Serial No. KN-50-19

Copy No. 4

V. Chalupski - Plant Files

E. I. DU PONT DE NEMOURS & COMPANY
KREBS PIGMENTS DEPARTMENT
256 VANDERPOOL STREET
NEWARK, NEW JERSEY

NEWARK PLANT
PIGMENT COLOR RESEARCH REPORT

Progress Report

THE FINISHING OF PHTHALOCYANINE PIGMENTS III
"MONASTRAL" FAST BLUE LB

Period Covered August 1, 1946 to April 1, 1948

FILE: 223.41

DATE: 8/31/50

SERIAL NO. EN-50-19

Copy No. 4

Copy to:

1 - Numerical File
2 - Research Office (223.41)
3 - Library File (223.41)
4 - V. Chalupski - Plant Files
5 - D. B. Killian, Newark
6 - J. H. Cooper, "
7 - P. D. Graham, Newport
8 - H. E. Schroeder, Jackson Lab.
9 - Extra
10 - Extra
11 - Extra

NEWARK PLANT

PIGMENT COLOR RESEARCH REPORT

Progress Report

TITLE: THE FINISHING OF PHTHALOCYANINE PIGMENTS III
"MONASTRAL" FAST BLUE LB

August 1, 1946 to April 1, 1948.

CHARGE: A-II-C-229

SUBMITTED BY: A. J. Stratton A. J. Stratton DATE SUBMITTED: 8/11/1950

APPROVED BY: J. H. Cooper DATE ISSUED: 8/31/50

TABLE OF CONTENTS

INTRODUCTION:

The first two reports in this series (See KN-46-19 and KN-46-55) discuss the historical background of the problem of finishing phthalocyanine pigments as proposed in the Pigments Department and cover the first nine months of work on the problem up to about the beginning of semi-works studies. A third report (KN-47-51) covers other aspects of the problem through to the end of 1947 and makes incidental mention of the finishing steps. This report will be limited to studies of finishing "Monastral" Fast Blue LB (4.0-4.5% chlorine introduced via 4-chlorophthalic acid in the synthesis) by milling methods and will cover the period from the beginning of semi-works studies (about August 1, 1946) to the start up of the Newport plant on April 1, 1948. Studies on finishing Blue LB subsequent to the plant start up and on finishing polychloro CPC Green will be covered in separate reports.

SUMMARY AND CONCLUSIONS:

The studies on finishing CPC-LB covered in this report fall logically into four divisions as follows:

A. Milling in hydrocarbon solvents

Continuing the studies reported in KN-46-55, semi-works operation was started on a process in which kerosene wet filter cake or slurry from the reactor was milled with more kerosene or mineral spirits in a ball mill. The process was reasonably successful as to grinding, but the problems involved in separating the ground pigment from the solvent proved insurmountable. Therefore, the use of hydrocarbon solvents as grinding media was ultimately abandoned.

B. Milling in isopropanol

When it became apparent that a water miscible solvent was required to permit complete removal from the pigment paste, attention was turned to the use of isopropanol as the grinding medium. Such a process was developed in the laboratory, was carried to reasonably successful semi-works operation and provided the basis for plant design. Most of the problems peculiar to solvent grinding were solved during this study, including:

1. The use of 1/8" balls in place of 1/2", or larger balls originally contemplated.
2. The requirement for low residual kerosene content of the crude pigment.
3. The demonstration of optimum mill loading at a material/void ratio of 1.5-1.6 and a pigment/liquid ratio of about 0.075; i.e. considerably less pigment and thinner slurries than contemplated in original plant plant design.

*The terms "milling" and "grinding" are used synonymously in this report with reference to processes of finishing phthalocyanines by mechanical reduction of particle size in contrast to the previously known chemical method of "acid pasting".

No treating agents of consistent value were found for improving the performance of grinds in isopropanol.

During this period the improved technique of isolating the crude by sulfation was adopted, and the improvements confirmed on fairly large scale grinds.

In spite of the many favorable aspects of grinding in isopropanol, attempts to translate the process into larger scale semi-works mills proved disappointing with respect to rate of grinding and ultimate strength. The troubles seemed to be related to the incompatibility of the residual kerosene in the crudes with the 91% isopropanol recoverable by distillation. Attention was then turned to acetone as a solvent readily recoverable in a purity with which kerosene is compatible.

C. Milling in Acetone

Direct comparisons of acetone with isopropanol as the grinding medium demonstrated the superiority of the acetone and, with its use, satisfactory performance was, for the first time, achieved in the large semi-works mills. Most of the variable data obtained with isopropanol were directly applicable to the use of acetone. The studies on mill loading were repeated, resulting in the specification of somewhat thinner slurries. In spite of this, however, the cycle was reduced and the strength increased so that actual production (in lbs. per hour) was materially increased.

Plant processes were specified and placed in operation in April, 1948, and have been thoroughly justified by successful operation with only minor modifications for over two years at capacities equal to or better than contemplated in design.

D. Treatment of Pigment after Milling

Procedures for extraction of the pigment after grinding were not exhaustively studied and the operations specified were based upon rather arbitrary laboratory procedures involving a combination of tradition from Orchem experience and expediency in reducing seemingly excessive times. These operations probably deserve more careful study.

The section on discussion of details includes a number of miscellaneous observations not directly in the line of this endeavor. The following deserve mention here:

1. The extreme greenness of chlorine-free CPC when ground in solvents, as contrasted to the redness of the same pigment on acid pasting or salt milling. This was traced to a new crystal phase of distinctive properties and is the subject of a separate report on "Monastral" Fast Blue BG.

2. The fact that crude pigments can be ground in hydrocarbon solvents or alkyd resin solutions to give pastes which can be converted to enamels of good strength and, usually, of outstanding resistance to flocculation.

PATENT SITUATION

Solvent grinding as a method of finishing phthalocyanine pigments is considered to be a new and novel process and a patent application (Lane and Stratton, case 1137-K) was filed early in 1949. The patent aspects of the new crystal form of chlorine-free CPC are discussed elsewhere.

No other patentable improvements are believed to exist in this work and no infringement of competitive patents is known.

DISCUSSION OF DETAILS

A. Hydrocarbon Solvents

When report KN-46-55 was written in July, 1946, the preferred process comprised grinding the pigment, preferably the kerosene wet cake or slurry from the reactor in a ball mill in either more kerosene or mineral spirits, followed by removal of the solvent by steam distillation, and extraction of the resulting paste. Dry toners, considered at that time to be of excellent quality, were obtained in the laboratory. Semi-works operation was started on this process and some excellent toners were made by grinding the reactor slurries in additional kerosene. However, experience demonstrated that this process was defective in several respects largely concerned with the isolation of the pigment after the ball mill operation was complete.

In the first place, the process gave wide variations in quality which were never completely explained. The steam distillation of the kerosene, or other hydrocarbon, from the ground slurry proved to be slow and required an excessive amount of steam. The conditions shown in the laboratory to be optimum resulted in serious foaming which was ultimately controlled to only a fair degree by a combination of mechanical and chemical variables. Finally, and probably of most serious import, complete removal of kerosene was never achieved and it became apparent that radical changes in processes would be required to make many of the lakes in our standard line and it seemed doubtful if water dispersible colors could be made at all. Some of the work on this phase of the problem is given in more detail in KN-47-51.

In view of these difficulties and some very promising results with water miscible solvents, particularly isopropanol, it was decided near the end of 1946 to abandon the use of hydrocarbon solvents and to concentrate on the use of isopropanol as the grinding liquid.

B. Milling in Isopropanol

The earlier reports (KN-46-19 and KN-46-55) record experiments on grinding in water miscible solvents including methanol, ethanol, isopropanol and acetone. The lower boiling points with consequent increased fire hazards as compared to kerosene and mineral spirits, the considerably higher costs and the extra operations involved in removing the kerosene used in the synthesis had militated against serious consideration of these solvents as grinding media. However, as the serious problems in the removal of hydrocarbons became apparent, laboratory investigation of water miscible solvents was again undertaken. For this work, isopropanol was chosen over methanol because of lower toxicity, over ethanol because of the freedom from control problems associated with the use of large amounts of ethanol, and over acetone because of higher boiling point and slightly lower cost. Experimental studies were carried out with both 99% isopropanol and 91% isopropanol which is the easily recoverable constant boiling mixture with water, with the major work on the latter.

In the early stages of the study of grinding in isopropanol, the method of isolating the crude CPC-LB from the reactor slurry had not yet been firmly established and attempts were made to grind a press cake containing roughly 65% solids (not all CPC) and 35% kerosene. Although fairly successful with 99% isopropanol, a two-phase liquid system resulted with 91% isopropanol, the pigment going into the phase containing the kerosene. In any case, the kerosene in the finished product posed the same problems encountered on attempting to remove it from slurries ground in hydrocarbons. Some dry crudes were made by evaporating the kerosene from the filter cakes in a high temperature oven and such crude pigments were successfully ground in 91% isopropanol even though relatively large amounts of impurities were present. However, experience in the semi-works pointed toward a conventional steam distillation as the efficient means of removing kerosene from the reactor slurries. Since this was followed by filtration and drying, a relatively pure extracted crude became available for grinding studies. It was from such a crude that the first really successful large scale laboratory grinds (1.8 gallon mills) were made in November, 1946. The resulting products were about 15% strong and slightly green vs BT-172-D, the then existing Orchem standard. The pastes were converted to "Ramapo" lakes approximating BP-173-D in strength but somewhat dull in hue. Linoleum lakes (BP-263-D) were green in hue. BL-220-D lakes showed acceptable dispersion in water and acceptable strength, but were dark in masstone and green in tint.

