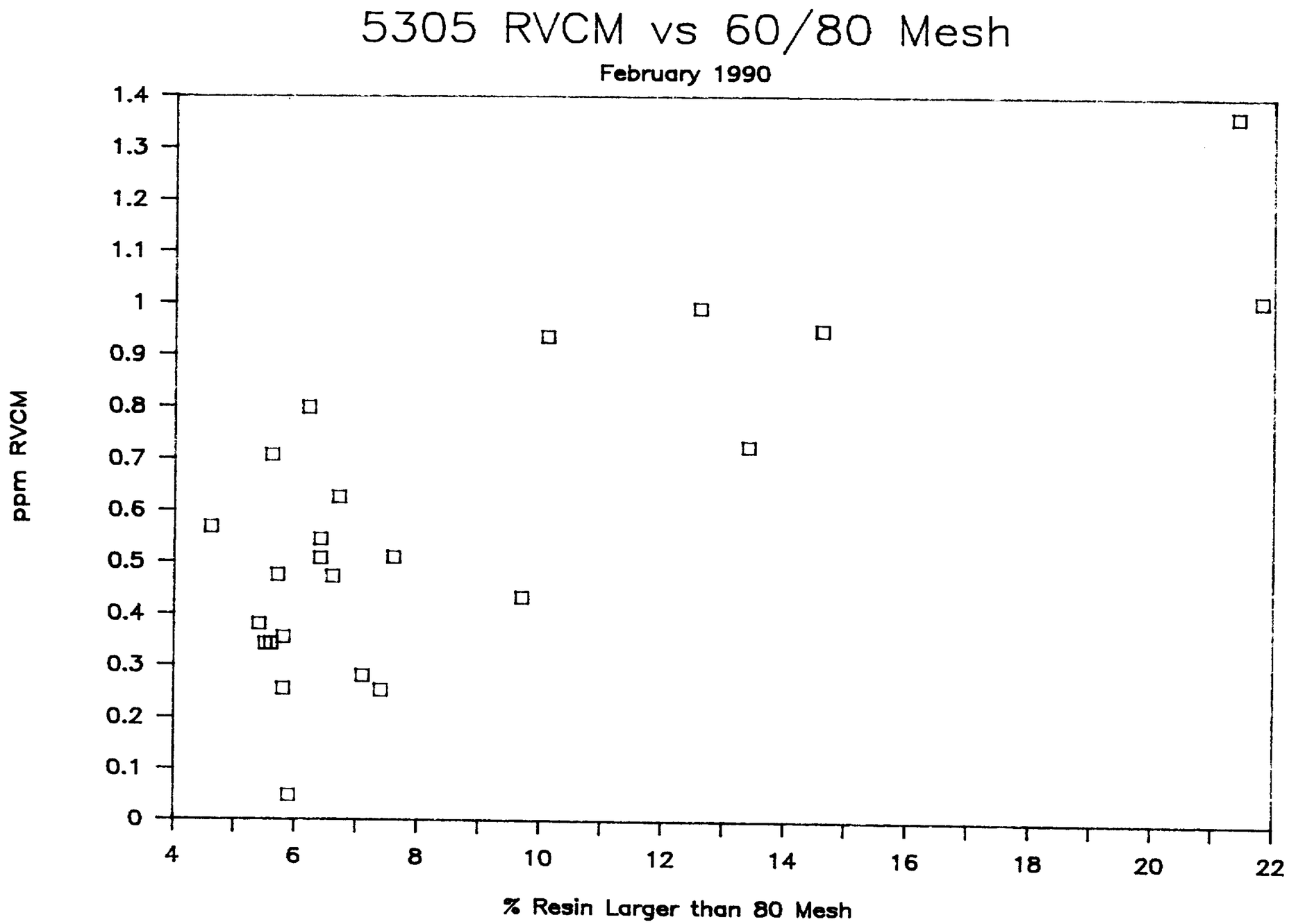

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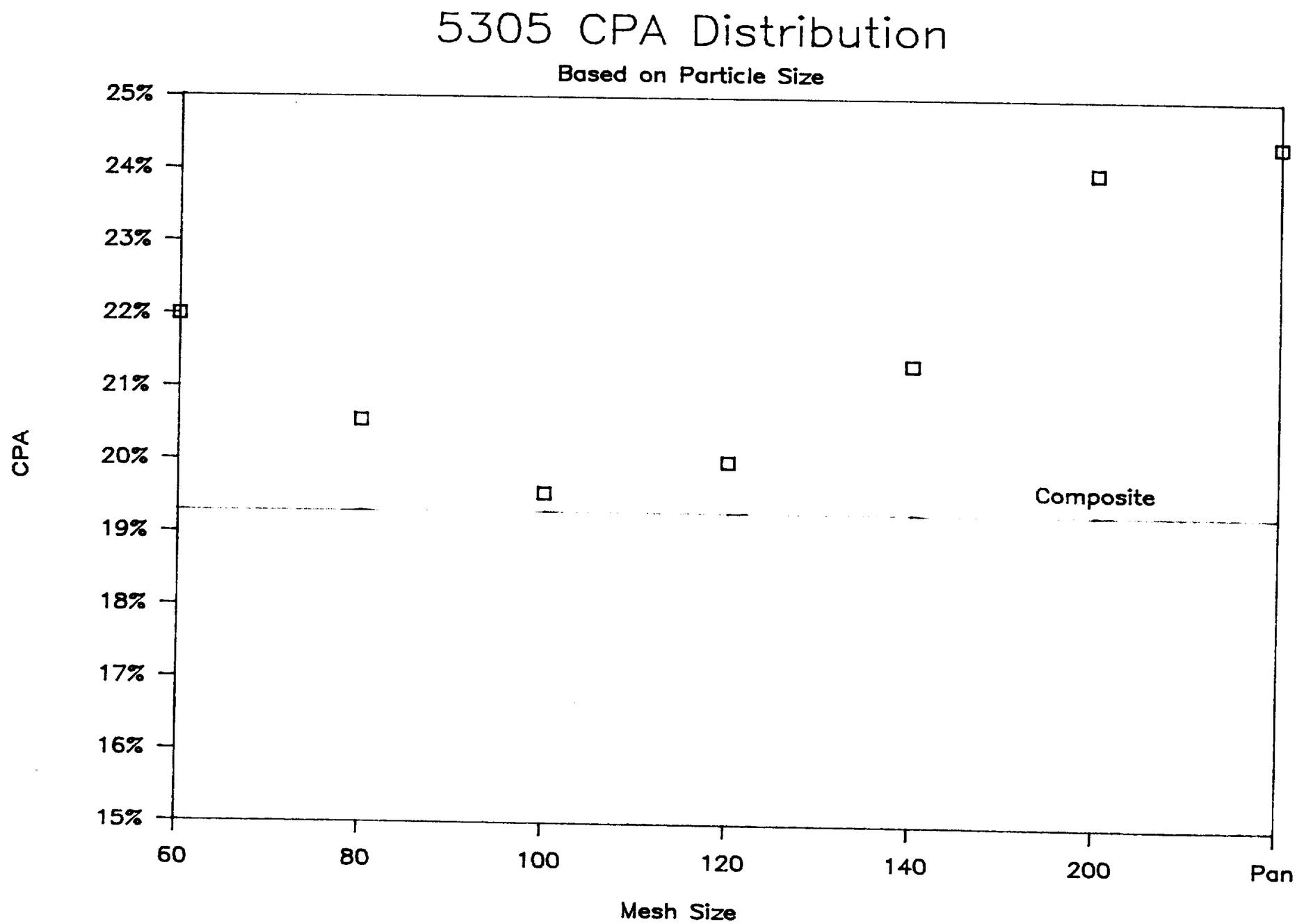

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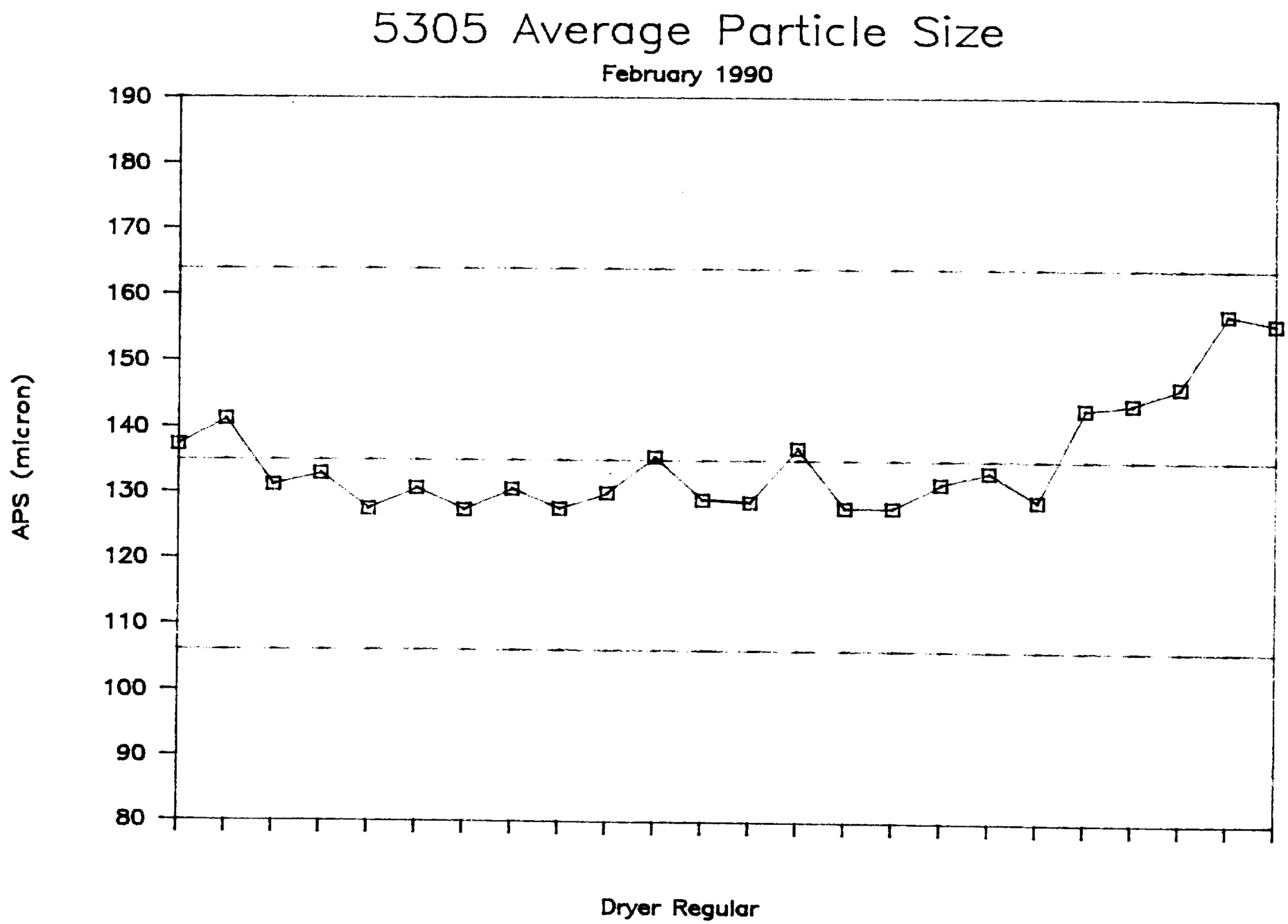

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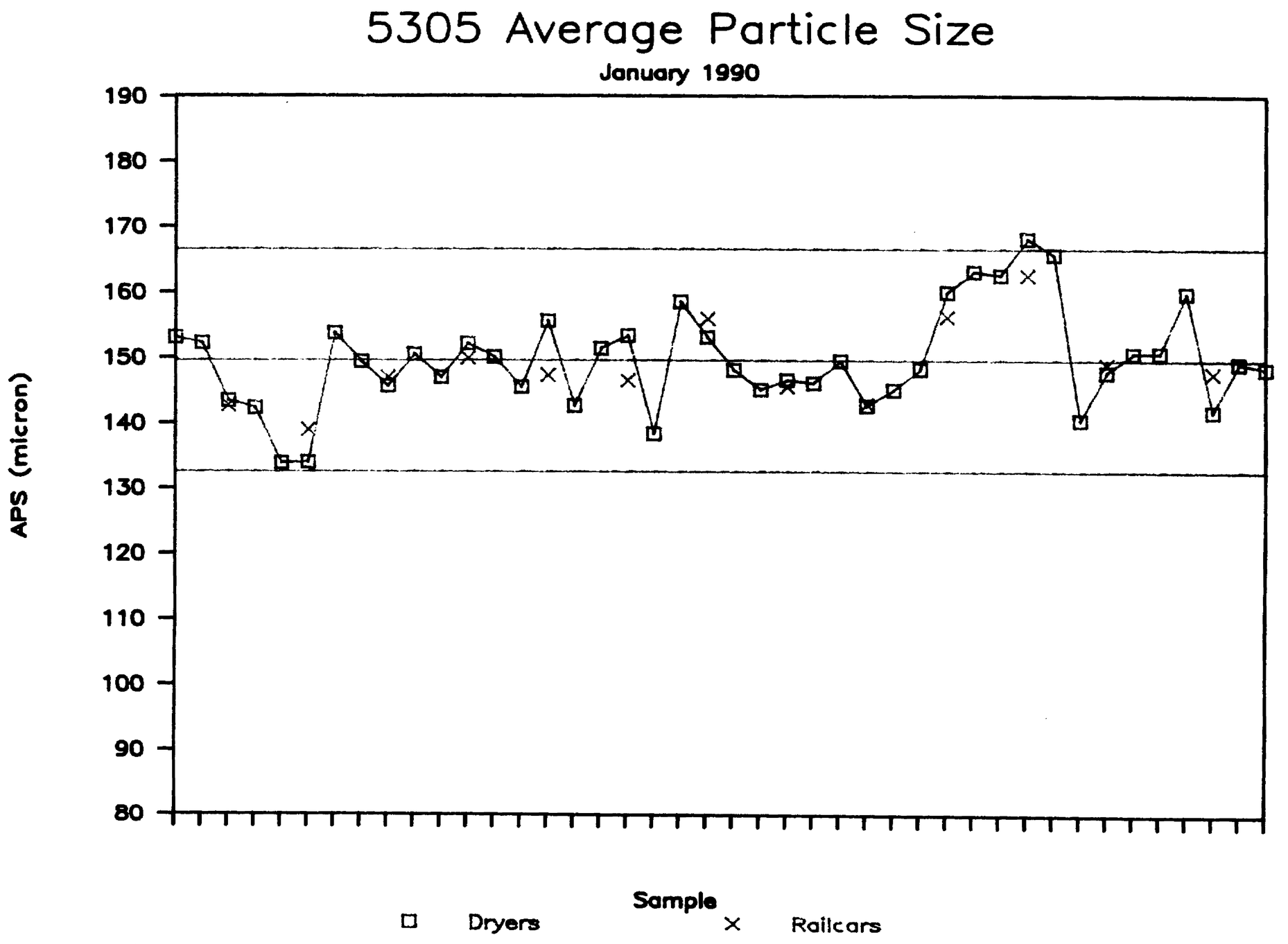

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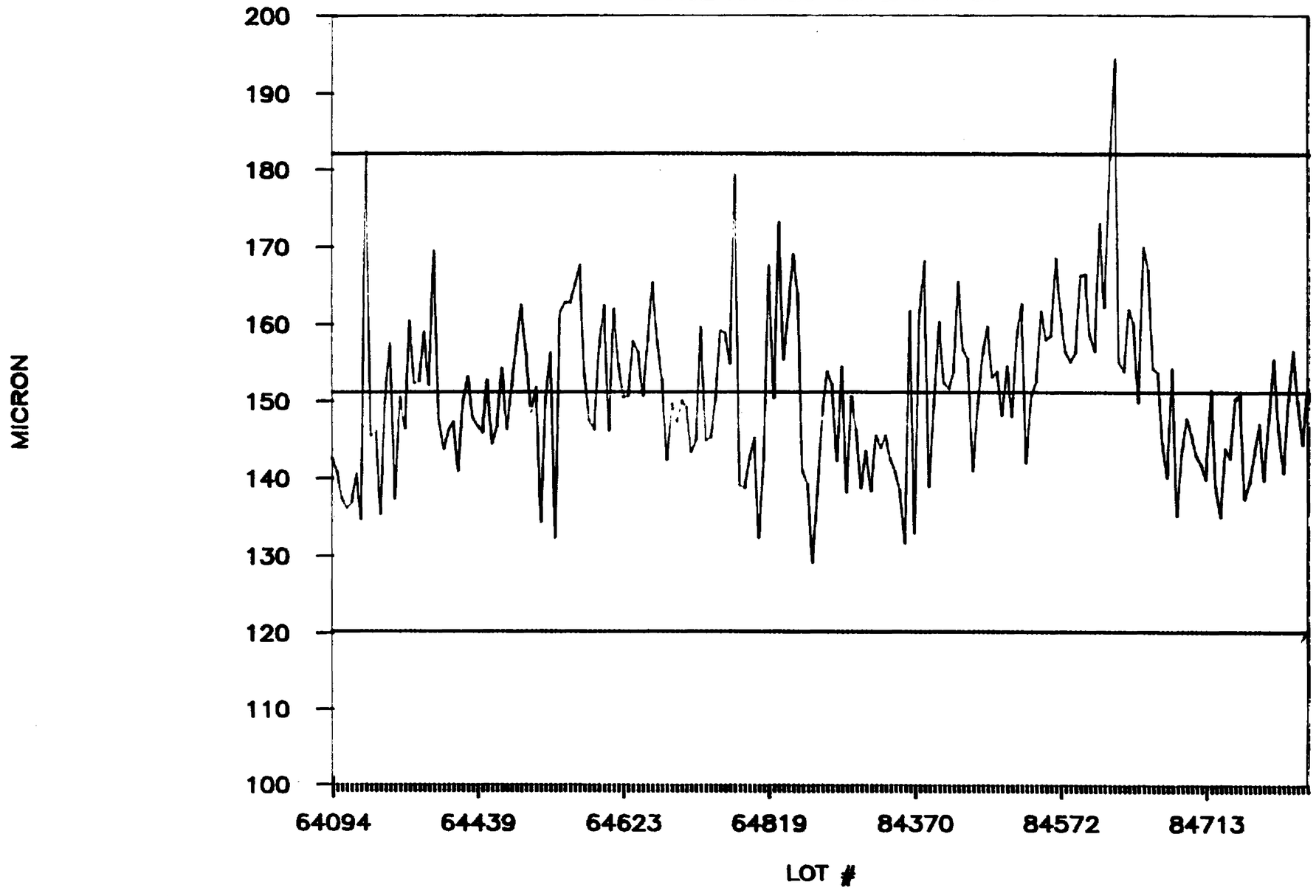
MEAN PARTICLE SIZE

LARGE RX DEC '89 & JAN '90



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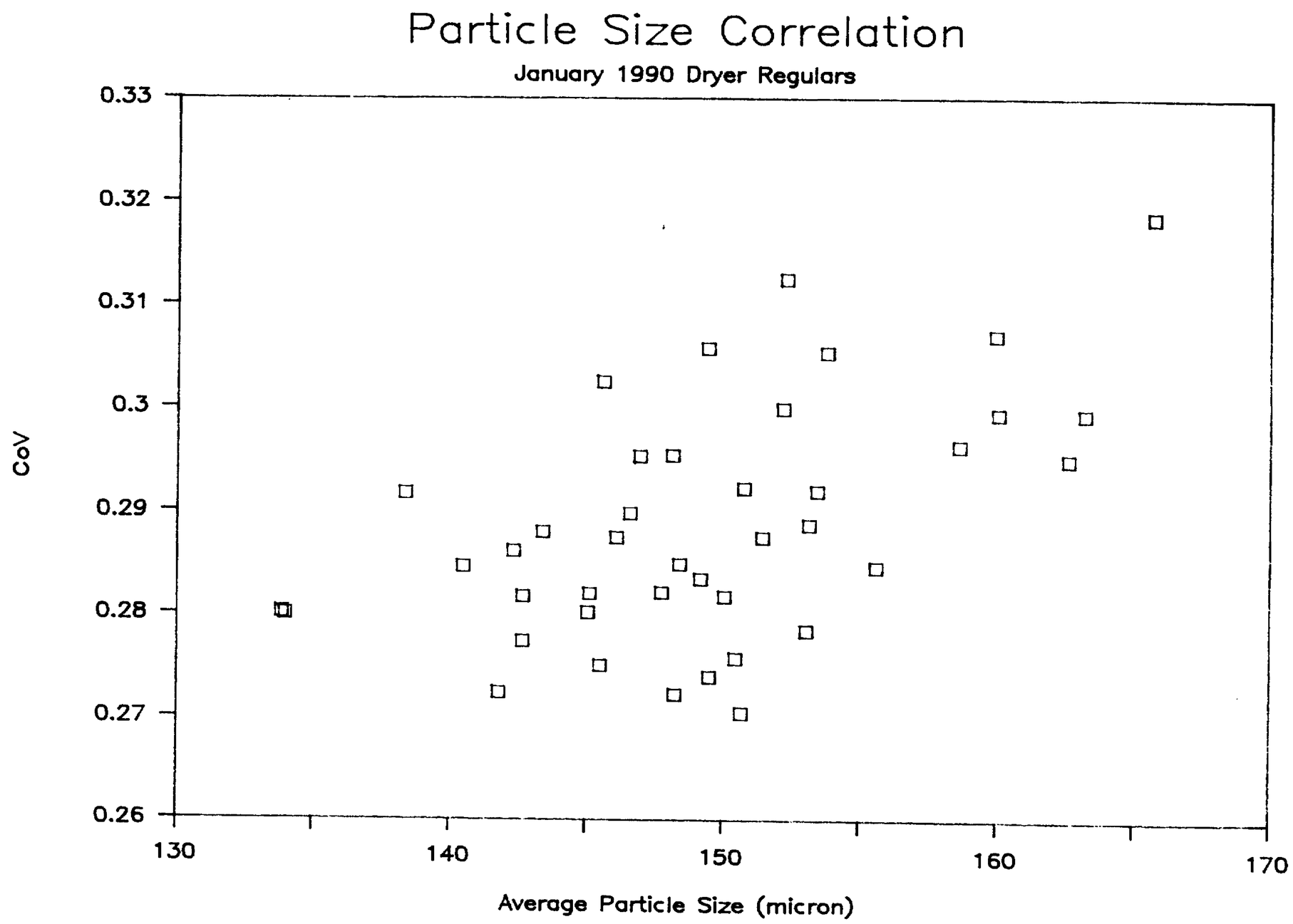

