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AN EXPERIMENTAL DEVICE FOR MEASUREMENT OF THE VISCOELASTIC PROP--ETC(U)
NOV 80 W J FENRICK, G A HILL

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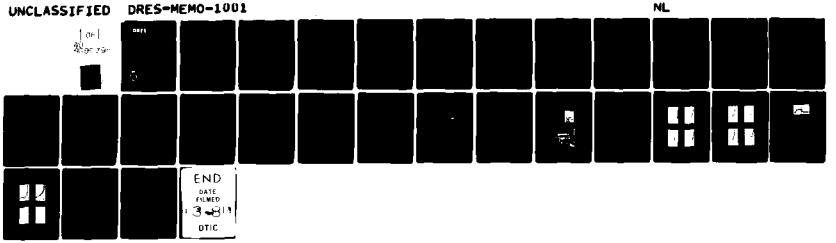

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AN EXPERIMENTAL DEVICE FOR MEASUREMENT OF THE VISCOELASTIC PROPERTIES OF THICKENED LIQUIDS (U)

by

W.J. Fenrick and G.A. Hill

ACN 13E10

November 1980

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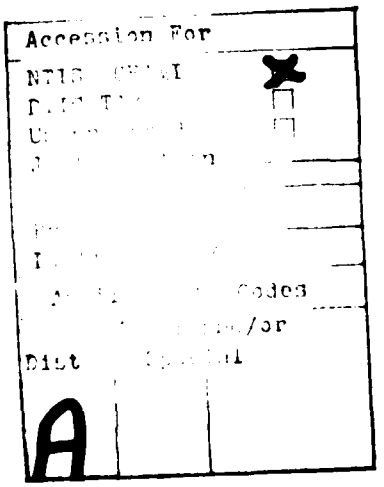
ACKNOWLEDGMENTS

The authors wish to acknowledge the valuable assistance of Mr. J.J. Vesso of DRES who mounted the strain gauges on the cantilever. Mr. Vesso also provided guidance in the selection, application and operation of the electronic equipment associated with the strain gauges.

The authors also wish to acknowledge the contributions of Mr. J.C. Muirhead of DRES who first suggested that a measuring device be built and who helped with the interpretation of the first signatures.



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AN EXPERIMENTAL DEVICE FOR MEASUREMENT OF THE VISCOELASTIC PROPERTIES OF THICKENED LIQUIDS (U)

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ABSTRACT

A novel device for measuring the viscoelastic properties of thickened liquids has been built and tested at DRES. The device involves the withdrawal of a thin metal rod from the liquid under test. The variation in force applied to the rod as the filament of liquid between the rod and the liquid surface relaxes and separates is monitored by strain gauges and displayed on an oscilloscope. The oscilloscope traces provide force time signatures unique to each combination of viscosity and elasticity, which can be photographed and compared. The apparatus and its development are described in detail and oscillographic signatures obtained from various viscoelastic liquids are shown. Some initial theory is discussed and some recommendations are set forth.

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INTRODUCTION

In order to compare the viscoelastic properties of various thickened liquids it has been customary for laboratory personnel to dip the pointed end of a pencil into the liquid and withdraw it at the rate of approximately 12 to 15 centimeters per second. If the liquid was thick and sticky a filament of the material would form stretching from the pencil point down to the surface of the liquid. The filament would then relax and separate over different lengths of time depending on the viscoelastic properties of the liquid. This report describes the development of a novel electromechanical device which performs essentially the same test but is capable of obtaining quantitatively reproducible measurements of the elasticity of the fluid.

The apparatus consists of a small, hollow metal rod suspended vertically from the end of a cantilever. The cantilever is fitted with

strain gauges that respond to its deflection when the rod is withdrawn from a liquid (Fig. 1). Because the cantilever and rod comprise an extremely sensitive assembly, the unit was designed having the cantilever and rod fixed while the withdrawal was accomplished by lowering a container filled with the liquid under test.

The device works on two separate principles, both of which can theoretically provide data on the viscosity and elasticity of a fluid. A diagramatic illustration of a typical curve obtained from the apparatus is shown in Figure 2. The first part of the curve is due to a stretching motion on the filament of liquid. The second part of the curve is due to a subsequent relaxation of the filament that has been formed after the stretching action has stopped. Middleman (Ref. 1) performs an analysis which is valid for Part 1 of this process; however, it requires some knowledge of the filament at time zero (before being stretched), and in this case there is no filament at time zero. Therefore, the analysis needs to be modified somewhat and it is hoped that this can be done in some subsequent research.

The part of the curve which is of interest to us, however, is Part 2 (Fig. 2). In our analysis we assume that this part of the curve is affected only by the elastic properties of the fluid. Initially this assumption ignores the fact that there is some viscous flow within the filament after it is stretched. Thus, the relaxing part of the curve (Part 2) is due to stress relaxation of the polymeric molecules. If we assume the Maxwell model for a viscoelastic fluid, then the stress relaxation is given by (Ref. 1):

$$\tau = \tau_o e^{-t/\lambda} \qquad (1)$$

where τ = stress

t = time

λ = relaxation time for viscoelastic fluid.

When $t = \lambda$, then the above equation gives:

$$\frac{\tau}{\tau_o} = \frac{1}{e} \qquad (2)$$

Thus the time taken for the stress to relax to 1/e of the initial value is the relaxation time for the polymeric liquid. This relaxation time is related to the viscosity of the fluid (η_o) and the elastic shear modulus (G) by (1):

$$\lambda = \eta_o/G \qquad (3)$$

Thus by measuring λ for a series of fluids with identical viscosities, we are measuring the differences in elastic properties of the fluid. This is the basic principle behind the device.

APPARATUS DESCRIPTION

The cantilever shown in Figure 1 is made of 0.25 mm thick phosphor bronze and has strain gauges mounted on either side of the narrow portion that are electrically connected to form a typical half-bridge configuration. Two lengths of a No. 17 hypodermic needle, 1.4 mm in diameter, are silver soldered together to form a "T" shape and the cantilever is designed so that it supports the "T" piece by both ends of its horizontal component. When the "T" piece is mounted it pivots on the cantilever at the points of contact and the vertical portion of it hangs down so that it can be dipped into the liquid under test. The tip of the "T" that dips into the liquid is plugged to prevent material entering the tube. The rod is hollow to reduce weight and thereby improve the signal from the strain gauges. When the container of thickened fluid is lowered, the tip of the vertical component of the "T" (the stylus) is pulled out of the fluid and a filament of fluid is formed. The weight of the filament on the stylus pulls down on the cantilever and as the filament slowly relaxes and separates the cantilever is slowly released. Tracings of the first two signals obtained during the initial testing of the apparatus are shown in Figure 3.