Direct comparisons of grinding the same crude in both kerosene and isopropanol demonstrated unmistakable superiority in the latter (see Figure I) and the first semi-works grind in isopropanol was made late in December, 1946. This grind was disappointing in the slow rate of strength development and in the failure to reach the strength of laboratory grinds. These results were ultimately traced to two factors, viz. ball size (1/2" balls in semi-works vs 1/8" balls in the laboratory) and residual kerosene in the supposedly dry crude.

Ball Size

The superiority of the small balls was demonstrated in the laboratory mills (see Figure I) and various expedients were tried to accomplish the same purpose in the larger mills. The addition of an inorganic salt, such as Na_2SO_4, (1-2 parts per pound of CPC) greatly improved the performance of 1/2" balls in the laboratory mills, but was much less successful in the semi-works and caused severe foaming in the alcohol recovery still. A charge of worn balls approximating 1/4" diam. was obtained from one of the F&F plants but resulted in no improvement over the 1/2" balls. Finally, a source of low cost cast steel shot designed for shot blasting and approximating 1/8" in diam. (though irregular in size and shape) was located. This material, known as "wheelabrator" shot, was successful in lab. trials and the first semi-works trial was made in the 25 gallon mill about April 1, 1947. It was quickly shown that strength equal to BT-172-D was attained in about 48 hours and that an ultimate strength about 20% above this level could be reached in about 90 hours.

Kerosene Content of Crude

Since the use of such small balls in large mills was regarded as highly unconventional, efforts were continued on means of using 1/2" balls in the 275 gallon mill but proved unfruitful. During this period, however, unexplainable peculiarities in the behavior of some of the batches of crude made in the semi-works led to the knowledge that the residual kerosene content of the supposedly dry crudes varied widely, some lots containing as high as 5-8% kerosene and invariably showing poor performance in the mills. A drying study, relating to the design of plant driers, thus led to the specification of a drying temperature of 150°C (300°F) and a maximum kerosene content of 0.5% in the dry crude.

Use of Treating Agents

It has been obvious from the early stages of the study of milling phthalocyanine pigments in organic solvents that the pigment is severely flocculated in the grinding medium. Such conditions are considered to make for inefficient grinding, and extensive efforts have been put forth in the search for a treating agent which would improve the efficiency of grinding. Most agents tried with isopropanol showed no benefit and some actual harm as judged by rate of grinding and, in many cases, by ultimate strength. The only exceptions judged worthy of semi-works trial were MP-189 (no longer commercially available), Petronate and Tetra-sodium pyrophosphate ($Na_4P_2O_7$) (See Figure II). However, the semi-works trials were not promising. Petronate caused severe foaming in the recovery still and $Na_4P_2O_7$ gave very inconsistent results even in the laboratory. At best, it promoted more rapid grinding in the early stages, but tended to a slight sacrifice in ultimate strength. In short, no treating agent was found to be of clear value in isopropanol grinding of CPC and none was adopted for use. Table I in the appendix lists the agents tried and the general results.

Use of NH_4OH in the Grinds

Conventional ball mill operation discourages the use of steel balls in aqueous liquids, particularly in closed mills, because of corrosion and the possibility of hydrogen liberation. To avoid such corrosion, the practice was adopted in the work with isopropanol of maintaining the contents of the mill rather strongly ammoniacal by the addition of aqueous NH_4OH to the charge. During the study of the use of treating agents, the wisdom of this practice from the standpoint of quality was questioned and it was demonstrated to be undesirable. It was first proposed to substitute NaOH but this was shown to be insoluble in isopropanol. Finally, no significant corrosion of the mill or ball charge in the absence of alkali was found and its use was abandoned.

Quality of product from Isopropanol Milling

As the study of grinding CPC in isopropanol proceeded, it became evident that the products obtained were far from perfection as to quality. On the good side were to be found strength substantially above any pigments commercially available at that time, relatively good texture, soft inks and paint mill bases, and relative freedom from flocculation in paint vehicles. On the negative side, one found a hue somewhat greener than was considered desirable and a serious tendency to dullness. As pointed out in KN-47-51, this greenness and dullness were largely traced to the quality of the crude used.

In this study a careful comparison of isopropanol grinding, salt milling and acid pasting was carried out on crudes from several sources. The conclusion was clear that solvent milling did not per se cause greenness nor dullness, but that it was less effective than either salt milling or acid pasting in purifying an impure crude. Thus, efforts were directed toward improving the quality of the crude by eliminating contamination with iron phthalocyanine and by isolation from the reactor slurry through the sulfation technique described in more detail in KN-47-51. This technique was adopted toward the end of 1947 and the improved type of crude like that ultimately made in the plant became available for experimental studies. Laboratory preparations under optimum conditions by this process were actually on the red side of "Ramapo" Blue BP-173-D and fully equal in intensity, though the semi-works never quite equalled this quality.

Mill Loading

With the process for the crude on relatively firm ground, it was possible to proceed with a detailed study of operating variables in the ball mill.

Mill speed was more or less fixed and the conventional optimum of 70% of the critical speed (the speed at which the balls are carried around the periphery of the mill) was accepted with little study. Some use of lower speeds was definitely unfavorable.

The ball charge was investigated in a limited manner over the range from 30% to 60% of nominal occupancy (gross volume of the bed of balls). There was little choice in the range of 40% to 50% with poorer results on either side. Most of the work at Newark was done at 40% ball loading and that figure was specified for the plant mills. After plant start-up, the ball loading was increased to 50% in two mills with no conclusive evidence of advantage.

The most important variables concerned the ratio of pigment to grinding liquid (P/Ve) and the ratio of total mill loading (pigment plus liquid) to the voids in the ball charge (M/V). Thus, at an M/V of 1.0 the ball charge is just covered. Obviously these two variables are inter-related and a ladder study was carried out in the range of M/V from 1.0 to 4.0 and P/Ve from 0.03 to 0.15. Curves and tables summarizing this study (see Table II and Figures V to VIII) are found in the Appendix to this report. In general, the conclusion was clear that the highest strengths were obtained at low M/V ratios and toward the lower end of the P/Ve range studied. However, when interpreted in terms of rate of grinding (viz. lbs. of pigment per hour of mill operation) there was a definite optimum at an M/V of about 1.5-1.6. The optimum P/Ve was less clear cut but was approximately 0.07. It was perhaps odd but very fortunate that the conditions thus specified were almost exactly those arbitrarily selected long before this study for use in laboratory grinds.

Semi-Works Experience with Isopropanol in Large Mills.

In spite of the favorable experience with isopropanol in the laboratory and in the 25 gallon semi-works mill, attempts to duplicate this experience in the larger semi-works mills (200 gal. and 278 gal.) proved disappointing both as to cycle required and in ultimate strength reached. Conventional scale-up formulas appeared to fail completely as to the predicted shorter cycle in the larger mills. This raised serious questions as to the capacity of the plant mills already nearing the installation stage, particularly as the indicated loading was approximately half that contemplated in the original design. A careful review of all factors together with additional experimental work, led to the conclusion that at least some of the difficulty lay in the inevitable slight amount of kerosene on the pigment particles and to insolubility in the 91% isopropanol being used. Contributing to this conclusion was the rather clear evidence that pigments with kerosene content below about 0.2% could be ground in isopropanol of 75%-80% concentration and that pigments containing appreciably higher amounts of kerosene required 99% isopropanol for efficient grinds. Since 99% isopropanol is not readily obtainable by recovery in a conventional still, the use of isopropanol as the grinding liquid thus fell into serious question.

C. Acetone Milling

As noted above, acetone had been previously considered as a grinding liquid but rejected in favor of isopropanol. However, the serious deficiencies found to exist in the use of isopropanol brought acetone back into the picture as a candidate. The DuPont Company has extensive experience in handling acetone and does not consider it significantly more hazardous than isopropanol. In spite of a lower

boiling point, it is said to have some points in its favor. It is readily recoverable by distillation in purities up to 99.5% at which point kerosene is completely miscible. Furthermore, it had already been shown (see KN-47-51) that acetone had potential advantages as a grinding liquid.

With this background, serious studies of milling in acetone were undertaken in December, 1947. In general, acetone proved to offer clear cut advantages in rate of grinding and, to a lesser degree, in ultimate strength (see Figure III) and, of particular importance, to eliminate some of the difficulties resulting from residual kerosene in the crudes. Calculations by the Design Division indicated that the recovery stills already under construction in the Newport plant could be used with minor modifications to recover 98% acetone at an acceptable rate. Higher purities, if needed, would seriously reduce capacity. (See Figure IV for effect of purity).

Studies of the mill loading were repeated using commercial 99.5% acetone and covering in more detail the range of M/V 1.25 to 2.0 and P/Ve 0.03 to 0.07. Curves and tables are appended summarizing these studies (see Table II and Figures IX to XII. The optimum M/V again was close to 1.5 with relatively minor differences up to M/V 2.0. The optimum P/Ve, on the other hand, was somewhat lower, around 0.05. In spite of this the rate in lbs. per hour was about 25% higher than with isopropanol and the ultimate strength in a reasonable cycle was about 5% greater.

The only apparent drawback to the use of acetone was a substantial increased usage of solvent per lb. of pigment due to (1) higher dilution during grinding, and (2) use of acetone instead of water for flushing the mill to avoid dilution by water left in the mill. Careful calculations indicated that the stills could handle the load and it was decided to proceed with use of acetone in the plant. This decision has been amply justified in the successful operation of the plant for over two years. The stills with slight modifications have more than met the original estimates.

