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An improved Couette high shear viscometer and its application to crystallization of polyethylene in simple shear.

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AN IMPROVED COUETTE HIGH SHEAR VISCOMETER AND ITS APPLICATION TO CRYSTALLIZATION OF POLYETHYLENE IN SIMPLE SHEAR

A Dissertation Presented

by

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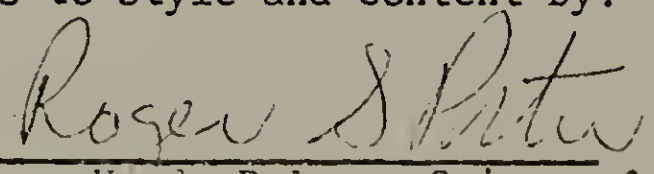
Approved as to style and content by:



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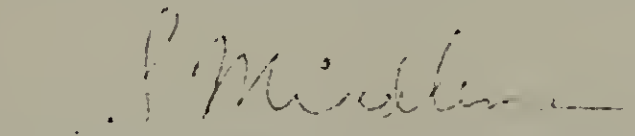

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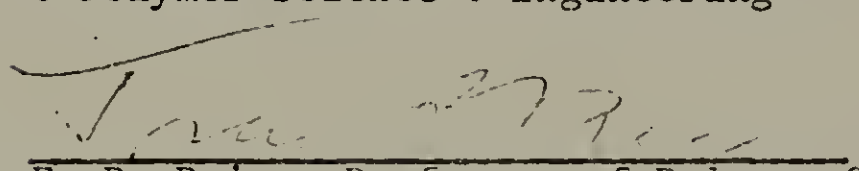

F. P. Price, Professor of Polymer Science & Engineering

May 1972

DEDICATION

To Merrilea, for her patience and collaboration in this effort

To David, Cheri and Wendy, for sharing the experience

To my parents, for their confidence in me

To Grant and Professor Porter, for their wise counsel

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ABSTRACT

Both the upper and lower shear rate ranges for a Couette-type high shear viscometer have been extended beyond those reported in the literature. This has been accomplished by:

1. Improved machining and lapping accuracy of precision steel cylinder (spindle and ring) combinations.
2. More effective temperature control and monitoring.
3. Standardized formats for test parameter and data recording.
4. Simplified, but statistically valid, data analysis techniques.

These and secondary improvements (self-aligning drive shafts of different diameters for different torques, a spindle depth placement tool, and computer compatible measurement output) have resulted in a high performance laminar shear viscometer of only modest complexity. Operational equations for this instrument are derived, and a complete operation procedure is given. Calibration data analysis shows a measurement accuracy of ± 3% or better over most of its shear rate range. Application of this viscometer to the crystallization of polyethylene under simple shear is also demonstrated.

I. INTRODUCTION

The first practical concentric cylinder rotational viscometer was developed by Couette in 1890.[1] It consisted of a rotational cup which placed a measurable shear stress on a torsion-wire-mounted inner cylinder, the shear stress being proportional to the viscosity of the fluid in the circular gap between them. The accuracy and versatility of the basic Couette design have been made apparent by its subsequent widespread use in original or modified form for over 50 years. Beginning in the 1940's, major design innovations began to appear in the literature with increasing frequency.[2]

It has been proposed that the Couette-type viscometer is the most satisfactory type for high shear rate measurements,[3] i.e. shear rates near or above 100,000 sec^{-1}. In contrast to capillary and other types of viscometers, concentric cylinder designs have the characteristic of producing uniform laminar shear fields over a wide range of shear rates. This was ably demonstrated with a Couette-type viscometer designed by Barber, et. al.[4] and later modified and extensively applied by Porter, et. al.[5-10] Noteworthy features in this design were: the use of narrow gap clearances between the cylinders, the elimination of end effects through the extension of the inner rotating cylinder above and below the ends of the torque measuring outer cylinder, and the control of temperature in both cylinders through the use of a common thermostatted heat transfer fluid.

In this study we have developed a new version of the Barber/Porter design which has extended both the lower and upper shear rate ranges of the earlier instruments. With this most advanced version of this type of viscometer, laminar shear rates significantly exceeding any reported in the literature to date have been achieved.

II. INSTRUMENTATION

The viscometer (Figs.1-2) consists of a main frame and drive assembly supported by a thermostatting system and an instrument readout console. A driven inner cylinder (spindle) imparts a rotational torque to a bearing mounted outer cylinder (ring)(Fig.3), and the force needed to restrain rotation of the ring is measured along a rigidly mounted torque arm by a horizontally mounted Statham Gold Cell.[11] transducer and strain gauge (Fig.4). The strain gauge assembly is precisely located along the torque arm by centering it over threaded holes in a slide holder and fixing it in place with an attached knurled screw. These fixed positions are on one inch (2.5 cm) centers, from 4 to 11 inches (10 to 28 cm) from the center of the spindle.

Four transducer/strain gauge combinations are used, capable of measuring forces ranging from 25 to 6,800 grams (force) and giving a torque measurement range of 60 to 190,000 gm-cm (.052 to 165 in-lbs). A needle mounted pulley is located near the end of the slide holder, allowing known weights to be attached to the torque arm for strain gauge calibration (Fig.5). The strain gauge is powered by a Statham Bridge Amplilier, model SC1100, and torque voltage is read out by a Hewlett Packard 3440A Digital Voltmeter equipped with a 3443A High Gain/Auto Range Unit.[12] The readout console (Fig.6) is also equipped with a Hewlett Packard 581A Digital-Analog Converter and a 680 Strip Chart Recorder. Besides providing a permanent

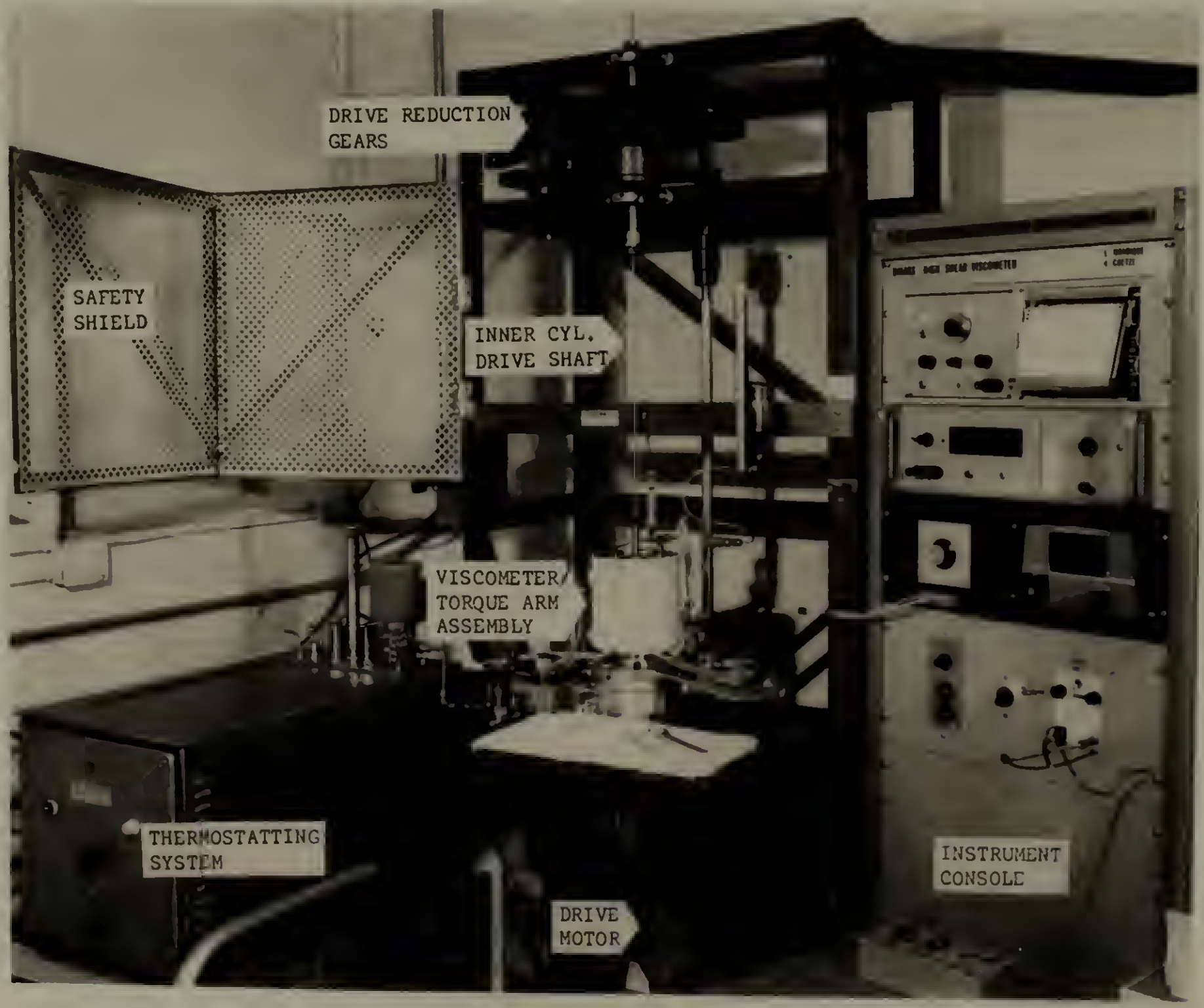


FIGURE 1 - UNIVERSITY OF MASSACHUSETTS HIGH SHEAR VISCOMETER

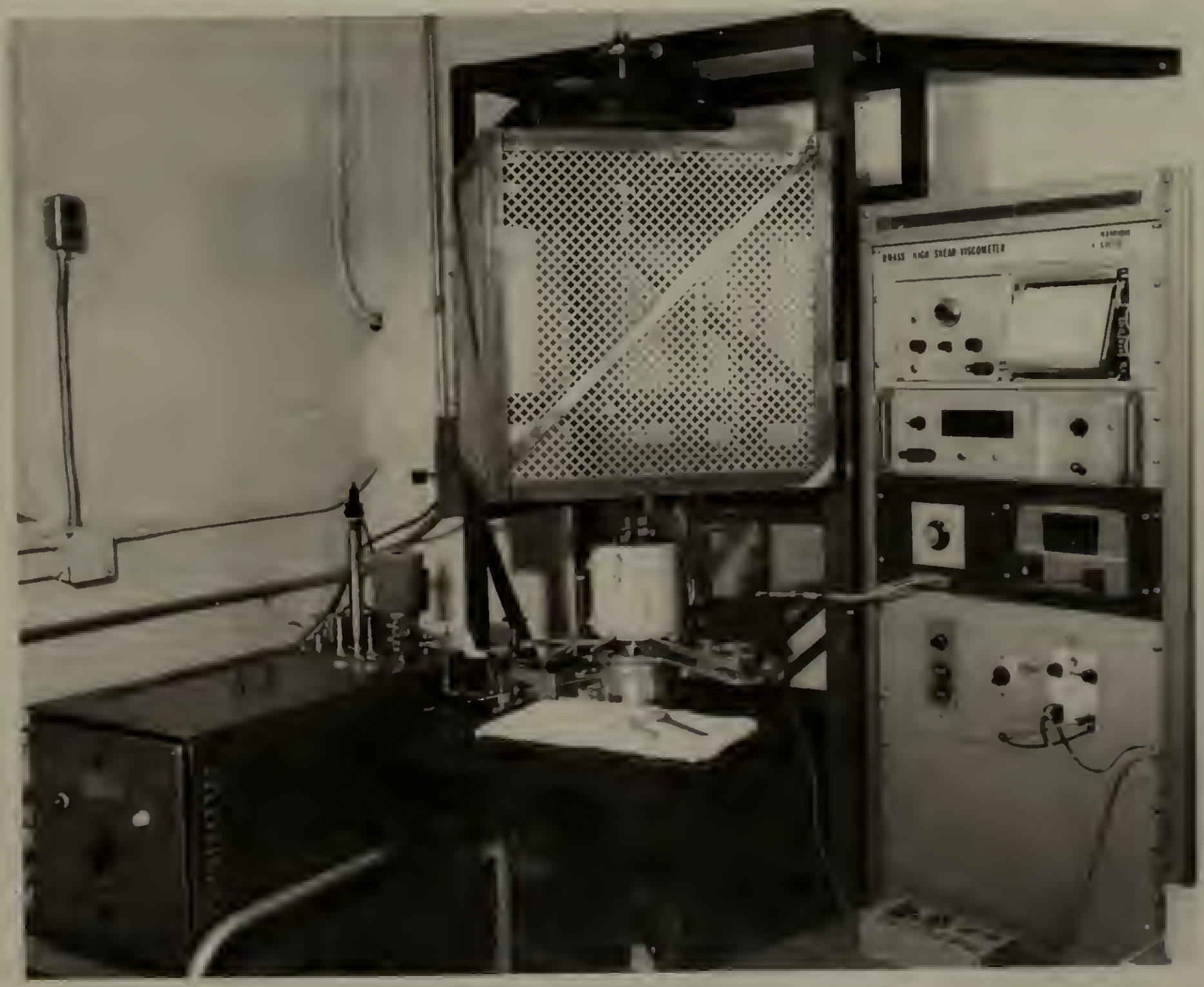


FIGURE 2 - HIGH SHEAR VISCOMETER, AS OPERATED

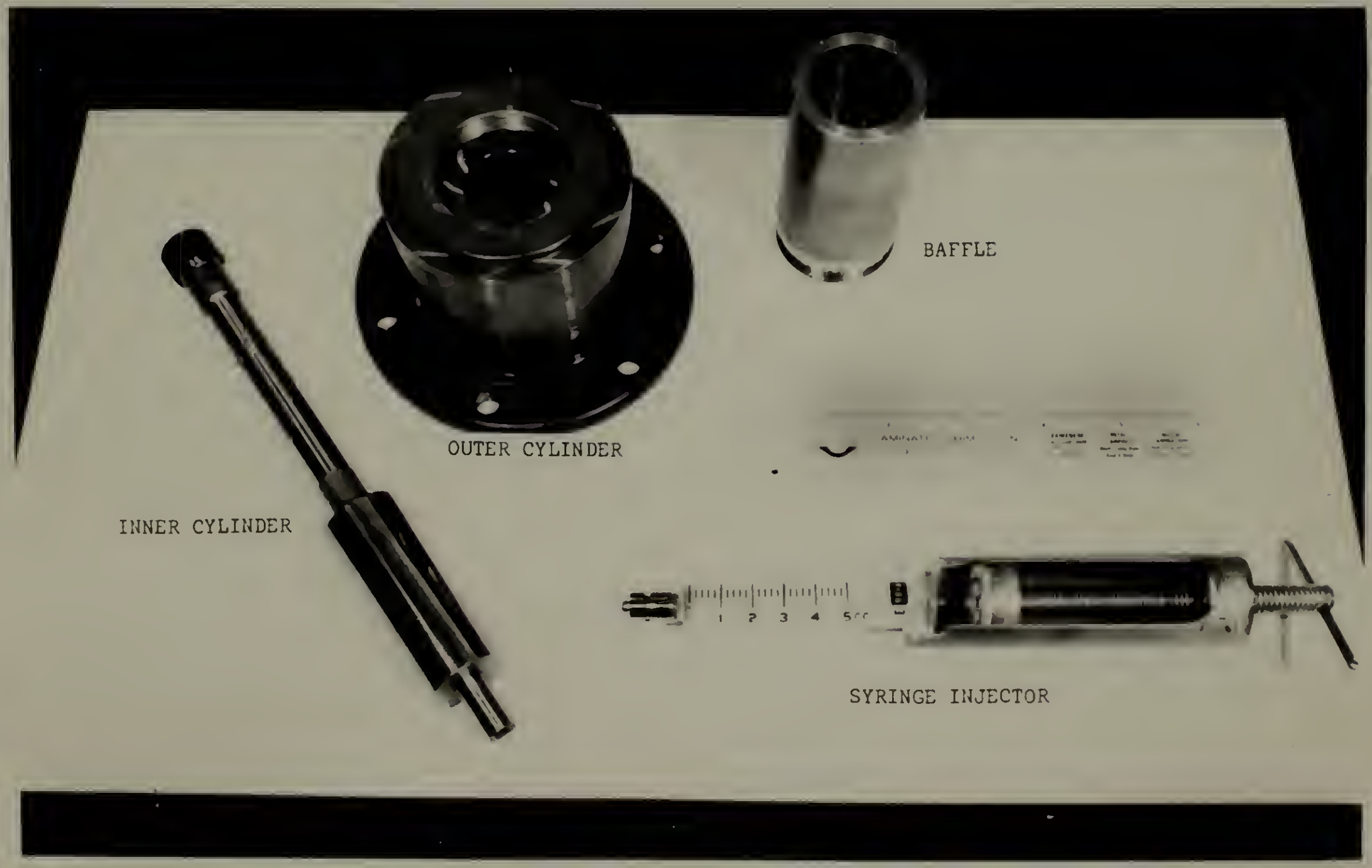


FIGURE 3 - VISCOMETER CYLINDERS AND SAMPLE INJECTOR

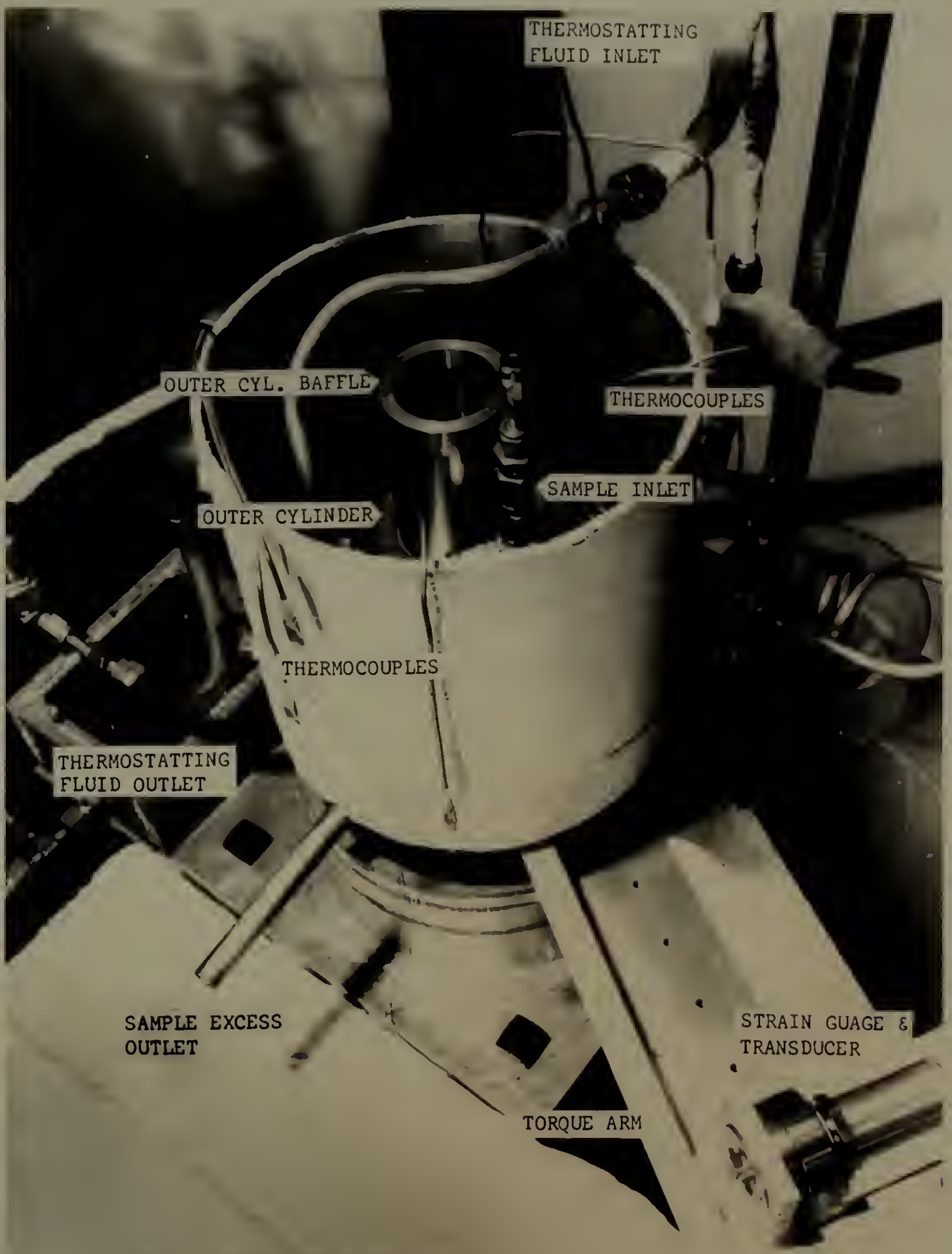


FIGURE 4 - VISCOMETER OUTER CYLINDER AND TORQUE ARM

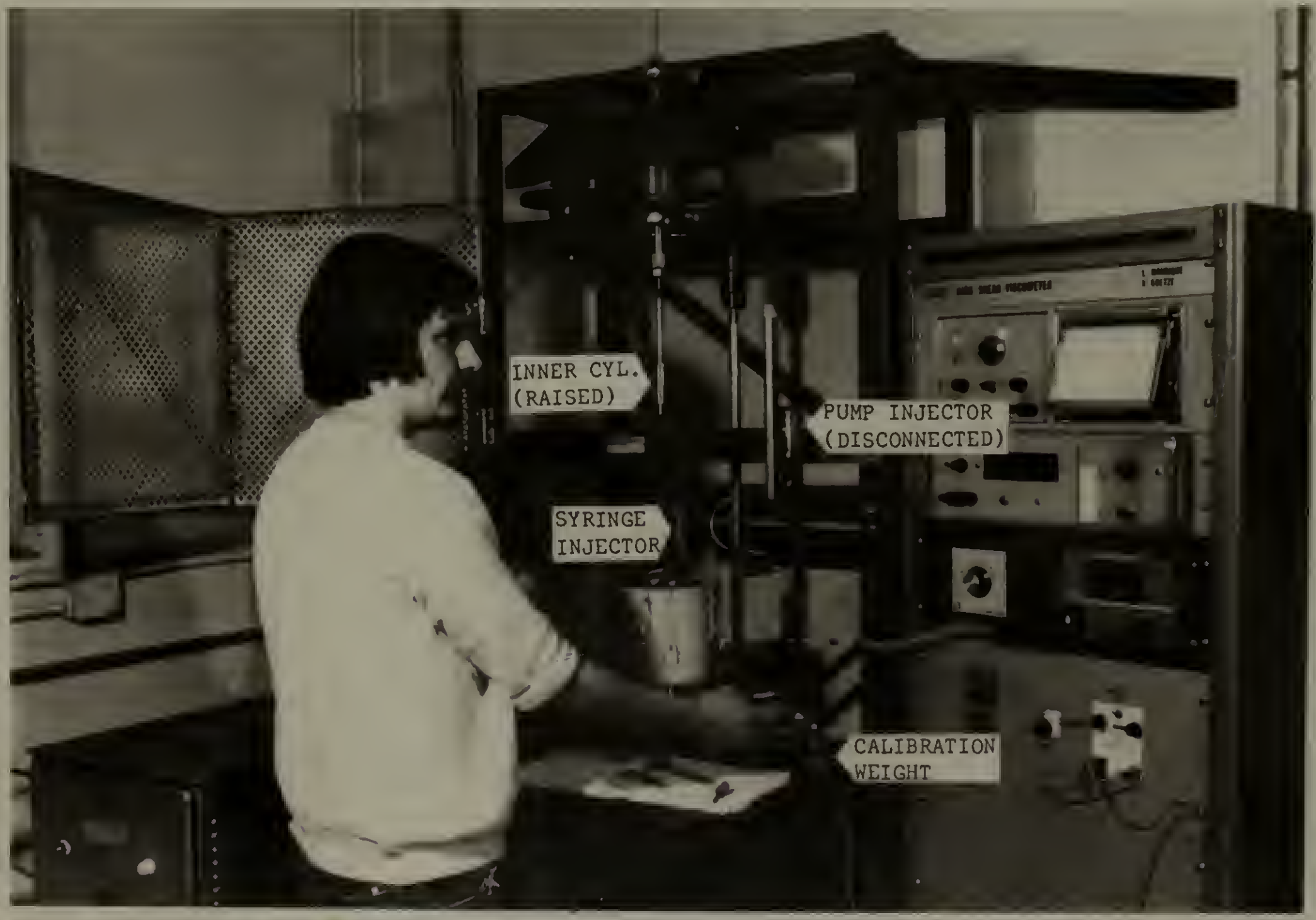


FIGURE 5 - TORQUE ARM CALIBRATION

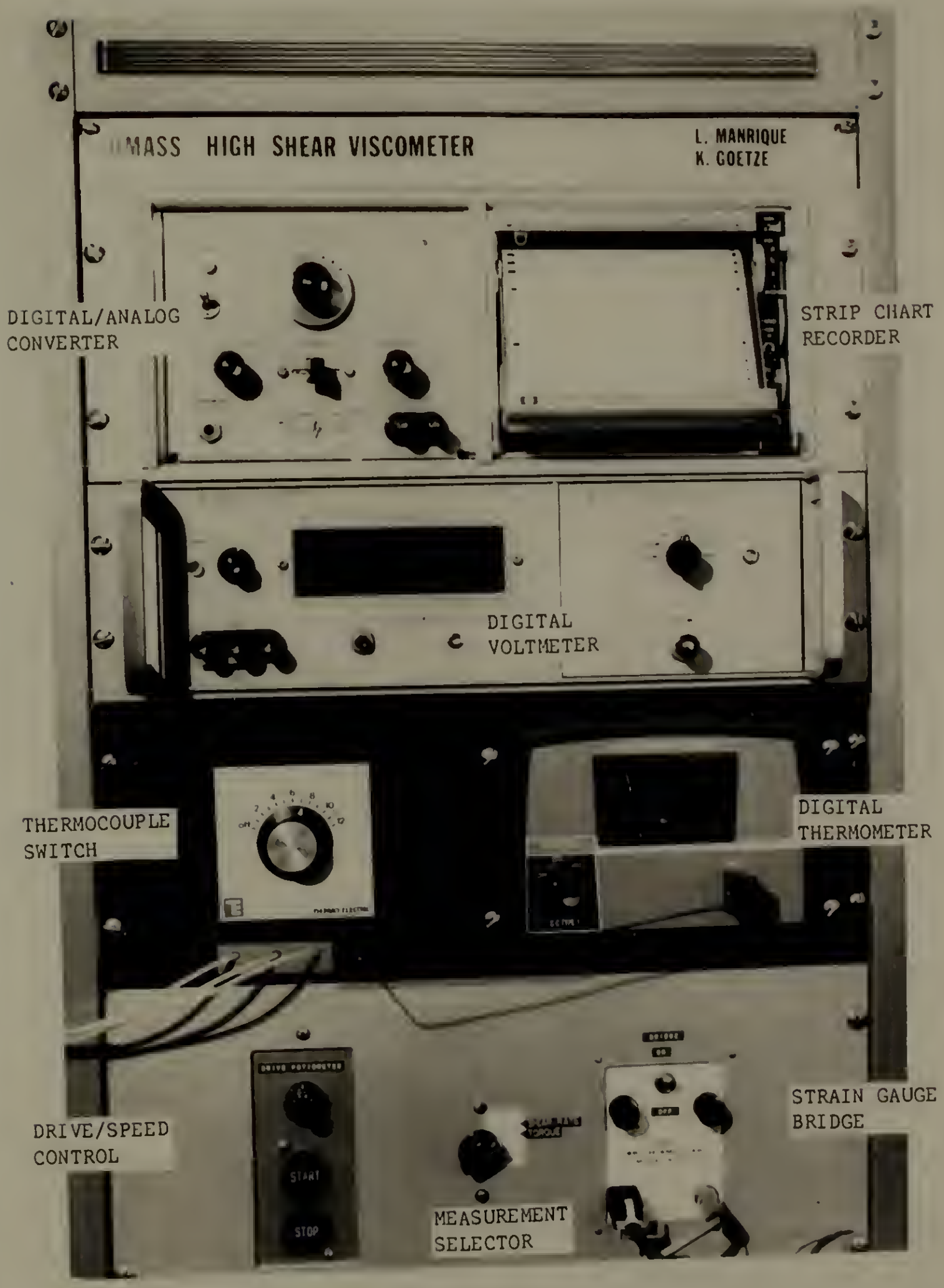


FIGURE 6 - INSTRUMENT CONSOLE DETAILS

chart recording of torque and shear rate voltages, the analog feature allows the early part of the torque curve to be observed before the digital voltmeter has reached equilibrium. It also allows a continous torque recording at constant shear rate, so that viscosity reduction due to viscous heating may be observed.

Temperature is controlled by a Tamson Thermostatic Bath Model TE 45,[13] using a General Electric Silicone Fluid 96 (50 cps)[14] as the thermostatting liquid. This fluid is held at a preset temperature and directed around the base of the ring by two "T" inlets. A cylindrical outer vessel, rigidly mounted to the ring, serves as a heating oil reservoir, and contains a return pipe mounted high enough to keep the ring fully immersed (Fig. 7). In addition, a second inlet pipe directs circulating fluid through the center of the hollow spindle and back out through the base of the viscometer assembly, maintaining the spindle at the same temperature as the ring. The feed-line to this inlet is extended into a coil which can be cooled in a water bath. This procedure allows the spindle to be cooled slightly more than the ring, assisting the free rotation of the narrowest clearance spindles.

