

From **MONSANTO CHEMICAL COMPANY**

At Springfield, Massachusetts

Date May 24, 1960

cc R. A. Coffey
F. A. Duston
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To A. O. Ericksberg ✓

Reference

Standards Dept.

Report on Limit Procedures:

under P92-19-1, Effect of PDR-1 Conc.
and pH Adjustment
P88-42, Addition of Lauric Acid and
Maleic Anhydride

Subject limited procedures have been reported in "Interim Report:
PDR-1 Characterization by Plant Trial" A.P.T./F.A.D., Feb. 25, 1960.

In brief, the concentrations of lauric acid and maleic anhydride normally encountered in raw materials ($L_2 O_2$, PDR-1, MGD) do not affect homopolymer quality.

PDR-1 concentration changes in homopolymer formulations do influence various properties such as heat stability, bulk density, and particle structure. The optimum PDR-1 concentration is 0.15%. The adjustment of the pH of the suspending medium with sulfuric acid or sodium hydroxide offers no PVC quality advantages.

APJ

Archie P. Torrenzano

Jmy

Al -

I have 9 copies of "Interim Report" available. However, since this information is classified I will await word from you before sending it over - if you want them. Also, I believe you have a "1" copy of this report now.

Archie

RSV0024070

At Springfield

P. H. Avon
M. O. Debacher

Date June 2, 1960

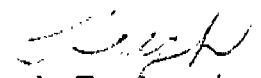
To A. O. Ericksberg*

Reference

Subject

Interim Report
Vinyl Resin Technical Project No. R-51

We are returning a copy of the subject report, sent to us by your Department for approval, for distribution of this report Overseas. Due to the nature of this report, even though it covers Limited Procedures P 92-19-1 and P 83-42, we feel that the contents of this report are of such a nature that it is not suitable for Overseas distribution.


Hugh F. Armstrong

/cvl
enc.*

RSV0024071

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Interim Report
PDR-1 Characterization by Plant Tial

Vinyl Resin Technical Project No. R-51

Work Done During: March, 1959 to December 1959

Report Prepared By: A. P. Torrenzano

Date Typed: February 25, 1959

1962

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I. Introduction

In May 1959 a PDR-1 characterization study was started with the objective of redefining PDR-1 quality requirements based on performance in plant PVC suspension polymerizations. This project was prompted by the erratic quality level of suspension homopolymer resins, the simultaneously poor to average performance of PDR-1 lots in the coke bottle acceptance test for copolymer usage, the critically low inventories of acceptable PDR-1, and the general belief that improved PDR-1 quality was needed to produce PVC for a more critical market.

The objective of redefining PDR-1 quality requirements imposed these specific problems:

1. To establish significant quality differences in PVC suspension homopolymer and copolymer resins caused by changes in PDR-1 lots.
2. To correlate a PDR-1 physical property with the PVC resin quality.
3. To establish PDR-1 specifications for both Springfield and foreign plants.

In addition to recommending a PDR-1 raw material specification, this report will review the plant studies on the effect of PDR-1 concentration changes and addition of acids and base to the PVC formulation.

II. Summary

- A. Fourteen PDR-1 lots were tested in Standard Opalon 300FM and Opalon 510 polymerizations to establish significant PVC quality shifts with PDR-1 lot changes. To maximize the probability of PVC quality shifts, the PDR-1 lots were selected to cover a wide range in properties: Seven lots were unacceptable by the coke bottle-copolymer acceptance test; the specific viscosity ranged from 3.9 to 9.2; the PDR-1 conversion ranged from 67 to 85 percent.

The polymerization results showed that Opalon 510 copolymer properties were completely insensitive to changes in the PDR-1 lots, while differences in the PDR-1 lots were responsible for very large shifts in Opalon 300FM particle size - equal to four times the specification range - and shifts in Opalon 300FM heat stability.

The Opalon 300FM particle size results, as measured by the screen analysis factor, correlated with (1) the specific viscosity of purified PDR-1, (2) the specific viscosity of unpurified PDR-1, and (3) the specific conductivity of a PDR-1 (unpurified) water solution. The results of experiments performed during this project and by Research showed that purified PDR-1 specific viscosity was the prime variable, while unpurified PDR-1 specific viscosity and specific conductivity were secondary variables. This fact was established when post polymerization treatment (purifying and increasing the impurity of PDR-1) did not alter its suspending power.

The Opalon 300FM heat stability level correlated with the specific conductivity of the PDR-1 (unpurified) water solution. A 10% improvement (50 to 55% transmission) in quality was measured for a 500 micromhos decrease in specific conductivity.

One anomalous PDR-1 lot was encountered during the project. Three different samples of PDR-1 lot 222 would not dissolve completely in acetone, during the specific viscosity test, indicating the presence of polyvinyl acetate (insoluble) and VAMA (soluble) polymer with excess vinyl acetate. A VAMA polymer with excess vinyl acetate - instead of an equimolar ratio of vinyl acetate to maleic anhydride - would exhibit a specific viscosity higher than "equimolar" VAMA of the same molecular weight, but not contribute to increasing the suspending power of the polymer. The problem of distinguishing between "equimolar" and "acetate rich" VAMA can be handled by rejecting any PDR-1 lot which contains insoluble polymer, or a high concentration of unreacted maleic anhydride - via a specific conductivity test.

- B. Four PDR-1 lots were studied at various concentrations in the PVC formulation to determine the possibility of improving homopolymer quality and/or compensating for lot to lot variations in PDR-1 quality by optimizing the PDR-1 concentration. The results showed that PDR-1 concentration had little effect on the homopolymer screen analysis and therefore could not be used to compensate for PDR-1 quality differences which had a major effect on resin particle size.

PDR-1 concentration changes had a significant effect on the shape and structure of the PVC particle agglomerate. Although the overall size of the agglomerate did not change, as measured by the screen analysis, the low PDR-1 concentration produced a more consistent, spherical agglomerate similar to successful competitive resins. This spherical agglomerate resulted in a resin of higher bulk density and consequently poorer Brabender fusion time. With high PDR-1 concentrations, the individual agglomerates consisted of 10 to 15 small particles fused together into myriad shapes thus resulting in a low bulk density resin and a broader screen analysis distribution.

At low PDR-1 concentrations the resin extrusion grade was affected adversely while the resin heat stability was improved markedly. As a result, the optimum PDR-1 concentration with the present method of operation was 0.12-0.15%, very close to Standard. Improvements in Brabender fusion time and extrusion grade could be obtained at the 0.18-0.20% PDR-1 concentration provided the "excess" PDR-1 could be removed by more efficient washing in the centrifuge to maintain an acceptable heat stability level.

- C. Lauric acid and Maleic Anhydride were added to the Opalon 300FM formulation to determine tolerable concentrations for these raw material impurities. The results of twelve Opalon 300FM batches showed no significant resin quality differences when Lauric Acid or Maleic Anhydride were present at concentrations as high as 10-12%, based on the weight of Lauroyl peroxide or PDR-1.

- D. The possibility of employing a combination of a PDR-1 concentration change and pH adjustment to control resin particle size and distribution was the subject of a designed 25 batch plant experiment in April, 1958. Sulfuric acid and sodium hydroxide were used to adjust the pH of the suspending medium to levels ranging from 1.7 to 4.6, while PDR-1 was employed at concentrations from 0.06 to 0.26 percent. This experiment was halted after 16 batches when the results of the acidified batches showed a drastic effect on resin heat stability.

In general, the results indicated no practical advantages to pH adjustment since the acidified batches exhibited poor resin heat stability and extrusion properties, while batches made with sodium hydroxide had a broad particle size distribution and, as expected, a lower volume resistivity level. The particle size level for the acidified batches was coarse, by comparison to standard batches, with a distribution that was approximately normal for coarse resin. The particle size level for batches with caustic was approximately unchanged, but as mentioned above, the particle size distribution was broadened.

III. Conclusions

- A. Opalon 510 copolymer resins are unaffected by PDR-1 lot changes ranging in specific viscosity from 4.0 to 9.2.
- B. An 8.0 minimum PDR-1 specific viscosity is required to minimize the effect of PDR-1 lot changes on homopolymer particle size.
- C. Resin heat stability level is dependent on PDR-1 specific conductivity (a measure of unreacted monomers) with the present method of washing PVC slurry in the centrifuge.
- D. PDR-1 concentration changes in the PVC formulation cannot be used to compensate for the effect of low specific viscosity PDR-1 on resin particle size.
- E. With the present method of operation, the optimum PDR-1 concentration is the standard 0.15%. However, further work should be directed towards improving centrifuge washing and better defining the various PVC particle parameter requirements.
- F. In concentrations normally encountered in raw materials, lauric acid and maleic anhydride, per se, do not affect homopolymer quality.
- G. The adjustment of the pH of the suspending medium with sulfuric acid or sodium hydroxide offers no PVC quality advantages.

IV. Recommendations

- A. From the results to-date, the following PDR-1 raw material specifications would be required to insure PVC quality control both for Springfield and foreign plants.

1. For Homopolymer Usage

- a. Specific viscosity of purified PDR-1...8.0 min. to *13.0 max.
in water
- b. Specific conductivity of a 0.4% PDR-1 water
solution, micromhos1600 Max.
- c. PDR-1 free of polymer insoluble in acetone

2. For Copolymer Usage

- a. Specific viscosity of purified PDR-1...6.0 min. to *13.0 max.
in water
- b. PDR-1 free of polymer insoluble in acetone
- c. Master blends for use in copolymer must consist of individual PDR-1
lots with a 6.0 minimum specific viscosity.

B. The excellent heat level for Opalon 300FM made with low PDR-1 concentrations - 0.06% - suggests this possibility for Opalon 306-69 resin where heat stability is of paramount importance.

C. The present centrifuge washing studies indicate a marked improvement in resin heat stability. This technique may cancel completely the effect of PDR-1 specific conductivity and PDR-1 concentration on resin heat stability. However, if the washing is not completely successful then PDR-1 purification should be evaluated and/or the PDR-1 specific conductivity specification lowered.

D. If the centrifuge washing permits, it is recommended that a 0.20% PDR-1 concentration be evaluated for captive resins.

E. A better definition of desirable, basic PVC particle parameters is required before a superior dry blending resin can be specified or manufactured. A few accurate, meaningful gauges of resin performance unbiased or unconfounded by the test equipment or other PVC variables are necessary. By contrast,--

1. A variety of different shaped particles will produce the same screen analysis
2. Resin bulk density changes can be achieved in any one of four ways, -
particle size level, particle size distribution, particle porosity
and particle shape
3. A good Brabender fusion time can be achieved with low bulk density
resin, regardless of most other factors.
4. Dry time results are as much dependent on resin particle size, particle
shape, and equipment variables as on resin porosity.

* Tentative until work in progress completed.

5. Resin Fisheye count, regardless of the test equipment, will vary markedly by small changes in the amount of intensive mixing due to the semi-Fisheye nature of Opalon resin (basically a problem of variable plasticizer absorption due to the inconsistency of the PVC particles).

In short, the present specifications can be met by a variety of PVC particles that do not necessarily produce good dry blending resins.

- F. The interesting effect on resin particle shape and structure by changes in the PDR-1 concentration indicates that two problems are encountered during the polymerization.
 1. Controlling the size of the individual particles which eventually agglomerate.
 2. Controlling the manner (shape and number) in which the individual particles agglomerate

It would appear rather fortuitous for one suspension system (susp. agents plus agitation) to be the optimum level for both problems. Altering the suspension system during the polymerization would appear necessary to produce the desired resin particle agglomerates.

V. Work in Progress

The following PDR-1 Characterization studies are in progress:

- A. Effect of high (above 10.0) PDR-1 specific viscosity on resin quality.
- B. Effect of blending PDR-1 lots of low and high specific viscosity.
- C. Effect of PDR-1 solution age on resin quality.

In addition, samples of Opalon 300FM will be microscopically inspected and tested in the porosimeter to better define particle parameters.

VI. REFERENCES

1. Suspension Polymerization of Vinyl Chloride, Part B., M.Baer .May 7,1958,Job. No. 696.
2. Preparation of Vinyl Acetate-Maleic Anhydride Heteropolymer as Suspending Agents for Vinyl Chloride Polymerization, Part A., M. Baer, Job No. 790
3. Process for Manufacturing Vinyl Acetate-Maleic Anhydride Heteropolymers- Part B, R.A. Hdmes, Aug. 31,1949 Job No. 790.
4. The Stability of Polymer Suspensions and the Control of Particle Size Distribution in Vinyl Chloride Polymerization, C.T.Chmiel, Dec. 1,1957, Job No. 1876.

5. Studies on the Stability of Polymer Suspensions and the Control of Particle Size Distribution - Part B., C.T.Chmiel; Feb.1, 1959, Job Nos.1876,1982.
6. Progress Report on PDR-1 Study; Vinyl PTS Project R-51, A.P.Torrenzano to F.A.Duston, July 21,1959.
7. Evaluation of PDR-14 Suspending Agent - Opalon 505, L.Vartanian to N.E.Aubrey, June 24,1957.

VII. DISCUSSION

A. NEED FOR PROJECT

In May 1959, Vinyl PTS Resin Project R-51 was initiated to redefine PDR-1 quality requirements for PVC suspension polymerization. The need for this project came from both direct and indirect problems associated with PDR-1 quality.

Directly: From January to May 1959 approximately 20 percent of the tested PDR-1 failed to meet product specifications, notably the coke bottle control lab test for PDR-1 acceptance as a suspending agent in Vinyl Chloride - vinyl acetate copolymerization. As a result, considerable quantities of unacceptable PDR-1 were held in inventory while the product was retested repeatedly in the unreproducible coke bottle test. The results of the coke bottle test could not be correlated with any known factors and therefore supplied no direction for improvement of PDR-1. Subsequently, inventories of PDR-1 for use as a copolymer polymerization suspending agent were dangerously low.

Indirectly: PDR-1 quality was a suspect in three copolymer coagulated batches and in homopolymer quality control problems. The three coagulated copolymer batches, which required a total of approximately 10 weeks to "Mine" out of the reactors, raised the fear that the entire 4 kettle line used for copolymer would be lost for a month if the four batches in process coagulated as a result of poor PDR-1. In homopolymer polymerization, the "poor to average" PDR-1 was not above suspicion for the simultaneously poor and erratic level in PVC blendability, extrusion, and heat stability.

Thus, Port Plastic was charged with the responsibility of improving PDR-1 quality level and variability while Springfield was commissioned with the project of determining the relationship between PDR-1 quality and PVC quality in order to redefine PDR-1 specifications. The marked improvement and continuing progress achieved by Port Plastics on this quality problem and of increased PDR-1 productivity had been documented in a series of progress reports by R.J.Grace. The results of work on the Springfield phase of the problem is the subject of this report.

B. REVIEW OF PREVIOUS WORK

It was considered essential to this present project to review past reports characterizing PDR-1 as a suspending agent for PVC polymerization in order to guide the experimental approach and help interpret results. A few previous reports pertinent to this project were paraphrased below.

Messrs. M. Baer and R.A. Holmes (in References 1, 2, and 3) defined five characteristics of PDR-1 with good suspending power, based on numerous coke tests and pilot plant studies.

1. Composition of VAMA: The best suspending power (defined by the minimum concentration of suspending agent needed to produce a PVC polymer passing a 60 mesh screen) was obtained with PDR-1 made with about equimolar proportions of Vinyl Acetate and partially esterified Maleic Anhydride.

2. Molecular Weight of VAMA: The suspending power increased with increasing molecular weight. VAMA of high molecular weight did not necessarily produce PVC of finer particle size than VAMA of lower molecular weight, but rather lowered the concentration of VAMA in PVC formulation needed to produce the optimum PVC particle size. As an example, 0.08% PDR-1 (based on the weight of VCM) of 6.0 specific viscosity was sufficient to produce the required PVC particle size, while 0.4 - 0.5% PDR-1 of 1.64 specific viscosity was required.

Only PDR-1 batches prepared by polymerizing VA-MA in equimolar proportions can be compared for specific viscosity since small changes in the ratio of the two monomers result in large changes in the viscosity of the resulting heteropolymer.

"Effect of Varying Monomer Ratio on the Viscosity of Resulting Heteropolymers"

<u>Vac (mols)</u>	<u>MA (mols)</u>	<u>*Specific Viscosity</u>
1.0	1.0	2.30
1.0	1.1	2.12
1.0	1.2	2.10
1.0	1.4	1.80
1.1	1.0	2.64
1.2	1.0	2.97
1.4	1.0	4.09

*Note: The above specific viscosity results were for a 0.4% concentration in water of unprecipitated PDR-1 made by the solvent-non solvent technique.

At ratios of higher than 1:1, VA polymerized by itself producing a water insoluble polymer which was emulsified by the VAMA resulting in a cloudy dispersion.

Infra-red absorption studies indicated that two polymers which differed only in their method of preparation had very different absorption spectras. In one of the polymers, the absorption band of the anhydride group was greatly diminished indicating a disappearance on anhydride groups possibly because of lactone formation. Any change in chemical structure of VAMA would, of course, have considerable influence on the viscosity of it's solutions, so that two polymers of identical molecular weight could display widely different specific viscosities. This could explain some discrepancies of results and some unexpected viscosity values.

The molecular weight of VAMA affected makedly the bulk density of suspension PVC. This correlation can be seen in the data on the following page.

Specific Visc.
(0.4% in Water)

2.63
2.37
2.30
1.91
1.64

PVC Bulk Density
(of 40 Mesh Material GR/CC)

0.615
0.554
0.516
0.490
0.354
0.294 - mat'l obtained by
blending PDR-1 and
1.64 spec. viscosity.

Rather surprising was the fact that when two VAMA batches of markedly different viscosities were blended the bulk density of the resulting PVC polymer was not only lower than the average of the bulk densities obtained, but also lower than the bulk density of either individual batch. If two VAMA batches of widely different specific viscosities were blended the PVC produced was coarser in particle size than that produced by either individual batch. This did not take place if the batches had about equal viscosities.

Initially PDR-1 was a copolymer of vinyl-acetate - maleic anhydride polymerized by a solvent (benzene) - non solvent technique. With continued development work the suspending action of PDR-1 was improved by modifying the balance of hydrophobic to hydrophilic groups in the polymer by partially esterifying some of the maleic anhydride with 2-ethyl hexanol prior to polymerization. In addition to the change in polymer composition, further developments improved the suspending action of PDR-1 by defining a modified mass polymerization technique which increased the level of the VAMA molecular weight than was above that possible with the solvent - non solvent technique and consequently improved suspending action.

3. Purity of PDR-1: Indications were that the more complete the polymerization of the VA-MA monomers the better the suspending action of the PDR-1. Low conversion PDR-1 which had been completely freed from unreacted monomers did not show any improvement in suspending action. Also, the suspending action of a good PDR-1 was not impaired by the addition of vinyl acetate or maleic anhydride in concentrations of those found in PDR-1 batches of poor conversion. It was concluded, therefore, that whatever caused conversion to be incomplete was also the cause for the poor suspending action. (Note: The PDR-1 produced by the recommended modified mass polymerization contained less than 2% unreacted maleic anhydride and had a specific viscosity greater than 1.7 for 1% unpurified PDR-1 in cyclohexanone).
4. Concentration of PDR-1 in PVC Formulation: The concentration of PDR-1 needed to produce PVC of desirable particle size depended on the molecular weight of the PDR-1 and the agitation of the PVC reactor. With constant agitation, the suspending action increased with increasing molecular weight. Although the molecular weight of PDR-1 did not determine the fineness of the PVC polymer but only the amount of suspending agent required to optimize PVC particle size, it had been found that PDR-1 of less than 2.0 - 2.5 specific viscosity (0.4% concentration in water) did not produce PVC polymers of satisfactory particle size.

If the concentration of PDR-1 as a suspending agent was plotted against the particle size of the resulting PVC, a curve showing a minimum was always obtained. The exact shape of the curve and the relative position of the minimum in the curve varied from batch to batch of PDR-1 but a minimum was always present. Three PDR-1 batches having specific viscosities of 1.6, 2.8, and 6.5 showed a minimum at a concentration of 0.4 - 0.5, 0.3 - 0.35, and 0.07 - 0.09 percent respectively.

5. Aging of PDR-1 Aqueous Solutions: On heating and long standing, chemical changes take place which cause the solutions to decrease in viscosity and the pH to drop, probably due to the hydrolysis of some acetate groups in VAMA with the liberation of acetic acid and by formation of lactones. PVC polymerization tests using aged PDR-1 solutions indicated that the suspending properties of PDR-1 became progressively poorer.

Age of VAMA Solutions at 50°C	% PVC Passing	
	60 Mesh	80 Mesh
0	97	87
1.5 hrs.	96	77
6.5 hrs.	88	63
22.5 hrs.	86	60
30.5 hrs.	80	52

Based on suspension stability studies in the Laboratory and pilot plant, C. T. Chmiel defined (in Ref. 4 and 5) a procedure to distinguish between "good" and "poor" suspending agents. In addition, a pH adjustment of the suspending medium was recommended to improve the suspending power of poor PDR-1 lots, and to narrow the particle size distribution of PVC homopolymer resin.

1. PDR-1 Characterization by Adsorption on PVC: By carefully dispersing PVC of approximately one micron in size in PDR-1 solutions of known concentration, and then filtering and titrating the filtrate with NaOH, an accurate measurement was obtained on the amount of adsorbed PDR-1 on PVC resin. The results indicated that a correlation existed between the amount of suspending agent adsorbed and the average particle size of PVC resin prepared in suspension polymerization. The adsorption isotherm for a PDR-1 - PVC system showed for two PDR-1 samples #42 and #46, that up to concentrations of 0.005 N PDR-1 the adsorption characteristics were the same. Beyond this concentration, the adsorption of sample #42 ("poor" PDR-1) decreased sharply whereas for sample #46 ("good" PDR-1) the adsorption remained reasonably constant. At a concentration of 0.01N PDR-1, which corresponded to the concentration employed for standard vinyl chloride polymerization, the adsorption of #42 was about two thirds that of sample #46.
2. Batch pH Adjustment: Polymerization studies showed that acidifying the suspending medium with mineral acid to 1.7 pH would improve "poor" suspending agents with a narrowing of the particle size distribution.

With "good" suspending agents, the pH adjustment resulted in an overall coarsening effect and narrowing of the particle size distribution. If NaOH was added instead of acid, a coarsening effect was also obtained as the pH increased from 3 to 5, but in general, the particle size distribution was broadened. At still higher pH large hard beads were formed.

C. EXPERIMENTAL APPROACH

Based on results of past studies it was decided that the objective of redefining PDR-1 specifications to meet the present day PVC requirements imposed three specific problems.

1. To establish significant quality differences in PVC suspension homopolymer and copolymer resins caused by changes in PDR-1 batches. In order to maximize the possibility for differences, batches of PDR-1 were selected which were acceptable by the coke bottle test, unacceptable by the coke bottle test, and covering a wide range of specific viscosity and conversion. The PVC properties to be considered were:

Opalon 300 FM

- a. Particle size
- b. Dry time
- c. Bulk density
- d. Color and heat stability
- e. Extrusion properties
- f. Brabender fusion time and fisheyes

Opalon 510

- a. Particle size
- b. Bulk density
- c. Melt index

2. To correlate a PDR-1 physical property with PVC resin quality. The first attempts at correlation were to be with the simpler tests such as (a) PDR-1 specific viscosity, using both purified and unpurified PDR-1, (b) PH and NaOH neutralization, and (c) conductivity of unpurified PDR-1, in water. If further testing was required, determinations could be made with (d) the polarograph for concentrations of free Maleic Anhydride, and (3) infrared analysis. Should these test techniques fail to correlate with PVC quality, then the suspending characteristics of PDR-1 could be explored using the adsorption technique developed by Chet Chmiel and lastly, the coke bottle test could be improved and possibly reduced in sensitivity to obtain a correlation.
3. To establish PDR-1 quality requirements for both Springfield and foreign plants. Since the quality problems at Springfield were not necessarily the same as those in foreign plants due to unavoidable basic differences (such as VCM and water quality) PDR-1 quality specifications different from those required at Springfield could, if economically justified, be utilized to improve PVC quality.

Another important phase of study which required simultaneous attention was the possibility of compensating for PDR-1 quality variations or even improving the PVC quality by altering the PDR-1 concentration in the PVC formulation. Conceivably PDR-1 specifications could be defined to permit an improved PVC quality at a PDR-1 concentration different from standard.

D. RESULTS AT STANDARD OPERATING CONDITIONS

The PDR-1 batches employed during this project were listed in Table 1. together with their pertinent data. Different groups of these 17 listed PDR-1 batches were utilized in a series of ten experiments designed to characterize PDR-1 performance.

1. PDR-I LOTS VERSUS PVC HOMOPOLYMER QUALITY (Exp. #2,#7)

PDR-1 lots 222, 225, 236, 237, and 242 (Group A) and later, lots 230, 240, 277, 278, 287, and 242 (Group B) were used in the polymerization of 50 acceptable batches of Opalon 300 FM by the X-57 process. Each batch was made in accordance with the Standard Operating Procedure. Any batch failing to adhere to the SOP was rejected and the run repeated. A sample of each batch was taken from the reactor, dried in the laboratory oven and tested for quality level. Since it was not possible to employ the same raw materials other than PDR-1) for the entire project, the comparison of results from one experiment to another, when necessary, was accomplished by using one PDR-1 lot as a control. In this case, Lot 242 was employed in both Groups. In any one experiment all variables not under study were held constant, including the lots of GMS, L2O2 and DAM.

- a. Particle Size: Differences in PDR-1 lots were responsible for very large shifts in particle size level. The severity of the shifts can best be gauged by inspecting Fig. 1 and noting that the spread of results was approximately four times as great as the Opalon 300FM specification range, which was ca. 90 to 140 SAF.

There was no significant difference in particle size variability associated with any PDR-1 lots.

The particle size results for the two groups of PDR-1 were compared on a common basis by adjusting the SAF results of Group B by an amount equal to the difference between lot 242 in Group A and in Group B.

<u>PDR-1 Lot</u>	<u>Average SAF</u>	<u>Adjusted Average SAF</u>
225	47	47
222	64	64
242	97	97
236	99	99
237	105	105
230	63	57
240	76	67
287	94	81
277	118	97
278	119	98
242	118	97

- b. Kitchen Mixer Dry Time: There was no change in dry time level with changes in PDR-1 Lots; see Fig. 2. The resin dry time averaged 4'40", with one run above 5'30".
- c. Bulk Density: There was no apparent shift in bulk density with PDR-1 lot changes except where PDR-1 coarsened resin particle size and consequently increased the bulk density; see Fig. 3
- d. Heat Stability: There was a significant (99%) variance in heat stability with changes in PDR-1 lots. The range of results of each PDR-1 were plotted in Fig. 4.
- e. Extrusion Properties: There was no apparent significant shift in resin extrusion grade or rate with PDR-1 lot change. The extrusion grade ranged predominately between a B and D rating; see Fig. 5.

It was rather surprising to encounter a reasonably good level of extrusion grade for the very coarse resin batches made with PDR-1 Lots 222, 225, 230 and 240. It was believed generally that good extrusion grades could not be obtained with resin coarser than 90 SAF.

For the 40 batches tested, 39 batches were within the specification limit of E maximum, with the average level at C. The extrusion rate averaged 145 GPM.

- f. Brabender Properties: Six of the seven Brabender fusion time results for PDR-1 Lot 287 were the highest values encountered at standard operating conditions - during this project. As can be seen in Fig. 6, two samples of Opalon 300FM made with PDR-1 lot 287 did not fuse (20+). The only known difference between lot 287 and the other tested lots of PDR-1 was in the method of manufacturing.

Lot 287 was representative of a series of PDR-1 batches made at Port Plastics to determine the effect of replacing 10% of the tertiary butyl per benzoate catalyst with benzoyl peroxide. Lot 277 and 278 represented PDR-1 batches made with 5% benzoyl peroxide, and all other PDR-1 lots tested were manufactured under standard operating conditions.

PDR-1 lot 287 was used again in a PDR-1 concentration study, which will be discussed in Section VI E, and showed a fusion time of approximately 25% higher than batches made with PDR-1 lot 277. It should not be concluded from these data that the 10% benzoyl peroxide catalyst system was responsible for the behavior of lot 287, but only that further testing of representative PDR-1 lots was warranted if the change to the 10% benzoyl peroxide catalyst system was considered desirable from a manufacturing point of view.

The Brabender fusion time results obtained when employing Group B PDR-1 lots ranged from ca. 5 to 11 chart units (excluding Lot 287) while Group A ranged from ca. 4 to 8 chart units, implying a change in an uncontrolled PVC process variable or a lack of understanding as to the meaning of Brabender fusion time values.

There was no correlation between Brabender Fisheye count and PDR-1 lots, Fig. 7.

2. PDR-1 LOTS VERSUS PVC HOMOPOLYMER QUALITY WHEN USING EDC VINYL MONOMER (Exp #4)

In an attempt to distinguish differences between PDR-1 lots 236, 237, and 242, which behaved very similarly when tested in Group A, eight batches of Opalon 300FM were manufactured using EDC vinyl chloride monomer.

About three months previously, in April 1959, two million pounds of vinyl chloride monomer made from ethylene dichloride, EDC, were received and used in the PVC operations with a simultaneous shift in resin particle size. The shift in particle size was to a coarseness level that could not be compensated for by changes in PVC operating conditions. As a result, over one million pounds of out-of-specification PVC was produced with a drastic effect on manufacturing costs. One tank of EDC monomer containing approximately 100,000 pounds was set aside for use again when the PVC particle size level returned to a normal level.