D. Treatment of Pigment after Milling

1. Separation from grinding liquid.

In all cases the grinding liquid has been removed by distillation with open steam. During the period of grinding in hydrocarbon solvents, various expedients were tried to make the steam distillation step more successful, particularly to avoid balling and to promote contact of the liquid and pigment with the water and steam. Emulsifying agents in strong alkaline solutions were more or less successful in the laboratory, but caused foaming in the semi-works. The final process involved simply a mechanical emulsification by vigorous agitation. This is no longer of interest in the problem.

With both isopropanol and acetone, no serious attempt has ever been made in the laboratory to recover the solvents. It has been removed by dilution with water and distillation in a hood with open steam. Recovery stills in the semi-works and, later, in the plant have operated as contemplated using both open steam via spargers and jacket heat on the still to give either 91% isopropanol or 98% (or better) acetone. In general, no chemical treatment has been included at this point and no serious problems of corrosion or the like have been encountered.

2. Extractions

The Pigments Department inherited a tradition from Orchem that all phthalocyanine pigments require both an acid and an alkaline, preferably ammoniacal, extraction to be satisfactory for all uses. No completely adequate studies of the required extraction procedure for CPC-LB have been made to this date in the Pigments Department. The extraction of the crude prior to grinding is in alkaline suspension (actually ammoniacal due to the urea decomposition products present) but is probably inefficient because of kerosene present. It is certain that there are copper salts and usually some iron salts present in the product discharged from the ball mill. There is also the probability of some metallic iron resulting from the abrasion of the balls and not removed by the magnetic filters. Accordingly an acid extraction is indicated and has always been applied.

The choice between HCl and H_2SO_4 has never been clear cut as to quality in the few trials made and has been resolved in favor of H_2SO_4 as the less expensive and the more easily handled acid. Concentrations in the range of about 2% to 4% have been used, again without clear cut evidence in support of any particular figure. The plant was designed to use 2% acid.

The laboratory arbitrarily selected and has continued to use a 30 min. period at the boil for the acid extraction. The semi-works and plant have used longer periods, from 1-2 hours, but the variable has not been fixed by study.

The evidence supporting the need for an alkaline (ammoniacal) extraction is conflicting. It was originally believed necessary to neutralize any residual acid and to complete the removal of cupric oxide not always soluble in acid. Analytical data indicate that the acid extraction is removing all the ionic copper and it is equally clear that the filter cake following acid extraction can be washed substantially acid-free so that the obvious need for this operation disappears and a number of plant charges in which it was omitted have recently been made with no apparent harm.

On the other side, it must be noted that during the semi-works operation preceding plant start-up, many samples were taken after acid extraction and were invariably weaker than the final samples. Some recent semi-works samples have also shown this tendency. To avoid a separate operation, the laboratory arbitrarily substituted an ammonia wash on the funnel to replace the separate extraction on all small samples. The wash water from this operation is always colored yellow but the impurity has never been identified. Since many of the codes made at Newark involve an additional filtration, it seems highly probable that the alkaline extraction is unnecessary and will ultimately be eliminated from the plant procedures.

3. Preparation of Lakes

Although not directly related to the manufacture of the phthalocyanine colors as such, the preparation of the various lakes offered for sale by Newark has been considered an essential part of the evaluation of the colors made. No significant changes in any of these processes were made during the period covered by this report. One new lake was prepared comprising a 50% lake of CPC-LB on a substratum of aluminum benzoate and was coded ABP-282-D. The process was a simple precipitation of the aluminum benzoate on the CPC. The process has since been modified and perfected and is reported elsewhere (KN-49-34).

In general, one can say that CPC-LB finished by solvent milling exhibits, in the various lakes, the properties to be anticipated from the properties of the toner. It is unfortunate and has been the source of endless confusion that the toner available to the Pigments Department as BT-172-D at the beginning of this work was inferior in both strength and brightness to the paste from which most of the lakes had been made. Consequently, the solvent ground CPC-LB has given toners markedly superior in strength and brightness to BT-172-D, but has given lakes barely equal and in some cases inferior to the lakes heretofore available.

E. Miscellaneous Observations

1. Chlorine-Free CPC

During the period of this report, a study on comparative methods of finishing phthalocyanine, mentioned above, brought out the extreme greenness of chlorine-free CPC when ground in isopropanol from a steam distilled crude. This greenness had been observed before but had usually been accompanied by serious weakness. In this case, however, the product was relatively strong and bright. It was, at first, viewed with little interest, but subsequent investigation of the properties of such products attracted much attention and demonstrated that it was a new crystal form (called the beta crystal form) of quite unexpected crystal stability. This product has subsequently been developed and is being sold under the name "Monastral" Fast Blue BG. It will be the subject of a separate report.

2. Grinding Crude Pigments in Paint Systems

In a preliminary consideration of the patent aspects of solvent milling, pastes ground in mineral spirits were diluted with alkyd resin to enamel consistency and compared with similar pastes made by grinding the crudes directly into paint vehicles. Surprisingly, crudes under these conditions returned enamel strengths substantially equal to that obtained with finished pigments. There was also a surprisingly increased resistance to flocculation but a tendency to greenness and dullness. The possibility of utilizing crude CPC in paint has not been widely explored, but has been confirmed to some extent by work of F&F. When sulfated crudes of higher purity became available, brighter enamels were obtained but there was considerably more flocculation. A conclusion was finally reached with respect to flocculation which has not yet been contradicted, nor fully explained. It is that the most flocculation resistant enamels of untreated CPC are obtained by grinding relatively impure crudes in paint vehicles. The enamels are, however, dull. Any steps which improve the brightness of the enamels result in more flocculation until one reaches the highly purified acid pasted CPC which, in untreated form, is the worst of the various known forms of CPC in flocculation.

3. Soft Texture Pigments.

During the period covered in this report relatively little work was done on treatments for improving texture of the CPC pigments. Some attempts to use the processes previously used by Orchen with acid pasted pigments met with only moderate success. Treatments are not required with solvent ground pigments, as they are with acid pasted pigments, to obtain full strength; but some improvements in working properties are required and have been effected in later studies.

REFERENCES:

The experimental details of the work covered in this report are recorded in the following notebooks.

N.B.	1206	Expt. 59 ff.
	1229	
	1251	
	1262	
	1275	Expts. 1-49
	1270	
	1286	Expts. 1-14

It should be noted that the results on mill loading covered in notebooks 1270 and 1286 together with many of the curves and tables presented herewith were obtained by Mr. J. S. Stage who is no longer connected with the DuPont Company. It is included in this report since it was done in close cooperation with the writer and has not been reported in detail elsewhere.

APPENDIX

TABLE I

Treating agents tried in grinds with isopropanol.

A. Those which offered some evidence of value.

Tetra sodium pyrophosphate
Disodium phosphate
MP-189
Petronate (particularly in dilute isopropanol)

B. Those which gave no evidence of improvement but which did not appear harmful.

Sodium sulfate (anhydrous)
Sodium tetra borate
Ammonium Naphthenate
Aerosol OT
Alkanol B
Caprolactam
DuPonol G
DuPonol WA
Glyceryl Salicylate
Victawet 12

C. Those agents which appeared harmful.

Caustic Soda (NaOH)
Aerosol C 61
Alkanol WXN
Alkaterge C
Ammonium Ricinoleate
Ammonium Styrene Maleamate
Armononyx 00
Armeen HT
Armid HT
Armeel HTD
Benzoic Acid
Blancol
Cation Active Compound D
O-chlor benzoic acid
CPC Sulfonate
Diethylene Glycol Monostearate
Diglycol Stearate
Fixanol
Glyceryl Monococate
Glyceryl Monoricinoleate
Glyceryl Monostearate
G-1209
Igepal CA Extra
Lissapol LS
Oleic Acid
Ortholeum 162
Para Soap
Polybenzyl sodium sulfonate
Propylene glycol monostearate
Soya Lecithin
Span 20
SP-717
TEA oleate
Tetradecylamine Acetate
Triton N-100
Triton 770
Victamine D

TABLE II

PIGMENT GRINDING RATES IN 91% ISOPROPANOL

Grams of pigment per hour in 1.8 gallon mills to strength level indicated.

M/V by vol.	Str. vs BT-172-D	P/Ve (by vol.) 0.030	0.040	0.050	0.075	0.100	0.125	0.150
1.0	110% Str.			3.32	5.10	5.79	5.67	5.85
	120% Str.			1.09	1.65	1.76	1.77	1.81
1.5	110% Str.			5.19	6.67	5.98	3.45	2.34
	120% Str.			1.79	1.95	1.74	-	-
2.5	110% Str.		3.27	4.38	4.31	3.32		
	120% Str.		-	-	-	-		
4.0	110% Str.	2.82	4.28	4.22	4.70			
	120% Str.	-	-	-	-			

IN ACETONE

M/V by vol.	Str. vs BT-172-D	P/Ve (by vol.) 0.03	0.04	0.05	0.06	0.07	0.08	0.09	0.10
1.25	115%	3.45	5.22	6.17	6.98	7.35	7.87	8.10	5.90
	124%	1.11	2.28	2.30	2.25	2.02	1.67	1.44	1.42
1.50	115%	9.93	12.13	14.08	15.77	16.68	14.18	7.17	
	124%	2.44	2.79	2.69	2.63	2.14	1.42		
1.75	115%	7.43	8.96	10.20	11.00	11.4			
	124%	2.33	2.64	2.66	2.26				
2.0	115%		8.10	9.55	10.42	11.11			
	124%		2.45	2.53	2.26				