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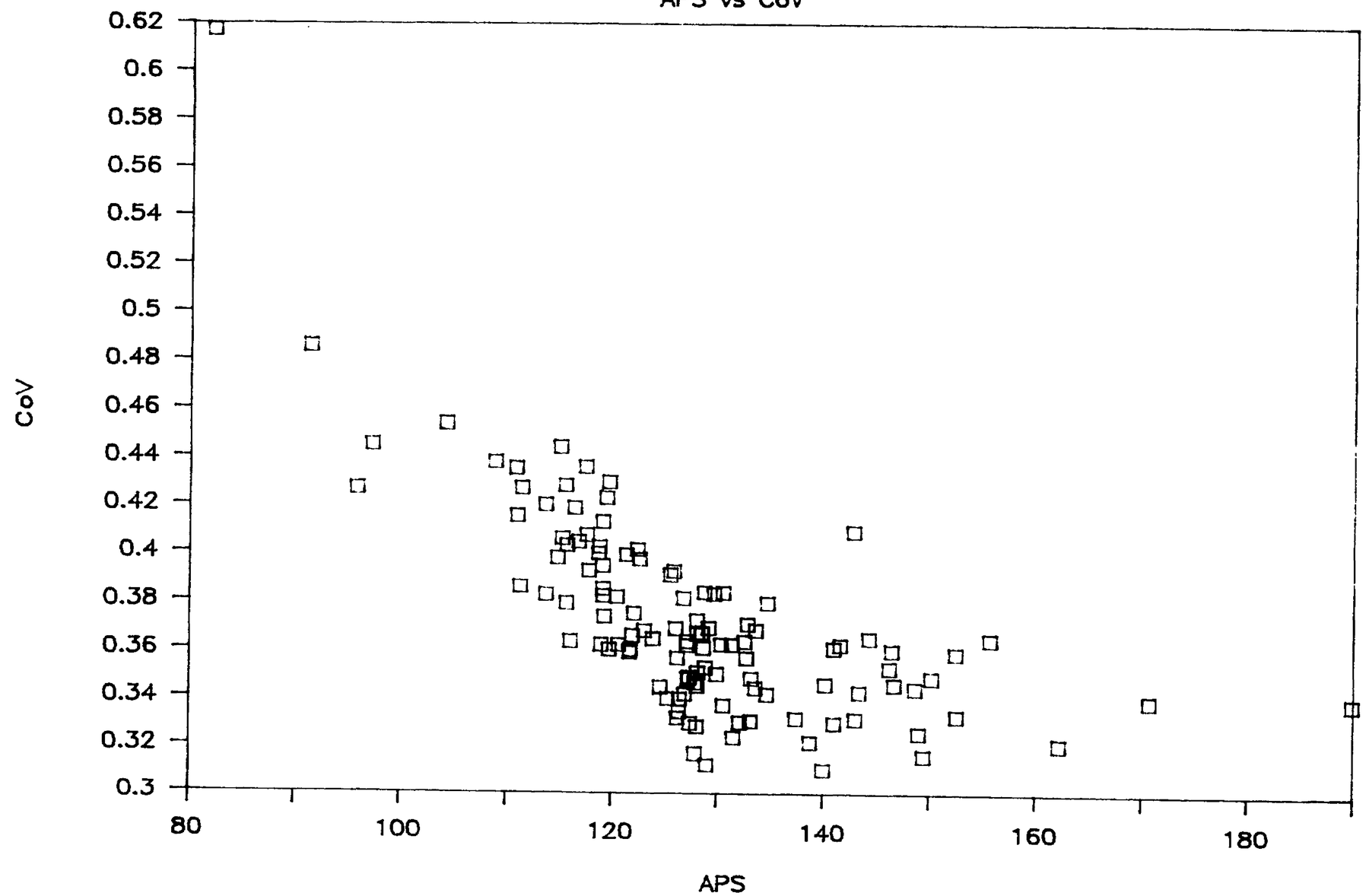
5305 Particle Size Correlation

APS vs CoV



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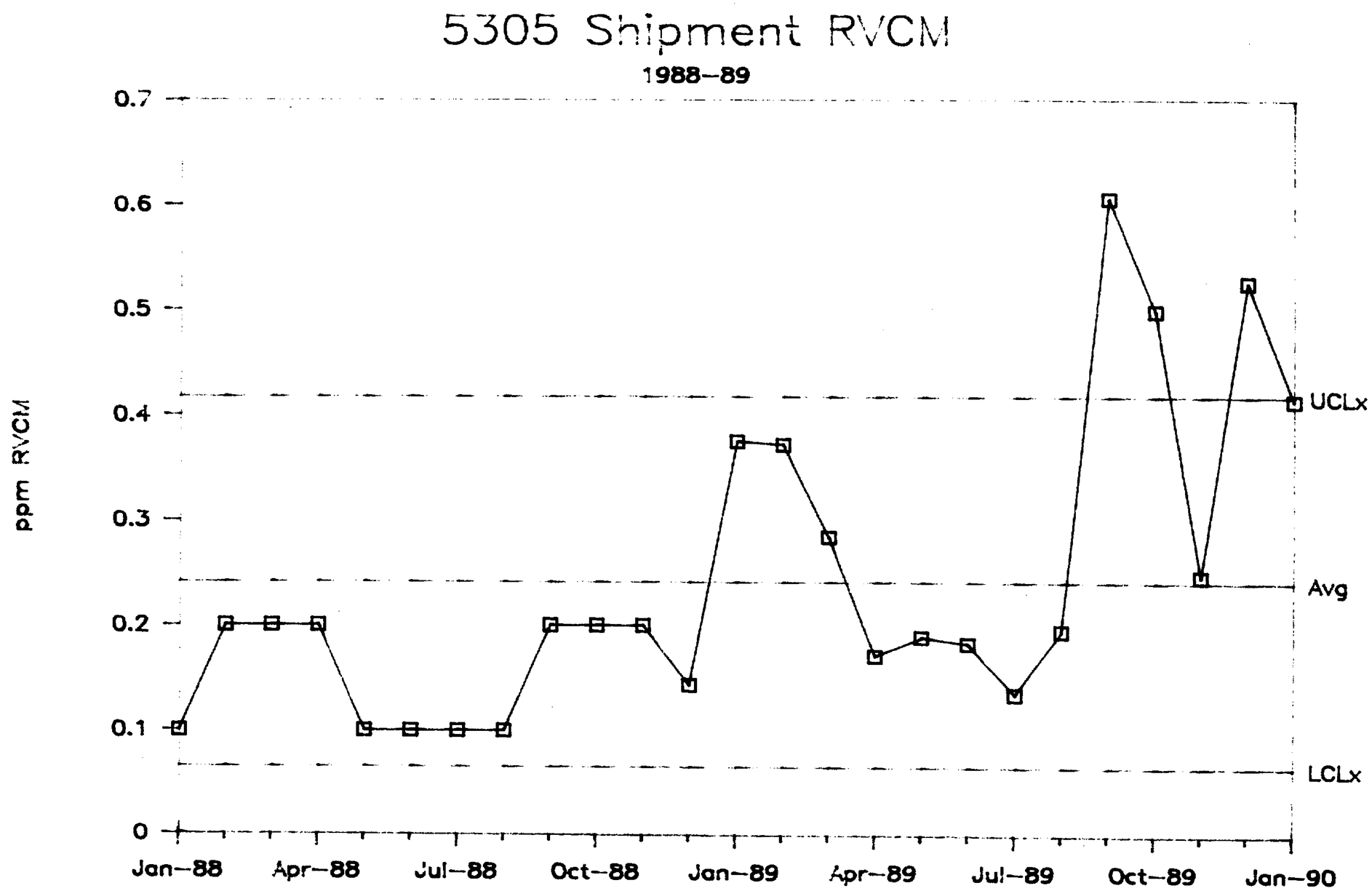
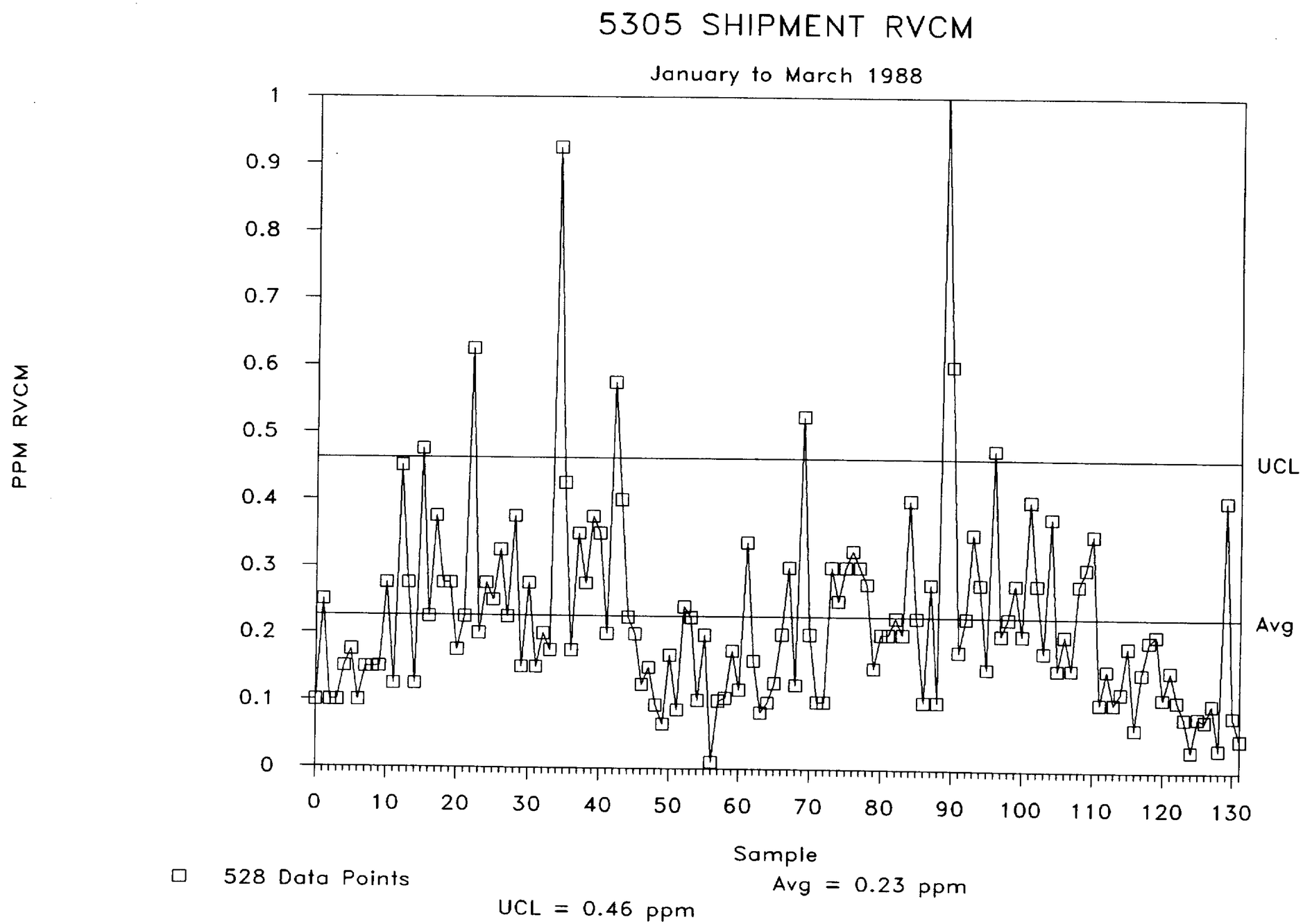

5305 Shipment RVCM
1988–89
ppm RVCM
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
UCLx
Avg
LCLx
Jan–88
Apr–88
Jul–88
Oct–88
Jan–89
Apr–89
Jul–89
Oct–89
Jan–90



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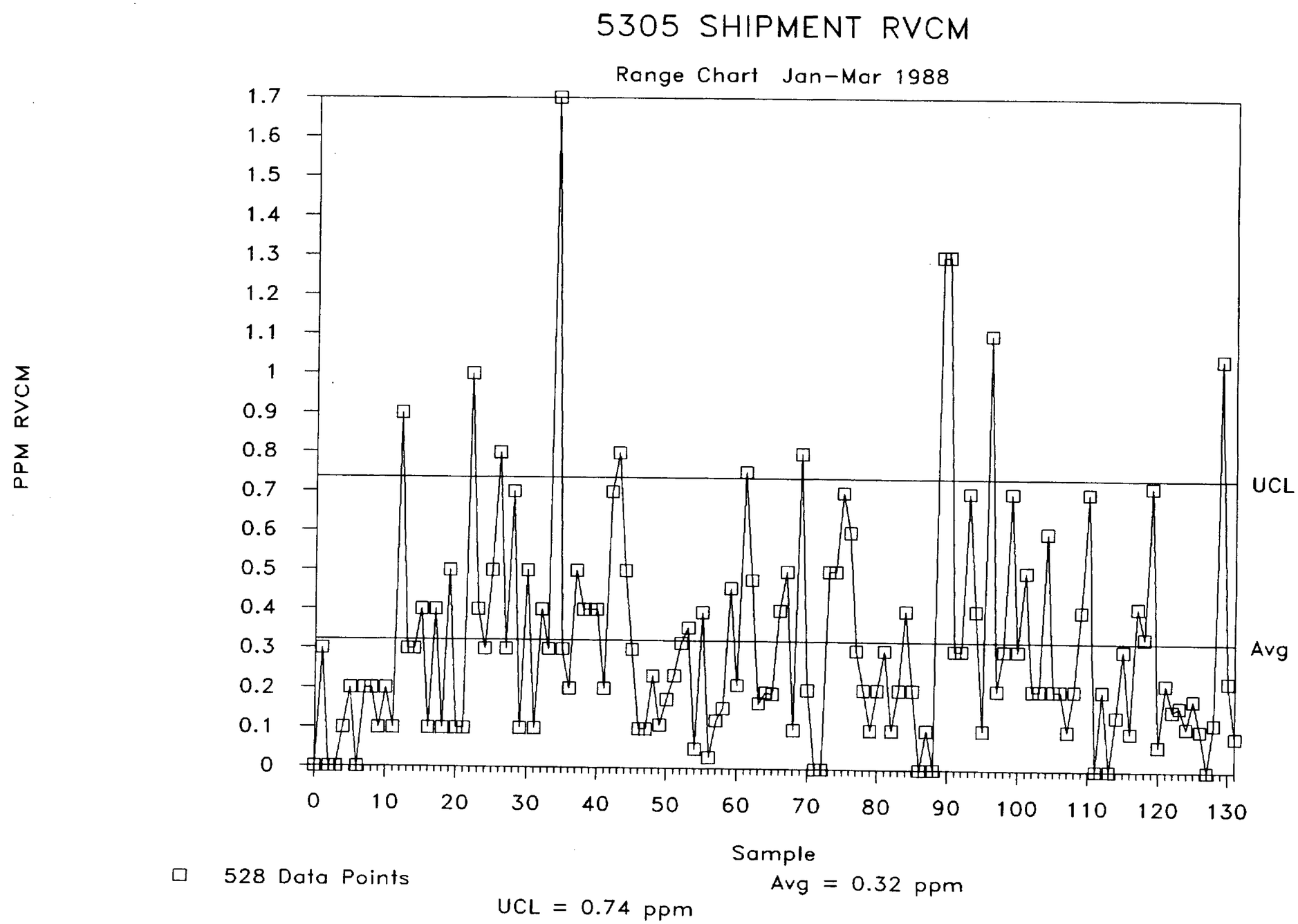