The incoming oil lines and the oil reservoir surrounding the ring are asbestos insulated to promote temperature uniformity and minimize heat loss through the viscometer base. In addition, there is a 1/4 inch thick Teflon[15] gasket (not shown in Fig. 7) between the ring and the surrounding oil reservoir for further

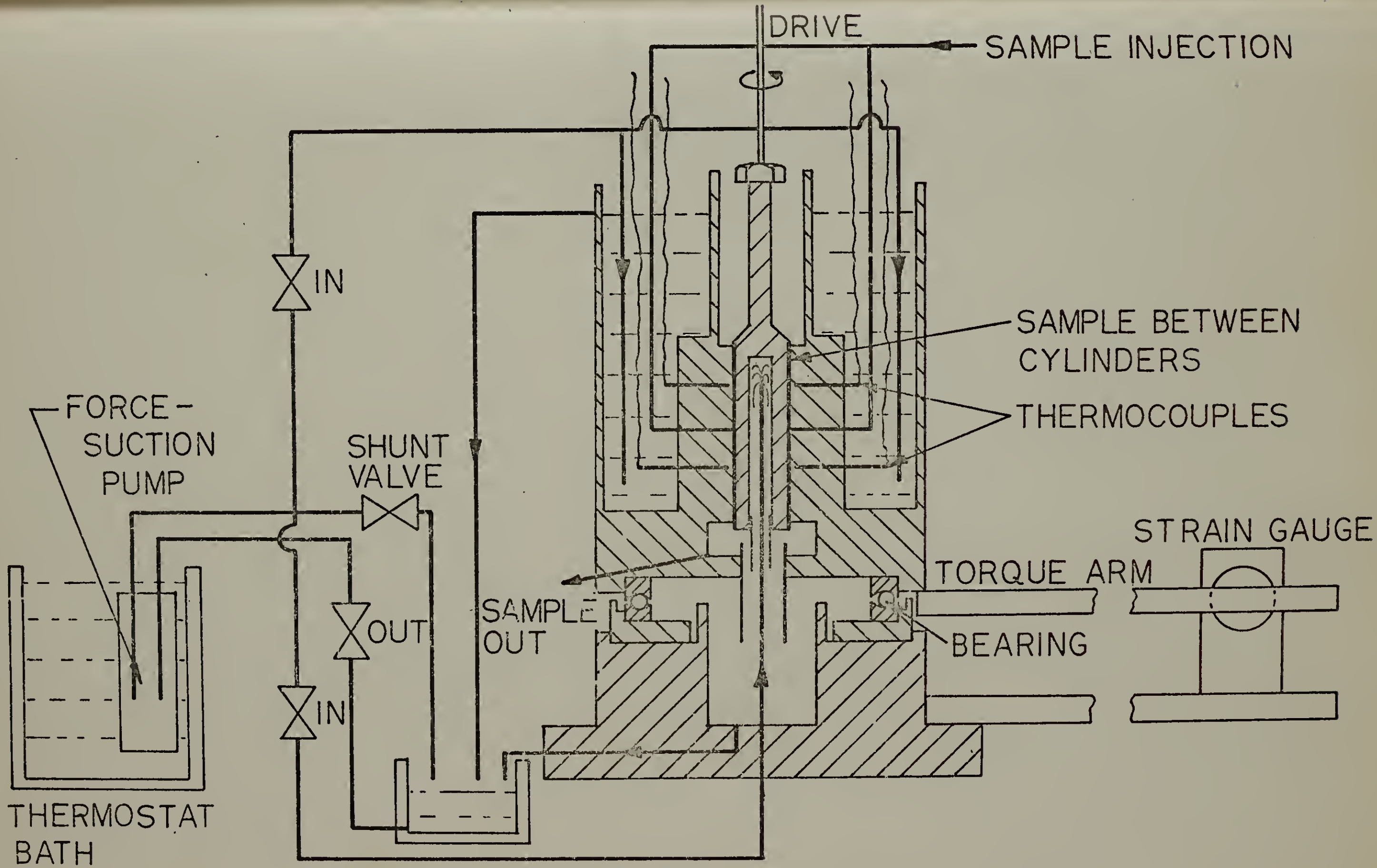


FIG. 7 - SECTION THROUGH HIGH SHEAR VISCOMETER AND FLOW SYSTEM

thermal insulation of the ring. A thin layer of silicone rubber caulking was placed on the surface of the gasket just before assembly to prevent thermostatting oil leakage at the seal.

These various heating bath modifications have minimized thermal gradients due to oil stratification, reducing them to less than 5°C through the cylindrical oil reservoir, and to less than 0.5°C through the ring. This has been accomplished without the use of torque producing mechanical stirrers.

There are six copper-constantan thermocouples permanently mounted at various heights and depths in the ring to monitor temperature (Appendix I, Fig. E-1) and two additional free thermocouples which can be used to check the temperature of the spindle when the latter is not rotating. All temperatures are read out visually at each position to 0.1°C with a Digitec Digital Thermometer[16] via a manually operated rotary switch. The thermocouples themselves are composed of fine guage Teflon coated copper-constantan wire, selected and positioned so that no measureable mechanical resistance to torque is observed when the ring assembly of the viscometer is rotated.

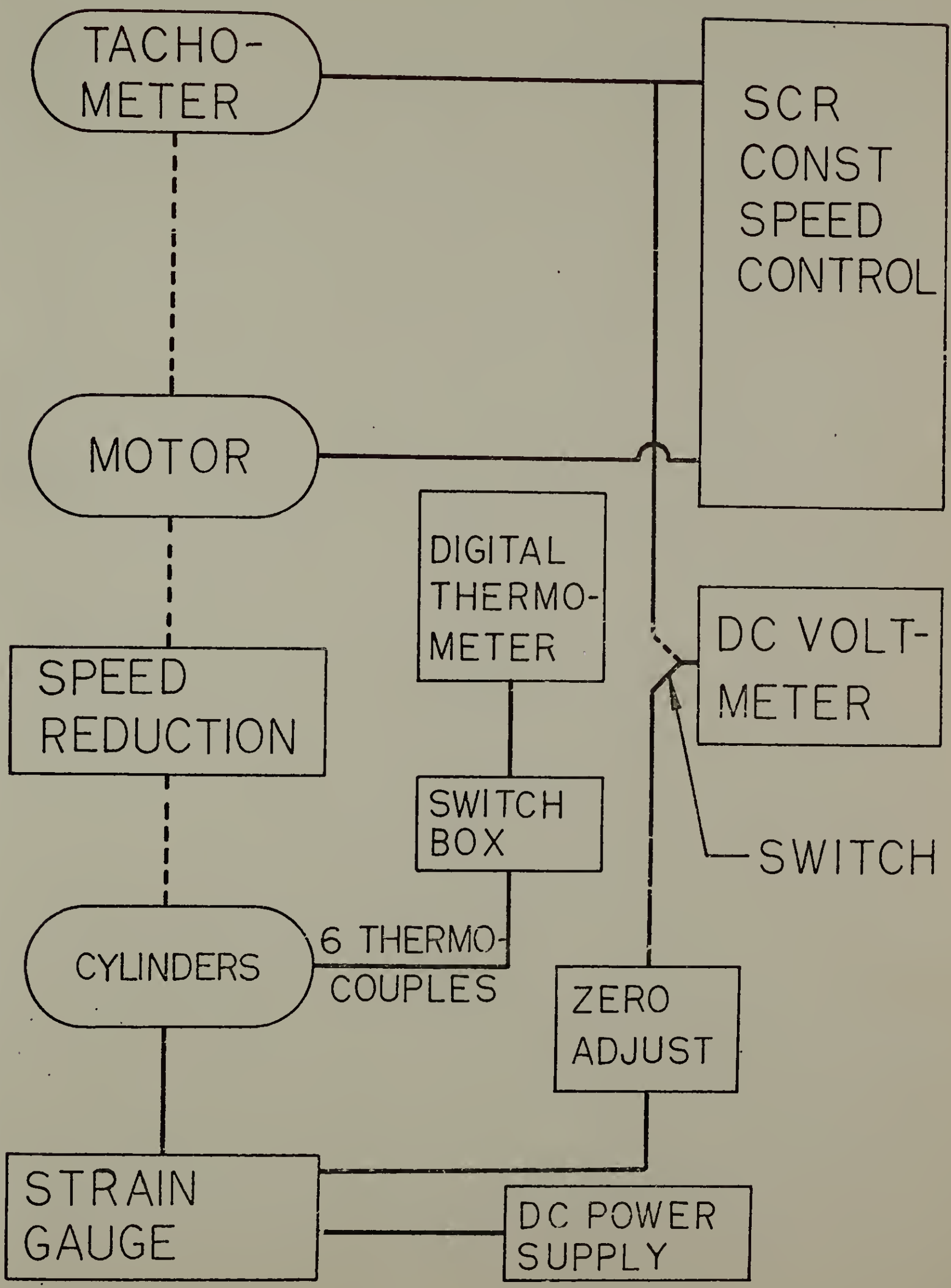
Rotational power to the spindle is supplied by a Century[17] 1 hp single phase electric motor, controlled by a Rotiotrol E-100 Full-Wave SCR Precision Speed Control.[18] The motor-mounted tachometer

feedback is recorded through the same voltmeter/converter/recorder system previously described, by means of a two-position selector switch. An electrical block diagram for the drive and readout systems is shown in Fig. 8. Not shown are the converter and recorder, which are connected directly to the voltmeter. A 1:10-1:1 clutch over-ride is included in the spindle drive network, giving a variable speed range for the spindle of 20 to 2,500 rpm.

The matched ring and spindles set was machined and lapped from SAE 8620 carbon steel by Bendix Automation and Measurement Division, Dayton, Ohio. Gap widths between the pairs range from .00002 inches to .005 inches, and are described in detail in the next section. The narrowest gap is at least 2X smaller than the narrowest reported in the literature[5,19] and represents a significant advance in precision machining and lapping. The widest gap set operating at low rotational speeds extends the lower shear rate range for this type of instrument also.

The spindles are mounted to the drive with a choice of 1/8, 3/16, or 1/4 inch diameter drive shafts and suitable collet chucks. This arrangement allows the drive shaft to be selected for maximum flexibility relative to the expected torque so that the spindle and ring can act as a self-aligning bearing on a flexible drive shaft.

FIG. 8 - HIGH SHEAR VISCOMETER
ELECTRICAL BLOCK DIAGRAM



III. OPERATIONAL EQUATIONS

For concentric cylinder viscometers, several operational equations have been described.[4,20,22] The equation used by Barber,[4] which he gives only in its final form, is particularly convenient and will be derived here.

Shear rate ($\dot{\gamma}$) is given for Newtonian fluids by the Margules equation[21] which relates angular velocity (Ω) of the rotating cylinder and the radii of each (r_o,r_i):

$$\dot{\gamma} = \frac{2\Omega}{r_i^2}\left(\frac{r_i^2 r_o^2}{r_o^2 - r_i^2}\right) = \frac{2\Omega r_o^2}{(r_o + r_i)(r_o - r_i)} \quad 1.$$

For narrow cylinder gaps, where $r_i \approx r_o$ and $r_o - r_i = h$ (the gap clearance), the subscripts for r may be dropped, and equation 1. becomes:

$$\dot{\gamma} = \frac{2\Omega r^2}{2rh} = \frac{\Omega r}{h} \quad 2.$$

The error in applying equation 1. to non-Newtonian fluids is small and has been shown graphically by Middleman.[22A]

Transforming angular velocity in radians/second to rotations per minute (N), equation 2. becomes:

$$\dot{\gamma} = \frac{2\pi N}{60} \frac{r}{h} \qquad 3.$$

In general, shear stress (S) is defined as a planer force (F) applied to a given area (A):

$$S = F/A \qquad 4.$$

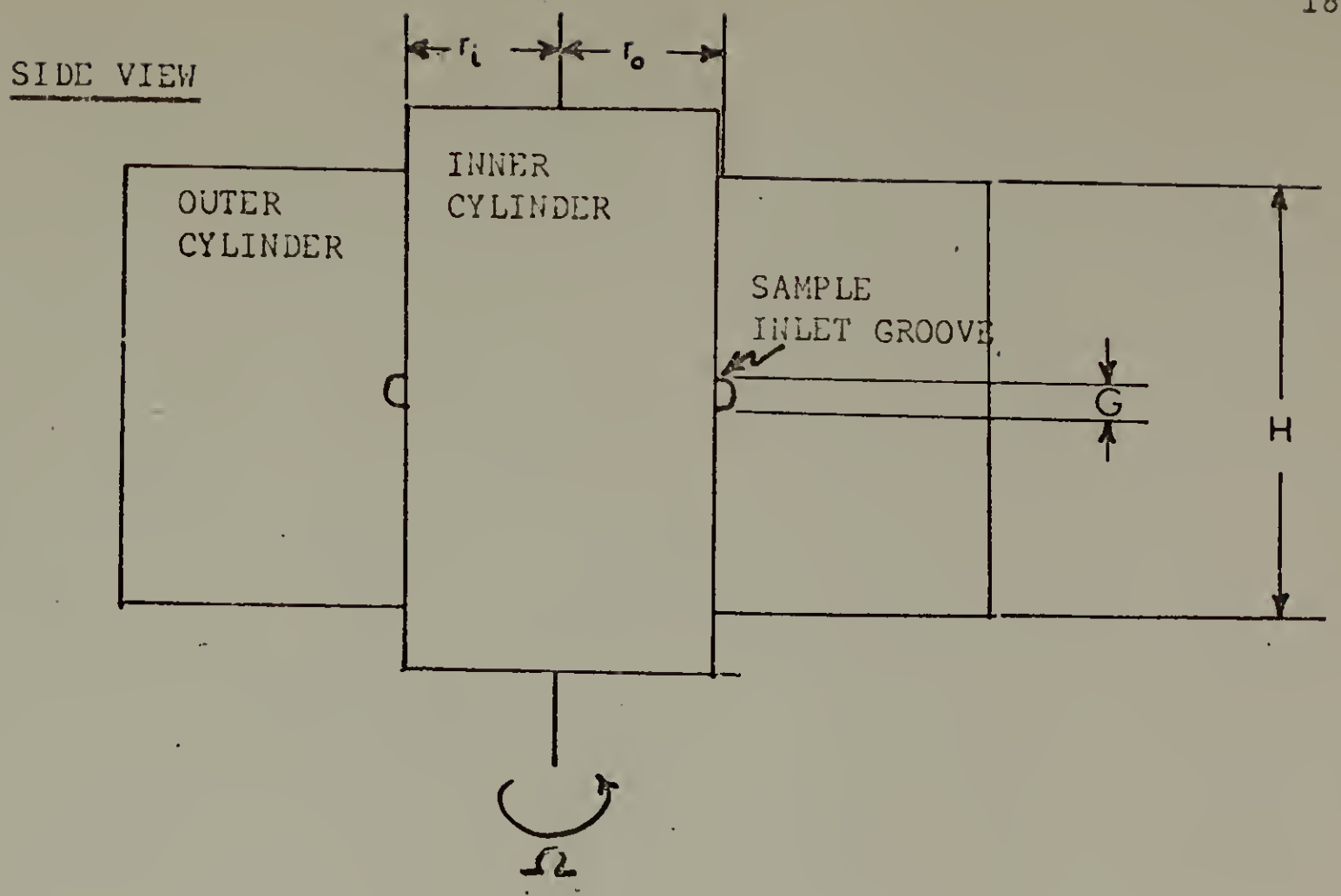
Referring to Figure 9, force can be expressed as the torque (τ) applied to the outer cylinder, divided by its radius (r_o), the effective torque arm. Area is given by the surface of the outer cylinder: the net length (L) of the outer cylinder times its circumference, thus:

$$S = \frac{\tau / r_o}{2\pi r_o L} = \frac{\tau}{2\pi r_o^2 L} \qquad 5.$$

By definitation, viscosity (η) is shear stress divided by shear rate. Thus using equations 3. and 5. we have:

$$\eta = \frac{S}{\dot{\gamma}} = \frac{\tau / 2\pi r^2 L}{2\pi N r / 60h} = \frac{15\,\tau\, h}{\pi^2 r^3 N L} \qquad 6.$$

SIDE VIEW



$L = H - G = 2.000 - 0.125 = 1.875"$

$r_i \approx r_o = 0.500"$

$\tau = 12.25 \times F_c = L' \times F_r = r \times F_s$ (in - lbs)

TOP VIEW

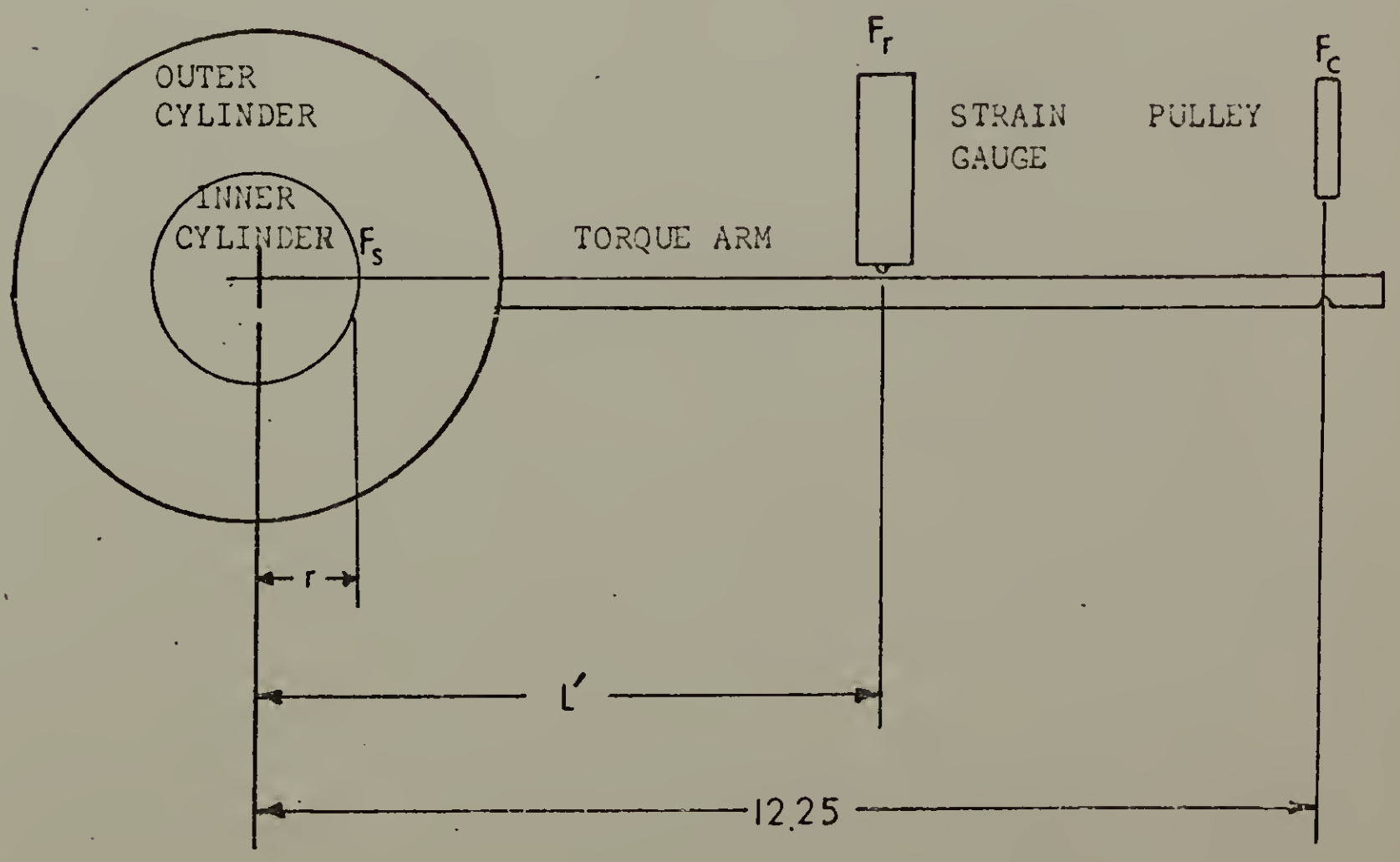


FIGURE 9 - CYLINDER AND TORQUE ARM DIMENSIONS

Inserting the appropriate dimensions for this viscometer, given in Figure 9, the operational equation used by Barber[4] and Porter[5] is obtained:

$$\eta = \frac{\tau\, h}{.154\, N} \qquad 7.$$

Where η is given in $\frac{lb/sec}{in2}$, τ in inch-pounds, and h in inches.

This general form will be modified for specific operating conditions in the next section.

IV. CALIBRATION

A. Measurement of Gap Clearances

Specification design data for various spindle and ring combinations can be used to calculate nominal gap clearances (Table I). A more refined calibration may be obtained with the previously given equations by measuring standard fluids of known viscosity. This is because micro-roughness in the cylinder surfaces becomes important to the narrower gap clearances.[23] Mechanical measurement of the spindle diameters tends to bias their dimensions in the + direction due to touching the "peaks" and missing the "valleys" of their surface roughness.[5] Thus, gaps calculated from such data tend to appear smaller than they would measure by a more accurate "averaging" technique, i.e. a technique based on the effective gap as "seen" by the fluid. Such a technique can be developed by rearranging equation 7.:

$$h = .154\,\eta\frac{N}{\tau} \qquad 8.$$

By shearing a fluid of known viscosity in a spindle/ring combination at a known rotational speed and measuring the resulting torque, gap clearance (h) may be calculated. In order to make equation 8. still more convenient for our application, it is possible to modify the machine constant .154 to a new constant (K), such that

TABLE I - RING AND SPINDLE SPECIFICATION SUMMARY

SPINDLE #	SPECIFICATION[4] DIAMETER (in)	RING[1,2] Δ DIAMETER	RING[1,2] GAP CLEARANCE	GAP VOLUME[3]
4	0.990 ± .001	0.010	5000 μ inches	.48 ml
1	0.9990± .0001	0.0011	550 "	.054 "
1A	0.9991 ± .0001	0.0010	500 "	.048 "
2	0.99970 ± .00003	0.00042	210 "	.020 "
2A	0.99982 ± .00003	0.00030	150 "	.014 "
3	0.999985 ± .000005	0.000140	70 "	.007 "
3A	1.000085 ± .000005	0.000040	20 "	.002 "

NOTES

1. Ring diameter = 1.000125 ± .000005 inches
2. A second ring, in storage as a reserve, is 1.000005 ± .000005 inches in diameter, and thus would give gap clearances 60 μ in less for each of the above spindles (except for 3A which does not fit).
3. Gap volume = $2\pi rLh = 2 \times 3.14 \times .5 \times 15/8 \times 16.4\ cm^3/in^3 \times h$

   $= 96.5\ h$
4. All measurements at room temperature

viscosity (η) is given in poise and rotational speed ($\dot{\gamma}$) and torque (τ) are given in volts, i.e. the actual readout units of the system.

Thus:

$$h_{(\mu\ \mathrm{INCH})} = K\,\eta_{\mathrm{POISE}}\,\frac{\dot{\gamma}_{\mathrm{VOLTS}}}{\tau_{\mathrm{VOLTS}}} \qquad 9.$$

Values of K for the available strain gauge and torque arm combinations are given in Table II, and are calculated from the slopes of the strain gauge graphs given in Appendix II. Torque arm linearity (i.e. accuracy of transducer placement positions) is demonstrated in Appendix III. For gap clearance calibration, both hydro-

carbon and silicone oil viscosity standards were used. Selected oil standards (lower viscosities for narrow gaps, higher viscosities for wider gaps) were introduced using a screw-driven syringe.(Fig.3). Low rotational speeds were used in order to minimize shear heating, and shear rate voltages vs. torque voltages were recorded. Using these data and equation 9. (along with the appropiate K values from Table II and viscosities from the viscosity/temperature plots in Appendix XI) ten to twenty gap clearances were calculated near room temperature for each spindle. The resulting measured gap clearances were then compared with manufacturer's specified gap clearances in Table III.

TABLE II - HIGH SHEAR VISCOMETER INSTRUMENT CONSTANTS (K)

TORQUE ARM POSITION L′ (inches)	STRAIN GUAGE USED			
	RATED LOAD	0.5 lbs	2 lbs	10 lbs
	W (lbs/v)	0.0539	0.270	1.58
4		242	48.4	8.27
5		194	38.7	6.62
6		161	32.2	5.51
7		138	27.7	4.72
8		121	24.2	4.13
9		108	21.5	3.68
10		96.9	19.4	3.30
11		88.1	17.6	3.00

NOTE: The above K values must be divided by 10 when the 1:10 drive reduction gears are used.

CALCULATIONS

Since $$h_{IN} = .154\,\eta_{\frac{LB\ SEC}{IN^2}}\,\frac{N_{RPM}}{\tau_{IN\ LB}}$$

then $$h_{\mu IN} = \frac{.154\,(1.45\times10^{-5}\ \frac{LB\ SEC}{IN^2}/_{POISE})(23.4\ ^{RPM}/_{VOLT})}{(W\ ^{LB}/_{VOLT})(L'_{IN})\ 10^{-6}\ ^{IN}/_{\mu IN}}\,\eta_{POISE}\,\frac{\ddot{\gamma}_v}{\tau_v}$$

$$= K\,\eta_{POISE}\,\frac{\ddot{\gamma}_v}{\tau_v}$$

where $$K = {}^{52.2}/_{WL'}$$

TABLE III - SPINDLE CALIBRATION SUMMARY [1]

SPINDLE #	SPECIFICATION [2] GAP CLEARANCE	MEASURED GAP [3]		
		HYDROCARBON OIL	SILICONE OIL	AVERAGE
4	5000	4910±180	5050±90	5010±190
1	560	505±5	527±18	514±22
1A	500	530±17	522±19	527±18
2	210	212±9	208±3	210±8
2A	150	152±3	157±2	155±3
3	70	78±4	78±1	78±2
3A	20	24±1	--	24±1

NOTES: 1. All gap measurements given in μ inches at room temperature

2. Ring diameter = 1.000125 inches

3. Data details and calculations are given in Appendices IV-X

Based on the above gap clearances and the range of available rotational rates for the viscometer, the range of available shear rates for each spindle/ring combination has been calculated (Table IV) and is shown graphically in Figure 10. Compared with earlier reported instruments,[4,5] these shear rates represent a 3 to 10X decrease in the low shear rate end and a 2 to 4X increase in the upper shear rate range capability.

B. Data Recording and Probability Plots.

In order to facilitate the collection and analysis of data, a standardized lab work sheet is utilized (Fig.11). Provision is made for the recording of nearly all operational parameters which might affect the precision or accuracy of the test. Calculations and operational notes may also be made directly on this work sheet for future reference. On the reverse side of the lab work sheet, provision is made for a τ vs. $\dot{\gamma}$ voltage plot in order to examine the raw data for non-Newtonian flow characteristics. There is also a grid to plot ring temperature vs. distance from the inner ring wall. In cases of significant temperature inhomogeneity due to viscous heating, a linear extrapolation of temperature vs. distance will help establish temperature at the film boundary.

TABLE IV - SPECIFICATION RANGE OF AVAILABLE SHEAR RATES

h_μ inch \ N_{RPM}	20	500	2500
5000	209	5,230	26,600
500	2,090	52,300	266,000
210 (2)	4,980	124,000	632,000
150	6,970	174,000	886,000
70 (3)	14,900	373,000	1,870,000
20	52,000	1,310,000	6,550,000

NOTE: All shear rates given in sec^{-1}

$$\dot{\gamma} = \frac{2\pi Nr}{60h} = \frac{.0523\ N}{h}$$

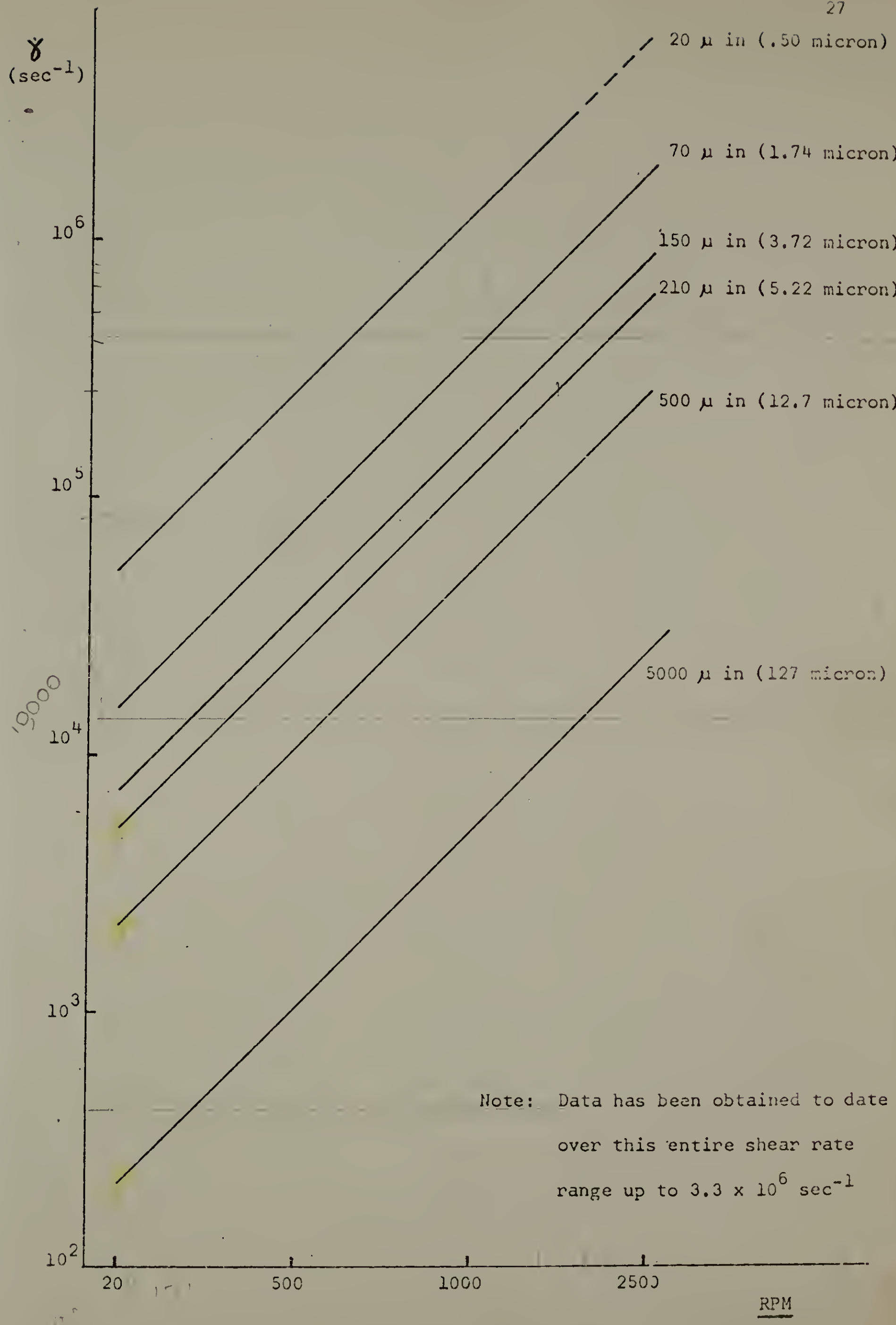


FIGURE 10 - SPECIFICATION SHEAR RATES FOR INDICATED GAP CLEARANCES

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO.