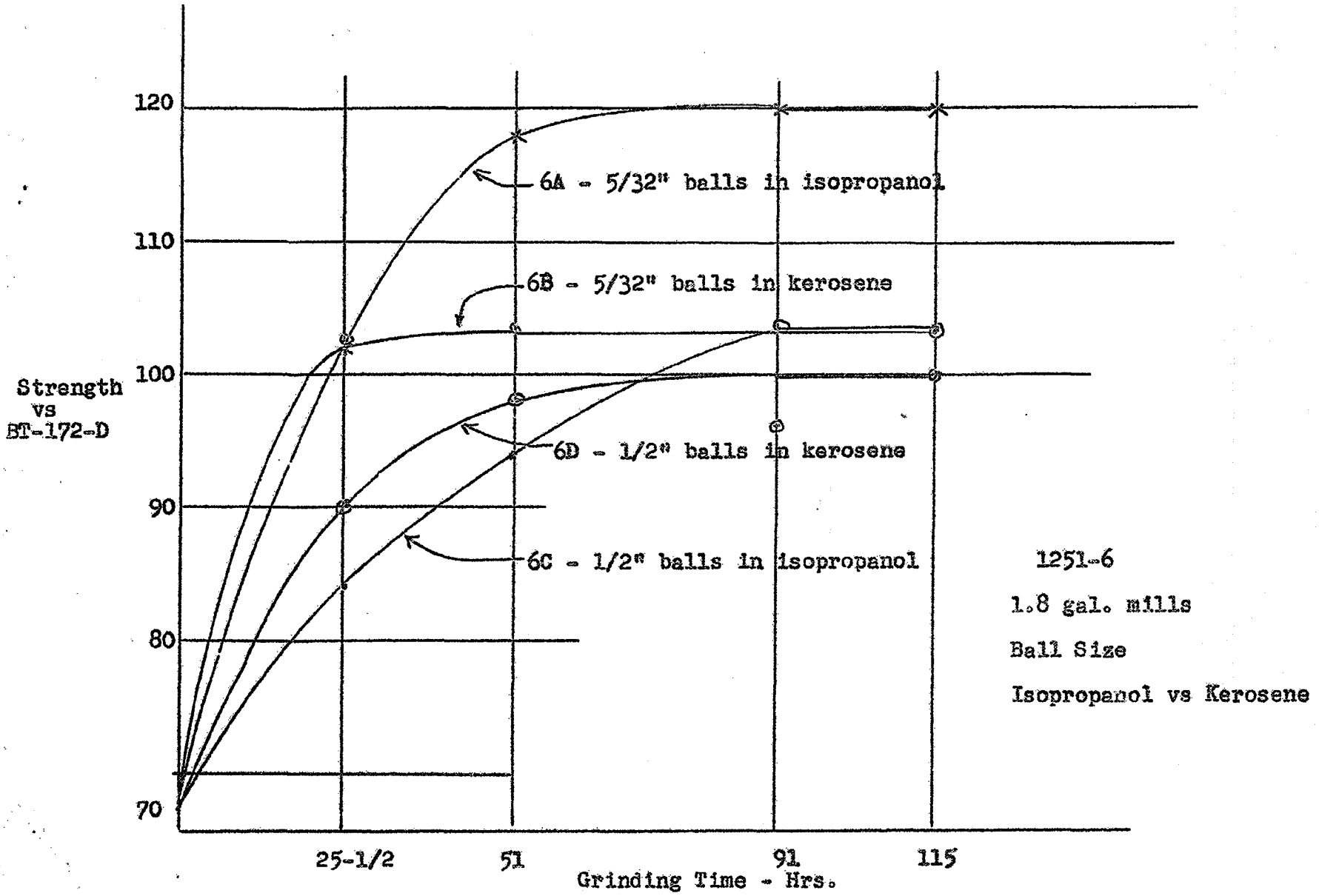
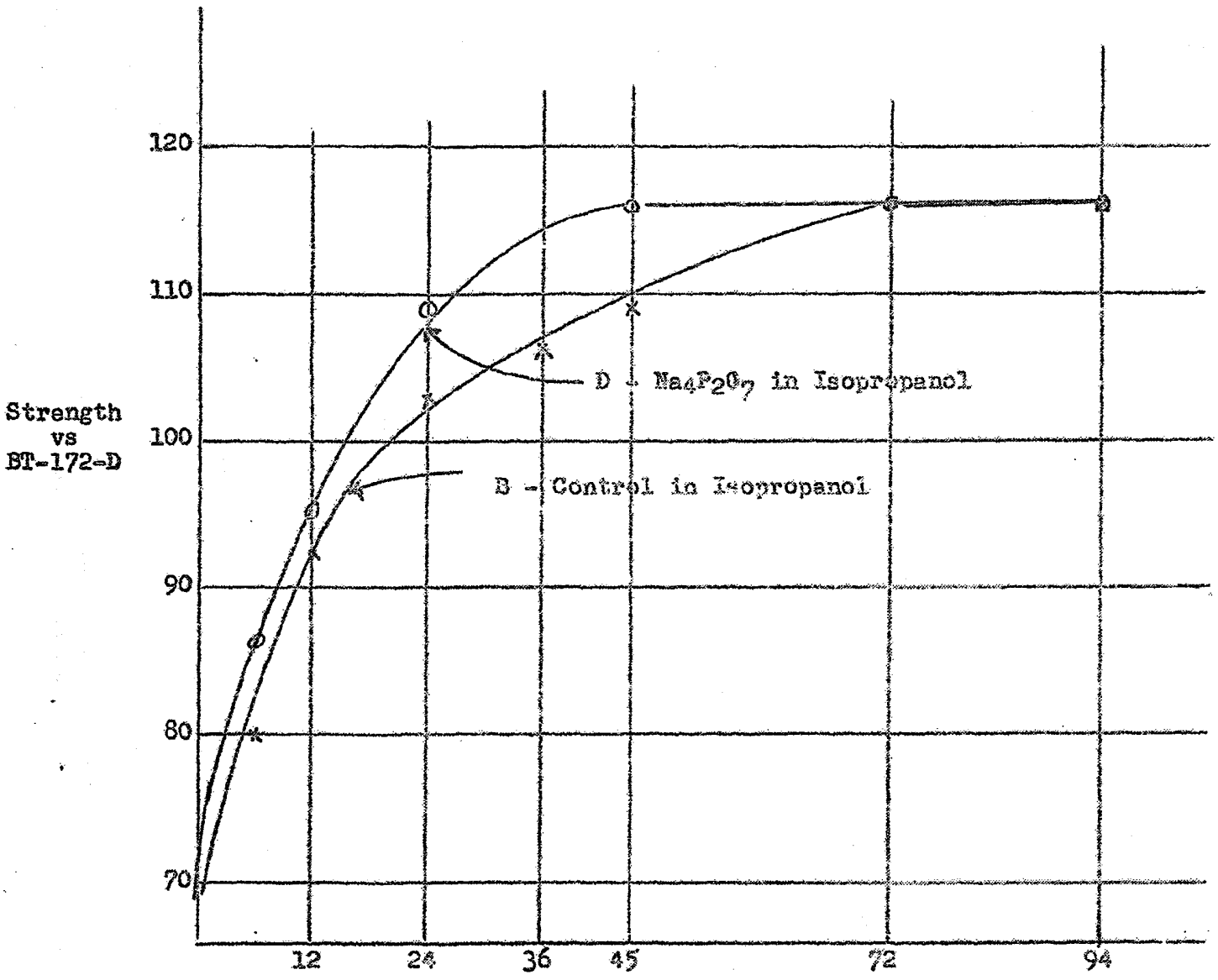