The eight batches of Opalon 300FM made with the three lots of PDR-1 were again very similar in all properties and comparable to the batches previously made with these lots when tested in Group A. The resin particle size level with the EDC monomer was only slightly coarser than normal production batches (sans EDC); see data sheets for experiment #2 and #4.

3. PDR-1 LOTS VERSUS PVC COPOLYMER QUALITY (EXP. #3 AND #5)

After PDR-1 lots 222, 225, 236, 237, and 242 had been tested in homopolymer polymerizations, the same five lots were used with the X-26 process to polymerize 18 batches of Opalon 510 copolymer. When no problems were encountered with these five lots of PDR-1, four lots of PDR-1 - 244, 247, 249, 251 - were tested which did not pass the coke bottle acceptance test but did have an acceptable specific viscosity and specific conductivity.

- a. Particle Size: Copolymer was unaffected by the changes in the PDR-1 lots tested. See Fig. 8, 9.
- b. Bulk Density: No shift in bulk density with PDR-1 lot changes; see Fig. 10.
- c. Melt Index: There was no change in melt index with PDR-1 lot changes; see Fig. 10.

E. CORRELATION OF PDR-1 QUALITY AND PVC QUALITY

The only properties significantly affected by more than one lot of PDR-1 were suspension homopolymer particle size and heat stability.

1. PARTICLE SIZE VERSUS PDR-1 QUALITY

After experimenting with PDR-1 lots in Group A, it appeared that the only correlation between particle size and PDR-1 quality was with specific conductivity (Ref. 6). However, after testing samples of the specific drum of PDR-1 used in the experiment and with further data from Group B, PDR-1 lots, it was evident that a significant correlation also existed with both purified and unpurified PDR-1 specific viscosity.

The relationship between resin particle size and purified PDR-1 specific viscosity, Fig. 11, showed that very little change in particle size occurred between a 7.0 - 9.0 specific viscosity level, while below a 7.0 specific viscosity the particle size coarsened rapidly. A very similar relationship, Fig. 12, was obtained between resin particle size and unpurified PDR-1 specific viscosity, with the critical point at a 3 specific viscosity.

The statement by M. Baer (Section VI, B) that only VAMA polymerized in equimolar proportions of vinyl acetate and maleic anhydride could be compared for specific viscosity was of particular importance in helping to interpret the experimental results obtained with PDR-1 lot 222. When tested in Group A, PDR-1 lot 222 resulted in very coarse homopolymer resin - 64 average SAF. This particle size level was inconsistent with the 7.8 specific viscosity resulted obtained for this lot. Two additional samples from the drum of PDR-1 used during the experiment were tested together with a repeat testing of the first sample. The results were inconsistent due to a difference in the PDR-1 quality for each sample tested.

Specific Viscosity For Lot 222

<u>Sample</u>	<u>Purified PDR-1</u>	<u>Unpurified PDR-1</u>
I-a	7.8	2.35
I-b	6.9	-
II	6.1	-
III	?	1.78

During the specific viscosity test, it was not possible to dissolve the PDR-1 completely in acetone before proceeding with the VAMA precipitation with benzene. It was necessary therefore to filter the mixtrue - removing the polyvinyl acetate solids - before completing the test. Samples II, I and III contained increasing amounts of polyvinyl acetate, with sample III producing insufficient quantities of benzene precipitated VAMA to test for specific viscosity. The

"Acetate Rich" VAMA polymer precipitated with benzene should exhibit a specific viscosity higher than anticipated from resin particle size data and Fig. 11. Therefore the SAF - specific viscosity data point for lot 222 was not added to Fig. 11.

To distinguish between "Equimolar" and "Acetate Rich" specific viscosity results would be a control test problem. Obviously any lot of PDR-1 which showed polymer insoluble in acetone should be rejected. Since, according to Ref. 1, VAMA containing as much as 20% excess vinyl acetate could be dissolved without a sign of polyvinyl acetate insolubles, and since this material would exhibit a 50% "higher" specific viscosity (which does not contribute to the suspending power of the VAMA) some additional safeguards were necessary. One solution would be to measure the presence of unreacted maleic anhydride to insure a minimum conversion of this monomer; a specific conductivity test would provide an approximate measurement. Adequate PDR-1 quality control could be expected with an 8.0 minimum specific viscosity, a 1600 micromhos maximum specific conductivity, rejection of PDR-1 lots exhibiting insoluble polymer, and the standard plant trial of one suspension batch. Note that lot 222 would have been rejected on all four counts.

The specific conductivity of a PDR-1 water solution was initially of considerable interest prior to establishing the specific viscosity correlations. To understand the significance of specific conductivity variations, samples of PDR-1 were purified and tested. The results showed a level of approximately 550 micromhos for VAMA that had been benzene precipitated. In addition, conductivity levels were synthesized by adding vinyl acetate, maleic anhydride, and both vinyl acetate and maleic anhydride to PDR-1 water solutions. These data showed that with only maleic anhydride added, the conductivity was affected as shown in Fig. 13, and the influence (the slope of the line) was approximately 50% that expected from the general correlation between PDR-1 conversion and specific conductivity in Fig. 14. When added alone vinyl acetate had a negligible effect on conductivity, while when added together with maleic anhydride, the vinyl acetate apparently hydrolyzed to acetic acid and acetaldehyde and contributed to the solution conductivity. The conductivity data for the PDR-1 solutions with added maleic anhydride and vinyl acetate showed the same slope as Fig. 14 up to a 9% monomer concentration, with a sharp leveling off as additional monomer was added, Fig. 15.

A significant correlation was obtained also between resin particle size and the specific conductivity of a PDR-1 (unpurified) water solution for the various PDR-1 lots tested, Fig. 16. The fact that when maleic anhydride added to the PVC formulation did not influence the resin particle size indicated that the conductivity was not the prime variable. The same was true for unpurified PDR-1 specific viscosity since research work had shown no change in suspending power when PDR-1 was purified prior to use in PVC polymerizations and no change when PDR-1 was made more "impure" by adding vinyl acetate and maleic anhydride. It was concluded, therefore, that the prime variable was the molecular weight of the VAMA, which was related to the specific viscosity of a purified PDR-1.

The secondary correlations with unpurified PDR-1 specific viscosity and specific conductivity were understandable since the polymerization of high molecular weight VAMA was synonymous generally with high monomer conversion.

2. HEAT STABILITY VERSUS PDR-1 QUALITY

There was a significant (99%) correlation between resin heat stability and PDR-1 specific conductivity, but not with PDR-1 specific viscosity. This indicated that the unreacted monomers affected the resin heat stability.

In figure 17 the calculated correlated curve for all the data was shown with a solid line; the two sigma confidence limits were $\pm 11\%$ transmission. Actually it was suspected that the correlation might be slightly steeper but for the variation in efficiency of washing slurry samples prior to testing. All the samples were washed in the same manner, employing a specified amount of water per specified weight of resin. The coarse resin with less total surface area probably was washed more efficiently than the finer resins. Since the PDR-1 specific conductivity also was inversely proportional to the resin particle size, it was possible that the heat stability correlation was masked partially. By eliminating the two very high specific conductivity data points - 2740 and 2355 micromhos - that were associated with very coarse resin and recalculating the correlation for the remaining points, a steeper correlation was obtained that was 99.9% significant (dotted line on Fig. 17).

Further experimentation with additional PDR-1 lots to exploit the accurate correlation was not considered justified since a specific viscosity and specific conductivity specification imposed to insure PVC particle size control would also satisfy the heat stability requirement.

Obviously the purification of PDR-1 prior to use in PVC polymerization would warrant consideration if the centrifuge washing studies do not eliminate the resin heat stability dependency on PDR-1 quality and quantity.

F. PDR-1 RAW MATERIAL SPECIFICATIONS

From the results todate, the following specifications would be required to insure PVC quality control.

1. FOR HOMOPOLYMER USAGE

- a. Specific Viscosity of purified..... 8.0 minimum
PDR-1 in water *13.0 maximum
- b. Specific conductivity of 0.4%..... 1600 maximum
PDR-1 Water Solution, Micromhos
- c. No PDR-1 Polymer Insoluble in Acetone

2. FOR COPOLYMER USAGE

- a. Specific Viscosity of Purified..... 6.0 minimum
PDR-1 in Water*13.0 maximum
- b. No PDR-1 Polymer Insoluble in Acetone
- c. Master Blends For Use In Copolymer Must Consist of Individual PDR-1 Lots Each With A 6.0 Minimum Specific Viscosity.

The reasons for the homopolymer usage requirements were discussed in the previous section. The safe, 6.0 minimum specific viscosity and blending PDR-1 specifications for copolymer usage were selected to prevent any boarderline PDR-1 quality which might result in coagulated copolymer batches.

No attempt was made to distinguish between Springfield and foreign plant requirements for PDR-1 quality.

G. PDR-1 CONCENTRATION IN PVC FORMULATION

The possibility of improving PVC quality and/or compensating for lot to lot variations in PDR-1 quality by optimizing the PDR-1 concentration in the PVC homopolymer formulation was explored during this project. Some previously unreported plant work will also be added to the discussion below to support the conclusions.

Back in March 1958, a study of PDR-1 concentration was started in 92 Building using Opalon 300 (X-57) as the subject. It should be noted that the agitative systems in 88 and 92 Buildings differed - and still do - by the type and position of the baffles in the reactor. This difference was pointed out since it was believed that agitation and suspending agent behavior were interrelated and conclusions based, for example, on coke bottle results, pilot plant reactor, or even 92 Building results, might not show up in a different agitative system.

The influence of baffling of the reactor can be highlighted from the particle size data below which were collected simultaneously in both 88 and 92 Buildings while all other known variables were held constant.

88 Building

Agitation: 54 inch span, retreating type impeller, rotating at 121 RPM, with two 4 $\frac{1}{2}$ " x 15" three fingered baffles positioned low in the reactor.

RUN NO.	PARTICLE SIZE % ON MESH						PAN	SAF
	40	60	80	100	140	200		
5-81	0	16	48	22	11	2	T	89
5-87	0	10	46	29	12	2	T	93
5-95	0	10	46	29	12	2	T	93
5-102	0	3	32	38	23	3	T	104
5-109	0	11	48	30	10	1	T	91
5-116	0	8	37	34	19	2	T	99
5-123	0	14	46	28	12	2	T	94

Ave. 95

*Tentative until work in progress completed.

RSV0024091

92 Building

Agitation: 54 inch span, retreating type impeller, rotating at 121 RPM, with two 8½" x 24" two fingered baffles positioned high in the reactor.

RUN NO.	PARTICLE SIZE, % ON MESH							SAF
	40	60	80	100	140	200	PAN	
8-6409	0	T	7	22	45	21	4	14-3
8-6422	0	1	4	20	50	20	4	144
8-6429	0	1	5	20	48	22	4	146
8-6437	0	1	8	24	45	18	4	140
8-6445	T	4	30	32	27	6	1	119
8-6453	0	2	11	29	44	13	2	132
8-6460	T	2	11	25	41	17	4	136

Ave.137

The larger baffles in 92 Building had approximately 408 square inches of surface area as compared to 203 square inches per baffle for the smaller baffles in 88 Building. This factor increased the baffling efficiency and resulted in a higher agitating intensity, as defined by the "Pfaudler Agitative System"; a 10.5 A.I. for 88 Building and a 12.3 A.I. for 92 Building. However, when the Agitative Intensity was changed a comparable amount by increasing the impeller speed in 88 Building very little effect was noted on resin particle size. It could be concluded only that the large baffles, positioned high in the reactor produced a finer resin due to a basic difference in flow pattern which probably provided a higher rate of vertical flow for the agitated fluid.

1. EFFECT OF PDR-1 CONCENTRATION ON OPALON 300 IN 92 BLDG. (Exp.#A)

The data in Table 2 were obtained from Opalon 300 batches using different concentrations for PDR-1 Master Blend #35.

Excluding runs 12 and 15 which appeared to be influenced by some other variable, these data indicated that PDR-1 concentration changes from 0.03% to 0.20% had very little effect on resin particle size level or distribution. With PDR-1 concentrations above 0.20%, the resin particle size distribution was broadened. This can be seen in Fig. 18 which shows the PDR-1 concentration plotted versus the Geometric Standard Deviation - defined as the slope of the particle size distribution curve when plotted on logarithmic probability paper, Ref. 7. Surprising was the fact that as little as 0.03% PDR-1 was sufficient to maintain an adequate suspension. Run 18, which was made using a 0.018% PDR-1 concentration, had over 20% resin of plus 40 mesh size and not tested for other properties.

With increasing PDR-1 concentrations the resin bulk density, Reed Mixer dry time, extrusion grade, and extrusion Fisheye count all decreased significantly. See Figures 19, 20, 21, and 22. Unfortunately no resin heat stability data was obtained during this experiment.

The rather promising resin processing characteristics for a "full conversion" type resin was pursued further in a study of PDR-1 concentration and batch pH, which will be discussed in Section VII, H.

2. EFFECT OF PDR-1 CONCENTRATION ON OPALON 300FM IN 88 BLDG. (Exp. #1)

During this present PDR-1 characterization project, further PDR-1 concentration studies were made and this time Opalon 300FM (X-57) in 88 Building was the subject.

Sixteen batches were polymerized in two kettles with slightly different baffle positions using PDR-1 master blend 47 and lot 237 at four levels of concentration - 0.06%, 0.12%, 0.18%, and 0.24%.

a. Particle Size: The particle size data, Fig. 23, for this experiment was extremely variable and could not be correlated with the variables under study, or for that matter, with any other variable. Therefore no useful particle size data was obtained from this experiment.

b. Dry Time: There was no apparent correlation between PDR-1 concentration and kitchen mixer dry time results Fig. 24, however the particle size variability could have confounded this relationship.

The relationship between the two dependent variables of kitchen mixer dry time and resin particle size can be seen in Fig. 25. It was hypothesized that the poor dry time levels for fine resins was primarily a result of the poor mixing action of fine resin in the type equipment used in this test and does not necessarily mean fine resin has less total void volume for plasticizer adsorption. The interaction of resin properties-other than the property under study - with test equipment can be extremely misleading especially if industrial equipment behaves differently.

c. Bulk Density: Increasing PDR-1 concentration decreased resin bulk density as seen in Fig. 26. The cause for the bulk density was apparent when samples were examined under a microscope. With low PDR-1 concentrations, the individual PVC agglomerates consisted of 3 to 4 particles fused together into a generally spherical shape; the agglomerates were fairly coarse. With high PDR-1 concentrations, the individual agglomerates consisted of 10 to 15 small particles fused together into myriad shapes thus resulting in a low bulk density.

d. Brabender Properties: Increasing the PDR-1 concentration had, in general, the same effect on Brabender fusion time as on bulk density. It was suspected that this again was a case where a dependent variable (bulk density) and test equipment were confounding the significance of fusion time results. See Fig. 27.

The Brabender Fisheye count showed no correlation with PDR-1 concentration, Fig. 28.

- e. Extrusion Properties: There was no correlation between PDR-1 concentration and Opalon 300FM extrusion grade, Fig. 29.
- f. Heat Stability: Kettle slurry samples were washed in the control lab prior to drying and testing. The heat stability of the samples were improved markedly by water washing, Fig. 30. In determining the effect of PDR-1 concentration on heat stability, all samples were washed equally. The results showed that decreasing the PDR-1 concentration improved resin heat stability, Fig. 31.

The importance of this correlation will depend on a centrifuge washing study now in progress. It has been established already that the present method of centrifuging Opalon 300FM was equivalent to approximately "one washing" in the laboratory (see Section VIII, A). Marked improvements were expected by improved washing in the centrifuge such that regardless of the PDR-1 concentration the same high level of heat stability might be attained by removing all excess PDR-1 adsorbed on PVC resin.

3. EFFECT OF PDR-1 CONCENTRATION OF OPALON 300FM in 88 Bldg. (Exp. #8.)

The PDR-1 concentration study was repeated but this time the kettle baffle variable was eliminated and the experiment run in triplicate using PDR-1 lot 277 and lot 287 at four levels of concentration - 0.06%, 0.12%, 0.18%, and 0.24%. PDR-1 lots 277 and 287 were selected since lot 277 represented the highest quality (specific viscosity) PDR-1 available at the time and lot 287 produced significantly higher Brabender fusion time results when previously compared in Group B (Section VII, D).

Within the range studied, PDR-1 concentration changes showed little effect on the resin particle size level (or more exactly, the screen analysis) except for an indication of a coarsening effect when PDR-1 lot 277 concentration dropped from a 0.12% to 0.06% level. No marked difference in screen analysis distribution was encountered at the high PDR-1 concentrations as had been the case with PDR-1 master blend 35 in 92 Building.

With both PDR-1 lots, PVC resin bulk density, Brabender fusion time and heat stability again showed a dependency on PDR-1 concentration. Results in general, agreed with the data previously obtained on Opalon 300 in 92 Building. See Figures 32, 33, 34, 35, 36, and 37.

4. SUMMARY AND CONCLUSIONS

PDR-1 concentration changes had a significant effect on the shape and structure of the PVC particle agglomerate. Although the overall size of the agglomerate did not change, as measured by a screen analysis, the low PDR-1 concentration produced a more consistent, spherical agglomerate similar to successful competitive resins made by Dow and Goodrich. This spherical agglomerate resulted in a resin of higher bulk density and consequently poorer Brabender fusion time.

Since the more consistent spherical particles did not have the total surface area possessed by the varied shaped particles made with high PDR-1 concentration, the dry time for full conversion resin such as Opalon 30Q was high. With the low conversion FM resins the internal void volume became the controlling factor in plasticizer adsorption rates and therefore there was little noticeable difference in dry time results for high and low PDR-1 made batches.

The myriad shapes encountered with Opalon 300FM particles made with high PDR-1 concentrations explained the tendency for a broader screen analysis distribution.

The extrusion grades obtained from PVC batches made with various PDR-1 concentration was contrary to what would be expected from the particle shape. Better extrusion grades were obtained from Opalon 300 as the PDR-1 concentration was increased and the particle shape made irregular and inconsistent. With Opalon 300FM a poorer extrusion grade was obtained at very low PDR-1 concentrations, with the more spherical, consistent particles. Possibly, with the large individual particles comprising the agglomerates (at the low PDR-1 concentration) any impervious particle was readily apparent in an extruded ribbon, while a small impervious particle would be much less noticeable.

Resin heat stability level was progressively poorer as the PDR-1 concentration was increased. A higher concentration of adsorbed PDR-1 in the dried PVC product was the explanation. This undesirable result can be improved by more efficient washing of the PVC polymer during the centrifuging operation.

By the present specifications for PVC, improved resin quality could be obtained at the higher PDR-1 concentrations, provided the excess PDR-1 was washed off the PVC resin. Although this approach would improve the PVC quality level as measured by the present specifications, and might also produce a more competitive resin, it was the author's opinion that basically this approach would never produce an excellent dry blending resin, since a very important requirement for proper performance in extrusion was a high bulk density resin (obtained with spherical shape not at a sacrifice of particle porosity) with good dry blend flowing properties.

H. ACID AND BASE ADDITIONS TO PVC FORMULATION

1. EFFECT OF PDR-1 CONCENTRATION AND pH ADJUSTMENT (Exp. 8)

Research work (Ref. 4 and 5) had shown that acidifying the suspending medium with mineral acids to a pH of 1.7 would improve poor PDR-1 lots, result in a narrowing of the resin particle size distribution, and a coarsening of the particle size level. If sodium hydroxide was added instead of acid, a coarsening effect would be obtained as the pH was increased from 3 to 5, with the particle size distribution broadened. At still higher pH, large hard particle beads would be formed.

The possibility of employing PDR-1 concentration and pH adjustment to control resin particle size and distribution was the subject of a designed 25 batch plant experiment in April 1958. Sulfuric acid and sodium hydroxide were used to adjust the pH of the suspending medium - prior to the addition of VCM - to pH levels of 1.8, 2.4, 3.0, 3.5, and 4.0 while PDR-1 Master Blend 38 was used at concentrations of 0.06, 0.10, 0.15, 0.20, and 0.25 percent.

The procedure employed during the experiment consisted of dissolving the specified amount of PDR-1 in the pre-mix tank and after charging to the reactor - which contained the process water - sampling the dilute solution and measuring the pH and titrating with sodium hydroxide. The titer was compared to a PDR-1 concentration - NaOH neutralization, calibration curve for the specific lot of PDR-1 being used. In this manner the exact PDR-1 concentration in the reactor was measured (Note: With the multi-kettle PDR-1 charging system gains or losses of PDR-1 were possible from one charge to another.) After the pH measurement the amount of 98% sulfuric acid or 50% sodium hydroxide required to adjust the pH to the specified level was estimated. The addition to the reactor was made, the reactor contents allowed to agitate for 5 minutes, and then a sample taken. After the polymerization was completed, a slurry sample was taken from the kettle, dewatered and dried in the laboratory. One very important operation was overlooked during this experiment, that of diluting and washing the slurry samples to simulate plant slurry handling operations. This omission must certainly have been partially responsible for the resin heat stability and volume resistivity results obtained.

After manufacturing 16 batches of Opalon 300 in 92 Bldg. the experiment was halted when the feed-back of quality results for the acidified batches showed an unexpected drastic effect on resin heat stability. Possible reasons for the poor heat stability were (1) the catalytic effect on thermal degradation by the sulfuric acid adsorbed on the PVC polymer and (2) the loss of the stabilizing effect of glyceryl monostearate by an accelerated acid-water hydrolysis of the ester to glycerol and stearic acid. The "loss" of the glyceryl monostearate could also account for the poorer resin extrusion grade of the acidified batches.

The quality results were summarized in order of increasing pH in Table 3.

- a. Particle Size: The addition of sulfuric acid caused a coarsening effect on the resin particle size level. The effect of sulfuric acid on the particle size distribution was negligible, in general, when compared to distributions for other coarse resin such as produced by low viscosity PDR-1. However, an accurate measurement of the screen analysis distribution for coarse resins can not be obtained with the size screens used in the standard test. This was due to the large difference in screen apertures for the 40 and 60 mesh screens, as compared to the screen aperture difference for the other screens.

<u>U.S. Screen Mesh</u>	<u>Aperature, Microns</u>
40	420
60	250
80	177
100	149
140	105
200	74

As can be seen from the above listing, the difference between a 40 and 60 mesh screen was approximately equal to the difference between a 60 and 200 mesh screen. Since there were three additional screens between the 60 and 200 mesh screens, the impression of a broader distribution was created, as shown below.

<u>Screens, Mesh</u>							<u>Screens, Mesh</u>			
40	60	80	100	140	200	PAN	40	60	200	PAN
1	84	12	2	1	T	T	1	84	15	T
1	93	3	T	1	1	T	1	93	5	T
T	1	14	24	38	18	5	T	1	94	5
T	8	26	17	23	15	11	T	8	81	11

Since there was an insufficient number of screens between the 40 and 80 mesh fractions, accurate distribution measurements, even with probability paper, were not possible. With the finer resins a more meaningful measurement was possible.

Increasing the PDR-1 concentration and/or adding sodium hydroxide had a broadening effect on the particle size distribution as can be seen by the increase in the Geometric Standard Deviation Values in Fig. 38.

The effect of adding sodium hydroxide was primarily on the particle size distribution with little change (possibly finer) on the particle size level. Not evident from the screen analysis for the 4.63 pH batch was the fact that this batch had approximately 5% agglomerated particles of plus 20 mesh size. Quite probably any batch polymerized at a pH of 5. or above would have been a total loss.

- b. Bulk Density: The lowering of the resin bulk density with increasing PDR-1 concentration was again quite significant, Fig. 39.
- c. Volume Resistivity: The expected effect on volume resistivity by the addition of sodium hydroxide was evident from Table 3. The addition of sulfuric acid had no marked influence on VR, with possibly a decrease in VR when the pH was lowered down to the 1.6 - 1.8 range.

- d. Extrusion Properties: The extrusion Fisheye count was increased markedly by the addition of sulfuric acid, as can be seen in Fig. 40. The limited data plotted could be somewhat misleading implying an improvement in the Fisheye count at low PDR-1 concentrations. It was suspected that additional data would show a generally worse and more variable level in extrusion properties as the PDR-1 concentration was decreased, with the average level at any one PDR-1 concentration becoming progressively poorer as the batch pH was lowered.

The extrusion grade was related directly to the extrusion Fisheye count.

- e. Heat Stability: The limited data on resin heat stability showed an extremely poor level for the batches made with sulfuric acid.

2. SUMMARY AND CONCLUSIONS

The results indicated no practical advantages to pH adjustment since acidified batches exhibited poor extrusion properties and heat stability, while batches made with sodium hydroxide had a broad particle size distribution and a lower volume resistivity level. The effect on resin particle size level by pH adjustment was of no practical value since low specific viscosity PRD-1 would produce coarse resin comparable to acidified batches, while the effect of sodium hydroxide on resin particle size could be duplicated by increased PDR-1 concentration.

3. ADDITION OF LAURIC ACID AND MALEIC ANHYDRIDE (Exp. #6)

There was reason to suspect that the concentration of acidic material in two PVC suspension raw materials was related to changes in homopolymer resin particle size. Previous plant work had established a correlation between the free fatty acid concentration in nine lots of lauroyl peroxide and changes in resin particle size. In addition, the acidity of methyl glucoside dilaurate was a suspect in the particle size control problem encountered when attempting to use this raw material as a replacement for glyceryl monostearate.

Lauric acid was added to the PVC formulation of nine Opalon 300FM batches to determine the maximum tolerable concentration. The following formulation was used during this study, with the reagent grade lauric acid charged to the reactor at the same time as the lauridyl peroxide.

OPALON 300FM, X-58 PROCESS

<u>Raw Material</u>	<u>Weight</u>	<u>Percent</u>
1. VCM	10,980. lbs.	100.
2. Water	14,820. lbs.	135.
3. PDR-1	16.5 lbs.	0.15
4. GMS	22.0 lbs.	0.20
5. L2O2	22.0 lbs.	0.20
6. DAM	1,600. lbs.	0.035
7. Lauric Acid	100 to 900 grams	1.0 to 9.0 based on L2O2

Following this lauric acid series, three batches of Opalon 300FM were made with maleic anhydride added to the formulation - in place of lauric acid - to determine if resin particle size was affected by the presence of unreacted maleic anhydride in PDR-1. Note that research work during the development of PDR-1 had shown no apparent effect on resin particle size by the purification of PDR-1 or addition of maleic anhydride to PDR-1 post-polymerization.

Monsanto technical grade maleic anhydride was dissolved in one gallon of water and added to the PDR-1 pre-mix tank at the start of the PDR-1 dissolving operation. The amount of maleic anhydride added was sufficient to increase the specific conductivity of a 0.4% PDR-1 water solution from 1520 to 2700 micromhos. If the presence of unreacted maleic anhydride per se was the cause for the large shifts in homopolymer particle size encountered with different lots of PDR-1, then the particle size level should be related to the specific conductivity as shown in Fig.

The particle size results of all the batches made using lauric acid or maleic anhydride showed no significant differences, as shown in Table 4. It was concluded, therefore, that neither lauric acid nor maleic anhydride was the prime variable in the raw material difficulties mentioned above.

VII. OPERATING PROCEDURE

The standard operating procedures as described in "Vinyl Chloride Polymerization, 92 Bldg., S.O.P., Manual Sections 630.0 through 639.0, Jan. 27, 1959" were employed during this study except for the defined formulation changes and the handling of the slurry for dewatering and drying. The salient features of the processes were summarized below.

A. SUSPENSION HOMOPOLYMER

1. FORMULATION:

<u>Raw Material</u>	<u>OPALON 300FM (X-57 PROCESS)</u>			<u>OPALON 300</u>		
	<u>Parts/100</u>	<u>Parts VCM</u>	<u>Weight</u>	<u>Parts/100</u>	<u>Parts VCM</u>	<u>Weight</u>
Vinyl Chloride Monomer	100		10,980. lbs.	100.		10,980 lbs.
Water	135		14,820. lbs.	135.		14,820 lbs.
Glyceryl Monostearate	0.2		22. lbs.	0.2		22 lbs.
Lauroyl Peroxide	0.26		28.5 lbs.	0.26		28.5 lbs.
Diallyl Maleate	0.20		0.20 lbs.	0		0
*PDR-1	0.15		16.5 lbs.	0.15		16.5 lbs.

RSV0024099

*Represented the amount used under standard operating conditions.

2. REACTION TEMPERATURE: $53 \pm \frac{1}{2}^{\circ}\text{C}$ for both products
3. CONVERSION: Opalon300FM was approximately 77% conversion polymer. To obtain this conversion, 1200 lbs. VCM was "slow vented" over a one hour period starting approximately 10 hours after the VCM charge to the reactor, and 1000 lbs. VCM was "final vented" over a 25 minute period starting at a 90 psig. level during the pressure drop. Approximately 330 lbs. of VCM were left in the polymer slurry.

Opalon 300 was approximately 91% conversion polymer, obtained by allowing the polymerization to near completion and then final venting 600 lbs. of VCM starting at a 50 psig. level during the pressure drop. Approximately 400 lbs. of VCM was left in the polymer slurry.

4. DEWATERING AND DRYING: This phase of the operation was performed in the Control Laboratory. A three gallon slurry sample of each PVC batch made was taken from the reactor immediately after the completion of the polymerization. The polymer in the sample was allowed to settle out and the liquid phase was decanted. The polymer was then transferred to porcelain trays and dried at air temperatures of 50-60°C for approximately 24 - 36 hours (or until dry to the touch).

When resin heat stability results were desired a portion (approximately one gallon) of the slurry sample was washed prior to drying. This was performed by decanting off the liquid from the settled out polymer, transferring the polymer to a Buchner funnel (muslin cloth as the filtering media) and allowing to drain. Then a 1000 grams of the wet polymer was reslurried in a clear gallon jar with 1000 milliliters of approximately 20 - 25°C tap water. The slurry, after being hand shaken for approximately 2 minutes, was again filtered in the Buchner funnel. This operation was termed "one washing", and by repeating the reslurrying of the wet polymer as many washing as specified were obtained.

