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A SONIC SHEAR METHOD FOR DETERMINATION OF SHEAR BREAKDOWN ON HYDRAULIC FLUIDS AND LUBRICATING OILS

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FOREWORD

This report was prepared by the Organic Materials Branch and was initiated under Project No. 7331, "Hydraulic Fluids", Task No. 73313, "Hydraulic Fluids", formerly RDO No. 613-15, "Hydraulic Fluids and Lubricants". The report was administered under the direction of the Materials Laboratory, Directorate of Research, Wright Air Development Center, with 1st Lt A. J. Gironda acting as project engineer.

ABSTRACT

A procedure has been devised which, within short test periods, will evaluate the shear stability of Hydraulic Fluids and Lubricating Oils containing polymeric materials. This procedure utilizes the Raytheon, 10KC, Magnetostrictive Oscillator Model DF-101. The test fluids are subjected to sonic vibration for a specified length of time, after which viscosity measurements are taken in order to determine the extent of molecular decomposition. Data of typical shear-time relationship of several hydraulic fluids are presented.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDER:

M. R. WHITMORE
Technical Director
Materials Laboratory
Directorate of Research

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I INTRODUCTION

In order for hydraulic fluid and lubricating oils to function efficiently, it is necessary that these fluids and oils withstand within limits any shearing conditions that would alter their molecular structure and consequently change their physical properties. Since this problem exists today and will be more critical in future programs, a test procedure was devised that would accurately and rapidly estimate the relative shear properties of these fluids.

All hydraulic fluids and some lubricating oils presently used in Aircraft equipment contain high molecular weight polymers, such as, Methacrylates, which are added for the purpose of improving and maintaining desirable viscosity- temperature characteristics.

The circulation of hydraulic fluids through a system subjects the fluid to very rapid and sudden changes in pressure as it passes through orfices and close tolerance areas. The fluid in consequence absorbs a large amount of energy which has the effect of stretching the polymeric molecules and rupturing some at the structural weak points. This is normally observed as a change in viscosity and acidity of the base fluid. Inasmuch as the physical changes caused by shear of fluids and oils may affect the efficient operation of aircraft hydraulic systems an accurate and rapid means of screening and separating from further studies those materials that shear readily or excessively was desired. Rohm and Haas Company of Philadelphia suggested the use of sonic vibration as rapid method of determining the shear characteristics of oil containing polymers.

II EXPERIMENTAL METHOD AND TECHNIQUES

A. Test Equipment

The test machine used is the Raytheon 200 Watt, 10 KC, Magnetostrictive Oscillator, Model DF-101 shown in Figure 1. The operation of this equipment is based on the fact that magnetic materials change dimensions when placed in a changing magnetic field. The magnetostrictive effect is utilized by attaching a sample cup Figure 4 to a magnetic rod Figure 4, and placing the rod within a coil of wire connected to an electronic oscillator.

The Magnetostrictive Oscillator consists of a treatment unit Figure 3 and a Driver Unit Figure 1. The treatment unit comprises a covered cup for holding the sample, a vibrating diaphragm serving as the bottom of the cup to which is attached a laminated nickel rod and a field coil incased in the stand that holds the rod and cup, which also includes a built-in circulating system for water-cooling. The acoustic transformer linkage then magnifies the small vibrations of the magnetostrictive rod into relatively large vibrations on the diaphragm, which in turns vibrates the sample of fluid at a frequency of approximately 10 kilocycles per second.