Figure I



1251-10
Use of $Na_4P_2O_7$

Figure II

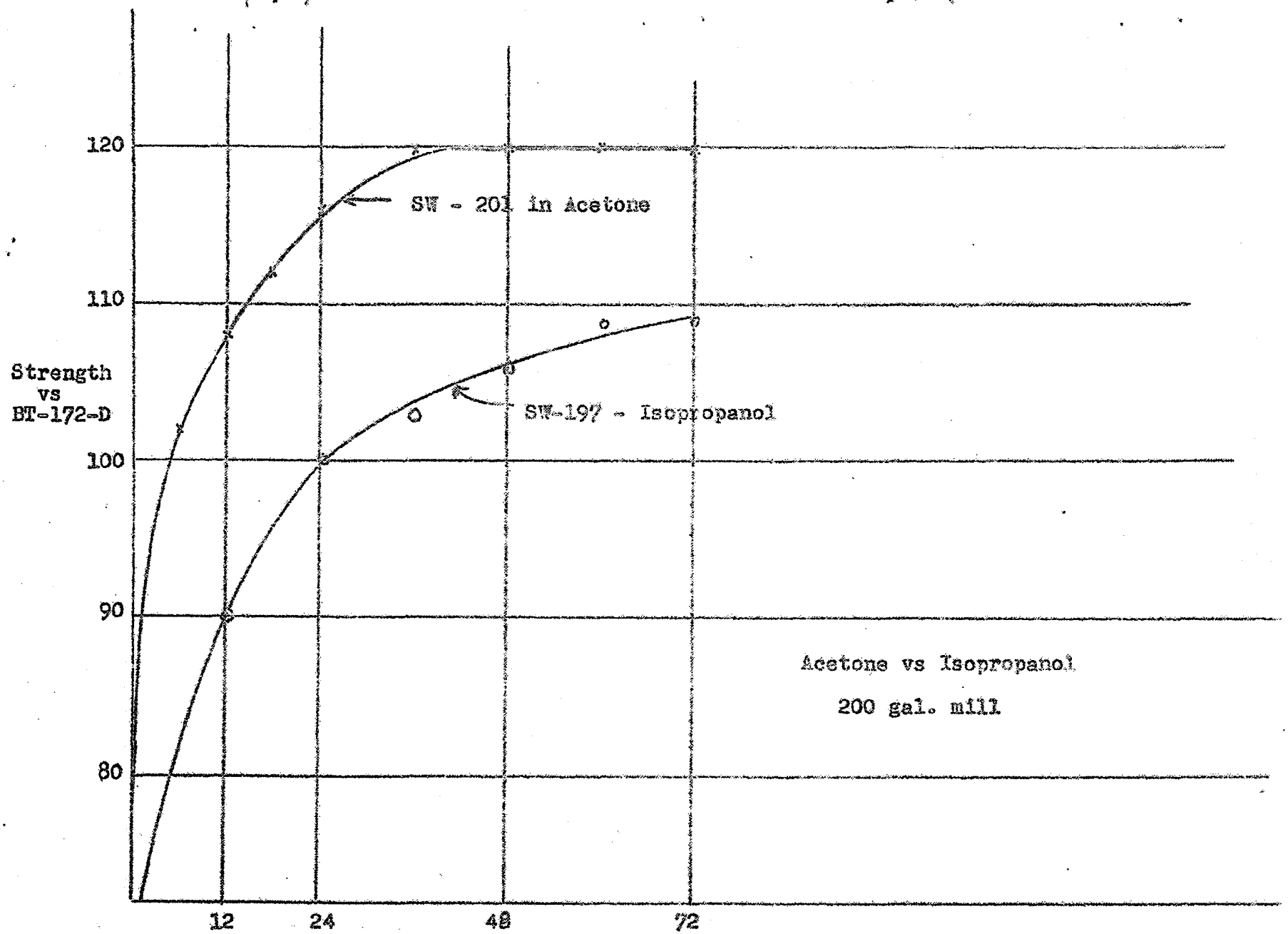


Figure III

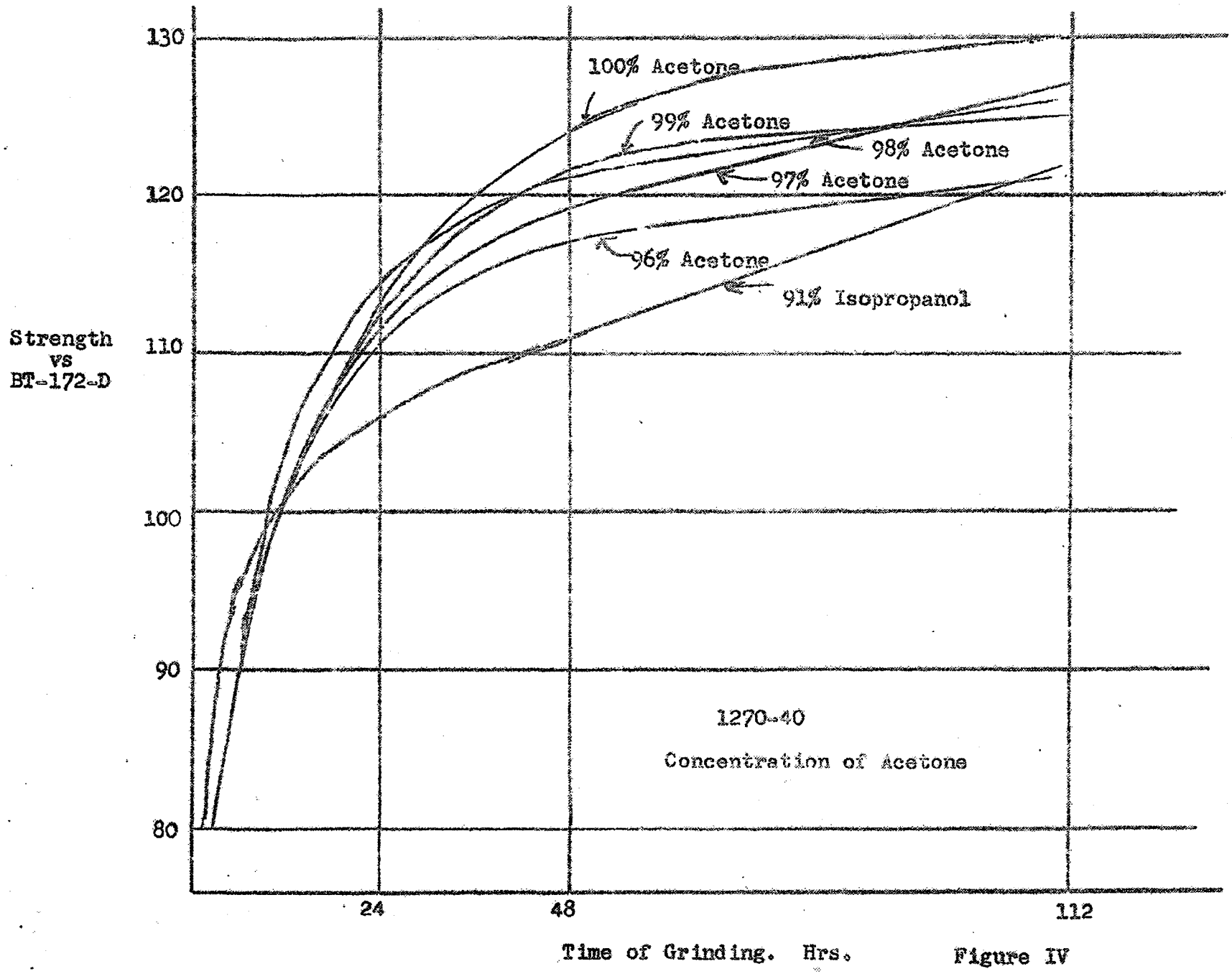