To facilitate more uniform withdrawal of the stylus from the liquid, the apparatus schematically illustrated in Figure 4 was designed. The sample cup was fitted to the plunger of a hypodermic syringe using a specially manufactured TEFLON® adaptor. A vacuum pump was connected to

the outlet of the syringe so that the barrel of the syringe could be evacuated causing the plunger to be forced into it by atmospheric pressure. The rate at which the plunger moved into the barrel of the syringe was controlled by installing a Hoke micrometer needle valve in the vacuum line between the syringe and the vacuum pump. In order to move the plunger upwards, and reimmerse the tip of the stylus into the fluid, the syringe barrel must be vented to atmosphere. Venting was accomplished by installing a solenoid valve in the vacuum line that was normally left open while the plunger was being reset and the rest of the apparatus was prepared for activation. When the solenoid was closed, the syringe barrel was evacuated and the plunger moved downward at a rate governed by the needle valve. The hypodermic syringe was mounted so that the plunger could be repositioned to a fixed height (Fig. 5), which provided a means of reproducing the immersion depth of the stylus into the fluid.

DETERMINING THE OPTIMUM NEEDLE VALVE SETTING

The speed at which the stylus is withdrawn from the fluid is an important factor in the determination of the shape of the force time signature. An experiment was conducted wherein the speed of the plunger was varied by setting the needle valve at different values ranging from 4 to 20 units. Several signatures were produced at each value chosen and the results are illustrated in Figure 6. At 4 units, the syringe was barely operational (Fig. 6A). The signature shown in Figure 6B was obtained at a higher plunger velocity and a needle valve setting of 18 units. Tests conducted using valve settings greater than 18 units showed only that the amplitude of the signal increased as the velocity of the plunger increased. The speed of the plunger when the needle valve is set at 18 units is between 12 and 15 centimeters per second. This is comparable to the speed used in conjunction with the pencil point test and will be discussed in detail later.

VIBRATION INTERFERENCE

It was discovered that the cantilever and stylus assembly

was so sensitive to vibration that it could be set into oscillation by the plunger of the hypodermic syringe contacting the bottom of the barrel when it was drawn into it. Other mechanical components which were found to cause vibrations sufficient to excite the cantilever into resonance were the solenoid, the solenoid switch and the vacuum pump. To prevent the excitation of the cantilever by these components the following actions were taken:

(a) the solenoid valve was shock mounted;

(b) the solenoid switch was located on the end of an electric cord (No. 1, Fig. 7); this switch also triggers the oscilloscope trace;

(c) the vacuum pump was located some distance from the apparatus on the floor with a vibration isolation pad under it; and

(d) the cantilever was mounted separately on a massive metal mount (No. 3, Fig. 7) which also rested on a vibration isolation pad.

Portions of the apparatus are shown in Figure 7. The components illustrated in Figure 4 are built into the general purpose instrument case (No. 2, Fig. 7). Not shown in the figure are the TEKTRONIX® oscilloscope, the strain gauge signal amplifier and the vacuum pump.

Assembly (No. 3, Fig. 7) consists of the cantilever and stylus mounted on a massive metal mirror mount which has two-dimensional adjustment. The mirror mount (UNERTL Optical Co.) is supported by a vibration isolation pad and is massive enough to provide stability. The cantilever and strain gauges are further protected from damage by robust guards, which are shown in Figure 7 and diagrammatically illustrated in Figure 8.

An attempt was made to damp the high frequency oscillations caused by the mechanical action of the equipment out of the signatures by placing a small amount of silicone grease between the upper movement limiter and one edge of the cantilever (Fig. 8). The diameter of the

column of silicone grease connecting the two components was approximately 1.0 to 1.3 mm. Subsequent tests confirmed that some of the high frequencies had been damped out and that the silicone grease damping had not significantly affected the shape of the signature.

Oscillographic signatures from four liquids of widely varying characteristics are shown in Figure 9. These signals exhibit far less interference from vibration but an oscillation caused by the plunger "bottoming" is still in evidence on the signal produced using "tap water". This oscillation was used to determine the velocity of the plunger.

THE VELOCITY OF THE PLUNGER

The distance over which the plunger travels (4.128 cm) is consistent and governed by stops attached to the syringe clamps (Fig. 5). A reasonable estimate of the time required for the plunger to travel the above fixed distance can be obtained from the signature labelled "tap water" (Fig. 9). Movement of the plunger commences at 0.1 seconds where the first influences of force are detected by the strain gauges on the cantilever. The stylus is released by the fluid and the cantilever returns to its original position at 0.2 seconds. The plunger strikes the bottom of the syringe barrel at 0.4 seconds and causes the cantilever to oscillate, as is indicated by the signature between 0.4 and 0.5 seconds. Using the above data, an average velocity of 13.7 cm per second can be calculated for the syringe plunger. To verify the velocity several additional measurements were made using faster oscilloscope sweep speeds; the velocities obtained ranged from 13.7 to 14.7 cm per second.

DEPTH OF IMMERSION OF THE STYLUS

The UNERTL® mirror mount, shown in Figure 7, has an adjustment which can raise and lower the platform on which the cantilever mount is fixed. The platform can be adjusted in increments of 0.025 mm and is employed to control the depth of immersion of the stylus. With the plunger in the maximum upward position, the platform is lowered 0.025 mm at a

time until the tip of the stylus contacts the fluid in the sample cup. The platform is then further lowered by the amount specified for the depth of immersion. Several tests were conducted using a single viscoelastic fluid and a variety of immersion depths. Figure 10 shows the results obtained using DOWANOL® thickened with ROHM and HAAS K125® polymer and immersion depths varying from 0.0 to 2.50 mm.

At an immersion depth of 0.0" (Fig. 10A), where the stylus first touches the liquid, the signature appears to decay to a level below that of the original base line. Figure 11 illustrates diagrammatically the capillary curve which forms around the stylus as it contacts the fluid. The oscilloscope trace, shown above the diagram in Figure 11, is the result of an experiment in which the stylus was lowered until the tip touched the surface of the fluid in the sample container, then the container was lowered, pulling the stylus away from the liquid. The sweep speed of the oscilloscope was decreased to 1.0 seconds per division so that various operations could be carried out during the sweep; an explanation of the signature is as follows. For the first second, after the scope trace was triggered (0.5 to 1.5 seconds), the stylus was being lowered toward the surface of the liquid. At 1.5 seconds on the time grid, the tip of the stylus touched the surface of the liquid and the cantilever was pulled down as the capillary curve was formed. The horizontal trace then moved upward to the center of the vertical calibration lines. At 5.2 seconds, the plunger of the hypodermic syringe was lowered and the tip of the stylus pulled away from the liquid. The resulting reaction of the cantilever is indicated by the signature shape between 5.2 and 6.0 seconds and if this portion of the signal could be elongated horizontally to 10 times its length, it would be very similar to the signature shown in Figure 10A. If the signature for 0.0" immersion is compared to the signature shown in Figure 11, an explanation for the decay of the signature in Figure 10A to a level below the original base line can be derived. It is apparent that the base line in Figure 10A is formed by that portion of the signature in Figure 11 which occurs between the 2.0 second and 5.0 second time lines. It would

be reasonable to assume that the signature would always decay to a point below the base line, except for the fact that a minute amount of liquid remains on the tip of the stylus after it is withdrawn. This material causes a residual loading on the cantilever that increases with the immersion depth. Signatures which were considered to be acceptable, such as Figure 10D, result when the residual loading balances the capillary curve loading.