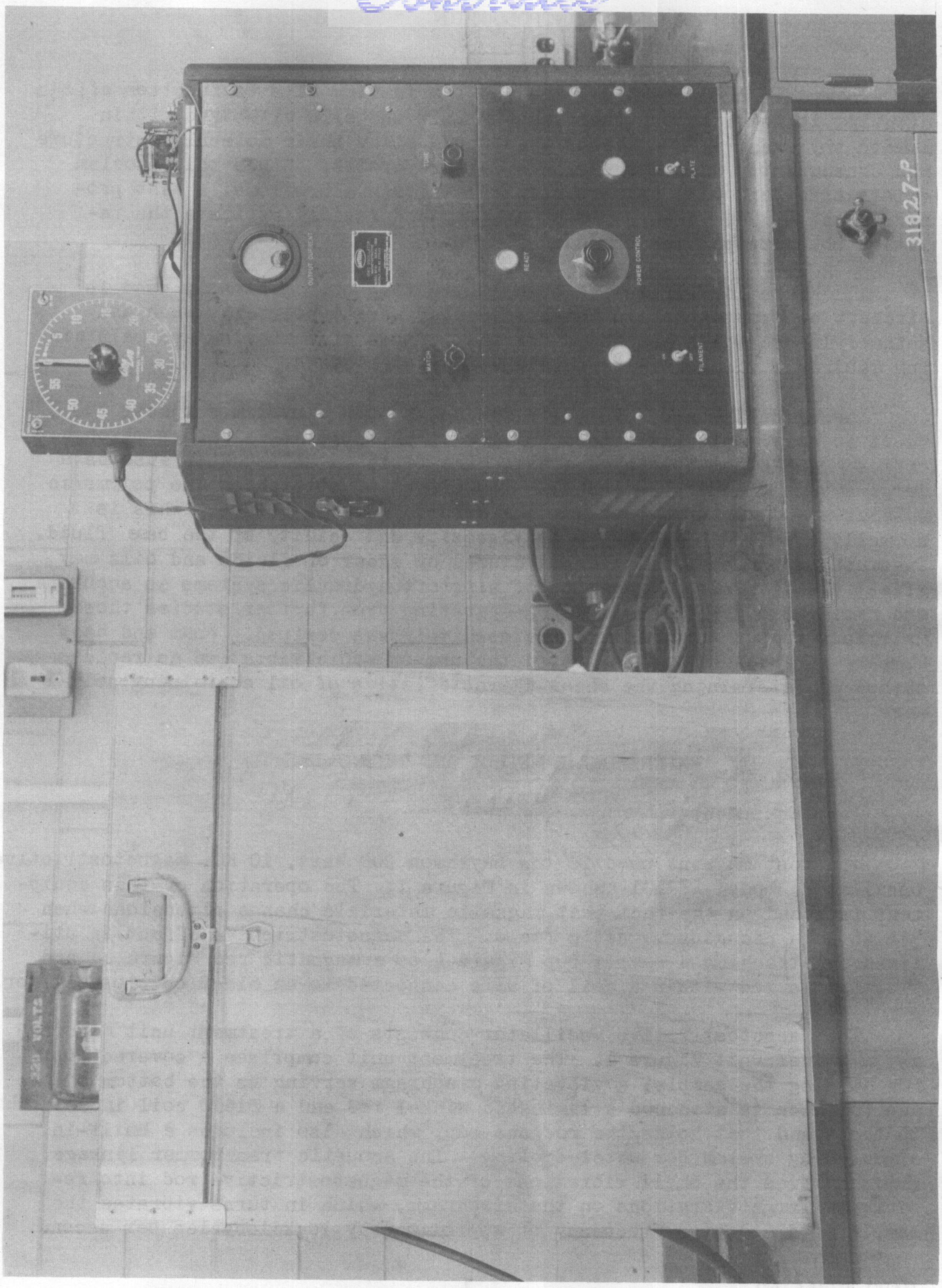

FIGURE 1 COMPLETE EQUIPMENT

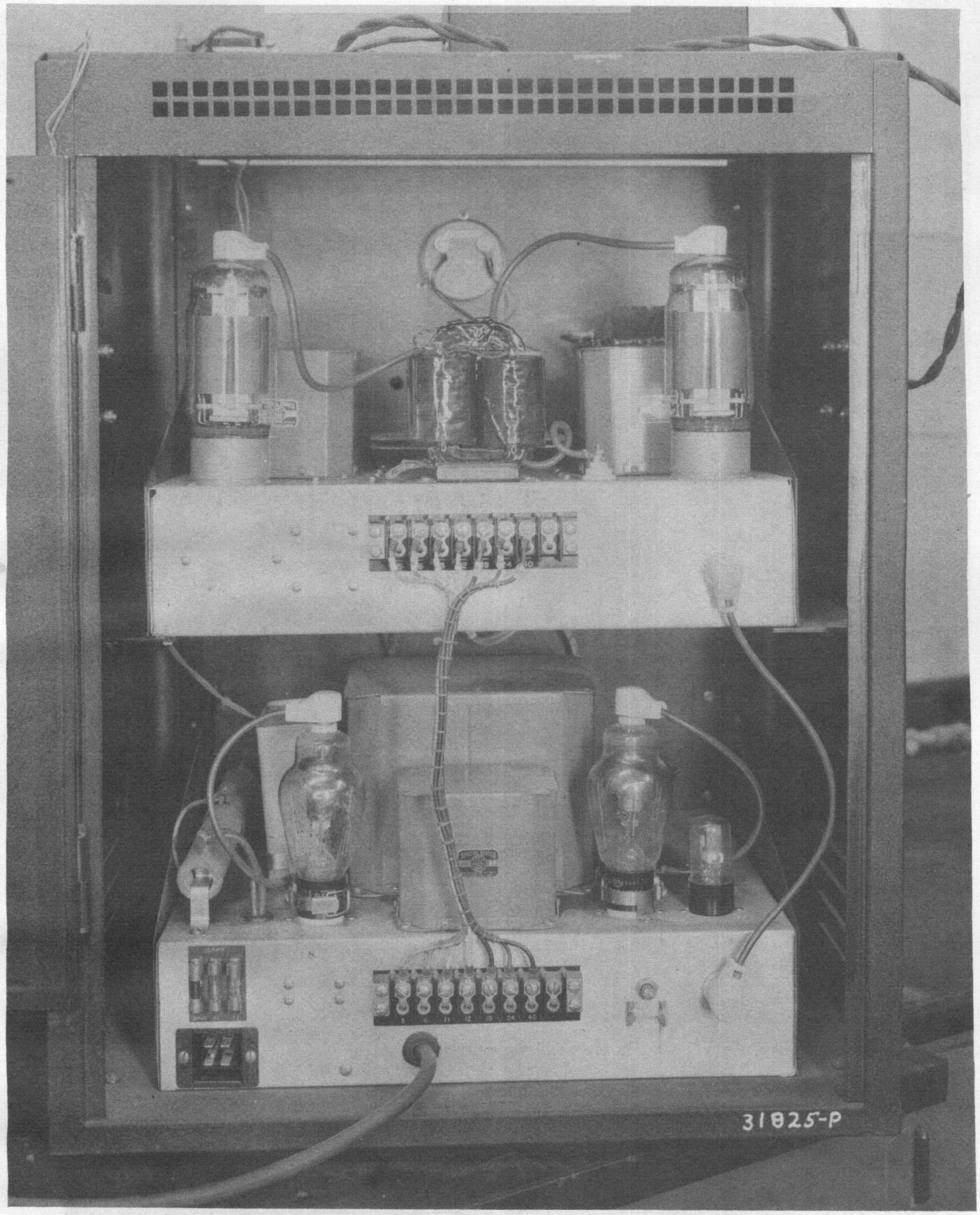

FIGURE 2 INTERIOR REAR VIEW

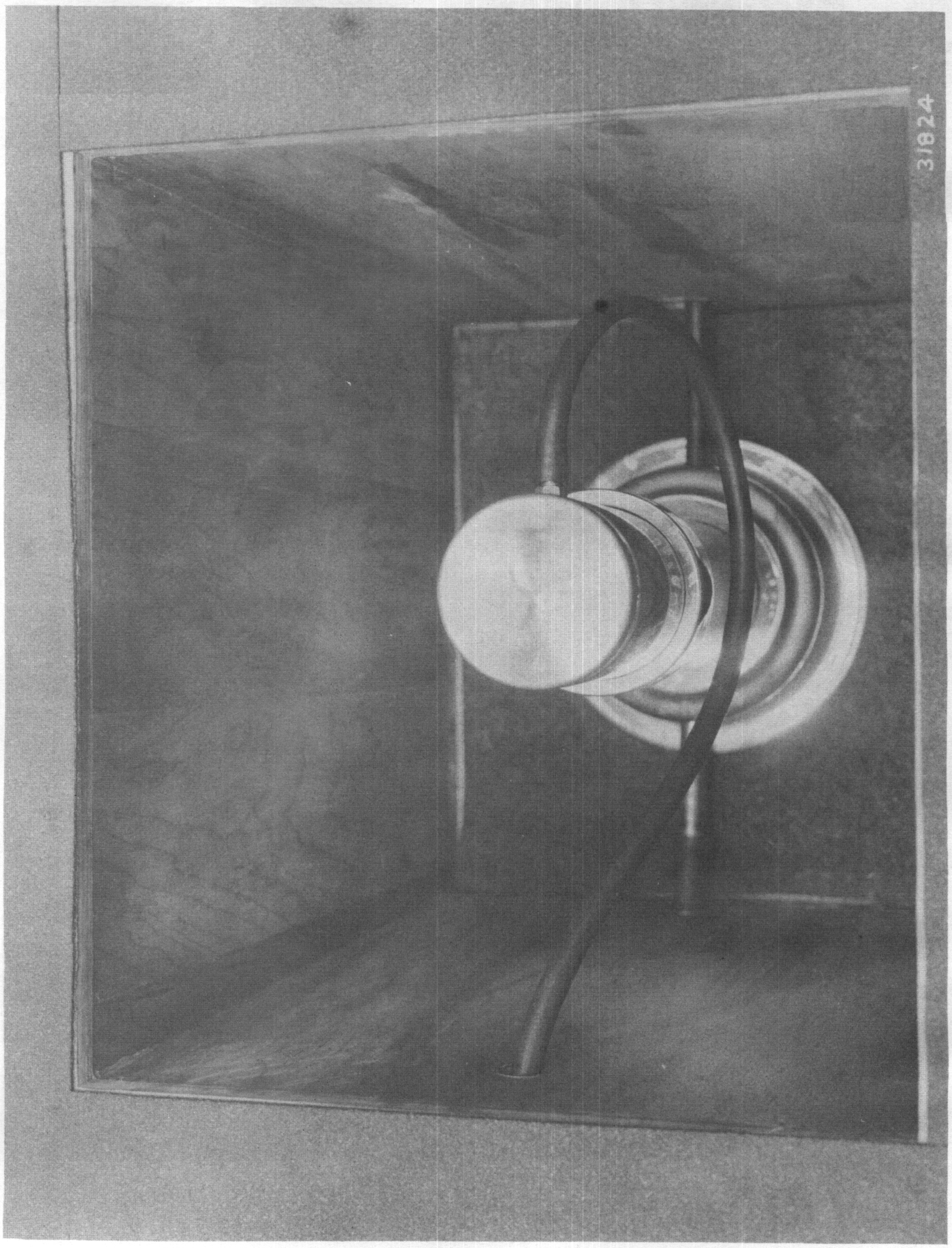

FIGURE 3 TREATMENT UNIT INSIDE OF THE SOUND DAMPENER

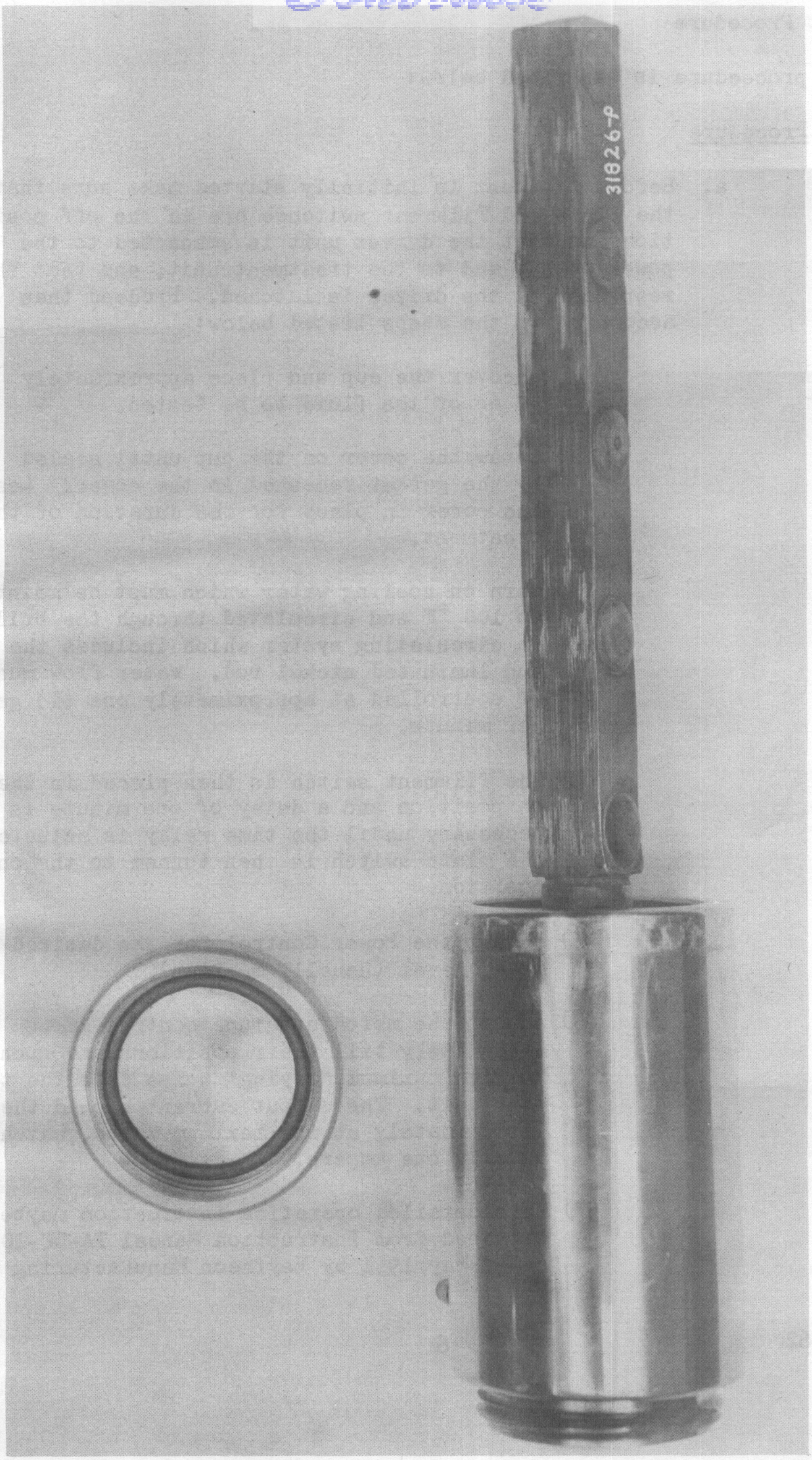

FIGURE 4 CUP AND ROD OF THE TREATMENT UNIT

B. Test Procedure

The proceedure is described below:

1. Procedure

a. Before the test is initially started make sure that the Plate and Filament switches are in the off position and that the driver unit is connected to the power outlet and to the treatment unit, and that the rear door of the driver is latched. Proceed then according to the steps listed below:

(1) Uncover the cup and place approximately 30 cc of the fluid to be tested.

(2) Screw the cover on the cup until sealed by the gasket recessed in the cover. Leave the cover in place for the duration of the treatment.

(3) Turn on cooling water which must be maintained at 100 °F and circulated through the built-in circulating system which includes the cup and laminated nickel rod. Water flow must be controlled at approximately one (1) gallon per minute.

(4) The filament switch is then placed in the on position and a delay of one minute is necessary until the time relay is actuated. The plate switch is then turned to the on position.