TEST CONDITIONS

sample

thermostat bath flow settings
heater: off ☐ boost ☐ max accuracy ☐
to pot:
to inner cyl:
" coil:
box inlet:

date 28
operator
temp.
spindle no.

drive shaft:
" ratio: 1:10 ☐ 1:1 ☐

TRANSDUCER

τ arm length:
load cell:
chart speed:

	VM zero	VM int. ck.	τ zero	τ ___ gm	
initial					
after run					

TEMPERATURE

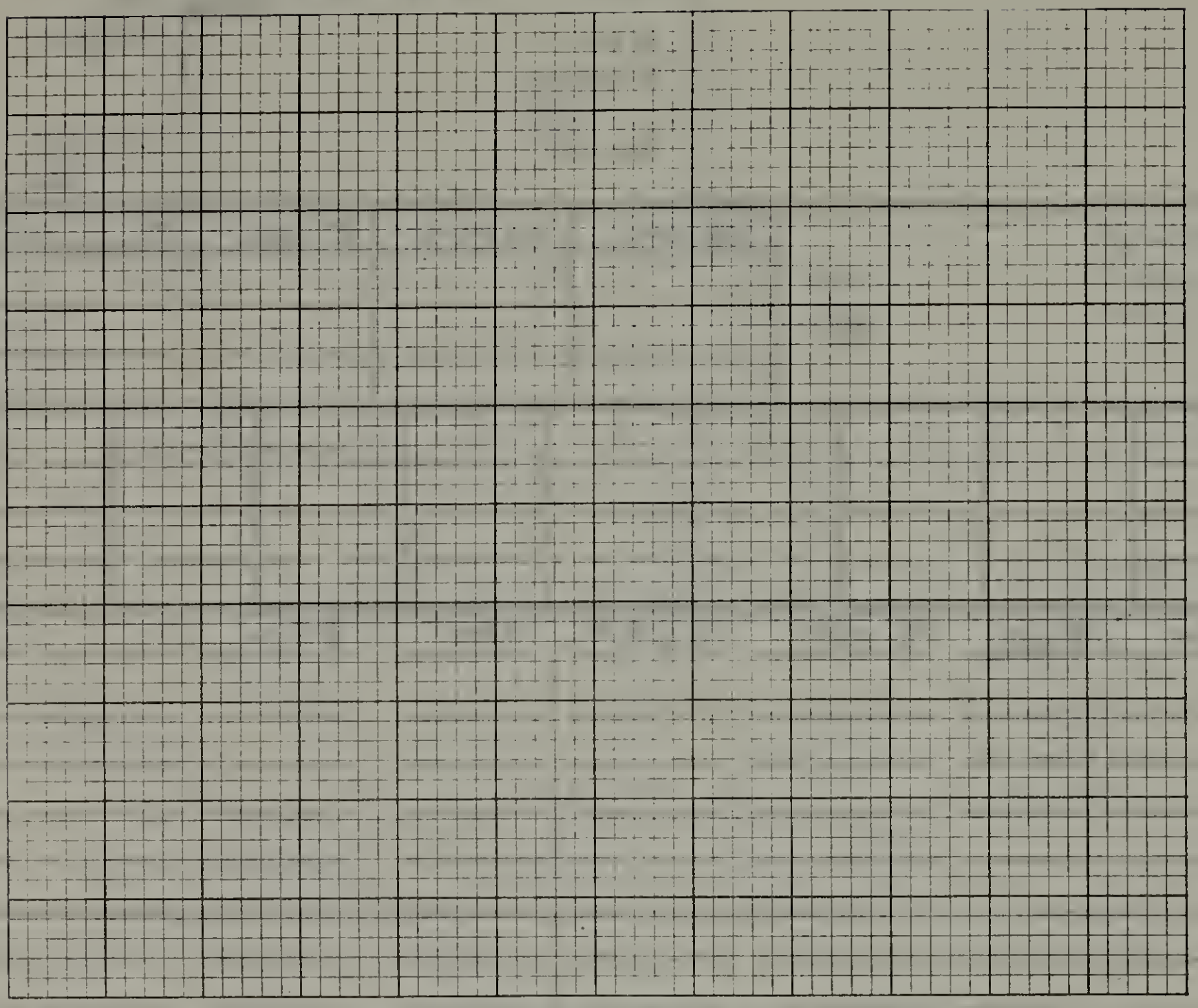
1	2	3	4	5	6	7 ___	8 ___		

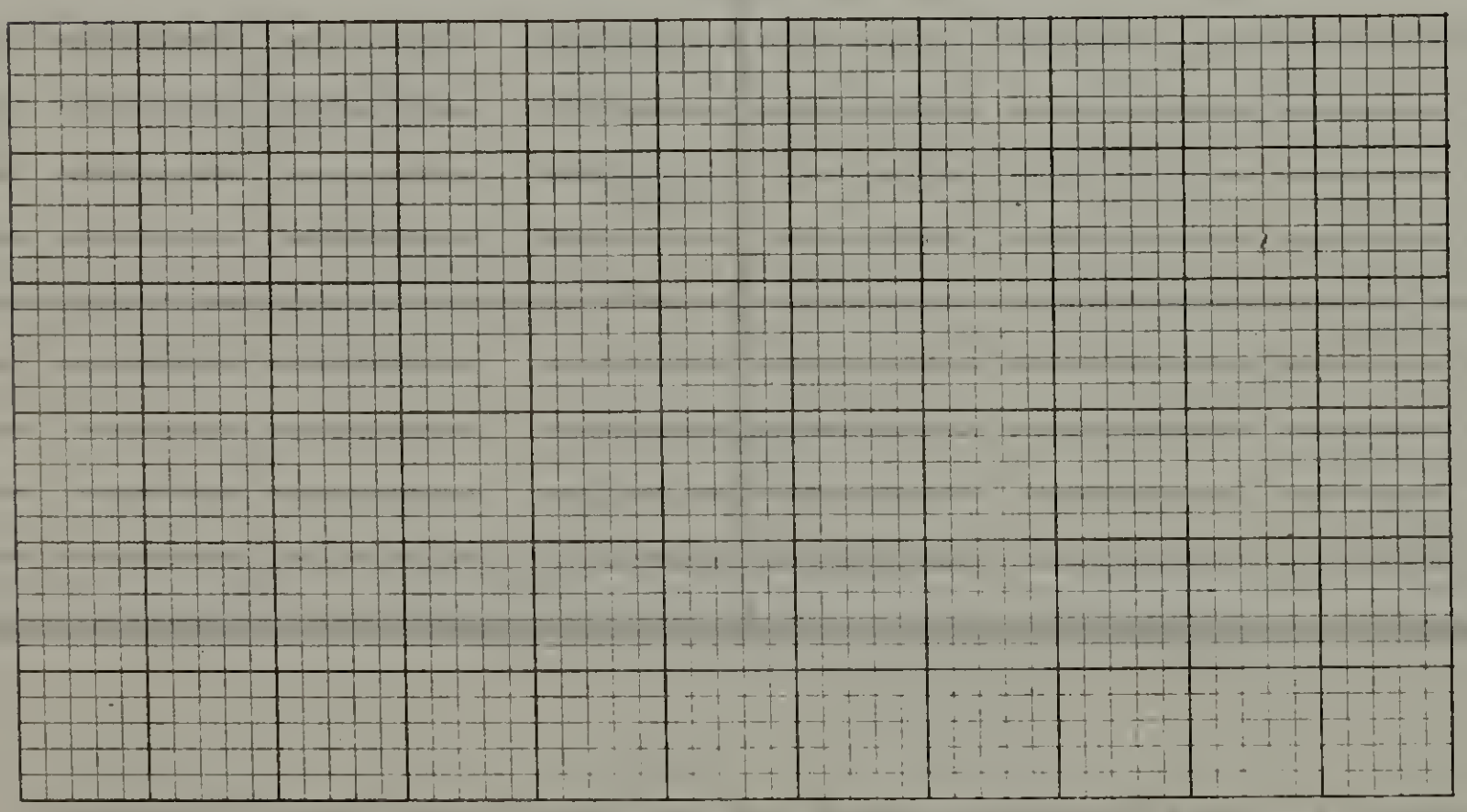
SHEAR DATA

pot. set.	$\dot{\gamma}$ volts	τ volts	°C at TC*	RPM	$\dot{\gamma}$ rate		

NOTES

FIGURE 11 - DATA RECORDING SHEET FOR THE UMASS HIGH SHEAR VISCOMETER





One method of convenient determination of experimental precision is through the use of probability graph paper.[26] This method was used in the measurement of spindle gap clearances (Appendices IV - X)and can be described as follows: For a series of n measurements of a value of h:

1) Arrange the values of h in ascending order.
2) Assign a numerical sequence i to these values h such that $i = 1$ for the smallest value and $i = n$ for the largest value.
3) Calculate $\frac{i-1/2}{n}$ for each value (note: for a given group of values, i.e. 10, 12, 20 etc., this need be done just once).
4) Plot h vs. $\frac{i-1/2}{n}$ on probability paper.
5) Fit with the best straight line, by eye.

The above operations are easier to perform than to describe, and allow these observations to be made:

1) Does the data distribution indicate normalcy? A normal (Gaussian) distribution of data can be concluded if the data points on the probability plot fall on or near a straight line.
2) If the criterion in 1) is met, then the intersection of the h axis with the 50th percentile line will give the mean value of h.
3) The difference between the h axis intersections with the 50th percentile and the 84th percentile gives the standard deviation (σ) of the distribution.

This technique of probability plotting of data was especially useful in justifying the combination of hydrocarbon and silicone oil data during spindle calibration. It should also be useful in comparing viscosity data obtained by different operators, or for determining the reproducibility of data obtained by the same operator.

C. Viscous Heating

One of the greatest difficulties encountered in viscosity measurements at high shear rates is uncertainty due to viscous heating. The thermocouple configuration and thermostatic bath arrangement to minimize gap temperature uncertainty have already been described. It can also be demonstrated that fundamental design considerations in this viscometer, namely small gap thickness for high shear rates, further minimizes viscous heating effects. Barber[4] illustrates this for a single SAE-10 oil film of several thicknesses at a single shear rate, using a formula derived by Hersey[27] for determining the lubrication film temperature rise in a cylindrical bearing, assuming a thin film under adiabatic conditions, that is, the inner surface adiabatic and the outer one isothermal.

This formula:

$$\theta_{oF} = \frac{\eta_R \dot{\gamma}^2 h^2}{2 k_E} \qquad 11.$$

where: θ_{oF} = maximum temperature rise in the film

η_R = viscosity (lb sec/in^2)

$\dot{\gamma}$ = shear rate (sec^{-1})

h = film thickness (inches)

k_E = thermal conductivity (lb/sec°F)

converts to cgs units as follows:

$$\theta_{oC} = \frac{.53\, \eta\, \dot{\gamma}^2 h^2}{k} \qquad 12.$$

where: η = viscosity (poise) h = film thickness (cm)

k = thermal conductivity (gm cm/sec^{3o}C)

Equation 12. was then used to calculate the temperature rise for several calibration standard oils at high shear rates in this viscometer. It can be seen from Table V that for all conditions except for higher viscosity fluids, viscous heating is not serious. Also noteworthy is that film temperature rise is independent of gap clearance if the rotational speed of the inner cylinder is constant.

TABLE V - TEMPERATURE RISE IN FILM - BARBER(HERSEY) FORMULA[1]

GAP CLEARANCE	20 μ inch	5000 μ inch
SHEAR RATE @ 2000 RPM	5.2×10^6 sec^{-1}	2.1×10^4 sec^{-1}
VISCOSITY(POISE)		
0.031^2	.07 °C	.08 °C
0.294^2	.72	.74
0.390^3	.92	.93
0.980^3	2.41°	2.3
12.96^2	shear stress exceeds instrument maximum	33

NOTES: 1. M. D. Hersey, Theory of Lubrication, J. Wiley & Sons, Inc., New York (1936) p.115.

2. Hydrocarbon oil (thermal conductivity 1.45×10^4 $\frac{gm\ cm}{sec^3\ °C}$)

3. Silicone oil (thermal conductivity 1.53×10^4 $\frac{gm\ cm}{sec^3\ °C}$)

A more refined calculation of temperature rise at the ring wall is recommended by Middleman.[28] This approach takes into account the curvature of the film and distinguishes between temperature rise when both of the cylinders are isothermal:

$$\Delta T_{MAX} = \frac{T_o \, Br \, Co^2}{4}\left[1 - \frac{f(s,n)}{2}\left(1 - \frac{\ln f(s,m)}{2}\right)\right] \qquad 13.$$

and where one of the cylinders is adiabatic:

$$\Delta T_{MAX} = \frac{T_o \, Br \, Co^2}{4}\left[1 - \frac{1}{S^2} - 2\frac{\ln S}{S^2}\right] \qquad 14.$$

where:

S = R/Ro

R = radius of inner cylinder

η = viscosity

Ro = radius of outer cylinder

$\dot{\gamma}$ = shear rate (sec^{-1})

N = rotations per minute

To = initial temperature

k = thermal conductivity

$$Co = S\left(\dot{\gamma}/\Omega\right)$$

$$Br = \text{Brinkman \#} = \frac{\eta V^2}{k T_o}$$

$$V = \text{linear velocity} = 2N\pi R/60 = \Omega R$$

$$f(s,m) = \frac{1 - 1/S^2}{\ln S}$$

The calculation of temperature rise using equations 13. and 14. has been greatly simplified through the use of a computer program developed by Fritzsche[29] and given in Appendix XII. Temperature rise calculated with these formula are given in Table VI. These data are in general agreement with those obtained using the Hersey equation, with the exception that the Hersey equation, in neglecting surface curvature, seems to overestimate heating effects. The Middleman equations are also more appropiate for this viscometer since the viscometer can be operated in either an adiabatic or an isothermal mode. Actual measurements in the high shear viscometer indicates small temperature rises consistant with these calculations.

TABLE VI - TEMPERATURE RISE IN FILM - MIDDLEMAN FORMULA[1]

GAP CLEARANCE	20 μ inch		5000 μ inch	
SHEAR RATE @ 2000 RPM	5.2×10^6 sec^{-1}		2.1×10^4 sec^{-1}	
CONDITIONS	ADIABATIC	ISOTHERMAL	ADIABATIC	ISOTHERMAL
VISCOSITY(POISE)				
0.031[2]	.08 °C	.02 °C	.08 °C	.02 °C
0.294[2]	.72	.18	.71	.18
0.390[3]	.90	.23	.90	.22
0.980[3]			2.2	.56
12.96[2]	shear stress exceeds instrument maximum		31	7.8

NOTES: 1. S. Middleman, The Flow of High Polymers, Interscience, New York (1968) p.36.

2. Hydrocarbon oil (thermal conductivity 1.45×10^4 $\frac{\text{gm cm}}{\text{sec}^3\,^\circ\text{C}}$)

3. Silicone oil (thermal conductivity 1.53×10^4 $\frac{\text{gm cm}}{\text{sec}^3\,^\circ\text{C}}$)

(cal/sec·cm·°K) × 4.184×10^7 = (g-cm/sec^3·°K)

ρ: 0°C 2.51[illegible]^4 (cal/sec·cm·deg)
50°C 2.78×10[illegible]

V. DISCUSSION

A detailed operation procedure for this instrument has been prepared (Appendix I), but once the user is familiar with its contents, the viscometer can be used quite routinely. Current measurement accuracy is estimated to be about $\pm 3\%$ for all but the most extreme operating conditions.

Limitations on further improving operational capability come from three major sources: 1) thrust bearing friction, 2) transducer signal amplification and 3) torque overload of the strain gauge system.

1) The thrust bearing supports the ring, its oil reservoir and its circulating oil. The downward force of this assembly results in bearing friction (against torque) of about 2 gms of force. This is not significant over most of the transducer range, but can introduce errors of $>10\%$ in the measurement region below 25 gms (force). Thus the transducer should always be used with a strain gauge in order to stay out of this low torque range. There is a way to circumvent this limitation since, for virtually any fluid to be measured, a gap clearance can be selected so that the magnitude of the resulting torque is greater than the "high error" region (Appendix II).

2) The components in the transducer readout system are all capable of the accuracy of $>$ 1%, with the exception of the Statham Bridge Amplifier. Although it has a rated accuracy of $\pm$.5% over the full range, this can be as poor as 5% in the lower range. This is most noticible in the inability to balance the bridge to precisely +5,000 v during calibration. The slightest adjustment in the sensitivity potentiometer often causes a jump of .01 v or more, which can cause an error of several % in signals of less than 1 v. A better quality signal amplifier would allow the higher accuracy capability of the remainder of the amplifier system to be properly utilized.

3) The upper limit of high shear torque measurement is primarily due to the maximum load limit of 6,800 gms (force) on our largest strain gauge (10 lbs). Simply using a larger strain gauge is no solution however, because at higher loadings torque arm and drive shaft overload can occur, resulting in an erratic and noisy signal background and possible hardware damage. There is a method of at least doubling the upper limit of shear stress measurement, namely by raising the spindle 1 3/16th inches above its normal depth of placement (after filling the gap with sample). This reduces the shear area (and the resulting torque) by 1/2 and doubles the measurable shear stress. A height block is used to insure that the spindle is raised

precisely 1 3/16th inches so that no new wear marks result (i.e. the wear marks in this position will correspond to those normally resulting from the bottom of the ring and the mid-line filling groove).

VI. VISCOMETER APPLICATION

A. Crystallization of Polyethylene in Simple Shear

The crystallization of Alathon 7050 and Phillips 6009 high density polyethylene under shear stress and pressure to produce highly oriented (transparent) strands has been widely published.[30-34] We have been able to show, through the use of the high shear viscometer, that under simple shear conditions alone, no comparable transparent morphology is produced. Although some crystallinity enhancement is observed, it is of a lower order than that of the transparent strand, and exhibits physical properties which lie between those of the transparent strand and the conventional bulk crystallized high density polyethylene morphology.

1) Properties of the Melt

Alathon 7050 crystallizes at ambient pressure in the range of 134 - 135°C, with a melt viscosity of approximately 10,000 poise[35] (Fig.12). With this information, and the computer program previously described (Appendix XII), it can be shown that for a .005 inch melt film thickness between concentric cylinders, less than 1°C viscous heating occurs for rotational speeds below 25 rpm (Fig.13). Thus, if a melt can be sheared

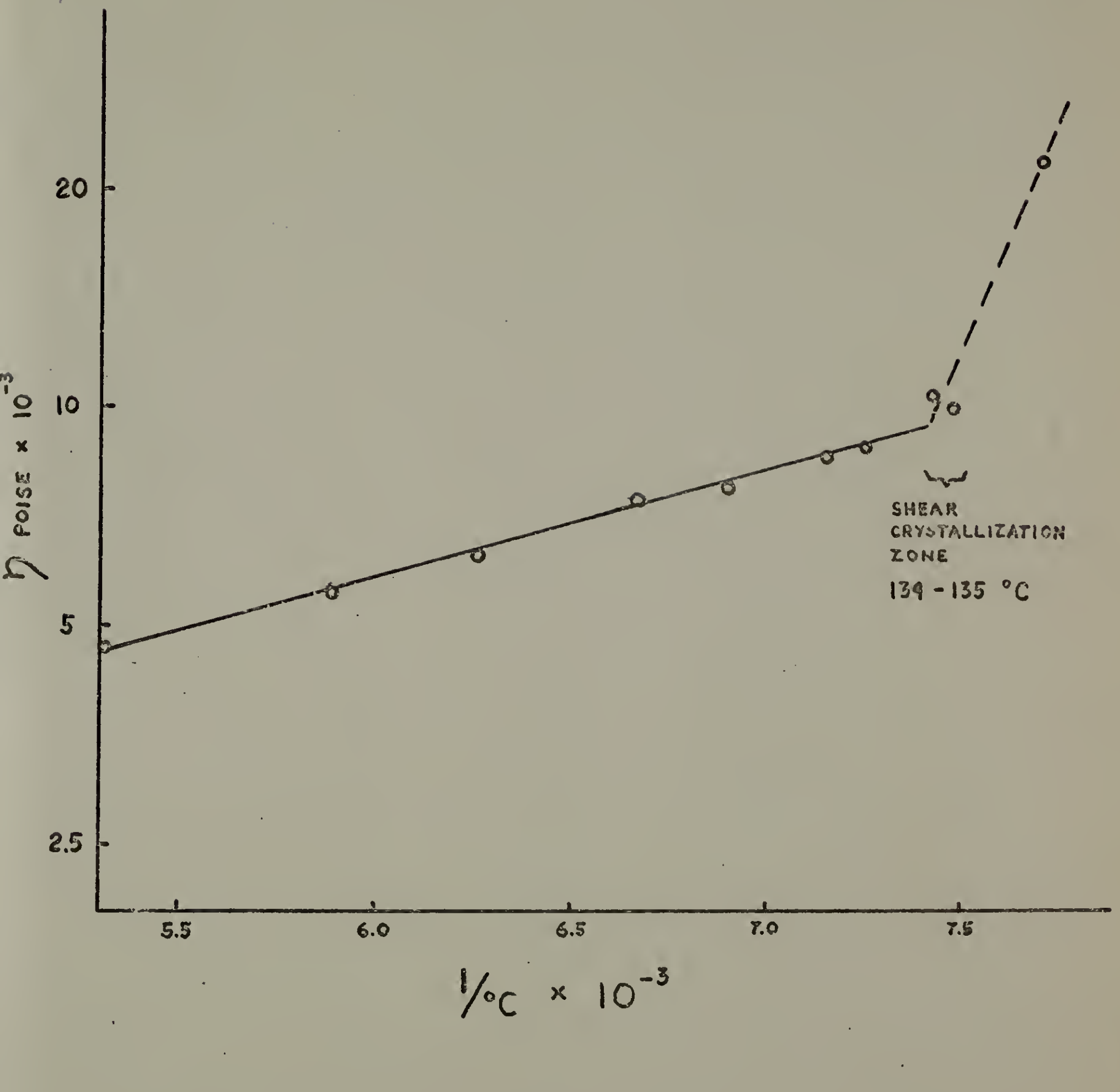


Fig 12 - ALATHON 7050 Viscosity vs. Temperature

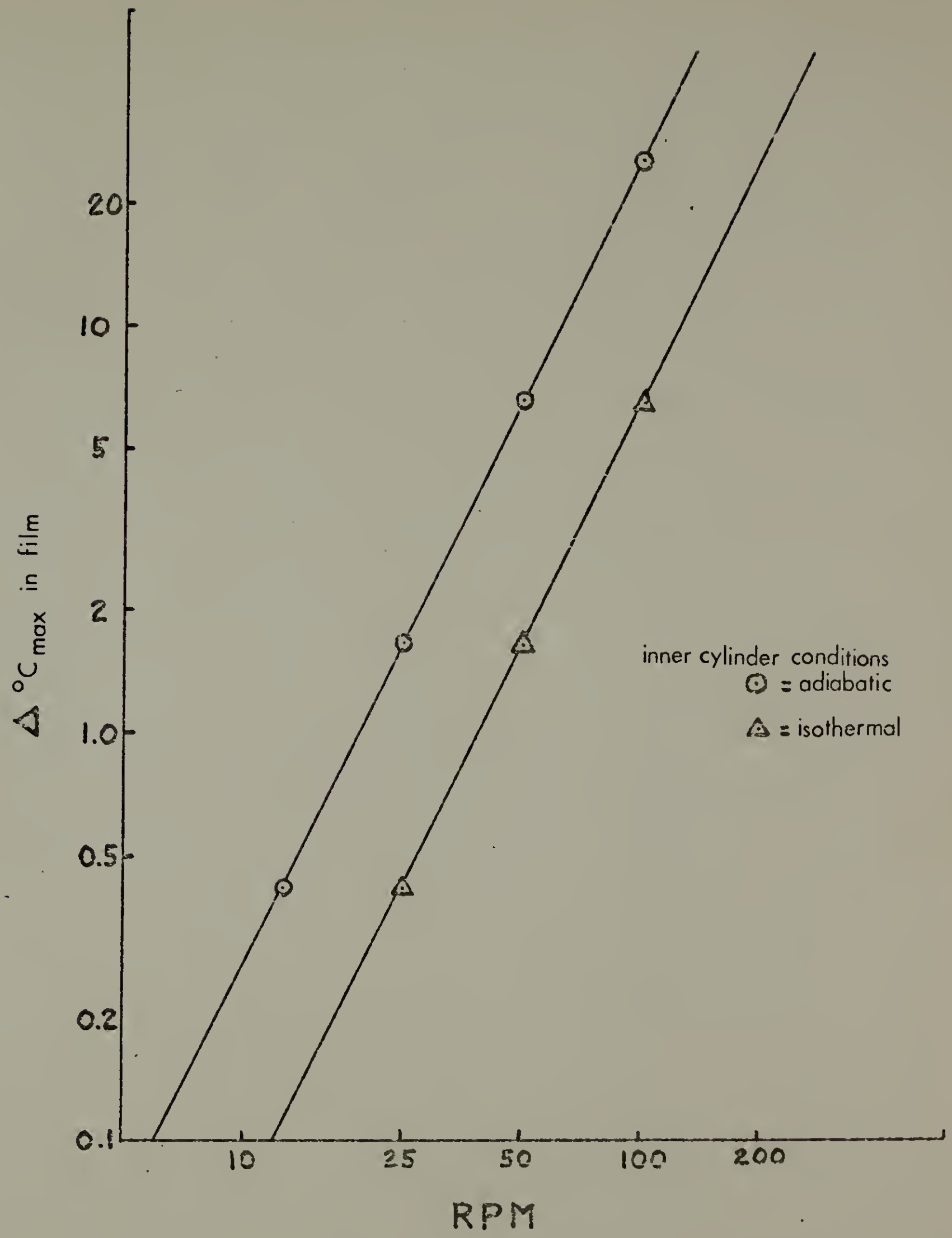
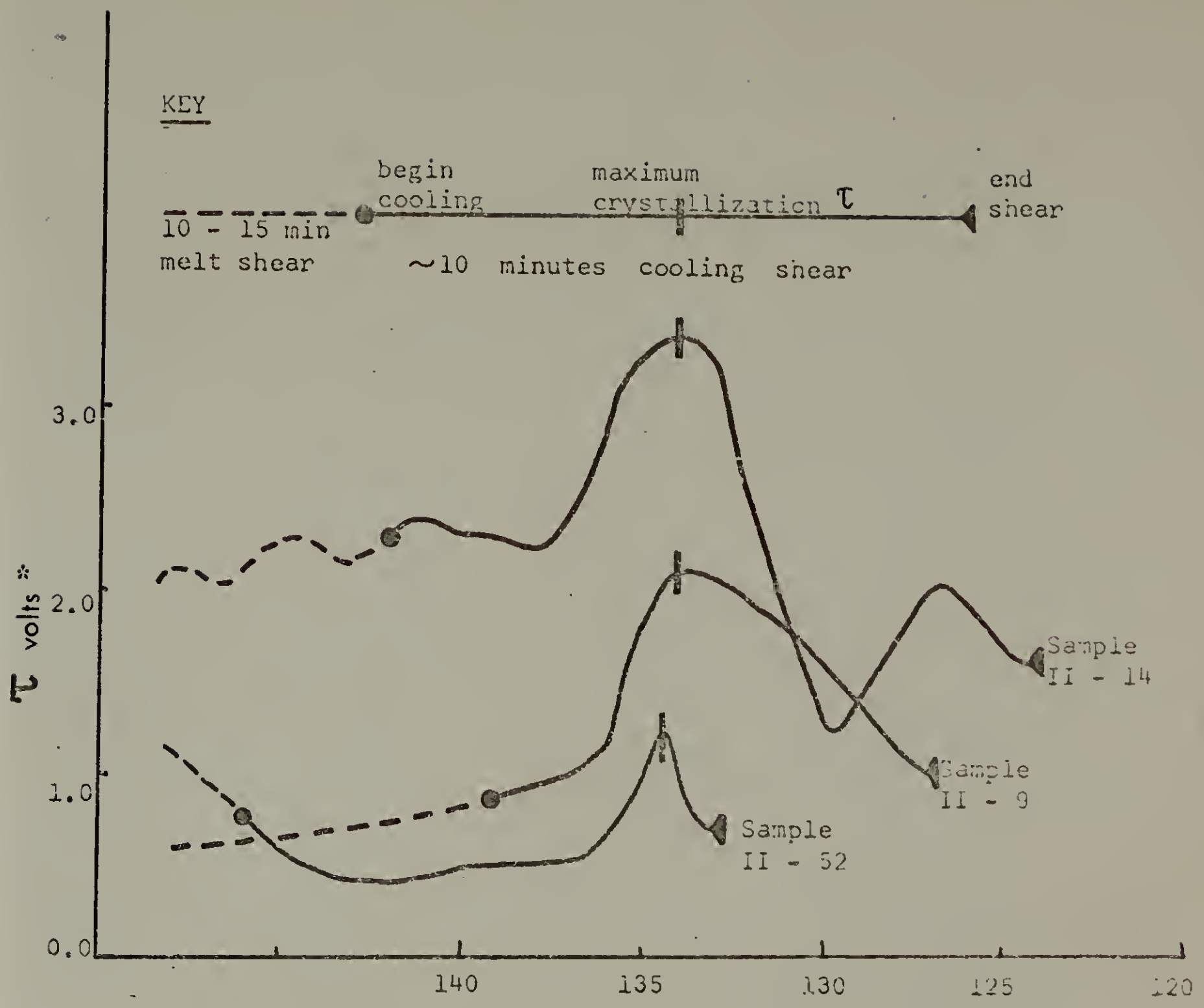


Fig 13- Viscous Heating During Shear – ALATHON 7050 at 134 °C

under these conditions and the film temperature is slowly lowered, simple shear crystallization of the film should occur. This corresponds to ~5X the estimated shear rate of capillary produced transparent strands (461 sec^{-1})[31] but without the pressure used in the latter (1900 ATM).[32]

2) Shear Crystallization Technique

Filling the cylinder clearance gap with a high viscosity polyethylene melt was a major difficulty and was accomplished as follows: With the ring and oil bath of the high shear viscometer held between 140 - 150°C, a special tamping tool is used to coat the inner surface of the ring and fill its sample inlet groove with melted polyethylene pellets. Care is taken to exclude as many air bubbles as possible. The spindle (#1) is then heated with a hot air gun to 140 - 150°C, and then slowly lowered into the ring. The spindle is locked in place and rotated by hand several times while a low pressure of N_2 (~90psi) is introduced via the sample inlet system. A polymer melt film of 1 - 2 in^2 in area can be formed in this fashion. After 1/2 hour equilibration under these conditions, shearing was begun at a constant rotational speed and continued until the torque reached a maximum (~10 minutes), at which time the heating bath and nitrogen pressure were cut off and ambient controlled cooling commenced (Fig.14). During this operation,



* Note: The absolute height of the torque voltage curve is not significant since it depends on the degree of partial filling of the cylinder gap with sample.