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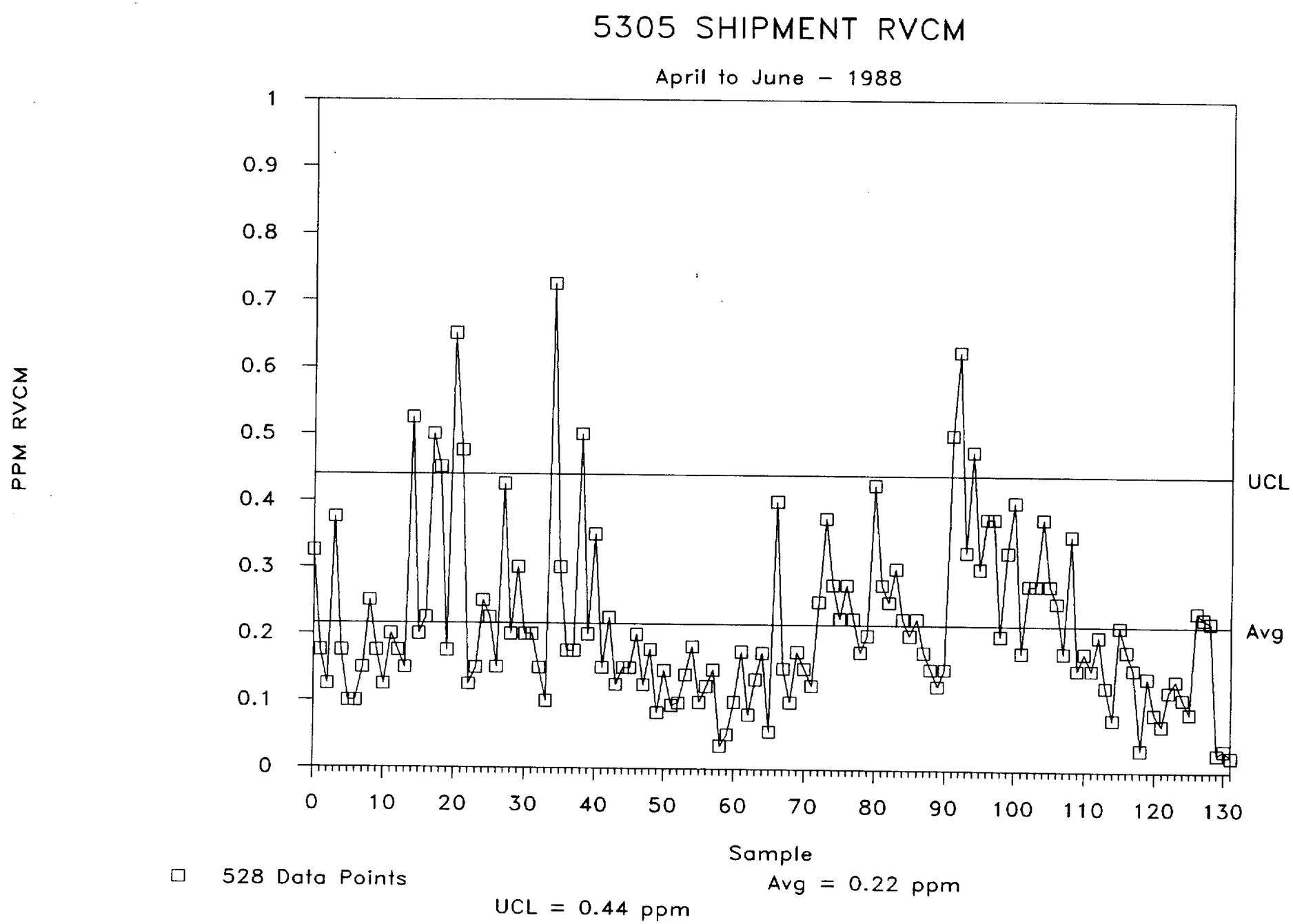
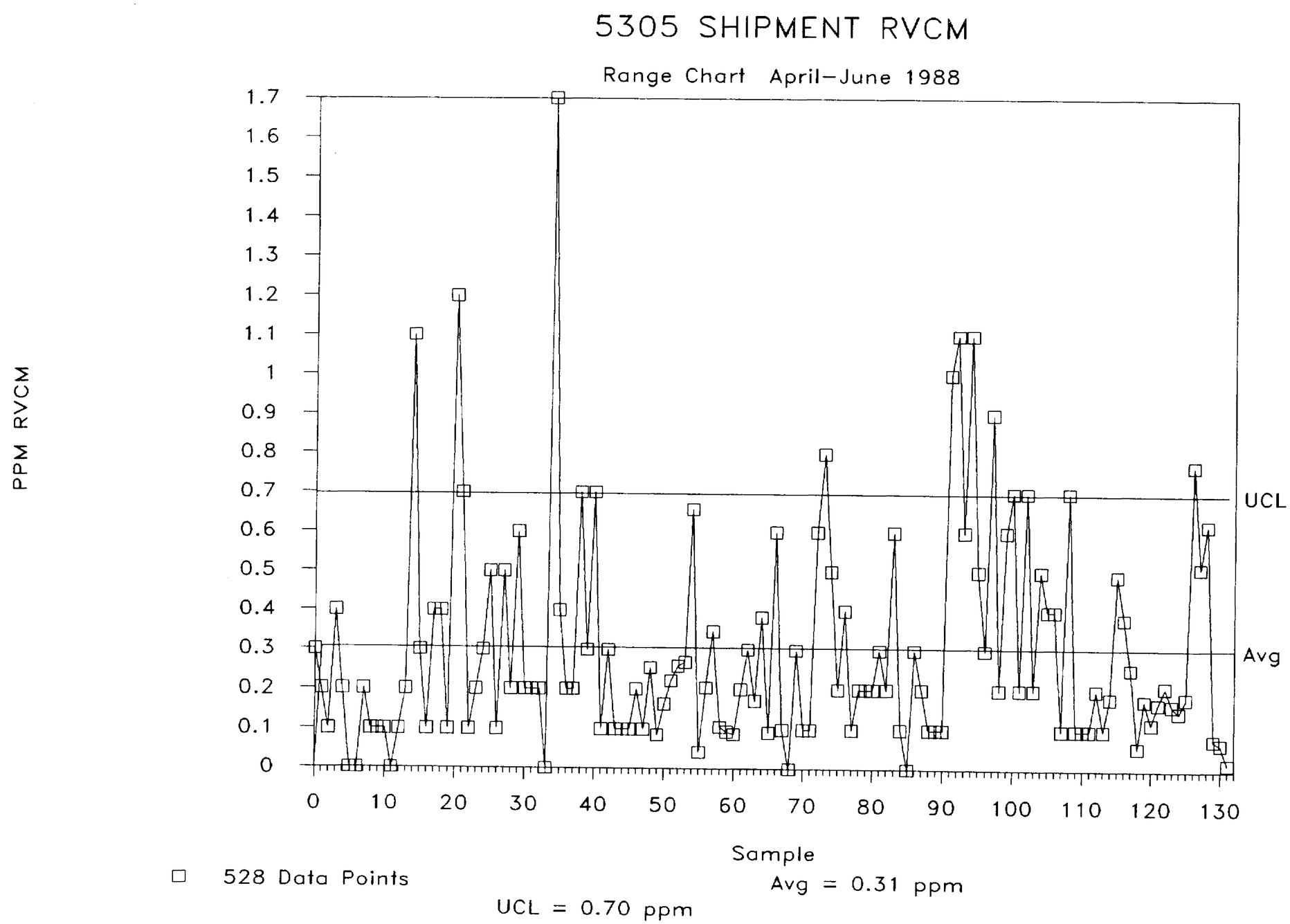
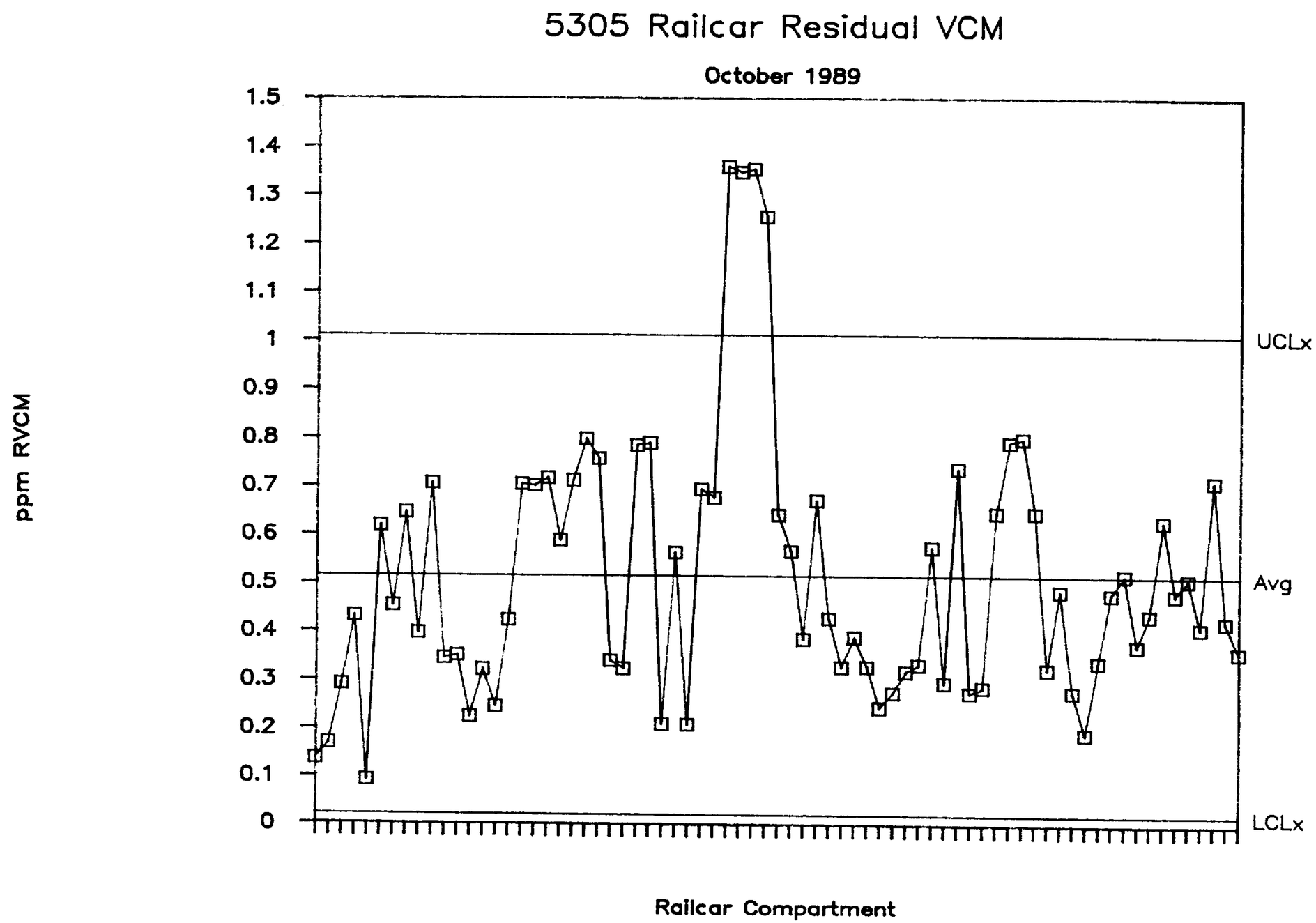

5305 SHIPMENT RVCM
April to June – 1988
PPM RVCM
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
UCL
Avg
0
10
20
30
40
50
60
70
80
90
100
110
120
130
Sample
528 Data Points
Avg = 0.22 ppm
UCL = 0.44 ppm


5305 SHIPMENT RVCM
Range Chart April–June 1988
PPM RVCM
1.7
1.6
1.5
1.4
1.3
1.2
1.1
1
0.9
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0
UCL
Avg
0
10
20
30
40
50
60
70
80
90
100
110
120
130
Sample
528 Data Points
Avg = 0.31 ppm
UCL = 0.70 ppm



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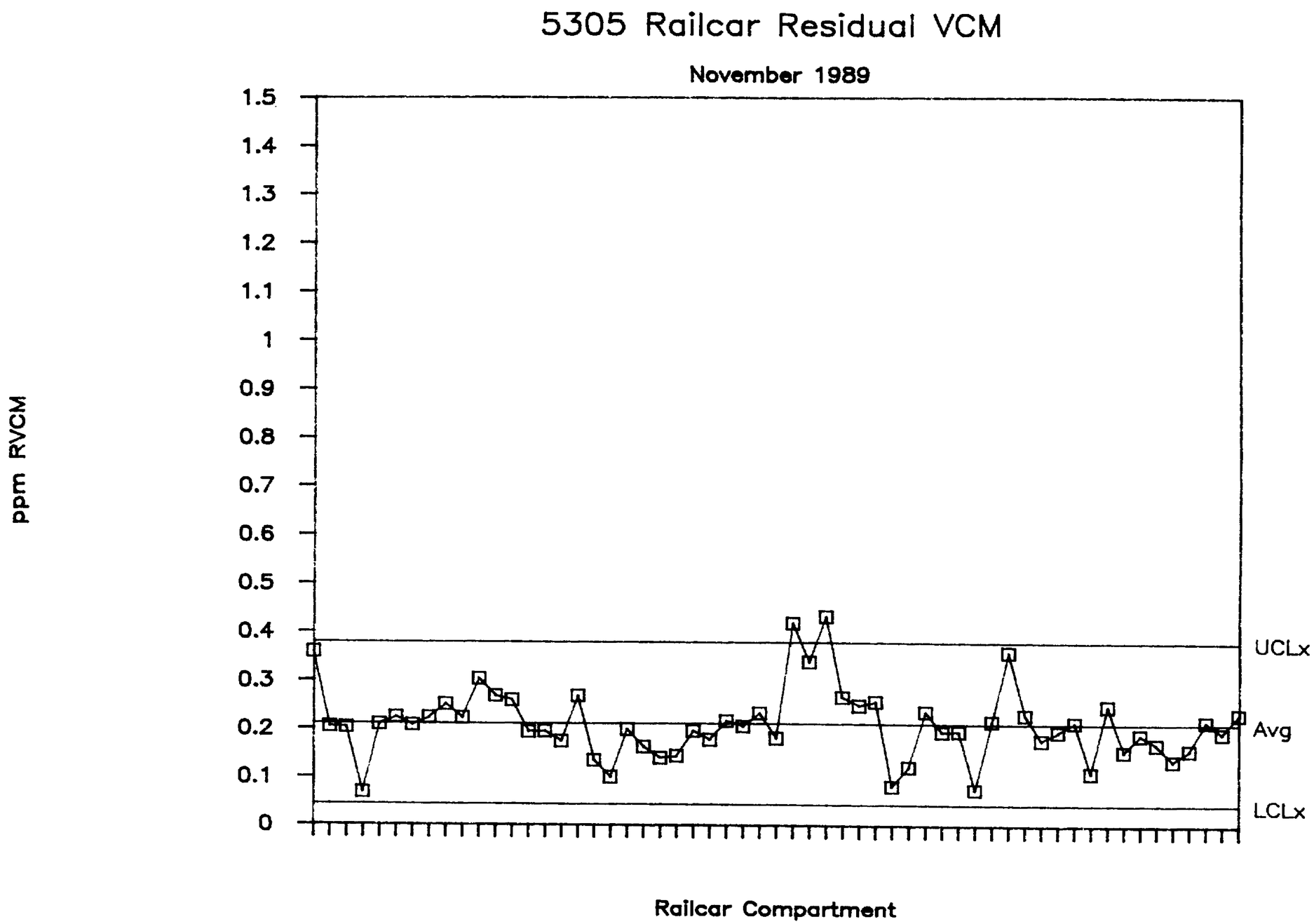

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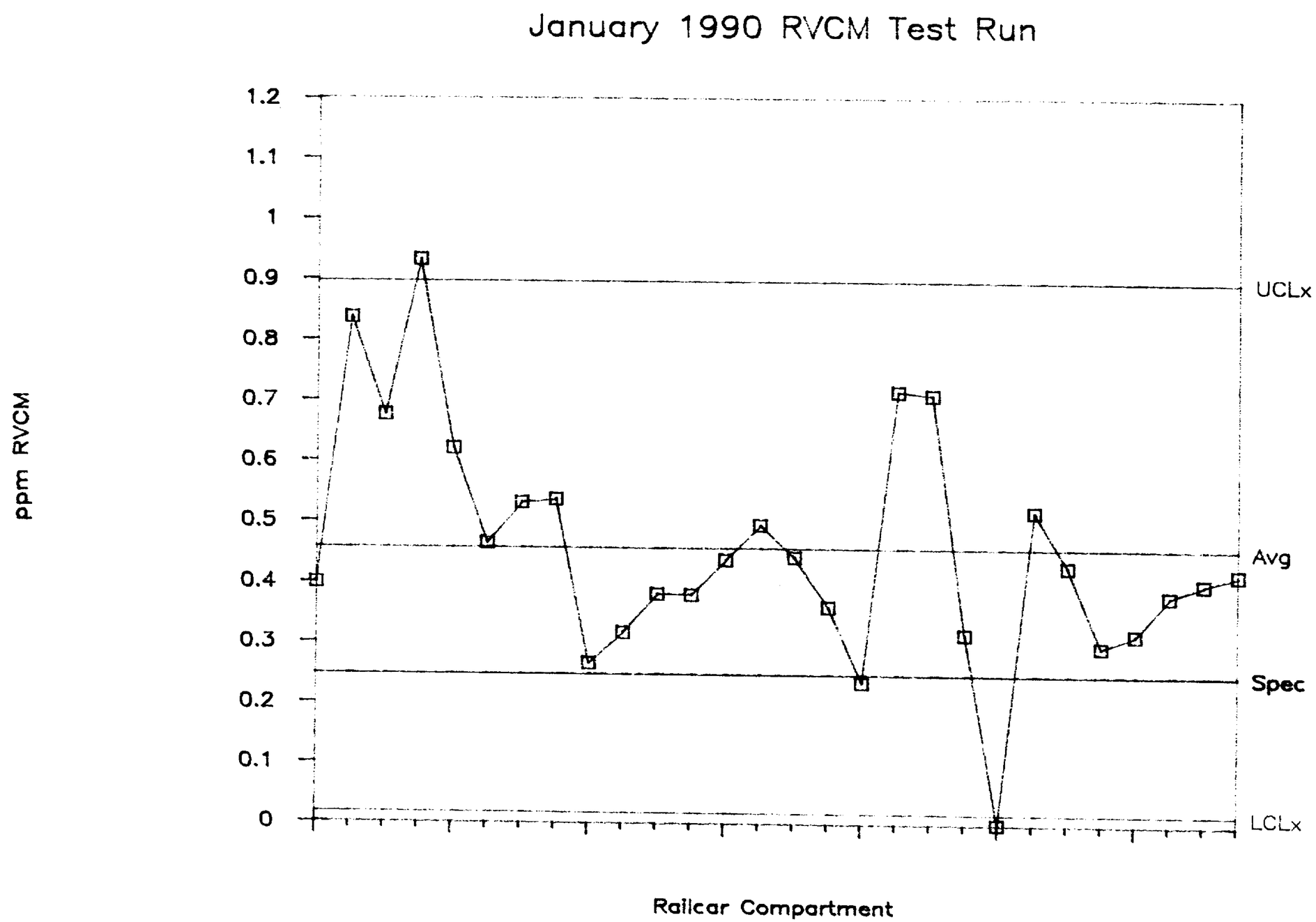


VAB.0001097562

Heated Silo Air

Appendix C

Heated Silo Air

Experimental Design Table
2^3 Designs

How Many Repititions? ==> 3
What is factor A? ==> Aeration Time
What is factor B? ==> Hot Silo Aeration
What is factor C? ==> Isonox Kill Amount

Is the effect of Aeration Time significant? ==> yes
Is the effect of Hot Silo Aeration significant? ==> yes
Is the effect of Isonox Kill Amount significant? ==> no
Is the combined effect of A & B significant? ==> no
Is the combined effect of A & C significant? ==> no
Is the combined effect of B & C significant? ==> no
Is the combined effect of A,B & C significant? ==> no
Is the Aeration Time variability significant? ==> no
Is the Hot Silo Aeration variability significant? ==> no
Is the Isonox Kill Amount variability significant? ==> no

RAW DATA TABLE

Repitition	1	2	3	4	5
Run 1	0.636	0.629	0.836		
2	0.527	0.590	0.722		
3	0.328	0.290	0.550		
4	0.179	0.165	0.469		
5	0.450	0.390	1.111		
6	0.354	0.316	0.960		
7	0.362	0.354	0.710		
8	0.310	0.272	0.588		

Run	Mean	A	B	C	AB	AC	BC	ABC
1	+	-	-	-	+	+	+	-
2	+	+	-	-	-	-	+	+
3	+	-	+	-	-	+	-	+
4	+	+	+	-	+	-	-	-
5	+	-	-	+	+	-	-	+
6	+	+	-	+	-	+	-	-
7	+	-	+	+	-	-	+	-
8	+	+	+	+	+	+	+	+

Run	Mean	A	B	C	AB	AC	BC	ABC
1	0.70	-0.70	-0.70	-0.70	0.70	0.70	0.70	-0.70
2	0.61	0.61	-0.61	-0.61	-0.61	-0.61	0.61	0.61
3	0.39	-0.39	0.39	-0.39	-0.39	0.39	-0.39	0.39
4	0.27	0.27	0.27	-0.27	0.27	-0.27	-0.27	-0.27
5	0.65	-0.65	-0.65	0.65	0.65	-0.65	-0.65	0.65
6	0.54	0.54	-0.54	0.54	-0.54	0.54	-0.54	-0.54
7	0.48	-0.48	0.48	0.48	-0.48	-0.48	0.48	-0.48
8	0.39	0.39	0.39	0.39	0.39	0.39	0.39	0.39
Sum+	4.03	1.82	1.53	2.06	2.01	2.02	2.18	2.04
Sum-	0.00	2.22	2.51	1.97	2.02	2.01	1.85	1.99
Overall	4.03	4.03	4.03	4.03	4.03	4.03	4.03	4.03
Difference	4.03	-0.40	-0.98	0.09	-0.01	0.01	0.32	0.05
Effect	1.01	-0.20	-0.49	0.04	-0.00	0.01	0.16	0.03
F		1.49	2.70	1.09	1.01	1.01	1.38	1.05

Deviation	0.222
MSFE	0.19
d2	1.693
V	16
v-num/dem	8
Table F	3.44
Table t	2.12

RJL
A

To: R. B. Quy, R&D, Ponca City

From: R. J. Lahiere, Polymers Research

Date: June 11, 1987

Subject: ENHANCED RVCM REDUCTION WITH HEATED RESIN IN BENCH SILO

Interoffice Communication

VISTA

Previous work with the bench-scale silo indicated that resin temperature influences the extent of RVCM removal from resin with aeration. Temperature effects are an important aspect of silo aeration because RVCM transfer rates have decreased during cold weather operation in the past.

This investigation confirmed that the RVCM transfer efficiency from resin during silo aeration is increased at higher resin temperatures. The temperature of the resin used in the silo was adjusted by drying the resin in a fluid bed drier, thus heating it to about 130°F. Silo aeration experiments employing heated resin were compared to previous work in which resin initially at ambient temperature was used. Conclusions of the study are discussed in greater detail below.

Steps should be taken to warm silo resin during winter conditions to prevent the reduction of RVCM removal. The resin can be warmed by (1) heating the aeration air and (2) improving air distribution in the silos with more efficient sparger designs.

More RVCM Removal in Heated Resin Aerated With Ambient and Hot Air

Silo aeration with air at 77°F and 150°F removed more RVCM from resin when the initial resin temperature was 130°F, rather than 77°F. This effect is shown in Figures 1 and 2.

The average temperature during the run of the heated resin was greater than that of the ambient-temperature resin. Since the heated resin stayed warmer, the diffusion of vinyl chloride out of the resin particle was increased. This diffusion mechanism is the rate-controlling step of the mass transfer process in the silo; higher diffusion rates resulted in more RVCM removed from PVC.

Less RVCM Removal in Heated Resin Aerated With Cold Air

Less RVCM was transferred from heated resin than from resin at ambient temperature when the silo was aerated with 40°F air, as Figure 3 shows. When heated resin was aerated, the silo was wrapped in insulation and a cooler average resin temperature was maintained with the cold aeration air. The resin temperature averaged about 46°F during the latter part of the experiment with hot resin and an insulated silo compared to 64°F for the run with ambient-temperature resin and a silo without insulation. Lower resin temperatures in the heated-resin experiment reduced the amount of RVCM removed.