(5) Adjust the Power Control for the desired power level (usually maximum).

(6) Adjust the match and tune control knobs alternately till their positions are such as to give maximum "frying" noise from the treatment unit. The output current should then be approximately at its maximum value, which is usually one Ampere.

(7) More detailed operating instruction maybe obtained from Instruction Manual TA-DF-101-2, dated May 1951, by Raytheon Manufacturing Co.

Note:

The direct current (for polarizing the Magnetostrictive element) is factory set at 7.4 Amperes (with 115 volt line supply) but over a period of many months this current will fall gradually with the aging of the selenium rectifiers. It should therefore, be checked occasionally and if it has fallen below 6.6 Amperes it should be adjusted upward to a value not exceeding 7.4 Amperes. This is very important for proper operation.

III TEST DATA AND DISCUSSION

A. Time VS Shear Relationship

The effects of increasing the quantities of energy in degrading the polymer were studied on two oils containing methacrylate polymers as followed:

a. Specification MIL-0-5606, Hydraulic Oil, Aircraft Petroleum

b. Specification AN-F-53, Reference Fluid, Shear Stability

During these determinations individual runs were made at fifteen (15) minute intervals. When the shear breakdown between intervals became small in percentage the time interval was increased to thirty (30) minutes and then later to sixty (60) minutes between test runs.

The data for the two test oils are contained in Tables 1 and 2 and plotted in Figure 5 and 6.

These curves (Figure 5 and 6) are similar in shape and shear severity to those obtained by mechanical shear in a hydraulic pump shear stand. Figures 7 and 8 show typical data on hydraulic fluids containing methacrylate viscosity index improvers.

B. Repeatability

Since the time versus shear curve provided evidence of the suitability of this equipment to investigate and rate the shear stability of polymer thickened fluids, a series of test runs were made to ascertain the repeatability.

Two test fluids were used, AN-F-53, reference fluid and a synthetic type hydraulic fluid conforming to Specification MIL-L-6387 requirements. A series of twenty five (25) runs were made for periods of one (1) hour each on the reference fluid. Ten runs each were also made on the synthetic type fluid for a period of forty five (45) minutes and for a period of one (1) hour. The data for these test runs are presented in Tables 3, 4, and 5.