It is apparent from Figure 10 that the amplitude of the signature τ_o is increased with the immersion depth but the relaxation time λ is not. The above is well illustrated by Figure 10D in which the vertical scale had to be doubled, from 0.1 to 0.2 volts/division, to contain the amplitude of the signature, while the relaxation time remained unchanged.

Theory suggests that the immersion depth of the stylus should not affect the relaxation time, and for verification the following experiment was conducted. Nine separate signatures were obtained using DOWANOL DPM thickened with ROHM and HAAS polymer K125, to a viscosity of 18.4 g/cm s. The immersion depth of the stylus was varied from 0.000 cm (Run No. 1) to 0.200 cm (Run No. 9). The results of the experiment are shown below.

Table 1. Reproducibility of Elasticity Measurements

Run No.	Immersion (cm)	λ (sec)
1	0.000	0.35
2	0.025	0.35
3	0.050	0.35
4	0.075	0.34
5	0.100	0.34
6	0.125	0.35
7	0.150	0.32
8	0.175	0.34
9	0.200	0.34

These data demonstrate that the signatures are reproducible and that the relaxation time is not a function of the immersion depth. Taking the average relaxation time from the above data as 0.34 sec and using the viscosity of our sample 18.4 g/cm s (at zero shear) yields a value of 54.1 g/cm s^2 for the elastic shear modulus of our fluid. This experiment indicates that the apparatus can provide a reliable, reproducible method of measuring an elastic property of a semi-viscous fluid.

Reproducibility of the signatures, when the immersion depth is held constant, is shown in Figure 12. During this series of tests, two different fluids were used, SAE No. 50 motor oil and DOWANOL thickened with ROHM and HAAS polymer. The immersion depth was held constant at 0.25 cm while the signatures were repeated 20 times and superimposed over each other. Figure 12 shows a photograph of the first single signature together with that of twenty signatures superimposed, for both No. 50 oil and thickened DOWANOL. The value for λ obtained from the No. 50 oil signatures was 0.035 $\pm$ 0.010 sec. When the DOWANOL signatures were obtained, the vertical and horizontal oscilloscope settings were left unchanged so that direct size comparison could be made. The DOWANOL signature was unfortunately not entirely contained within the photo and the most accurate value that can be estimated for λ is 0.21 $\pm$ 0.01 sec.

CONCLUSION

It has been shown (Fig. 9) that the apparatus produces signatures of varying characteristics when liquids with various viscoelastic properties are tested. Much is yet to be understood about the signatures and the forces that govern their shape, but before further experiments are undertaken more sophisticated apparatus should be designed. The hypodermic syringe used to lower the sample cup could be replaced by a double action pneumatic cylinder, both lowered and raised pneumatically. Also, a cantilever of optimum size, shape and thickness could be designed. A study has already been undertaken by Bayly (Ref. 2) during which com-

puter programs were produced with which the optimum specifications for the cantilever could be computed. Although it has been shown that the silicone grease damping of the cantilever did not significantly alter the overall shape of the signatures, it would be an improvement if a cantilever not requiring such damping could be designed. In the future it is hoped that a cantilever, built to the specifications of Bayly, will be produced and tested.

REFERENCES

1. Middleman, S. "The Flow of High Polymers". J. Wiley and Sons, 1968.

2. Bayly, D.A. "Dynamic Simulation of a Cantilever Beam Type Force Transducer (U)". Suffield Technical Note No. 440. February 1980. UNCLASSIFIED.