A

Richard J. Lahiere

Richard J. Lahiere

attachments

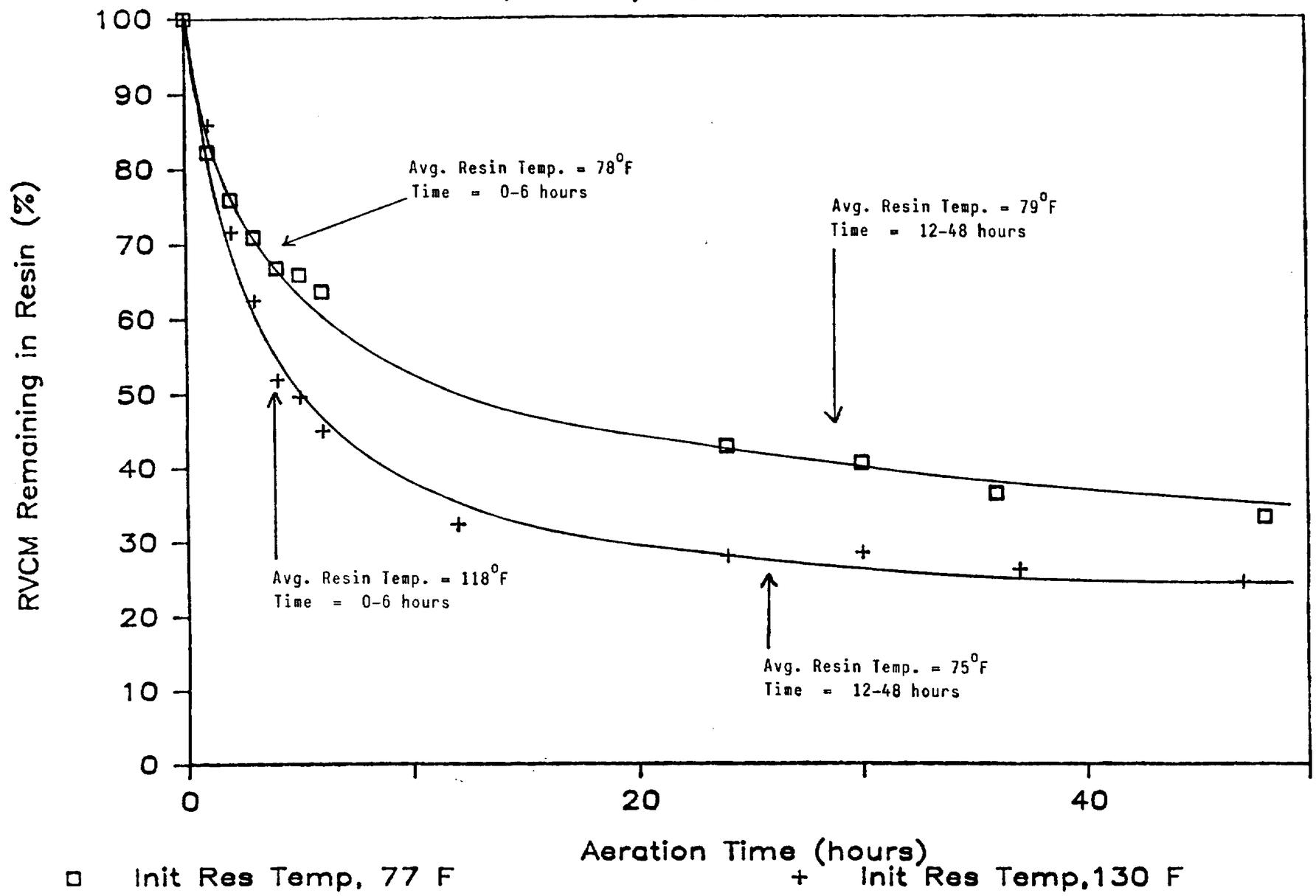
cc/attachments:

Ponca City: SEM CJMc AWW MEO WCT RRH JLC
Aberdeen: DWH VEM FGJ
Houston: LZK JFN
Oklahoma City: JDO RP
Saddlebrook: RDJ

Figure 1

Effect of Init Resin Temp in Bench-Silo

77 F Air, 0.95 scfm Flow Rate



VAB.0001097569

Figure 2

Effect of Init Resin Temp in Bench-Silo

150 F Air, 0.38 scfm Flow Rate

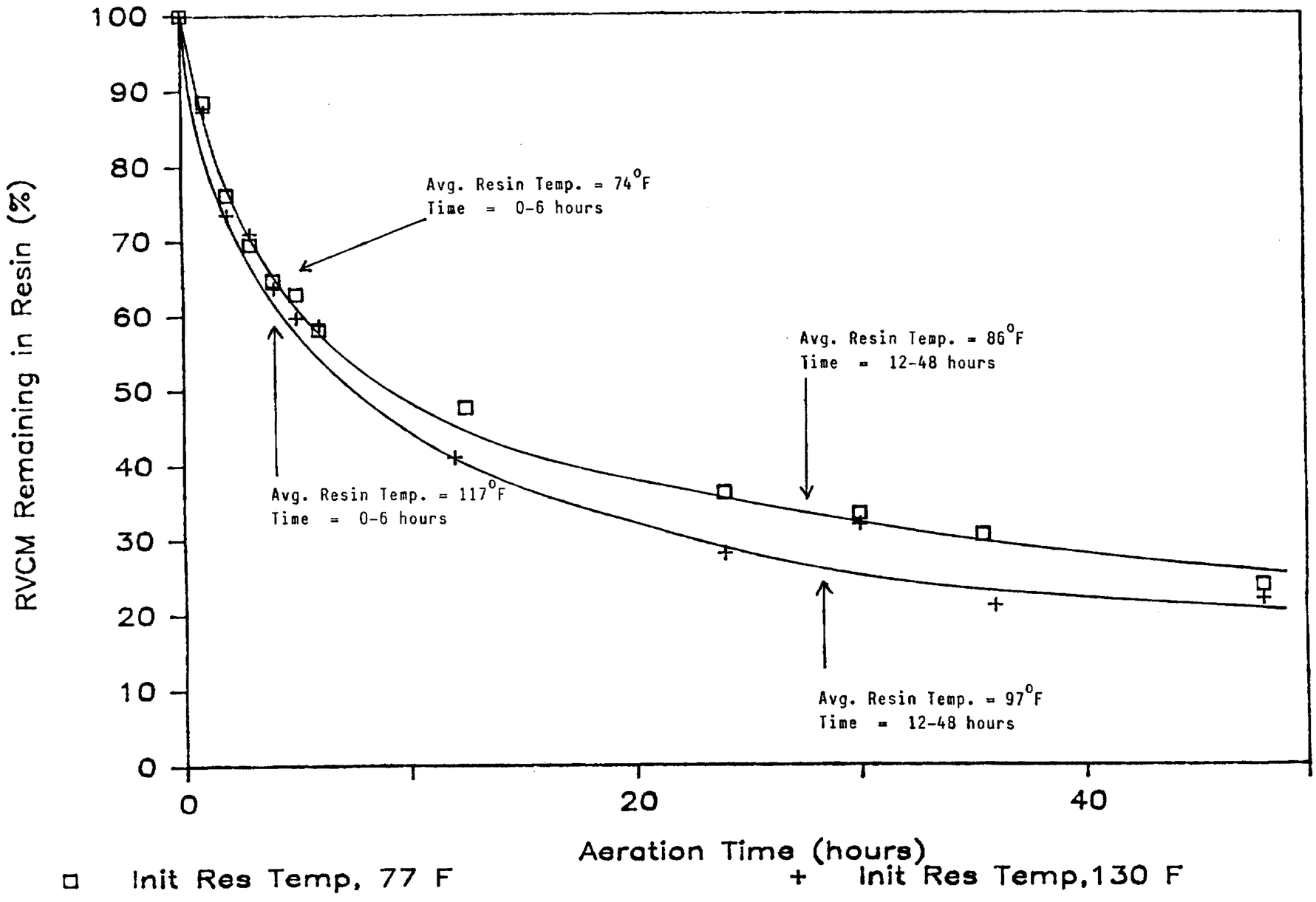


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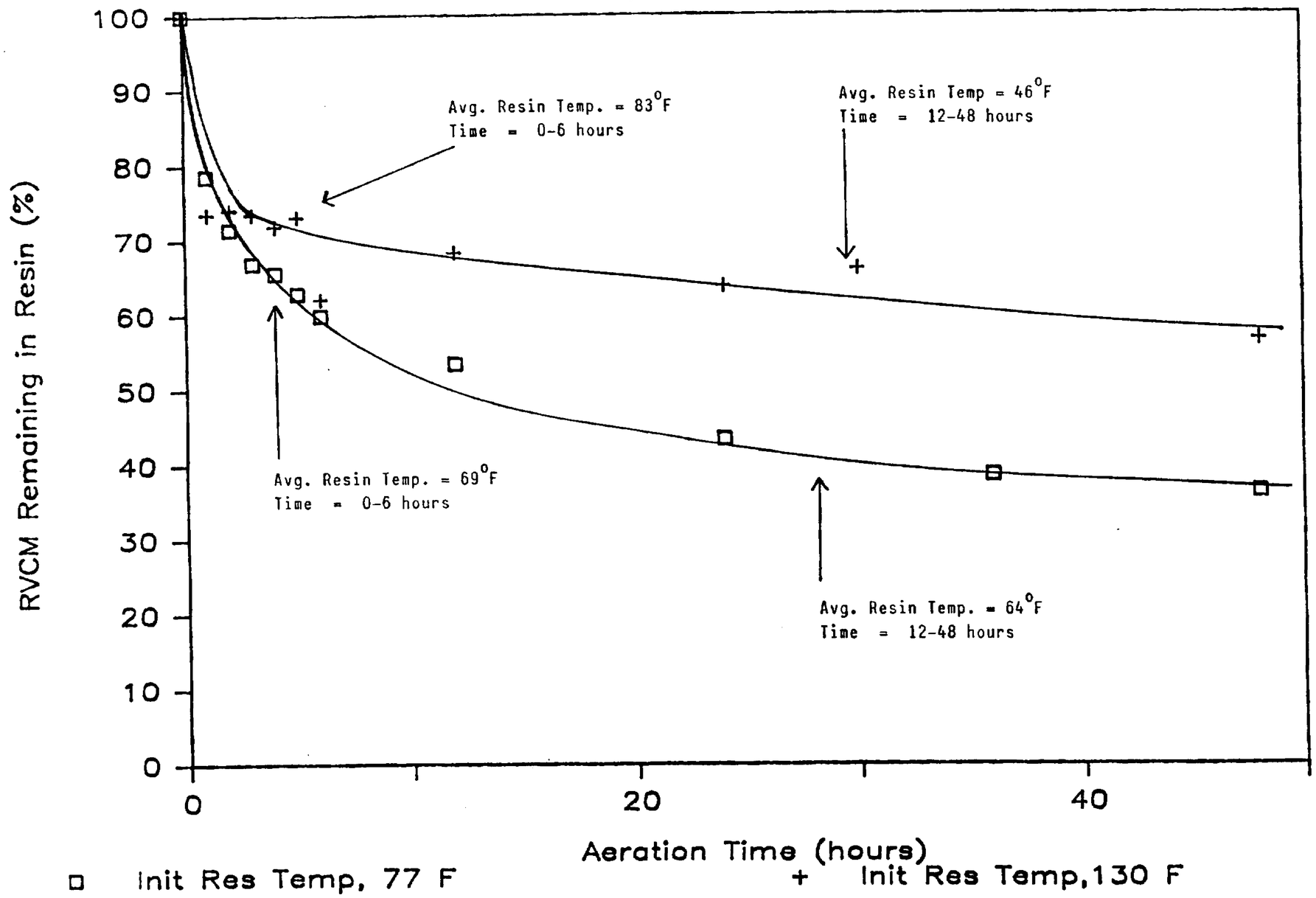
Figure 3

Effect of Init Resin Temp in Bench-Silo

40 F Air, 1.90 scfm Flow Rate



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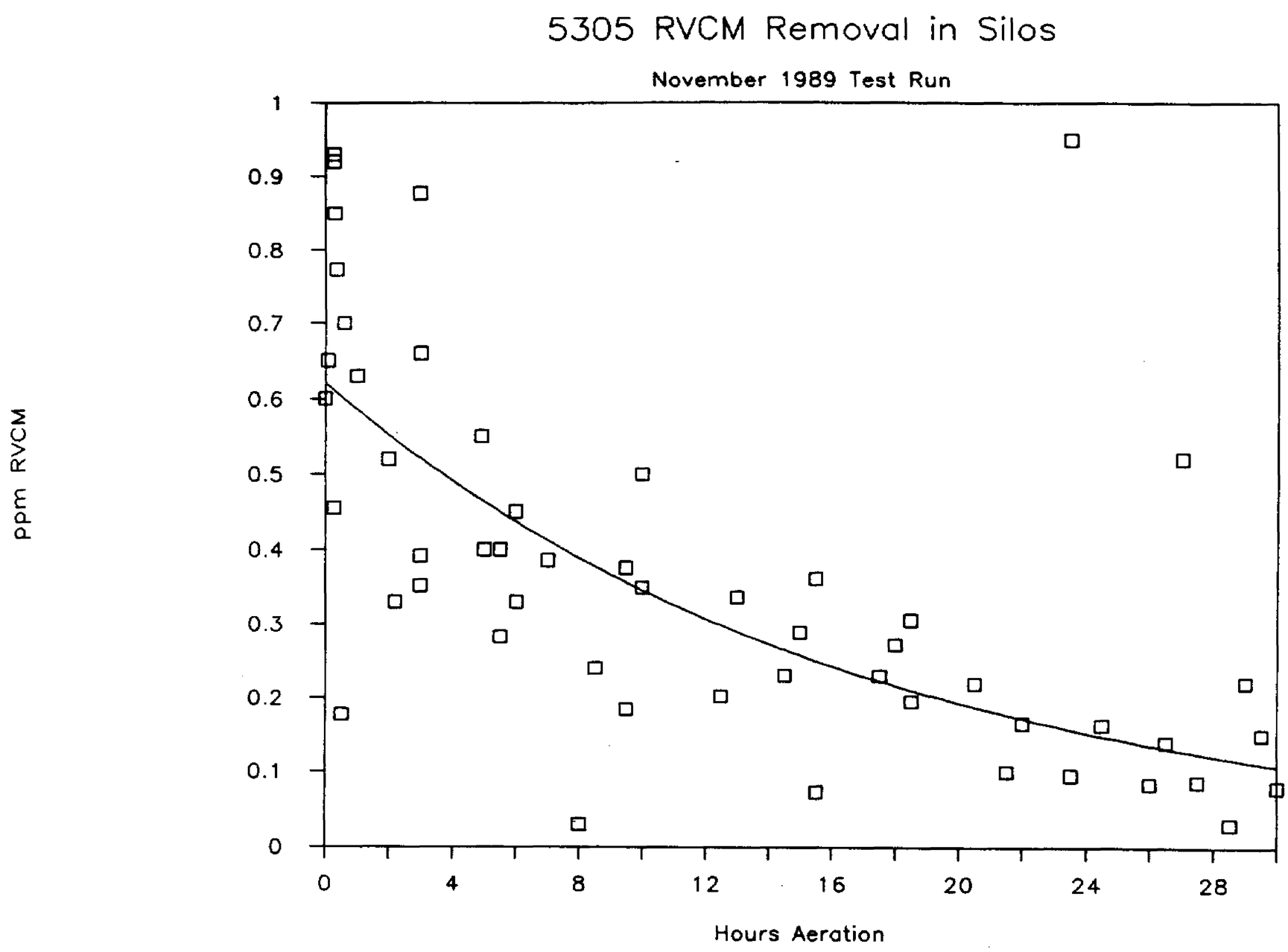

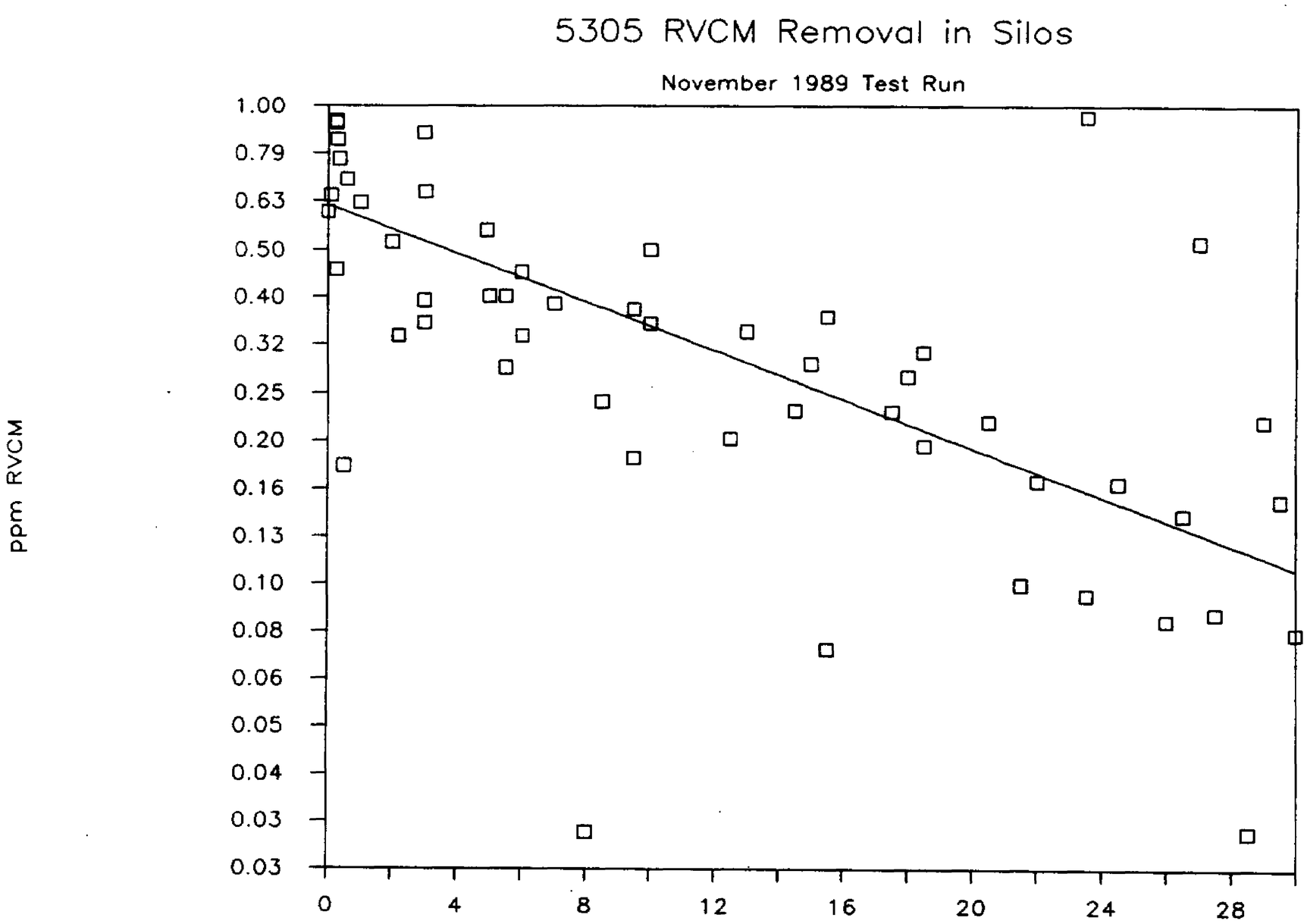

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Heated Blend
Tanks

Appendix D

Heated Blend Tanks

A

2/2 FACTORIAL DESIGN

PROJECT TITLE: Small Blend Tank RVCM Removal DATE: Janurary 25, 1990

RUN#		MEAN		A		B		AB	Y-BAR	R
1	+	1.09	-	1.09	-	1.09	+	1.09	1.09	0.73
2	+	0.40	+	0.40	-	0.40	-	0.40	0.40	0.42
3	+	0.62	-	0.62	+	0.62	-	0.62	0.62	0.53
4	+	0.39	+	0.39	+	0.39	+	0.39	0.39	0.29

SUM+	2.50	0.78	1.01	1.48
SUM-	0.00	1.71	1.49	1.02
OVERALL	2.50	2.50	2.50	2.50
DIFFERENCE	2.50	-0.93	-0.48	0.47
EFFECT	0.62	-0.46	-0.24	0.23
STD. DEV.	0.21	MSFE	0.20	

T-VALUE= 2.12 F-VALUE= 3.44 d2= 2.326

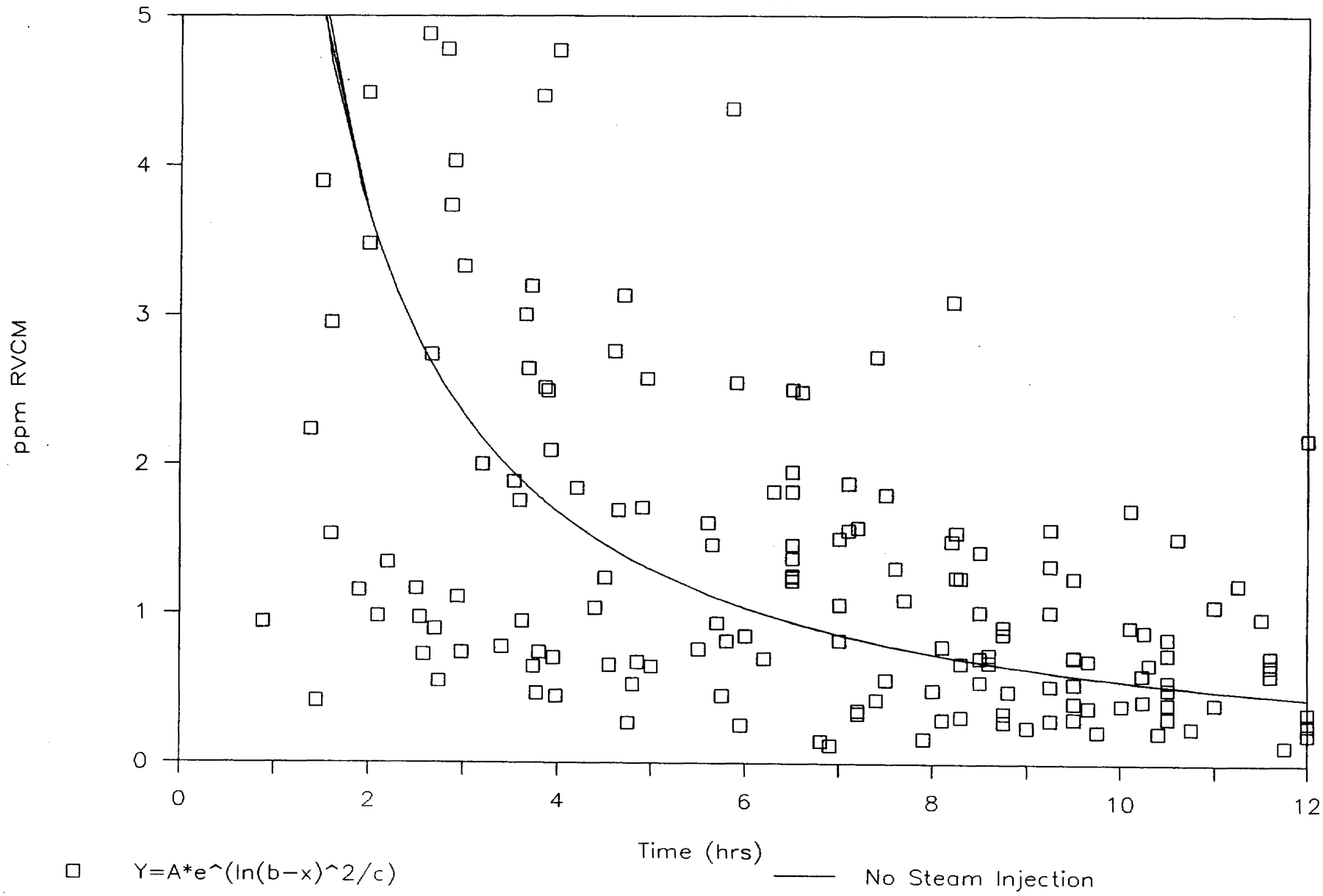
IS THE EFFECT OF A (Steam Injection) SIGNIFICANT? YES
IS THE EFFECT OF B (Hot Water Rinse) SIGNIFICANT? YES
IS THE EFFECT OF A (Steam Injection) & B (Hot Water Rinse) SIGNIFICANT? YES
IS THE VARIABILITY OF A (Steam Injection) SIGNIFICANT? NO
IS THE VARIABILITY OF B (Hot Water Rinse) SIGNIFICANT? NO
IS THE VARIABILITY OF A (Steam Injection) & B (Hot Water Rinse) SIGNIFICANT? NO