TABLE I

EFFECT OF SONIC IRRADIATION ON VISCOSITY OF A SPECIFICATION MIL-0-5606

HYDRAULIC FLUID

Irradiation Time Minutes	Viscosity At 130 °F Centistokes First Run	Second Run	Third Run	Percent Viscosity Decrease Average
0	10.19	10.19	10.19	0
5	9.48	9.51	9.50	6.81
10	8.93	8.93	8.97	12.21
15	8.49	8.49	8.56	16.40
30	7.79	7.89	8.00	22.52
45	7.31	7.24	7.24	28.72
60	6.82	6.92	7.01	32.11
75	6.67	6.69	6.74	34.28
90	6.51	6.58	6.60	35.59
105	6.29	6.32	6.34	38.02
120	6.15	6.21	6.26	39.08

TABLE II

EFFECT OF SONIC IRRADIATION ON VISCOSITY OF SPECIFICATION AN-F-53

Irradiation Time Minutes	Viscosity At 130 °F Centistokes First Run	Viscosity At 130 °F Centistokes Second Run	Percent Viscosity Decrease Average
0	9.61	9.61	0
15	7.66	7.72	20.00
30	7.01	7.01	27.06
45	6.66	6.60	31.01
60	6.28	6.34	34.07
75	6.09	6.05	36.84
90	5.99	5.92	38.04
120	5.70	5.62	41.12
150	5.50	5.50	42.77
180	5.29	5.31	44.85
240	5.09	5.18	46.57
300	5.00	4.99	48.03