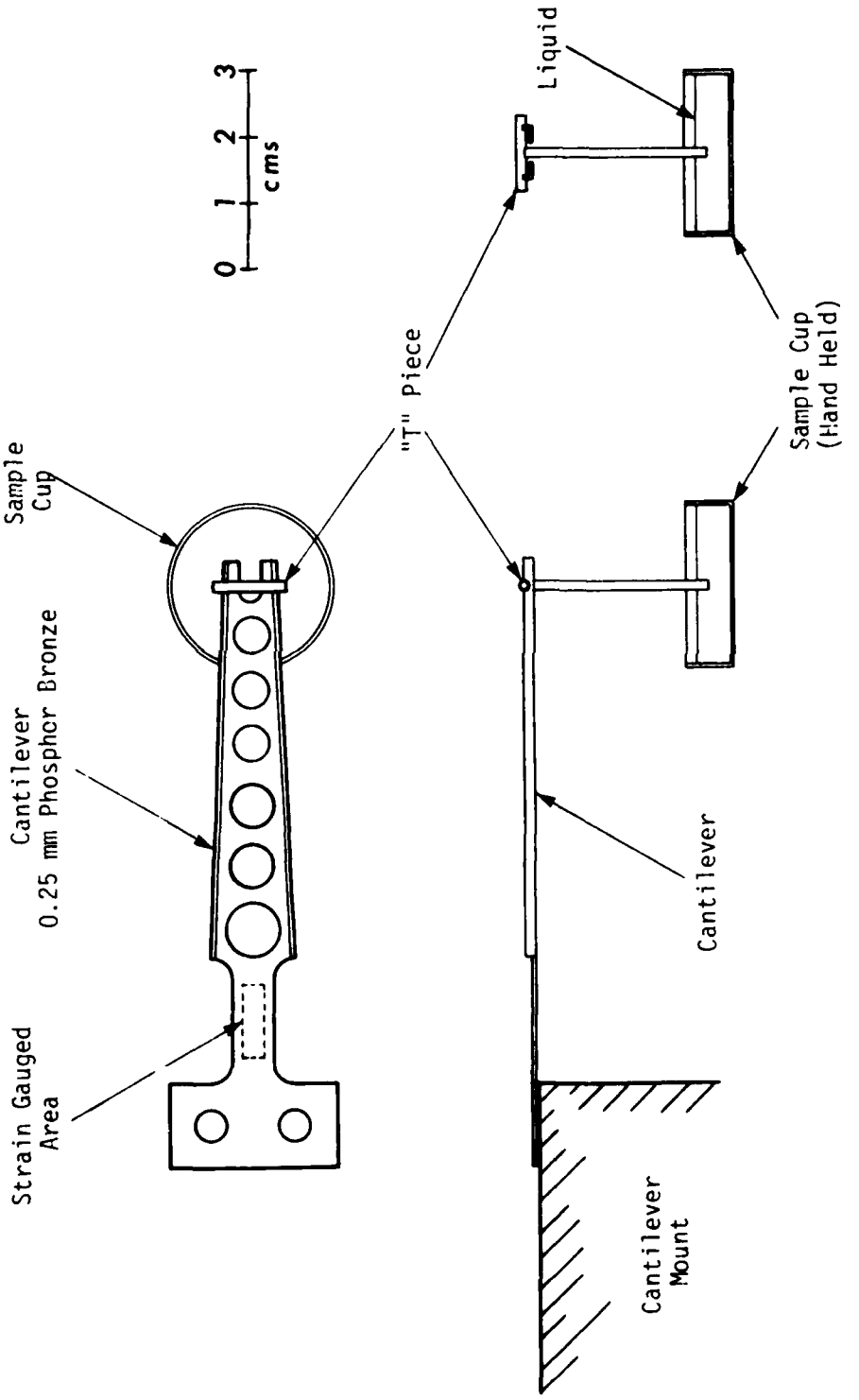


Figure 1. Apparatus Used for Initial Feasibility Tests

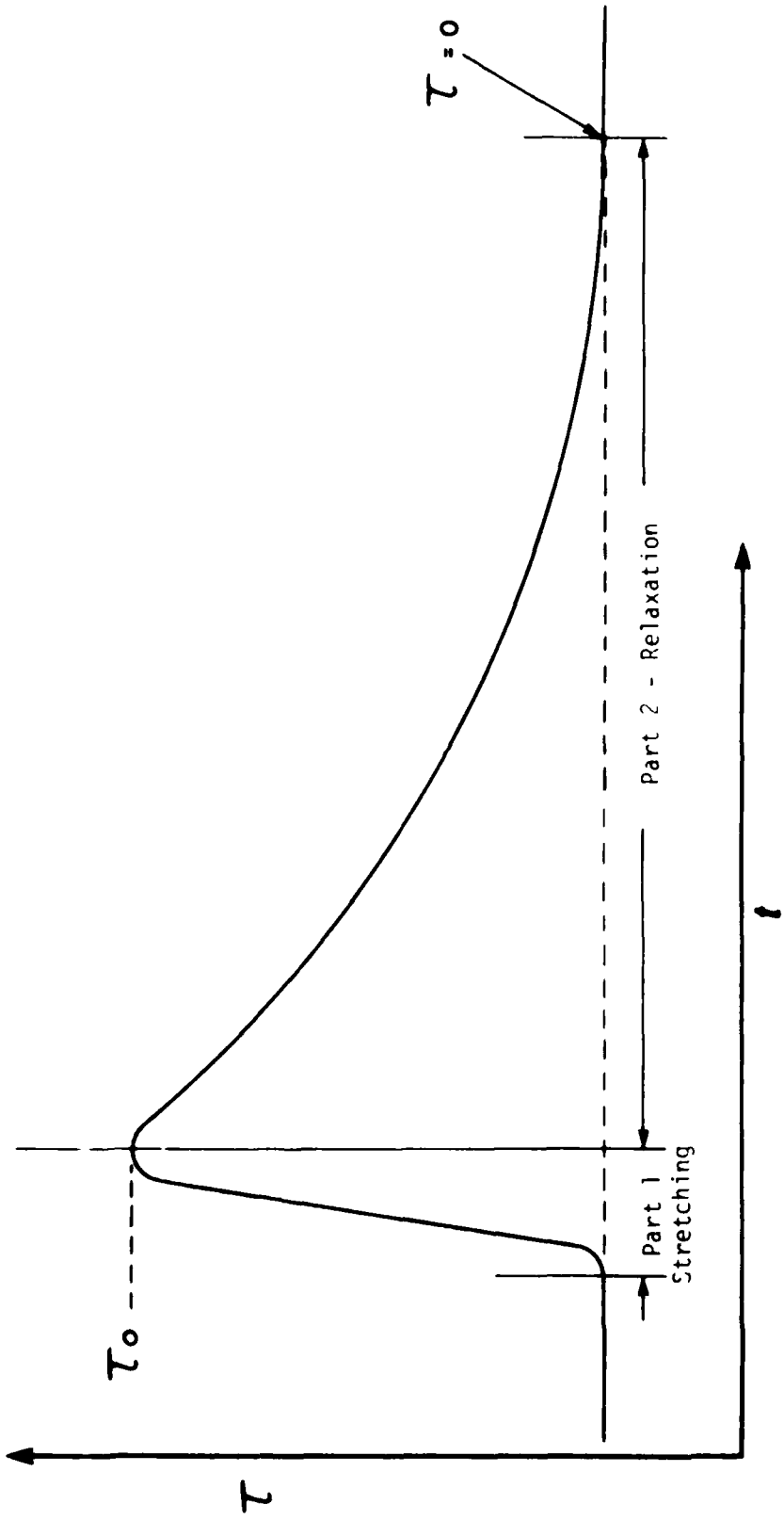
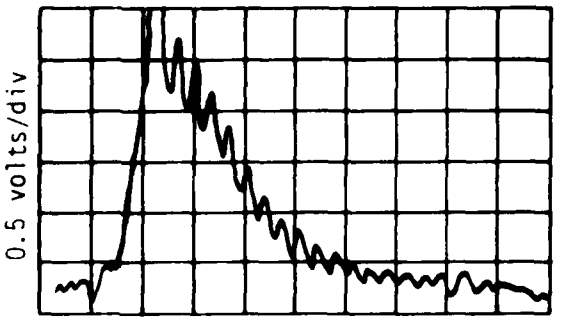
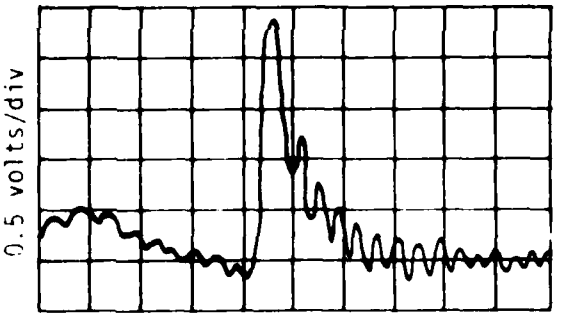