B. SUSPENSION COPOLYMER

1. FORMULATION:

<u>Raw Material</u>	<u>OPALON 510 (X-26 PROCESS)</u>	
	<u>Parts/100 Parts VCM+VAc</u>	<u>Weight</u>
Vinyl Chloride Monomer	82.	9,020 lbs.
Vinyl Acetate Monomer	18.	1,980 lbs.
Water	126.	13,860 lbs.
Lauroyl Peroxide	0.20	22 lbs.
Trichloroethylene	1.0	110 lbs.
PDR-1	0.127	14 lbs.

2. REACTION TEMPERATURE: $67 \pm \frac{1}{2}^{\circ}\text{C}$

3. CONVERSION: Opalon 510 was approximately 90 conversion polymer, obtained by allowing the polymerization to proceed until the batch pressure dropped to 25 psig and then cooling the batch (via the jacket water system) as rapidly as possible until the batch pressure reached opsig.
4. DEWATERING AND DRYING: A one gallon sample of the slurry in the reactor was taken to the Control Laboratory for decanting and oven dried with 40-50°C air.

IX. DESCRIPTION OF EQUIPMENT

All the Opalon 300 FM experimental batches were polymerized in 88 Bldg. equipment while the 92 Bldg. equipment was used for the Opalon 300 and Opalon 510 batches. The only known major difference in the equipemnt was the agitative systems for the reactors.

Type PVC Resin	Kettle Bldg.	<u>AGITATIVE SYSTEMS</u>		Type Baffles	Baffle Position	Agitative Intensity
		<u>Impeller</u> Size	<u>Speed</u>			
Op. 300 FM	#8, 88 Bldg.	54"	126 RPM	A, A	X	12.0
	#5, 88 Bldg.	54"	126 RPM	A, B	X	12.0
	#6, 88 Bldg.	54"	126 RPM	A, A	X	12.0
	#7, 88 Bldg.	54"	126 RPM	A, B	Y	12.0
Opalon 300	#5, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#6, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#7, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#8, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
Opalon 510	#1, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#2, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#3, 92 Bldg.	54"	121 RPM	C, C	Z	12.0
	#4, 92 Bldg.	54"	121 RPM	C, C	Z	12.0

Notes

A= $4\frac{1}{2}$ " x 15" three fingered upward deflecting baffle

B= $4\frac{1}{2}$ " x 15" three fingered downward deflecting baffle

C= $8\frac{1}{2}$ " x 24" two fingered upward delecting baffle

X= Baffle positioned perpendicular to flow. Baffle length into kettle ranged from 100 to 106 inches

Y= Baffle positioned perpendicular to flow. Baffle length into kettle ranged from 78 to 81 inches

Z= Baffle positioned perpendicular to flow. Baffle length into kettle was 69 inches

Test Procedures

The quality results reported, were obtained with the below listed test procedures.

A. PDR-1 QUALITY

1. PURIFIED SPECIFIC VISCOSITY.....SDP-508-72 B
Prior to testing, the sample was purified by dissolving in acetone (to remove acetone, vinyl acetate, and maleic anhydride) and precipitating the polymeric VAMA with benzene.
2. UNPURIFIED SPECIFIC VISCOSITY
Same test procedure as above was used, except for the elimination of acetone dissolving - benzene precipitating technique for sample preparation. The PDR-1 was tested "as is".
3. DILUTE SOLUTION SPECIFIC VISCOSITY
The sample was not purified. Efflux times were measured for a 0.10% PDR-1 water solution at 60°C. The two viscosity procedures listed above employed a 1.0% PDR-1 water solution at 25°C.
4. % CONVERSIONSDP-508-72-4
5. SPECIFIC CONDUCTIVITYSDP-508-72-5
- B. OPALON 300 FM QUALITY SDP-508-1359-1A
 1. PARTICLE SIZE.....SDP-508-1359-1A

Note: $SAF = \frac{(\% \text{ on } 40 \text{ mesh})(40) + (\% \text{ on } 60 \text{ mesh})(60) + \dots + (\% \text{ on Pan})(250)}{100}$

 2. BULK DENSITY.....SDP-508-1500 A
 3. DRY TIME
 - a. Reed.....SDP-508-1368
 - b. Kitchen Mixer.....SDP-508-1368-1
 4. HEAT STABILITY.....SDP-508-1363A
 5. EXTRUSION PROPERTIES.....SDP-508-1366A
 6. BRABENDER PROPERTIES
As described in "Opalon Technical Service Application Laboratory Report #3: Development of a Test Procedure for Determination of Performance Requirements of Dry Blending Resin", A.B.Bonneville, J.L.Young, Sept. 14, 1959.
- C. OPALON 510-26 QUALITY
 1. PARTICLE SIZE.....SDP-508-1359-1A
Note: SAF for copolymer was calculated the same as above except that the "% on Pan" was multiplied by 300.
 2. BULK DENSITY.....SDP-508-1500 A
 3. MELT INDEX.....SDP-508-1358 plus
SDP-508-1061B

DATA SHEET: EXPERIMENT #1; PDR-1 LOTS, PDR-1 CONCENTRATION, AND TWO DIFFERENT KETTLES
VERSUS OPALON 300 FM QUALITY

RSV0024103

BATCH NO.	PDR-1 LOT.	KETTLE NO.	DESIRED PDR-1 CONC.	ACTUAL PDR-1 CONC.	BULK DENSITY GR./CC		DRY TIME			BREMENDER		PARTICLE SIZE								EXTRUSION		HEAT STABILITY	
					RESIN	DRY BD.	REED VIS.	MIN: SEC. REED ADJ.	KITCH. MIXER	FUSION TIME	V.F.F.	% RETAINED ON MESH								SAFE	RATE GR/HW	GRADE	% TRANE
3244	M.B. 47	6	.06	.074	.471	.583	3:15	3:00	6:00	6:25	6	T	2	30	27	27	1	0	102	135	B	69	
3309	"	"	.12	.12	.430	.562	2:30	2:30	4:15	6:50	4	T	27	47	19	7	1	T	85	128	ROUGH	-	
3237	"	"	.18	.19	.427	.558	2:30	2:30	4:30	5:15	10	T	79	17	2	1	T	0	64	137	B	46	
3281	"	"	.24	.22	.388	.475	3:00	2:45	4:45	4:30	7	1	6	39	27	20	5	2	105	117	ROUGH	21	
3316	"	7	.06	.068	.489	.585	3:00	3:00	6:30	20:30	②	T	2	20	39	34	6	T	116	136	B	65	
3246	"	"	.12	.14	.435	.553	3:00	3:00	6:00	4:45	17	T	5	34	24	21	3	2	105	141	B	62	
3284	"	"	.18	.18	.417	.538	2:30	2:15	4:45	4:45	10	T	38	38	12	8	1	0	78	130	C	39	
3322	"	"	.24	.24	.414	.571	2:45	2:30	4:45	4:45	4	9	77	9	3	2	1	T	65	142	B	16	
3315	BA 237	6	.06	.088	.449	.558	3:45	3:15	4:45	13:15	③	T	2	35	39	21	4	1	106	127	ROUGH	66	
3289	"	"	.12	.12	.400	.495	3:15	3:15	6:45	5:25	13	T	1	5	17	35	29	10	158	97	E	52	
3321	"	"	.18	.19	.390	.500	2:00	2:15	4:00	4:00	3	1	13	46	25	10	3	1	90	115	ROUGH	50	
3296	"	"	.24	.24	.415	.550	3:45	3:30	4:45	4:00	3	T	70	17	6	3	1	T	68	126	B	46	
3303	"	7	.06	.074	.453	.550	3:30	2:45	5:45	17:15	④	T	1	3	23	52	19	3	144	123	C	59	
3290	"	"	.12	.14	.420	.520	3:15	3:00	5:30	5:30	17	T	T	1	9	45	31	11	162	110	F	63	
3240	"	"	.18	.20	.400	.507	3:30	3:15	7:30	4:00	15	T	2	16	25	36	15	7	137	142	D	47	
3310	"	"	.24	.23	.398	.474	7:30	4:30	11:30	2:30	6	T	T	2	12	32	32	20	173	140	D	45	

DATA SHEET : EXPERIMENT #2 ; DIFFERENT LOTS OF PDR-1 VERSUS OPALON 300 FM QUALITY

ROW NO.	BATCH NO.	PDR-1 LOT	BULK DENSITY GR/KC.		DRY TIME MIN: SEC			BRA PENDER		PARTICLE SIZE							EXTRUSION		HEAT STABILITY	
			RESIN	DRY BLEND	REED VIS.	REED ADD	KITCHEN MIXER	FUSION TIME	FLANGES	40	60	80	100	140	200	POW	SAF	GR/MIN. RATE	GRADE	% TRANS.
2	3629	225	436	593	2:45	2:15	4:45	4:45	15	64	29	4	0.5	0.3	T	0	47	144		39
9	3648	"	432	570	2:15	1:45	4:15	4:00	3	70	23	4	1	T	T	A	45	144	B	47
11	3650	"	447	596	2:30	2:15	5:00	4:15	5	67	25	4	2	1	T	T	49	148	B	51
1	3628	222	432	579	2:30	2:30	4:30	7:45	44	3	90	6	1	T	0	0	61	146	B	38
8	3645	"	418	554	2:30	2:15	4:30	5:00	9	1	86	9	2	2	1	T	65			51
15	3659	"	429	549	2:30	2:15	4:15	5:30	8	1	83	12	3	1	T	T	65	142	B	44
19	3665	"	-	-	-	-	4:15	5:00	8	T	85	12	2	1	T	T	64	148	B	
21	3678	"	429	540	2:00	2:00	4:45	4:45	5	T	84	9	3	3	1	T	66	132	A-R	45
5	3637	242	419	530	2:30	2:15	4:45	5:15	17	2	10	44	26	13	5	T	94	139	C-B	58
10	3649	"	428	545	3:00	3:00	5:15	7:15	10	T	19	47	22	8	3	1	81	151	B	51
14	3657	"	432	549	3:00	3:00	4:45	5:30	9	T	7	46	30	11	4	1	98	143	B-R	50
17	3664	"	417	529	2:30	2:30	4:45	5:45	14	T	4	28	39	18	9	1	110	149	C	54
23	3686	"	410	525	2:45	2:30	4:30	5:00	30	T	8	45	30	11	5	2	101	147	C	54
3	3633	236	430	548	2:45	2:45	5:30	6:30	23	1	15	47	26	8	2	1	90	148	E	53
7	3644	"	440	558	3:15	3:00	7:00	7:45	95	1	9	34	37	7	11	1	103	143	D	53
12	3654	"	420	535	2:45	2:30	4:15	4:45	10	1	7	47	28	10	6	2	101	143	B	54
20	3672	"	423	535	2:45	3:00	4:30	-	-	T	22	46	22	7	2	T	87	152	B	54
22	3681	"	405	570	3:15	3:00	5:00	5:00	26	1	3	27	38	20	10	1	112	157	D	41
4	3635	237	400	529	3:00	2:45	4:45	5:00	11	1	8	38	32	16	5	2	103	150	D-R	43
6	3642	"	377	478	2:30	2:15	4:15	3:45	2	1	5	28	32	18	15	3	120	99	E	50
13	3656	"	420	530	2:45	2:30	4:45	5:00	17	1	9	40	31	14	5	1	101	149	B	58
16	3662	"	422	530	3:00	2:45	5:00	7:00	11	T	8	40	32	11	8	2	103	143	B	54
24	3687	"	405	509	2:15	2:15	4:45	5:30	13	1	12	41	28	10	6	2	99	143	C-R	44

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DATA SHEET: EXPERIMENT #4, DIFFERENT LOTS OF PDR-1 VERSUS OPALON 300FM QUALITY
(USING EDC VINYL CARBAZIDE MONOMER)

RUN NO	BATCH NO	PDR-1 LOT	BULK DENSITY		DRY TIME			BRABENDER		PARTICLE SIZE						EXTRUSION		HEAT STABILITY		
			GR/CC RESIN	GR/CC DRY BLEND	REED VIS	MIN:SEC		FUSION TIME	VFHCHMS	% RETAINED				ON MESH		SAF	RATE GR/MIN	GRADE	% TRANS	
						REED ADJ	KITCHEN MIXER			40	60	80	100	140	200					PAN
2	3886	242	440	560	2:15	2:30	4:45	6:30	25	T	53	31	9	4	2	T	75	146	D	NAT
5	3889	"	443	555	2:45	2:45	4:30	7:15	30	T	26	44	18	12	6	1	90	146	D	TESTED
8	3891	"	446	557	3:15	3:00	4:45	6:45	47	T	13	46	25	13	5	T	98	147	D	"
3	3887	236	453	579	4:00	3:15	5:00	4:30	25	O	33	40	14	7	4	1	86	149	C	"
7	3890	"	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
1	3885	237	453	561	3:00	2:45	5:00	7:30	20	1	17	38	26	12	6	2	101	145	D	"
4	3888	"	405	531	2:30	2:45	4:45	4:15	12	O	18	45	13	12	6	1	96	148	C	"
6	3892	"	400	516	3:30	3:30	4:45	6:00	32	1	5	30	31	20	12	3	118	148	D	"

DATA SHEET: EXPERIMENT #3, DIFFERENT LOTS OF PDR-1
VERSUS OPALON 510 QUALITY

RUN NO.	BATCH NO.	PDR-1 LOT	PARTICLE SIZE								BULK MELT		
			% RETAINED ON MESH								DENSITY	INDEX	
			40	60	80	100	140	200	PAN	SAF	GR/CC.	GR/MIN	
1	1254	2/3 242 + 1/3 225	T	5	20	18	22	24	10		.605	.220	..
2	1260	1/3 242 + 2/3 225	2	7	18	21	24	22	8		.614	.171	
4	1268	2/3 242 + 1/3 222	T	7	20	19	28	18	8		.604	.216	
5	1277	1/3 242 + 2/3 222	T	10	20	17	22	24	8		.623	.284	
3	1264	225	-	0	6	18	20	24	19	146	.601	.277	
11	1304	"	T	3	12	18	24	29	19	175	.613	.205	
18	1329	"	T	3	12	17	19	31	19	173	.618	-	
6	1284	222	T	3	11	14	18	36	19	175	.611	.280	
13	1315	"	T	3	11	10	20	32	24	185	.613	.323	
15	1321	"	T	1	10	13	19	31	26	188	.623	.278	
10	1299	242	T	T	3	4	11	36	45	229	.647	.201	
19	1330	"	T	2	8	10	16	30	35	205	.637	.229	
8	1291	236	T	2	11	15	20	34	21	184	.651	.196	
14	1318	"	1	3	10	12	14	35	28	195	.638	.240	
17	1326	"	0	2	9	12	18	31	27	188	.681	.317	

DATA SHEET: EXPERIMENT #5, DIFFERENT LOTS OF PDR-1
VERSUS OPALON 510 QUALITY

RUN NO.	BATCH NO.	PDR-1 LOT	PARTICLE SIZE								SAF	MELT INDEX
			40	%	60	80	100	140	200	PAN		GR/Min
				60 MESH								
1	1414	249	T	6	10	12	19	37	15	169	.370	
2	1422	247	1	8	13	15	18	32	14	162	.286	
3	1430	244	1	2	14	13	21	31	17	168	.412	
4	1437	251	T	5	10	8	14	32	30	193	.331	
Control	1415	H.B. 59	1	7	19	19	20	26	8	143	.318	

DATA SHEET: EXPERIMENT #7, PDR-1 LOTS VERSUS OPEN 300 FH QUALITY

RUN NO	BATCH NO	PDR-1 LOT	BULK DENSITY GR/L		REES VISC	DRY TIME MIN: SEC		KIRKEN MIXER	BRA BEINDER		PARTICLE SIZE % RETAINED ON MESH										SAF	EXTRUSION		SPEC. VISC.	HEAT STABILITY	
			RESIN	DRY WELD		REED ART	FUSION TIME		FISHPOX	40	60	80	100	140	200	PAN	RATE GR/MIN	GRADE	% TRANS							
6	5036	230	.447	.573	-	1:30	2:00	4:00	-	-	1	72	17	6	1	1	T	67	154	G	-	-				
7	5038	"	.350	.455	-	-	-	5:15	9:00	65	3	81	14	1	1	T	T	63	135	D	.548	-				
23	5070	"	.438	.560	-	-	-	4:00	7:00	12	T	84	12	2	T	T	T	62	145	D	-	-				
26	5076	"	.454	.578	-	3:00	2:45	4:45	6:00	3	3	92	14	1	T	T	T	61	144	C	-	-				
1	5023	240	.425	.550	-	-	-	4:00	10:00	6	T	32	43	18	4	1	1	82	150	C	.558	-				
11	5048	"	.433	.565	-	2:30	3:45	4:30	10:15	9	T	38	39	18	5	2	T	84	157	B	.556	-				
13	5051	"	.430	.570	-	2:30	3:15	4:45	10:45	8	T	66	24	7	2	1	1	73	154	C	-	-				
24	5072	"	-	-	-	-	-	5:00	10:00	5	1	56	37	8	T	T	T	72	-	-	-	-				
29	5081	"	.472	.610	-	-	-	4:30	11:00	5	T	68	24	7	1	1	T	70	146	D	-	-				
2	5026	277	.410	.540	-	-	-	4:45	5:00	10	2	5	28	44	16	5	1	165	144	ROUGH	-	-				
10	5046	"	.382	.502	-	3:15	2:30	4:15	4:45	10	3	7	22	32	23	10	2	114	149	ROUGH	-	-				
21	5066	"	.405	.485	-	3:15	4:00	5:15	10:00	3	T	2	4	8	20	35	30	185	151	A	.565	-				
25	5075	"	.400	.536	-	-	-	4:00	-	-	1	6	10	38	26	16	2	123	140	C	-	-				
36	5137	"	.363	.555	-	-	-	4:30	7:15	7	T	1	10	27	30	23	9	125	148	B	-	-				
4	5030	278	.453	.600	-	-	-	4:15	5:00	8	2	4	16	33	27	14	4	125	145	ROUGH	.588	-				
8	5042	"	.387	.500	-	2:45	2:45	4:30	5:00	7	2	6	22	40	19	9	3	114	147	ROUGH	.562	-				
22	5069	"	.378	.506	-	-	-	4:15	6:15	13	T	3	14	30	30	18	4	131	147	D	-	-				
32	5131	"	.444	.564	-	3:15	3:15	4:30	-	-	T	7	38	35	13	3	1	96	-	-	-	-				
34	5134	"	.430	.525	-	-	-	5:00	7:45	4	1	2	17	40	28	11	3	123	152	D	-	-				
5	5032	287	.460	.576	-	2:30	2:45	4:15	19:30	7	T	13	36	33	13	3	1	96	150	B	.556	-				
9	5044	"	.460	.570	-	2:15	3:15	3:45	11:45	2	1	37	38	17	5	1	T	79	95	C	.559	-				
14	5053	"	-	-	-	-	-	4:15	13:00	3	T	25	48	20	4	T	1	82	-	-	-	-				
19	5064	"	.455	.585	-	-	-	5:15	20+	5	1	20	44	28	7	2	T	89	151	C	-	-				
31	5132	"	.398	.520	-	2:15	2:30	5:00	6:00	7	1	8	36	34	16	6	1	105	150	D	-	-				
35	5135	"	.430	.530	-	2:30	2:45	4:15	20+	-	1	4	31	36	17	8	2	111	150	C	-	-				
3	5028	242	.353	.473	-	2:15	2:30	4:30	-	-	T	2	17	40	28	11	3	124	-	-	.561	-				
20	5065	"	.425	.545	-	-	-	4:45	9:00	6	1	16	44	28	6	6	T	94	146	C	-	-				

DATA SHEET: EXPERIMENT #8; PDR-1 LOTS AND PDR-1 CONCENTRATION VERSUS OPALON 300FM QUALITY.

RUN NO	BATCH NO.	PDR-1 LOT	PDR-1 CONC.	BULK DENSITY GR/CC		DRY TIME MIN: SEC			BRA BENDER		SCREEN ANALYSIS % ON MESH										EXTRUSION			HEAT STABILITY % TAIL
				RESIN	DRY BEHT	REEL VLS	REEL ADT	K.M.	FUSION TIME	JFE	40	60	80	100	140	200	240	SAF	RATE	GRADE	JFE			
11	5418	277	.06	461	.565	-	-	4:15	20+	-	T	1	8	22	43	22	3	141	150	-	-	49		
14	5420	"	"	470	.583	2:45	3:30	4:45	10:45	18	T	2	16	30	26	21	4	133	145	E	4	57		
21	5445	"	"	461	.565	-	-	4:00	16:15	-	T	7	29	31	26	3	6	116	-	-	-	51		
1	5392	287	"	500	.608	2:30	2:45	6:15	19:15	36	T	2	23	28	33	13	T	114	146	E	3	58		
10	5413	"	"	498	.604	5:30	4:30	5:30	20+	-	T	1	16	32	38	13	2	130	147	E	-	58		
22	5426	"	"	515	.620	5:30	5:30	7:45	20+	-	T	1	19	29	37	15	1	129	150	E	-	64		
7	5406	277	.12	411	.501	-	-	-	8:15	22	T	T	T	4	30	49	16	134	149	C	-	31		
19	5440	"	"	383	.489	2:45	2:45	4:15	7:30	8	T	T	3	13	35	28	23	178	149	B	-	42		
9	5412	287	"	REJECTED		-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-		
12	5419	"	"	445	.580	2:20	3:15	5:15	9:00	15	T	10	22	29	21	7	2	109	147	D	2	45		
15	5425	"	"	428	.535	-	-	5:00	10:00	-12	T	1	4	21	32	31	8	3	118	148	C	3	53	
4	5399	277	.18	374	.489	3:15	2:20	4:30	6:15	8	T	1	11	20	28	29	10	152	147	B	4	40		
6	5405	"	"	409	.498	8:30	8:30	18+	11:30	30	T	T	T	4	26	55	15	186	149	B	4	42		
18	5439	"	"	400	.497	6:15	6:15	18+	6:45	22	T	1	3	9	30	27	28	178	149	B	-	37		
3	5398	287	"	430	.550	2:45	3:45	5:15	10:45	2	T	21	37	19	14	5	2	96	149	B	-	86		
5	5404	"	"	411	.501	-	-	5:15	9:00	3	T	25	41	19	12	4	1	94	+	-	-	40		
8	5411	"	"	403	.535	2:30	2:45	4:30	6:15	22	T	1	3	22	30	29	12	3	122	146	C	2	-	
13	5434	277	.24	355	.435	4:00	-	4:00	6:15	-	T	1	3	11	30	33	22	177	108	D	-	10		
16	5432	"	"	335	.448	3:15	4:00	5:15	6:15	26	T	2	14	19	28	26	12	153	144	B	3	10		
17	5438	"	"	361	.480	2:45	2:45	4:15	6:00	7	T	T	6	15	32	29	16	163	140	C	-	12		
2	5394	287	"	431	.519	2:30	2:45	6:30	8:45	64	T	10	33	24	20	11	3	112	152	D	-	18		
20	5427	"	"	411	.535	-	-	4:00	6:30	-	T	3	23	29	30	11	5	126	149	B	-	19		

TABLE I

SUMMARY OF PDR-1 LOTS TESTED

Group	PDR-1 Lot	Specific Viscosity			% Conversion	Spec. Conductivity Micromhos	Coke Bottle Acceptance Test	
		A	B	C			Copolymer	Homopolymer
A	222-I,a	7.79	2.35	0.60	75.1	1855	NG	OK
A	222-I,b	6.90	-	-	-	-	-	-
A	222-II	6.10	-	-	-	-	-	-
A	222-III	?	-	-	-	-	-	-
A	225	3.86	1.21	0.28	70.1	2740	OK	-
A	236	6.79	9.97	0.93	82.3	1510	OK	-
A	237	8.21	3.73	1.25	77.0	1410	OK	-
A	242	8.48	3.26	1.11	78.4	1560	OK	-
B	230	4.19	1.25	-	67.0	2355	NG	OK
B	240	4.30	1.36	-	69.0	1992	NG	OK
B	277	8.90	4.36	-	83.7	887	OK	-
B	278	8.98	4.77	-	85.4	922	-	-
B	287	5.75	2.52	-	79.9	1283	OK	-
C	244	7.7	-	-	84.1	1142	NG	OK
C	247	9.0	-	-	86.5	985	NG	OK
C	249	9.2	-	-	83	1012	NG	OK
C	251	6.0	-	-	81.5	1397	NG	OK
MB	35	8.87	-	-	-	-	-	-
MB	38	6.56	-	-	-	-	-	-
MB	47	5.45	-	-	-	-	-	-

Notes:

Specific Viscosity= PDR-1 lots in Group A and B were tested by the East Control Lab for specific viscosity using three procedures.

A = 1% PDR-1 water solution at 25°C, using PDR-1 that was purified by dissolving in acetone and precipitating with benzene. Present S.D.P. test.

B = 1% PDR-1 water solution at 25°C, using unpurified PDR-1, i.e. material as received and used in the plant.

C = 0.1% PDR-1 water solution at 60°C, using "unpurified" PDR-1.

Three different samples of PDR-1 Lot 222 would not dissolve completely in acetone, showing a variable amount of insolubles (polyvinyl acetate). There was insufficient precipitated PDR-1 - Lot 222 III - to complete the specific viscosity test.

TAB 2.

EFFECT OF PDR-1 CONCENTRATION CHANGES ON OPALON 300 RESIN IN 92 BLDG.

PDR-1 CONC.	RUN NO.	SCREEN ANALYSIS, % ON MESH							SAF	BULK DENSITY, GR./CC.		REED DRY TIME, MIN.: SCC		EXTRUSION		
		40	60	80	100	140	200	PAN		RESIN	DRY BLEND	VISUAL	ADJUSTED	RATE	GRADE	FISHEYES
.02	18	Very coarse							-	-	-	-	-	-	-	-
.03	14	T	8	31	27	24	6	3	110	.576	.671	9:45	9:45	152	V	39
.04	16	T	16	41	23	17	2	1	96	.575	.675	8:15	6:15	150	IV	22
.06	8	T	8	37	32	18	T	3	98	.520	.635	7:15	8:00	160	V	43
.06	11	T	5	37	33	24	3	T	105	.545	.640	9:15	6:45	151	III	13
.07	10	T	4	36	38	21	2	T	102	.530	.628	8:30	9:00	156	V	34
.07	13	1	10	38	29	22	1	T	99	-	-	-	-	-	-	-
.08	7	1	12	36	31	18	2	T	97	.505	.650	8:15	8:00	159	V	40
.09	4	0	2	25	36	32	4	1	119	-	-	-	-	-	-	-
.12	15	1	59	26	9	4	1	T	-	.505	.620	4:15	3:30	150	I	5
.16	6	1	12	42	28	14	3	T	95	-	-	-	-	-	-	-
.17	1	T	3	19	28	35	14	2	107	-	-	-	-	-	-	-
.18	12	3	56	18	10	6	5	2	84	.435	.554	4:15	4:30	155	III	12
.20	17	1	7	31	29	26	5	1	107	.450	.580	6:45	4:30	152	III	15
.21	5	1	30	32	14	12	10	T	95	-	-	-	-	-	-	-
.25	9	2	22	34	20	18	6	T	98	.415	.553	4:00	4:45	154	II	13
.28	2	1	5	19	24	29	20	2	126	-	-	-	-	-	-	-
.30	3	1	11	18	15	22	18	14	145	-	-	-	-	-	-	-

EFFECT OF PDR-1 CONCENTRATION AND pH ADJUSTMENT ON OPALON 300 RESIN IN 92 BLDG.

pH	PDR-1 CONC.	AMOUNT OF ACID(A) OR BASE(3)ADDED	SCREEN ANALYSIS, % on MESH 40 60 80 100 140 200 PAN	RESIN BULK DENSITY, GR./CC	REED DRY TIME MIN: SEC	EXTRUSION GRADE F.E	V.R.	HEAT STABILITY % TRANS.
1.61	.070	21# 5 oz.	A	T 52 24 8 4 11 T	.533	5:15	IV 57	57 -
1.67	.185	18#	A	38 60 2 T T T	.472	4:45	IV 52	52 -
1.88	.128	18#	A	1 93 3 T 1 1 T	.500	8:30	V 57	64 0
2.21	.060	3# 12 oz.	A	T 46 36 12 4 1 T	.544	6:30	IV 27	107 -
2.29	.096	4# 5 oz.	A	1 84 12 2 1 T T	.493	8:45	V 54	80 5
2.30	.163	2# 8 oz.	A	T 45 36 11 5 2 T	.470	12:45	IV 35	94 -
2.37	.225	1# 4 oz.	A	8 71 14 5 4 T T	.465	4:45	IV 27	107 -
2.71	.124	0		T 3 24 22 38 12 1	.465	5:45	III 30	89 -
2.76	.141	0		T 25 44 22 10 T T	.455	10:00	IV 25	101 40
-	.205	0		T 5 21 19 21 10 22	.413	8:00	II 13	89 36
2.74	.260	1# 8 oz.	B	1 17 32 19 18 2 10	.405	3:30	II 8	73 -
3.36	.114	1#	B	T 1 14 24 38 18 5	.455	4:45	II 12	53 -
3.51	.228	3# 2 oz.	B	T 8 26 17 23 15 11	.420	8:45	III 13	18 -
3.67	.155	1# 15 oz.	B	9 6 6 10 23 25 19	.435	4:30	II 14	24 -
3.94	.103	2# 6 oz.	B	T 3 10 18 30 2 37	.455	4:15	III 14	12 42
4.63	.180	4# 8 oz.	B	1 16 37 20 16 15 3	.430	4:15	III 16	9 -

A = 98% Reagent Sulfuric Acid

B = 50% Technical Sodium Hydroxide

TABLE 4.