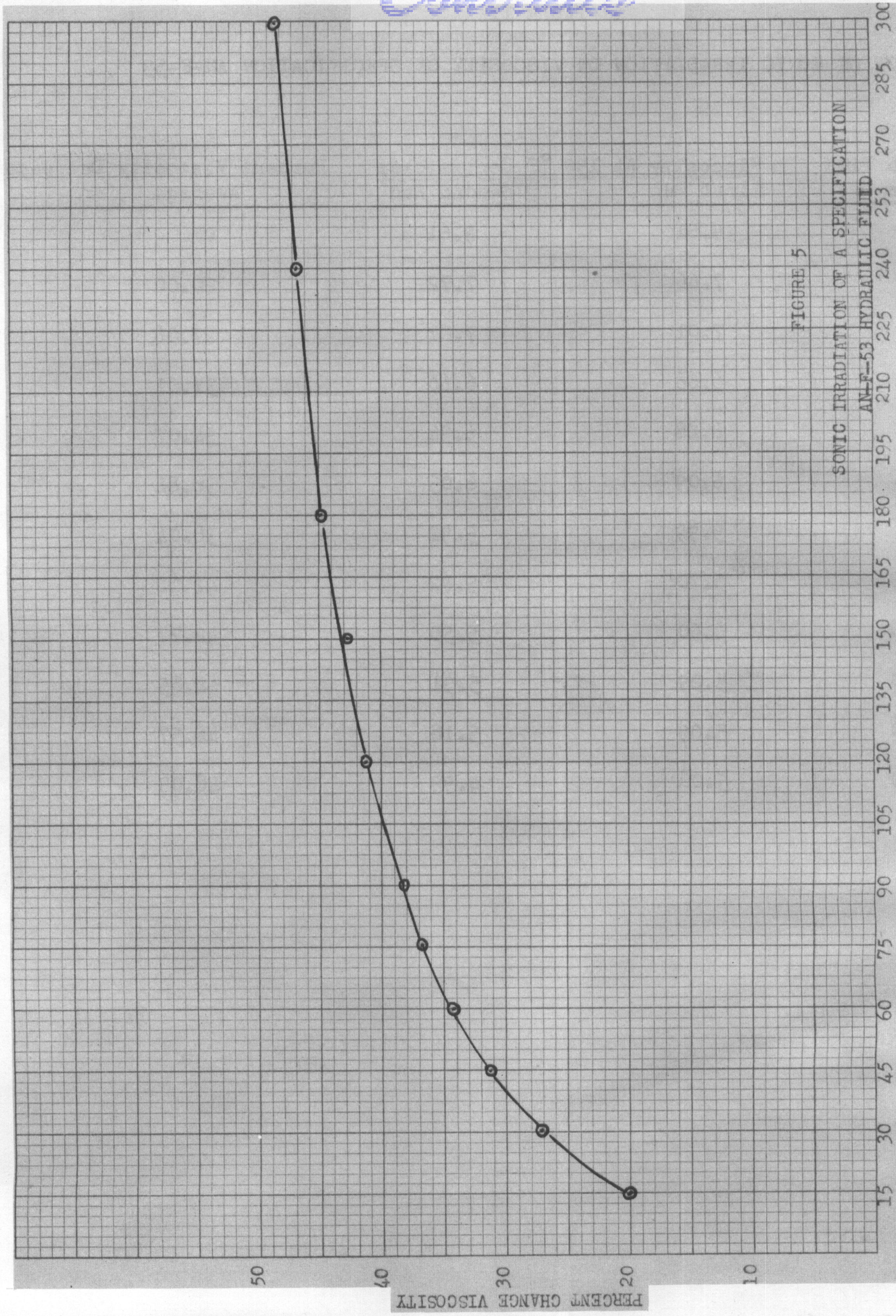

FIGURE 5
SONIC IRRADIATION OF A SPECIFICATION AN-F-53 HYDRAULIC FLUID

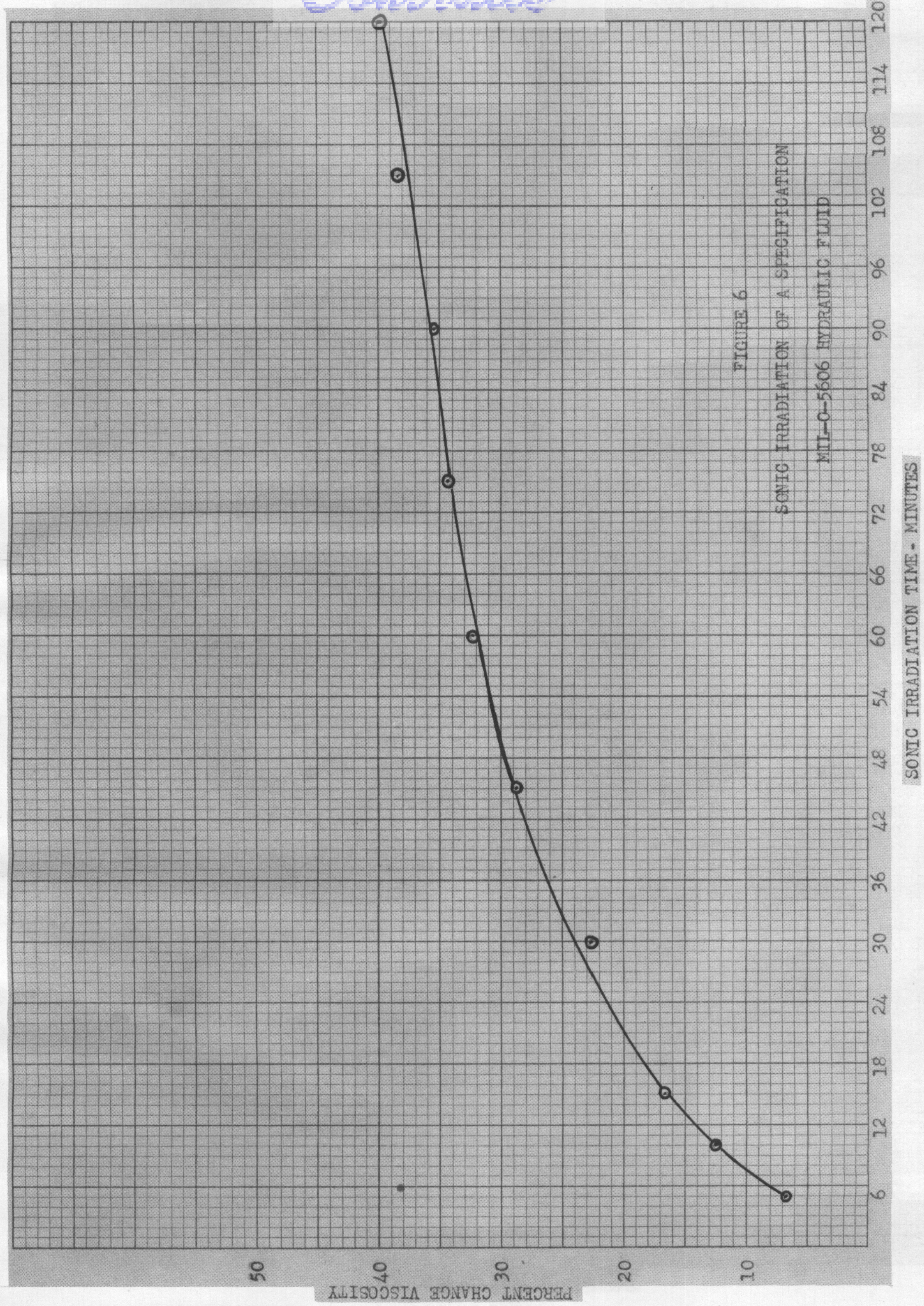

FIGURE 6

SONIC IRRADIATION OF A SPECIFICATION MIL-O-5606 HYDRAULIC FLUID

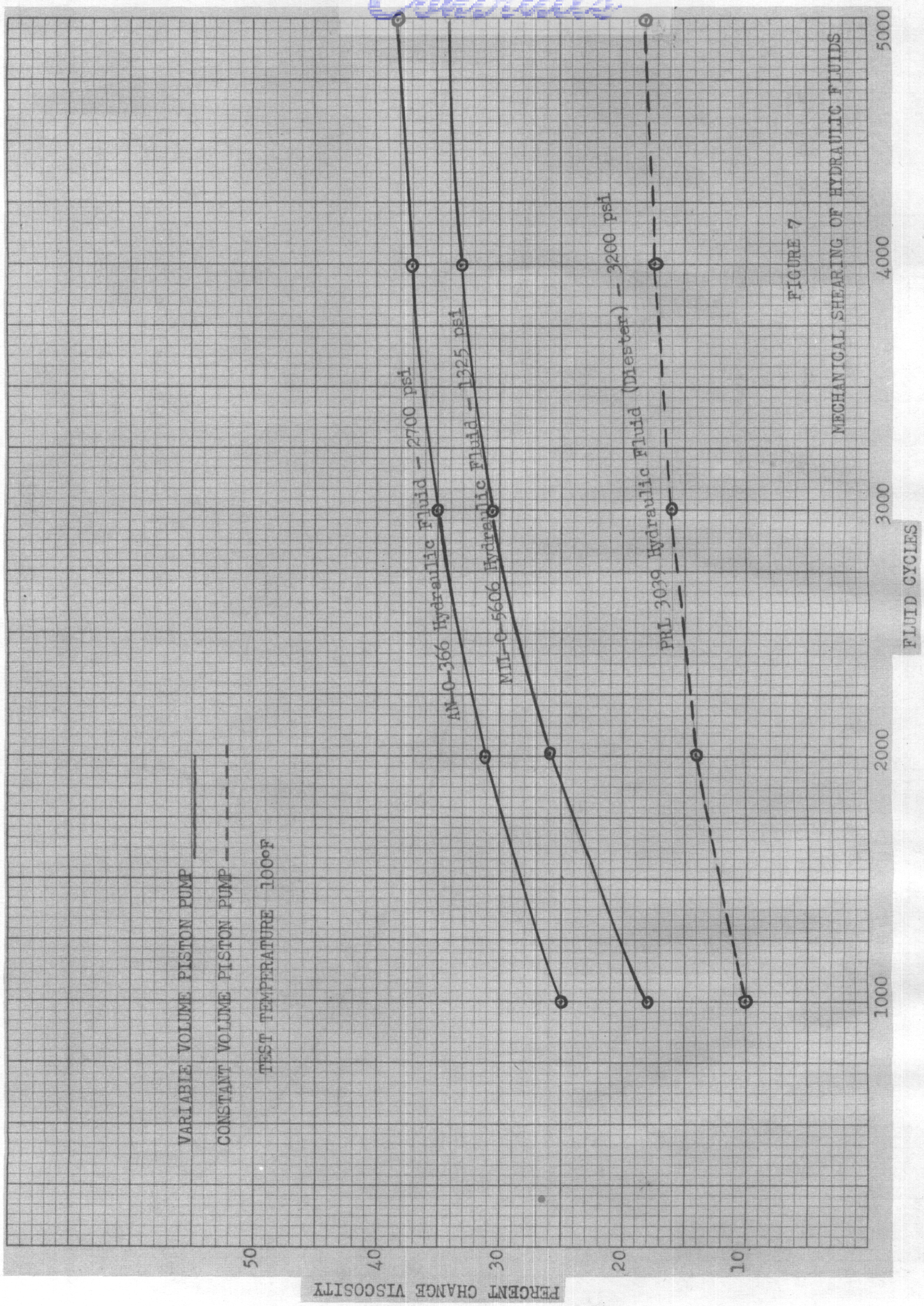

FIGURE 7
MECHANICAL SHEARING OF HYDRAULIC FLUIDS

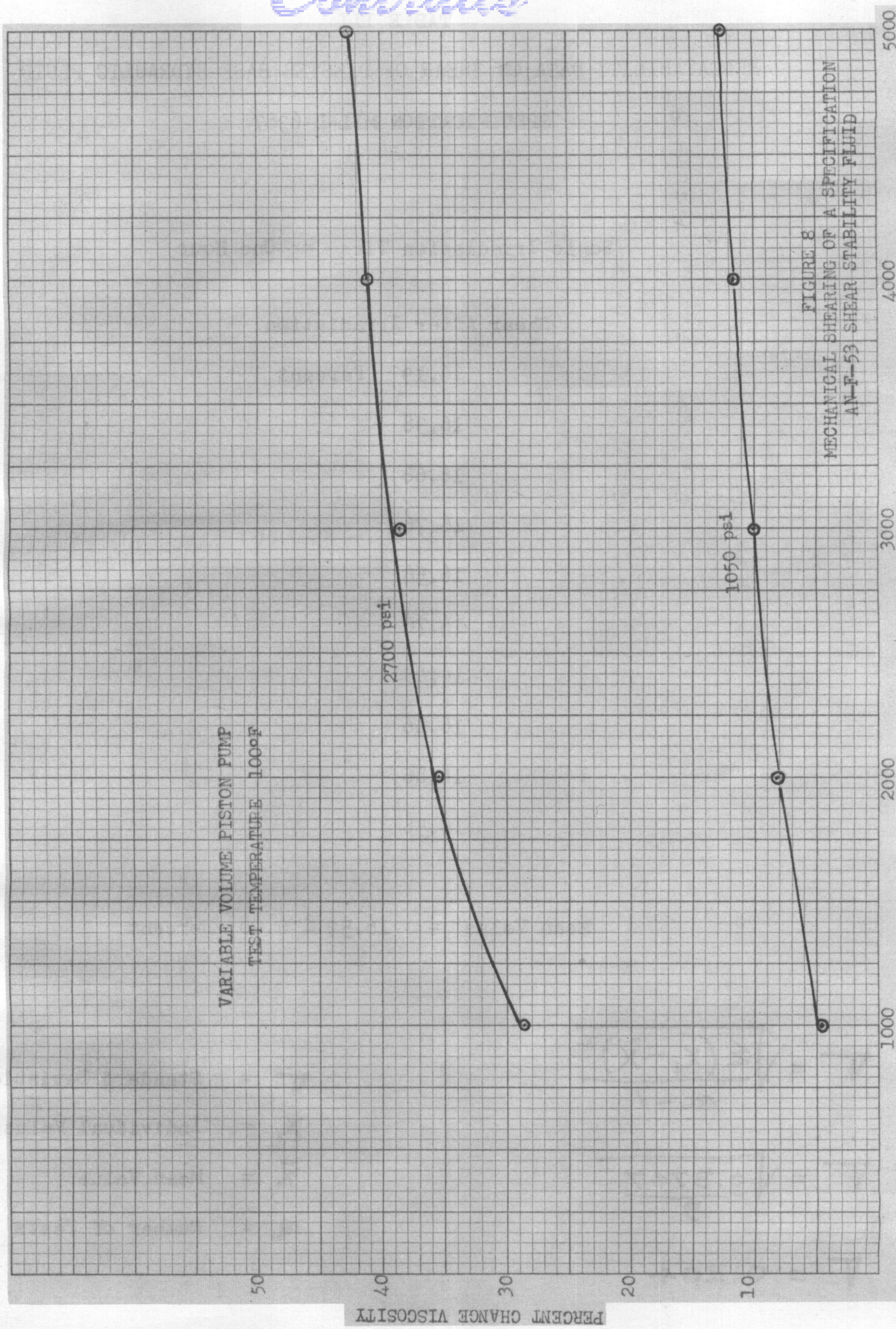

FIGURE 8
MECHANICAL SHEARING OF A SPECIFICATION AN-F-53 SHEAR STABILITY FLUID

TABLE III

REPEATABILITY DATA ON SHEAR OF DIESTER BASE HYDRAULIC FLUID

SPECIFICATION MIL-L-6387

Sonic Irradiation Time = One Hour

Shear After Irradiation

16.19 Percent

16.38

16.66

16.76

16.56

16.76

16.48

16.66

16.86

16.56

Mean Value = 16.59 ± 0.20 Percent

$$\sigma = \sqrt{\frac{\Sigma (X_1 - \bar{X})^2}{n-1}}$$

$$\sigma = \sqrt{\frac{0.3747}{9}}$$

$$\sigma = 0.204$$

σ = Standard Deviation

X_1 = Individual Values

$\bar{X}$ = Mean Value

n = Number of Tests

TABLE IV

REPEATABILITY DATA ON SHEAR OF DIESTER BASE HYDRAULIC FLUID

SPECIFICATION AN-F-53

Sonic Irradiation Time = 45 Minutes

Shear After Irradiation

Shear After Irradiation	
15.14	Percent
15.05	
14.86	
14.86	
14.38	
14.95	
14.95	
15.33	
14.95	
14.95	

Mean Value = 14.94 ± 0.24 Percent

$$\sigma = \sqrt{\frac{\Sigma (x_1 - \bar{x})^2}{n-1}}$$

σ = Standard Deviation

n = Number of Runs

$$\sigma = \sqrt{\frac{0.5310}{9}}$$

$$\sigma = 0.243$$

TABLE V

REPEATABILITY DATA ON SHEAR OF REFERENCE FLUID

SPECIFICATION AN-F-53

Sonic Irradiation Time = One Hour

Shear After Irradiation

33.81	Percent	34.11	Percent
33.64		34.81	
33.72		33.56	
33.72		34.90	
33.56		34.36	
34.27		34.11	
33.72		34.19	
33.56		34.11	
34.54		34.11	
34.27		34.27	
34.27		33.72	
34.11		34.19	

Mean Value = 34.07 ± 0.38 Percent

$$\sigma = \sqrt{\frac{\Sigma(x_1 - \bar{x})^2}{n-1}}$$

$$\sigma = \sqrt{\frac{3.2611}{23}}$$

$$\sigma = 0.377$$

σ = Standard Deviation

n = Number of Runs

TABLE VI

STATISTICAL ANALYSIS OF REPEATABILITY DATA