RAW DATA

REP #	1	2	3	4	5
RUN 1	1.48	1.43	0.95	0.86	0.75
RUN 2	0.62	0.49	0.45	0.22	0.20
RUN 3	0.74	0.89	0.55	0.36	0.56
RUN 4	0.24	0.23	0.52	0.45	0.50
CENTER PTS	0.00	0.00	0.00	0.00	0.00

5305 Blend Tank RVCM Removal

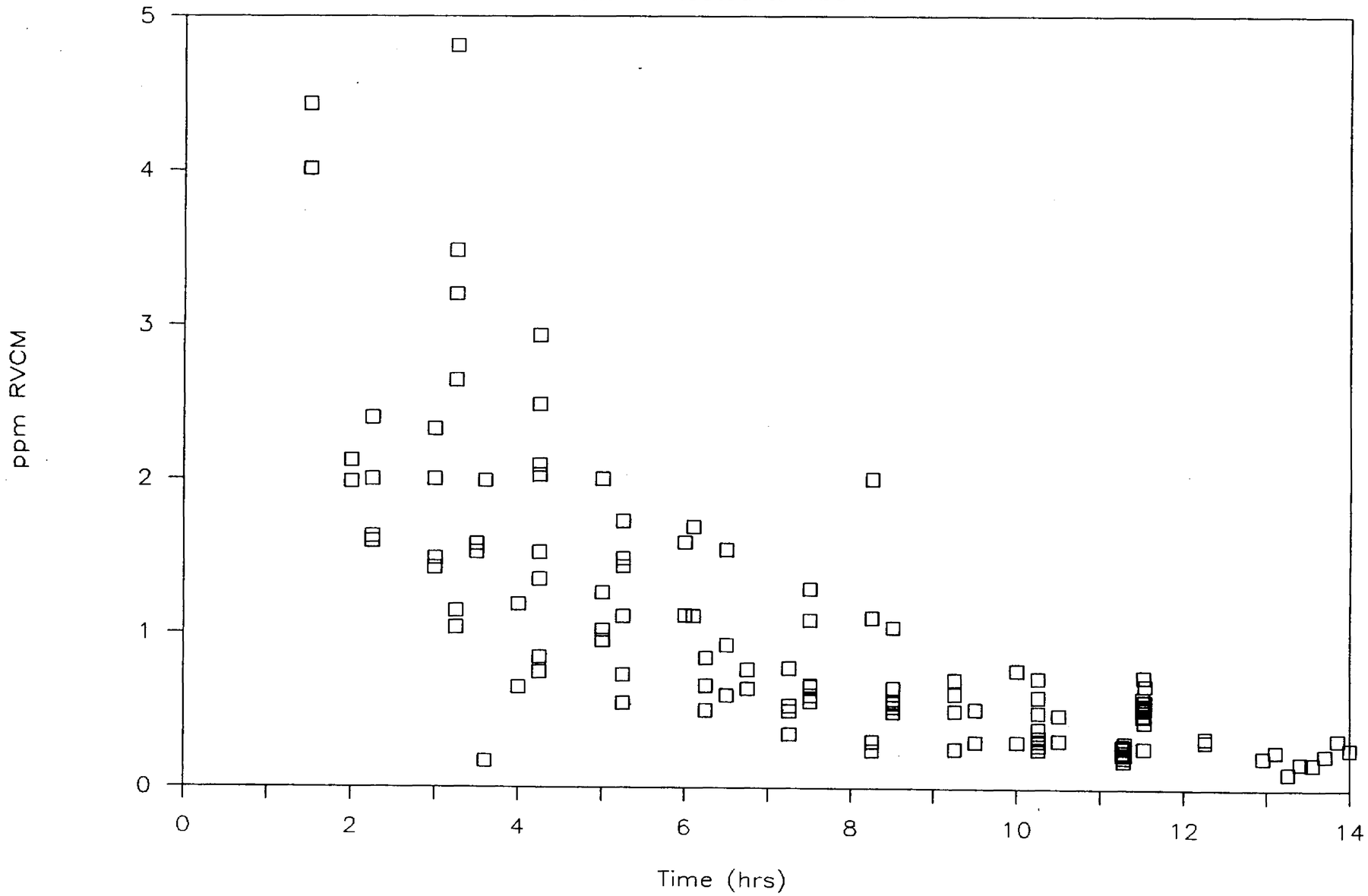
November 1989 Test Run



VAB.0001097576

5305 Small Blend Tank RVCM Removal

Steam Heated to 150 F



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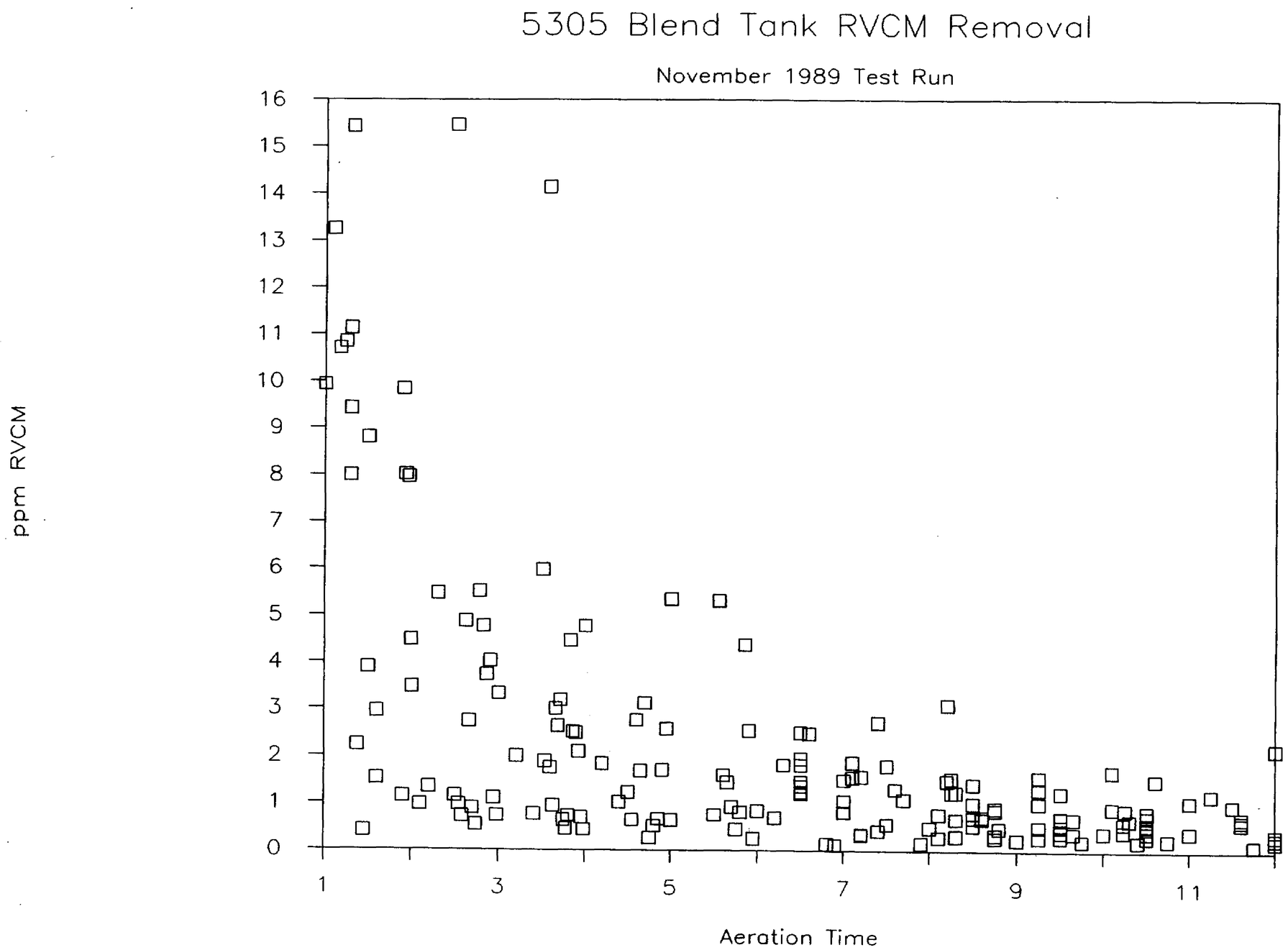

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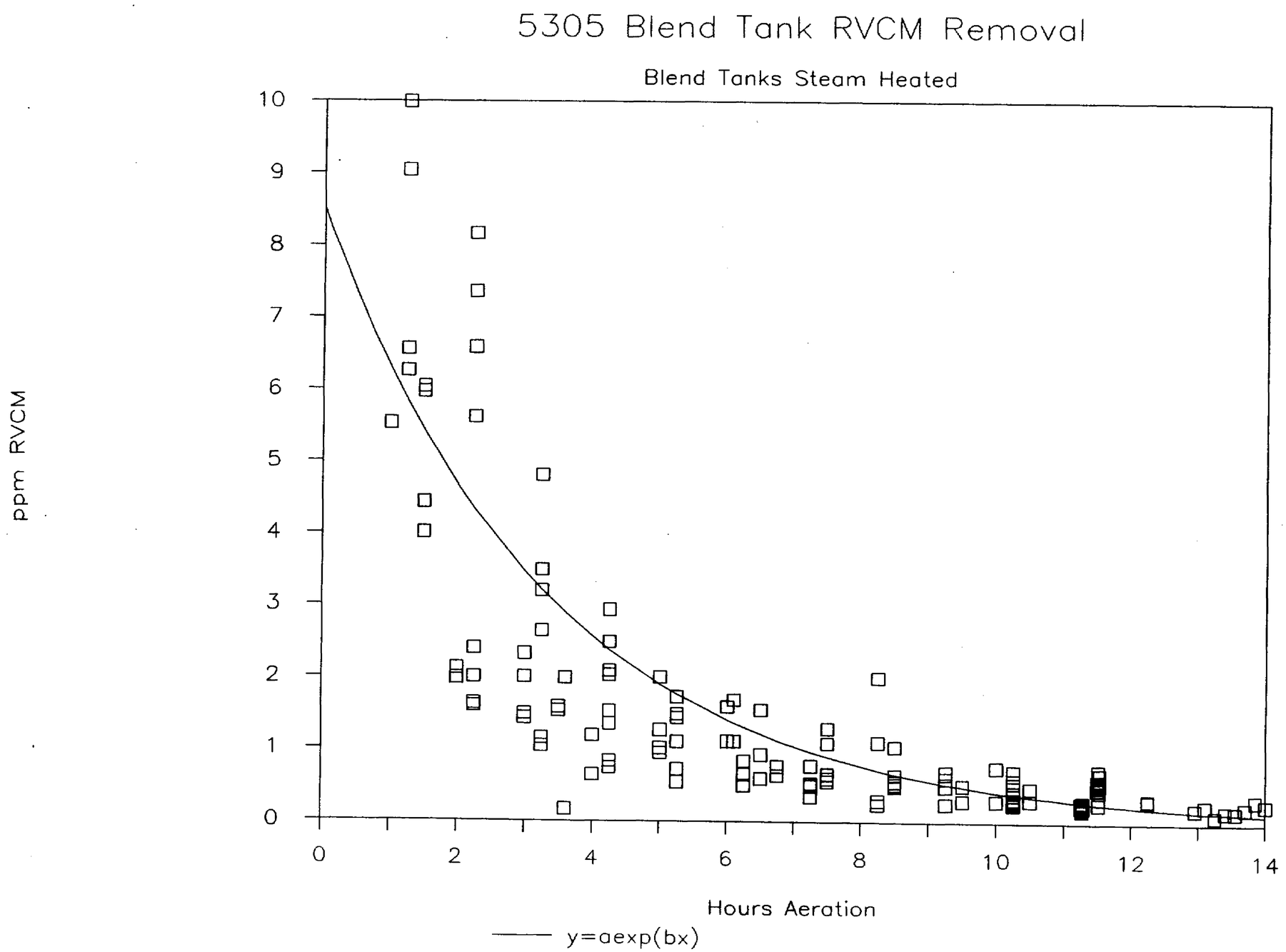
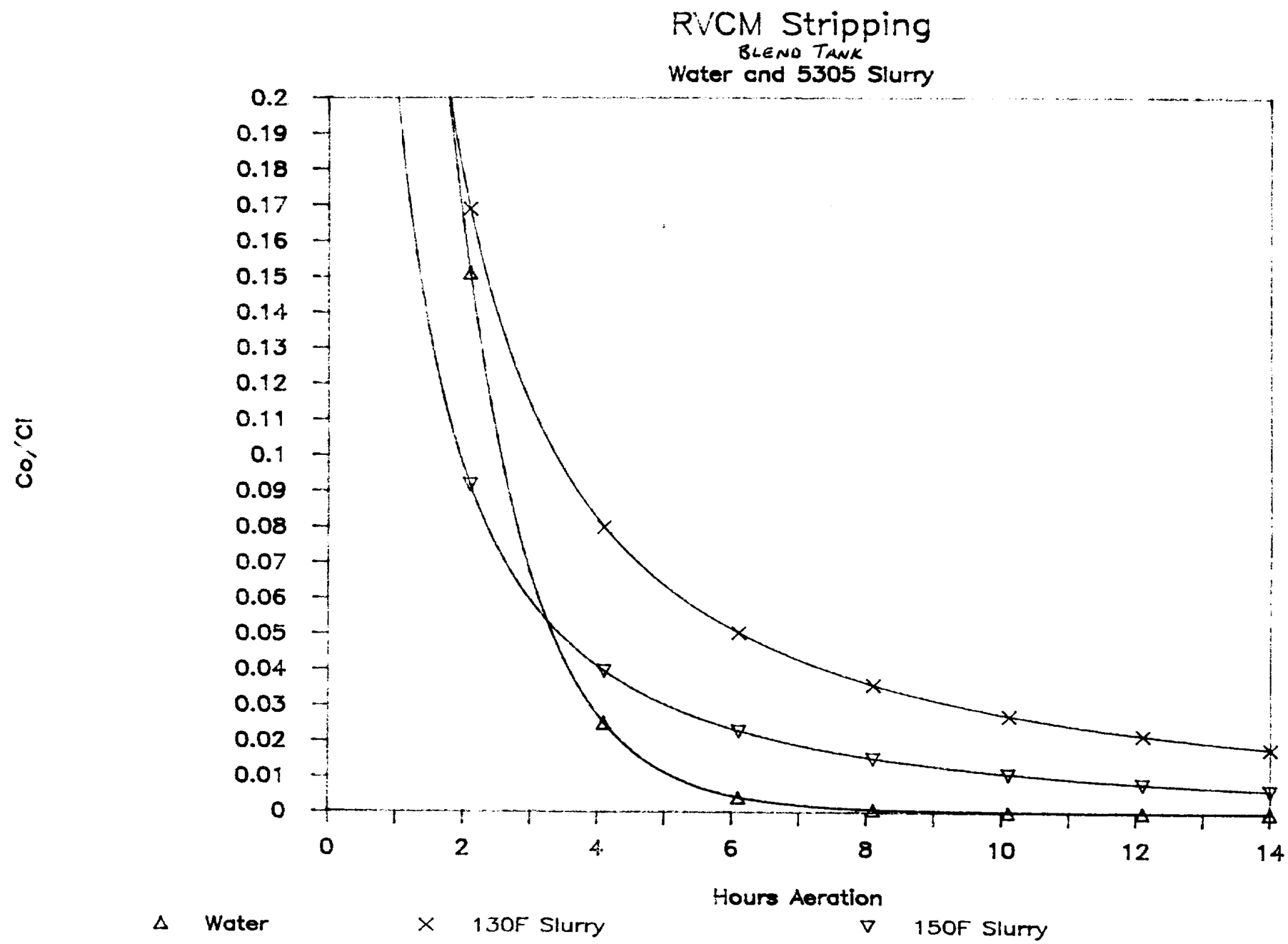

5305 Blend Tank RVCM Removal
Blend Tanks Steam Heated
ppm RVCM
10
9
8
7
6
5
4
3
2
1
0
0
2
4
6
8
10
12
14
Hours Aeration
y=aexp(bx)



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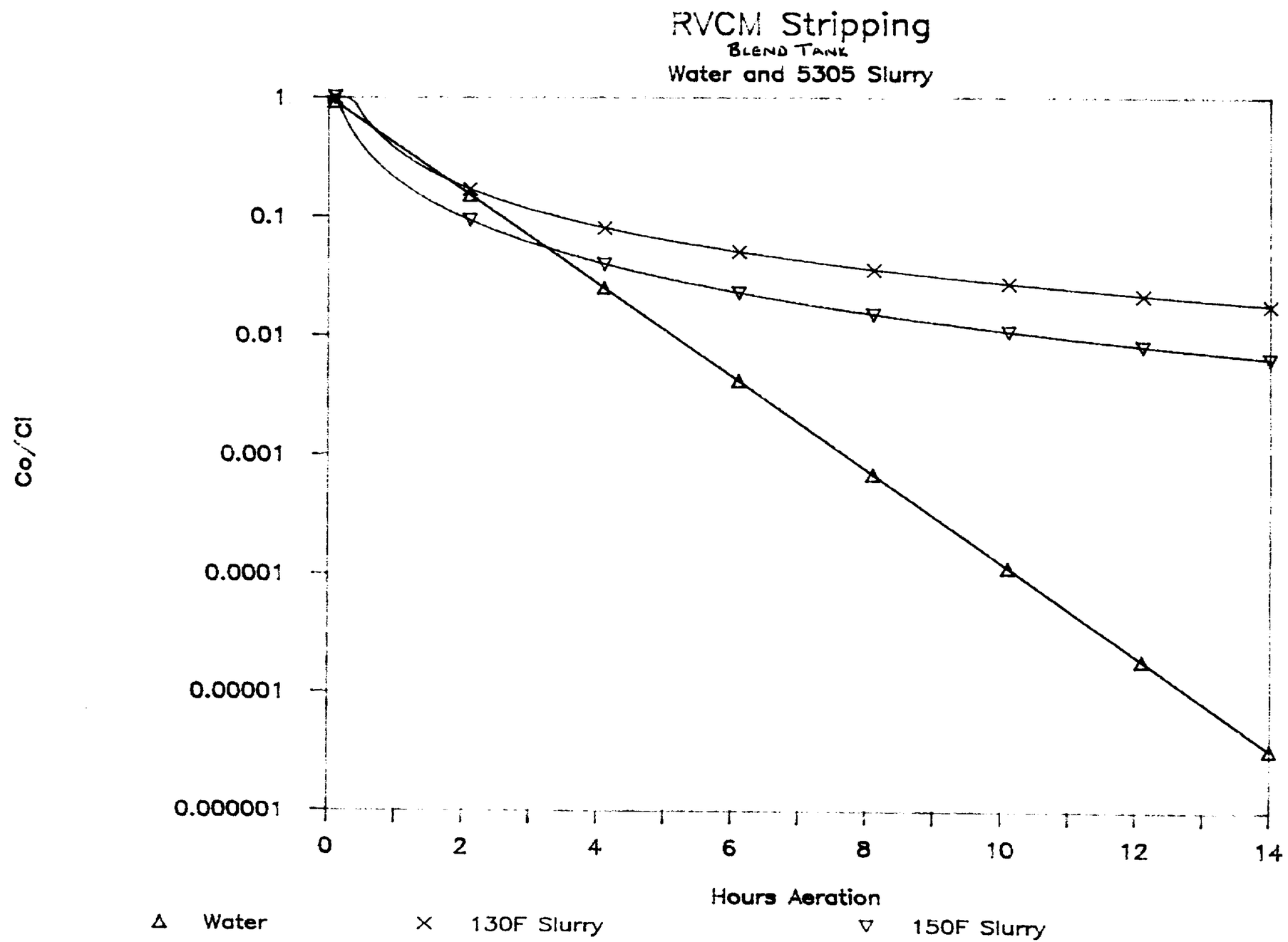