FIGURE 14 - SHEAR CRYSTALLIZATION OF POLYETHYLENE @ 2500 sec^{-1}

the heating bath "T" inlets acted as siphons, removing the heating oil from the cylinder reservior. Cooling was continued for about 10 minutes, at a rate of $\sim$ 1 1/2°C/minute. Shearing was continued until well below the crystallization (abrupt torque increase) region of the film, whereupon rotation was halted and cooling continued to near room temperature. A small open-end wrench was carefully applied to the spindle to remove it so that the polyethylene film could be removed and examined. In some cases it was necessary to heat the outer ring somewhat (to $\leq$ 80°C) in order to facilitate spindle removal.

3) Results

Properties of simple shear crystallized Alathon 7050 are summarized in Table VII, along with corresponding properties of capillary rheometer crystallized (shear and pressure) and bulk crystallized (no shear and no pressure) material. Most noticible was that transparency was not achieved under simple shear alone. The film had a mottled, fibrillar appearance. Inspection under polarized light (Fig.15) revealed regions of higher order orientation scattered in a less highly oriented matrix, indicating some degree of crystalline orientation in the shear direction. The melting peak and % crystallinity, determined by differential scanning calorimetry (DSC) also placed the properties of the shear crystallized material intermediate

TABLE VII - COMPARATIVE PROPERTIES OF SHEAR - CRYSTALLIZED ALATHON 7050 POLYETHYLENE

	CRYSTALLIZATION CONDITIONS		
PROPERTY	SIMPLE SHEAR ALONE[1]	SHEAR AND PRESSURE[2]	NO SHEAR AND NO PRESSURE[3]
APPEARANCE	OPAQUE, FIBRILLAR	TRANSPARENT	OPAQUE
MELTING PEAK[4]	135.7 °C	138.7 °C	131.6 °C
% CRYSTALLINITY[5]	82 %	85 %	70 %
THICKNESS	.0005 in	.05 in	.01 in est.
BIREFRINGENCE	.005	.054	-

NOTES:
1. Concentric cylinder viscometer crystallized
2. Capillary Rheometer crystallized (N. Weeks)
3. Bulk crystallized (as received)
4. By DSC at a 5 °C/min heating rate
5. Determined by area under the DSC curve; some heat loss suspected

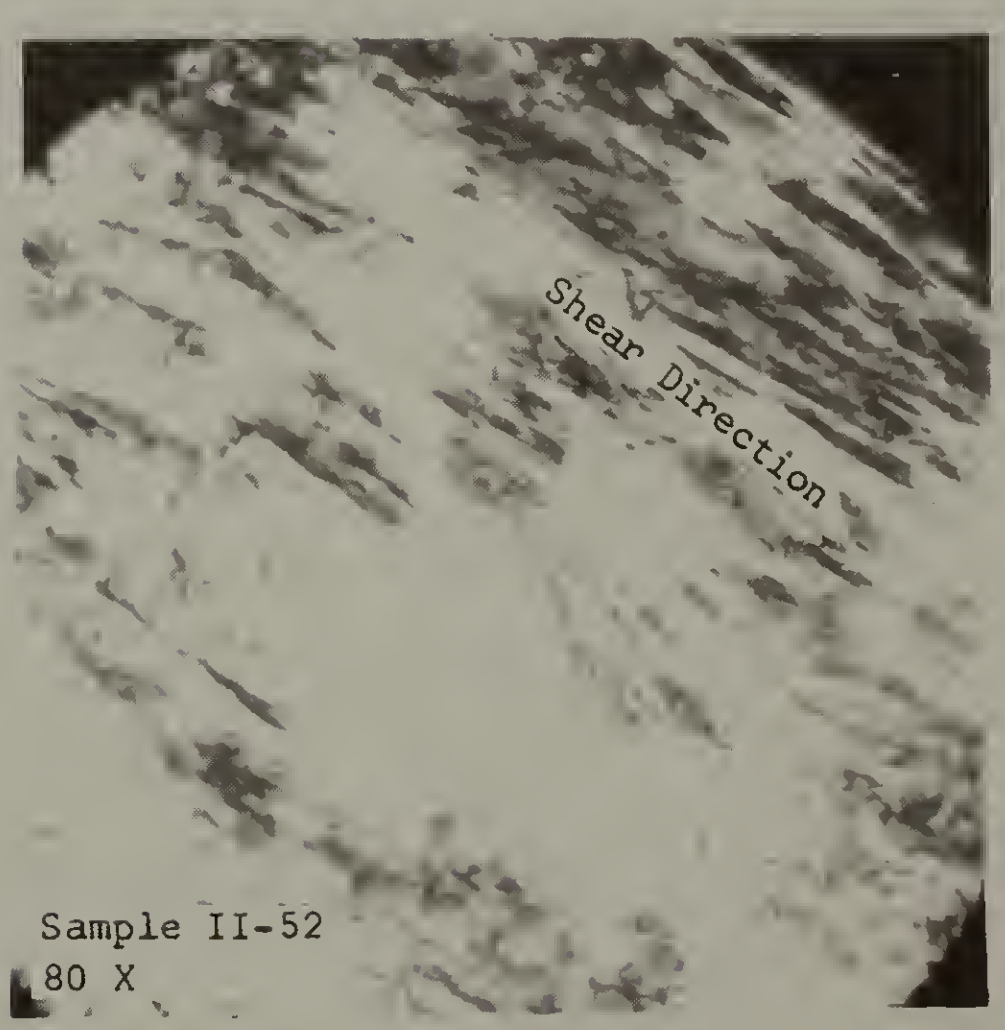


FIGURE 15 - SHEAR CRYSTALLIZED POLYETHYLENE SAMPLES UNDER POLARIZED LIGHT.

between the transparent strand and bulk crystallized polyethylene.. The thinness of the shear crystallized sample (.0005") prevented conventional molecular orientation measurements such as those using X-ray diffraction. Birefringence measurements were possible however, and again showed some orientation but significantly below that produced by shear and pressure together in the capillary rheometer. Because DSC measurements proved most sensitive to the property differences melting peak vs. heating rate curves were also obtained. Southern has previously demonstrated [31] that highly oriented transparent strands show 1) steeper superheating slopes, and 2) no evidence of low heating rate annealing. Again it can be seen that the properties of simple shear crystallized material lie intermediate between transparent and bulk crystallized material (Fig.16).

4) Conclusions

Melts of Alathon 7050 polyethylene crystallized under simple shear, in the absence of pressure, exibit some orientation, but at a level lower than that achieved by the combination of shear and pressure in the capillary rheometer. This leads to the conclusion that both shear stress and pressure are required to produce the transparent strand phenomenon,

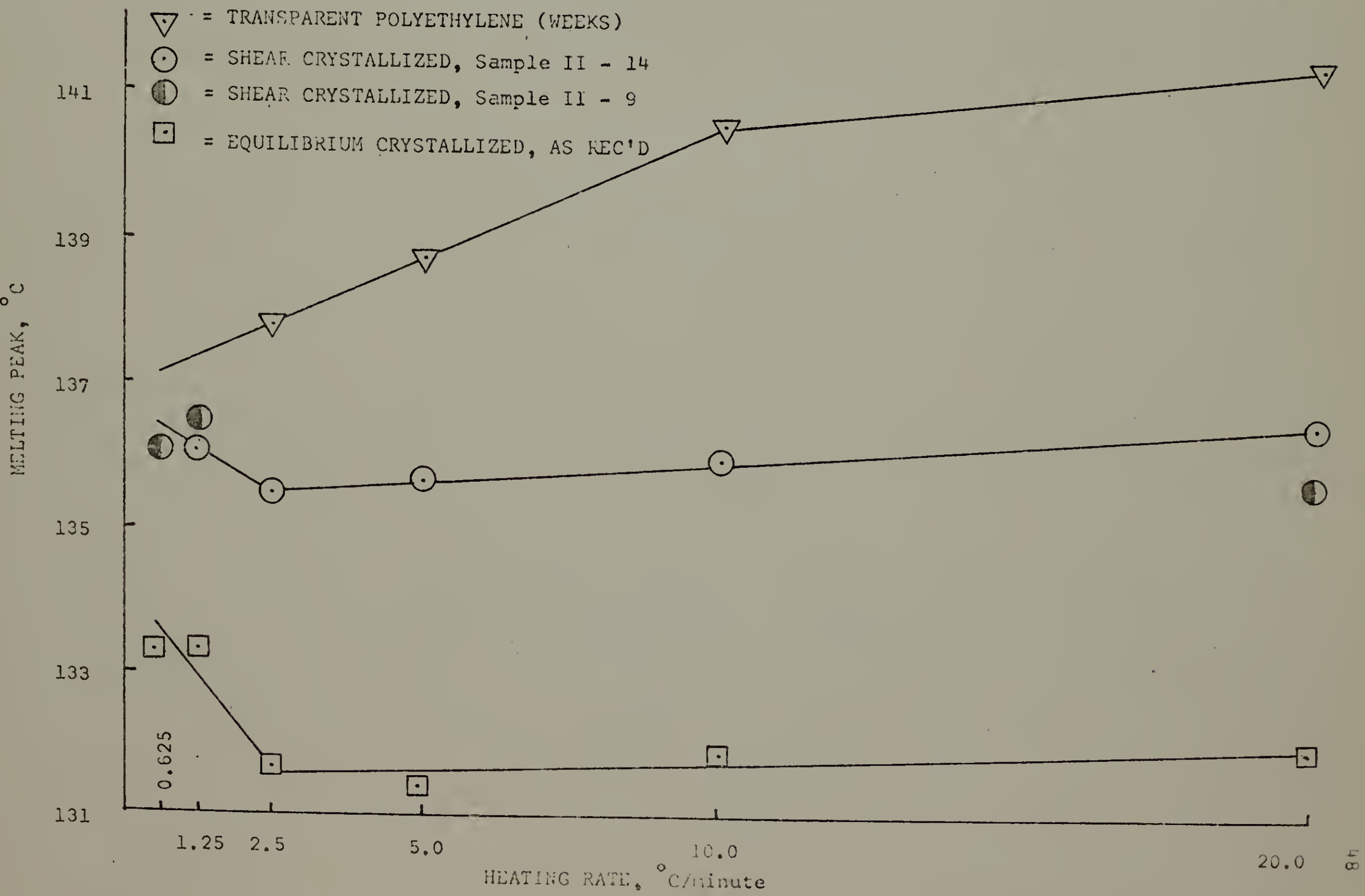


FIGURE 16 - EFFECT OF HEATING RATE ON THE PEAK MELTING TEMPERATURE OF ALATHON 7050 (by DSC)

since the shear rate is higher and more homogeneous in the concentric cylinder viscometer. Differences may be attributable to the drawing of partially crystalline material in the case of capillary extrusion and the elevation of melting temperature due to pressure effects. In any event, simple shear alone produces anisotropic but not highly oriented material in the case of high density polyethylene crystallized from the melt.

Note: Since this work was completed, two additional papers on the preparation of sheared transparent polyethylene have appeared. Kwei, Wang and Bair (Bell Laboratories, to be published) described a method of quench rolling molten film between cold metal rollers. An ill defined, though high shear rate of 10^5 sec^{-1} gave a transparent film with a high degree of molecular orientation in the flow direction. D. L. Krueger and G. S. Y. Yeh (University of Michigan, to be published) reported the preparation of a thick (.134"), partially transparent material at shear rates between 0.4 and 1.0 sec^{-1} in a Couette device under a hydrostatic pressure of $<$400 psi. While no extended chain material appears to have been produced in the latter case, electron microscope and x-ray studies revealed a fibrous morphology of ill-defined lamellae, well oriented in the direction of flow.

VII. FUTURE WORK

Recommended future work lies in two areas: hardware improvements in the viscometer and applications utilizing its high shear capability.

A. Hardware Improvements

1) A major improvement in viscometer operation can be achieved through tying in its readout system directly to some form of computer input. The viscometer electronics have been selected for compatibility with paper tape, punch card or on-line terminal inputs. A suitable program would also have to be developed for this purpose.

2) It has been pointed out earlier that an improved signal amplifier for the strain guage, replacing the currently used resistance bridge, would eliminate this unit as the weak link in an otherwise sophisticated readout system. This would also reduce readout error to .15%. The cost for this change should be less than $300.

3) Spindle alignment can be improved in two ways. A peelable shim laminate disk can be added between the spindle plug and its chuck. Thin sections of the laminate may then be removed so that upon tightening the chuck against the plug screw,

improved alignment will result (Fig.17). This can best be done while slowly rotating the spindle on a lathe. An alternative would be to use a more flexible drive shaft, such as the Stow Flexshaft (Stow Manufacturing Co., Binghamton, New York).

4) While the cylinder gap clearances determined near room-temperature are satisfactory for most viscosity measurements, calibration should be repeated for the narrower gaps at elevated temperatures. This is because the coefficient of expansion of the cylinder steel alloy (11×10^{-6} in/°C) could affect gap clearance due to any small amount of temperature inhomogeneity.

5) A further improvement to the self-contained syringe injection system would be achieved by machining a new syringe barrel and plunger from stainless steel, in order to allow higher injection rates without danger of syringe breakage. The fit between the plunger and barrel must be exceptionally good in order that the injected sample does not back-flow while it is being injected into the narrowest spindle/ring combination which gives the highest back-pressure. A light-weight spring-wound clock mechanism may be added to the syringe injector so that a continuous sample supply can be maintained during constant shear rate operation.

6) An interlock should be added so that the safety shield (Fig.2) cannot be opened while the viscometer is operating.

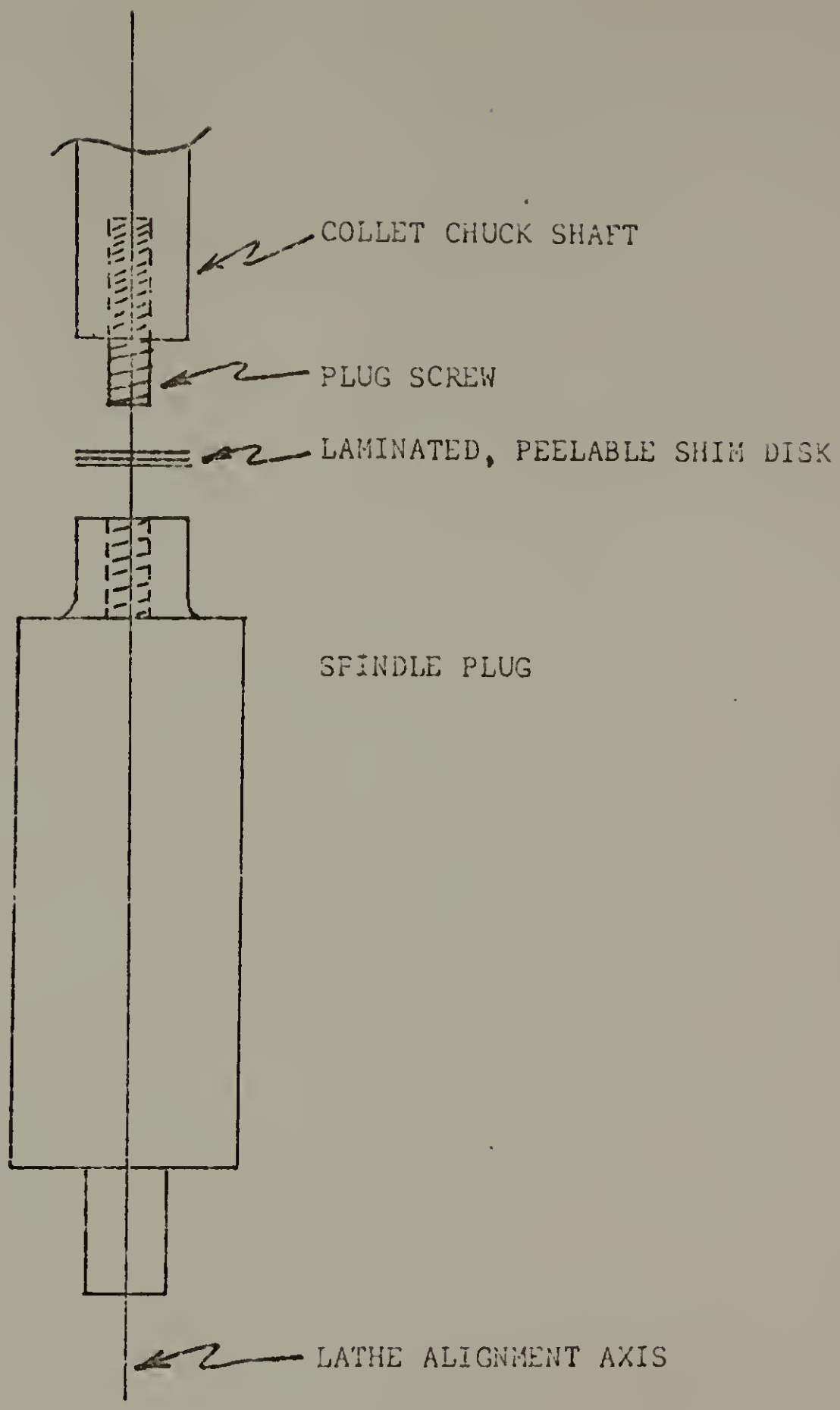


FIGURE 17 - IMPROVED SPINDLE ALIGNMENT WITH A LAMINATED SHIM DISK

B. High Shear Applications

1) So little has been published on the viscosity characteristics of polymers and solutions above 1,000,000 sec^{-1}, that studies on nearly any material in this shear rate range will give new state-of-the-art data. Polymers used in high-speed printing and paper-coating processes would be particularly suitable candidates.

2) There is special interest in polyethylene glycols as friction reducers in high-speed water flow conditions. This viscometer may be unique in its ability to examine this phenomenon under controlled laminar shear conditions.

[36]

3) Arai, et. al., has studied the effect of solvents on the high shear degradation characteristics of polystyrene solutions, but under highly turbulent conditions. It would be useful to extend this work to examine the effects of laminar shear on polystyrene degradation in solution.

4) The ability of this viscometer to monitor sample temperature at or near the film interface makes it suitable for study of viscous heating effects and rates over four decades of shear rate. Some preliminary data with Araclor [37] 1260, a 30,000

poise chlorinated polyphenyl plasticizer, indicates that this instrument is especially suitable for measuring viscous heating at high Brinkman Numbers.[38]

VIII. APPENDICES

APPENDIX I. HIGH SHEAR VISCOMETER OPERATION MANUAL

High Shear Viscometer Operation Manual

Operation of the high shear viscometer will be described in full detail in this manual. After you are familiar with these details, the OPERATION SUMMARY at the end of this manual can be used as an actual operation procedure. Reference to Figures 1 - 6 in the text, or to the viscometer itself will facilitate the use of these instructions.

A. Stand-By

1. The voltmeter and digital converter should be left on, with the voltmeter sampling rate on HOLD, unless the instrument will not be used for several weeks. If total shut-down has occurred, the voltmeter should be calibrated only after 1/2 hour warm up, and then several times thereafter in the first 24 hours of operation.

2. During shut-down and stand-by, the red torque arm guard should be kept in place on the viscometer measurement assembly.

B. Voltmeter Internal Calibration

1. With the sampling rate on maximum and the range selector on 100 MV, set the ZERO adjust screw with a small screwdriver or

spatula so that the readout cycles between + and - for 0.00 MV. This is most readily done when the selection switch is on SHEAR RATE and the throw lever to the drive motor power supply is OFF, thereby minimizing electrical leakage across the ON/OFF button switch. A $\pm$ oscillation of several hundredths of a millivolt is acceptable.

2. Similarly, set the INT. CHECK to read -8.000 V when the internal check button is depressed. The position of the decimal place is not significant for this check.

3. Repeat the above procedure at a minimum of twice per operating day, preferably at the beginning and at the end of a series of measurements.

4. Refer to Hewlett-Packard Manual 3440A/3443A for additional detail.

C. Digital/Analog Converter

1. The power switch for this unit may remain ON, as with the voltmeter. The COLUMN SELECTOR is set on columns 2-3-4 for high shear viscometer operation, due to the internal wiring of the rear plug-in connector. Calibration is done with the recorder and is given in section D.

2. Refer to Hewlett-Packard Manual 580A/581A for additional detail.

D. Recorder

1. The recorder should be OFF when not actually in use, although the clutch lever (MIN/HR) may be used to interrupt operation for short periods of time. Normal operation is on the 100 MV scale.

2. Set the zero by placing the Digital/Analog Converter throw switch on GALV. ZERO and bring the recorder pen in line with the chart zero using the ZERO adjustment wheel on the recorder.

3. Swing the converter throw switch to the CALIBRATE position, and use the CALIBRATE knob on the Converter to set the recorder pen to full scale.

4. Repeat steps D-2 and D-3 until both zero and full-scale are accurately set. Return the throw switch to OPERATE.

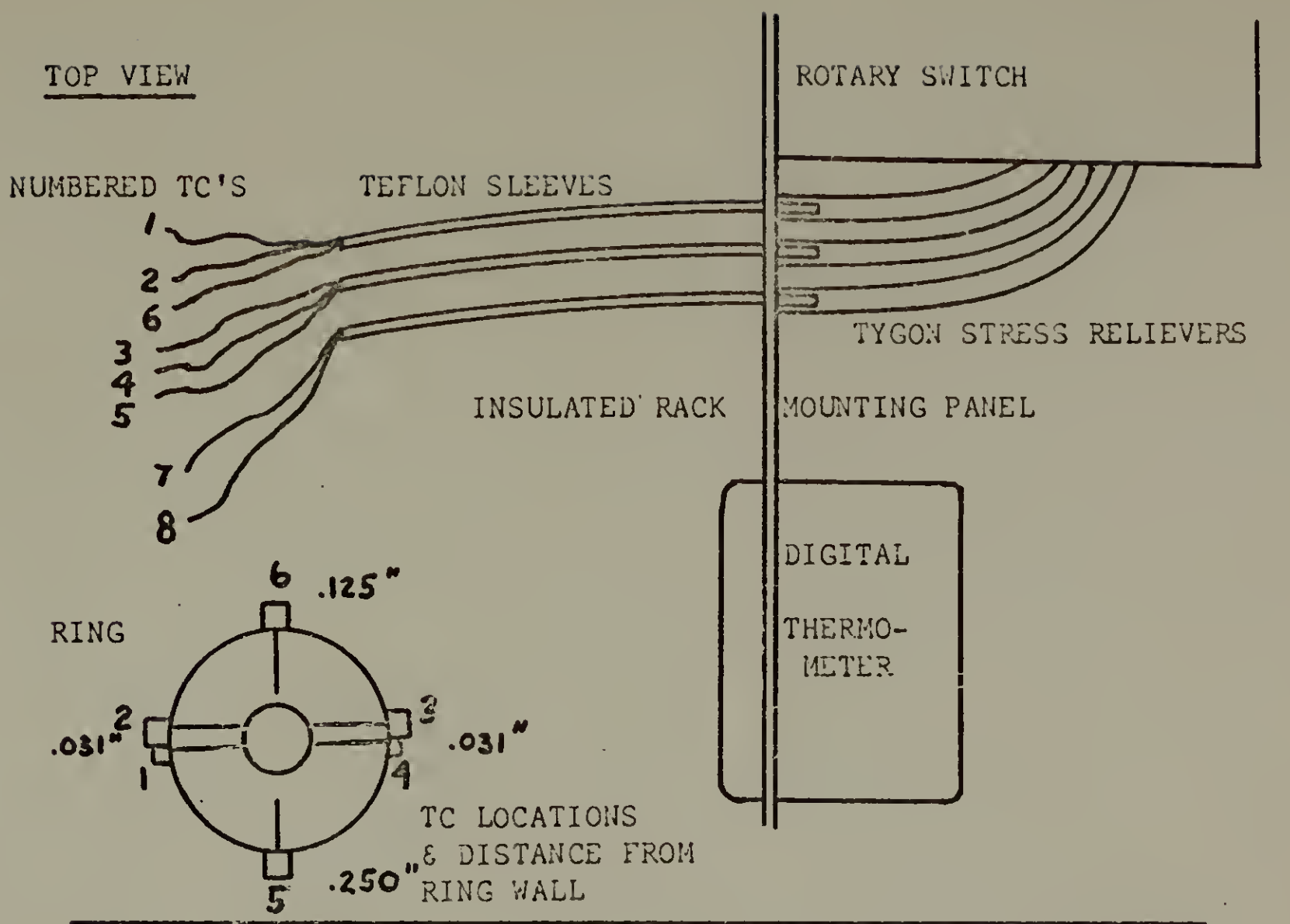
5. Additional detail may be obtained from Hewlett-Packard Manual HP680.

E. Digital Thermometer

1. The digital thermometer should be turned on at least 15 minutes before using. Since this is an electro-mechanical device, it is desirable to minimize unnecessary wide temperature swings; therefore, the thermocouple selector switch should either be OFF or set on a stable ring thermocouple position.

2. The copper-constantan thermocouples are delicate because of their fineness and the brittleness of their welds. Electrical opens develop at times and show up as wide swings in the read-out or by a rapid excursion of the readout to a maximum (meter-pegging). A properly welded thermocouple of this type will show an internal resistance of 67Ω. A resistance higher than this indicates a poor (though possibly "functioning") weld and will give non-linear errors in temperature readout. Thermo-couple construction is detailed in Figure E-1.

3. The thermocouple/digital thermometer system functions best if it is floating, i.e. grounded to neither the console chassis nor the viscometer ring. Floating prevents the development of ground loops (Hewlett-Packard Application Note 123, Floating Measurements and Guarding, June 20, 1970). Care must be taken to insure that the thermometer panel is insulated from the chassis mounting screws, and that the thermocouple tips them-selves are coated with a thin layer of epoxy.