Test Series	N-1	Mean Value $\bar{X}$	Standard Deviation σ	σ^2	"F" Values 3 vs 4	3 vs 5	4 vs 5	Precision of Estimate of the mean (99%) -Percent
Table 3	9	16.59	0.20	0.040	1.44	3.60*		1.17
Table 4	9	14.94	0.24	0.058			2.52	1.56
Table 5	24	34.07	0.38	.144				0.81

N = Number of test runs

* Indicates a probability of .05 or less.

Figures based upon calculation of the precision are according to the procedure given on Page 64 of Snedecor - "Statistical Methods", Iowa State College Press, 1946, with the ± deviation given as a percentage of the mean.

Table VI contains a summary of the statistical computations to which the data were subjected. The repeatability was examined from the viewpoint of variation within a population group (or series of tests) and with respect to variation between different population samples subjected to the same operation.

The F - test ($F = \frac{V_1^2}{V_2^2}$) which compares the variance of the samples is a means of determining whether the variation of individual determinations within each of two population samples is significantly different. The F - test is based on the proposition that if there is no difference between the variance of two populations the expected F-value is equal to one. The five percent and one percent levels are considered to be significant. The observed F- value distributions of F has been established and are available in a number of books on statistics. These tables are entered with particular values of F (which may vary with sample size) and give the probability of obtaining values of this magnitude due to chance alone. The five percent and one percent probability levels are considered significant. If the observed F value is greater than the five percent or one percent critical value, the hypothesis that the variances are equal is rejected.