EFFECT OF LAURIC ACID AND MALEIC ANHYDRIDE ON OPALON 300FM PARTICLE SIZE

RUN NO.	AMOUNT OF LAURIC ACID (L) AND MALEIC ANHYDRIDE(M) ADDED GRAMS.		PERCENT*	PARTICLE SIZE, % ON MESH							SAF
				40	60	80	100	140	200	PAN	
3	0	(Control)	-	4	36	29	16	6	2	1	85
7	0	(L)	-	1	23	47	20	5	2	T	85
8	100	(L)	1.0	1	35	42	17	5	1	1	84
1	200	(L)	2.0	T	30	50	14	5	3	T	85
5	200	(L)	2.0	1	16	46	25	8	3	1	92
10	200	(L)	2.0	1	20	50	20	6	2	T	85
2	300	(L)	3.0	T	30	48	15	6	1	T	82
4	400	(L)	4.0	T	9	42	28	16	4	1	100
9	500	(L)	5.0	1	47	37	11	3	1	T	75
11	600	(L)	6.0	1	20	48	22	5	1	T	81
6	900	(L)	9.0	1	11	42	29	13	4	2	101
2	0	(Control)	-	T	4	32	35	19	6	2	107
4	0	(Control)	-	T	1	20	39	28	9	3	120
1	315	(M)	4.2	T	13	46	27	9	2	1	91
3	630	(M)	8.4	T	9	43	32	11	3	1	96
5	945	(M)	12.6	T	3	30	37	22	5	1	106

* Weight percent for Lauric Acid was computed using the weight of L_2O_2 as the basis, while for Maleic Anhydride, PDR-1 was the basis.

FIG. 1. EFFECT OF TEN LOTS OF PDR-1 ON
 OPALON 300 PM PARTICLE SIZE
 EXPERIMENT 2 of 7.

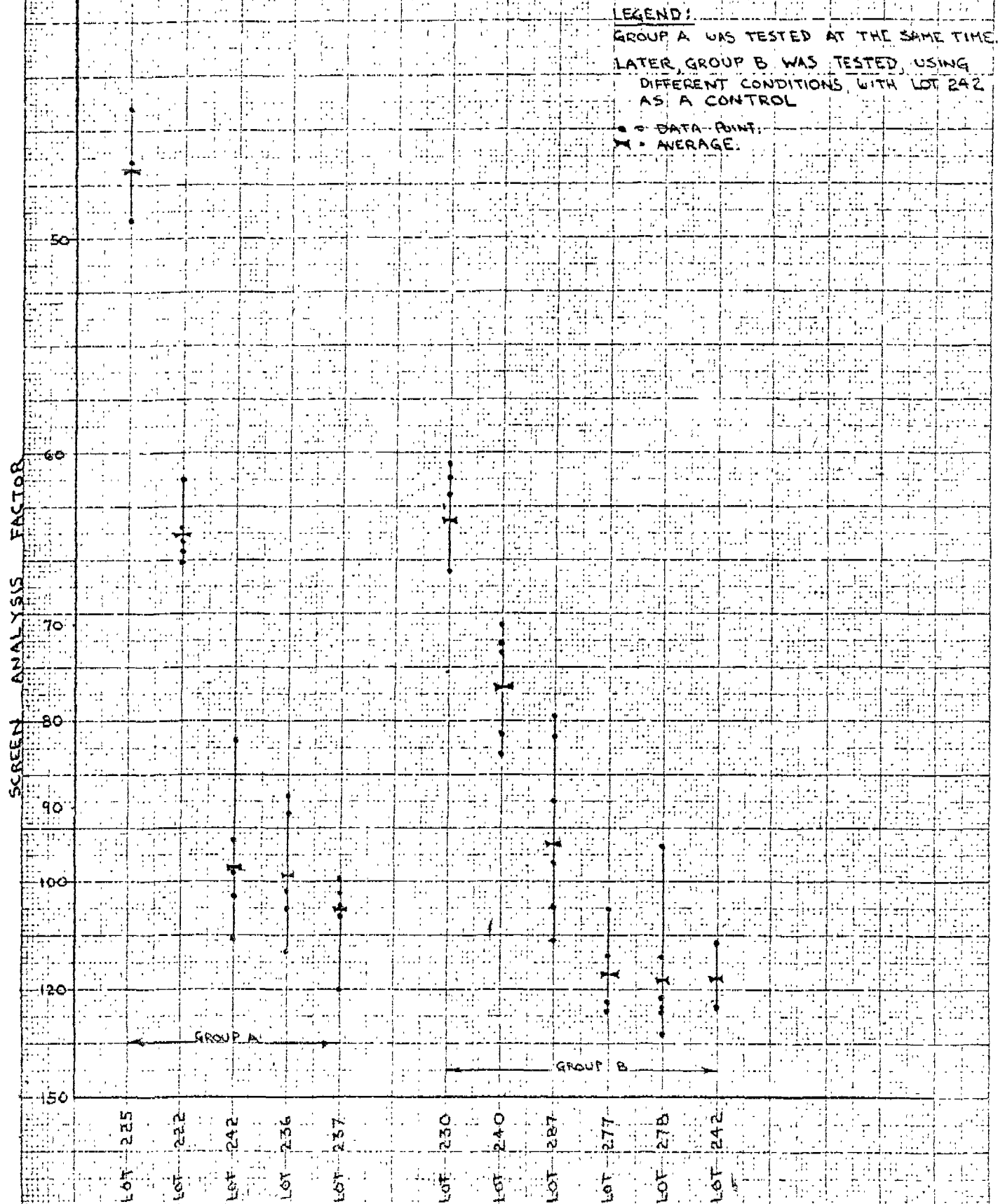


FIG. 2. EFFECT OF TEN LOTS OF PDR-1 ON
OPALON 300 FM. KITCHEN MIXER DRY TIME.
EXPERIMENT 247

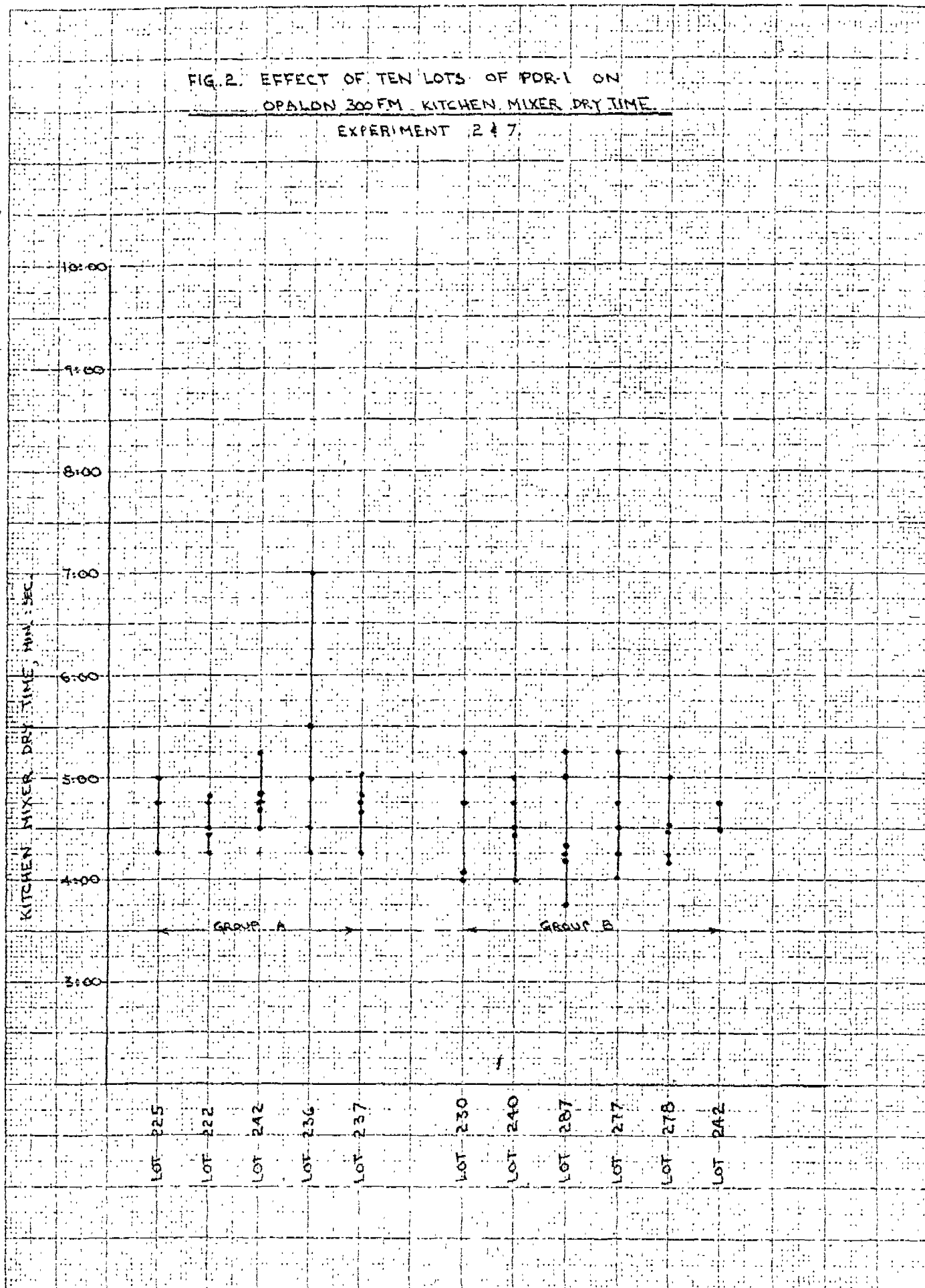


FIG. 3. EFFECT OF TEN LOTS OF PDR-1 ON
OPALON 300 FM BULK DENSITY
EXPERIMENT 2 & 7.