Figure IV

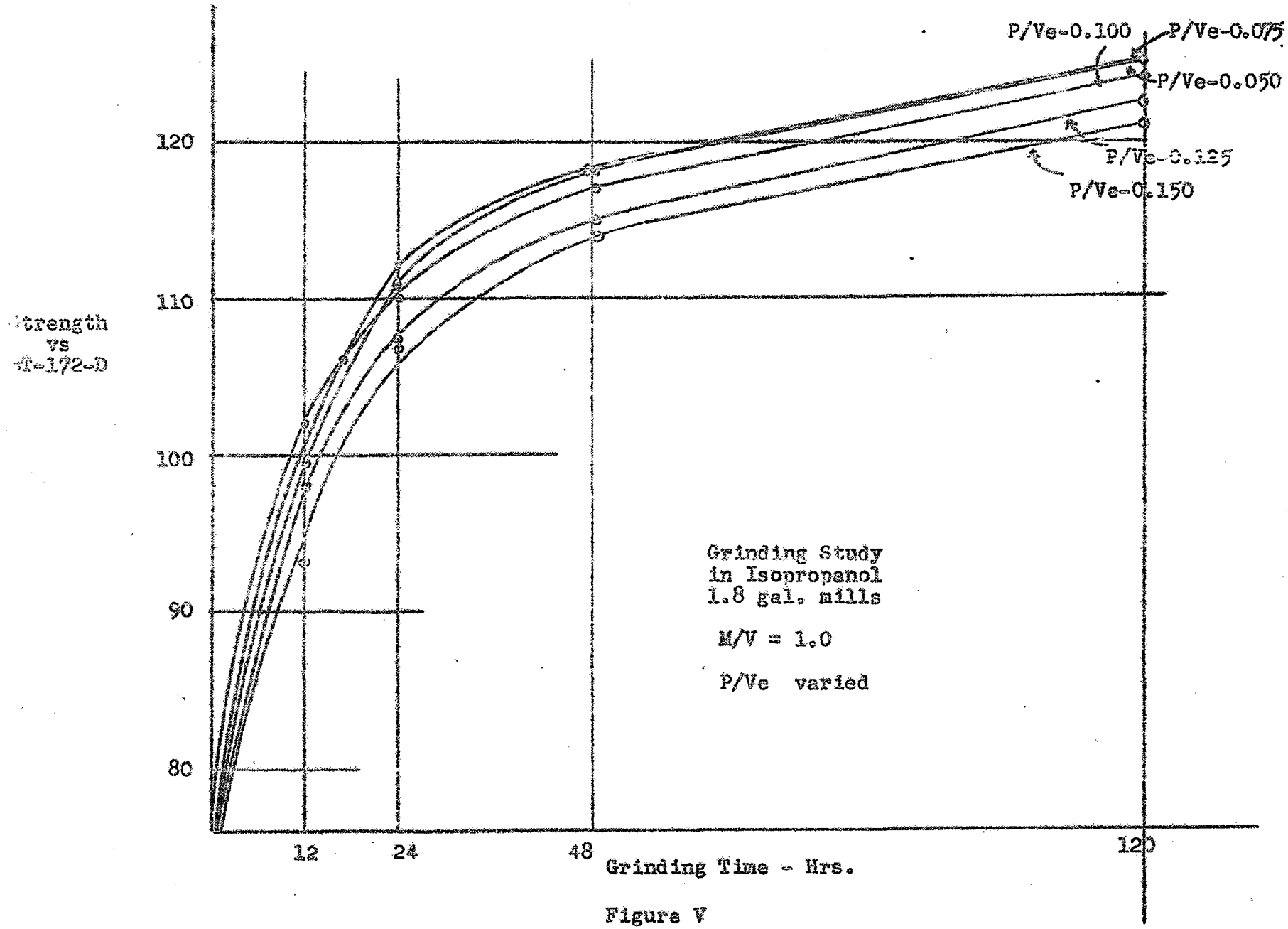


Figure V

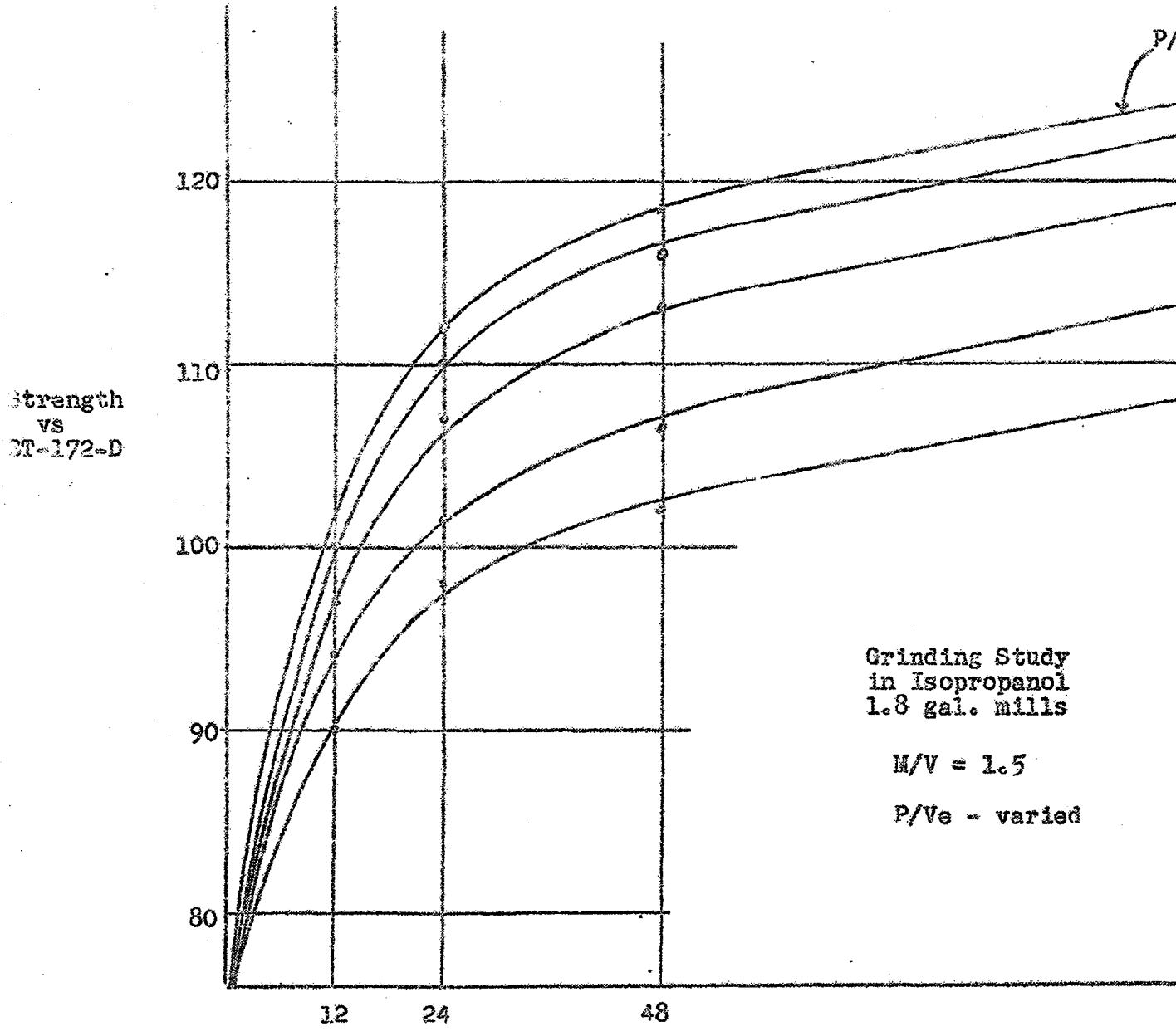


Figure VI