When the variance of the sample of Tables III and V are calculated (See Table VI) and their ratio is compared to the F - values of the probability table, the probability falls between the five percent and one percent values (Table values $F_{.05} = 2.90$; $F_{.01} = 4.73$). On this basis it was found that the difference between the above series of test runs is significant at the five percent level. However, the same considerations applied to comparison of the ratios of Tables IV and V reveals no significant differences in the variances of the samples.

Although there is a statistically significant difference in the variance of Tables III and V, it is contended that the five percent level of significance is not sufficient reason to reject the hypothesis that there is no real difference between the variances of Tables III and VI. Had the probability level been $\leq$ 0.01, the hypothesis would have been rejected.

The basis for the foregoing statement is the fact that the F values indicate that there is no difference between samples 3 and 4 and between 4 and 5. Consequently, it does not seem unreasonable to require the one percent significance level before rejecting the hypothesis.

The following conclusions may then be tentatively drawn:

1. The sonic shear test procedure gives repeatable results over a wide range of viscosity change (or shear).

2. A change in the test procedure (45 minutes vs 60 minutes of sonic irradiation) did not affect the repeatability of this series of tests.

3. Two different test materials subjected to the same test procedure showed the same magnitude of percent variation of the means indicating that the percent dispersion of data may be constant.

IV CONCLUSIONS

Conclusions:

1. That the sonic equipment is an accurate and rapid mean of studying oils containing polymers.

2. The Sonic Shear Method offers the following advantages over the Mechanical Shear Method.

(a) Utilization of small quantities of fluid (30 to 50 cc) compared to the requirements of two to three gallons in mechanical test apparatus.

(b) The test at any desired level of shear severity can be performed in very short time periods.