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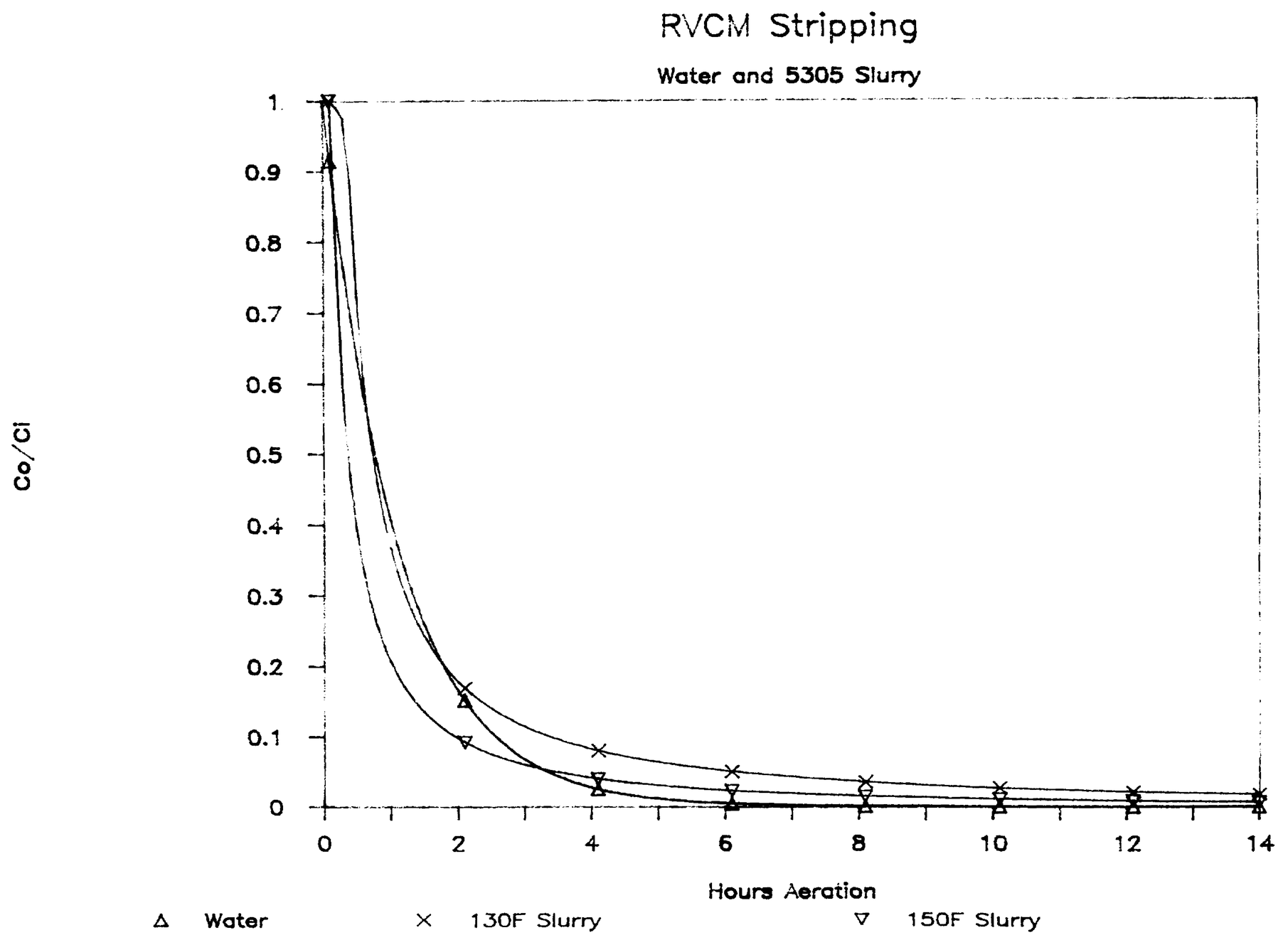

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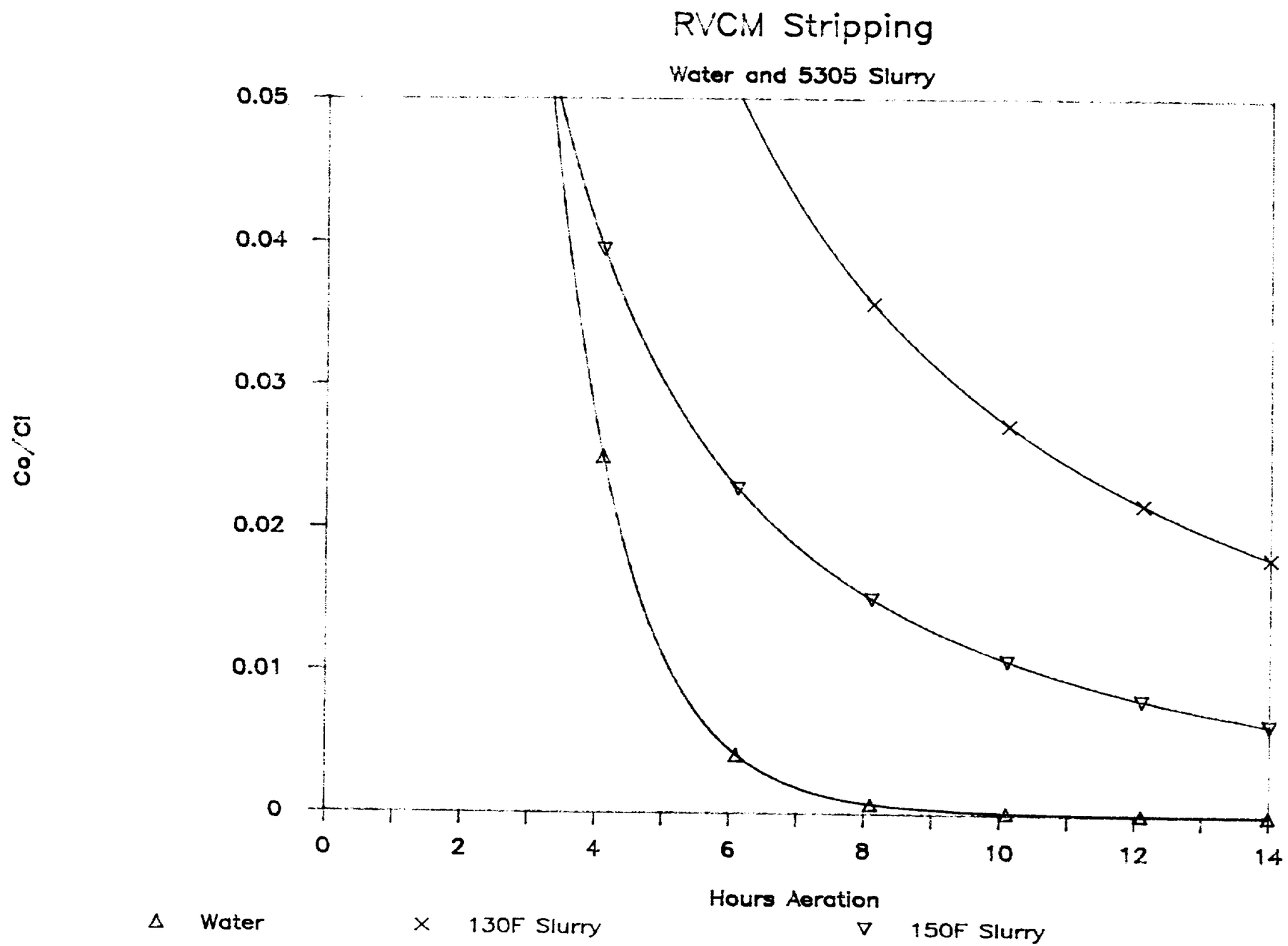

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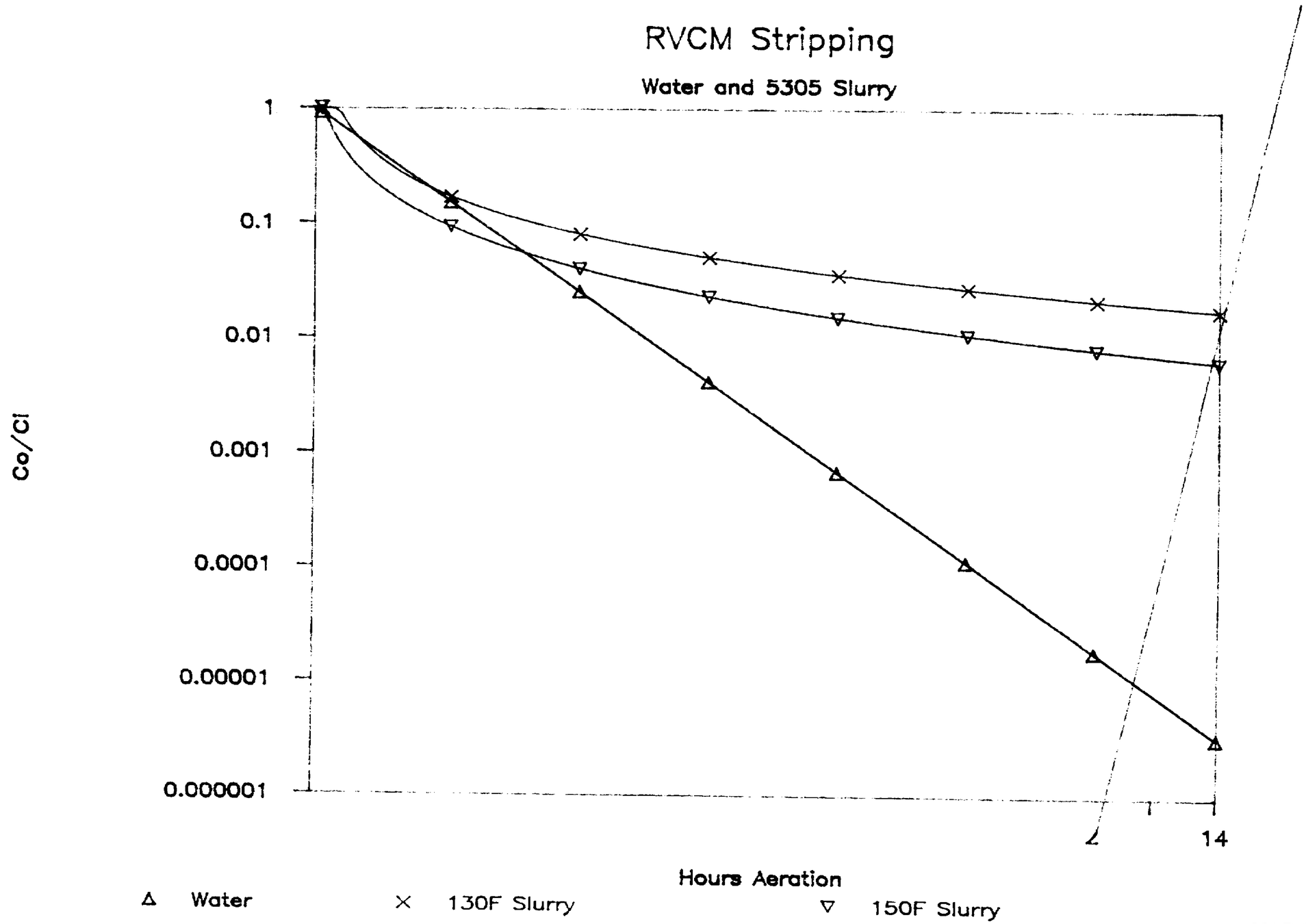

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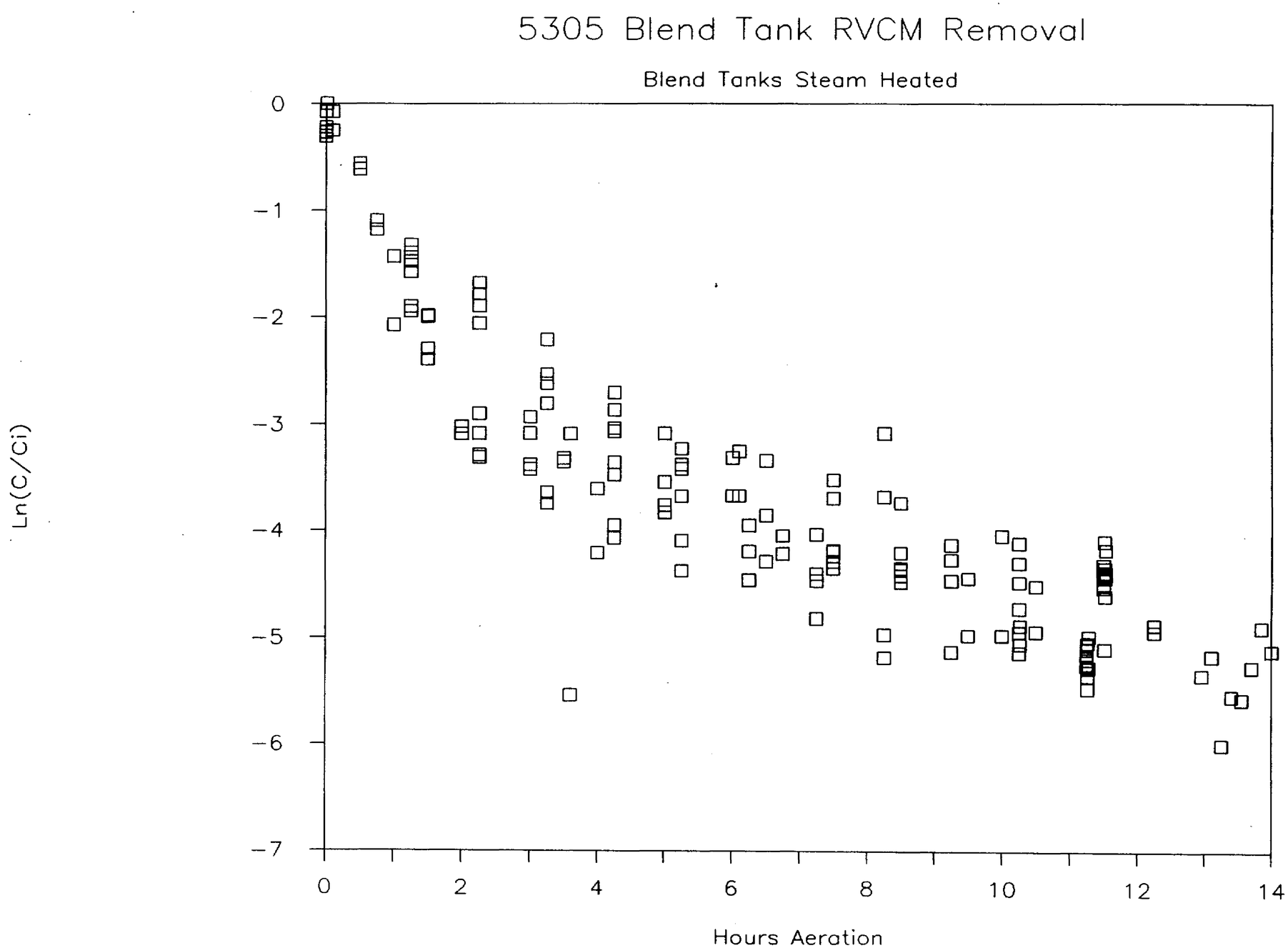

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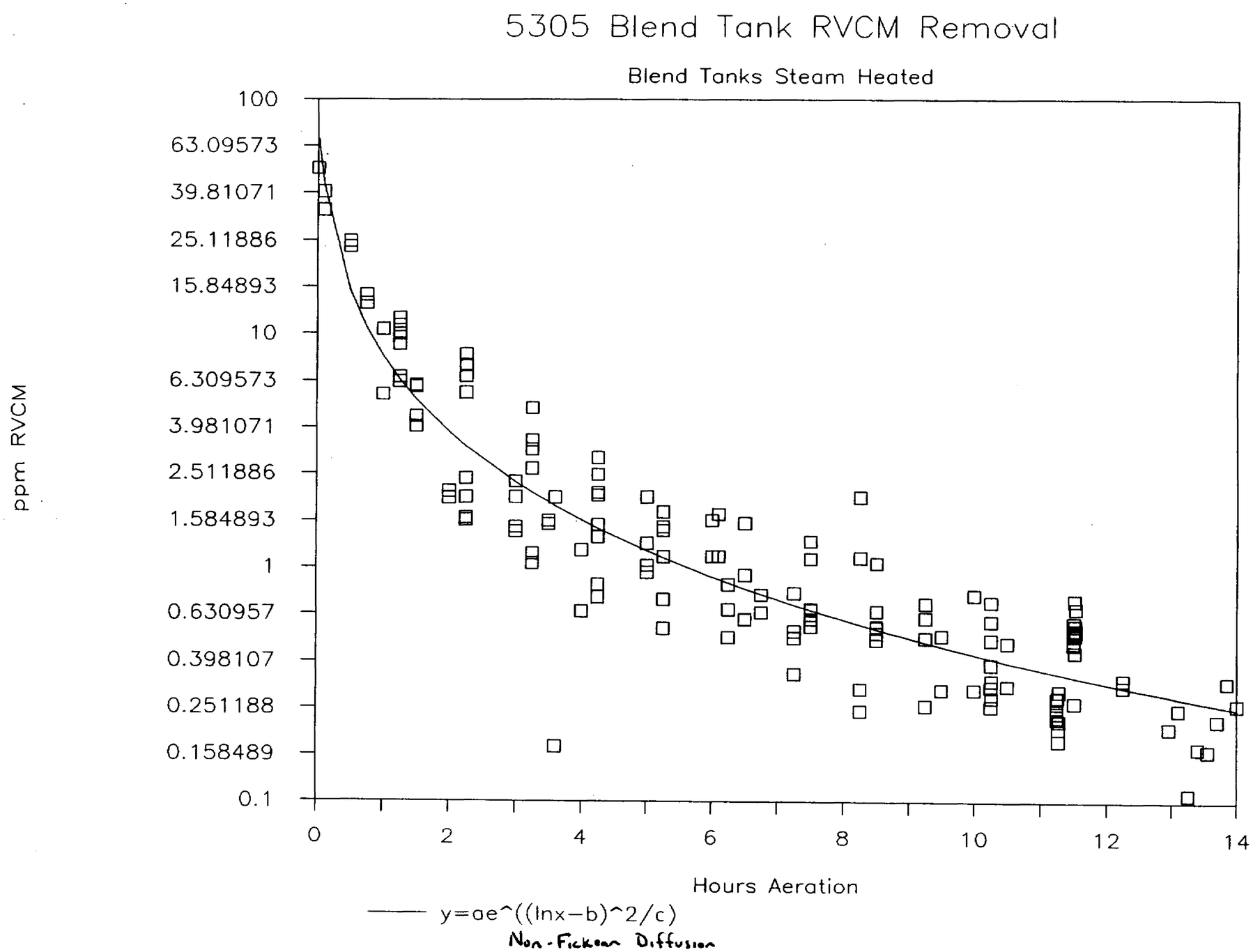
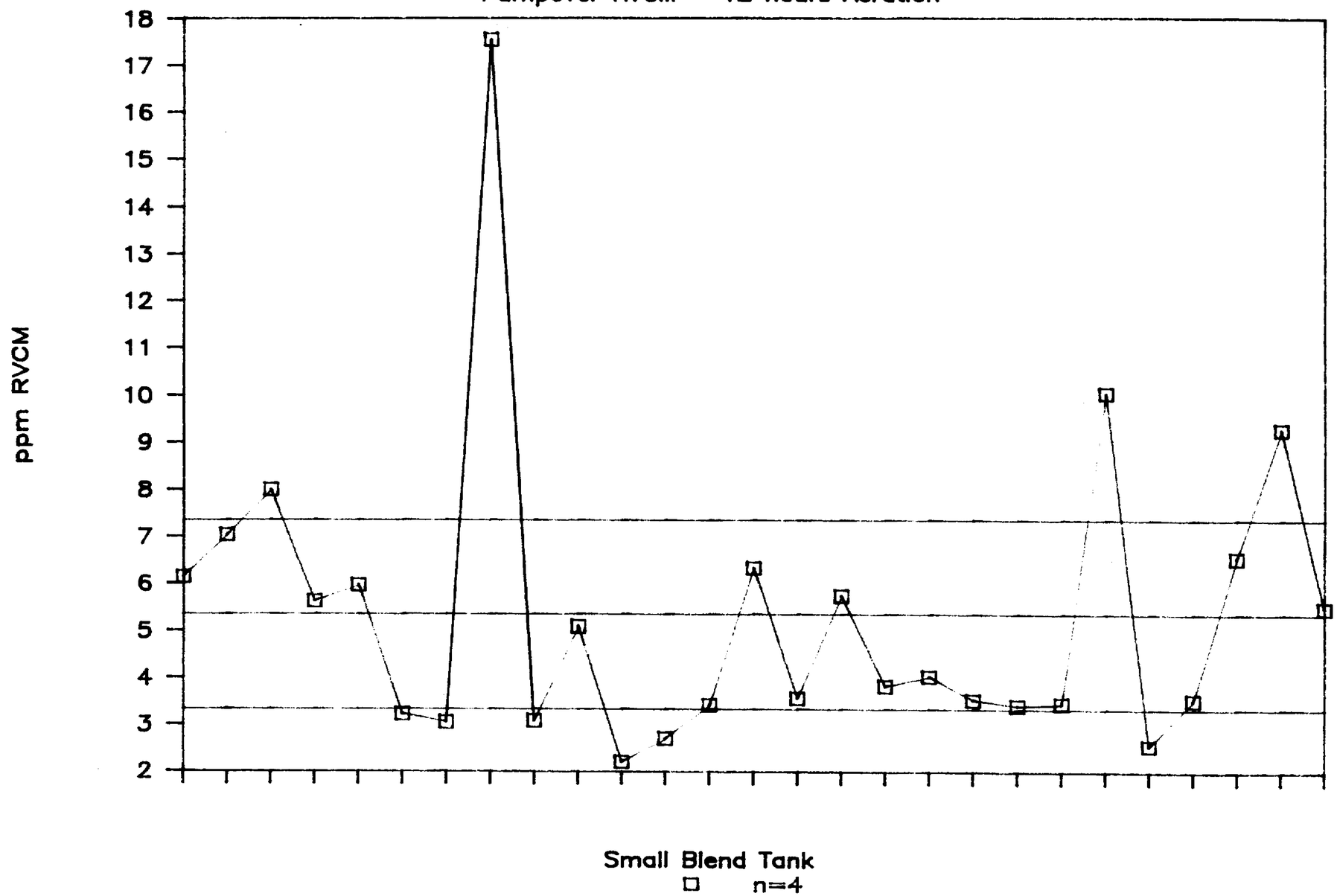