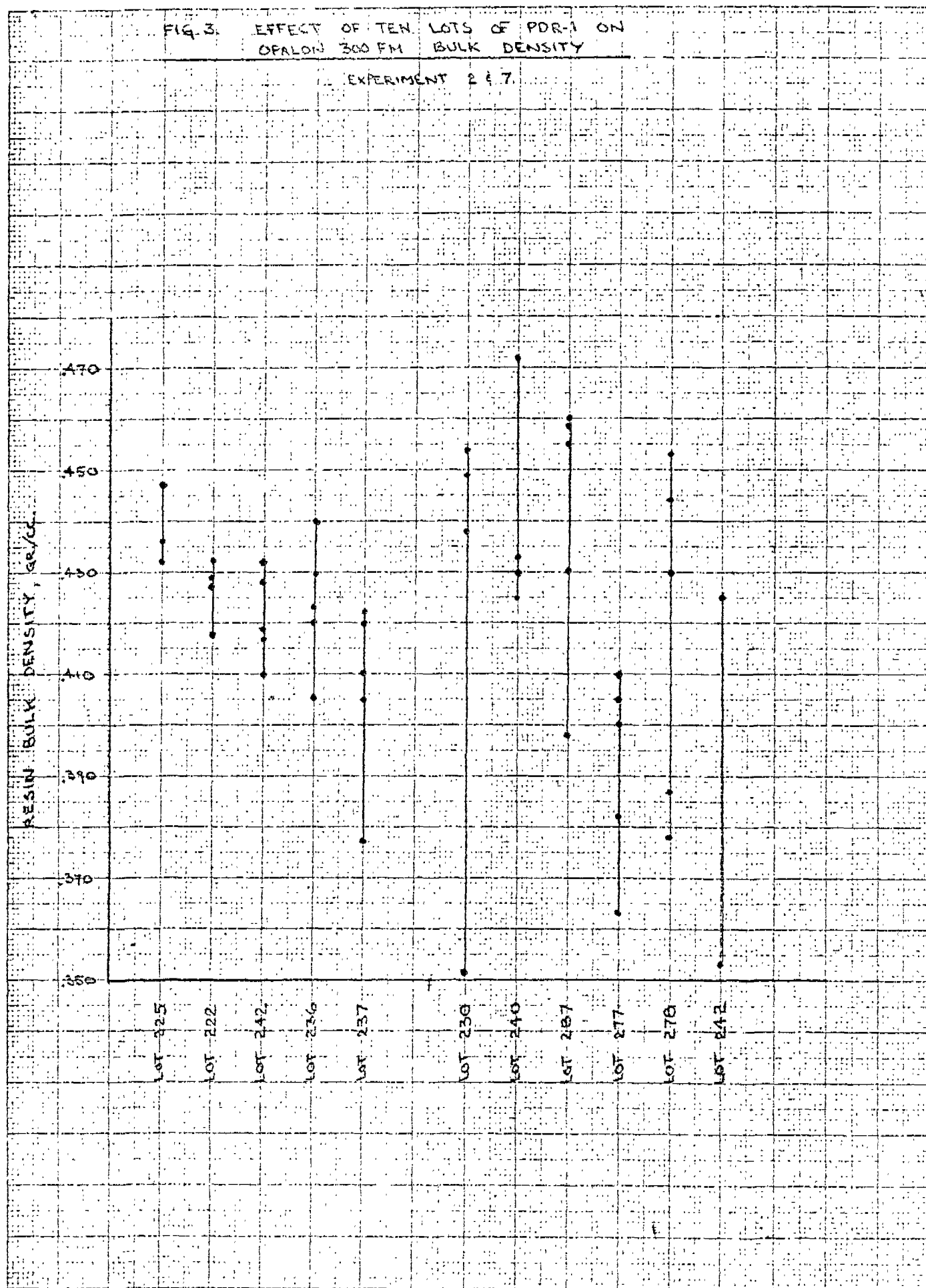


FIG. 4. EFFECT OF TEN LOTS OF POR-1 ON
OPALON 300 FM HEAT STABILITY

EXPERIMENT 2 & 7

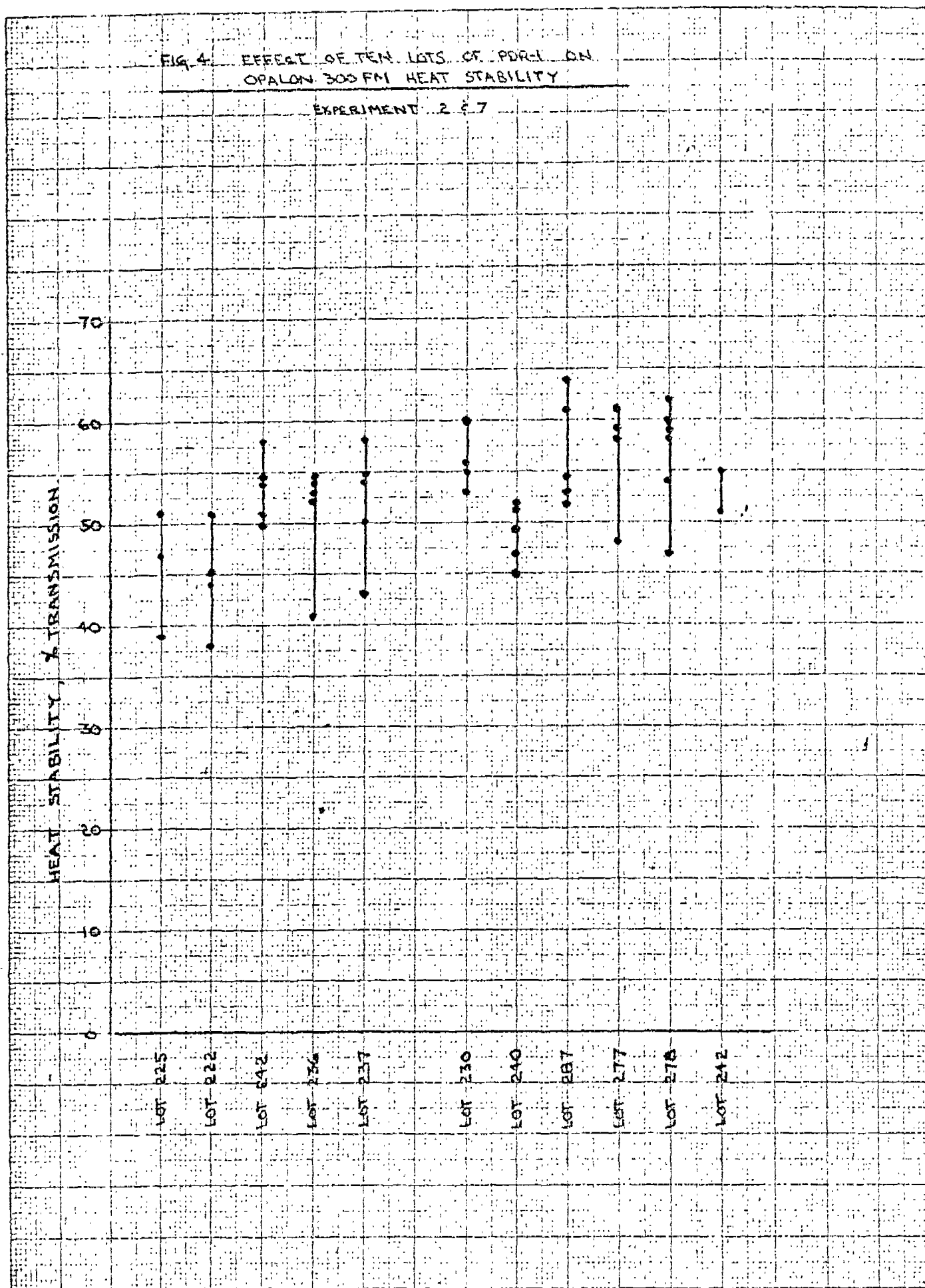


FIG. 5. EFFECT OF TEN LOTS OF PDR-1 ON
OPALON 300 FM EXTRUSION GRADE

EXPERIMENT 2 & 7

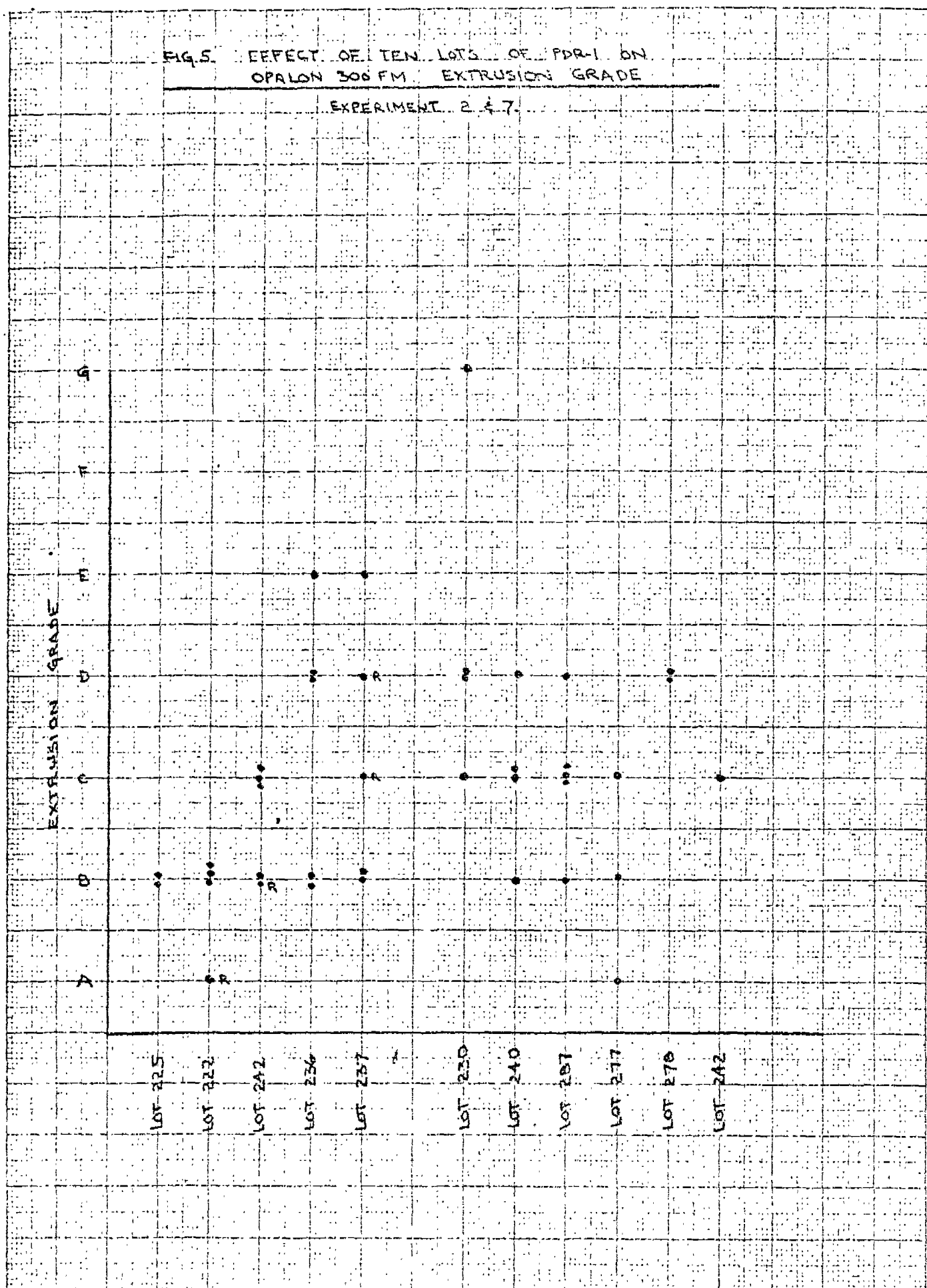


FIG. 6. EFFECT OF TEN LOTS OF PDR-1 ON OPALON 300FM
BRABENDER FUSION TIME

EXPERIMENT 2-7

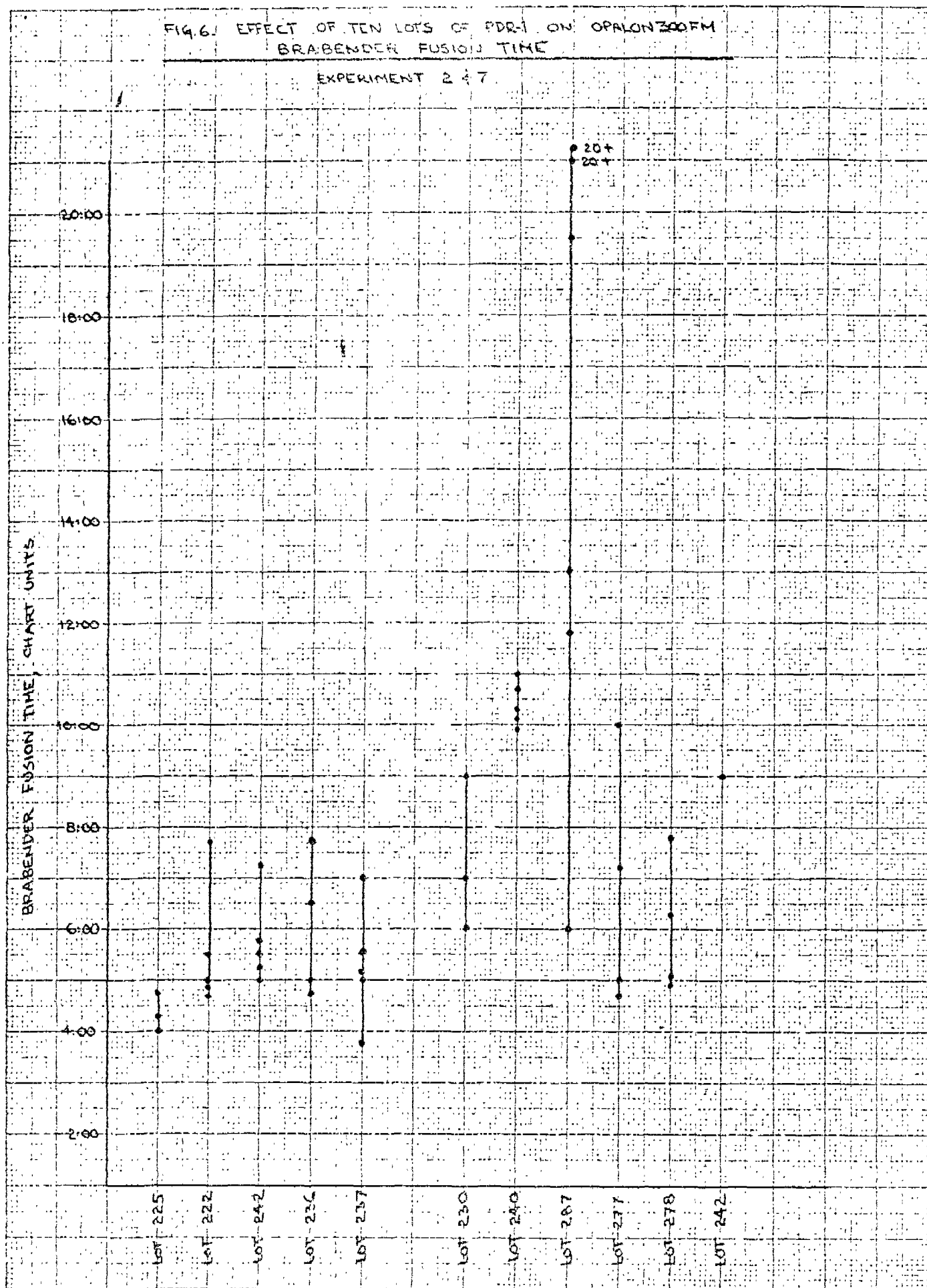


FIG. 7. EFFECT OF TEN LOTS OF PDR-1 ON OPALON 300 FM FISHEYES

EXPERIMENT 2:7

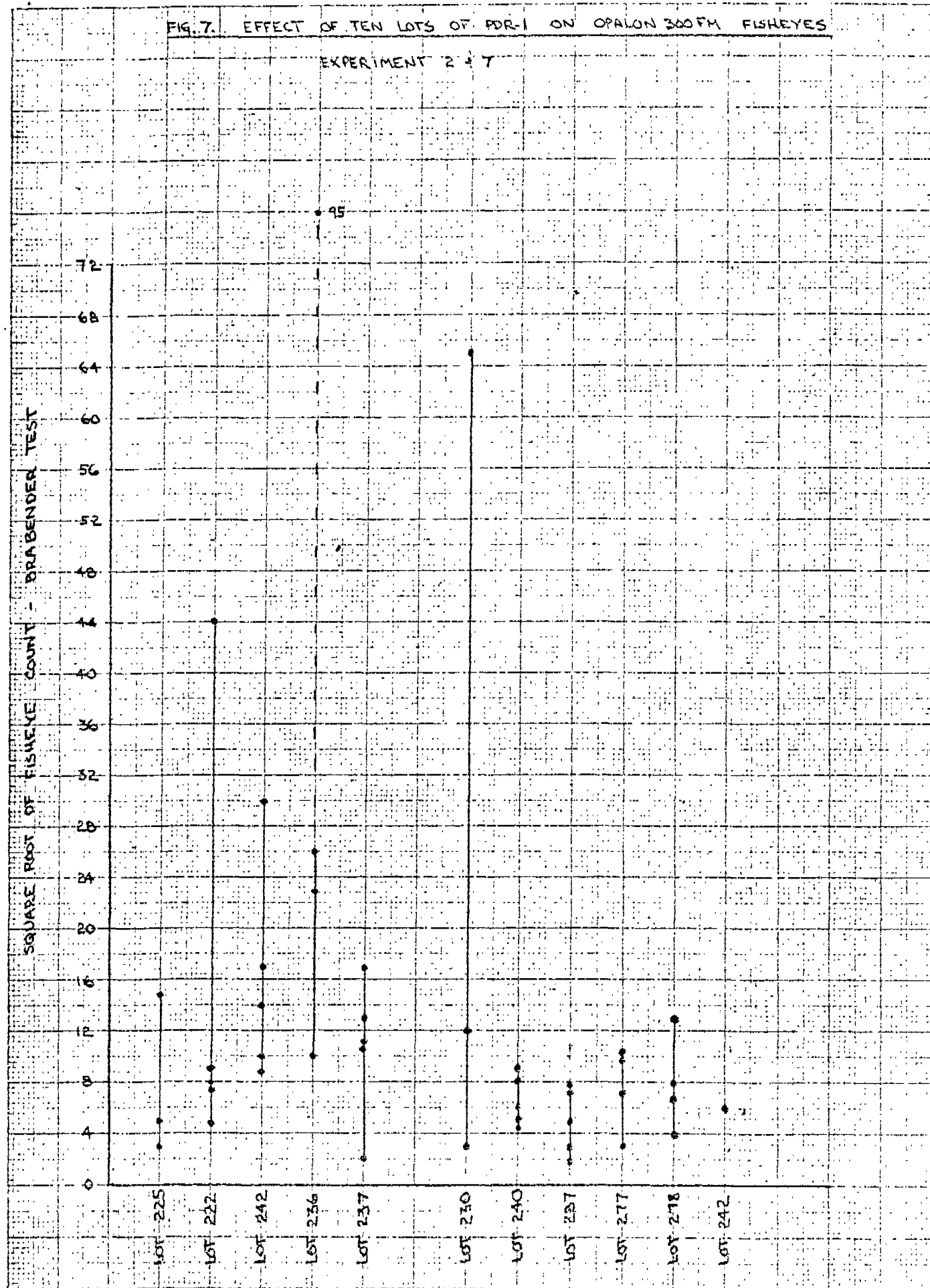


FIG. 8. EFFECT OF FIVE LOTS OF PDRT ON
OPALON 510 PARTICLE SIZE

EXPERIMENT 3

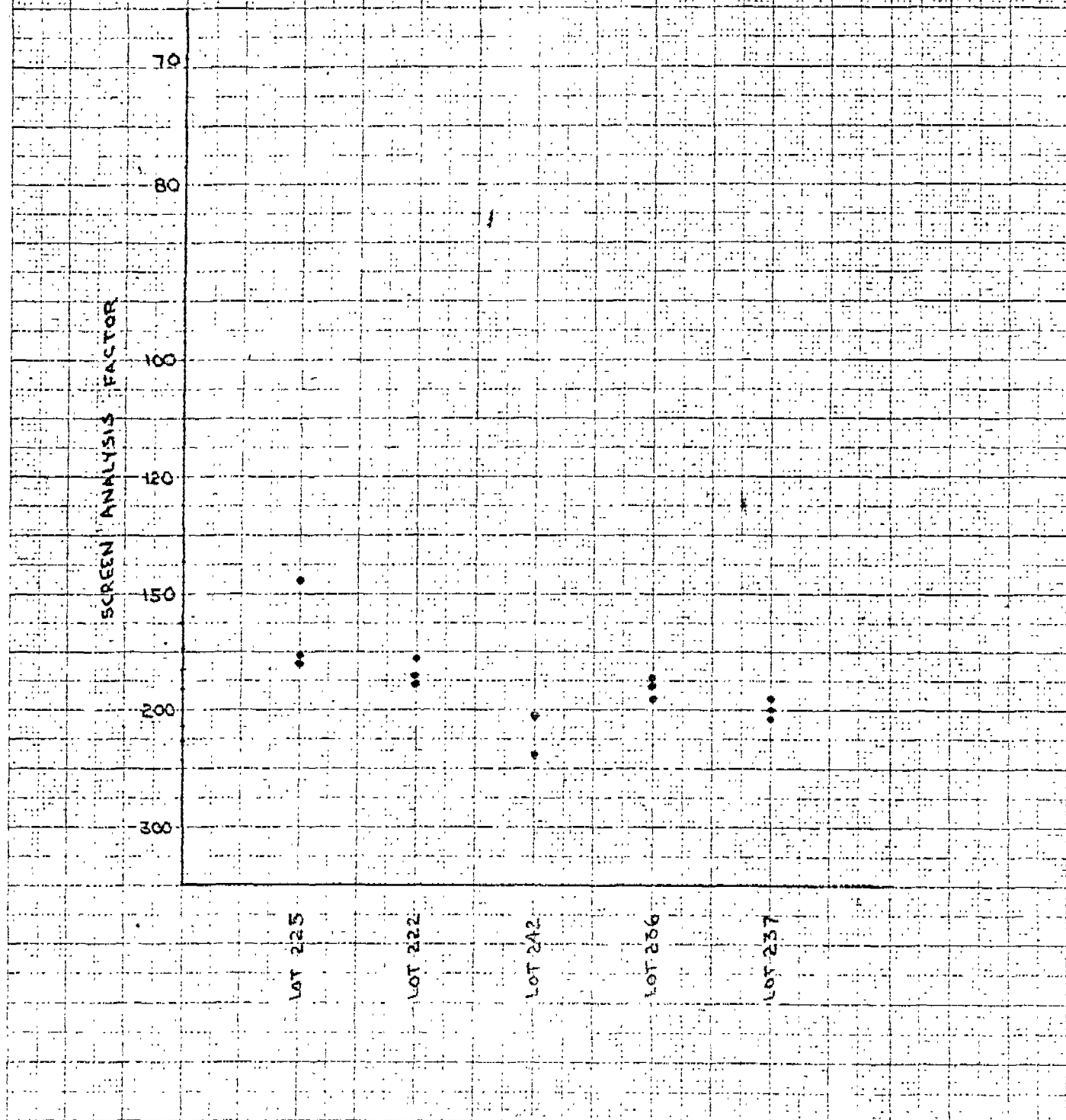


FIG. 9 EFFECT OF FOUR LOTS OF COKE BOTTLE REJECTED PDR-1
ON OPALON 510 PARTICLE SIZE

EXPERIMENT 3

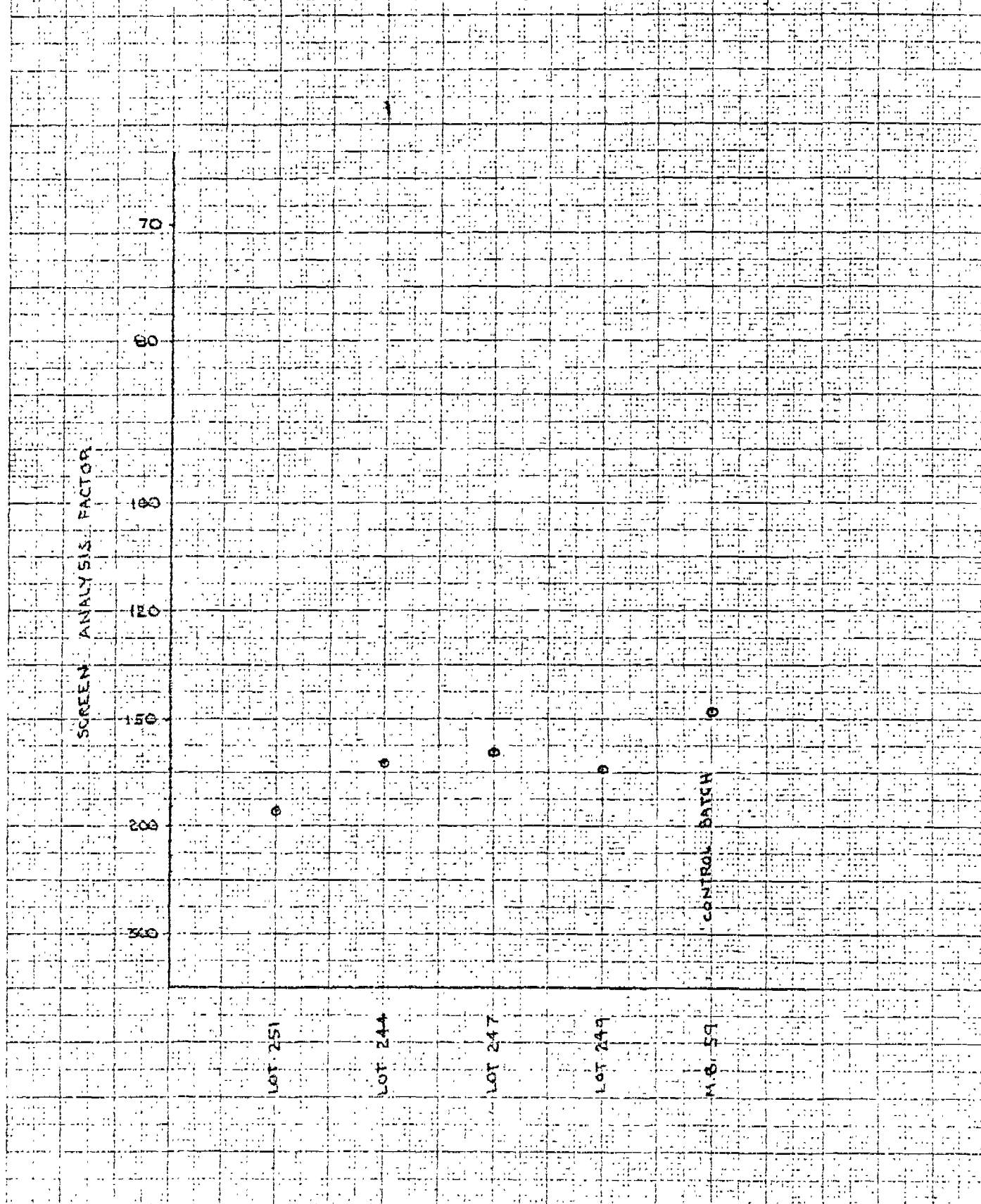


FIG. 10. EFFECT OF FIVE LOTS OF PDR-1 ON OPALON 510
BULK DENSITY AND MELT INDEX.

EXPERIMENT 3

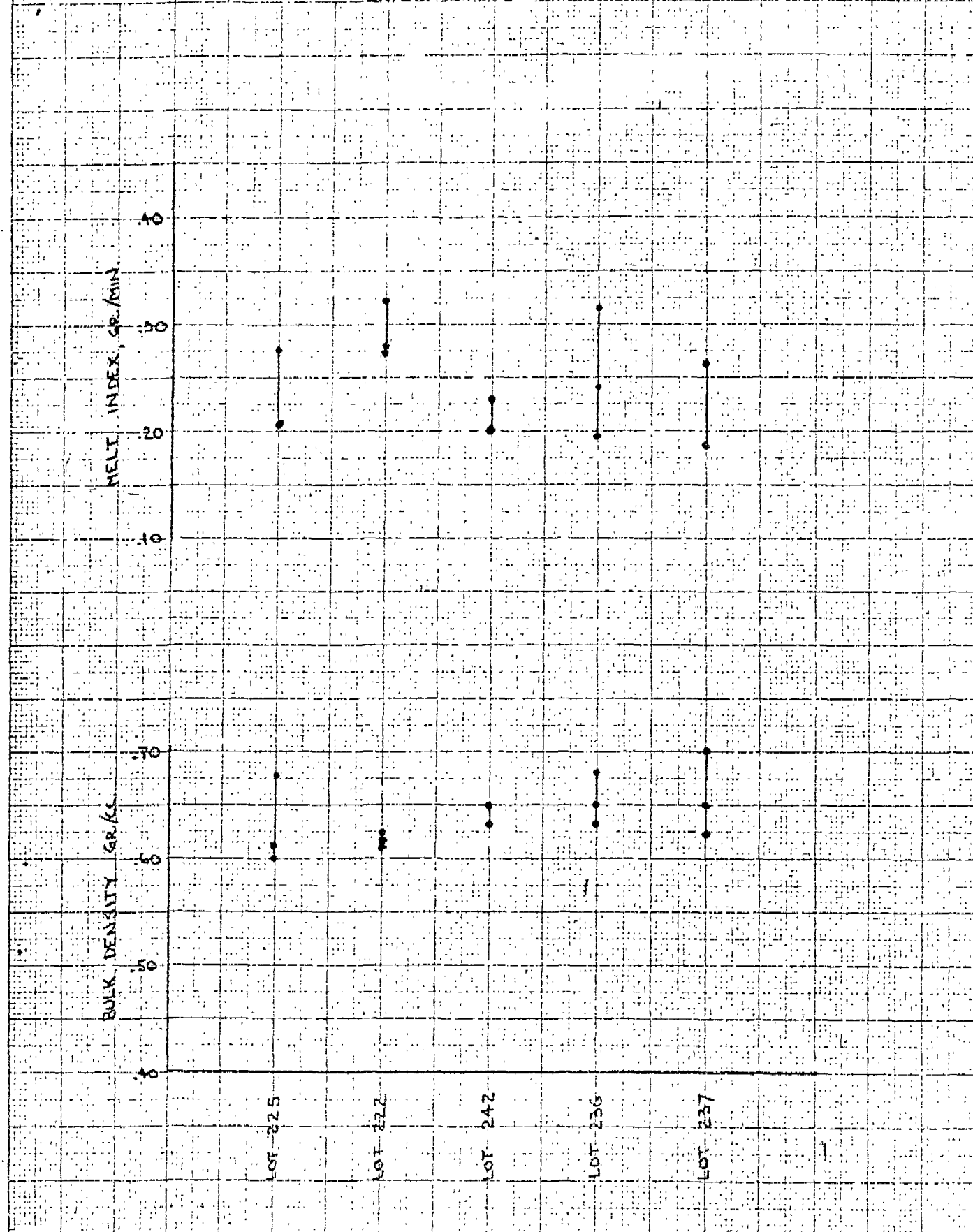


FIG. II. EFFECT OF PRECIPITATED PDR-1 SPECIFIC VISCOSITY ON OPALON 300 FM PARTICLE SIZE

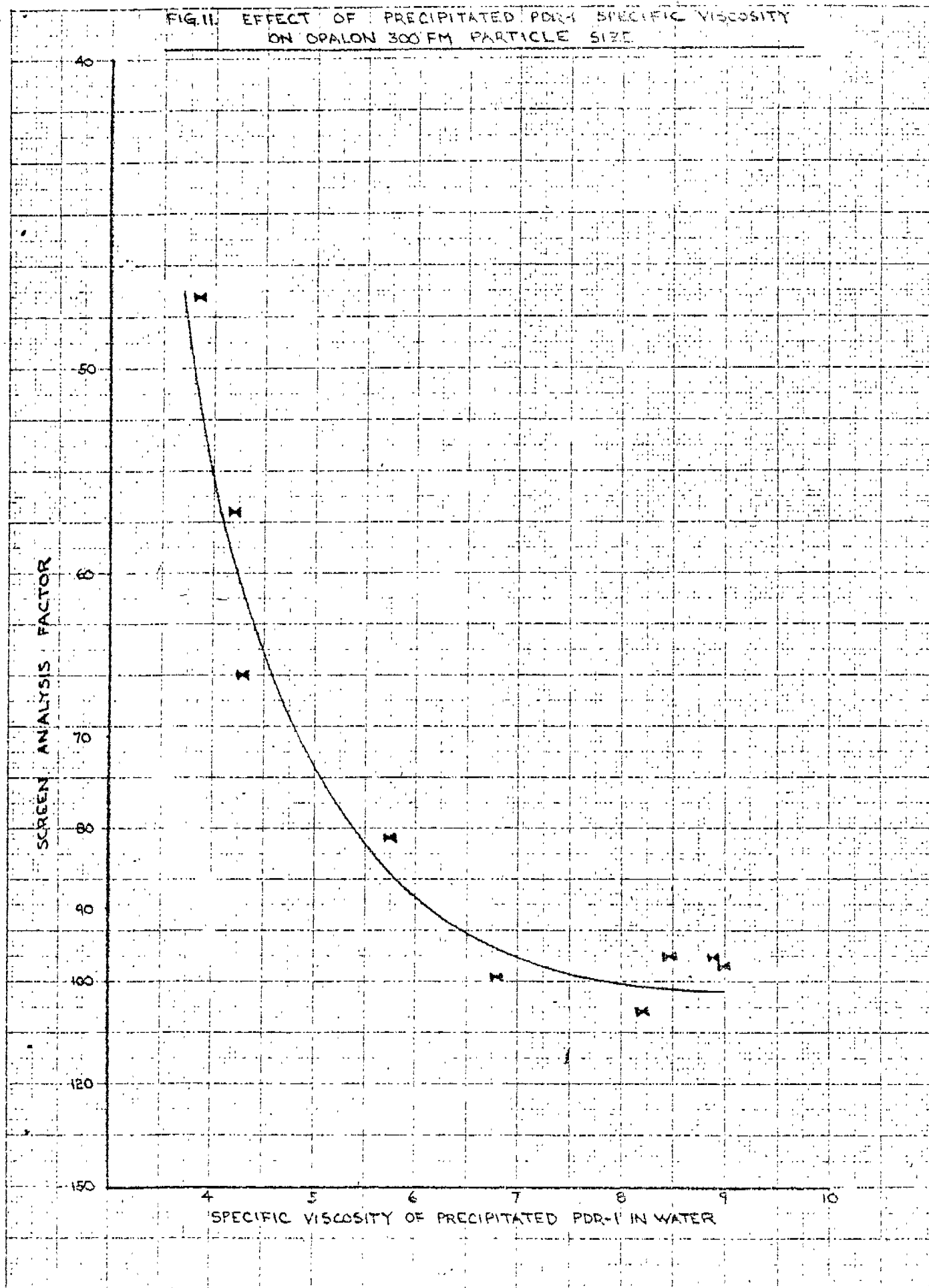


FIG. II. 10 X 10 TO THE CM. 359-14.
 R. S. V. NEUTRON-SCATTERING CO. U.S.A.

12.12 10 X 10 TO THE CM. 359-14
K. P. L. & C. S. P. C. L.

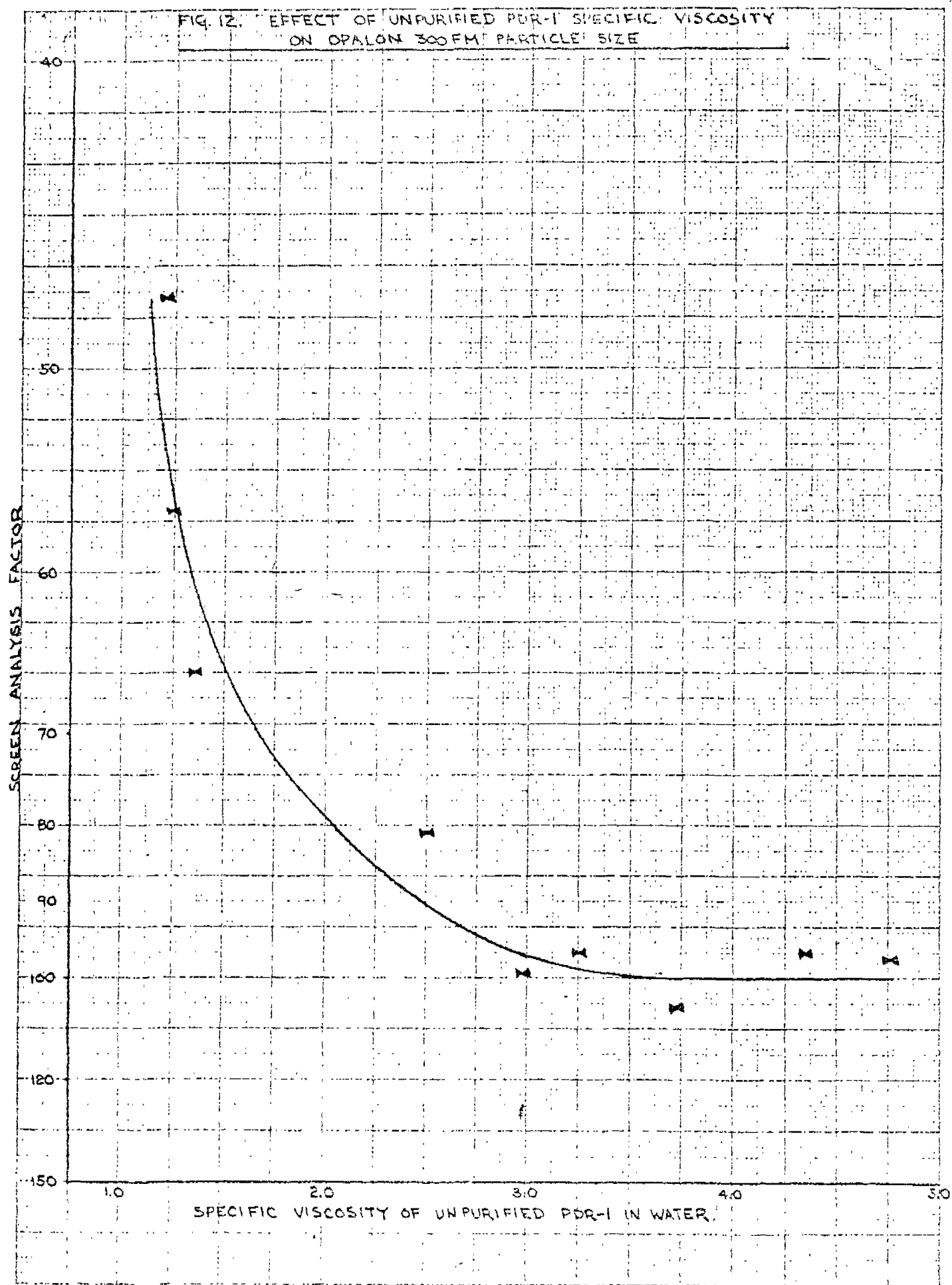
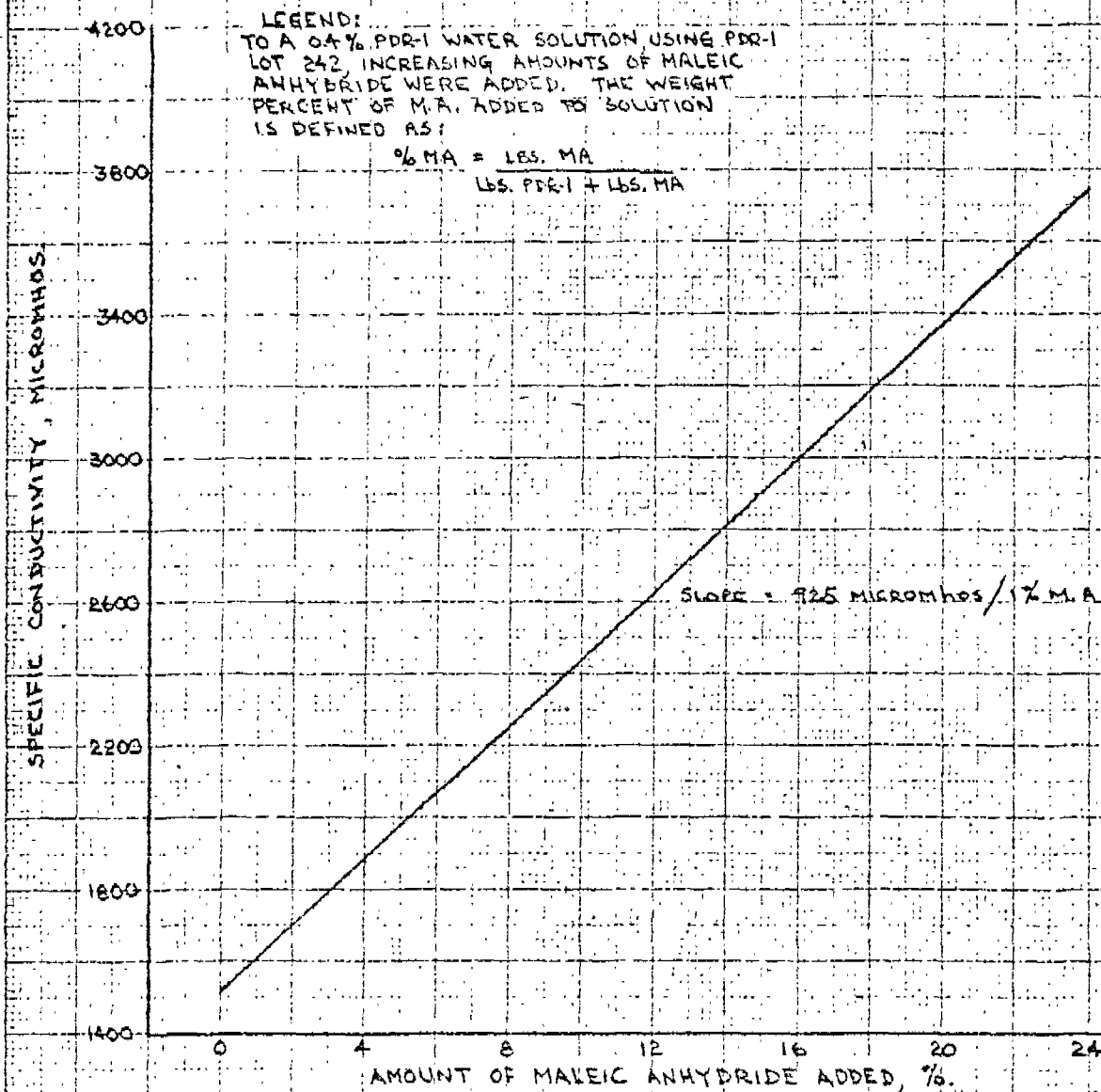
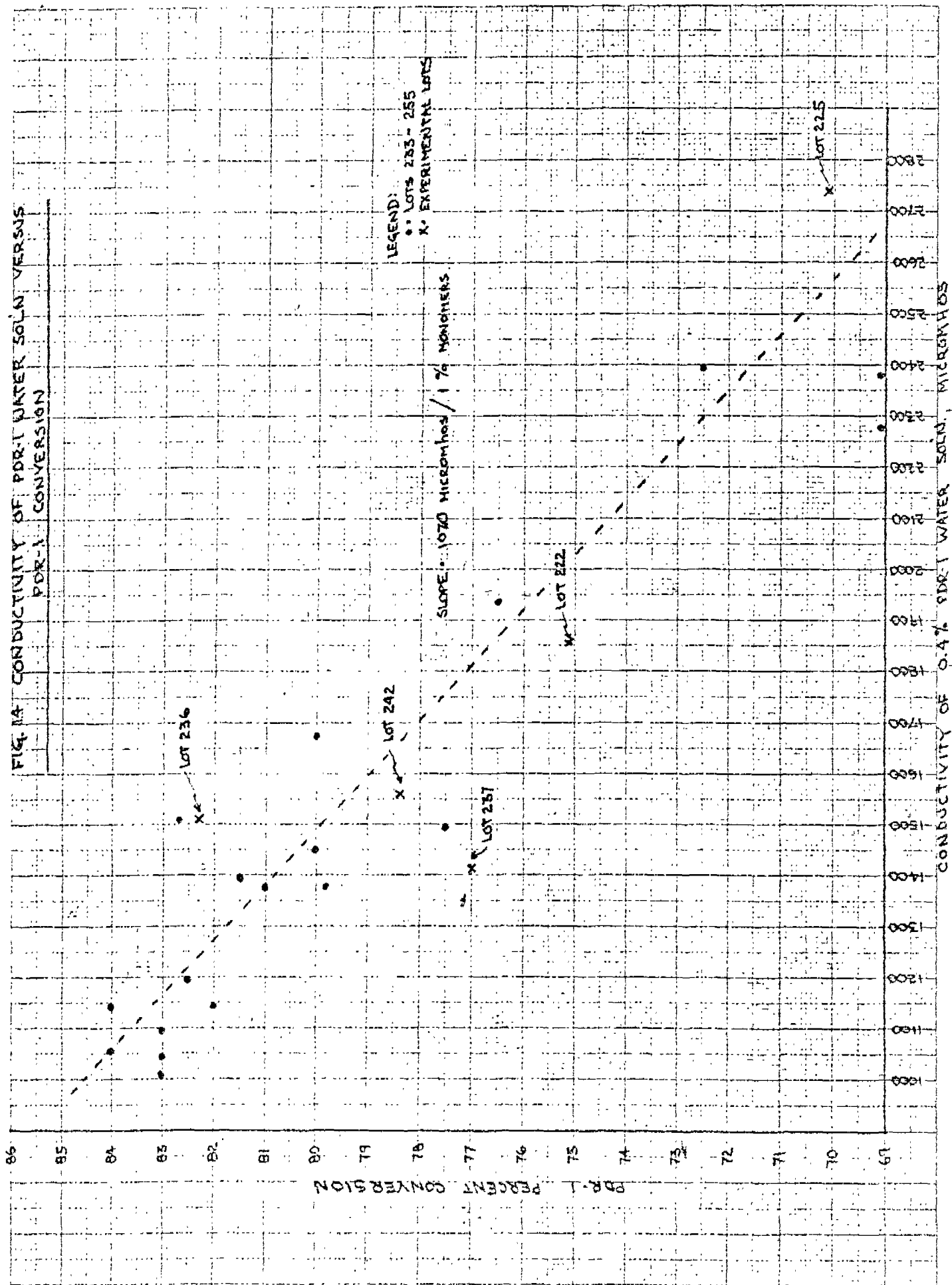
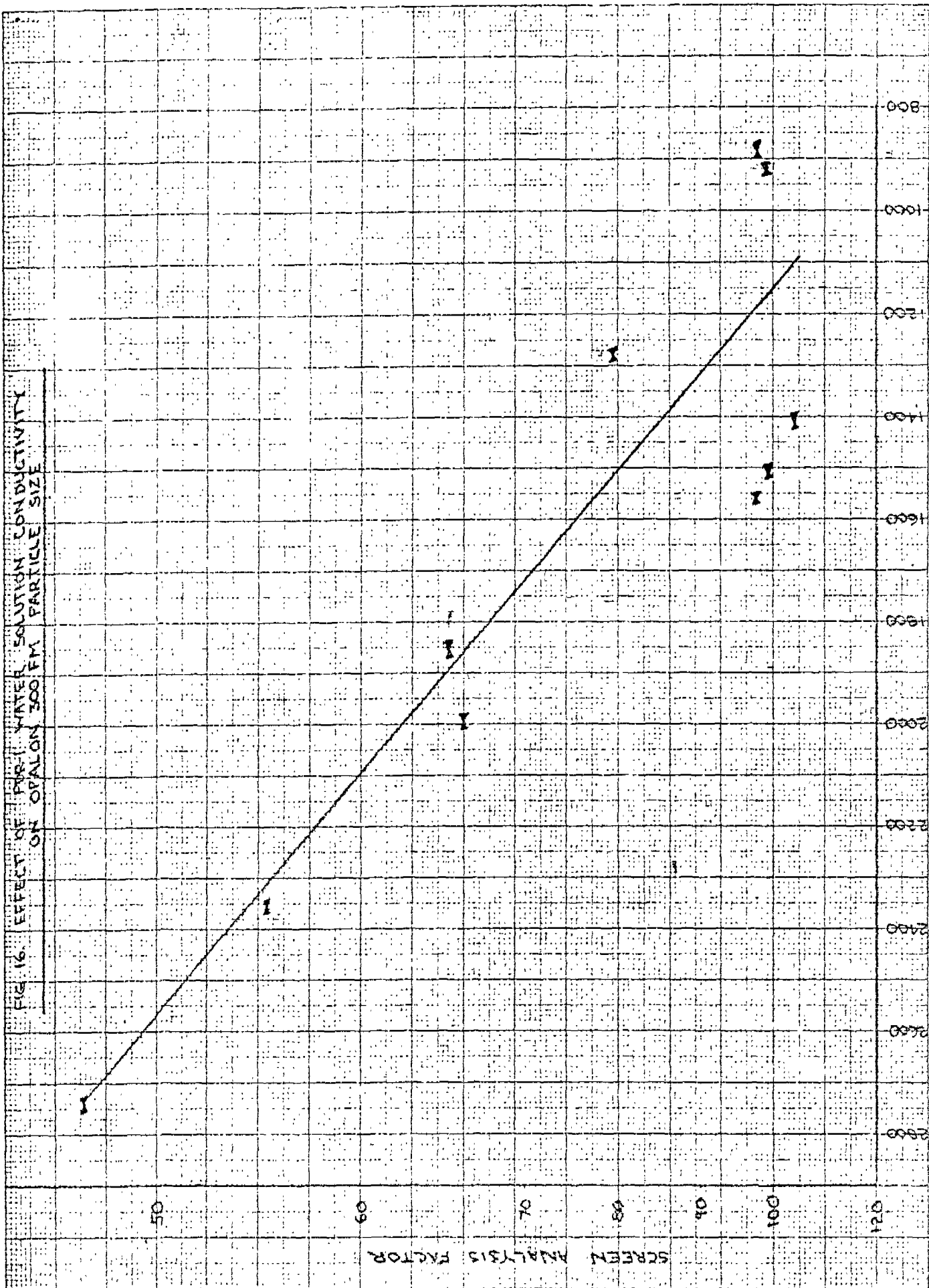


FIG 13. THE EFFECT OF ADDING MALEIC ANHYDRIDE TO PDR-1 WATER SOLUTION ON THE SPECIFIC CONDUCTIVITY OF THE SOLUTION.