Figure 2. Diagram of a Typical Signal Obtained from the Apparatus



Thickened DOWANOL 6.2 Poise at 20°C



Thickened Diethyl Phthalate 6.6 Poise at 20°C

Figure 3. Tracings of Force Time Signatures Obtained During Initial Experiments

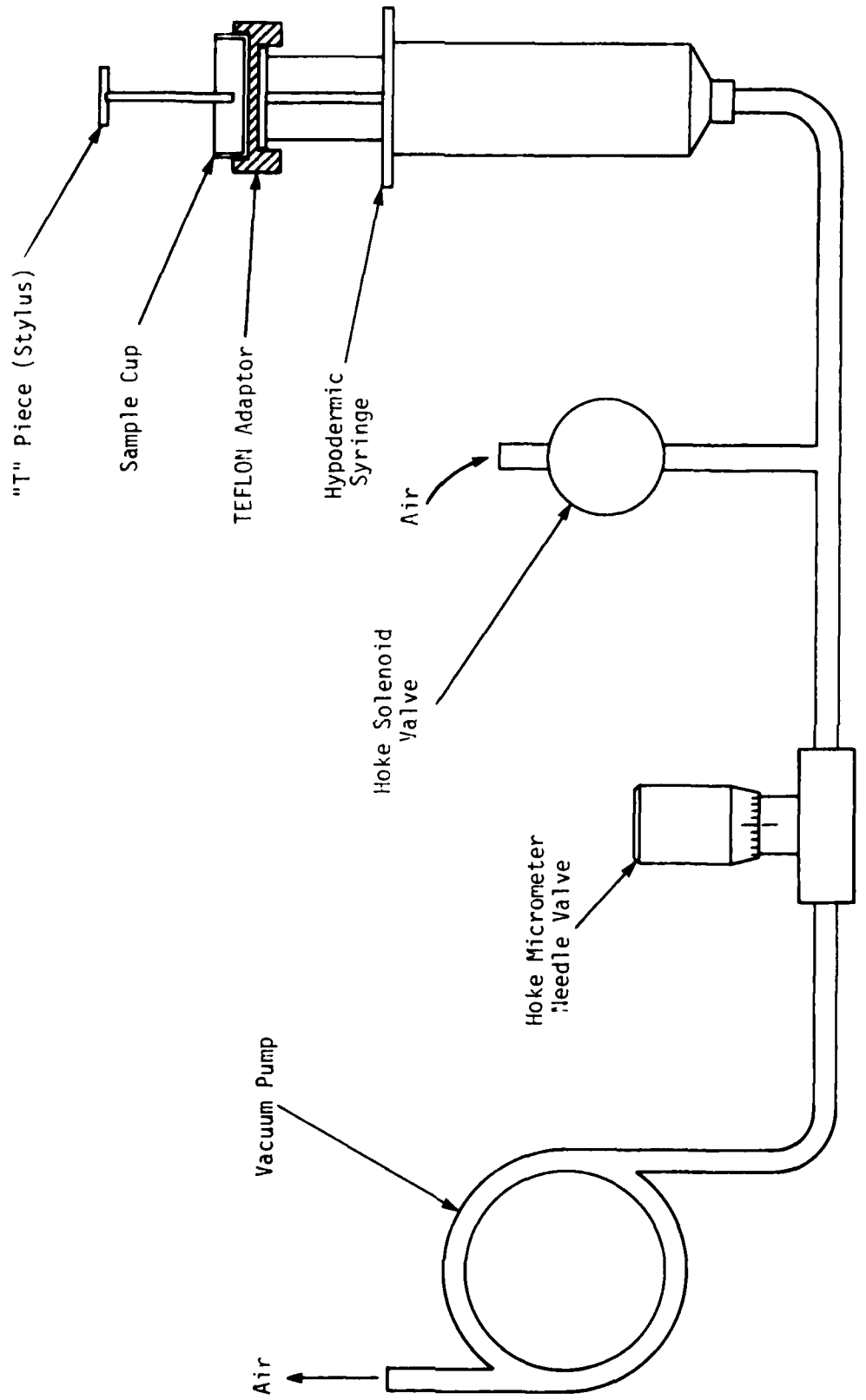


Figure 4. Schematic of Vacuum Operated Hypodermic Syringe

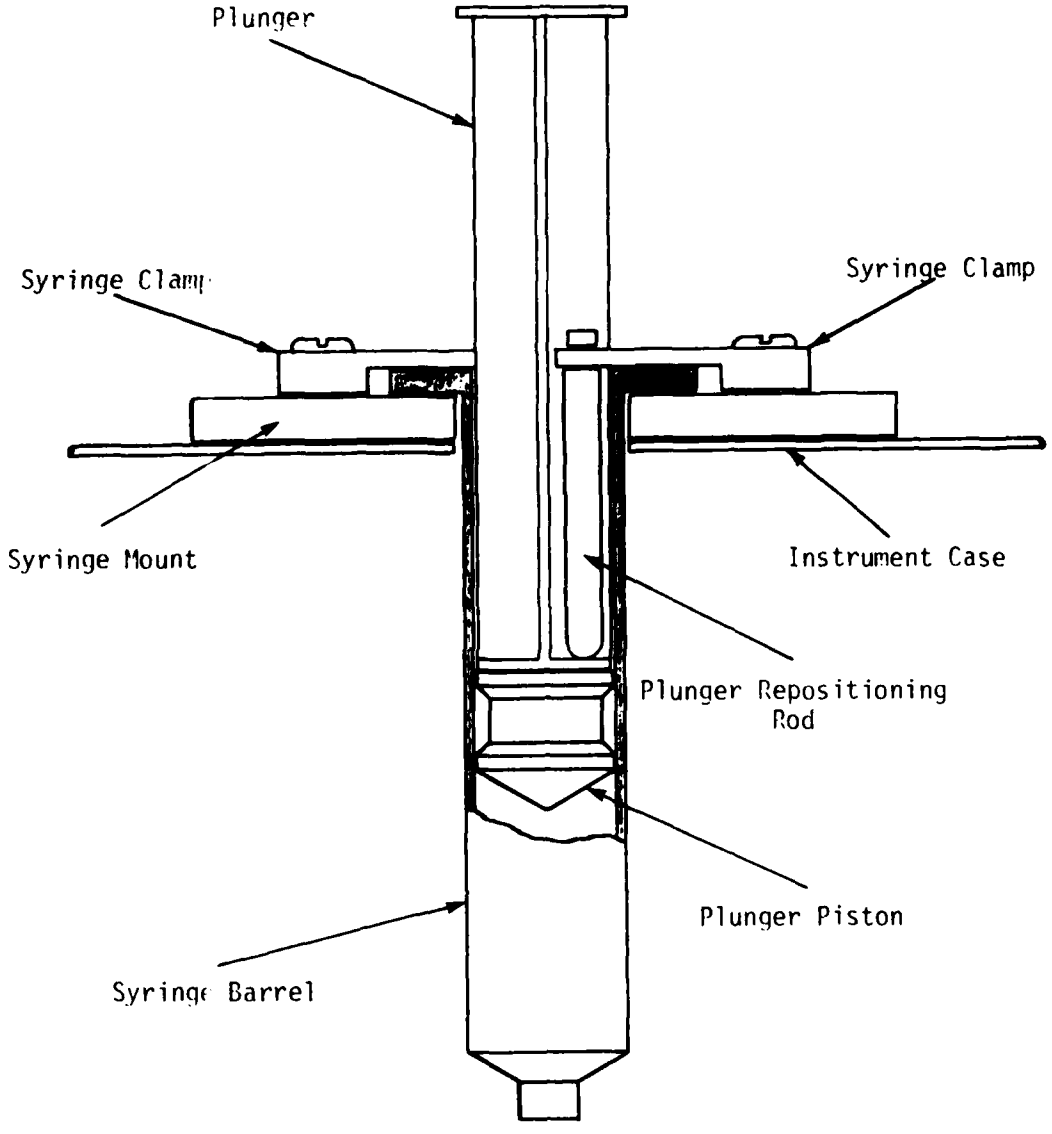
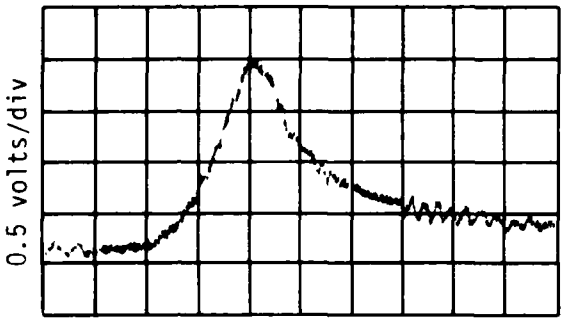
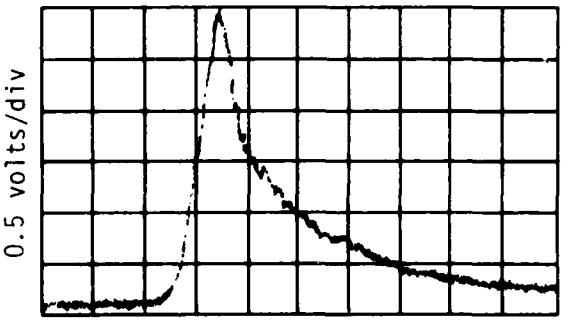