VAB.0001097586

5305 Small Blend Tanks

Pumpover RVCM – 12 hours Aeration



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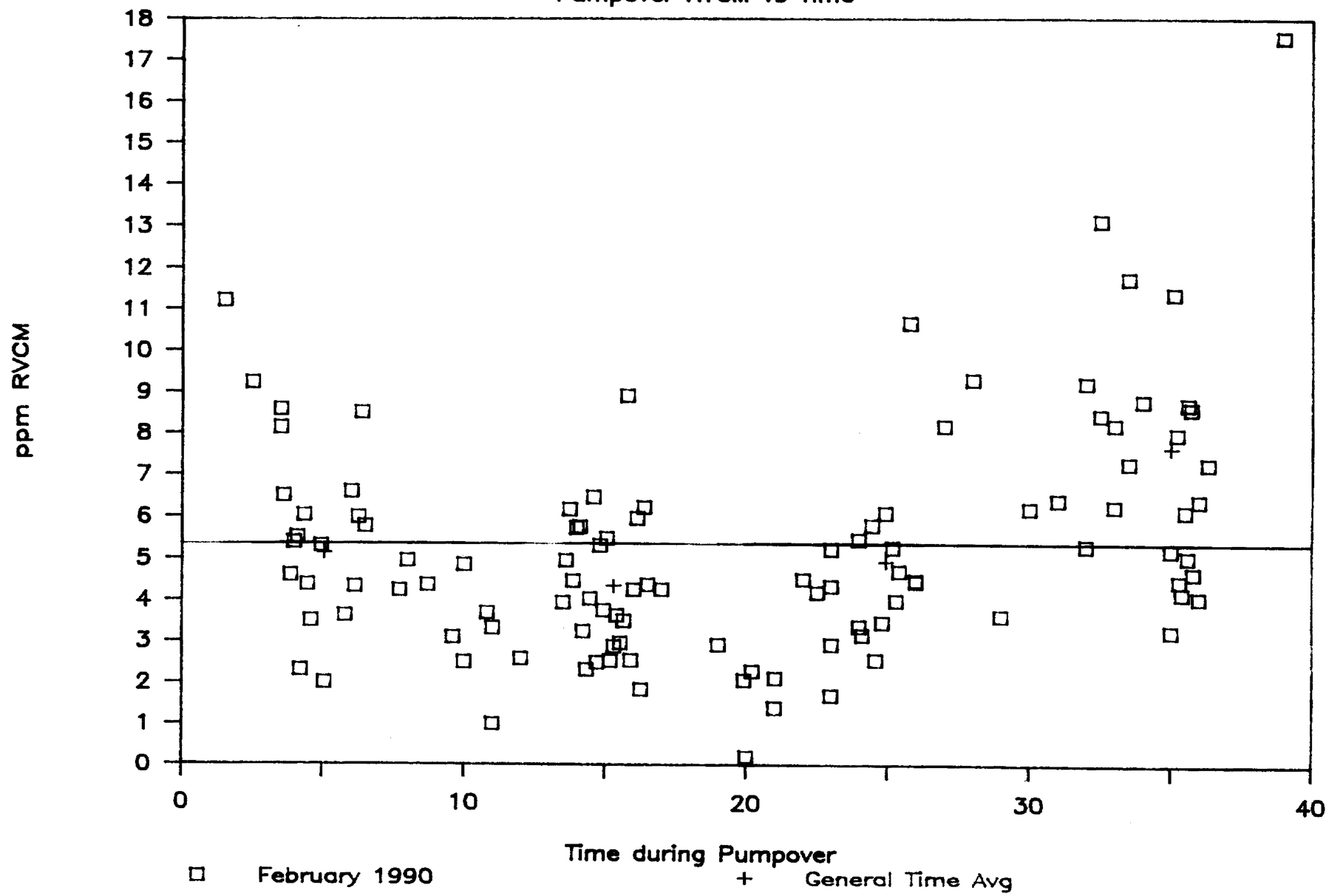
5305 Small Blend Tanks

Pumpover RVCM vs Time



VAB 0001097588

Agitation

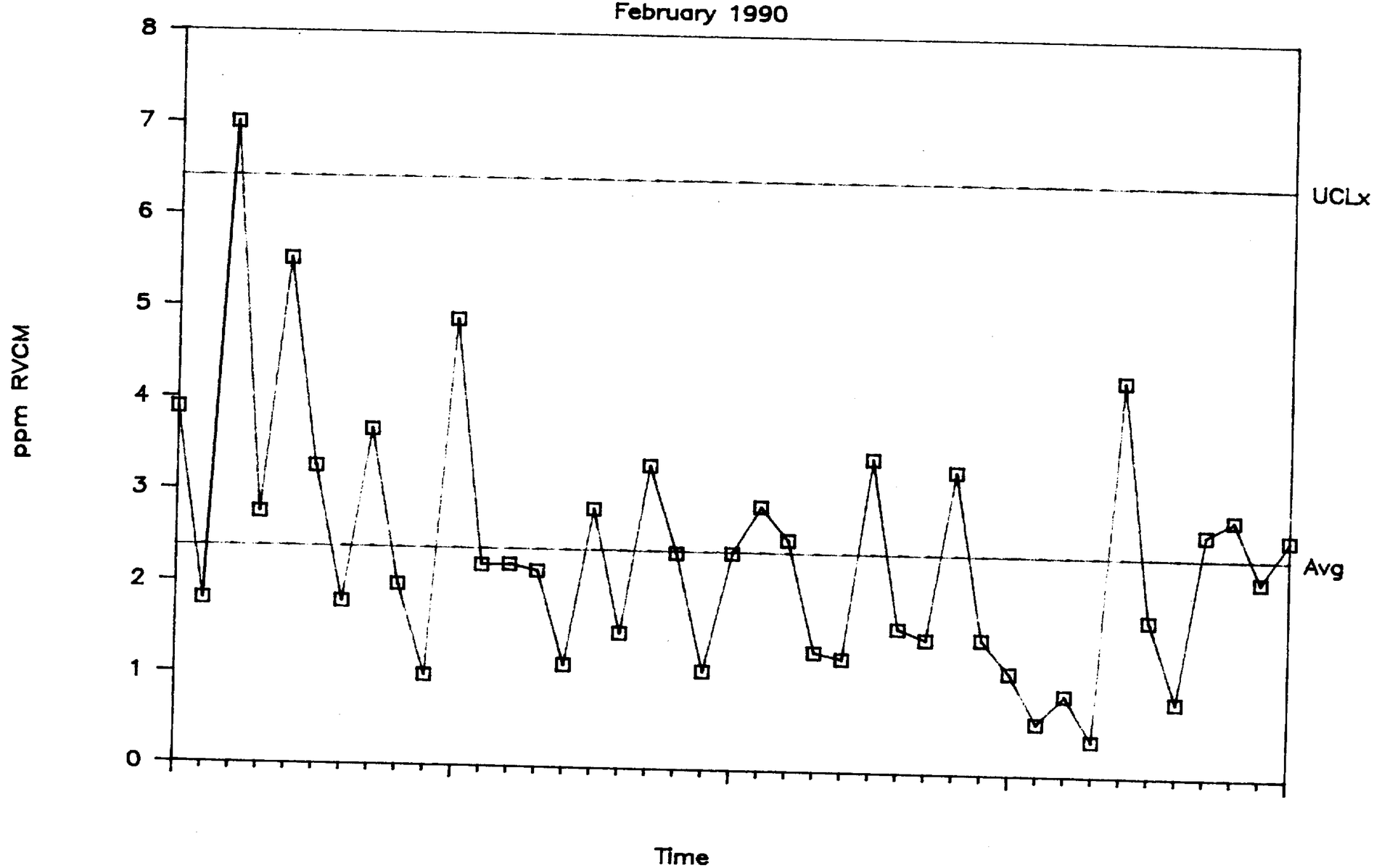
A

Appendix E

Blend Tank Aeration/Agitation

5305 Large Blend Tank RVCM

February 1990



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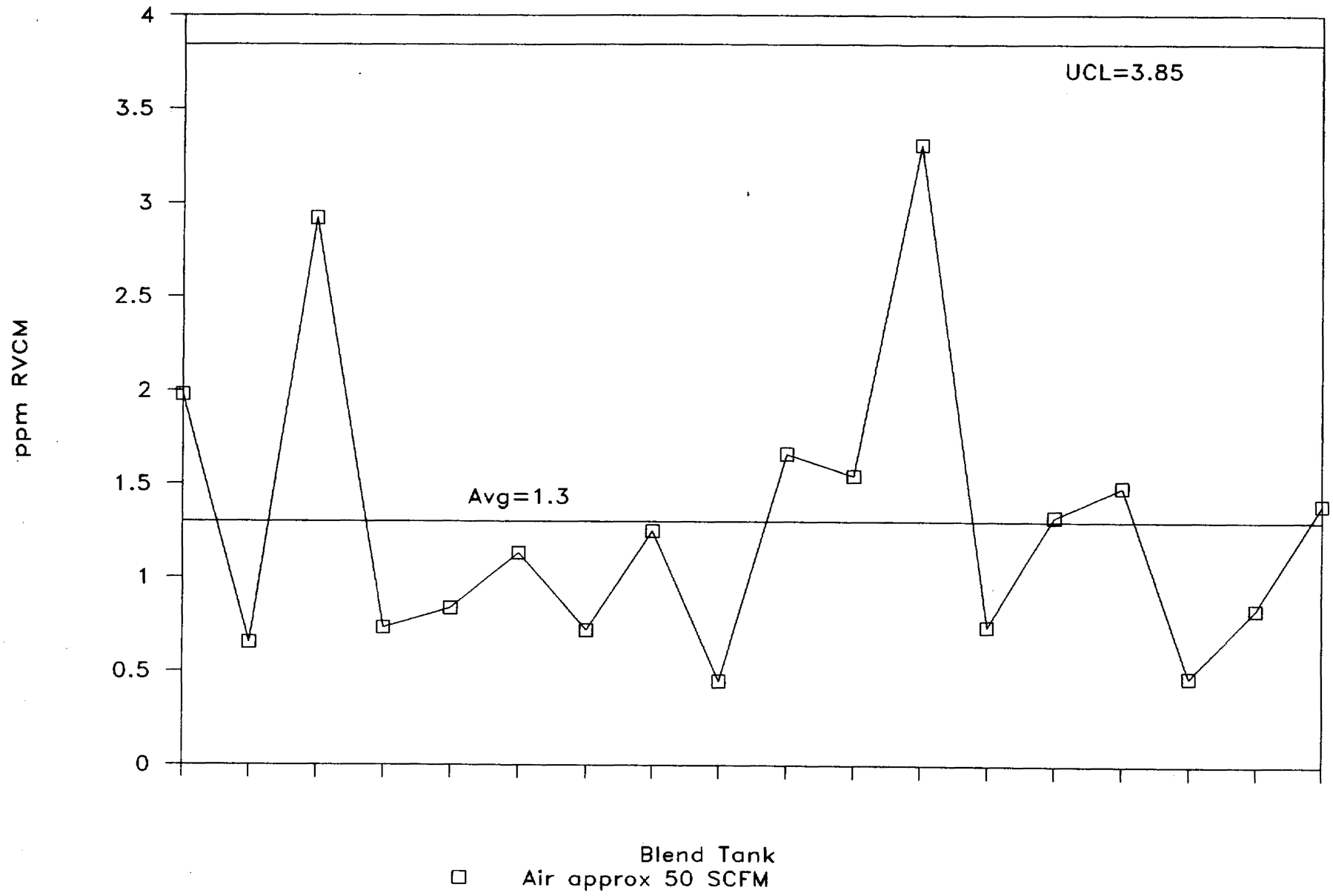
5305 Small Blend Tank RVCM