CONDUCTIVITY OF 0.4% PDR-1 WATER SOLUTION.

10 X 10 TO THE CM. 359.14
1.00 1.00 1.00 1.00 1.00 1.00

FIG. 17. EFFECT OF PDR-1 SPECIFIC VISCOSITY AND SPECIFIC CONDUCTIVITY ON RESIN HEAT STABILITY

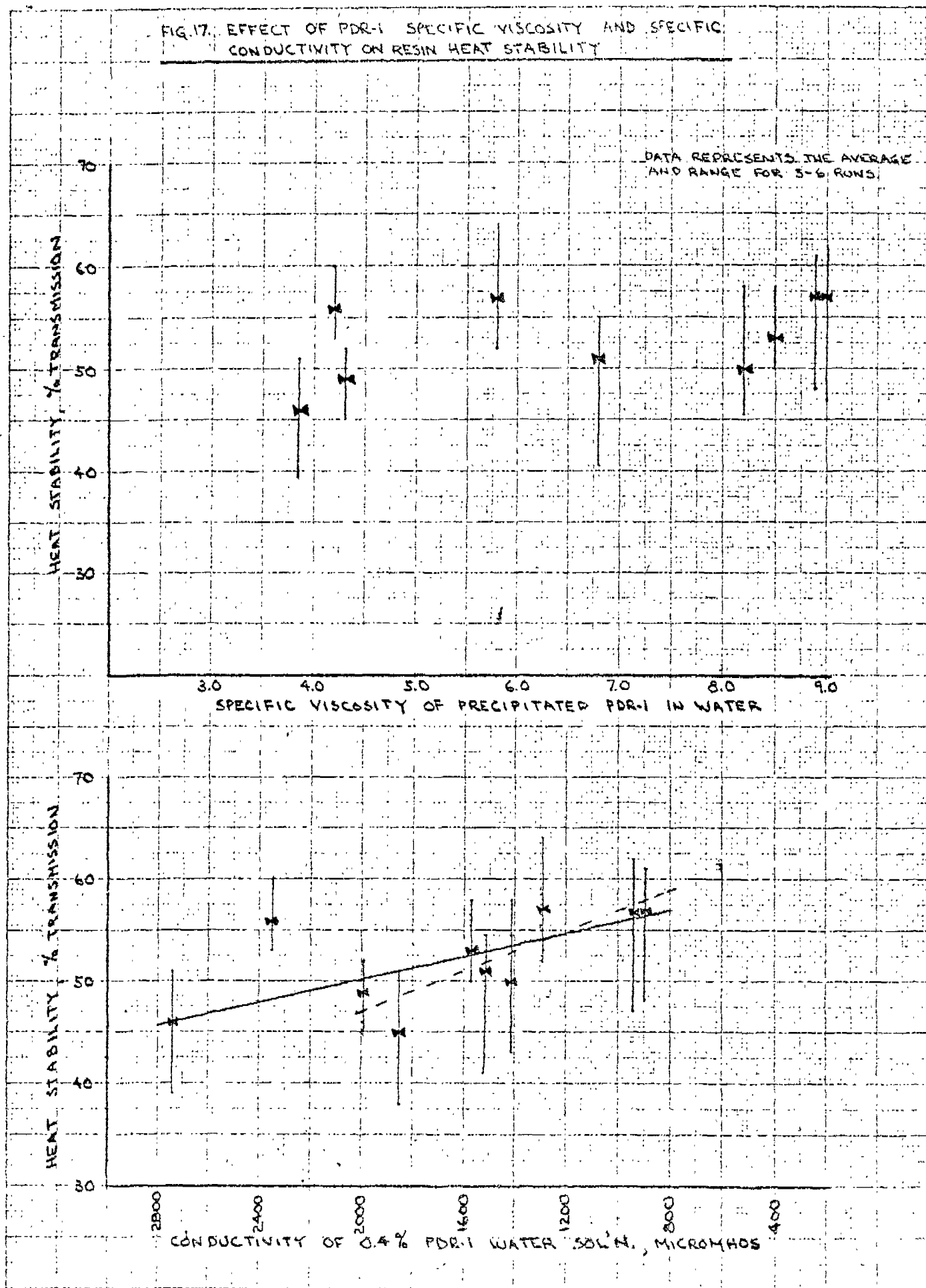


FIG. 18. EFFECT OF PDR-1 CONCENTRATION ON
OPALON 300 PARTICLE SIZE DISTRIBUTION

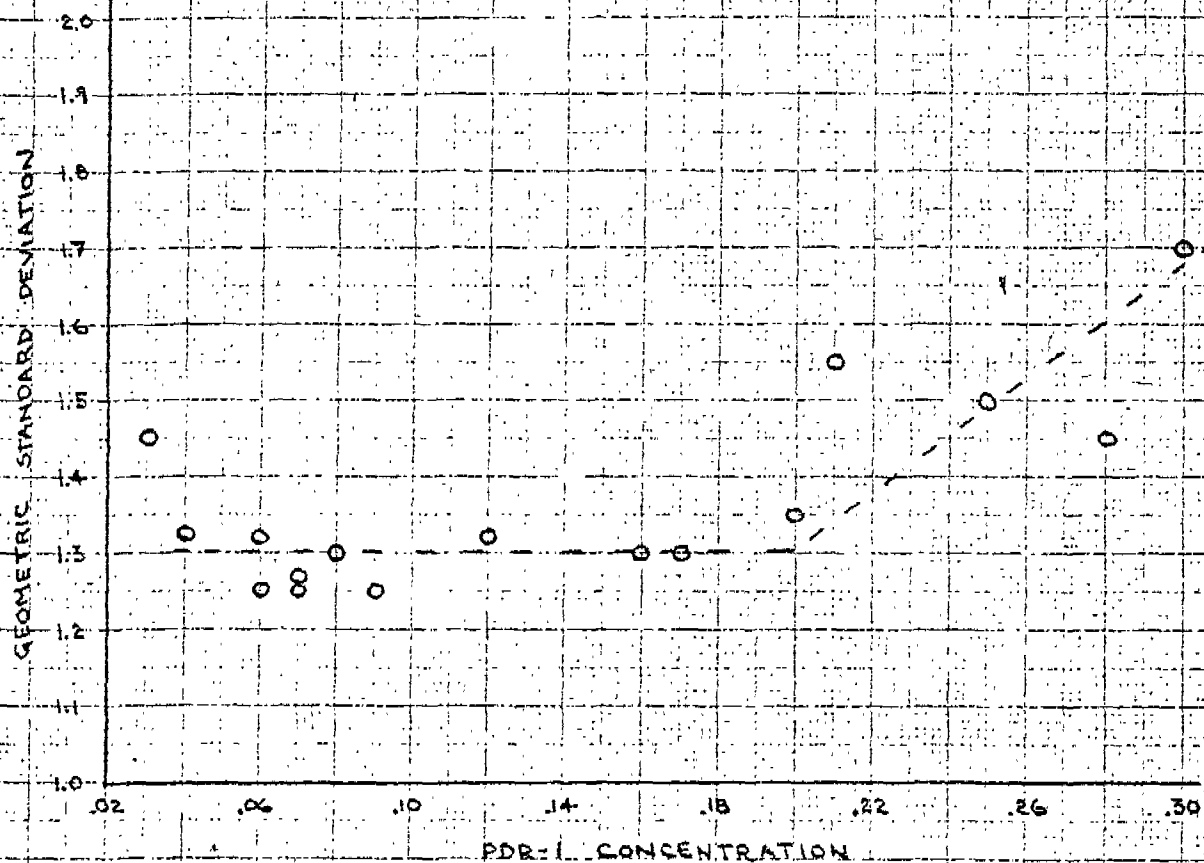


FIG 19. EFFECT OF PDR-1 CONCENTRATION ON
OPALON 300 BULK DENSITY.

EXPERIMENT A.

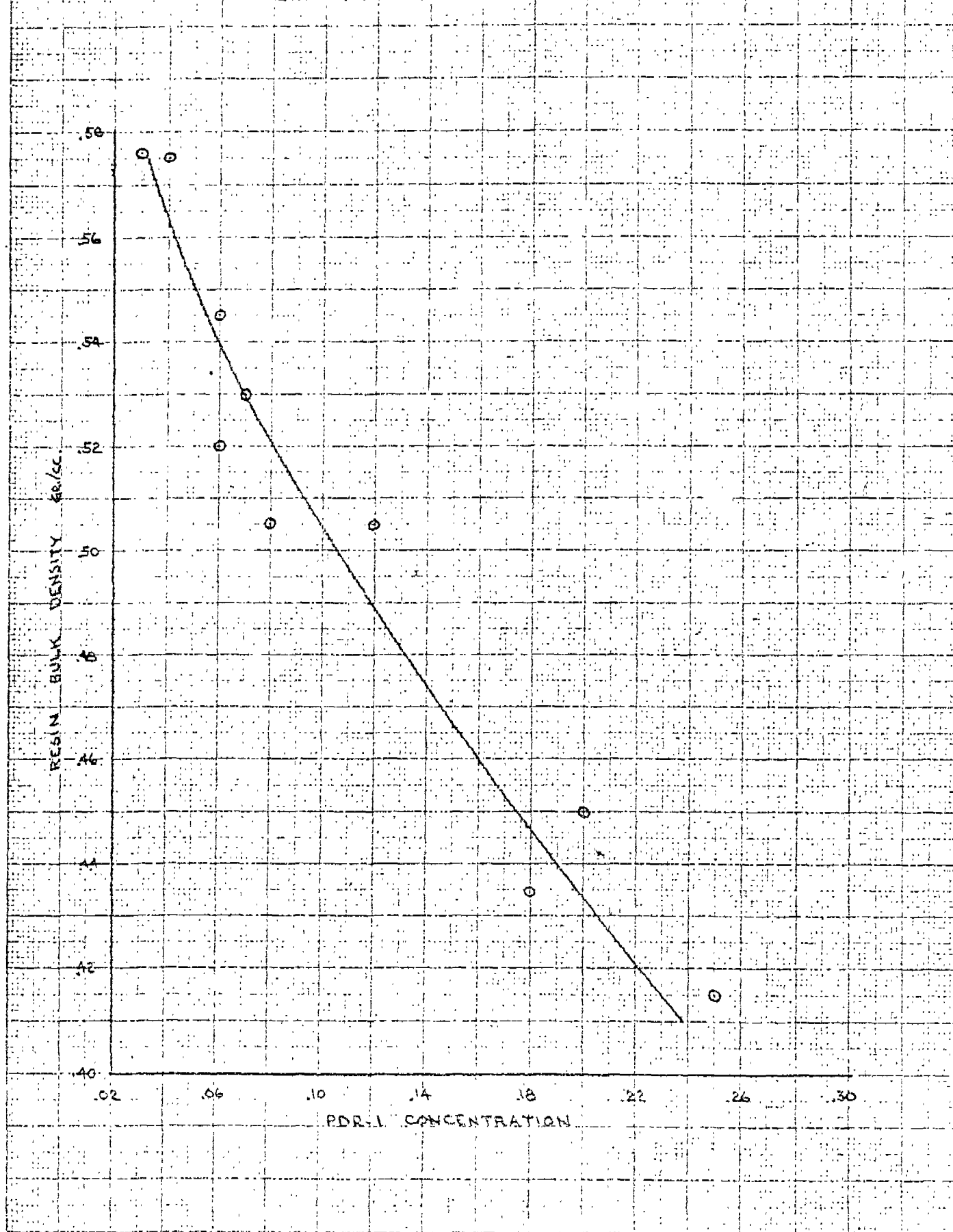


FIG. 20 EFFECT OF PDR-1 CONCENTRATION ON
OPALON 300 DRY TIME.

EXPERIMENT - A.

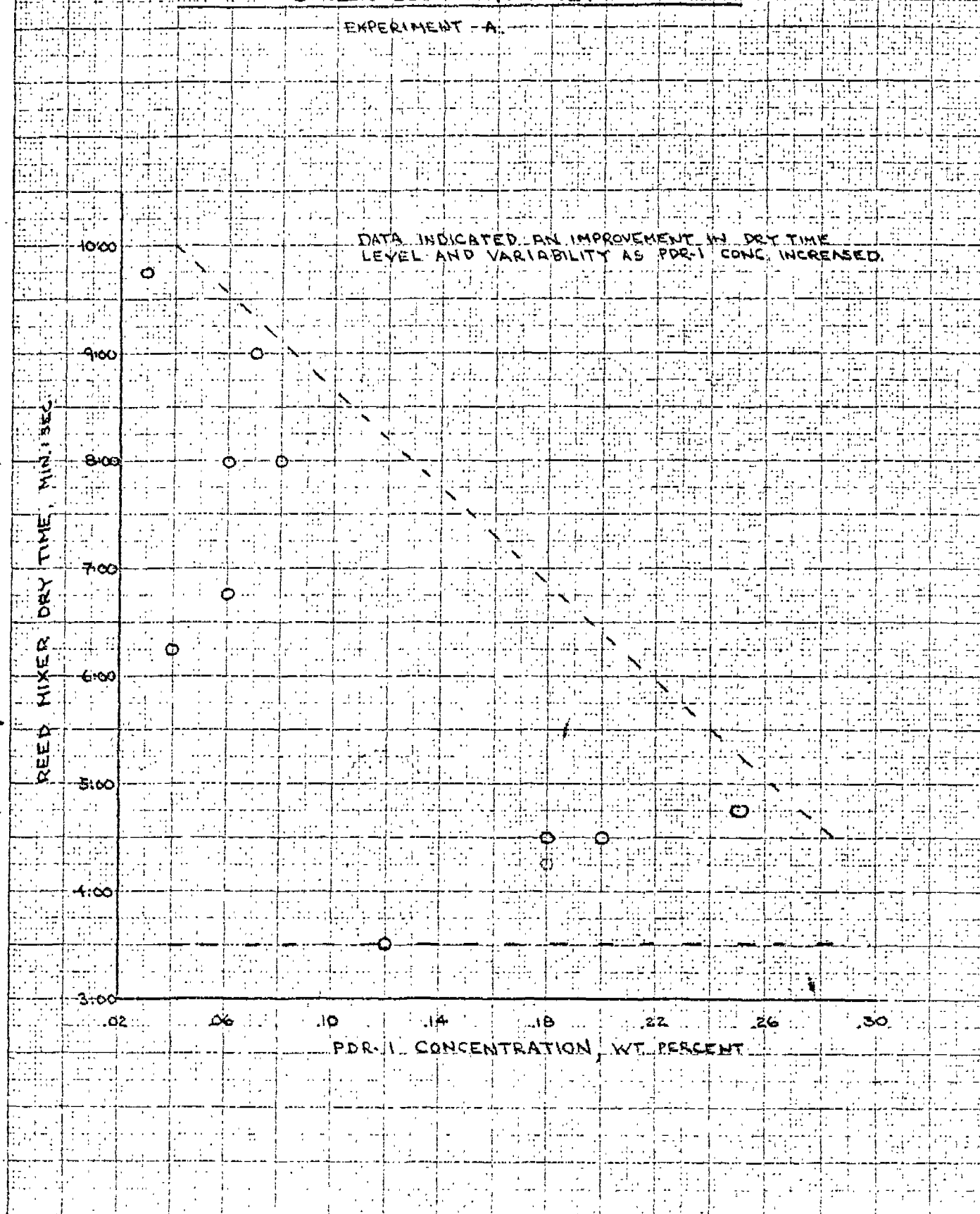


FIG. 21. EFFECT OF PDR-1 CONCENTRATION ON APRILON 300
 EXTRUSION FISKEYE COUNT.

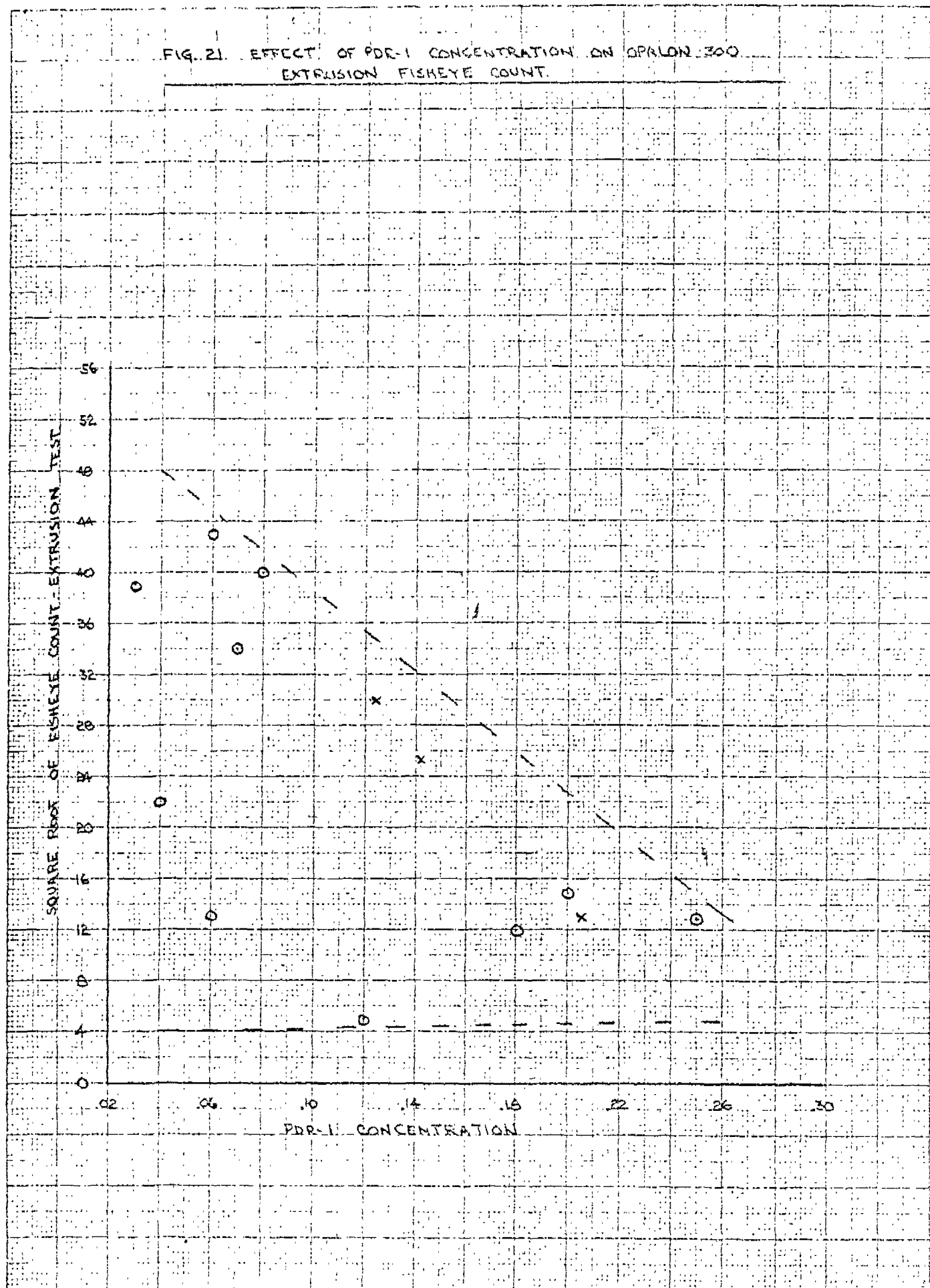


FIG. 22. CORRELATION BETWEEN OPALON 300 DRY TIME
AND EXTRUSION FISHEYE COUNT.

EXPERIMENT A

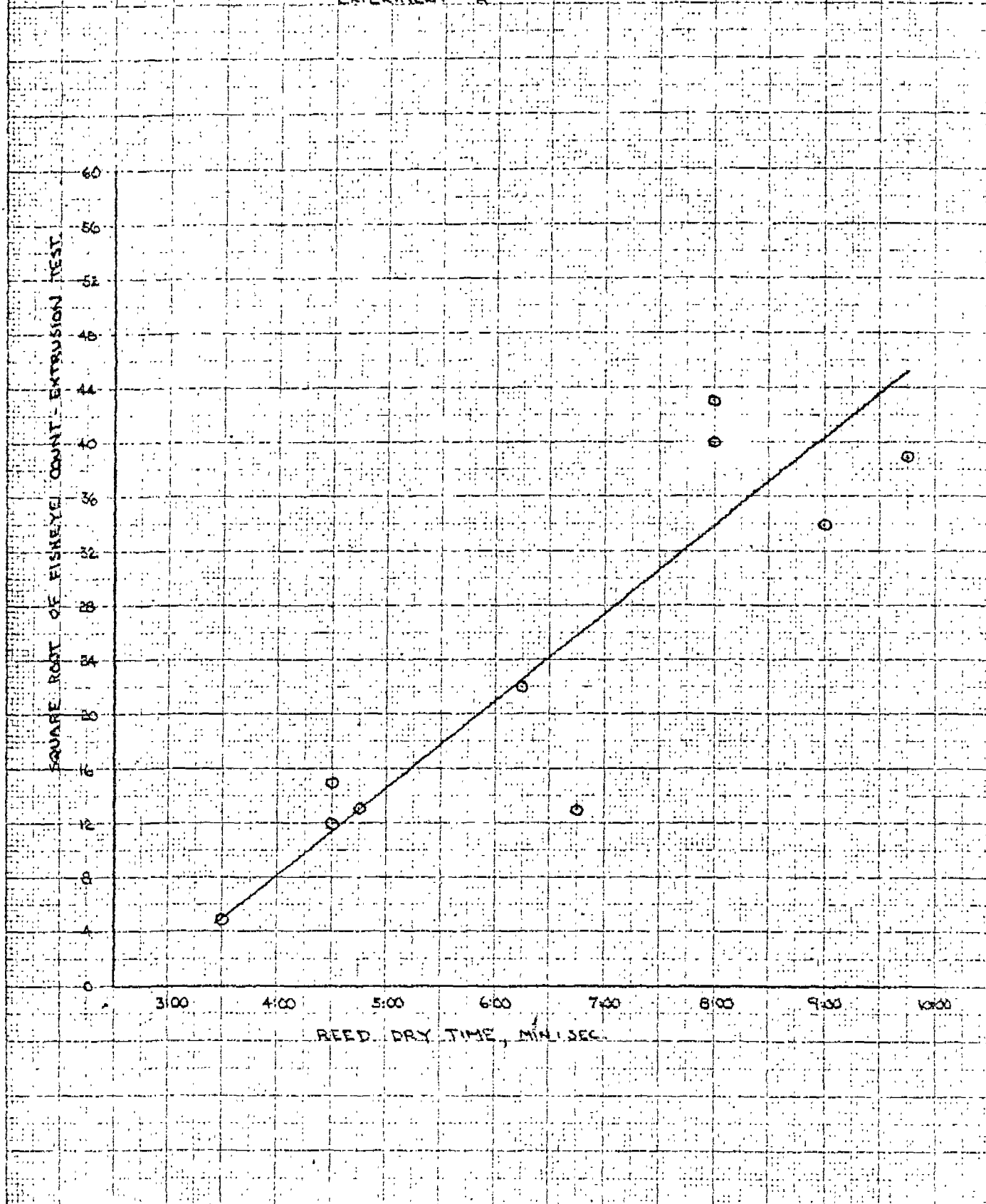


FIG. 23. PDR-1 CONCENTRATION VERSUS OFALON 300PM PARTICLE SIZE.

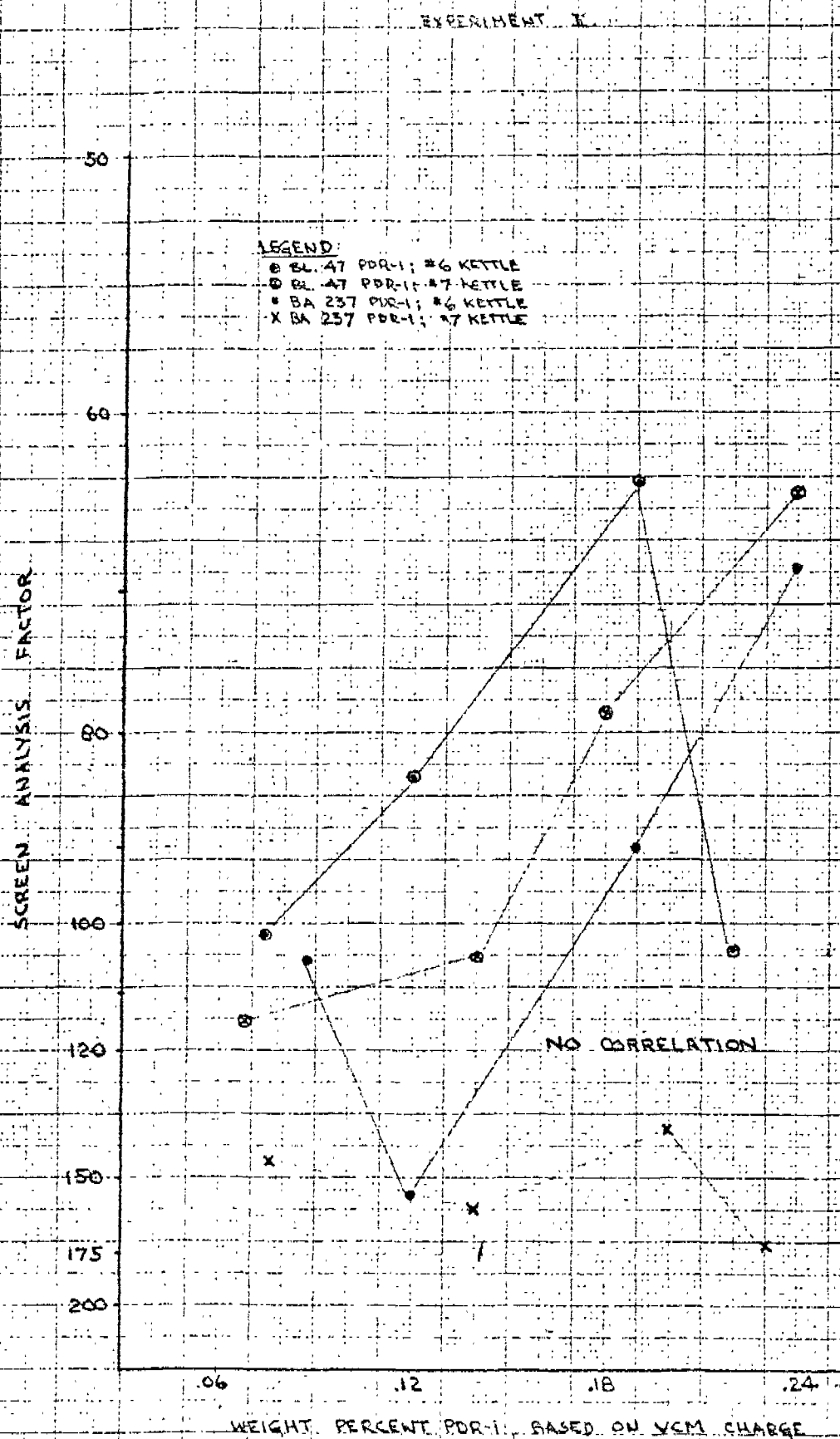


FIG 24 PDR-1 CONCENTRATION VERSUS APALON 300 FM
KITCHEN MIXER DAY TIME
EXPERIMENT I

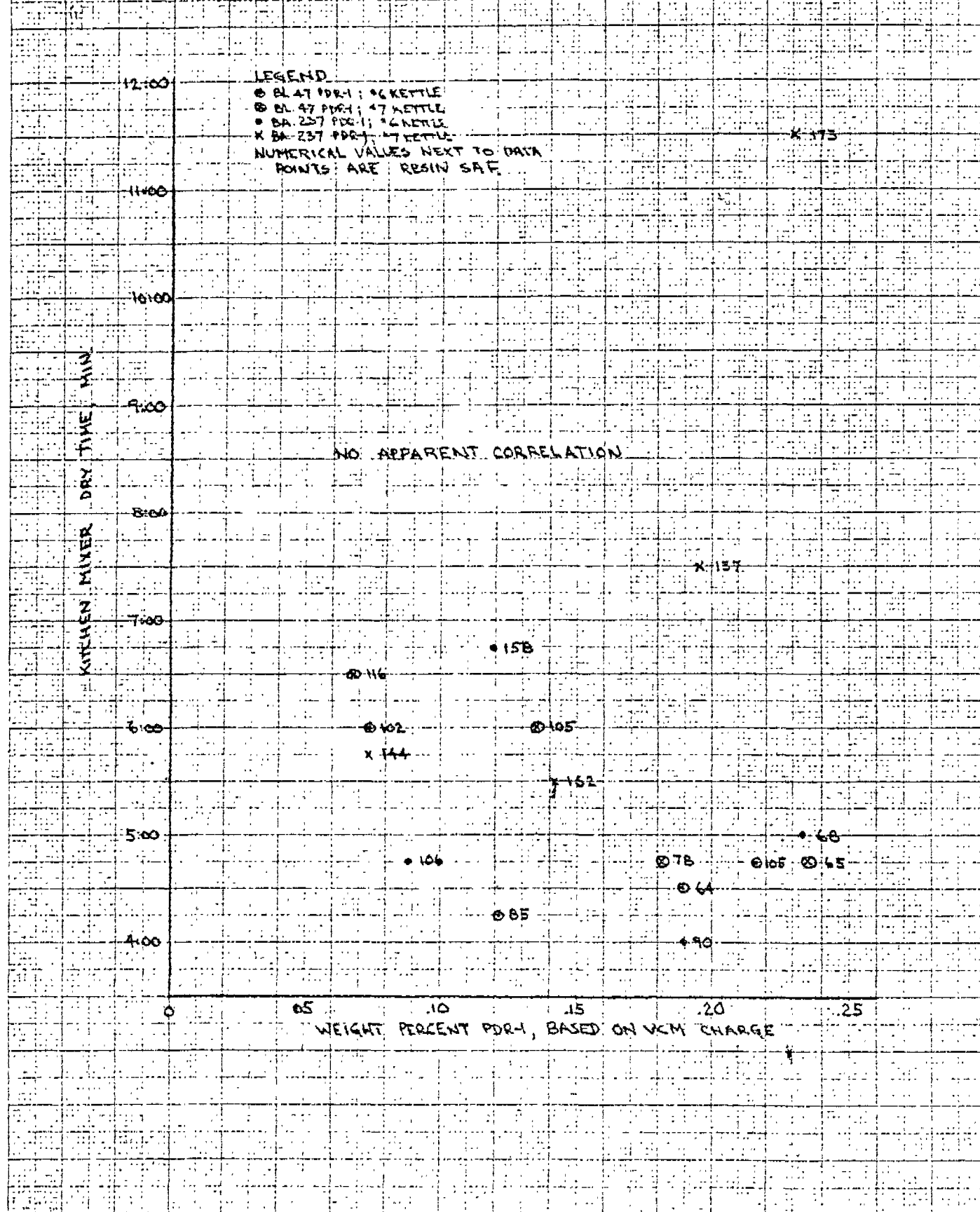


FIG. 25. RELATIONSHIP BETWEEN THE TWO DEPENDENT QUALITY VARIABLES
DRY TIME AND RESIN PARTICLE SIZE

EXPERIMENT II

LEGEND:
 O BL 47 POR-1 #6 KETTLE
 O BL 47 POR-1 #7 KETTLE
 P BA 237 POR-1 #6 KETTLE
 X BA 237 POR-1 #7 KETTLE

KITCHEN MIXER DRY TIME, MIN.