Figure 5. Diagram Illustrating Plunger Height Repositioning System



A - Needle Valve Setting 4 Units



B - Needle Valve Setting 18 Units

Figure 6. Signatures Generated Using Thickened DOWANOL and Different Withdrawal Speeds

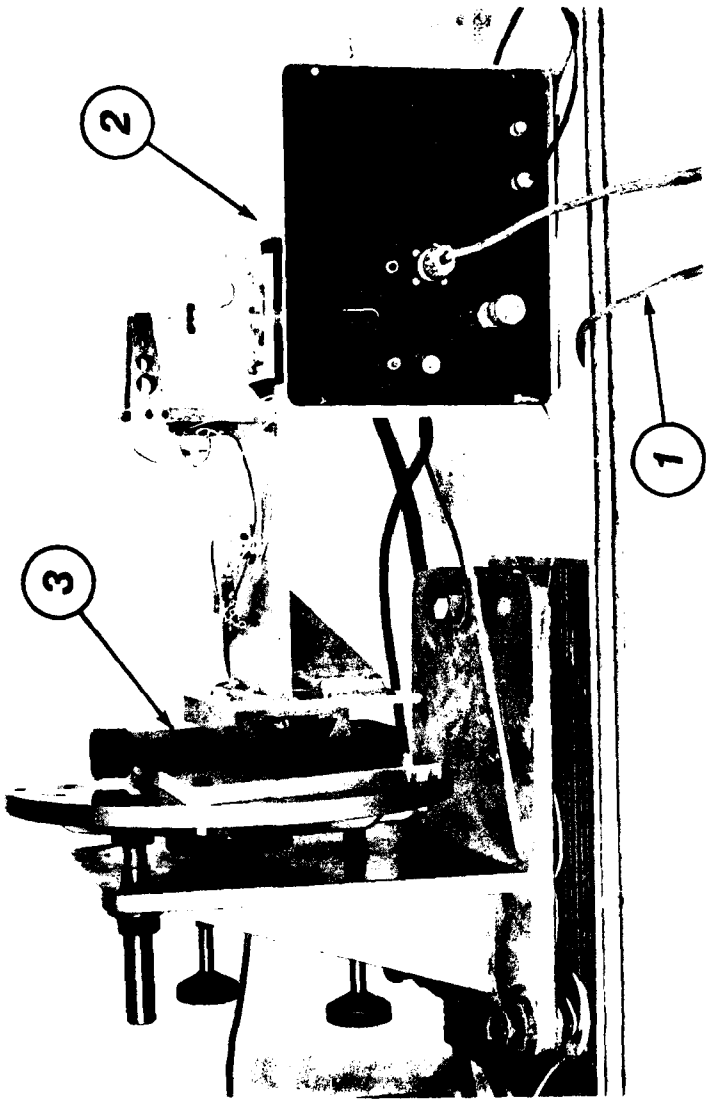


Figure 7. The Filament Forming Apparatus

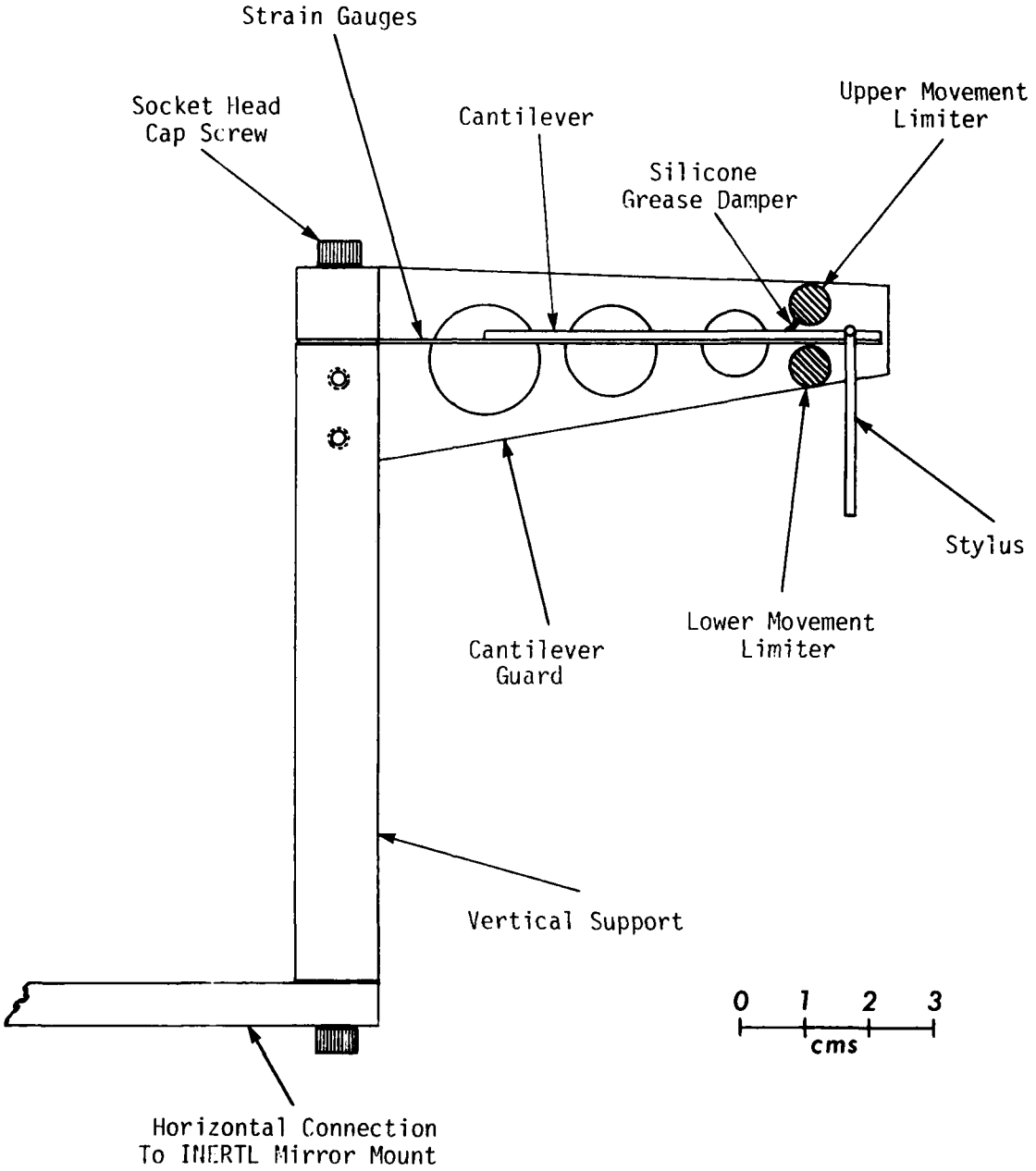
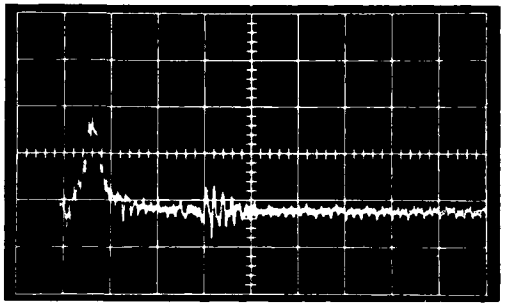