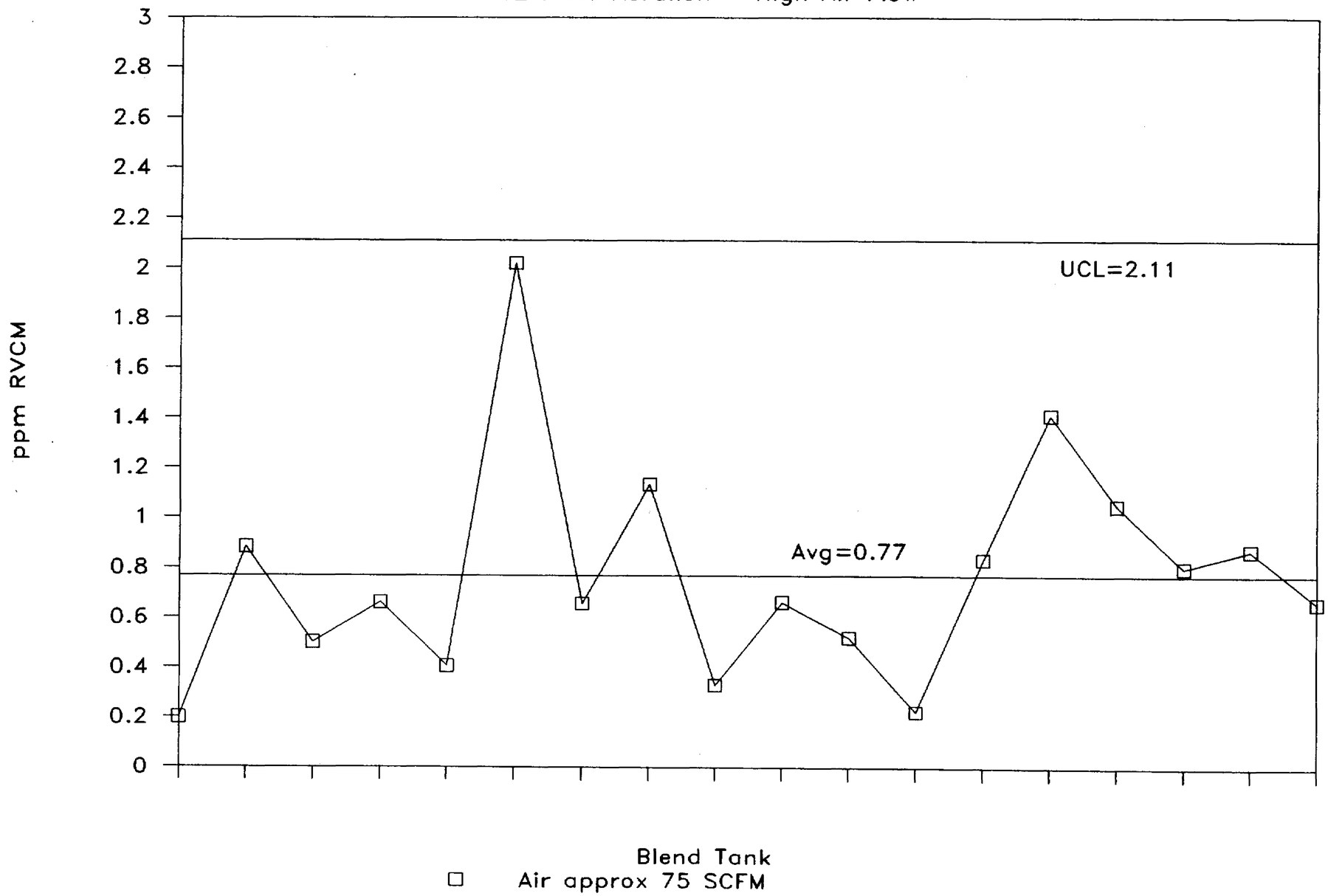
12 Hour Aeration – Low Air Flow



VAB.0001097592

5305 Small Blend Tank RVCM

12 Hour Aeration – High Air Flow



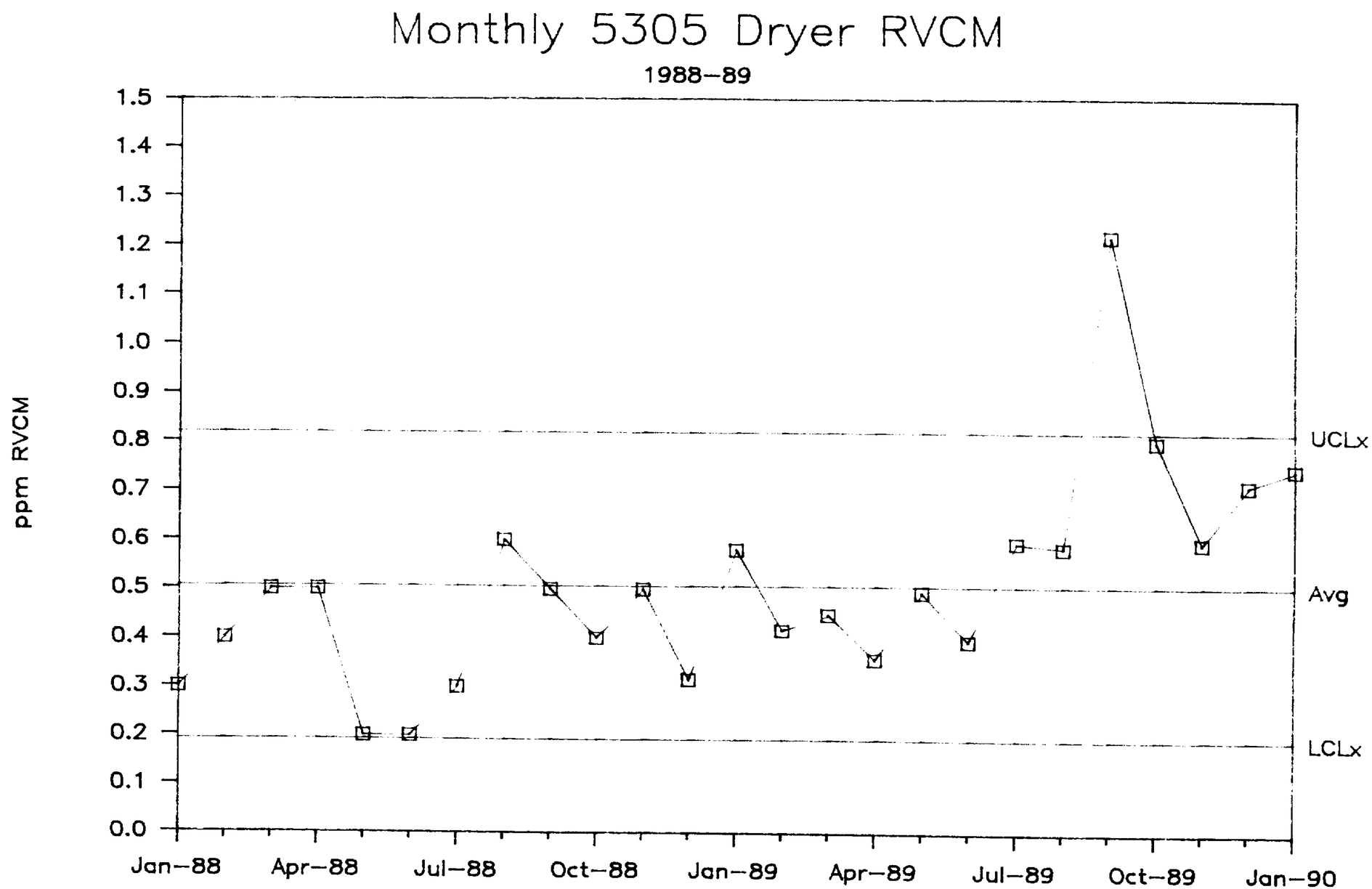
VAB.0001097593

Dryer Controls

A

Appendix F

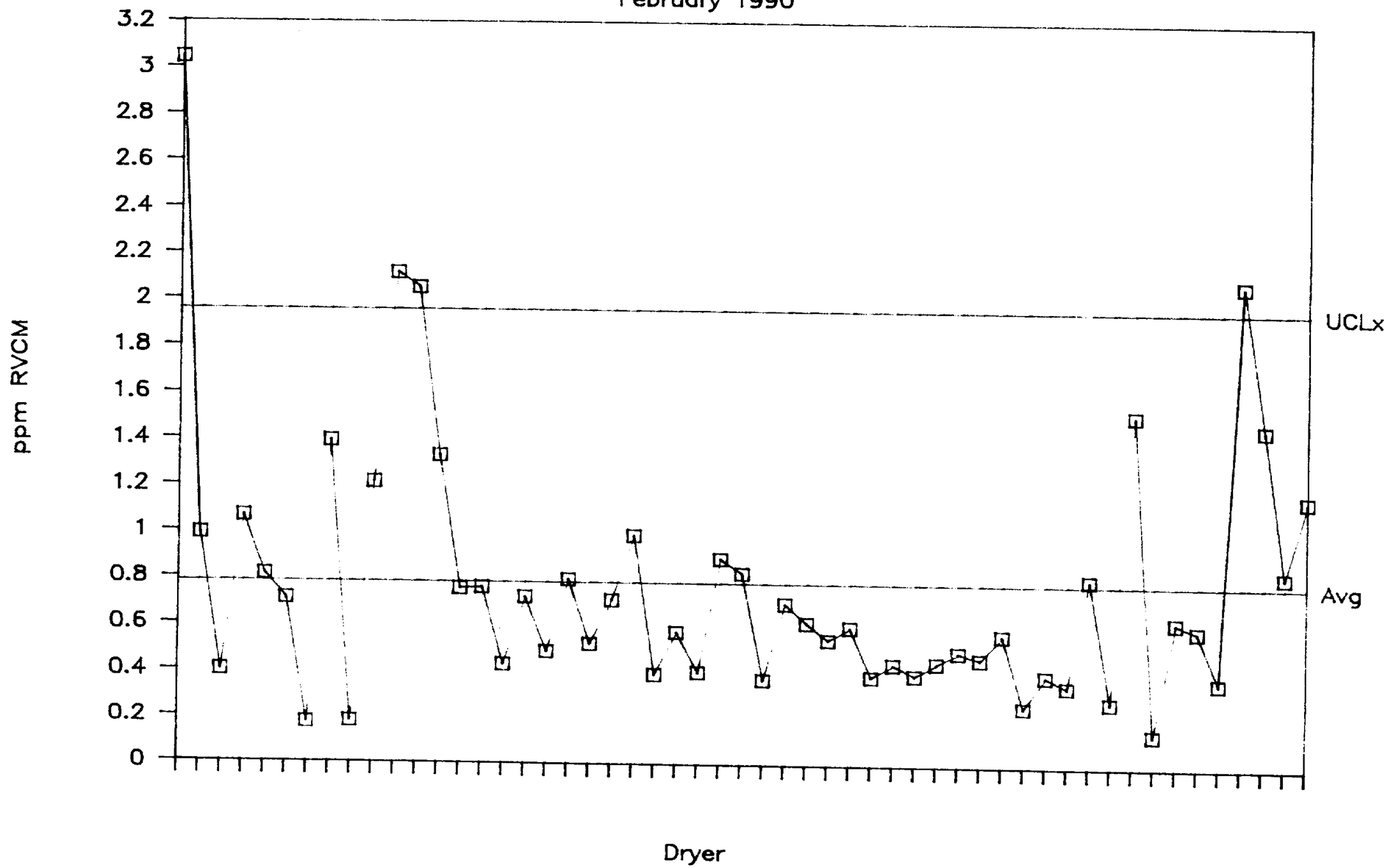
Dryer Controls



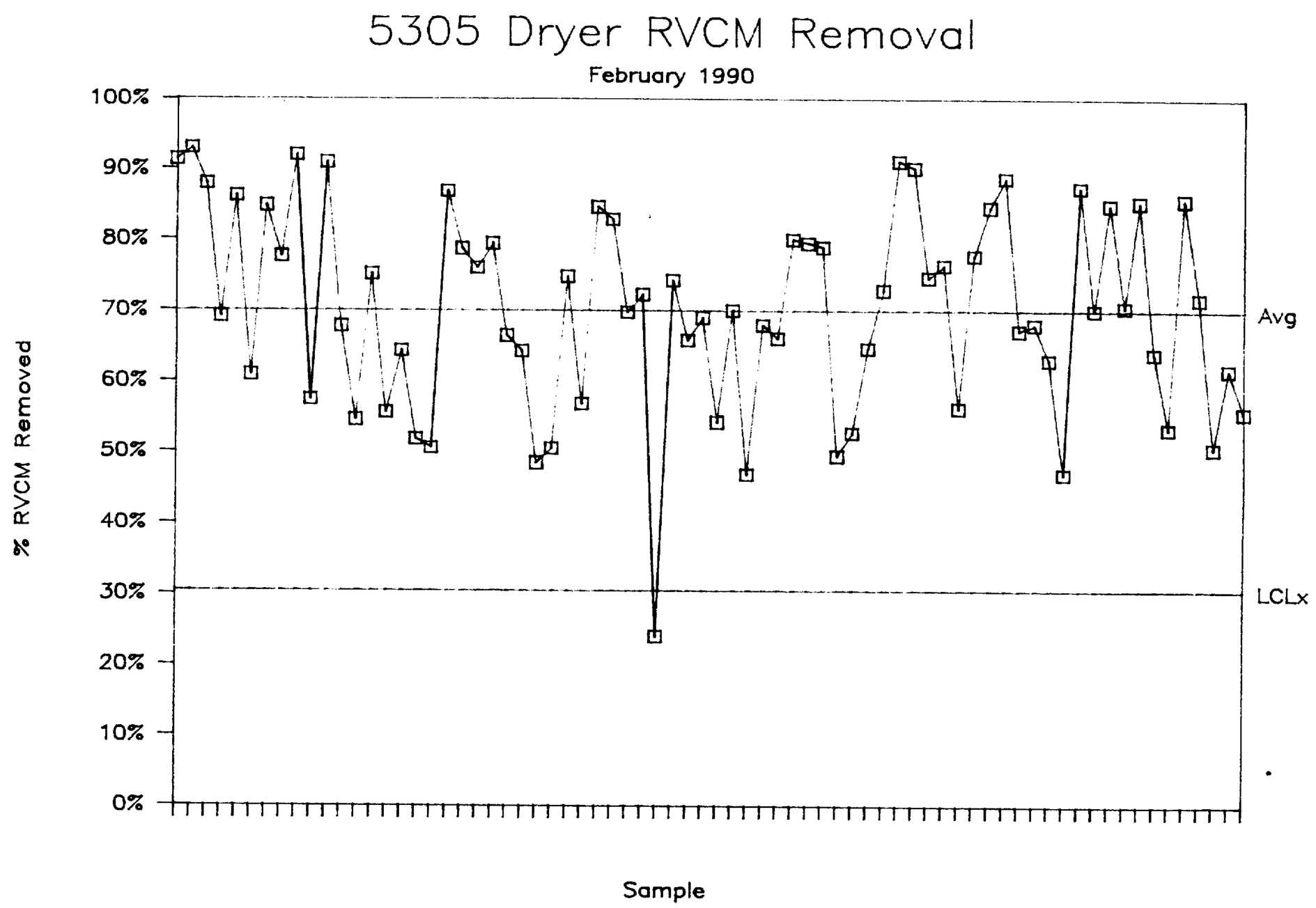
VAB.0001097596

5305 Dryer RVCM

February 1990

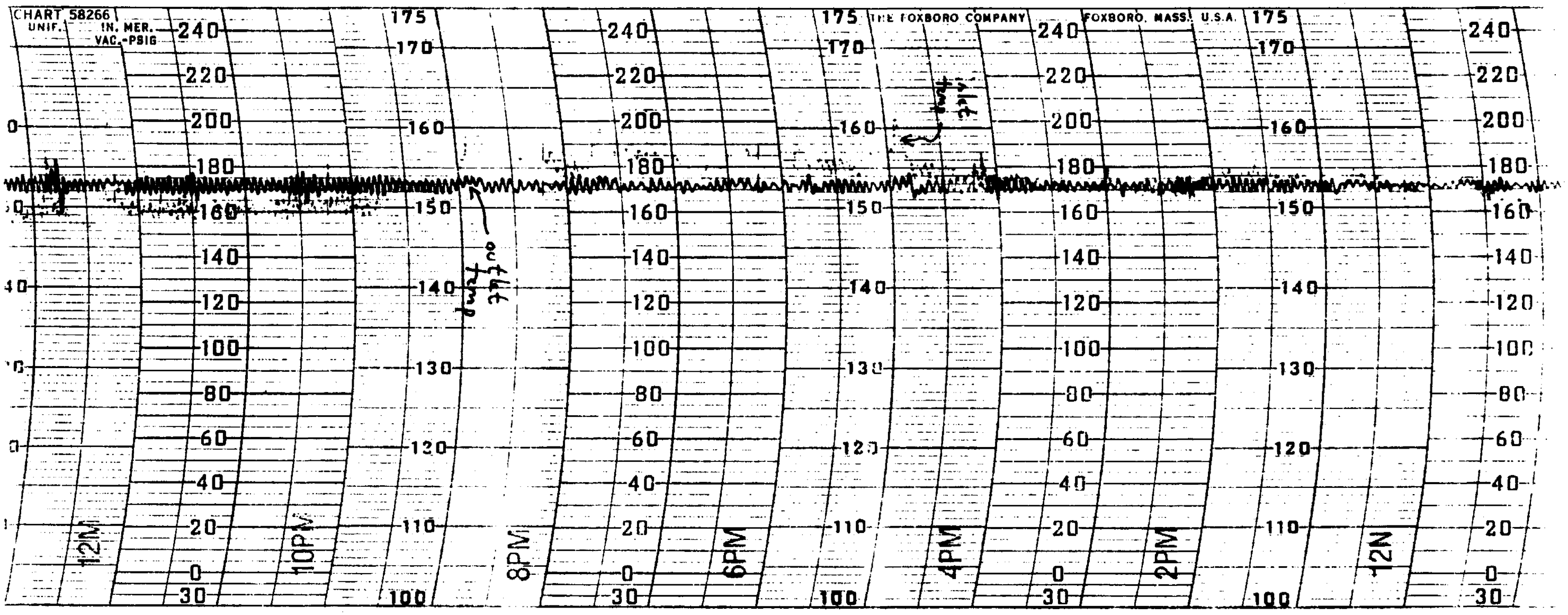


VAB.0001097597


5305 Dryer RVCM Removal
February 1990
100%
90%
80%
70%
60%
50%
40%
30%
20%
10%
0%
% RVCM Removed
Avg
LCLx
Sample

Dryer #4
2/26/90
5385 resin

Temperature Control Strip-chart



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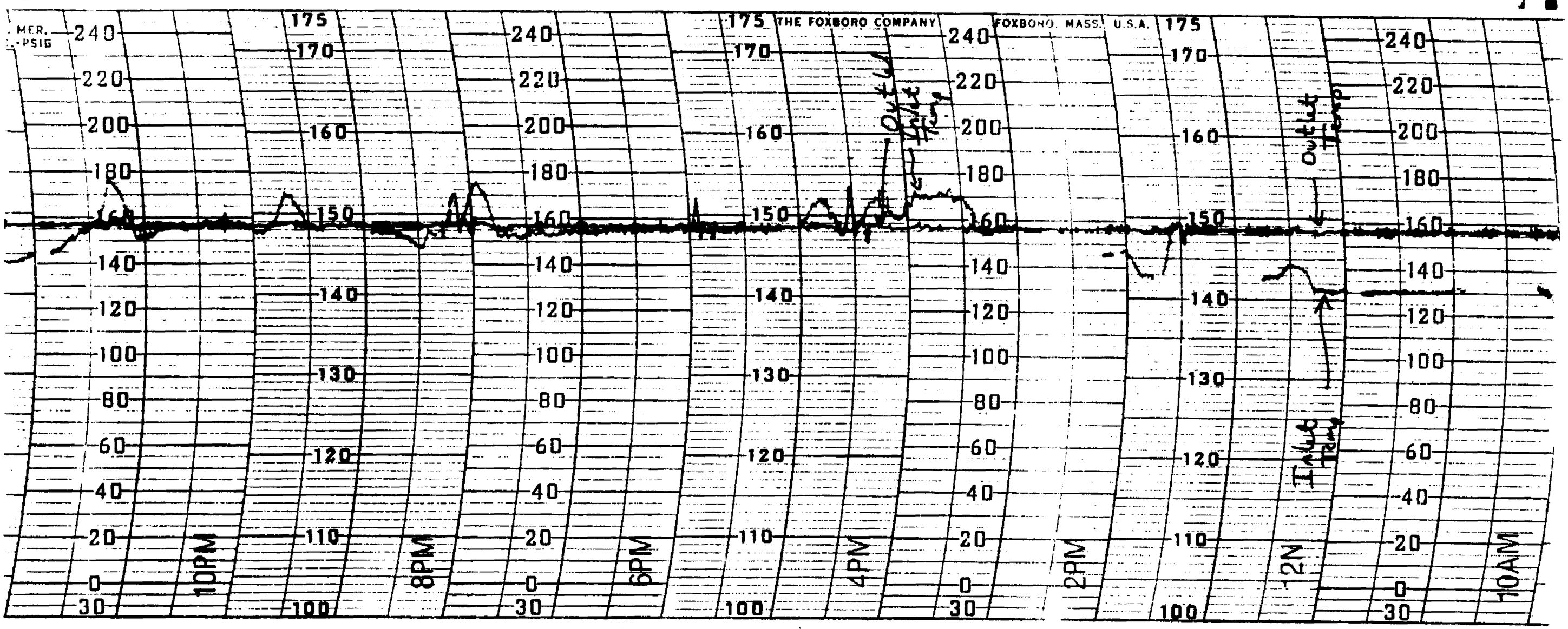
VAB.0001097600



Dryer #5
2/27/90
S385
resin

Temperature Control
Strip Chart

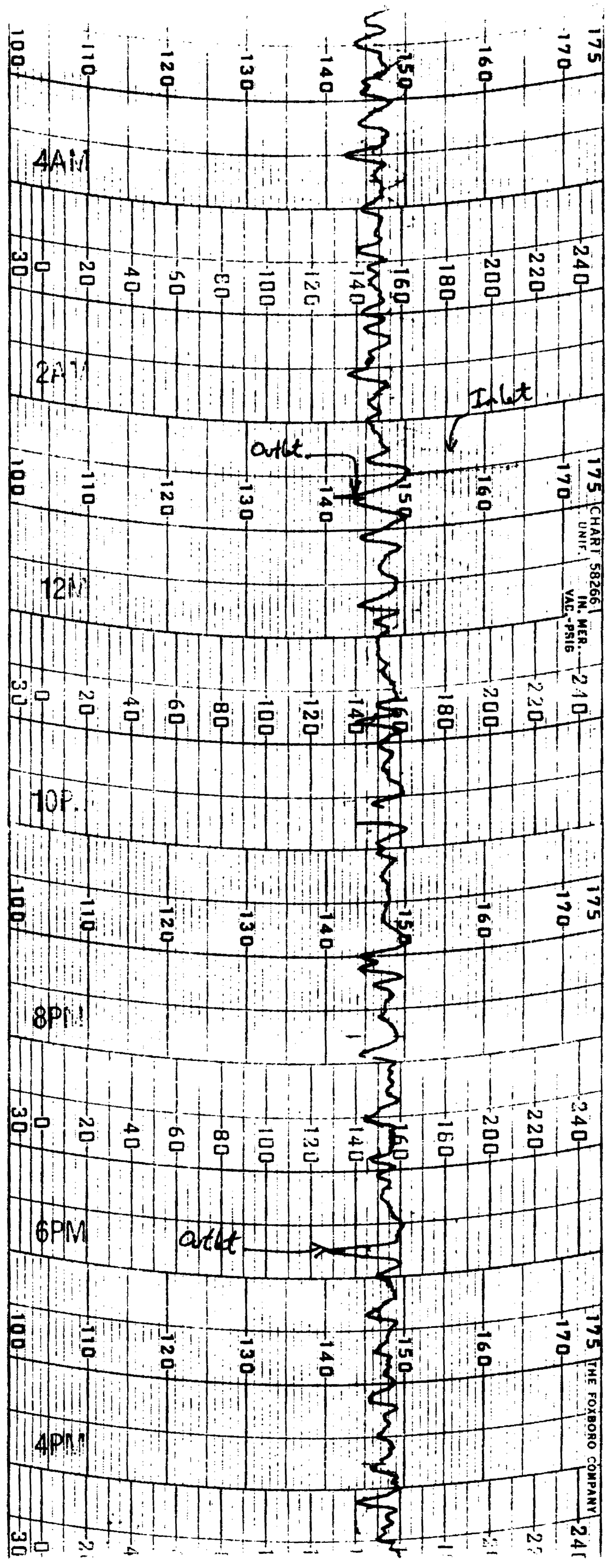
A

Dryer #6

2/26/90

5305 resin

Temperature Strip Chart



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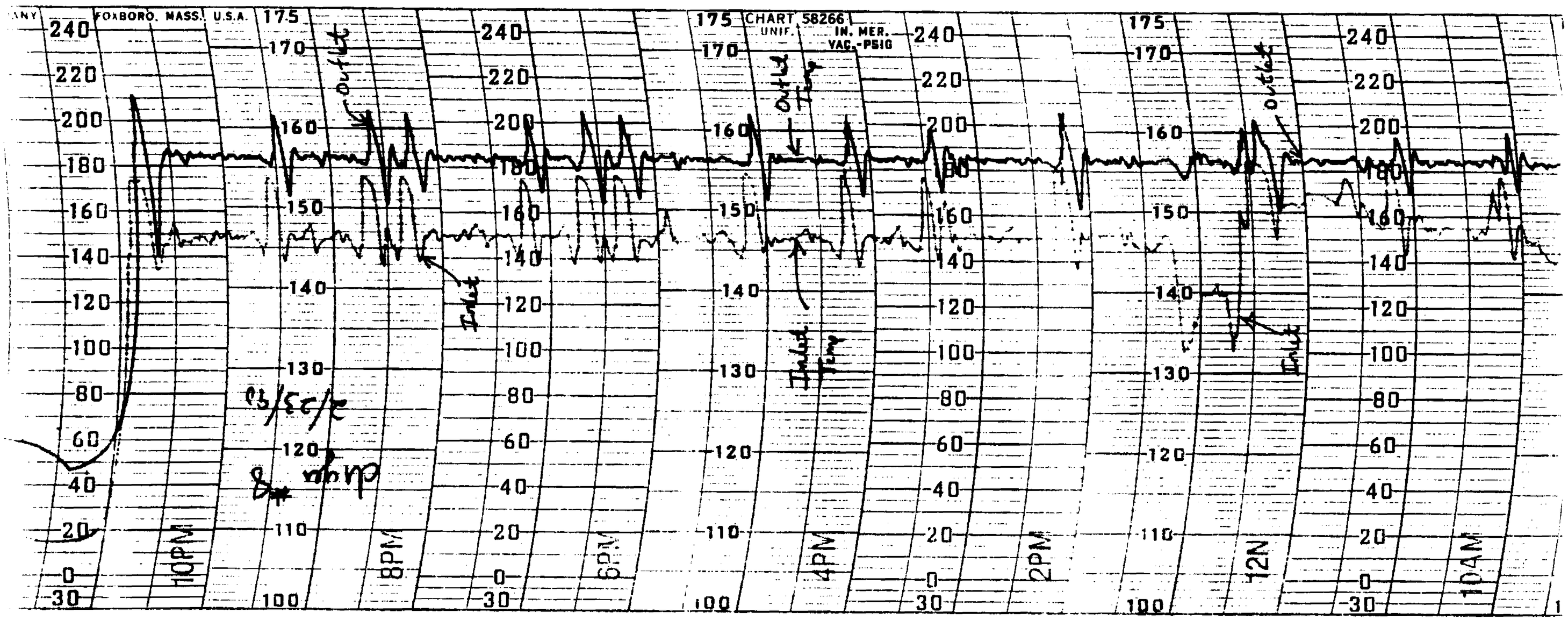


Dryer #8
2/23/90
S305 resin
Temperature
Strip Chart

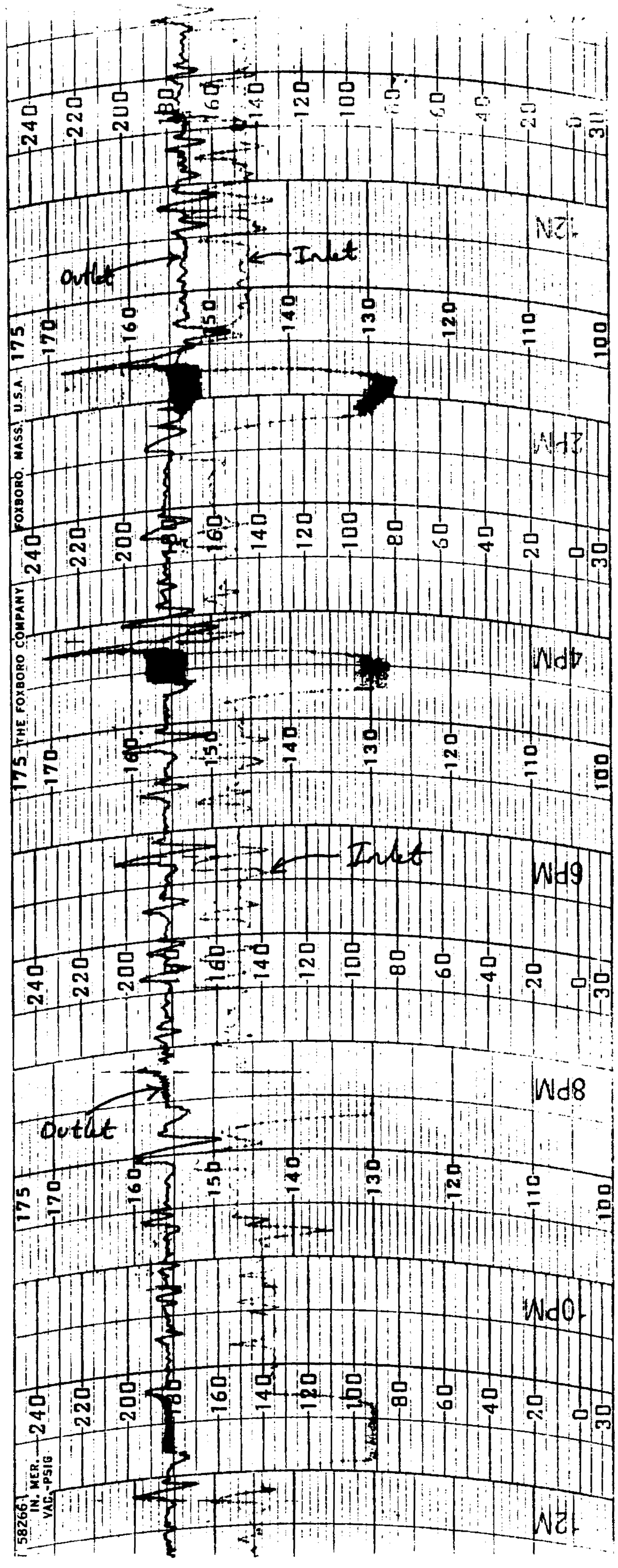
VAB.0001097602

Dryer #8

2/26/90

5305 resin

Temperature Strip-Chart



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Silo Aeration

Appendix G

Silo Aeration