ESTIMATED CORRELATION

SCREEN ANALYSIS FACTOR

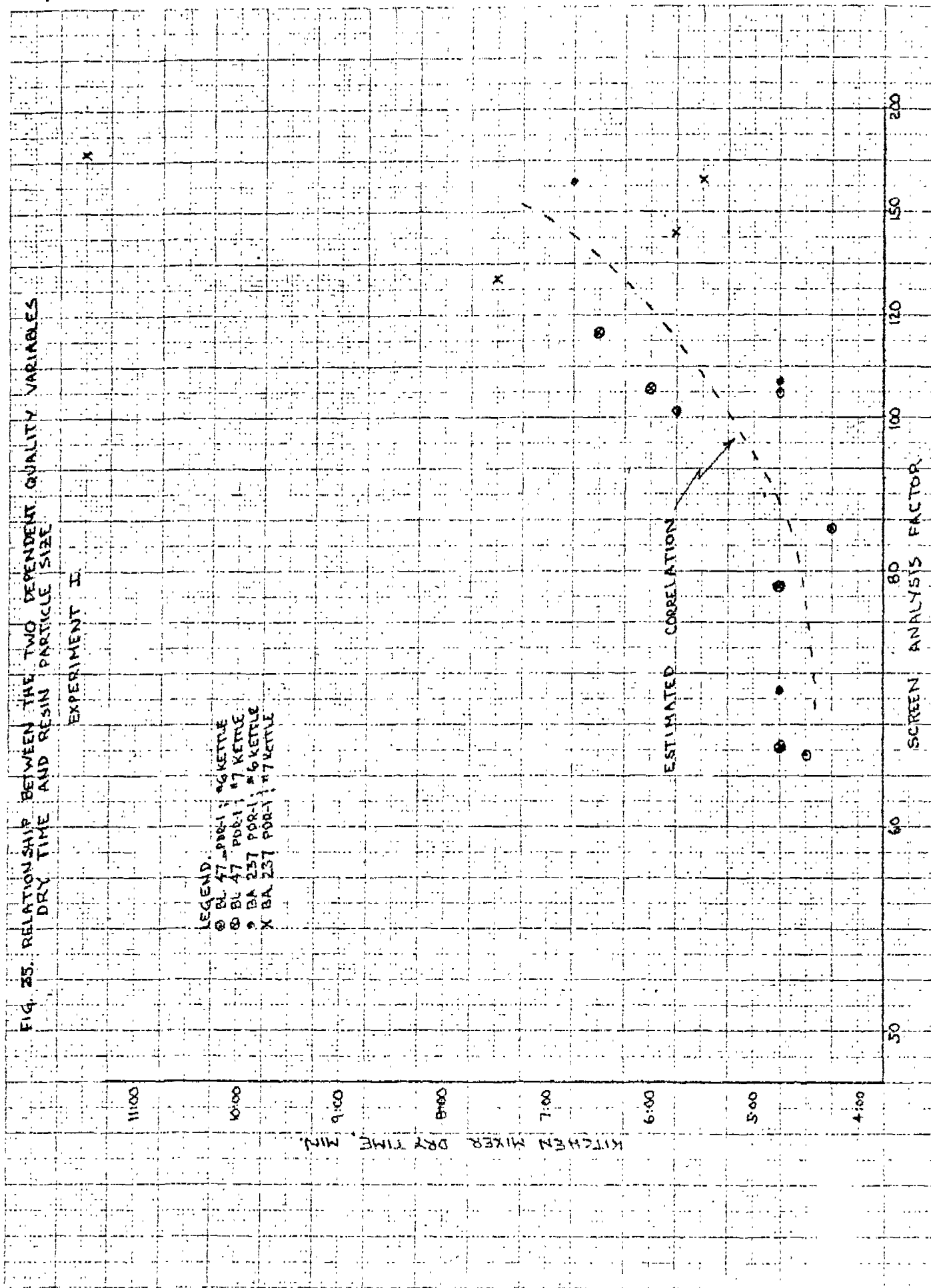


FIG. 26. PDR-1 CONCENTRATION VERSUS ORALON 300 FM. BULK DENSITY

EXPERIMENT I

LEGEND:
 O BL 47 PDR-1; *6 KETTLE
 O BA 47 PDR-1; *7 KETTLE
 O BA 237 PDR-1; *6 KETTLE
 X BA 237 PDR-1; *7 KETTLE
 NUMERICAL VALUES NEXT TO
 DATA POINTS ARE RESIN SAT.

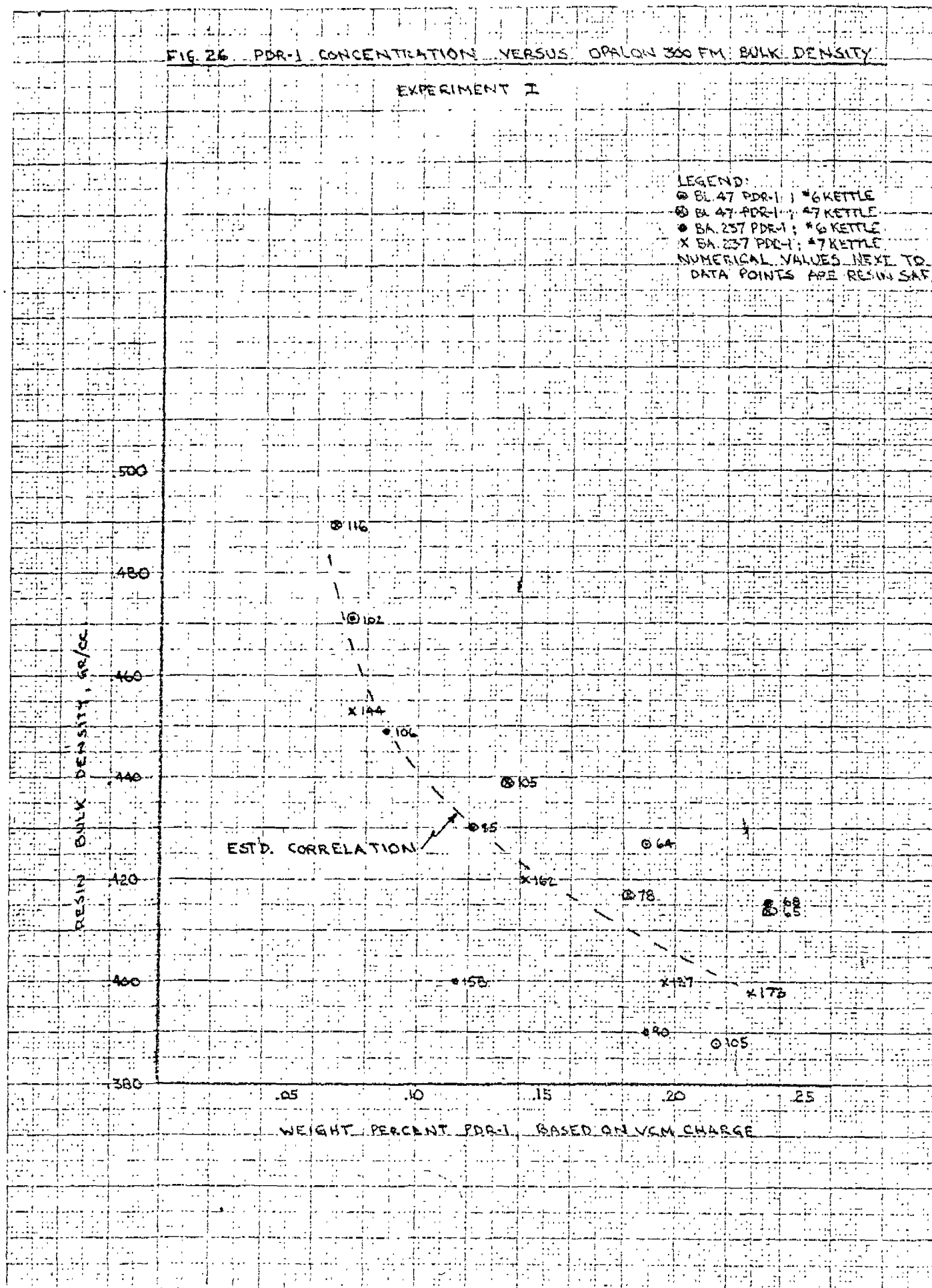


FIG. 27. RELATIONSHIP BETWEEN PDR-I CONCENTRATION
AND BRABENDER FUSION TIME

EXPERIMENT I

LEGEND:

- BATCH 237 PDR-I ; *6 KETTLE
- X BATCH 237 PDR-I ; *7 KETTLE
- ⊙ BLEND 47 PDR-I ; *6 KETTLE
- ⊗ BLEND 47 PDR-I ; *7 KETTLE

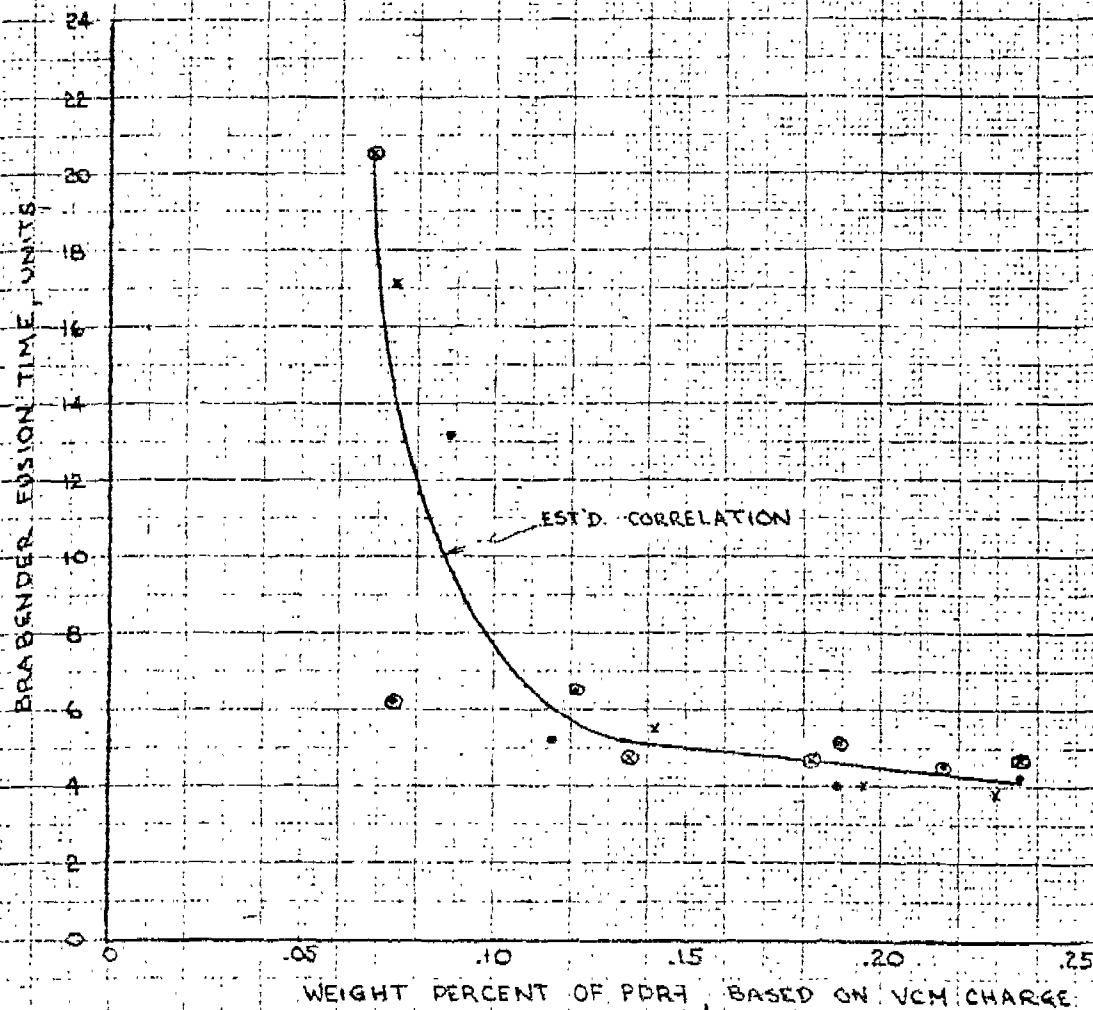


FIG. 28 PDR-I CONCENTRATION VERSUS OPALON 300 FM
BRABENDER FISHEYE COUNT

EXPERIMENT I.

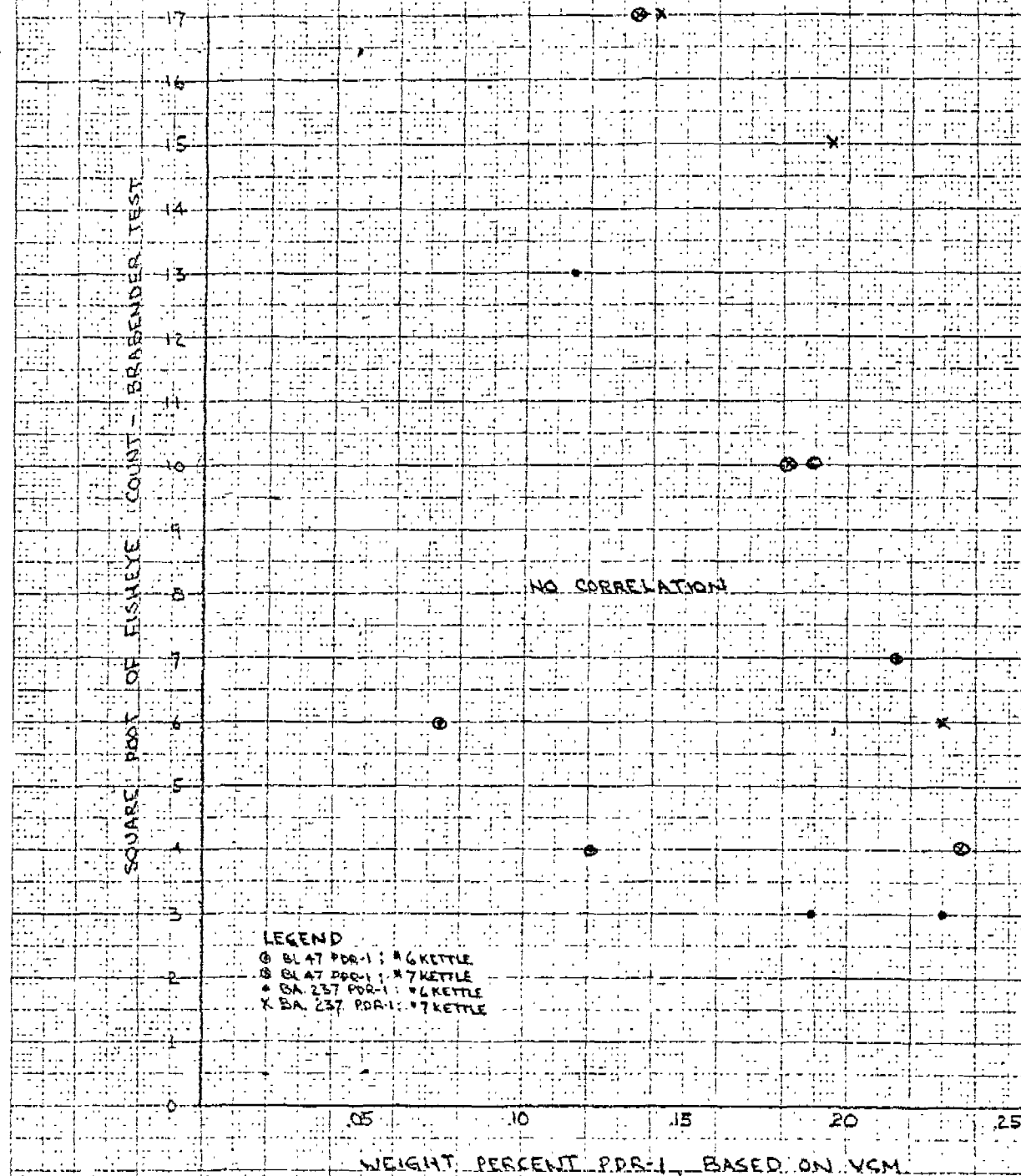


FIG. 29. PDR-1 CONCENTRATION VERSUS OPALON 300FM
EXTRUSION RIBBON GRADE

EXPERIMENT I

LEGEND
 ○ BL 47 PDR-1; #6 KETTLE
 ⊙ BL 47 PDR-1; #7 KETTLE
 ● BA 237 PDR-1; #6 KETTLE
 × BA 237 PDR-1; #7 KETTLE

EXTRUSION RIBBON GRADE

NO CORRELATION WITH PDR-1 CONC.
 POSSIBLE CORRELATION WITH PDR-1 LOTS

WEIGHT PERCENT PDR-1, BASED ON VCM CHARGE

FIG. 30 EFFECT ON OPALON 300 FM HEAT STABILITY BY
COLD WATER WASHING

LEGEND

SAMPLES OF OP. 300 FM MADE IN
#7 KETTLE WITH BL 47 PDR-1 WERE
WASHED ZERO (•), ONE (X) AND
(Δ) THREE TIMES WITH COLD WATER
IN CONTROL LAB TO DETERMINE
THE EFFECT ON HEAT STABILITY.

THE 0.24 PDR-1 RESIN SAMPLES WHICH
WERE WASHED ZERO AND ONE
TIME WERE TOO DISCOLORED TO
OBTAIN TRANSMISSION READINGS.

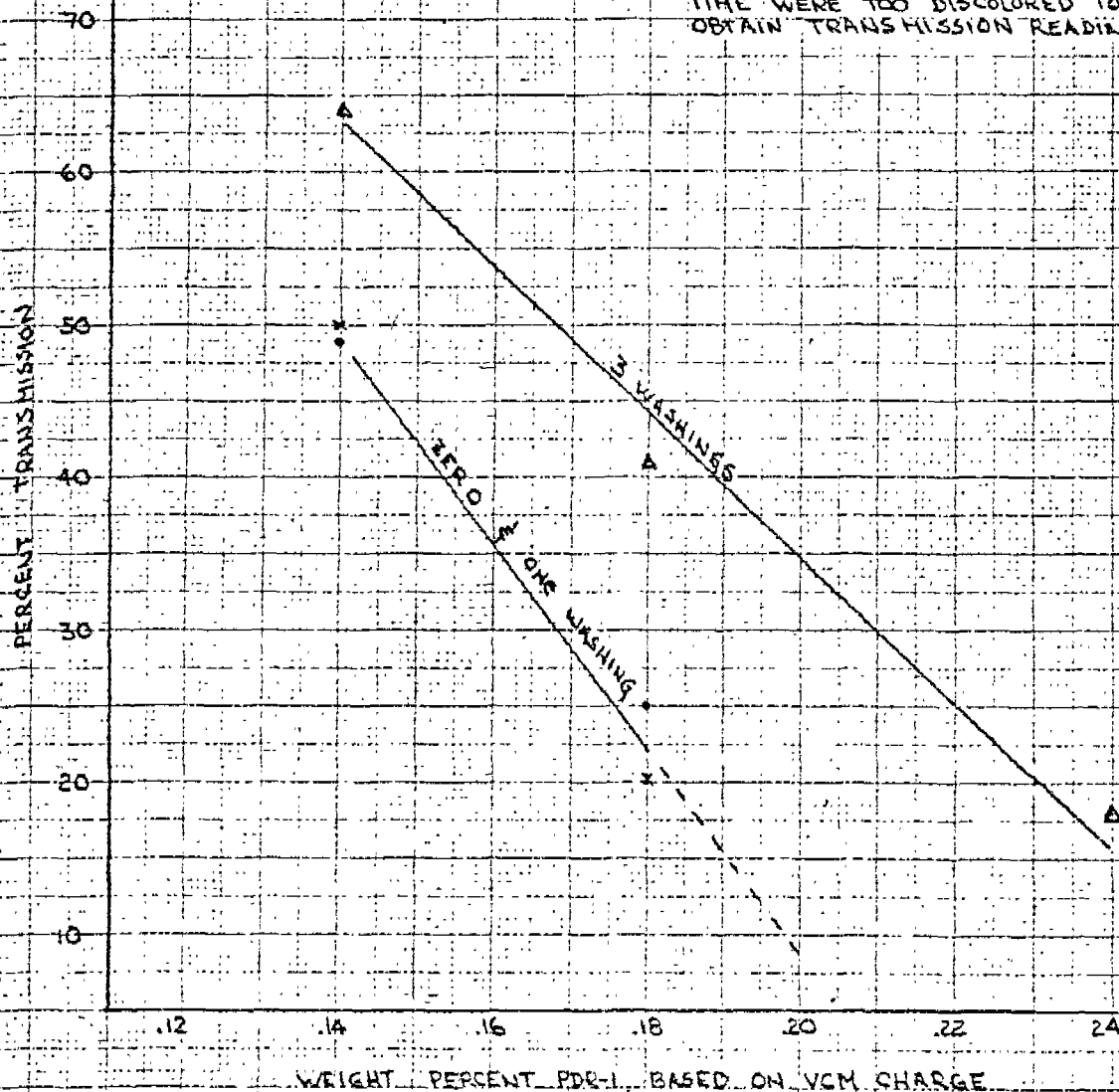
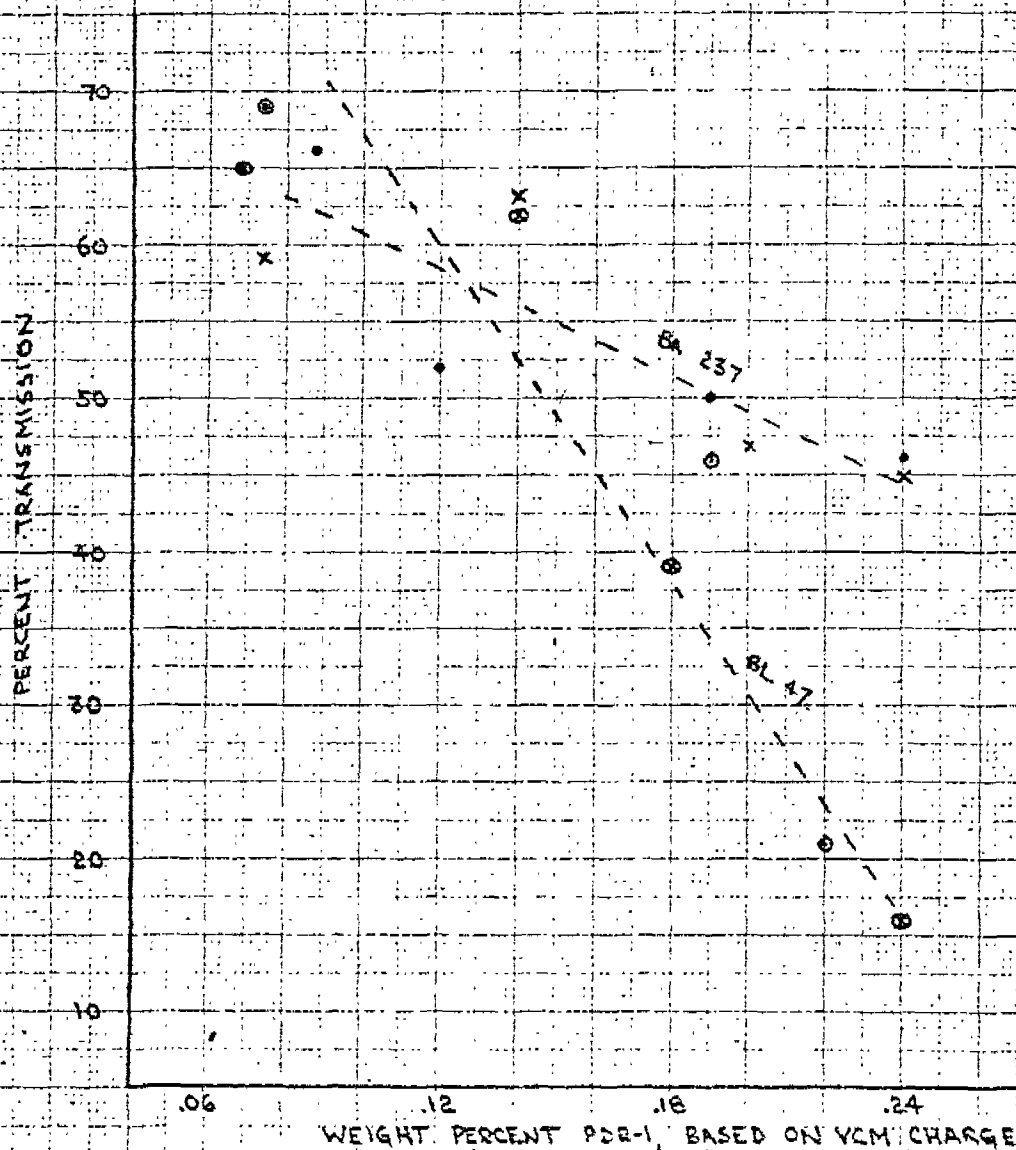


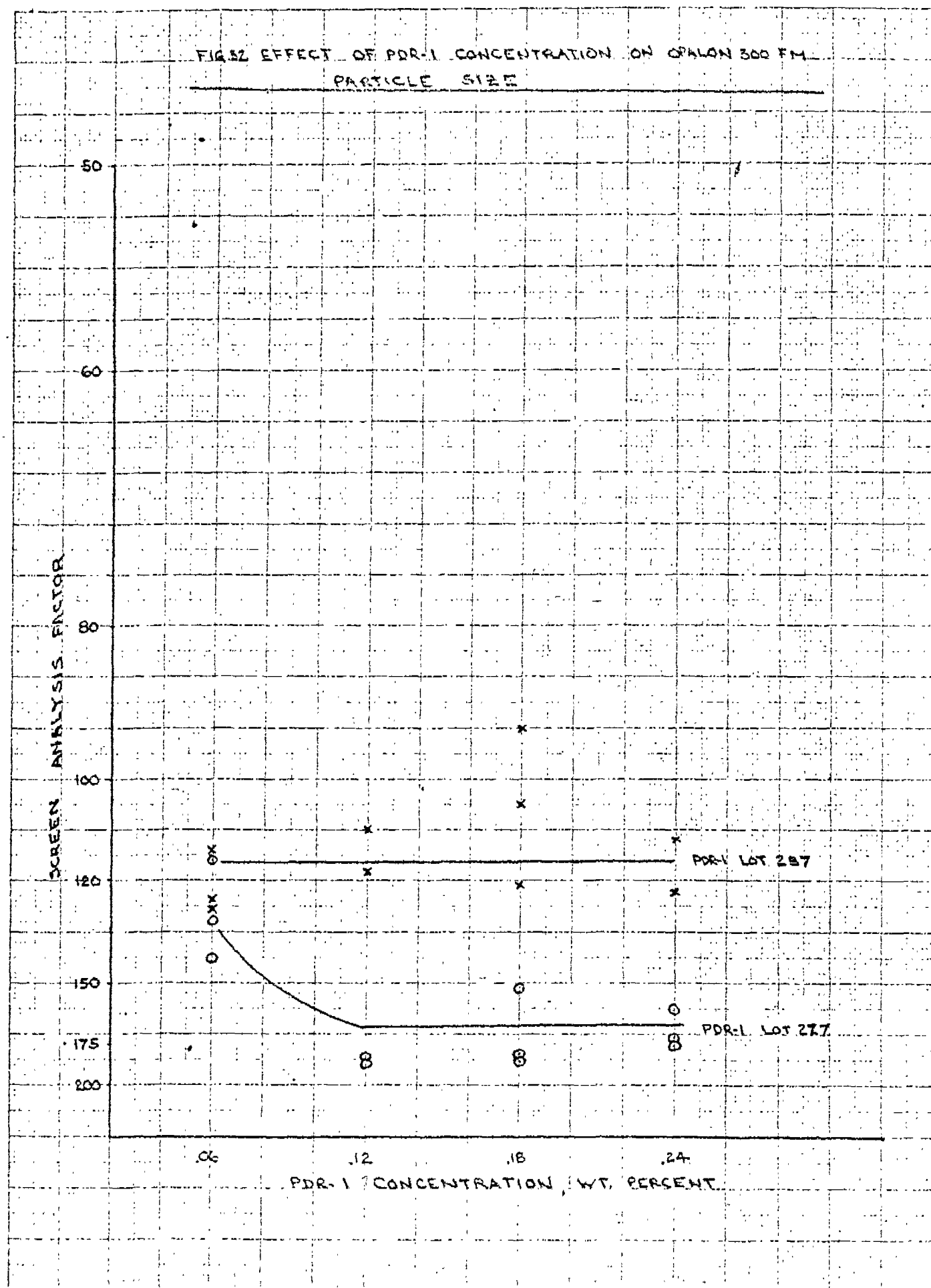
FIG. 31. EFFECT OF PDR-1 CONCENTRATION ON ORALON 300 FM
HEAT STABILITY.