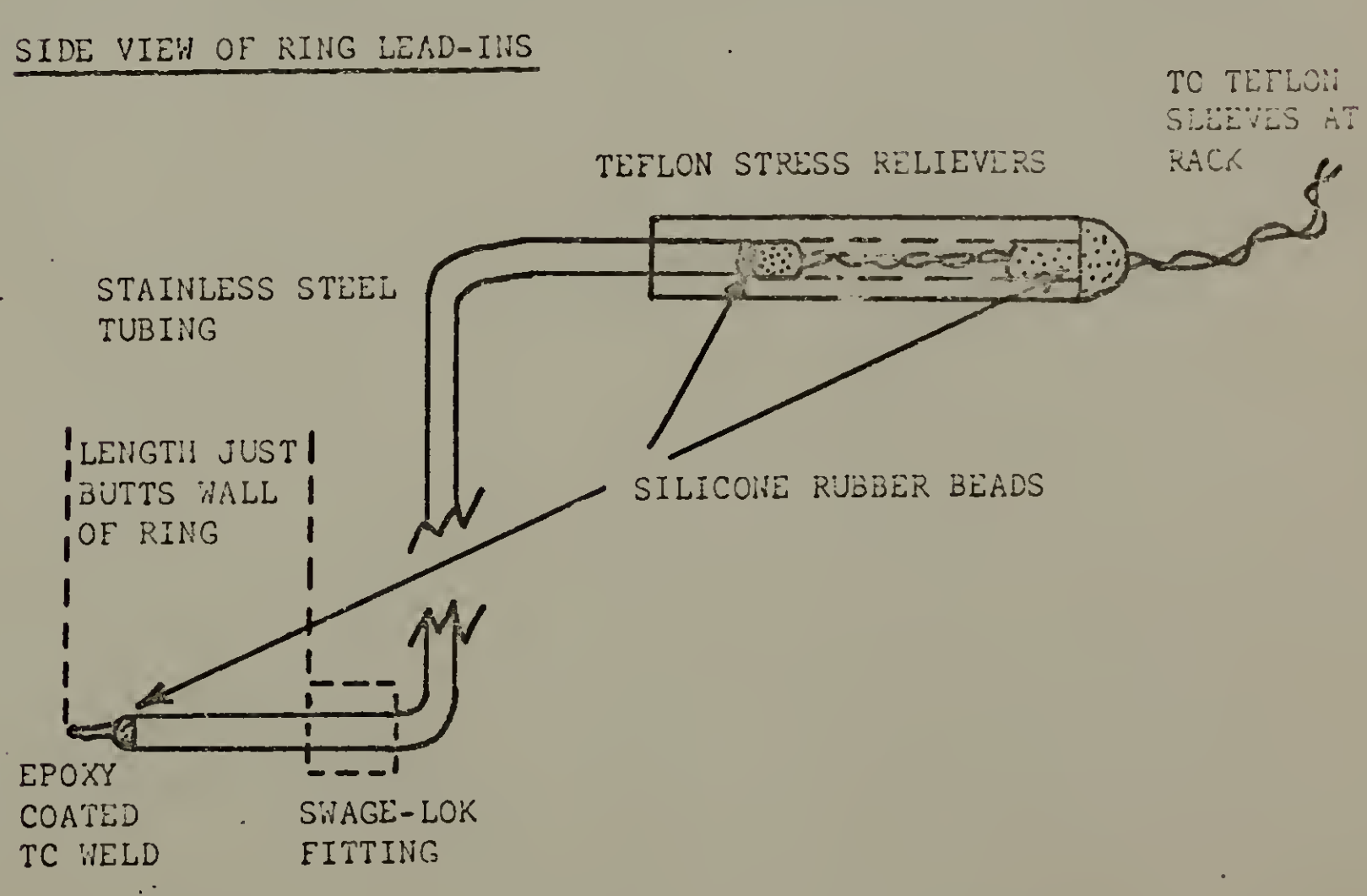


FIGURE E-1 - THERMOCOUPLE CONSTRUCTION DETAILS

4. The thermocouples and digital thermometer may be calibration-checked in an ice bath, using a National Bureau of Standards mercury thermometer as a cross check. The readout should be within ± 0.2°C of the bath for all couples.

F. Strain Gauge

1. The red torque arm guard should be in place at all times, except when the strain gauge is actually in use. This is necessary in order to protect the transducer from overstress.

2. The Statham Bridge Amplifier should be turned on at least 15 minutes before use. A satisfactory operating range is obtained by turning the BALANCE to the full clock-wise direction, and setting the SENSITIVITY to read 5.00X volts on the "10 V" volt-meter range scale. All of this is done with no load on the strain gauge. The BALANCE control is then backed off to read +.001 to +.005 V on the same scale. This slight positive voltage is desirable in order not to undershoot the zero setting. During the course of a run, a zero drift of +.01 to +.02 V is not significant so long as the magnitude of the torque measurements made are > 1 V.

3. The amount of weight used on the torque arm to set the SENSITIVITY knob of the bridge is as follows:

STRAIN GAUGE/WT	ARM LENGTH	BRIDGE SENSITIVITY SET TO READ:
0.5 lb / 100 gms	10 in	5.00 V
2 lb / 500 gms	10 in	5.00 V
10 lb / 700 gms	4 in	3.00 V

In the last case, the 200 gm weight should be hung from the 500 gm weight so that only a single filiment is actually attached to the torque arm. The above bridge settings may actually exceed those recommended by +.02 V without serious error in the calibration.

4. If additional weights are used to check calibration, the resulting torque curves should match those in Appendix II.

5. Force on the transducer is calculated as follows:

$$F_R = \frac{12.25\, F_C}{L'}$$

where: F_C = calibration weight used in grams (force)

L' = torque arm transducer position setting (in)

F_R = grams (force) on transducer.

6. Shear stress at the cylinder wall is given by:

$$S_{dyn/cm^2} = \frac{F_R \times L'_{cm} \times a}{A \times r} = 20.3\, F_R \times L'_{cm}$$

where: a = acceleration due to gravity (980 cm/sec^2)

A = shear area of cylinders (38.0 cm^2)

r = radius of inner cylinder (1.27 cm)

7. Additional details of operation are given in the strain gauge instruction booklet.

G. Drive Motor

1. For reasons of safety, the high voltage throw switch on the side of the viscometer support frame should be open (off) at the end of each days operation as well as whenever the drive gears are handled, and/or whenever a spindle is attached or removed.

2. Once the high voltage throw switch is closed (on) several minutes should be allowed to pass before the START-STOP control switch is utilized. This allows controller electronics to charge up and prevents surge upon starting the motor. Surge control may be further adjusted through the internal PEDESTAL ADJUST potentiometer in the controller. See the Ratiorol E Series Instruction Manual for additional details.

3. The motor should always be stopped with the STOP-START buttons, and never with the controller potentiometer. Potentiometer settings below 5 V may stop the motor, but can also cause motor insulation failure due to overheating.

4. Motor speed has been confirmed with a strobe light to better than ±1% (Fig.G-1).

H. Gear Reduction

1. Drive rate reduction is accomplished mechanically in a 1:10 ratio by removing two hex screws from the override clutch at the uppermost rear of the gear system. To prevent operator injury, the drive motor high voltage throw switch should be OFF when this adjustment is made.

2. Instrument calibration constants (K in Table II) should be divided by 10 when gear reduction is used.

3. The clutch, located above the drive shaft, should be topped off with 20W oil at least annually. The gear grease fittings may also be lubricated at this time.

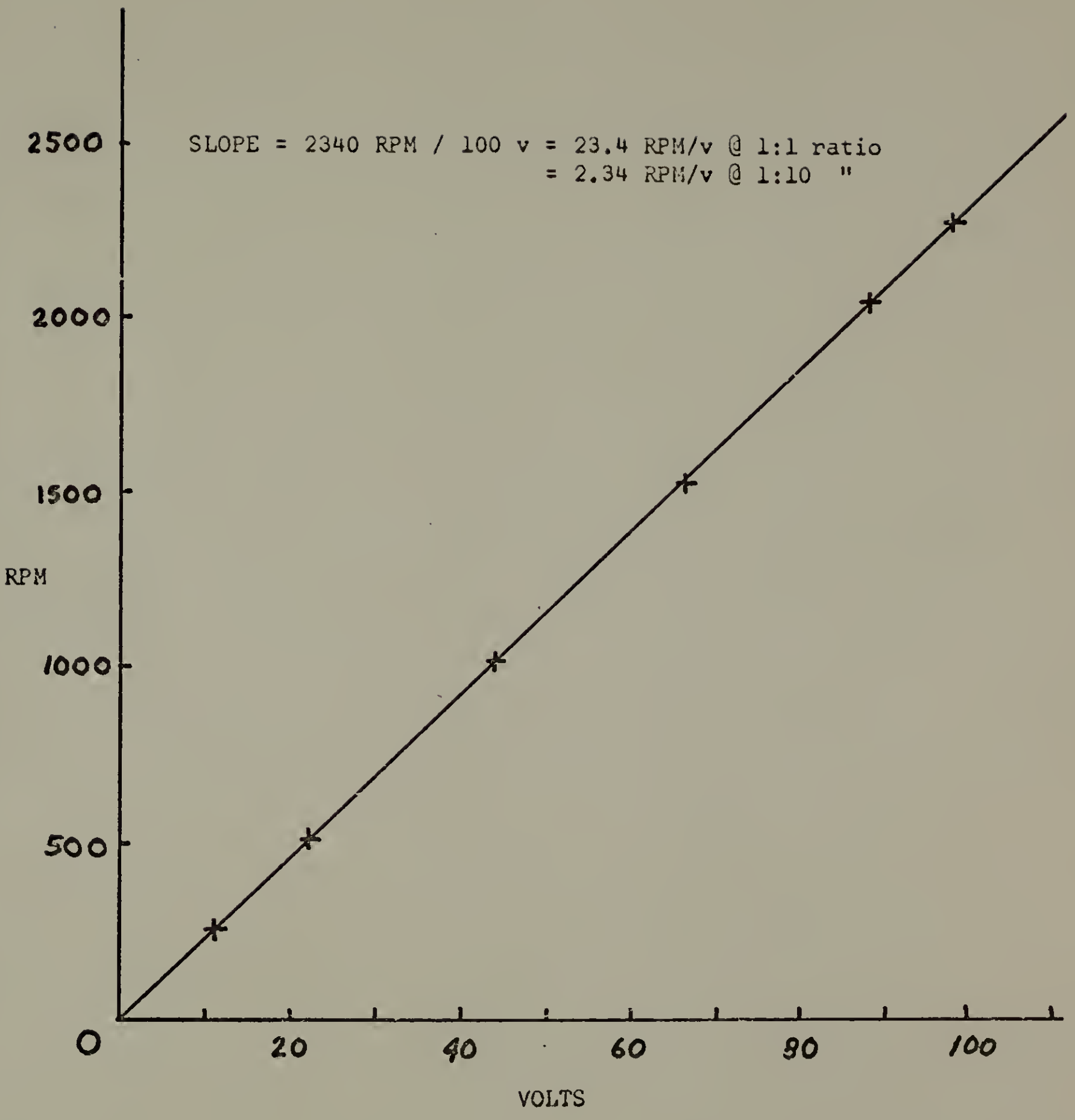


FIGURE G-1 - CALIBRATION OF DRIVE MOTOR SPEED

I. Spindle Placement

1. The spindles and drive shafts are installed by means of high precision collet chucks of 1/8, 3/16 and 1/4 inch sizes. The latter should be carefully tightened with two opposing wrenches. Slippage of the drive shaft in either chuck may score the drive shaft and make its removal from the chuck very difficult. New shafts may require a light emery-cloth buffing in order to fit them to their collets. (SAFETY NOTE: Drive shafts may shatter and whip if they break during high shear operation, therefore always operate with the safety shield closed. It is also advisable to wear safety glasses or goggles).

2. Collet chucks are most easily untightened if opposing wrenches are used at an angle of 15° or less with each other. The wrenches can then be operated with one hand, preventing uncontrolled slipping of the spindle and possible damage to its lapped surface.

3. Spindles are placed at the proper depth in the ring by slipping the SPINDLE LOCATOR tool in the locator groove on each spindle shank. This tool insures that wear marks on the spindle surfaces always occur in the same location and avoids spindle surface damage. The HEIGHT BLOCK is used with the LOCATOR when just 1/2 of the shear area is used.

4. The spindles and ring should be coated with a thin film of oil when they are to remain exposed to air for more than 24 hours at a time, in order to avoid rust formation on their surfaces. When not in use, the spindles should be stored in a dessicator filled with an active drying agent such as "Dri-Rite"(W.A. Hammond Co., Xenia, Ohio).

5. Avoid touching spindle surfaces with fingers. If fingerprints are deposited, wipe them immediately with a clean tissue to avoid corrosion.

J. Sample Injectors

1. Either the screw driven syringe (Fig.3) or the hydraulic pump (Fig.5) may be used to introduce sample, through the use of proper Swage-Lok fittings.

2. Of the two available sample injectors, the syringe has the advantage of easier cleaning, but care must be used so that its glass housing is not shattered through over tightening of the screw holder. The syringe may also be left in place during shear measurement operation.

3. The hydraulic is capable of higher sample injection pressure, but it is difficult to clean, and must be disconnected during

shear measurements. A "quick-disconnect" fitting is used for this purpose, but the latter introduces a small amount of air each time it is re-connected. Thus, after re-connection, care must be taken to pump in enough fresh sample in order to expel all air.

4. Excess sample of low viscosity quickly drains from the base of the viscometer and also collects at the top of the spindle. For high viscosity fluids (>10 poise) care must be taken to avoid excessive build up of fluid at the top of the spindle, since this may affect torque readings.

5. There is "dead volume" of about 1/2 cc on each side of the ring where the sample injector is connected. Thus, during sample changeover, sufficient "new" sample must be flushed through the injector to clear this of old material.

K. Thermostatic Bath

1. The HEAT CAPACITY setting knob should be set at full counter-clockwise (off) when turning on the circulation bath in order to prevent fuse overload. After bath is running, HEAT CAPACITY may be turned up.

2. For mercury switch thermometer settings below 70°C, a source of cooling water must be provided in order to achieve accurate temperature control. A container of "dry ice" in the bath will also accomplish this.

3. Whereas silicone bath oil should be odorless up to 150°C, some odor may be present at high temperature due to contaminents which can get into the circulation oil when improper draining of the sample collector (under the ring) occurs. Therefore, when operating at elevated temperature, it is best to keep all circulation valves closed in order to confine hot oil to the main reservoir while it heats up. Once proper temperature is attained, valves may be opened and the ring will reach equalibrium in 20 to 30 minutes.

4. The spindle must not be raised when its oil supply valve is open. Otherwise silicone oil contamination of the ring walls will result.

5. Circulation of heating oil through the viscometer assembly should only be done when an operator is in attendance. This is because overflow of the catch basin can occur as the oil heats up and undergoes viscosity reduction.

L. Cleaning

The injector, ring and spindle system may be cleaned with any suitable solvent. A tissue held by wooden-tipped tongs will assist ring cleaning. Adequate flushing will insure that all dead volume in the injector system is cleared. A final cleaning with acetone or some other volatile solvant, followed by multiple syringe injections of air will remove solvent traces.

M. High Shear Viscometer - Operation Procedure Summary

1. Stand-by

   a) voltmeter and converter ON

   b) sampling rate on HOLD

   c) recorder, thermometer, bridge amplifier and drive motor throw switch OFF

   d) bridge and thermometer warm up time 15 minutes

2. Voltmeter Calibration

   a) ZERO set to $\pm$0.00 MV on SHEAR RATE selector setting with drive motor throw switch open (OFF)

   b) INT. CHECK to -8.000 V

   c) repeat a) and b) twice a day

3. Recorder/Converter

   a) adjust recorder ZERO with converter in GALV. ZERO position, using adjustment wheel on the recorder.

   b) adjust full scale with converter in CALIBRATE position, using CALIBRATE knob

   c) return converter to OPERATE

4. Strain Gauge

   a) red TORQUE ARM GUARD in place at all times, except during measurement

   b) BALANCE at full clockwise position and SENSITIVITY set at +5.00X V on 10 V scale

   c) turn BALANCE counterclockwise until voltage is +0.005 V or less

   d) refer to section F.3 of Appendix I or to calibration charts (Appendix II) for load/voltage settings

5. Drive Motor/Gear Reduction

   a) use the STOP/START buttons (never the controller potentiometer) to shut off drive motor

   b) main drive throw switch open (OFF) during stand-by, gear changeover and shutdown

   c) two hex screws removed from rear uppermost gear for 1:10 drive reduction (adjust instrument constants accordingly)

6. Spindle Placement

   a) avoid touching surfaces

   b) locate depth with special tool

   c) store spindles in desiccator when not in use

7. Sample Injectors

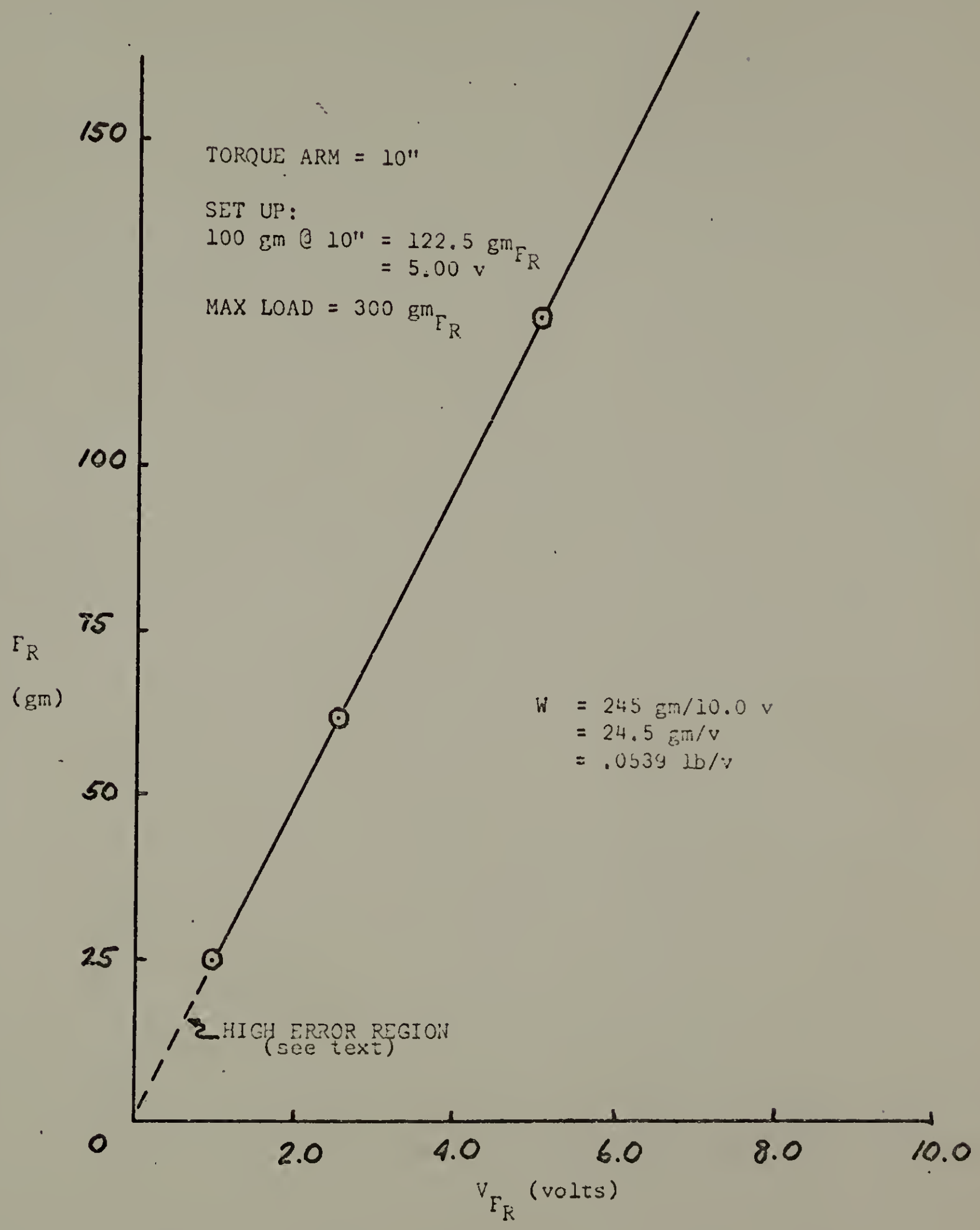
   a) inject sufficient sample to fully expel air entrapped when hydraulic quick-disconnect is attached

8. Thermostatic Bath

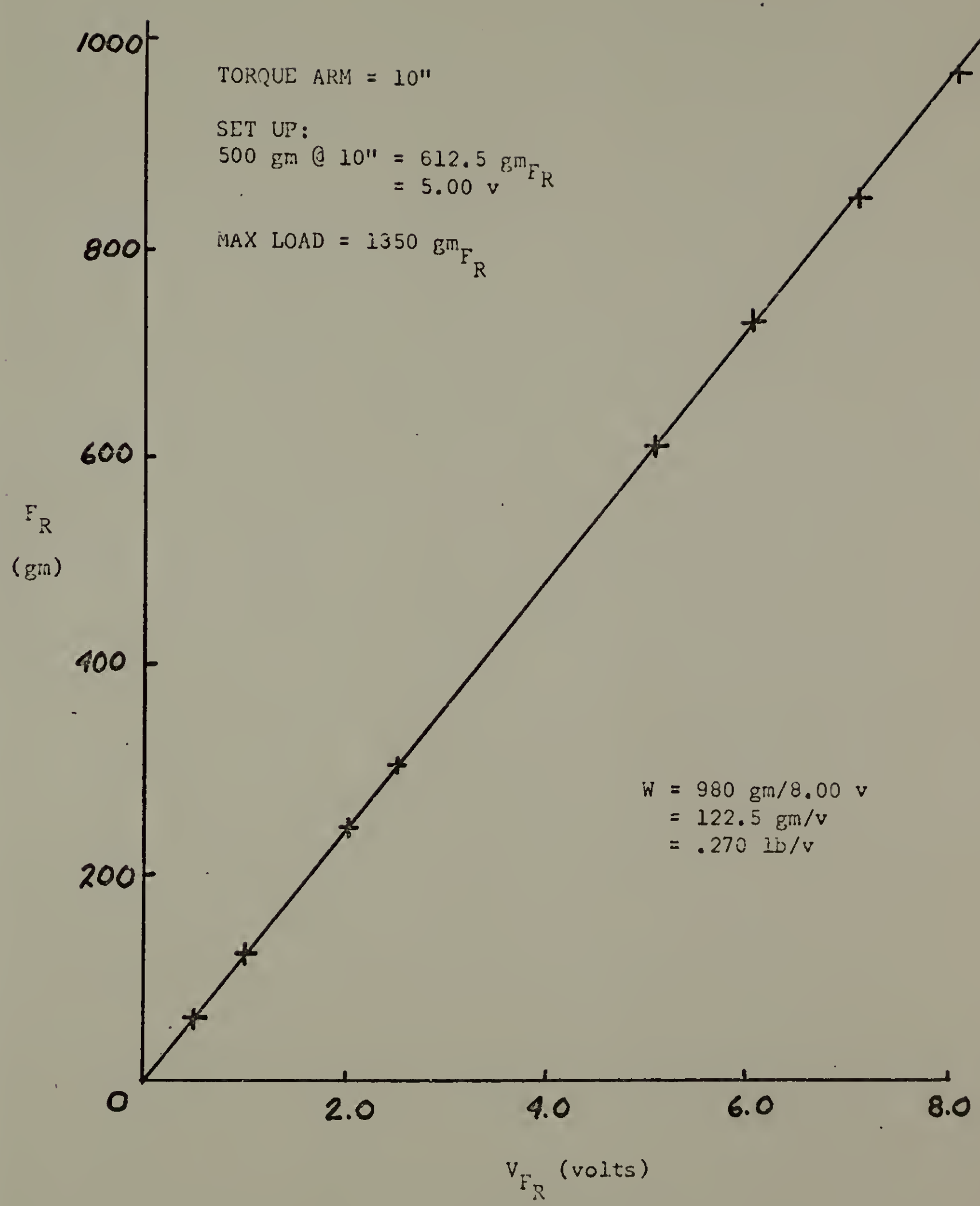
   a) HEAT CAPACITY switch off when starting bath