DUP050068370

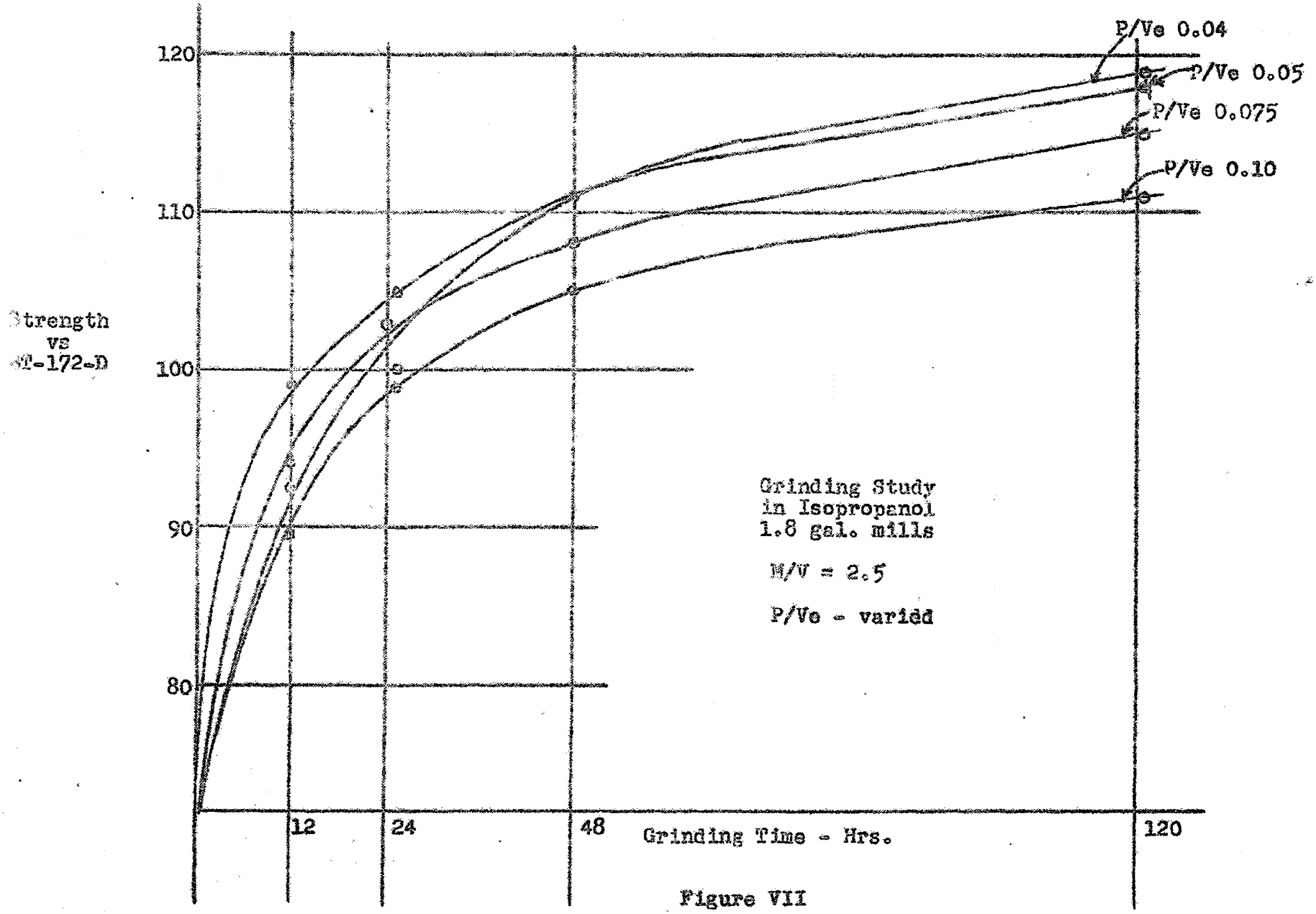
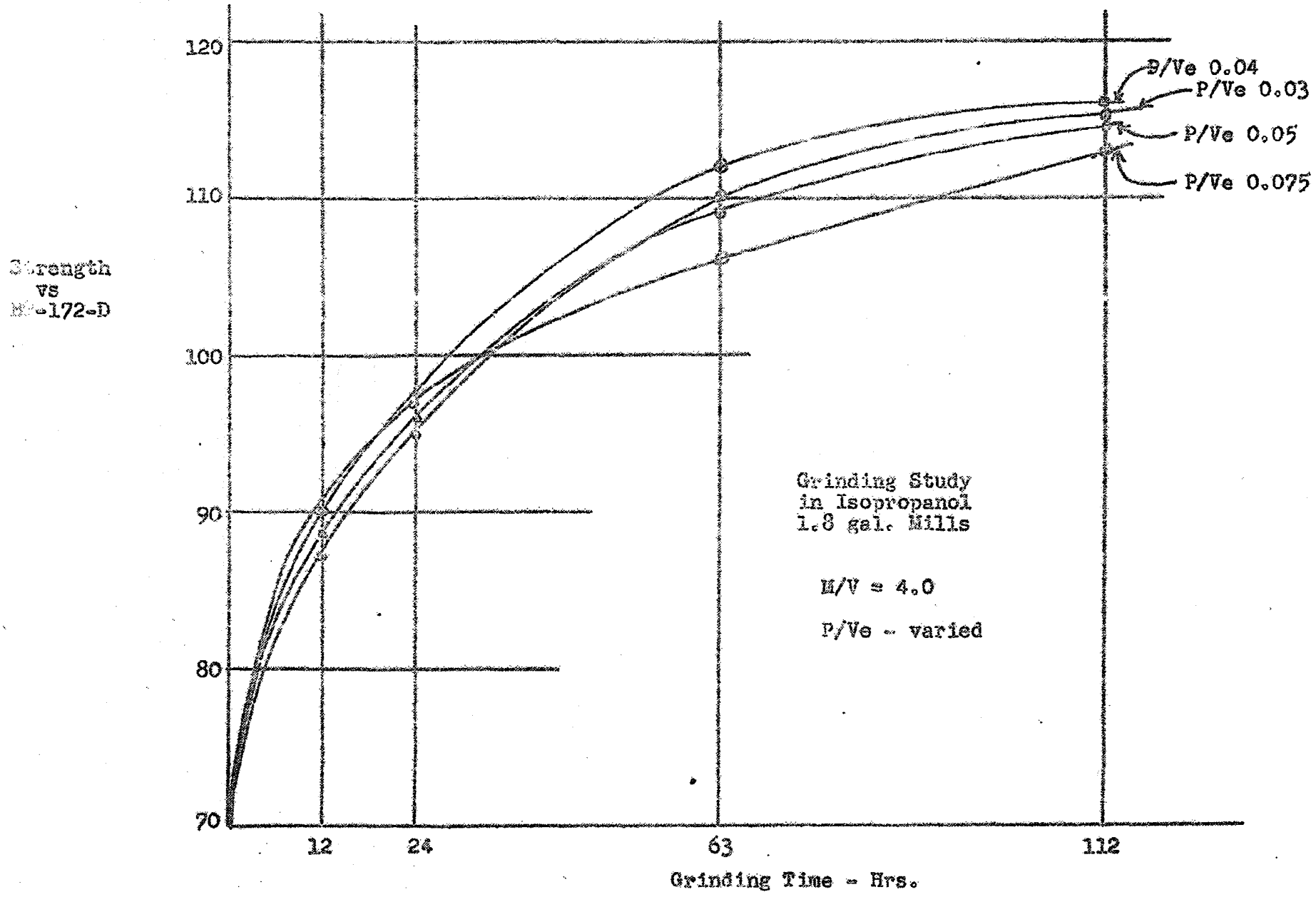
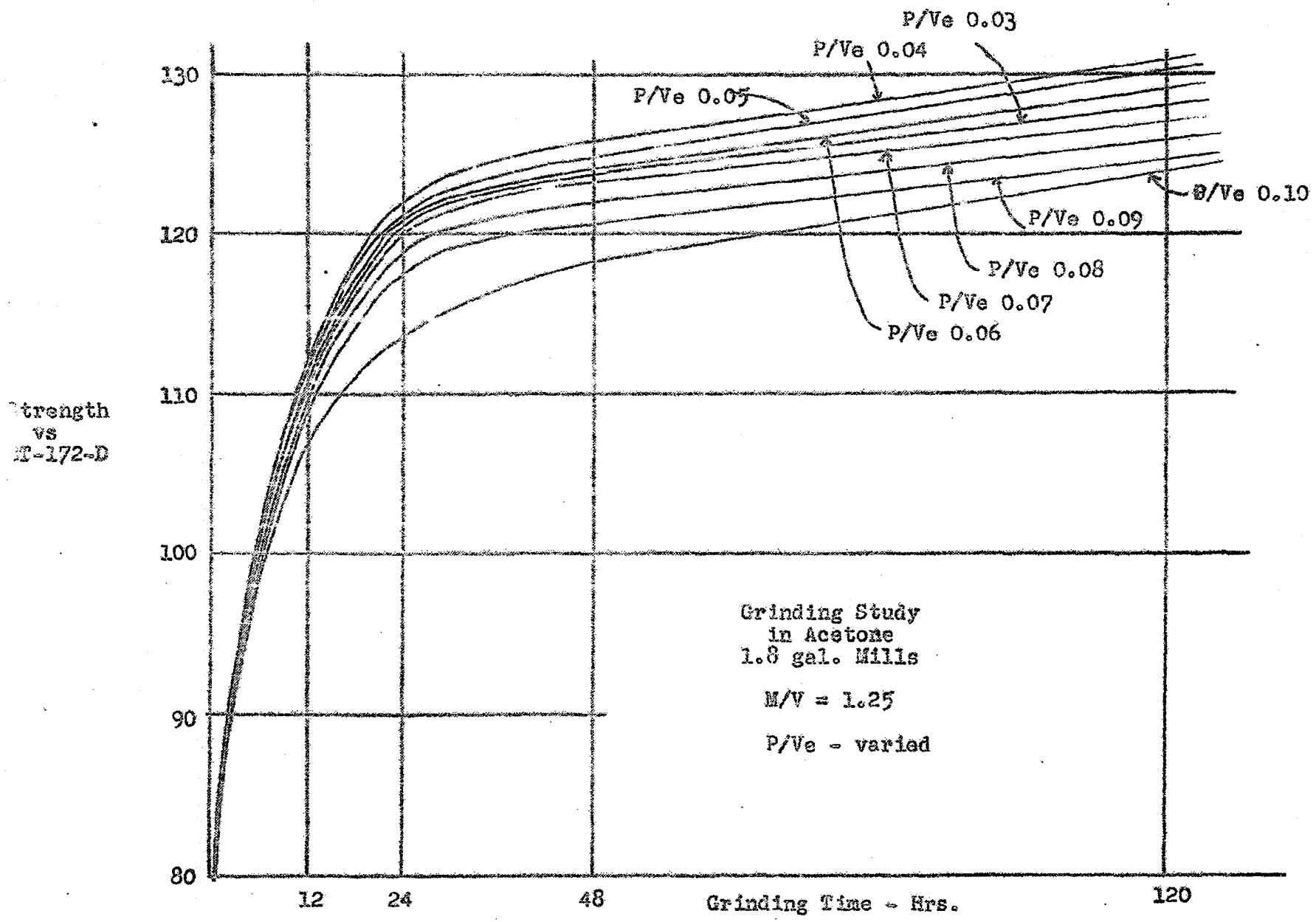