EXPERIMENT I

LEGEND
ALL SAMPLES WASHED THREE TIMES
○ - BL 47 PDR-1, 96 KETTLE
⊗ - BL 47 PDR-1, 47 KETTLE
● - BA 237 PDR-1, 96 KETTLE
X - BA 237 PDR-1, 47 KETTLE



7.7.65 10 X 10 TO THE CM. 250 14
1.002 15.00 15.00 15.00 15.00 15.00



10X10 TO THE CM. 350-14
COPY 145000 ON 10X10

FIG. 33. EFFECT OF PDR-1 CONCENTRATION ON
OPALON 300FM PARTICLE SIZE DISTRIBUTION

EXPERIMENT 8, IN BB BLDG.

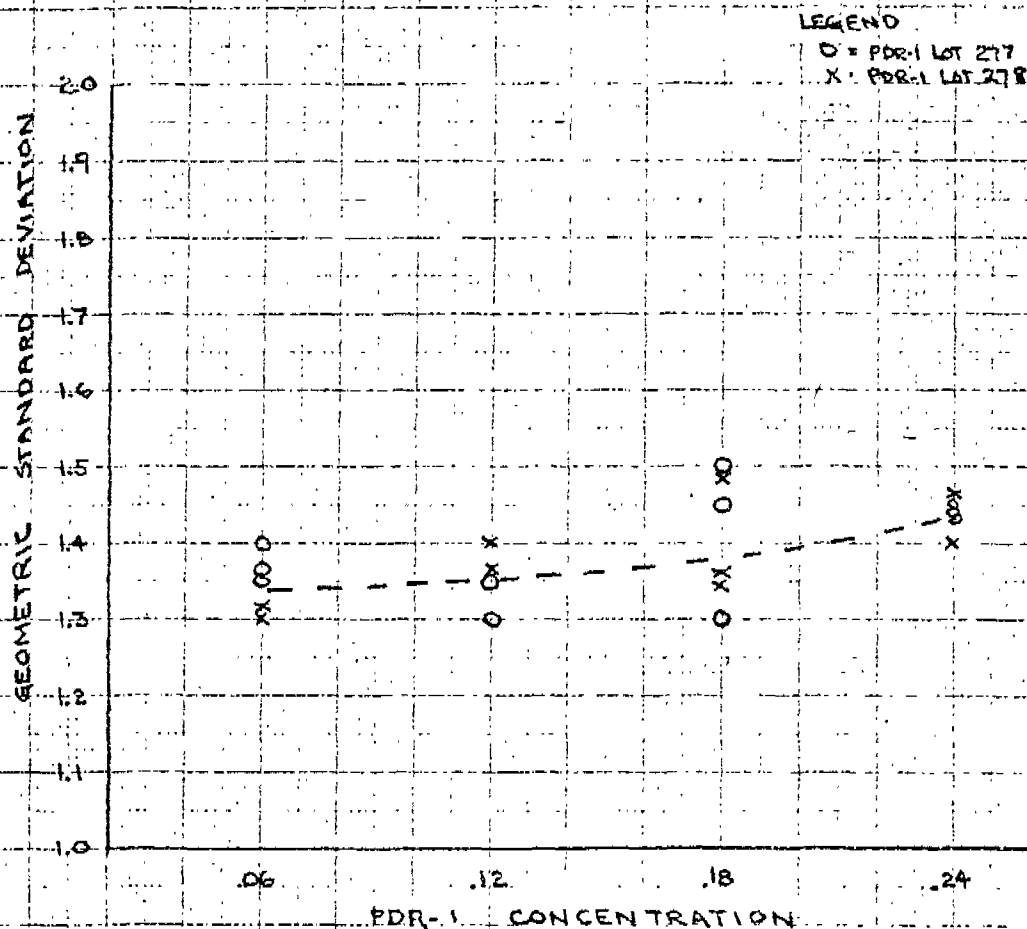


FIG. 34. EFFECT OF PDR-1 CONCENTRATION ON
OPAL ON 300FM BULK DENSITY

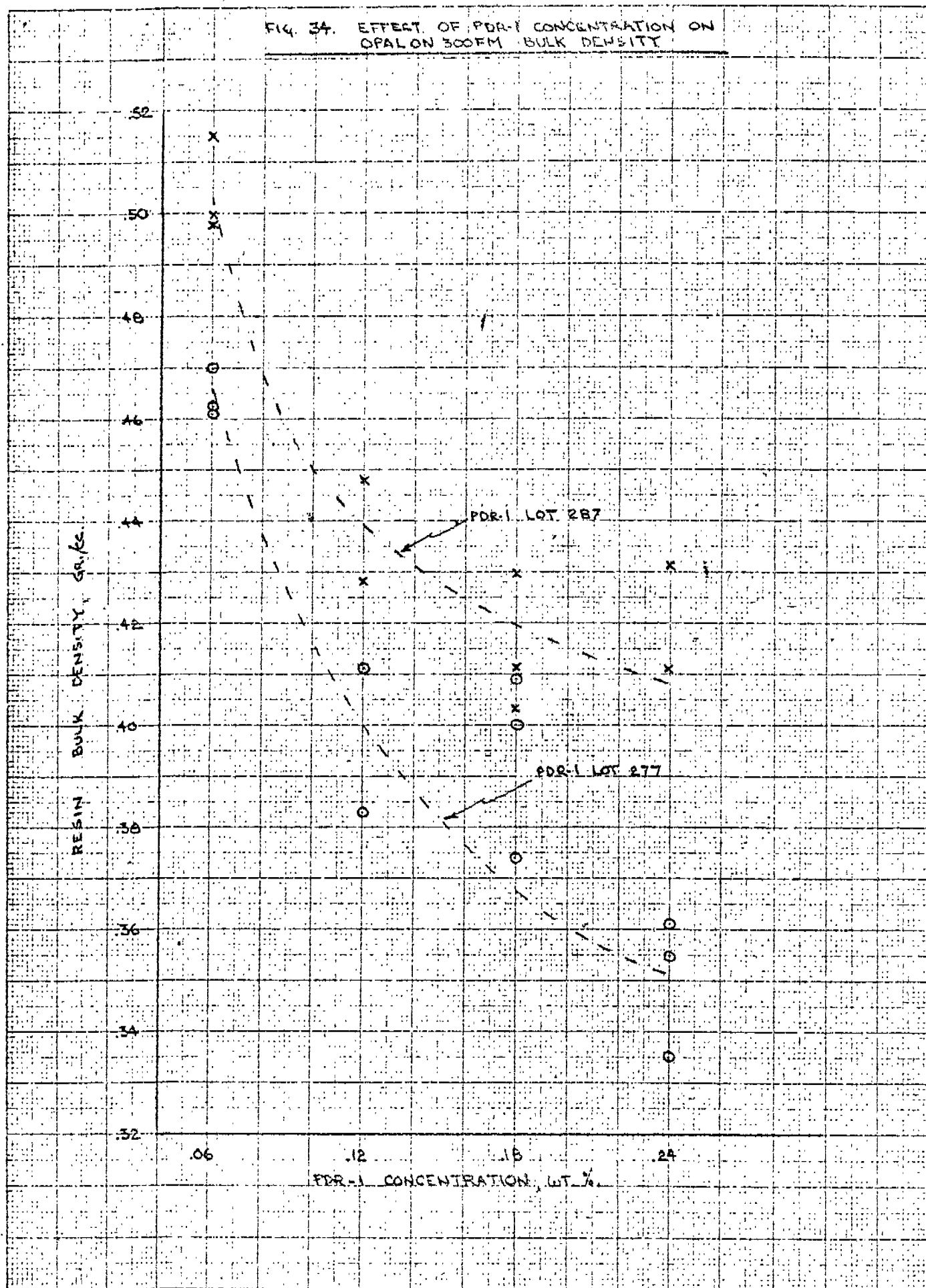


FIG. 35 EFFECT OF PDR-1 CONCENTRATION ON OPALON 300 FM
BRABENDER FUSION TIME

EXPERIMENT B.

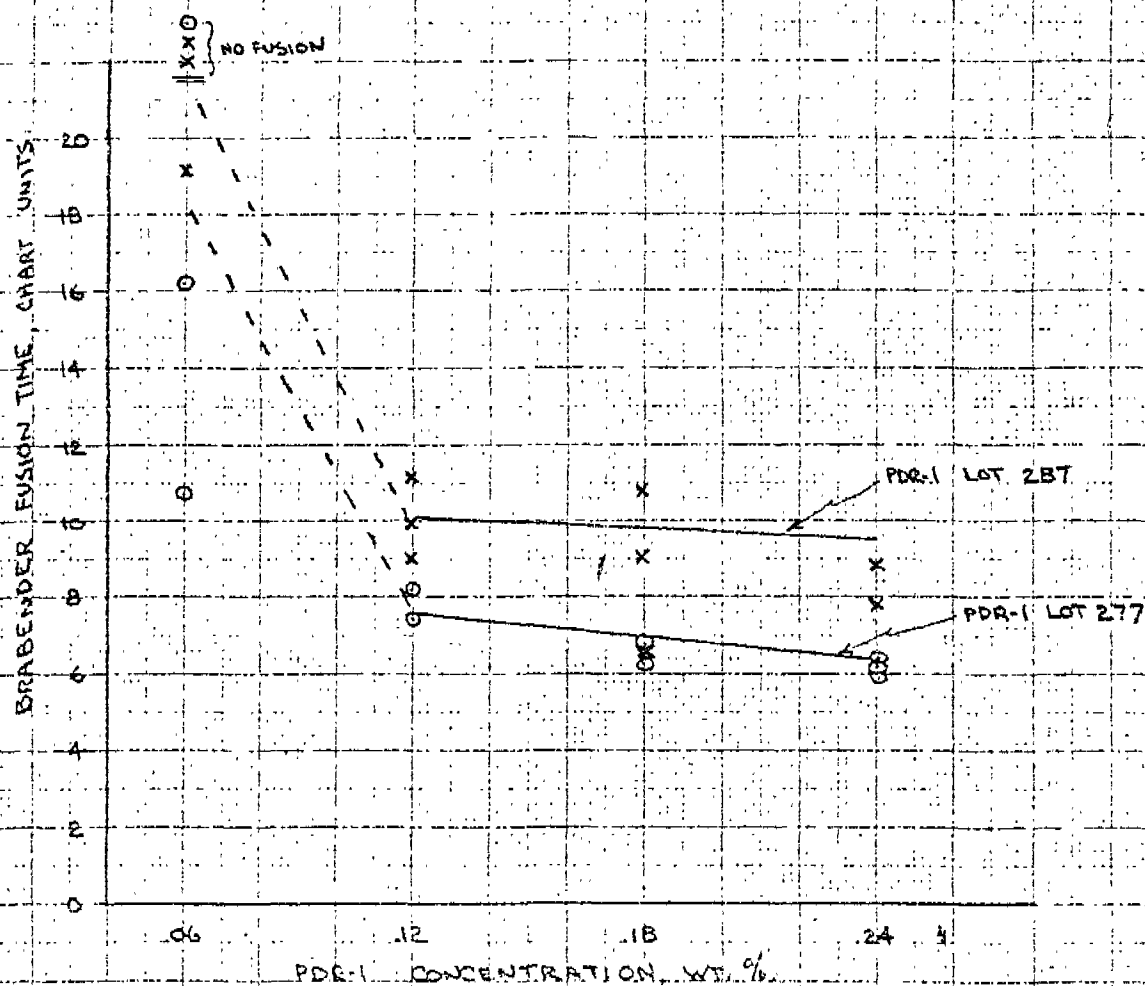


FIG 36. EFFECT OF PDR-1 CONCENTRATION ON
OPALON 300FM HEAT STABILITY

EXPERIMENT 8

LEGEND

X PDR-1 LOT 287

O PDR-1 LOT 277

NOTE THAT LOT 287 PRODUCED
COARSER PVC RESIN AND PROBABLY
WASHED MORE EFFICIENTLY

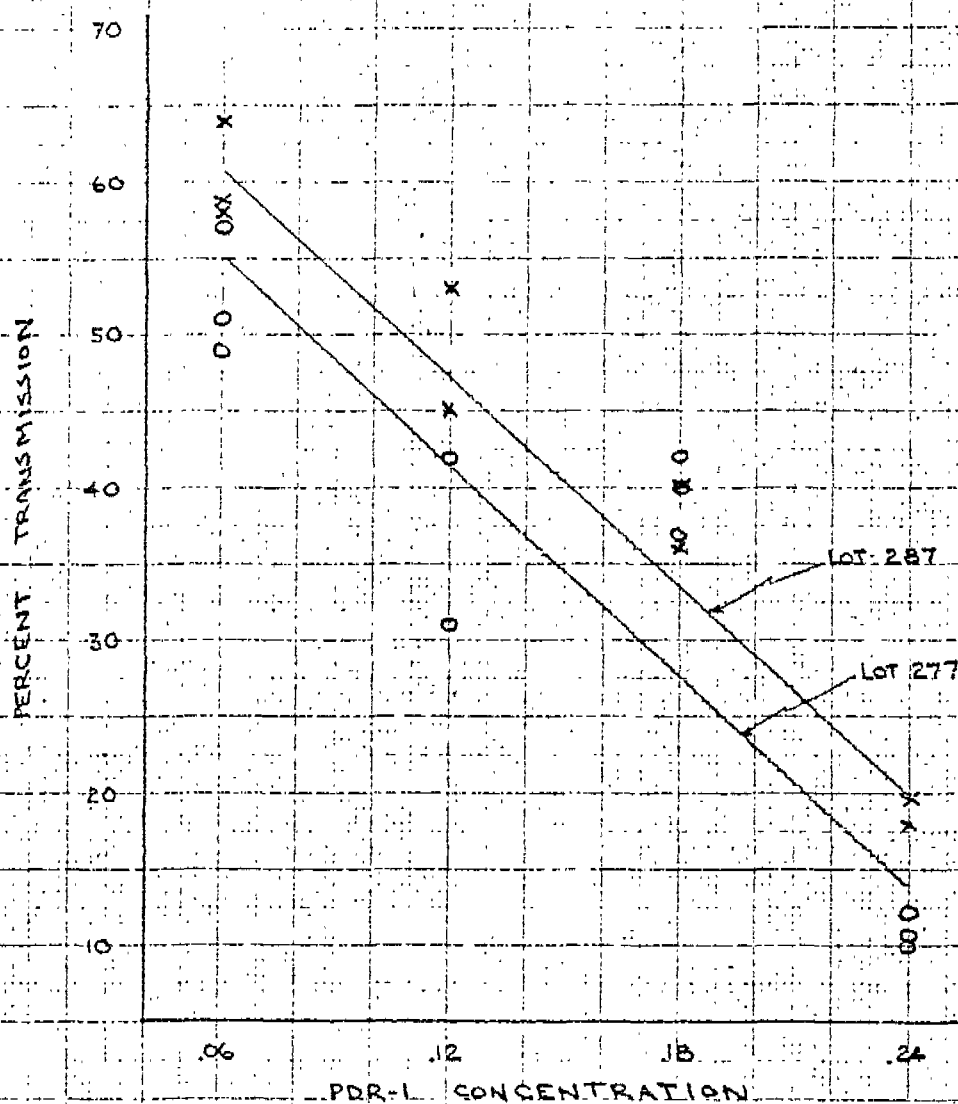


FIG. 37 EFFECT OF PDR-1 CONCENTRATION ON OPALON 300FM
EXTRUSION RIBBON GRADE

EXPERIMENT B.

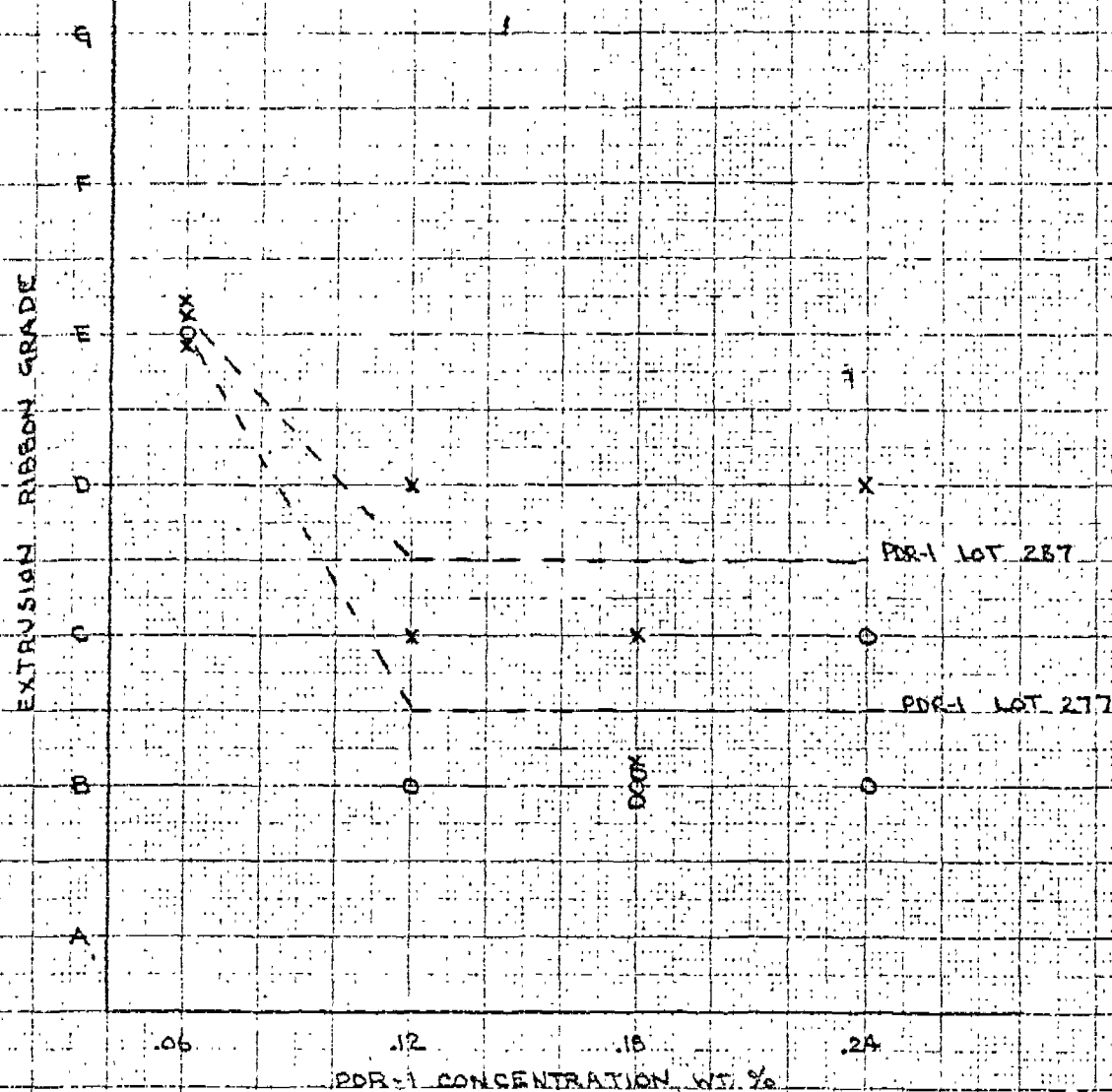


FIG 38. EFFECT OF PDR-1 CONCENTRATION AND BATCH PH ON OPALON 300 PARTICLE SIZE DISTRIBUTION.

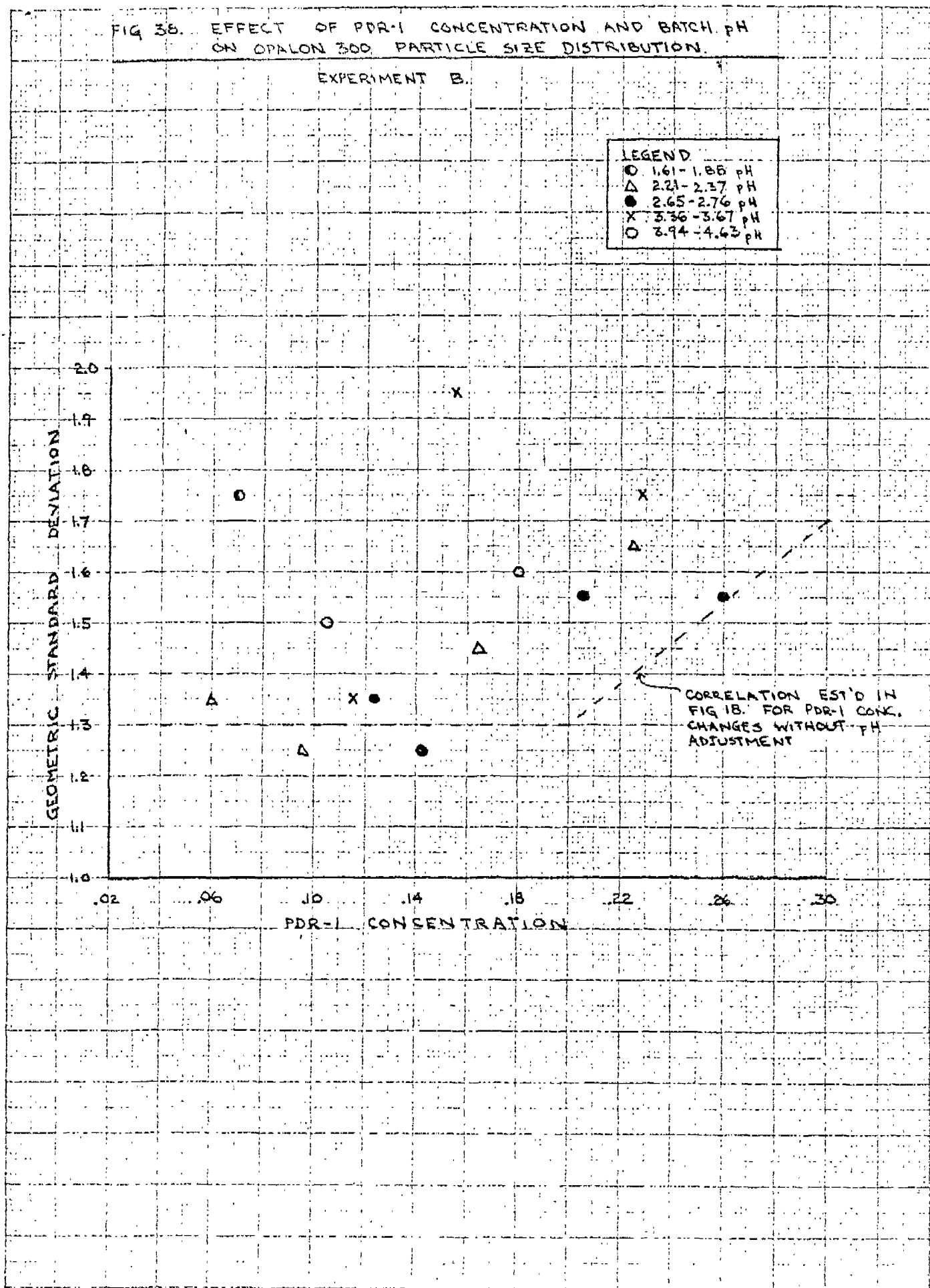


FIG 29. EFFECT OF PDR-1 CONCENTRATION AND BATCH pH
ON OPALON 300 BULK DENSITY

EXPERIMENT B

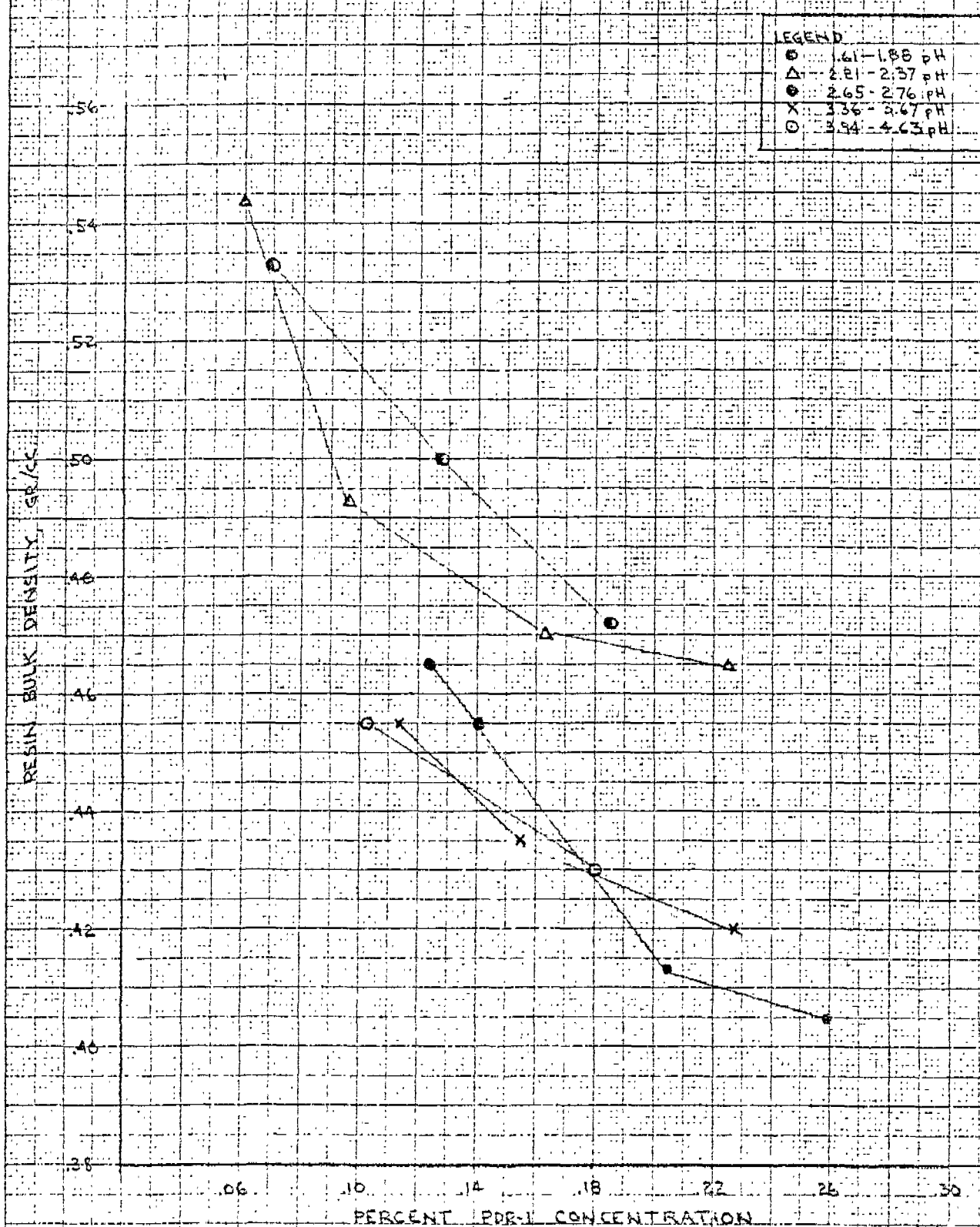


FIG. 40. EFFECT OF PDR-1 CONCENTRATION AND BATCH pH
ON OPALON 300 EXTRUSION FISHEYE COUNT

EXPERIMENT B