   b) never raise spindle without first shutting off its oil supply valve

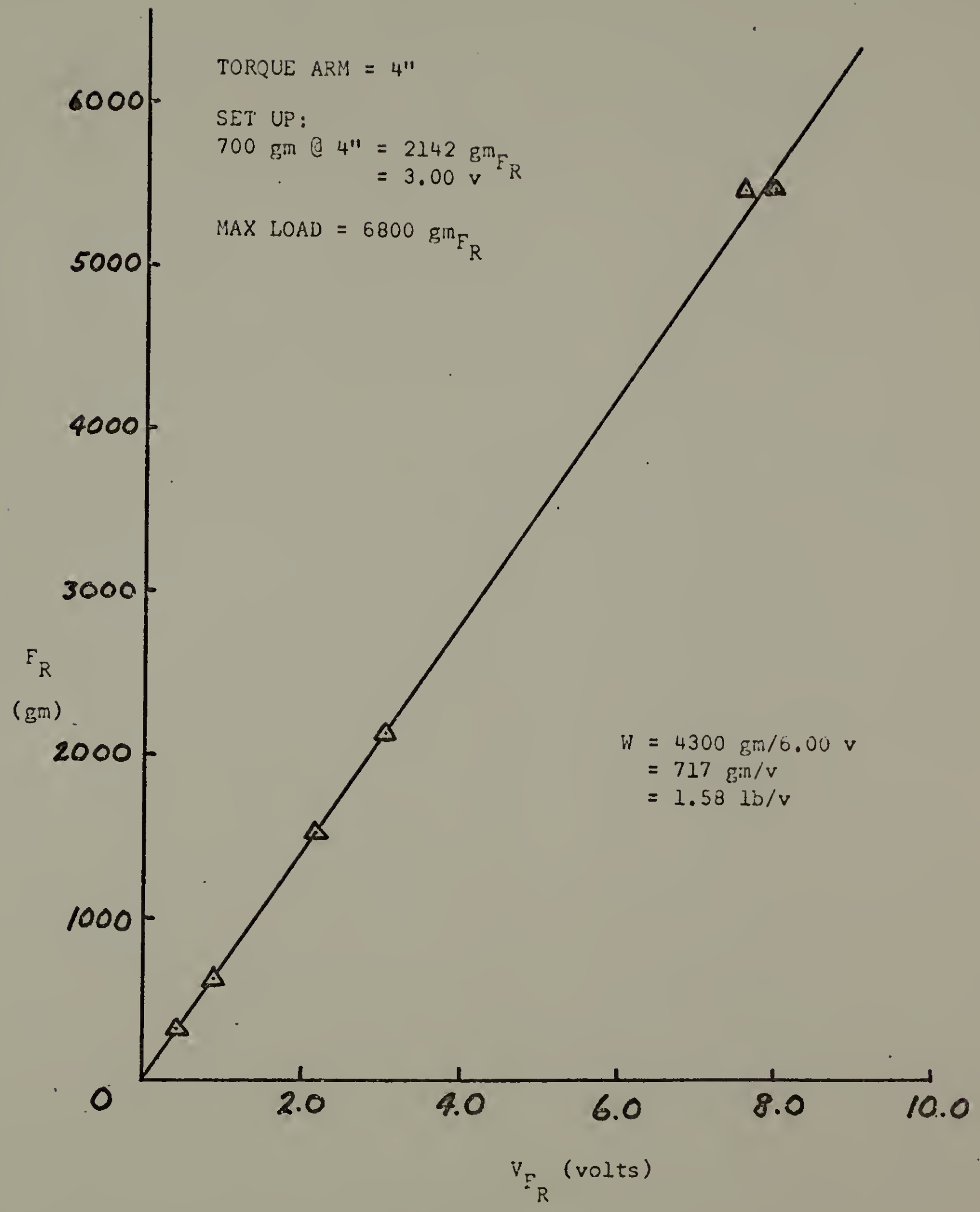
APPENDIX II - STRAIN GUAGE CALIBRATION CHARTS



CALIBRATION OF 0.5 lb STRAIN GUAGE

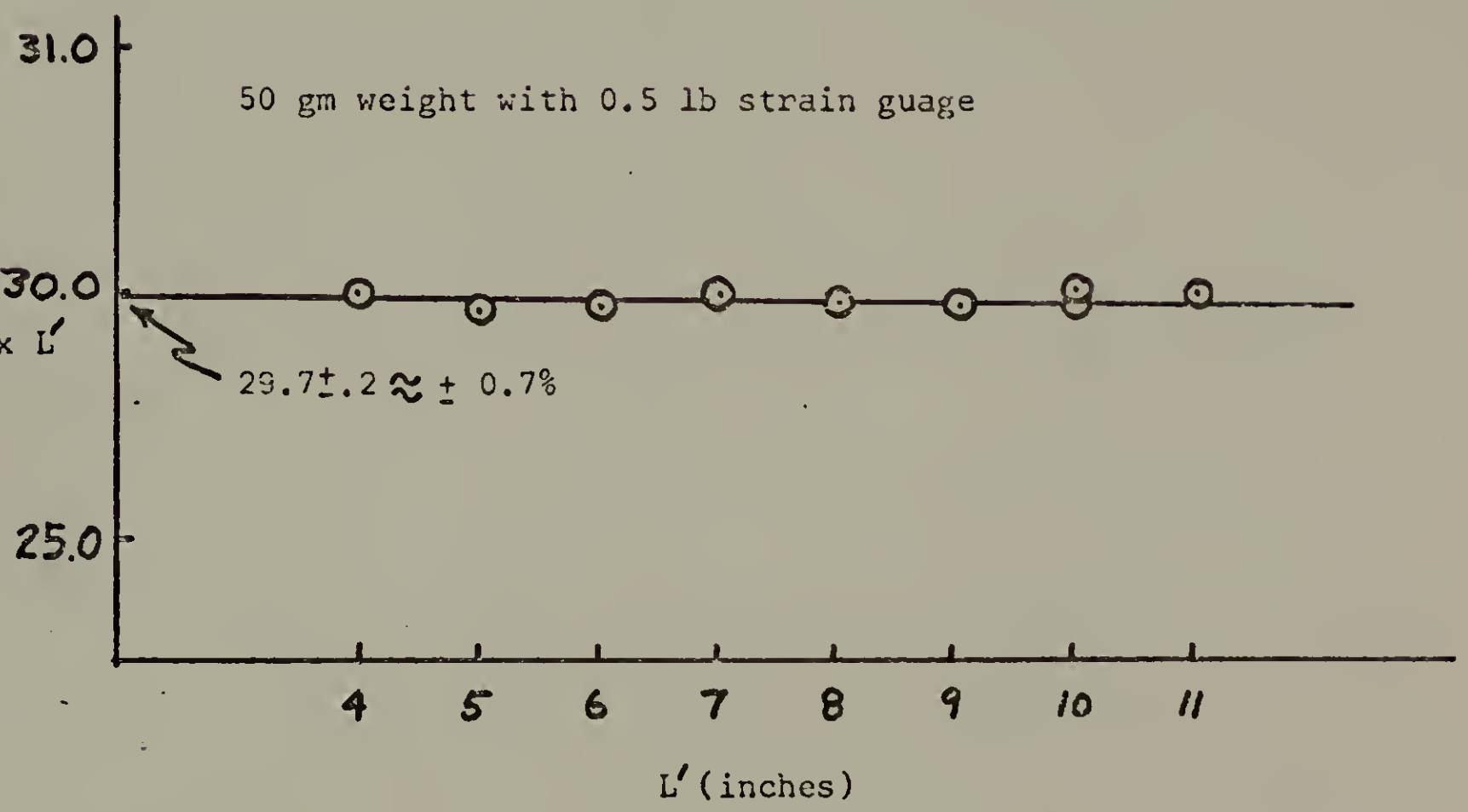


CALIBRATION OF 2 lb STRAIN GUAGE

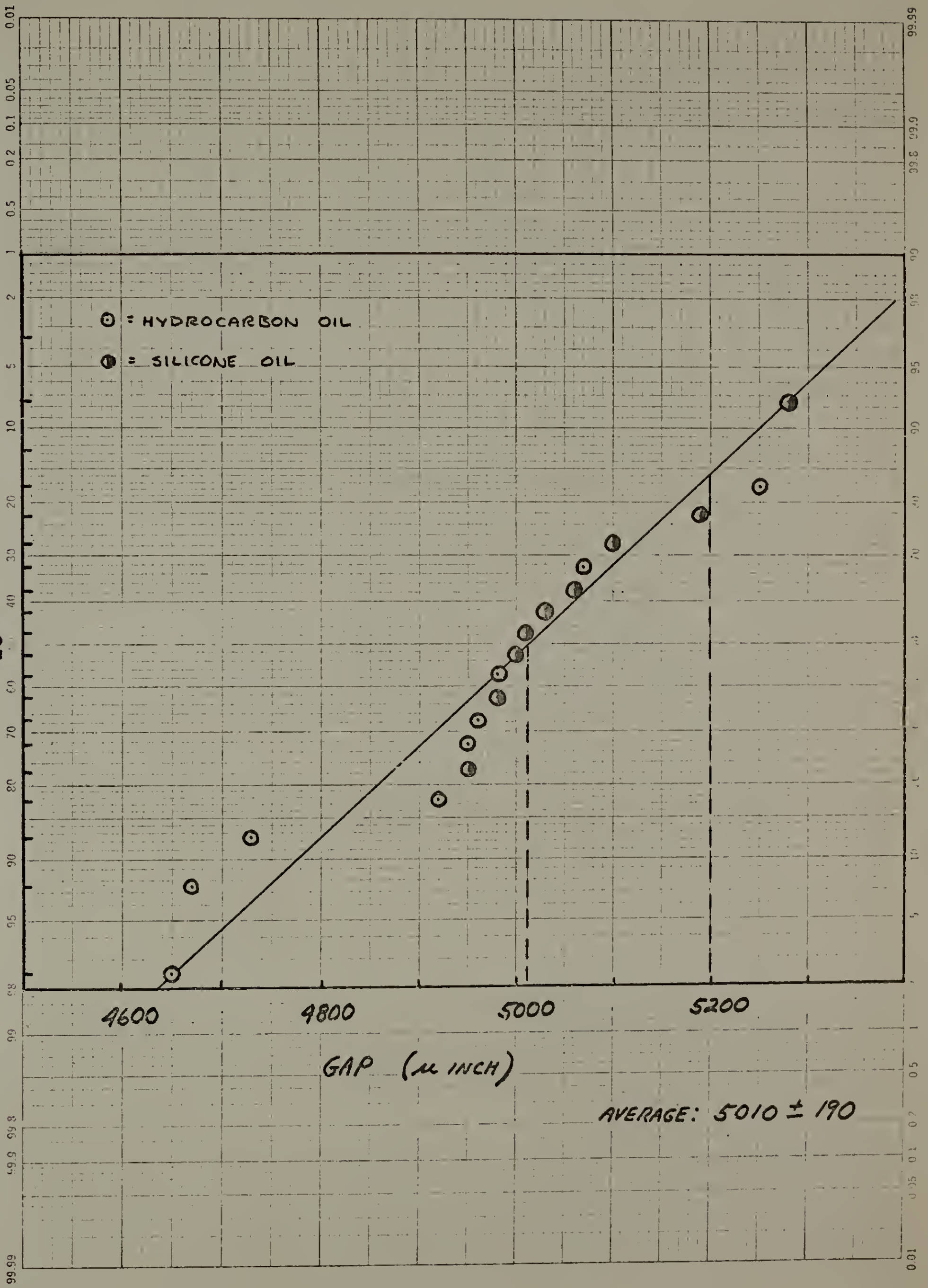


CALIBRATION OF 10 lb STRAIN GUAGE

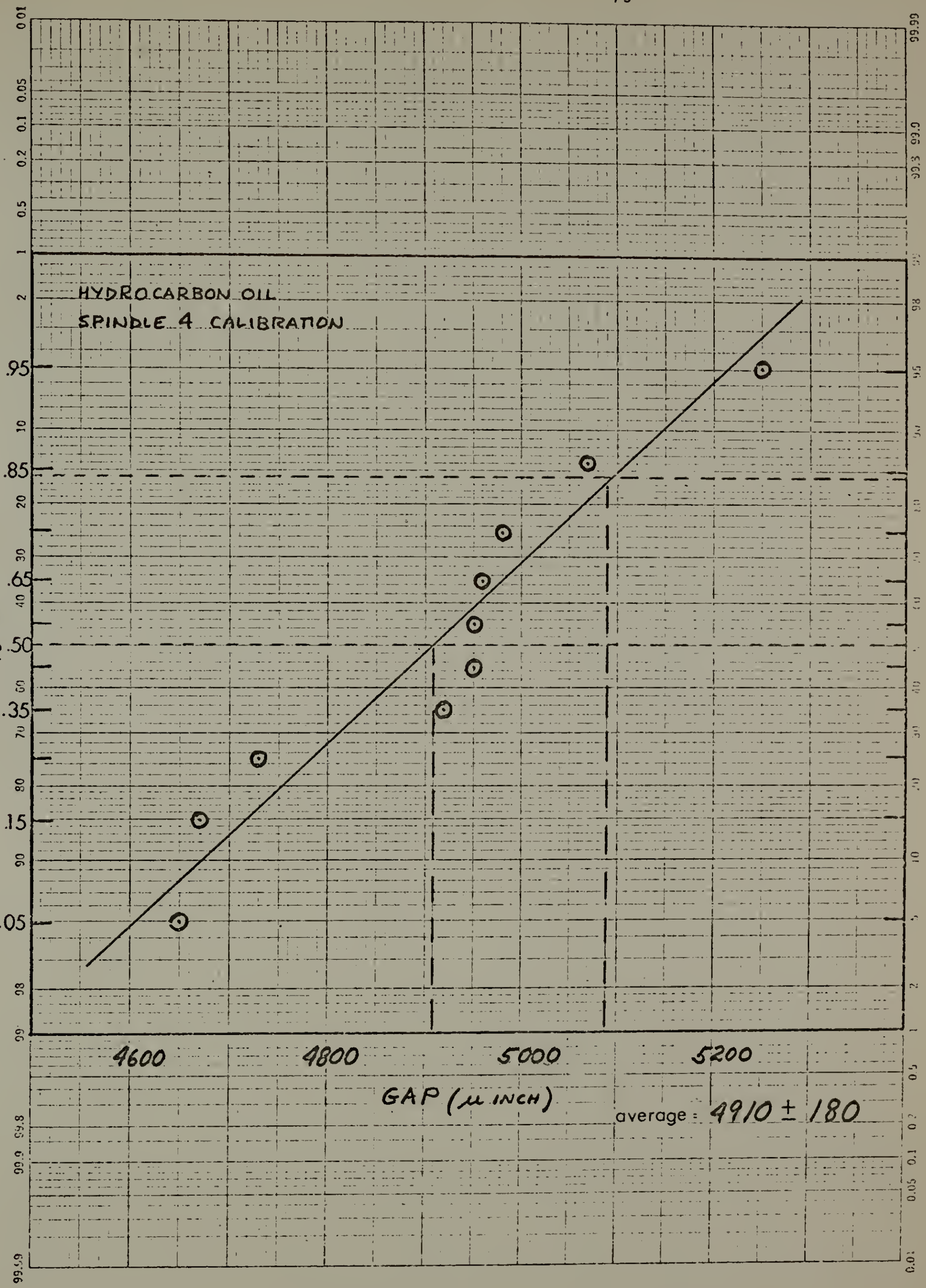
APPENDIX III - TORQUE ARM LINEARITY



APPENDIX IV - SPINDLE 4 CALIBRATION



Probability Scale x 90 Divisions



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. II-98 A,B

80

TEST CONDITIONS

sample: 1300 cps HYDROCARB. OIL S-600-65-2d

thermostat bath flow settings

heater: off ☒ boost ☐ max accuracy ☐

to pot: OPEN

to inner cyl: OPEN

" coil: AIR

box inlet: CLOSED

date 3/21/72

operator SM

temp. RT

spindle no. 4

drive shaft: 1/8"

" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 5"

load cell: 0.5 LB

chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 50 gm	100/20/10
initial	± 0.00 MV	-8.000	+ 0.01 V	+5.03	10.35/1.82/.7
after run	± 0.00	-8.000	+ 0.01	+5.04	10?/1.88/.8
NEW SPL	± 0.00	-7.999	+ 0.01	+5.00	10.1/1.82/.7

TEMPERATURE

1	2	3	4	5	6	7 RT	8	POT	
28.3	—	28.2	—	28.2	28.2	27.0		27.5	

DATA

SHEAR

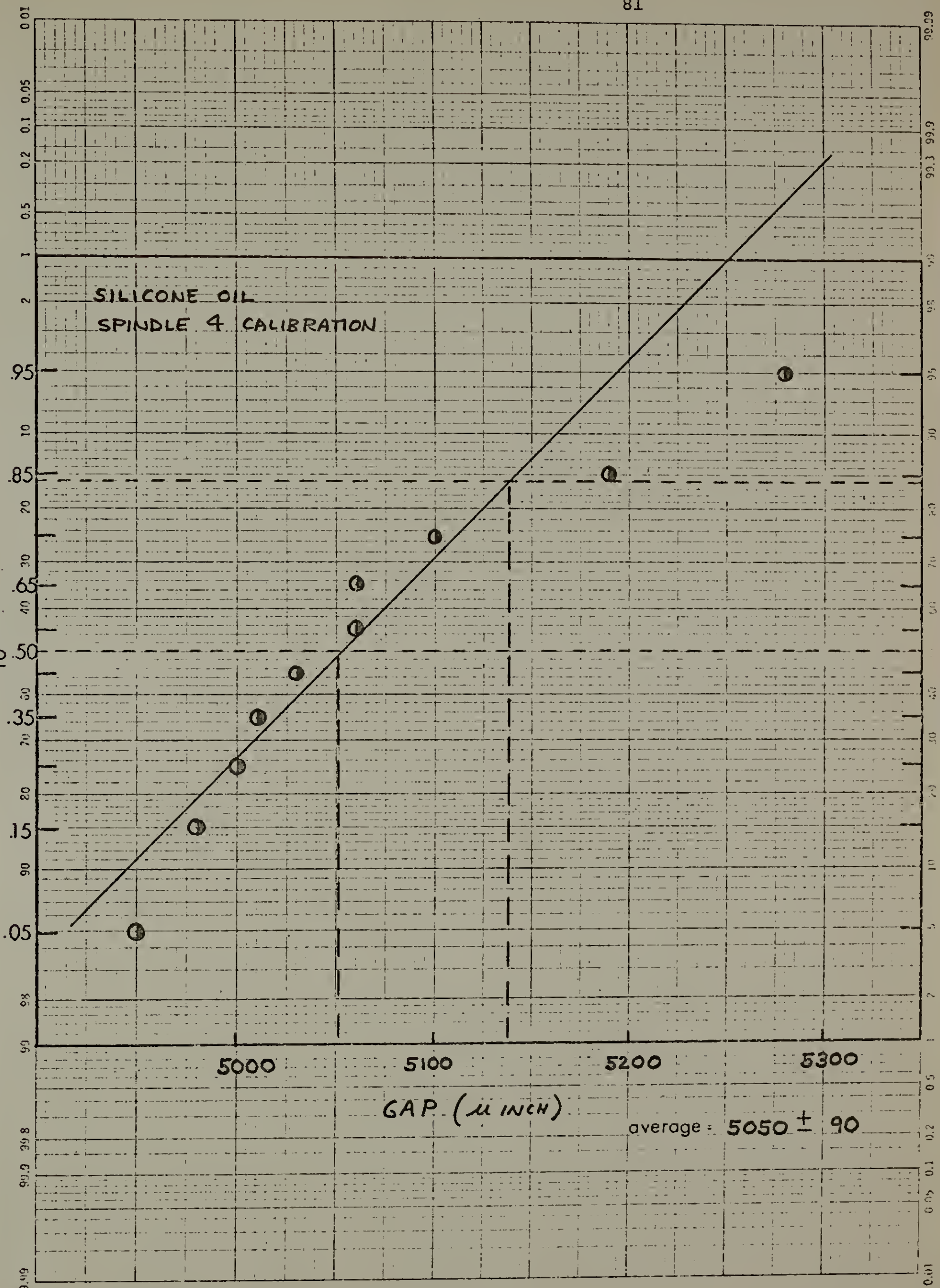
pot. set.	γ̇ volts	τ volts	°C at TC#3	RPM	γ̇ rate	η	h ‡
1:10 95	102.9	4.25	28.6			9.90	4650
1:1 10	9.20	2.90	31.2			8.10	4980
15	14.7	4.55	31.5			7.90	4950
20	20.4	6.06	32.0			7.60	4960
10*	9.15	2.50	32.9			7.15	5070
NEW SPL.							
1:10 75	80.6	4.07	25.7			12.3	4730
99	103.8	5.17	26.0			12.0	4670
1:1 10	8.90	4.00	26.8			11.4	4920
15	14.4	6.20	27.2			11.0	4950
10	9.10	3.60	27.5			10.7	5250

NOTES

* addnl. sample added

‡ $h = 19.4\,\eta\,\frac{\dot{\gamma}_r}{\tau_v}$ or $194\,\eta\,\frac{\gamma_r}{\tau_v}$ for 1:1 ratio

LM 4-72



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. II-97 B,C

TEST CONDITIONS

sample: SILICONE OIL 1000 cps

thermostat bath flow settings

heater: off ☒ boost ☐ max accuracy ☐

to pot: OPEN
to inner cyl: OPEN
" coil: AIR
box inlet: CLOSED

date 3/20/72
operator ym
temp. RT
spindle no. 4

drive shaft: 1/8"
" ratio: 1:10 ☒ 1:1 ☒

TRANSDUCER

τ arm length: 5"
load cell: 0.5 LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 50 gm	100/20/10
initial	± 0.00 mv	−8.000 v	+0.002 v	+5.04 v	10.18/1.89/.8
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8	POT	
26.4	–	26.3	–	26.4	26.4	26.6		25.0	

DATA

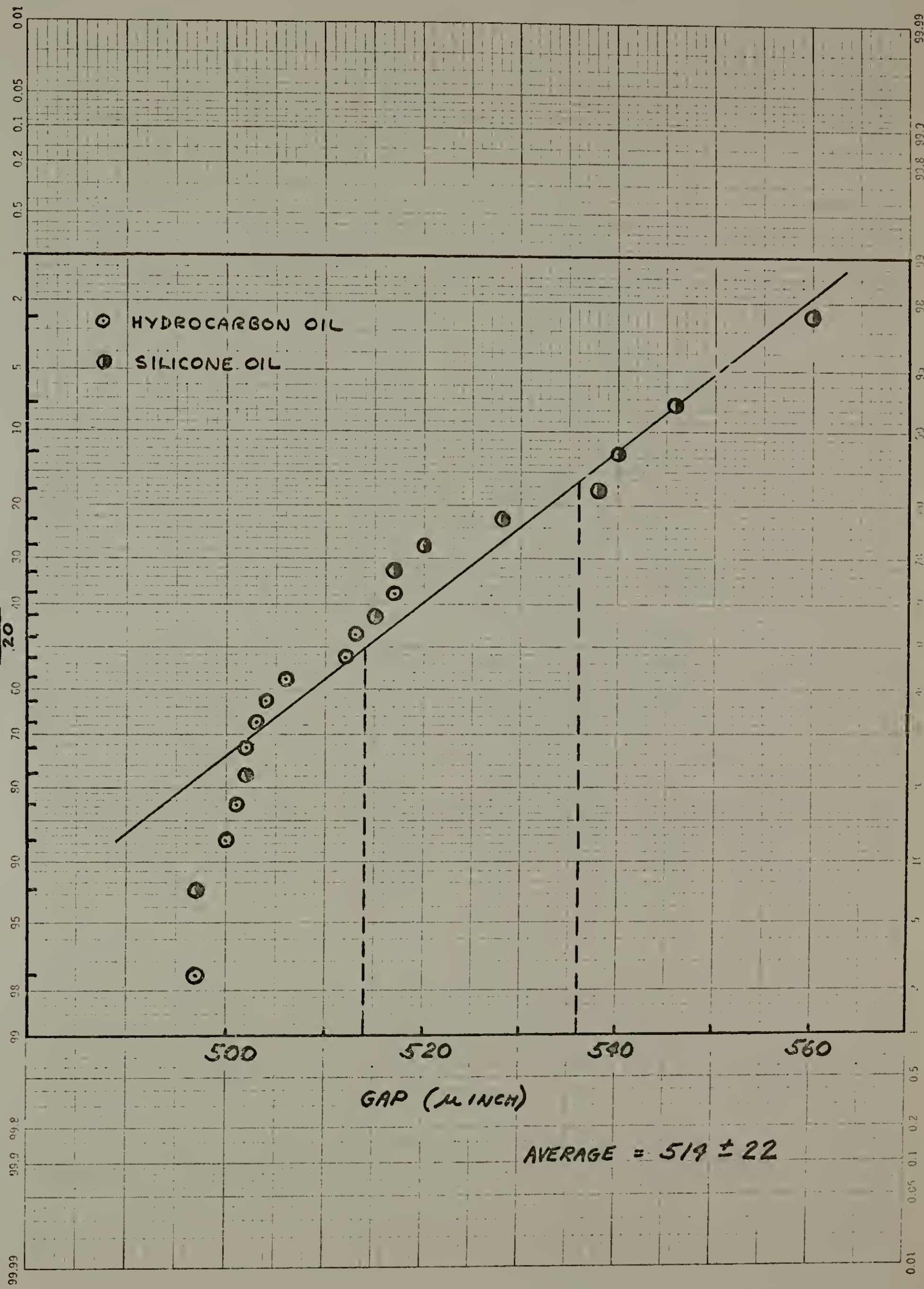
pot. set.	γ̇ volts	τ volts	°C at TC*	RPM	γ̇ rate	η	h‡
75	80.7	2.88	26.4			9.70	5280
99	104.1	3.76	26.6			9.65	5190
NEW SPL 1:1							
10	9.15	3.30	28.5			9.35	5030
15	14.7	5.30	28.7			9.30	5000
20	20.3	7.20	29.0			9.25	5060
25	26.0	9.05	29.5			9.15	5100
10	9.15	3.21	29.4			9.15	5060
1:10 75	81.3	2.88	29.6			9.15	5010
99	107.4	3.83	29.6			9.15	4980
99	108	3.83	30.2			9.05	4950

SHEAR

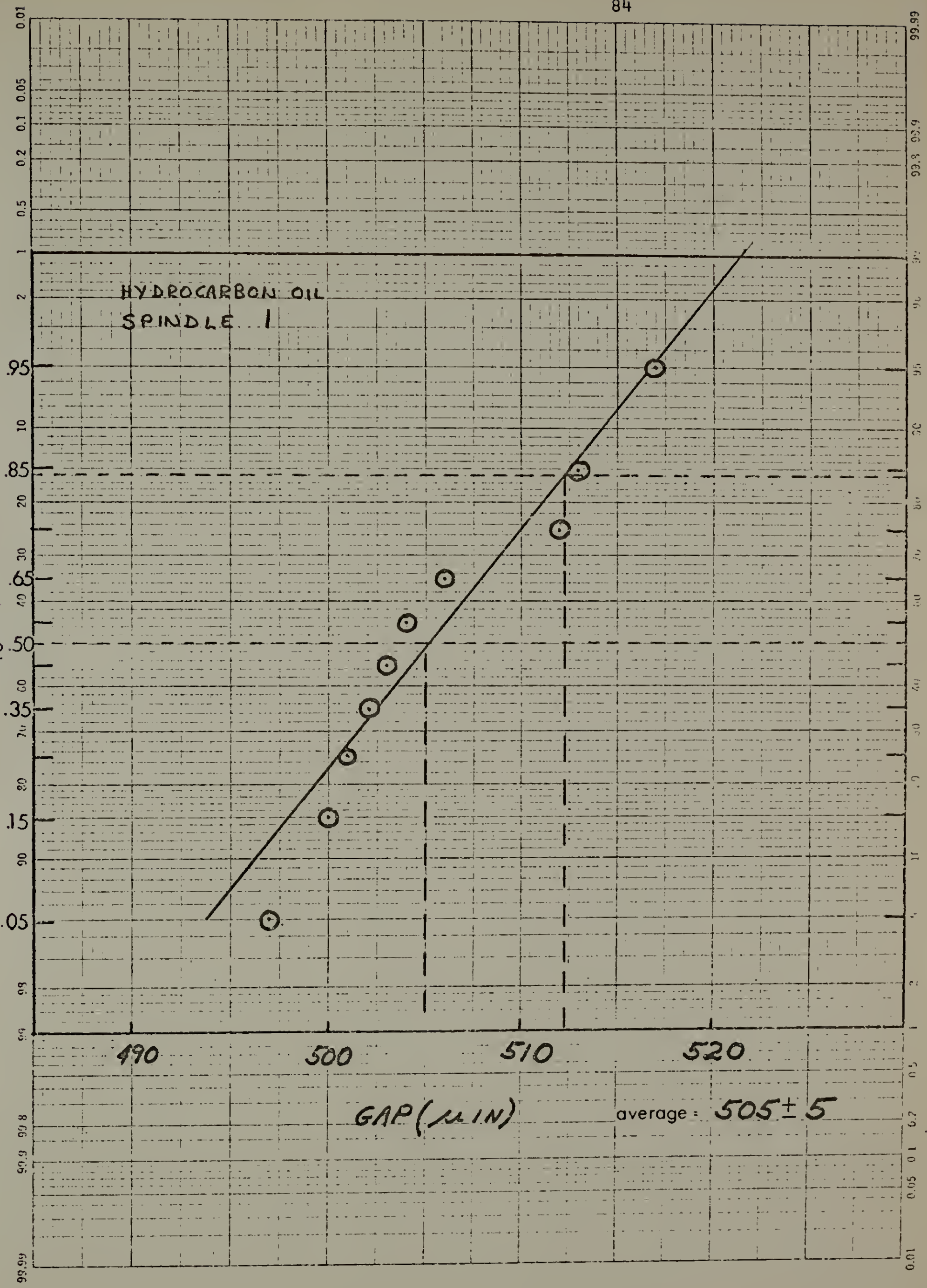
NOTES

‡ $h = 19.4\, \eta \frac{\dot{\gamma}_v}{\tau_v}$ or $194\, \eta \frac{\dot{\gamma}_v}{\tau_v}$ @1:1 ratio

APPENDIX V - SPINDLE 1 CALIBRATION



Probability Scale x 90 Divisions



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. II-40-B, C

TEST CONDITIONS

sample: 1300 cps HYDROCARB. OIL S-600-65

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:
to inner cyl: STATIC
" coil:
box inlet:

date 2/8/72
operator sm
temp. RT
spindle no. 1

drive shaft: 1/8"
" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 5"
load cell: 2 LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 200 gm	50/500
initial	± 0.01 MV	-8.000	+0.002 v	+4.02 v.	0.97/10.04
after run					
SPL 4	± 0.01	-8.000	+0.002 v	4.02	.97/9.98

TEMPERATURE

1	2	3	4	5	6	7 RT	8		
22.7	—	22.7	—	22.7	22.7	21.7			
21.8	~	21.8	~	21.8	21.8	20.2	SPL 4		

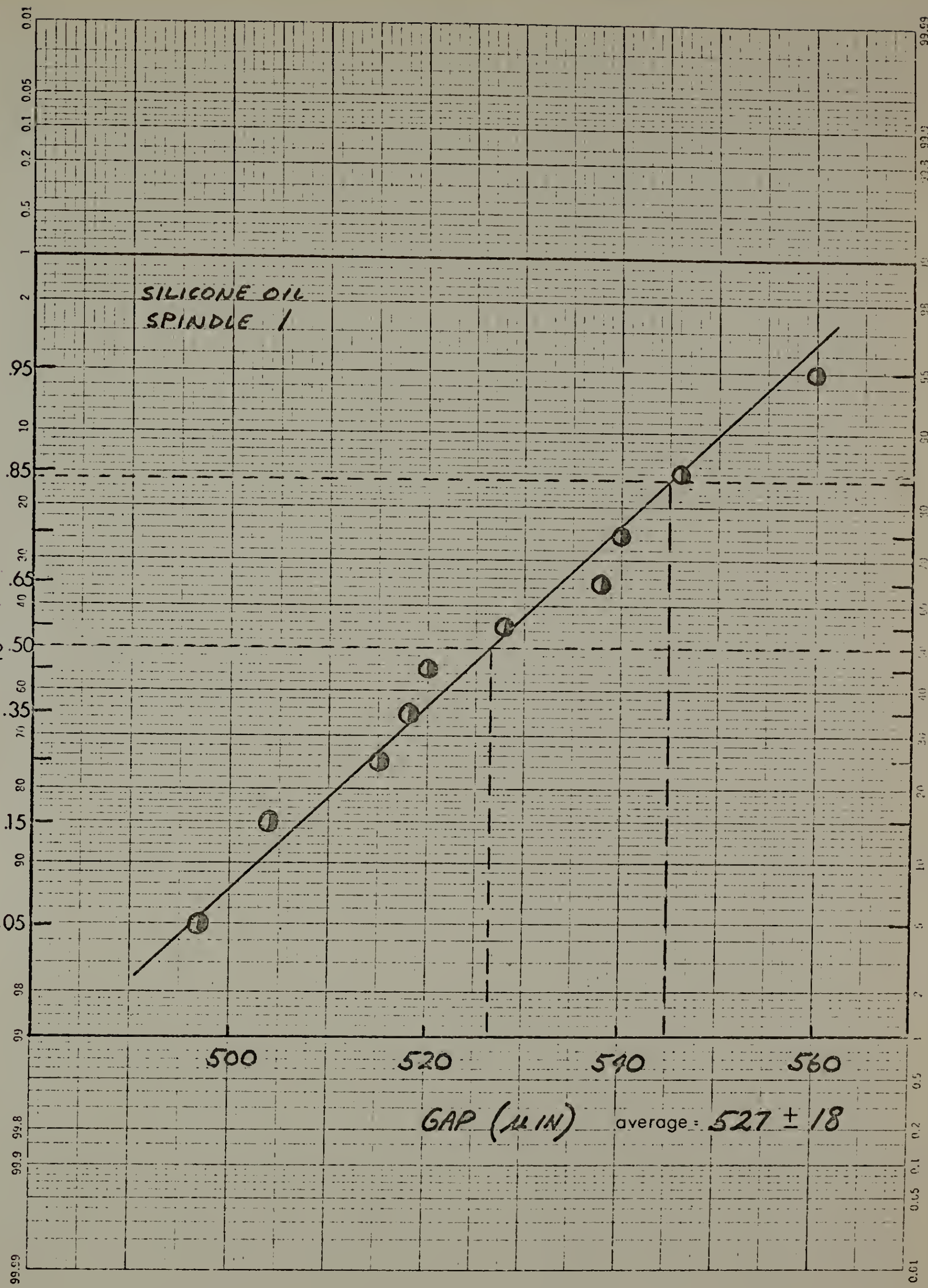
SHEAR DATA

	pot. set.	γ̇ volts	τ volts	°C at TC#3	RPM	γ̇ rate	η	h*
	10	9.05	1.05	22.6			15.5	517
SPL 2								
	10	9.05	1.06	22.6			15.5	512
	10	9.08	1.09	22.5			15.6	503
SPL 3	10	9.10	1.10	22.5			15.6	500
	15	14.65	1.78	22.5			15.6	497
SPL 4	10	9.10	1.17	21.8			16.7	502
	15	14.65	1.88	21.8			16.7	504
	10	9.10	1.15	21.8			16.7	506
SPL 5	10	9.15	1.18	21.8			16.7	501
	10	9.15	1.15	21.8			16.7	513