Figure VII



Strength
vs
M-172-D

Figure VIII



trength
vs
T-172-D

Figure IX

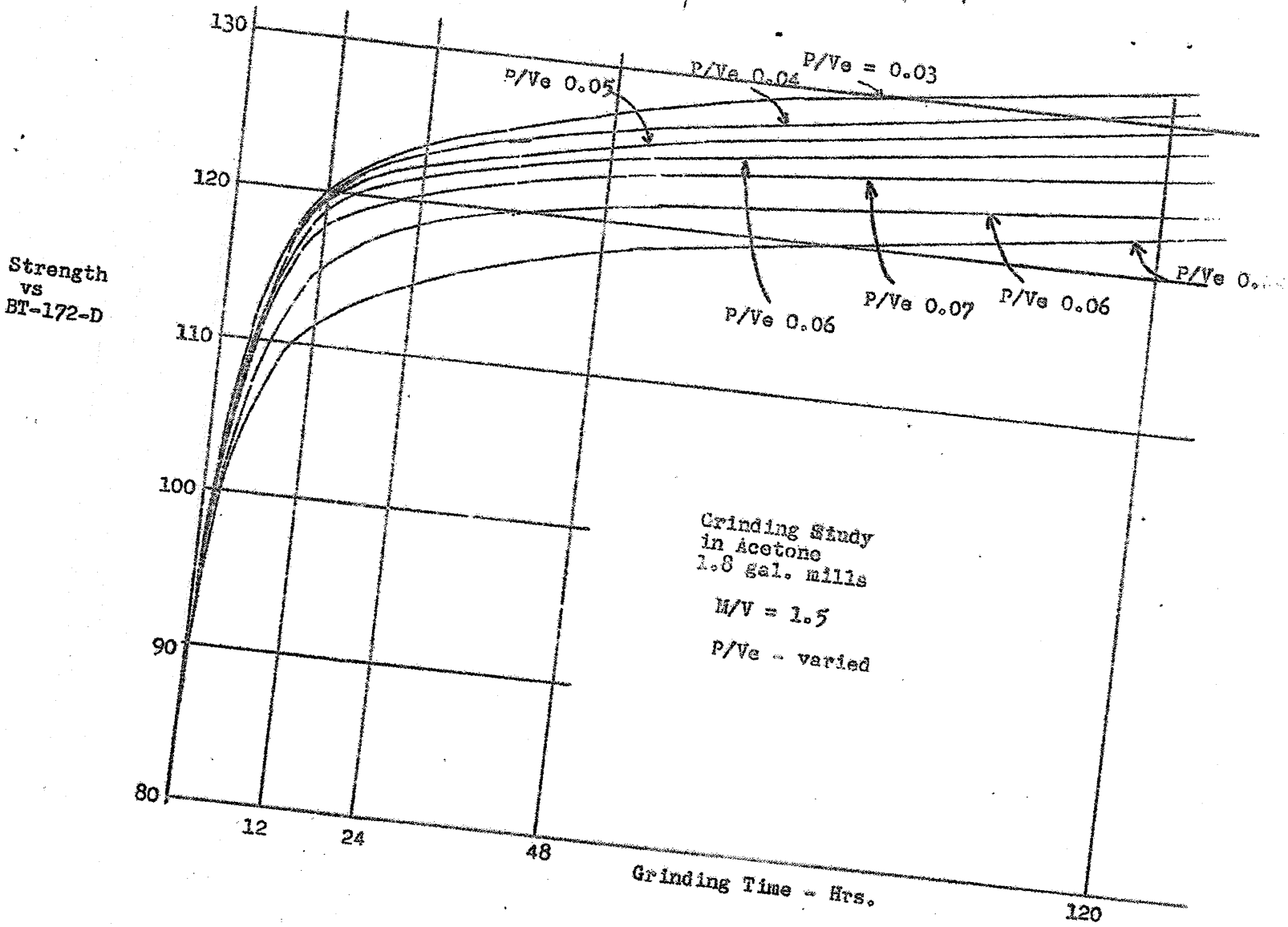


Figure X

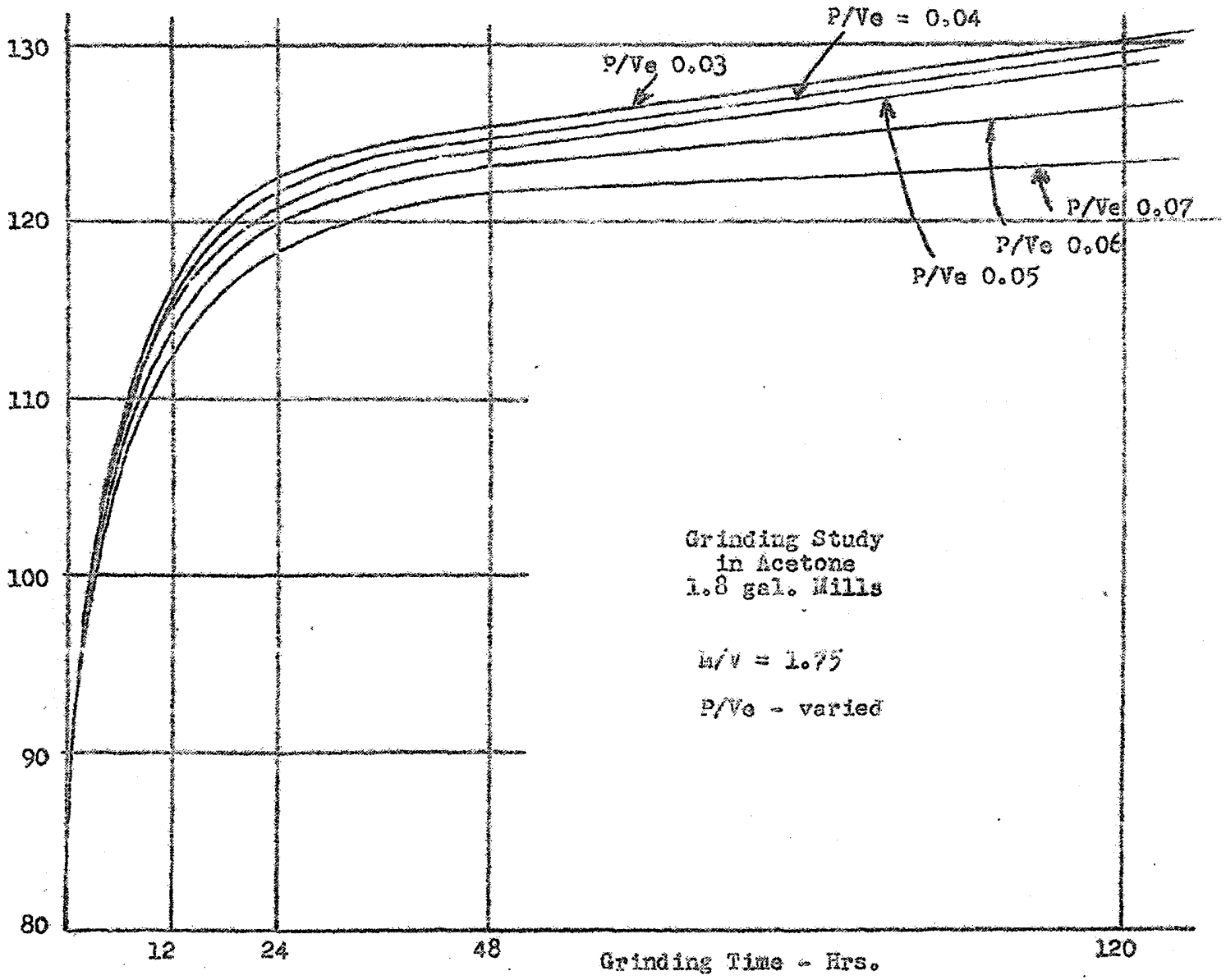


Figure XI

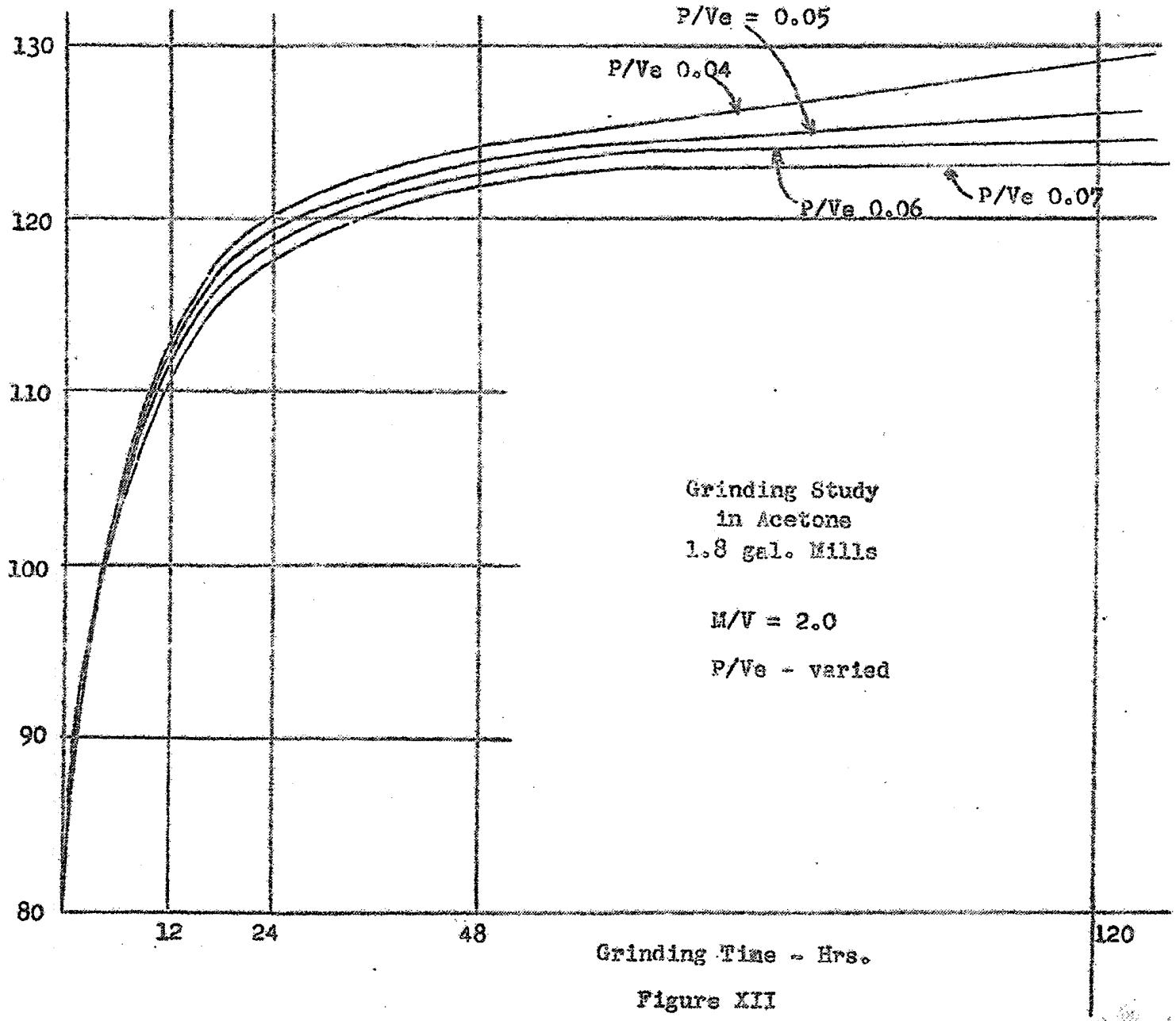
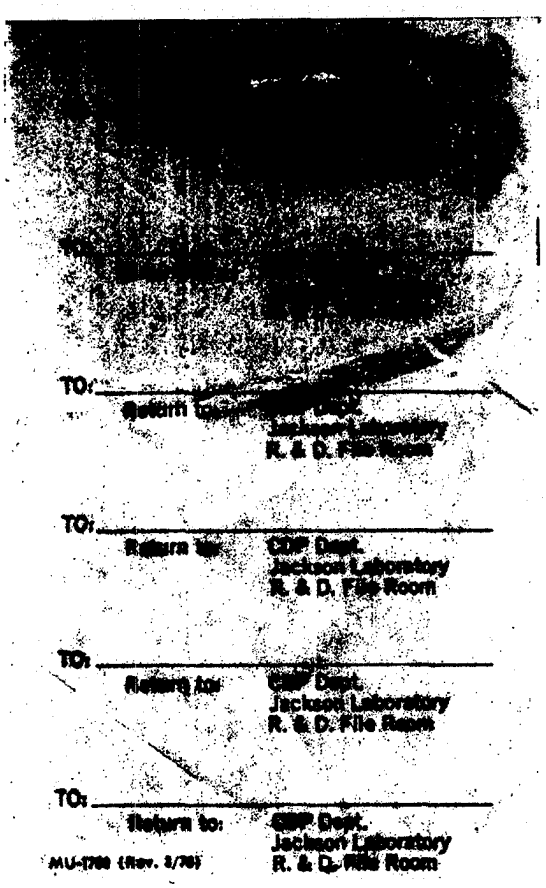


Figure XII



TO:

Return to: CBP Dept.
Jackson Laboratory
R. & D. File Room

TO:

Return to: CBP Dept.
Jackson Laboratory
R. & D. File Room

TO:

Return to: CBP Dept.
Jackson Laboratory
R. & D. File Room

TO:

Return to: CBP Dept.
Jackson Laboratory
R. & D. File Room

MU-1788 (Rev. 3/78)