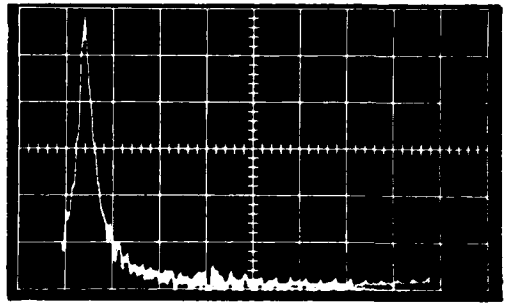


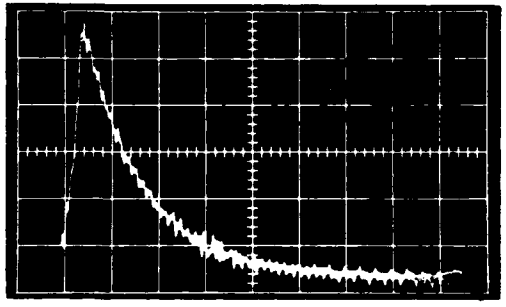
Figure 8. Detail of Cantilever Mount and Silicone Grease Damping



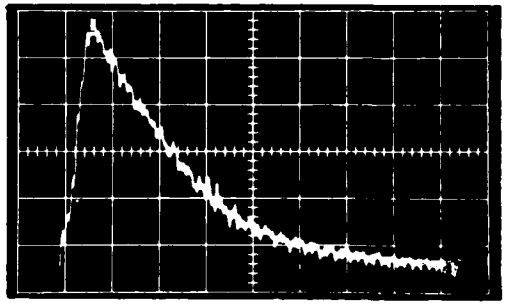
Tap Water



Number 50 Oil



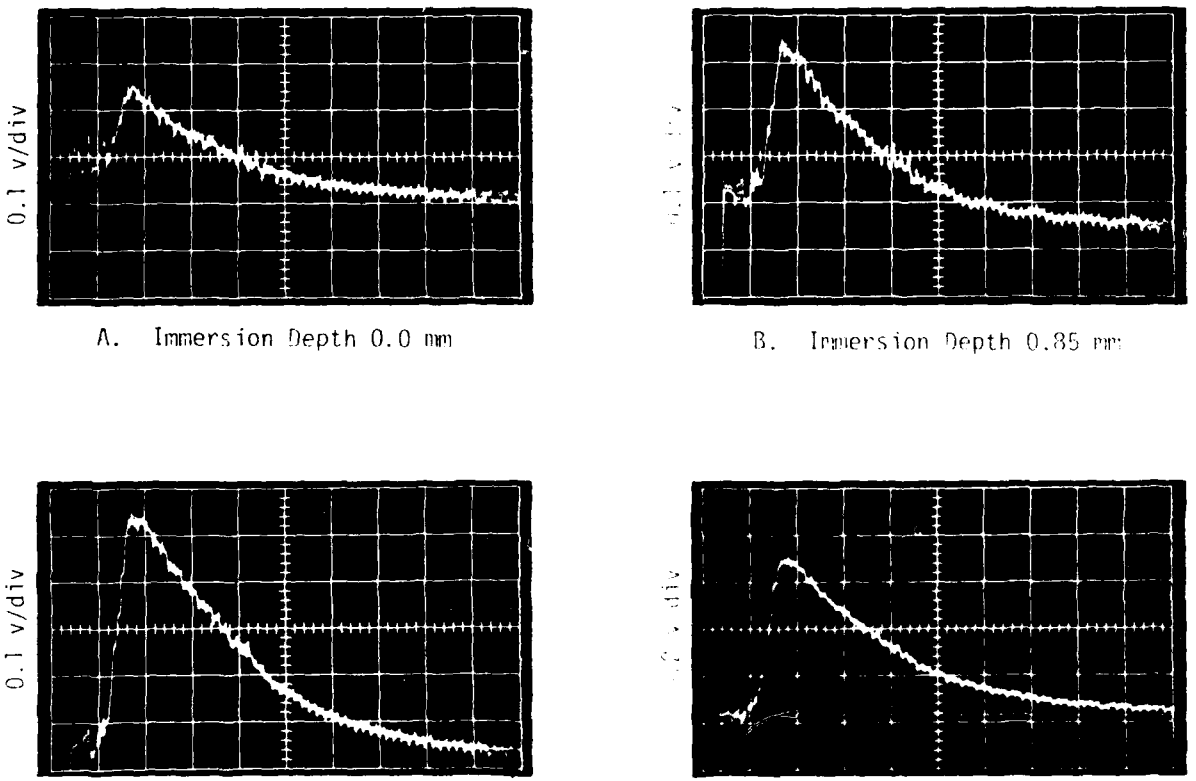
Thickened Diethyl Phthalate



Thickened DOWANOL

Figure 9. Signatures Obtained from Liquids of Differing Viscoelastic Properties Using:

(A) Silicone Damped Cantilever
(B) Needle Valve Setting - 18 Units
(C) Sweep Speed 0.1 sec/div
(D) Vertical Sensitivity 0.1 volts/div



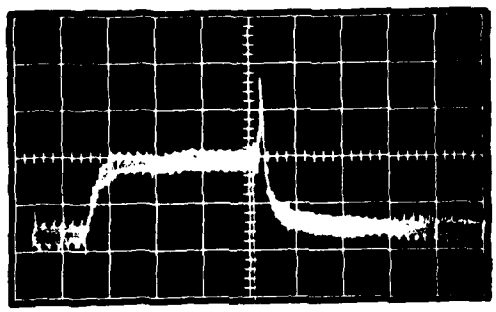
A. Immersion Depth 0.0 mm

B. Immersion Depth 0.85 mm

C. Immersion Depth 1.7 mm

D. Immersion Depth 2.5 mm

Figure 10. Signatures Obtained Using Thickened DOWANOL and Different Immersion Depths (Sweep Speed 0.1 sec/div)



1.0 sec/div

1.0 to 5.0 sec Capillary Loading
5.0 to 10.0 sec Normal Signature

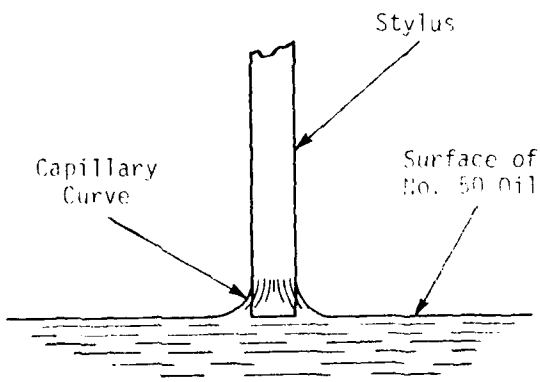
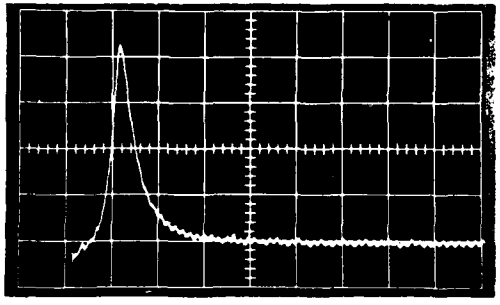
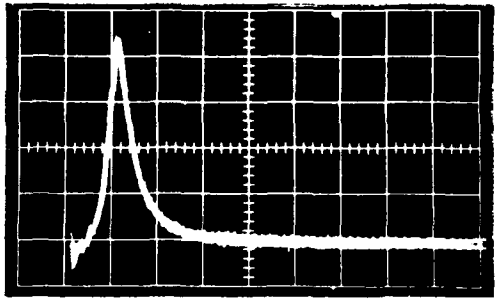


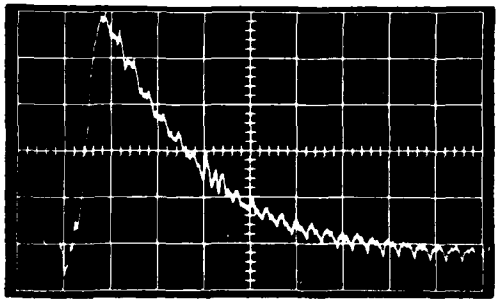
Figure 11. Capillary Curve Loading Cantilever



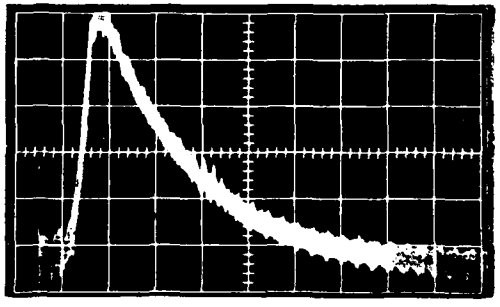
No. 50 Oil Single Signature



No. 50 Oil 20 Signatures



hickened DOWANOL Single Signature



Thickened DOWANOL 20 Signatures

Figure 12. Repeatability of Signatures - (Vertical 0.1 volts/div; Horizontal 0.1 sec/div)

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1 ORIGINATING ACTIVITY

DEFENCE RESEARCH ESTABLISHMENT SUFFIELD

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UNCLASSIFIED

2b. GROUP

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13. ABSTRACT

A novel device for measuring the viscoelastic properties of thickened liquids has been built and tested at DRES. The device involves the withdrawal of a thin metal rod from the liquid under test. The variation in force applied to the rod as the filament of liquid between the rod and the liquid surface relaxes and separates is monitored by strain gauges and displayed on an oscilloscope. The oscilloscope traces provide force time signatures unique to each combination of viscosity and elasticity, which can be photographed and compared. The apparatus and its development are described in detail and oscillographic signatures obtained from various viscoelastic liquids are shown. Some initial theory is discussed and some recommendations are set forth.

(U)

DSIS
78-078

KEY WORDS

Viscosity

Ligand

Rheology

Viscoelasticity

Relaxation

Measurement

Strain Gauges

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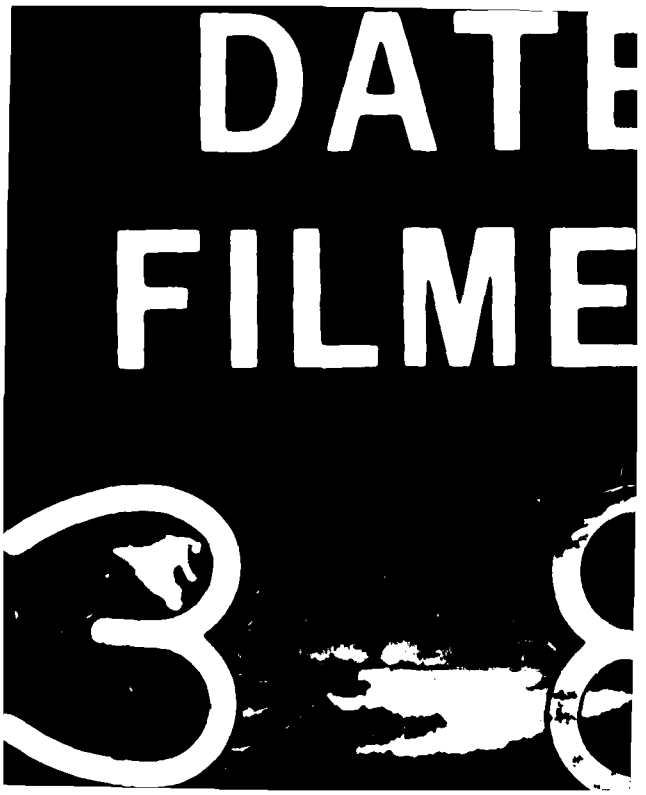
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