NOTES

$$^{*}h = 3.87\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$

LM 4-72



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. II-85-A,B

date 1/27-1/28-'72

operator gm

temp. RT

spindle no. 1

drive shaft: 1/8"

" ratio: 1:10 ☒ 1:1

TEST CONDITIONS

sample

1000 cps SILICONE OIL 81871

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:

to inner cyl:

" coil: STATIC

box inlet:

TRANSDUCER

τ arm length: 10"

load cell: .5 LB

chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 100 gm	50
initial	±0.00 mv	-8.000	+.001 v	+5.01	+2.49
after run					
SPL 3					

TEMPERATURE

1	2	3	4	5	6	7 RT	8		
22.7	—	22.7	—	22.7	22.7	22.7			

DATA

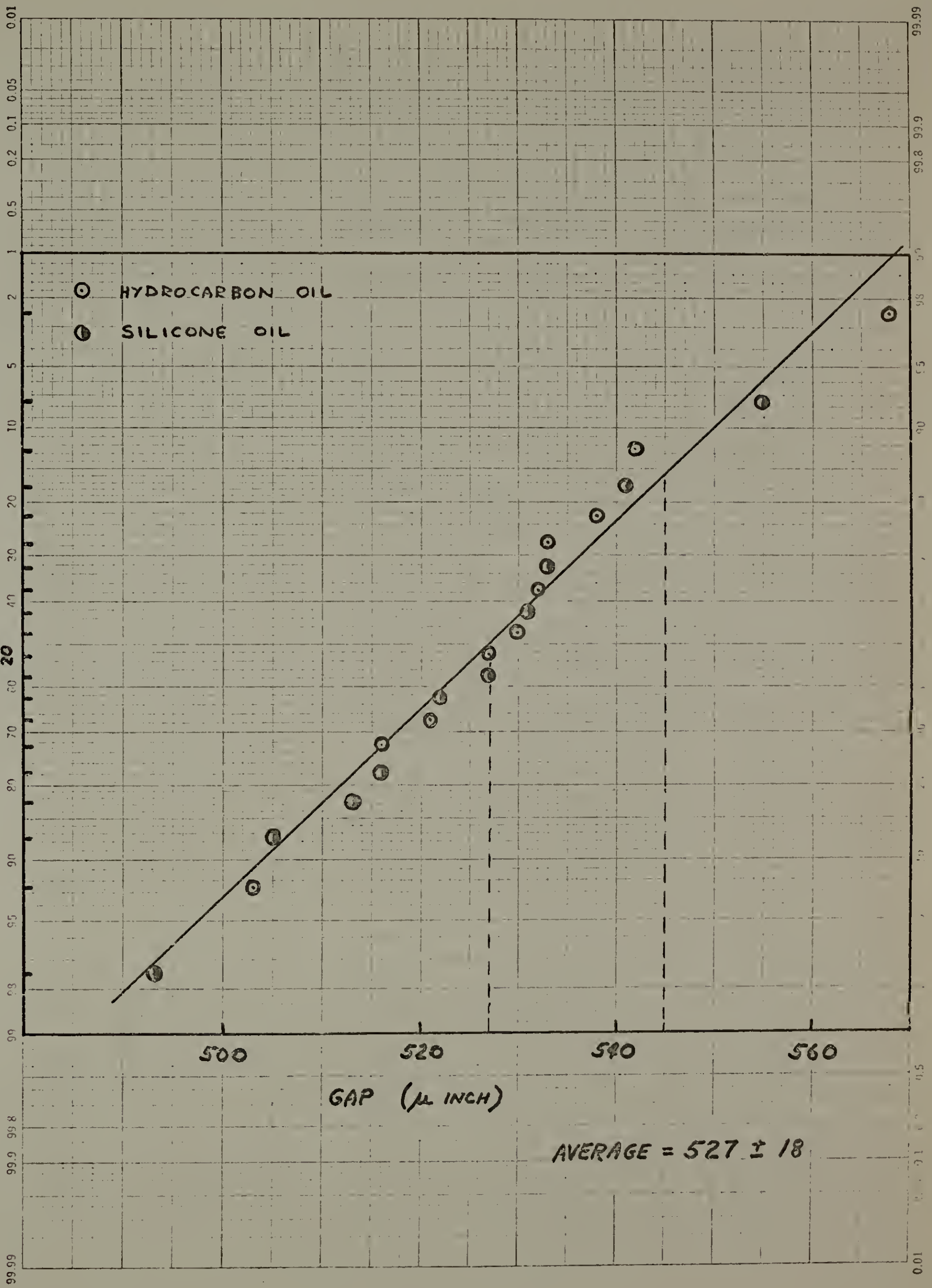
SHEAR

pot. set.	γ̇ volts	τ volts	°C at TC# 3	RPM	γ̇ rate	η	h
10	9.15	1.73	22.8@#1			10.3	528
10	9.15	1.70	23.0			10.3	538
SPL 2 10	9.18	1.70	23.1			10.3	540
SPL 3 10	8.95	1.82	22.4			10.4	497
25	25.8	4.65	22.7			10.4	560
SPL 4 10	9.15	1.77	22.9			10.3	517
10	9.08	1.78	22.7			10.4	515
10	9.10	1.83	22.7			10.4	502
25	26.1	4.78	22.8			10.3	546
10	9.10	1.75	22.8			10.3	520

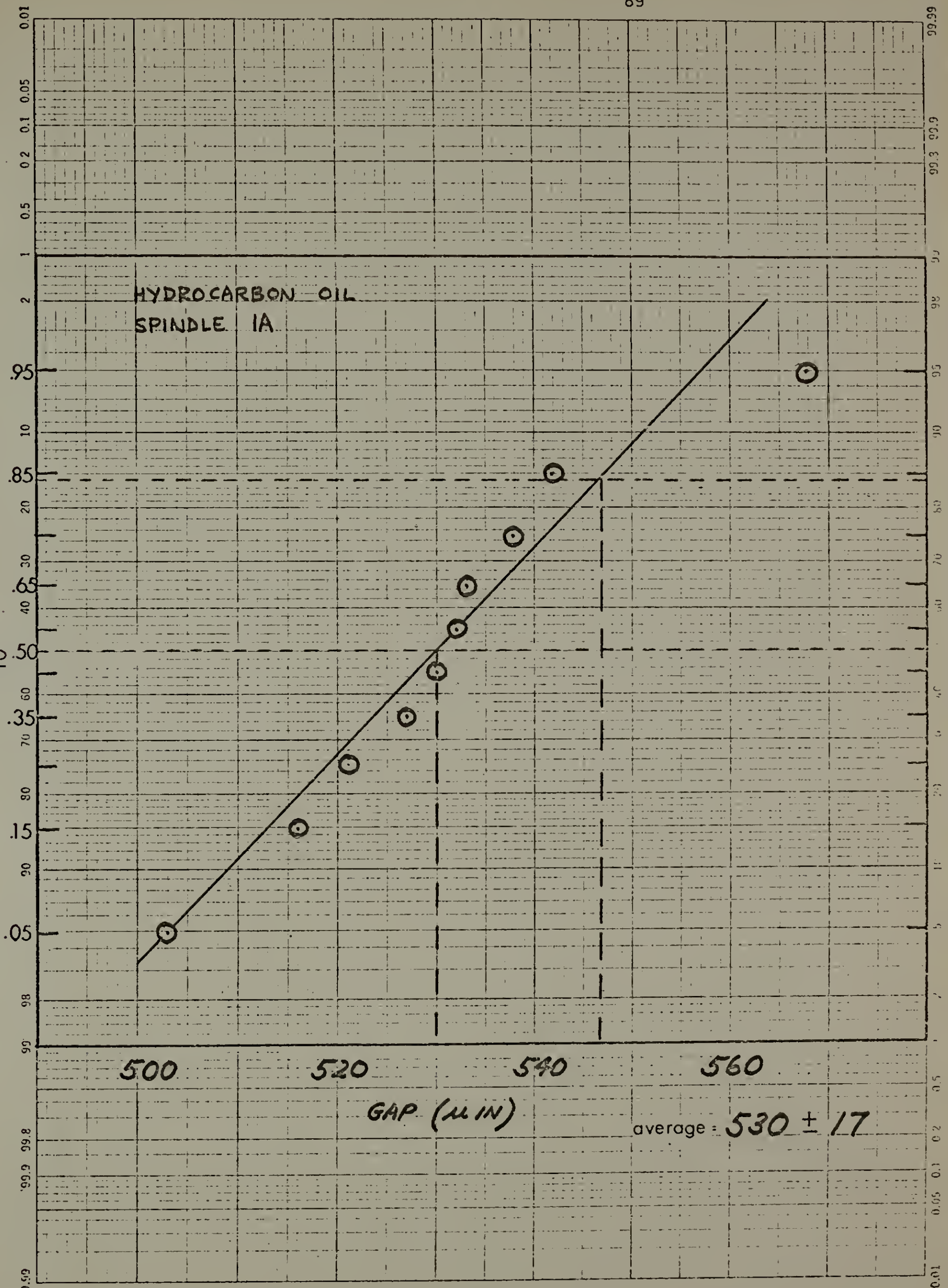
NOTES

$h = 9.7 \eta \frac{\dot{\gamma}_v}{\tau_v}$

APPENDIX VI - SPINDLE 1A CALIBRATION



Probability Scale x 90 Divisions



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet 90

RUN NO. II-89, 90

date 2/6 - 2/7/72

aperator gm

temp. RT

spindle na. 1A

drive shaft:

" ratia: 1:10 ☒ 1:1 ☐

TEST CONDITIONS

sample

HYDROCARBON OIL
1300 cps
S-600-65-2d

thermostat bath flow settings

heater: off ☐ baost ☐ max accuracy ☐

to pot:
to inner cyl: STATIC
" cail:
box inlet:

TRANSDUCER

τ arm length: 5"
load cell : 2LB
chart speed : 1"/MIN

	VM zera	VM int. ck.	τ zero	τ 200 gm	50/500
initial	±0.01 mv	-8.000	+.002	+4.02	+.99/10.02
after run					
SPL 3	±0.01 mv	-8.000	+.002	+4.01	+.98/+10.0

TEMPERATURE

1	2	3	4	5	6	7 RT	8 ____		
23.3	—	23.3	—	23.2	23.2	23.3			

DATA

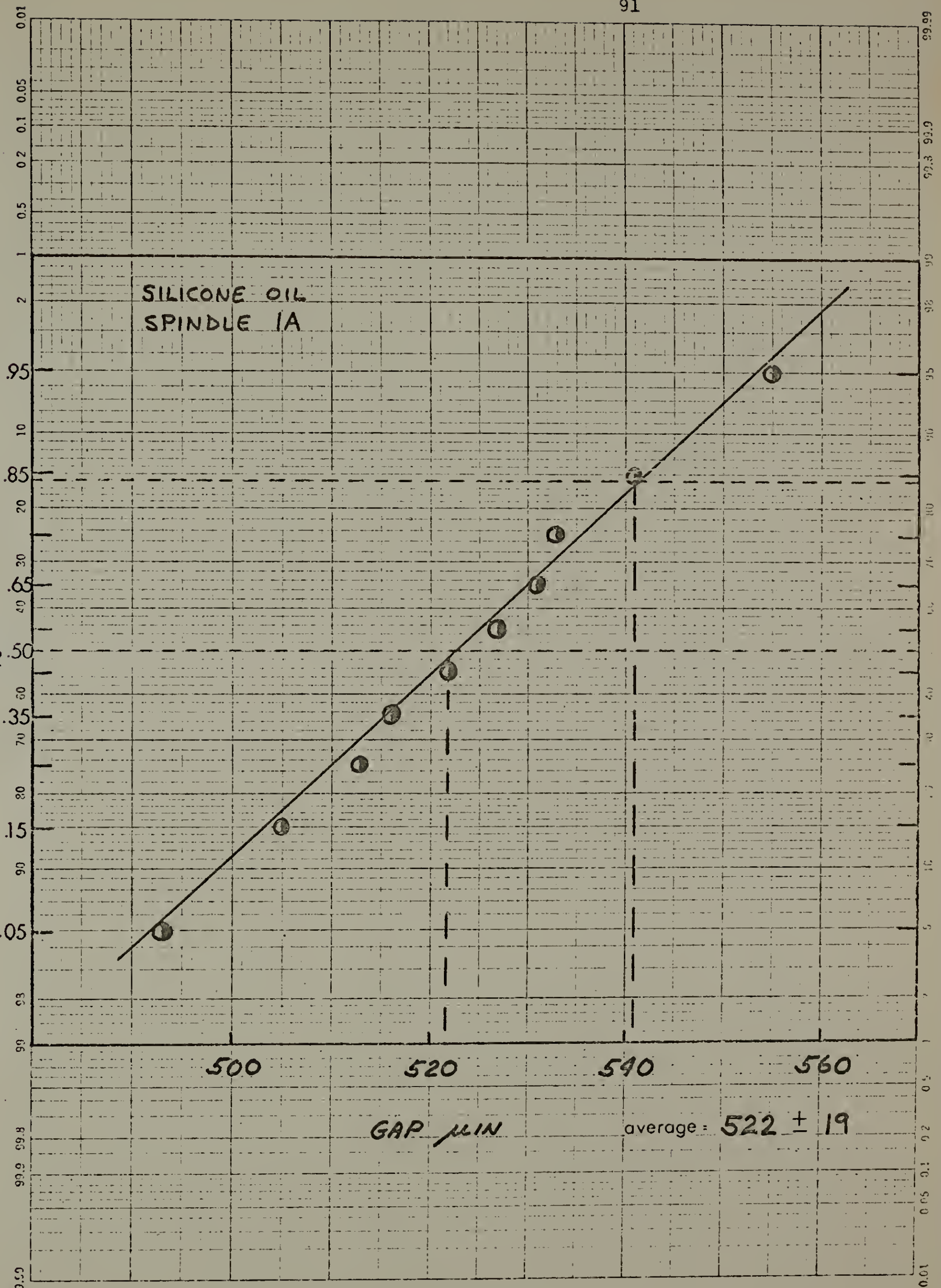
pot. set.	ϑ volts	τ volts	°C at TC#3	RPM	ϑ rate	η	h *
10	9.15	0.98	23.4			14.6	527
10	9.10	0.88	23.8			14.4	519
SPL 2 10	9.10	0.98	24.0			14.0	5.03
15	14.65	1.54	24.0			14.0	516
10	9.10	0.94	24.1			13.9	521
SPL 3 10	9.15	0.85	25.0			13.0	542
SPL 4 10	9.20	0.87	25.1			12.9	538
20	20.3	1.89	25.2			12.8	532
10	9.10	0.85	25.2			12.8	530
SPL 5 10	9.15	0.85	25.2			12.8	525

SHEAR

NOTES

$$*h = 3.87\,\eta\,\frac{\gamma_v}{\tau_v}$$

LM 4-72



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. II-85 C, D

TEST CONDITIONS

sample: SILICONE OIL 1000 cps 81871

thermostat bath flow settings *
heater: off ☒ boost ☐ max accuracy ☐
to pot: OPEN
to inner cyl: CLOSED
" coil: AIR
box inlet: CLOSED

date 1/28 - 1/29/72
operator JM
temp. RT
spindle no. 1A
drive shaft: 1/8
" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 10"
load cell: 0.5LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 100 grm	SO
initial	±0.00 mv	-8.000 v	+0.002 v	+5.00 v	+2.49 v.
after run	±0.01 mv	-8.000 v	+0.002 v	+4.92 v	+2.91 v.
SPL4	±0.01 mv	-8.000 v	+0.002 v	+5.00 v	+2.47 v.

TEMPERATURE

1	2	3	4	5	6	7 RT	8		
22.6	-	22.6	-	22.6	22.6	21.0			
21.8	-	21.8	-	21.8	21.7	22.2			SPL 4

DATA

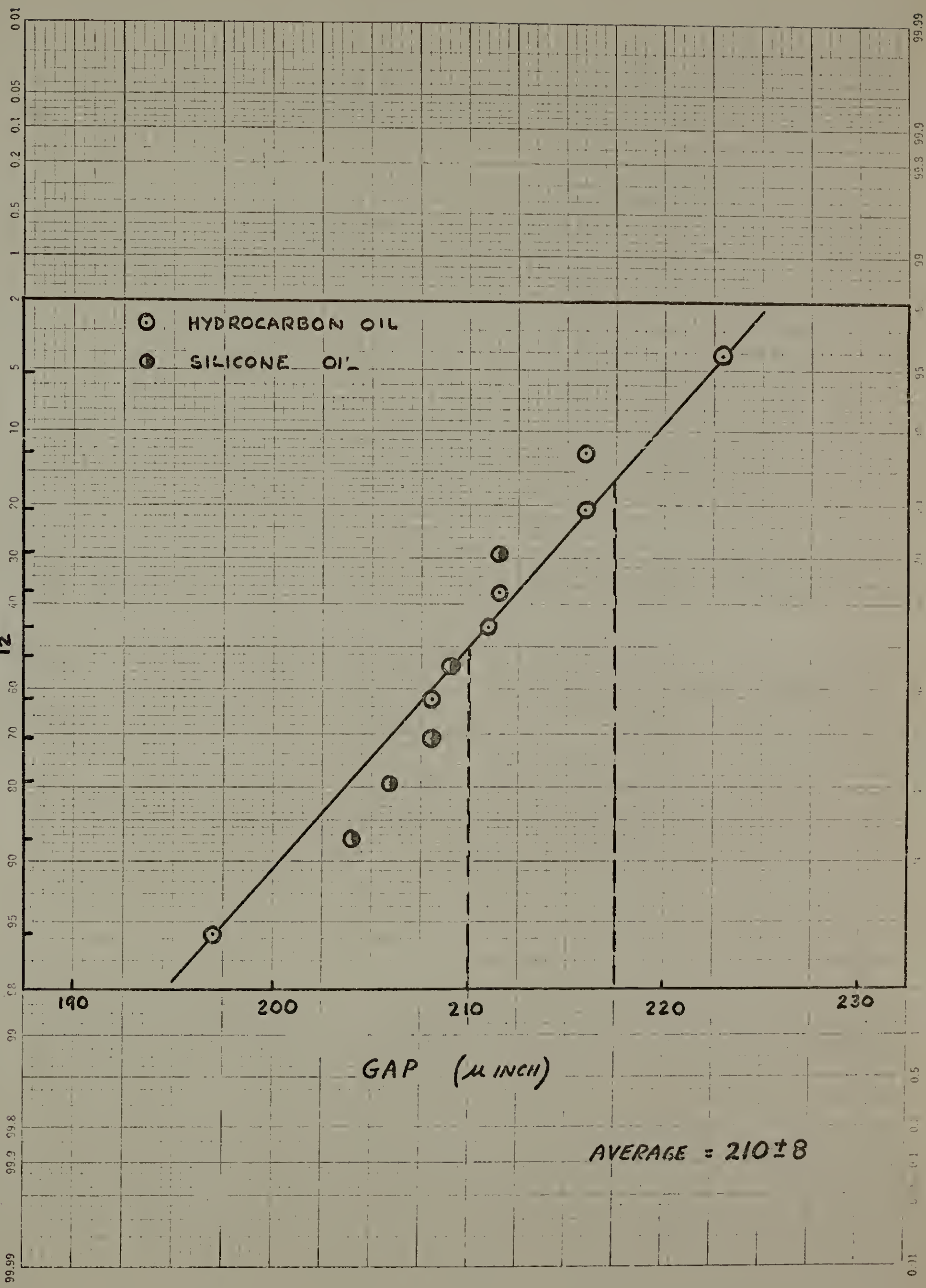
SHEAR

pot. set.	γ̇ volts	τ volts	°C at TC #3	RPM	γ̇ rate	η	h
			STATIC				
10	8.95	1.83	22.6			10.4	493
18	17.9	3.58	22.6			10.4	505
SPL 2 10	9.05	1.78	22.7			10.4	513
10	9.05	1.75	22.9			10.3	516
SPL 3 10	9.05	1.75	22.7			10.4	522
18	17.9	3.53	22.7			10.4	527
10	9.05	1.70	23.0			10.3	531
SPL 4 10	9.05	1.73	* BATH FLOW 22.0			10.5	533
SPL 5 10	9.15	1.71	22.6			10.4	541
10	9.05	1.63	23.0			10.3	555

NOTES

* APPLIES TO D ONLY; C STATIC

APPENDIX VII - SPINDLE 2 CALIBRATION



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

94

RUN NO. II-80 A

date 1/14/72

operator JM

temp. RT

spindle no. 2

drive shaft: 1/8"

" ratio: 1:10 ☒ 1:1

TEST CONDITIONS

sample: 30 cps HYDROCARBON OIL 71106

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:

to inner cyl: STATIC

" coil:

box inlet:

TRANSDUCER

τ arm length: 10"

load cell: 1/2 LB

chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 100 gm	
initial	± 0.00 mv	-8.000 v	+.002 v	+5.00 v	
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8	POT	
28.4	28.4	28.5	—	28.5	28.5	26.9		28.5	

DATA

pot. set.	γ̇ volts	τ volts	°C at TC*2	RPM	γ̇ rate	η	h *
99	105.7	1.21	28.5			0.255	216
99	106.7	1.24	28.6			0.254	211
70	75.5	0.90	28.5			0.255	197
50	54.0	0.60	28.4			0.256	208
35	37.2	0.43	28.4			0.256	223
99	107.5	1.35	28.5			0.255	216
SPL 2 50 *	54.1	0.70	25.7			0.283	212
						AVG	212 ± 9

SHEAR

NOTES

$$*h = 9.7\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$

‡ RUN II-79

UMASS HIGH SHEAR VISCOMETER - lab work sheet 95

RUN NO. II-89B

TEST CONDITIONS

sample: SILICONE OIL 100 CPS 81871

thermostat bath flow settings
heater: off ☐ baost ☐ max accuracy ☐
to pot:
to inner cyl:
" coil: STATIC
box inlet:

date 1-26-72
aperator YM
temp. RT
spindle no. 2
drive shaft:
" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 10"
load cell: ½ LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 100 gm	
initial		-8.000	+0.002v	+5.00	
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8		
20.7	—	20.7	-	20.7	20.7	20.0			

DATA

pot. set.	γ̇ volts	τ volts	°C at TC #3	RPM	γ̇ rate	η	h*
						1	
25	26.1	1.28	20.7			1.06	209
50	54.0	2.71	20.8			1.06	208
75	81.0	4.05	21.0			1.06	206
99	106.2	5.25	21.5			1.04	204
25	26.0	1.25	21.2			1.05	212
						AVG	208±3

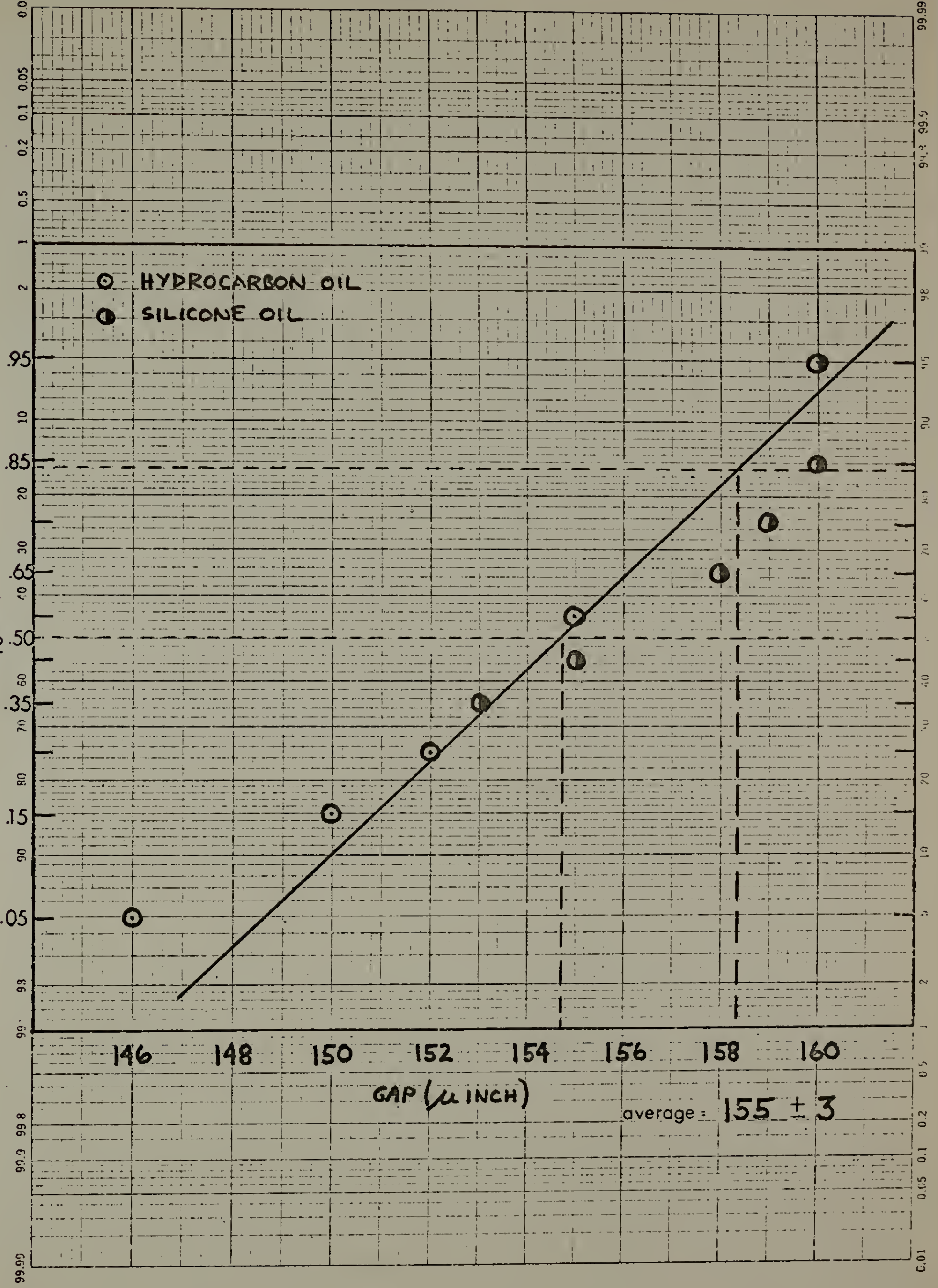
SHEAR

NOTES

$$*h = 9.7\,\eta\,\frac{\gamma_v}{\tau_v}$$

LM 4-72

APPENDIX VIII - SPINDLE 2A CALIBRATION



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

97

RUN NO. II-80-D

date 1-17-72

operator GM

temp. RT

spindle no. 2A

drive shaft: 1/8"

" ratio: 1:10 ☒ 1:1 ☐

TEST CONDITIONS

sample

30 cps HYDROCARBON OIL 71106

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:

to inner cyl: STATIC

" coil:

box inlet:

TRANSDUCER

τ arm length: 10"

load cell: 0.5 lb

chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 100 gm	
initial	±0.01mv	-8.000	+0.002v	+5.00v	
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8		
23.5	23.5	23.5	-	23.5	23.5	22.4			

DATA

pot. set.	$\dot{\gamma}$ volts	τ volts	°C at TC*2	RPM	$\dot{\gamma}$ rate	η	h^*
50	54.0	1.10	23.5			.316	150
25	25.9	0.55	23.2			.320	146
50	53.9	1.10	23.2			.320	152
75	80.7	1.60	23.4			.317	155
						AVG	152±3

SHEAR

NOTES

$$*h = 9.7\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$

UMASS HIGH SHEAR VISCOMETER - lab work sheet 98

RUN NO. II-89C

date 1/26/72

operator gm

temp. RT

spindle no. 2A

drive shaft:

" ratio: 1:10 ☒ 1:1 ☐

TEST CONDITIONS

sample

SILICONE OIL 100 CPS
81871

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:

to inner cyl: STATIC

" coil:

box inlet:

TRANSDUCER

τ arm length: 10"

load cell: 0.5 LB initial

chart speed: 1"/MIN after run

VM zero	VM int. ck.	τ zero	τ 100 gm
—	-8.000	+0.003v	+5.00

TEMPERATURE

1	2	3	4	5	6	7 RT	8
20.5	—	20.4	—	20.4	20.4	20.2	

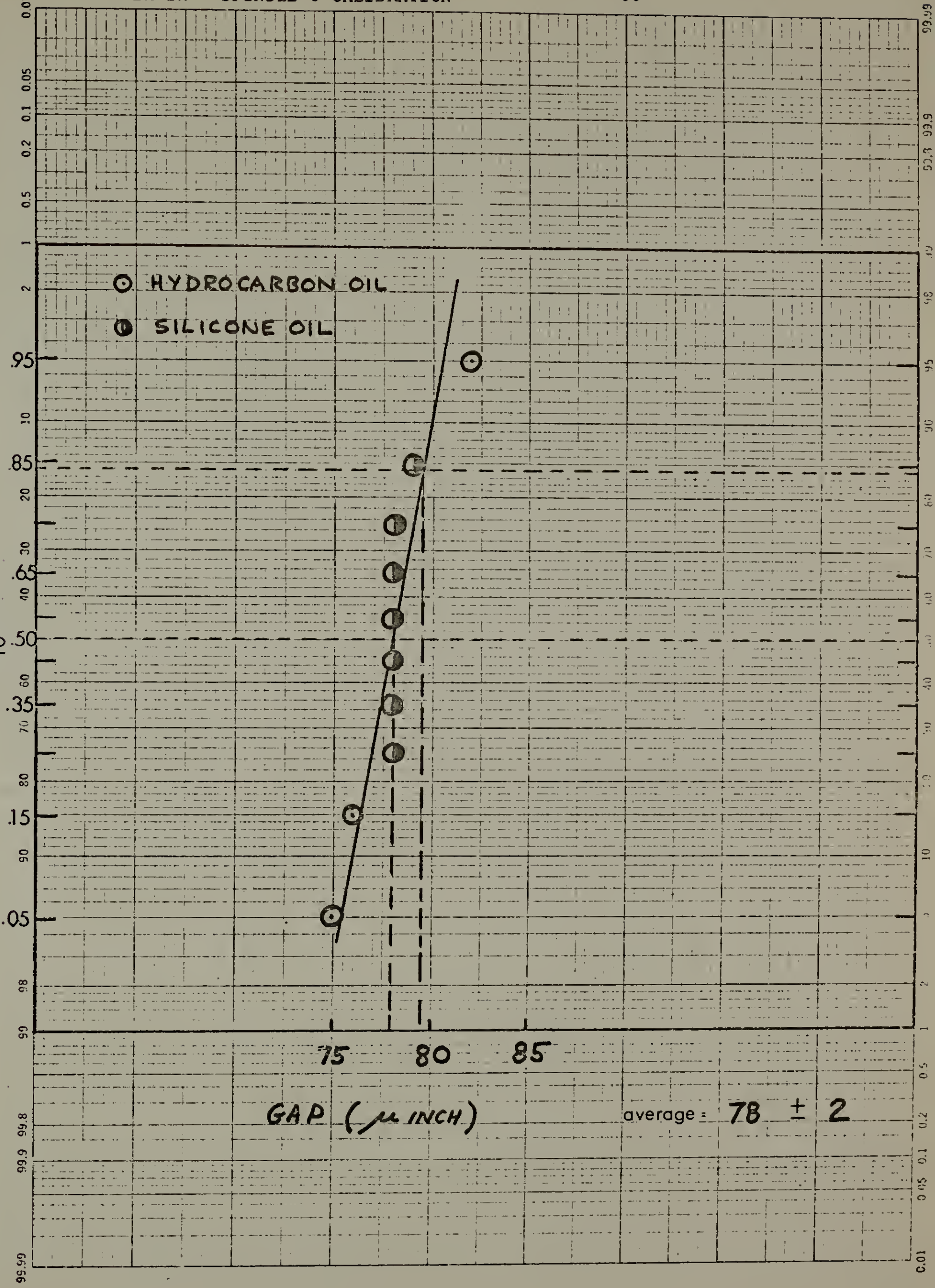
DATA

pot. set.	$\dot{\gamma}$ volts	τ volts ≠	°C at TC#3	RPM	$\dot{\gamma}$ rate	η	h^*
25	26.2	1.68	20.7			1.06	160
50	54.0	3.50	21.0			1.06	159
25	26.3	1.66	21.5			1.05	160
SPL 2							
50	54.0	3.60	21.5			1.05	153
25	26.1	1.68	21.5			1.05	158
75	81.0	5.31	21.6			1.05	155
						AVG	157 ± 2

SHEAR

NOTES

$* h = 9.7 \eta \frac{\dot{\gamma}_v}{\tau_v}$ ≠ all are plateau values



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

100

RUN NO. II-80 E

TEST CONDITIONS

sample: 30 CPS HYDROCARBON OIL 71106

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:

to inner cyl: STATIC

" coil:

box inlet:

date 1-17-72

operator YM

temp. RT

spindle no. 3

drive shaft:

" ratio: 1:10 ☐ 1:1 ☐

TRANSDUCER

τ arm length: 10"

load cell: 2LB

chart speed: 2"/MIN

	VM zero	VM int. ck.	τ zero	τ 600 gm	*
initial	±0.0 MV	-8.000	0.00V	+4.99 V	
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8
23.5	23.5	23.3	—	23.4	23.5	24.0	

DATA

pot. set.	$\dot{\gamma}$ volts	τ volts	°C at TC*2	RPM	$\dot{\gamma}$ rate	η	h*
10	8.85	0.55	23.5			.315	82
25	25.5	1.65	24.4			.303	76
10	9.0	0.55	26.0			.283	75
						AVG	78 ± 4

SHEAR

NOTES

* NON-STANDARD SET-UP

$$h = \frac{.154\,(1.45\times10^{-5})(23.4)}{(.3245)(10)(10^{-6})}\,\eta\,\frac{\gamma_v}{\tau_v} = 16.2\,\eta\,\frac{\gamma_v}{\tau_v}$$

$$W = \frac{600 \times 12.25}{10 \times 4.99} = 147.5\ \text{gm/V} = .3245\ \text{LB/V}$$

LM 4-72

UMASS HIGH SHEAR VISCOMETER - lab work sheet

101

RUN NO. II-84 D

TEST CONDITIONS

sample: SILICONE OIL 100 CPS 81871

thermostat bath flow settings

heater: off ☐ boost ☐ max accuracy ☐

to pot:
to inner cyl: STATIC
" coil:
box inlet:

date 1-26-72
operator ym
temp. RT
spindle no. 3

drive shaft: 1/8"
" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 10"
load cell: 0.5 LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ ____ gm	
initial		-8.000	+0.002v		
after run					

TEMPERATURE

1	2	3	4	5	6	7 RT	8 ____		
21.4	–	21.4	–	21.4	21.4	19.8			

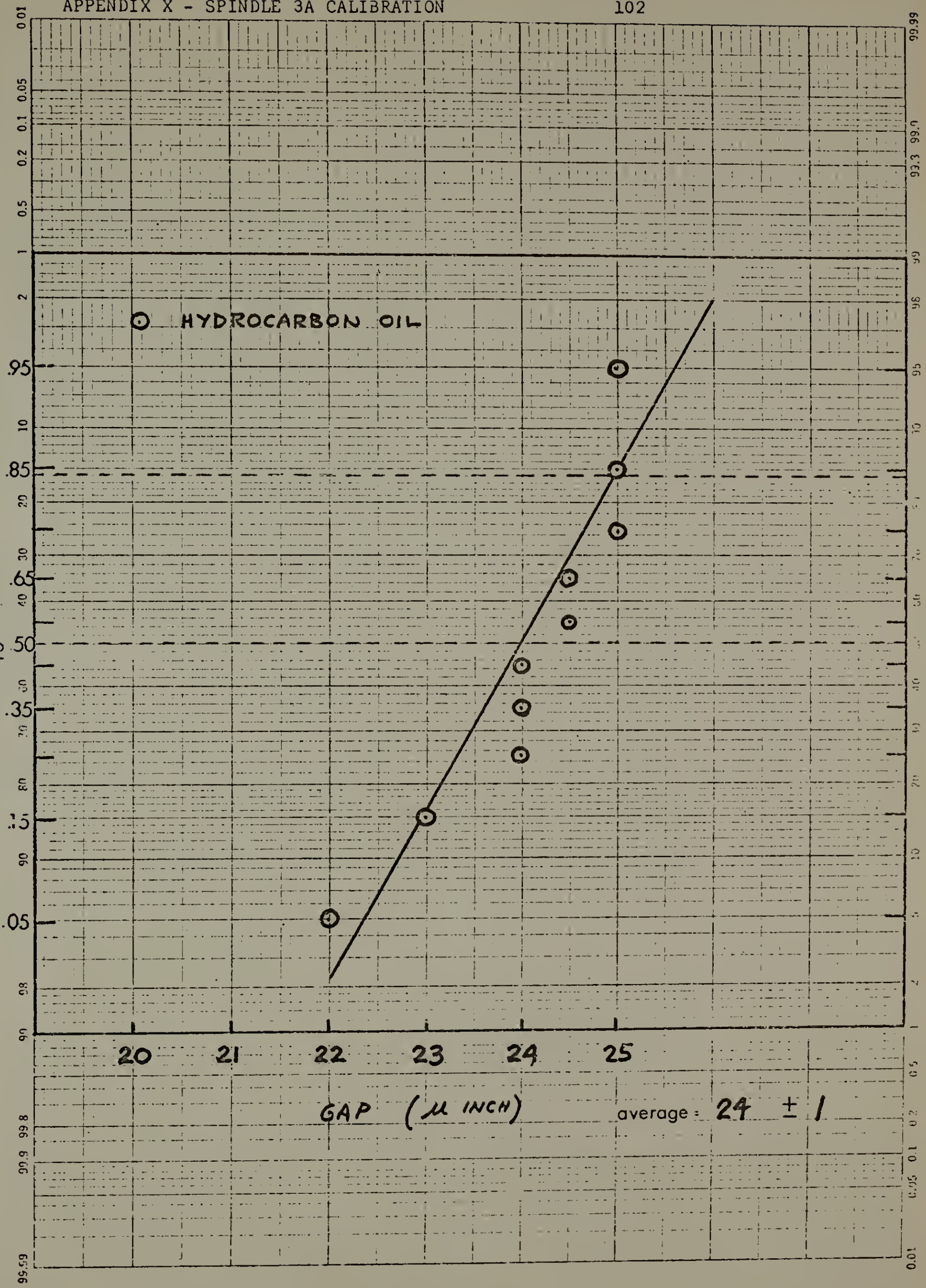
DATA

SHEAR

poi. set.	$\dot{\gamma}$ volts	τ volts	°C at TC*3	RPM	$\dot{\gamma}$ rate	η	h*
25	25.9	3.35	21.5			1.05	79
50	54.0	6.95	21.8			1.04	78
10	9.10	1.17	21.7			1.04	78
25	26.2	3.38	21.7			1.04	78
SPL 2							
50	54.0	6.95	22.0			1.03	78
25	26.2	3.36	22.0			1.03	78
10	9.15	1.18	21.8			1.04	78
						AVG	78 ± 1

NOTES

$$* h = 9.7\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$ at τ equilibrium plateau



Probability Scale x 90 Divisions

UMASS HIGH SHEAR VISCOMETER - lab work sheet

RUN NO. 201, 202

TEST CONDITIONS

sample

30 CPS HYDROCARBON
OIL 71106

SPINDLE AT 1/2 DEPTH*

thermostat bath flow settings

heater: off ☒ boost ☐ max accuracy ☐

to pot: OPEN 5 TURNS
to inner cyl: FULL OPEN
" coil: AIR
box inlet: CLOSED

date 3/31, 4/3/72
operator gm
temp. RT
spindle no. 3A

drive shaft: 3/16"
" ratio: 1:10 ☒ 1:1 ☐

TRANSDUCER

τ arm length: 5"
load cell: 2 LB
chart speed: 1"/MIN

	VM zero	VM int. ck.	τ zero	τ 200 gm	100/50/20
initial	—	-8.000 v	+.004 v	—	—
after run				+4.00	2.00/1.00/.40
4/3 ‡	±0.00 mv	-8.000	+.002 v	500gm/+5.00v	.99/.48/.1

TEMPERATURE

1	2	3	4	5	6	7 ____	8 ____	POT	
26.9	—	26.7	—	26.8	26.8			25.5	

DATA — SHEAR

pot. set.	γ̇ volts	τ volts	°C at TC#3	RPM	γ̇ rate	η	h *
15	15.2	0.28	29.8			.240	25
30	32.1	0.60	30.0			.240	25
45	49.3	0.95	30.1			.240	24
60	65.3	1.35	30.3			.238	22
30	32.0	0.60	30.3			.238	25
45	49.3	0.93	30.4			.238	24.5
15	15.1	0.28	31.0			.232	24
NEW SPL 4/3 ‡							h ‡
15	15.1	0.17	26.2			0.28	24
30	31.8	0.38	26.2			0.28	23
50	54.2	0.60	26.4			0.28	24.5

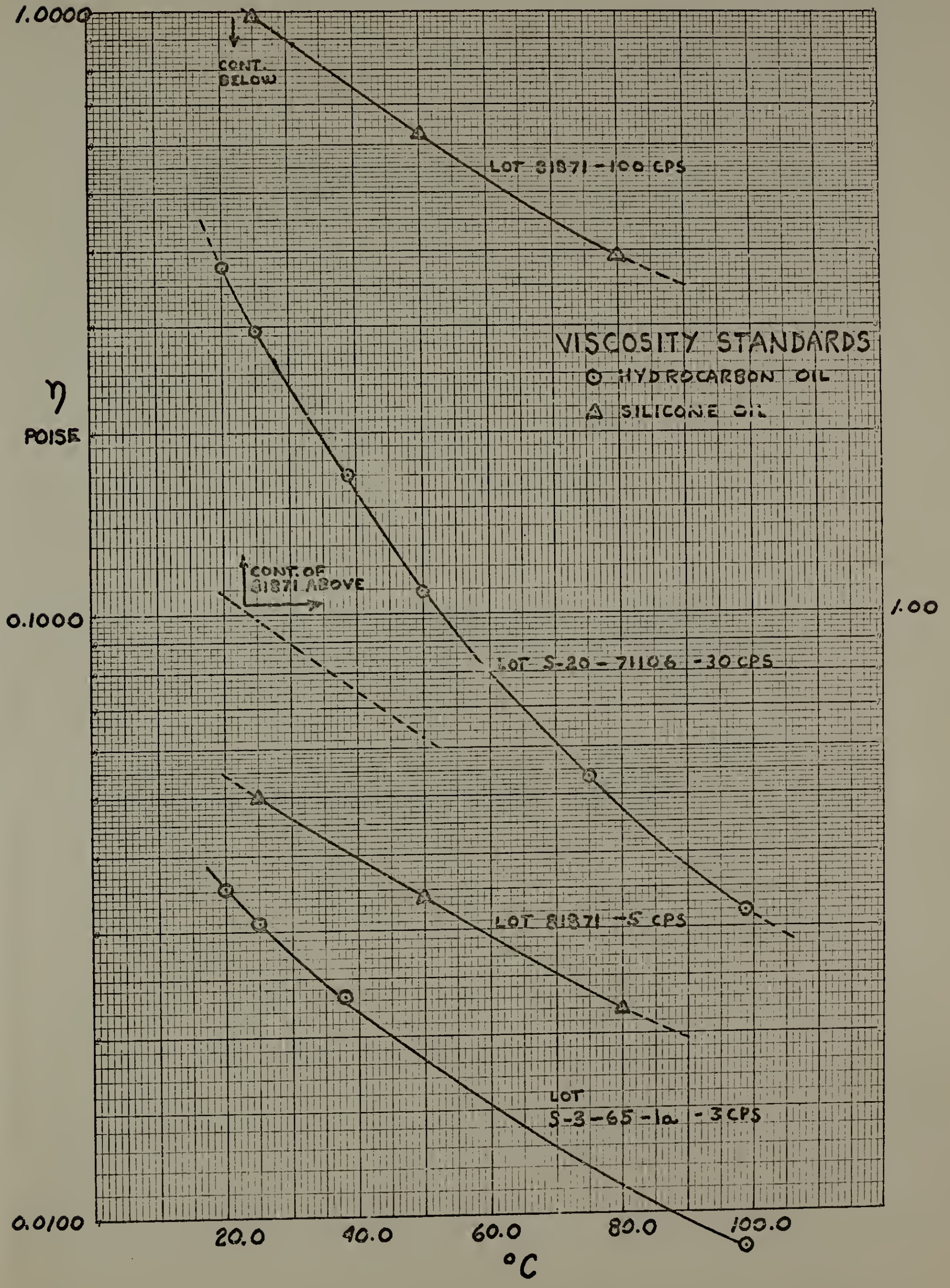
NOTES

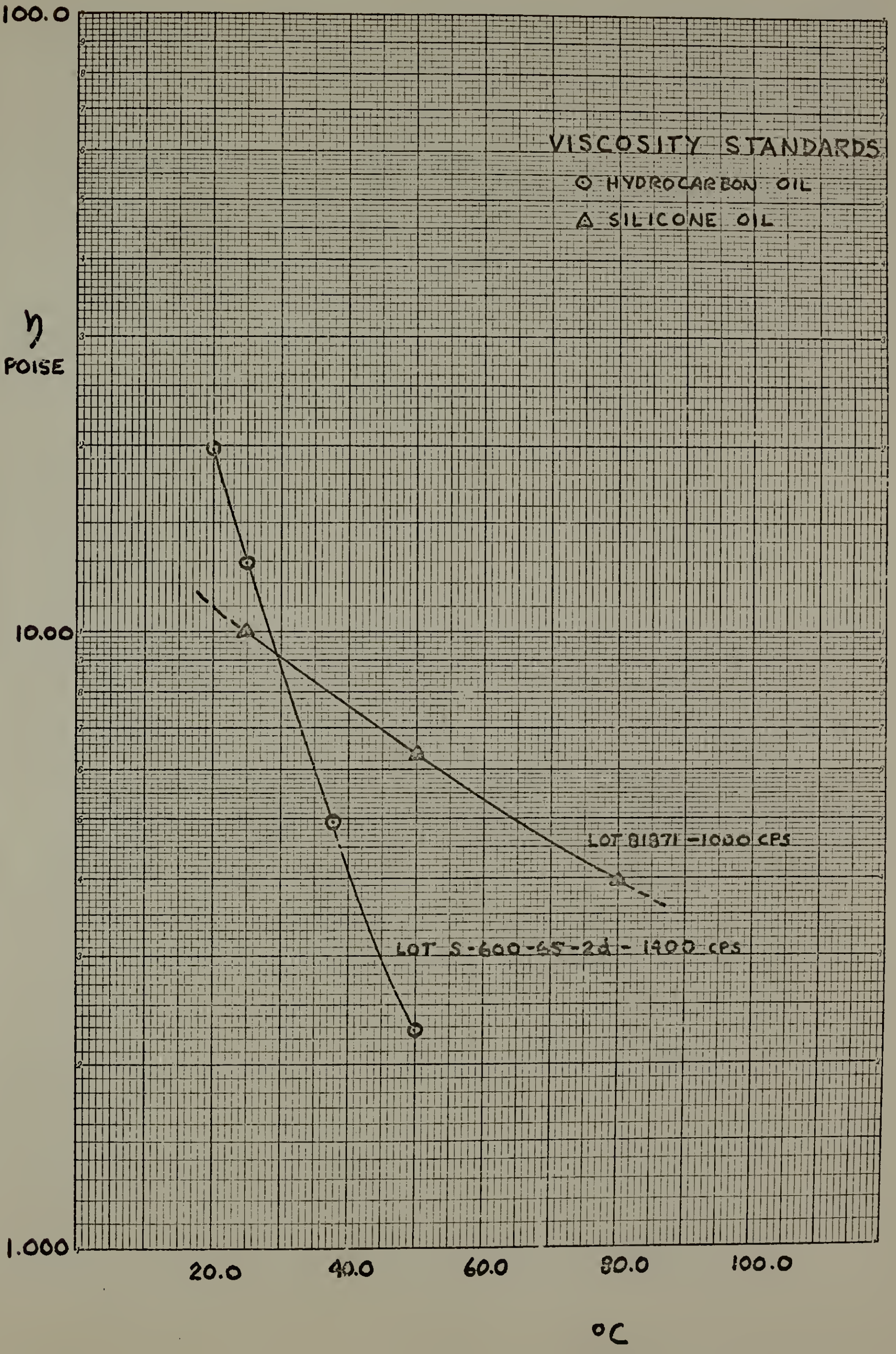
* SPINDLE AT 1/2 DEPTH

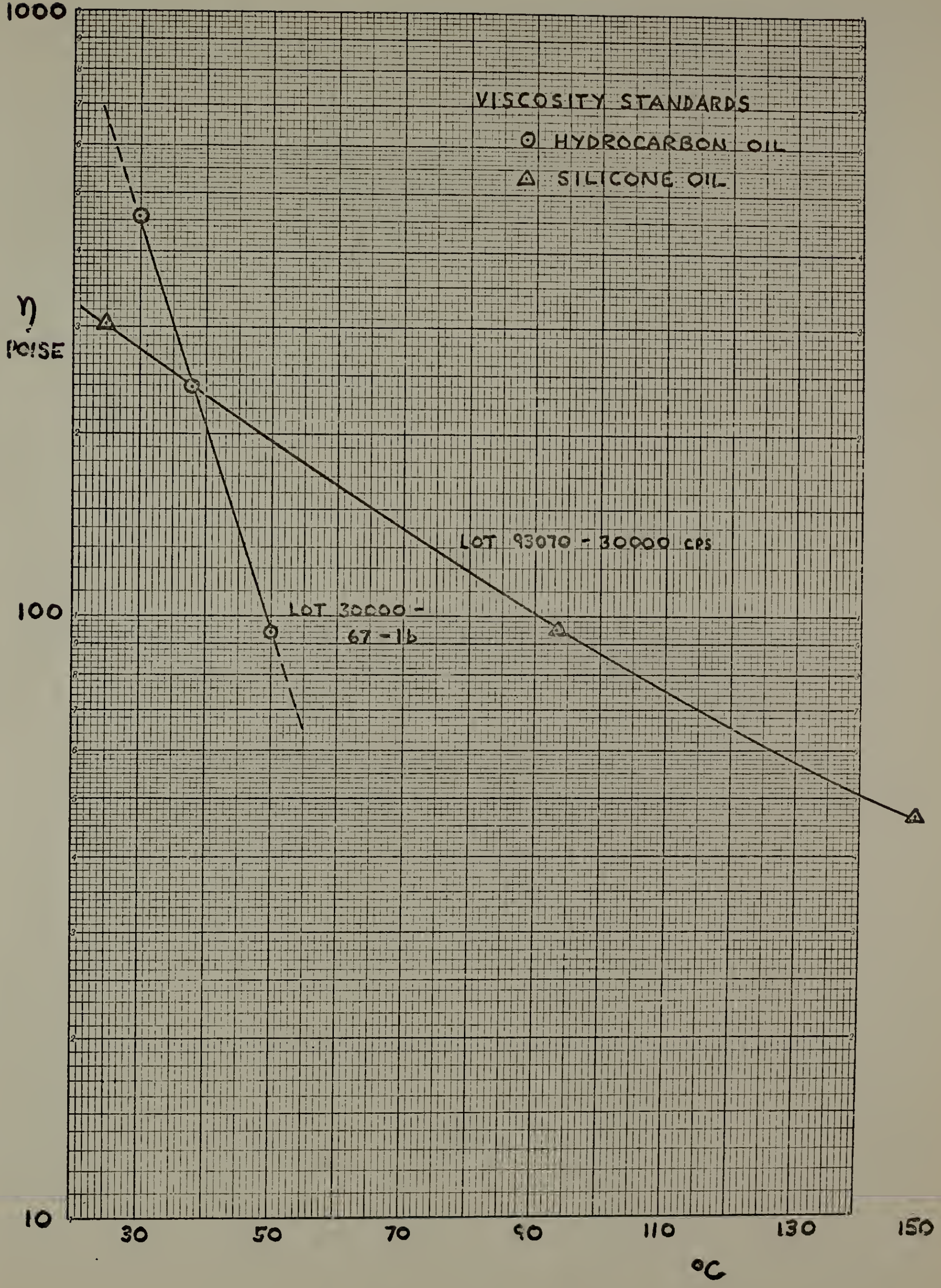
$$h = 1.94\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$

‡ τ arm @ 10", spdl @ 1/2 depth

$$h = 0.97\,\eta\,\frac{\dot{\gamma}_v}{\tau_v}$$


1.0000
0.1000
0.0100
η
POISE
CONT. BELOW
LOT 81871 - 100 CPS
VISCOSITY STANDARDS
HYDROCARBON OIL
SILICONE OIL
CONT. OF 81871 ABOVE
1.00
LOT S-20 - 71106 - 30 CPS
LOT 81871 - 5 CPS
LOT
S-3-65-1a - 3 CPS
20.0
40.0
60.0
80.0
100.0
°C


VISCOSITY STANDARDS
HYDROCARBON OIL
SILICONE OIL
LOT 81371 - 1000 CPS
LOT S-600-45-2d - 1400 CPS
100.0
η
POISE
10.00
1.000
20.0
40.0
60.0
80.0
100.0
°C


VISCOSITY STANDARDS
HYDROCARBON OIL
SILICONE OIL
LOT 93070 - 30000 CPS
LOT 30000 -
67 - 1b
1000
100
10
η
POISE
30
50
70
90
110
130
150
°C

```
1    PROGRAM TEMP
2    T=(NOTE 1)
5    A=(NOTE 2)*2*3.1416/60.0
10   R=(NOTE 3)*2.54/2
15   RI=(NOTE 4)*2.54/2
20   VIS=(NOTE 5)
25   U=(NOTE 6)
30   S=(R/RI)
35   V=A*R
40   BR=(VIS*V*V)/(U*T)
45   W=2*A/(1-S**2)
50   C=S *  (W/A)
55   FS=(1-1/(S*S))/LOG(S)
60   D=1-LOG(FS/2.0)
65   D1=FS*D/2.0
70   D2=(C**2/4.0)*(1.0-D1)
75   DT=T*BR*D2
80   DD=(1.0-1.0/(S*S)-2.0*LOG (S)/(S*S))
85   DE=BR*T*C**2*DD/4.0
90   PRINT 95,DE
95   FORMAT(5X,*TEMP INCREASE ADIABATIC WALL*,1X,F7.2,
96A  1X,*DEG*)
100  PRINT 105,DT
105  FORMAT(5X,*TEMP INCREASE ISOTHERMAL WALL*,1X,F7.2
106A ,1X,*DEG*,/,/)
110  END
220  END PROGRAM
```

NOTES

1. Temperature in $^{\circ}C$
2. Rotations per Minute of Inner Cylinder
3. Inner Cylinder Diameter in Inches
4. Outer Cylinder Diameter in Inches
5. Viscosity in Poise
6. Thermal Conductivity in $gm\ cm\ /\ sec^3\ ^{\circ}C$

APPENDIX XII - FORTRAN Program for Viscous Heating

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ADDENDA - VI. VISCOMETER APPLICATION*

B. Viscosity Measurement at High Brinkman Numbers

The equations of motion and energy for the non-isothermal flow of a Newtonian liquid have been solved recently by Gavis and Laurence[39], based upon assumptions of an exponential dependence of viscosity on temperature and no-pressure flow between moving, parallel surfaces. They made an interesting observation that the temperature field arising under such conditions should be double valued for a given applied shear stress, but that this had not yet been shown experimentally "because no one has extended [such measurements] to large enough Brinkman numbers". The Brinkman number of a Newtonian fluid is defined as a dimensionless parameter which characterizes the tendency of a system to display viscous heating effects[40]:

$$Br = \frac{\beta \eta_o V^2}{k T_o}$$

where: β = dimensionless exponent in viscosity/temp. equation

η_o = viscosity (poise) k = conductivity $(\frac{gm\ cm}{sec^3\ ^oK})$

V = linear velocity (cm/sec) T_o = temperature (^oK)

---

* This section was added with the approval of the thesis committee, after the defense of the thesis proper.

It may also be described as the ratio of heat generated by viscous dissipation to heat removed by conduction. The experimental difficulty in extending such measurements to large Brinkman numbers is that exceptionally high viscosities must be measured at substantial laminar shear rates, a combination of conditions particularly suitable for the high shear viscometer. We have, in fact, observed this double valued viscous heating phenomenon in our viscometer with Araclor 1260[37], a temperature sensitive 30,000 poise chlorinated polyphenyl plasticizer, thus supporting the prediction made by Gavis and Laurence.

1) Experimental Considerations

The details of this application are reported elsewhere, as part of a larger study by Sukanek[38]. In general, the shear stress developed by Araclor 1260 was measured under adiabatic conditions at a variety of shear rates. The widest gap cylinder pair (5000 μ inches) was used, and the Araclor sample was injected by a heated syringe and then cooled to a preselected temperature. Shear rates were chosen which promoted viscous heating, and τ, $\dot{\gamma}$ and T_o (at the wall) were recorded when equilibrium was reached.

2) Results and Discussion

Figure 18 shows the results obtained when Brinkman numbers calculated at selected shear rates are plotted against the measured (dimensionless) shear stress. It clearly demonstrates the double valued nature of the Brinkman number for a given applied shear stress. The observed shear stress values fall somewhat below those of the theoretical curve of Gavis and Laurence, possibly due to incomplete filling of the cylinder gap. Nevertheless, it is believed that this is the first experimental demonstration of shear stress inversion at high Brinkman numbers.

FIGURE 18 - THE REDUCTION IN DIMENSIONLESS SHEAR STRESS AT HIGH BRINKMAN NUMBERS FOR ARACLOR 1260 IN PLANER COUETTE FLOW WITH AN ADIABATIC INNER CYLINDER.