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(6) MEASUREMENT OF THE VISCOSITY OF PLASTIC PROPELLANT INGREDIENTS BY PARALLEL PLATE PLASTOMETRY.

by

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SUMMARY

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The viscosities of three liquids used as ingredients of plastic propellant have been measured at room temperature, as a function of strain-rate, by parallel plate plastometry. The liquids were (a) Vistanex LM, a polyisobutene with a falling-sphere viscosity at very low shear-rates of about 100 kPa s (10^6 poise), (b) a mixture of polyisobutene, plasticiser and surfactants known as PSB, used as the binder for many propellant formulations, and (c) an unplasticised mixture of polyisobutene with surfactants, known as USB, similar to another propellant binder.

The strain-rate was varied over a range from 10^{-3} to 10 s^{-1}, and the measured viscosities ranged from 100 to 1 kPa s (10^6 to 10^4 poises). The viscosity of each liquid, within the wide experimental error, was found to be inversely proportional to the square-root of the strain-rate at which it was measured.

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1 INTRODUCTION

Plastic propellant is a stiff paste typically containing (by weight) 89 per cent solids (ammonium perchlorate and perhaps aluminium and a burning catalyst), 1 per cent synthetic surface-active agent and 10 per cent polyisobutene. The polyisobutene has a nominal (viscosity-average) molecular weight of about 4×10^4 and a viscosity of about 100 kPa s (10^6 poise) at 25°C, when measured at a very low shear-rate by the falling sphere (Stokes) method. The paste itself is rheologically similar to modelling clay or Plasticine, and may be considered to a first approximation as a Bingham solid subject to shear-hardening.[1]

As part of a programme aimed at characterising rheologically both plastic propellant and its ingredients, detailed consideration has been given to the methods by which the fundamental rheological properties such as viscosity, plastoviscosity, yield stress and shear-hardening coefficient may be measured. Separate reports deal with plastic propellant:[1,2,3] this report is confined to a consideration of the liquid ingredients, that is (a) the polyisobutene alone, (b) the mixture of polyisobutene with plasticiser and surfactants which is designated PSB (this mixture is the liquid medium for a series of propellant formulations), and (c) a mixture of polyisobutene with surfactants (that is, containing no plasticiser), designated USB, which is similar to the liquid medium of another series of propellant formulations. The three materials are described in Appendix A.

Routine measurements on these very viscous liquids are made with a falling-sphere viscometer, normally using a steel ball of diameter 2.38 mm (3/32 inch) falling through a sample in a tube of 25 mm internal diameter for periods as long perhaps as 24 hours, before a distance is passed which may be measured with sufficient accuracy. When the liquid is opaque, X-rays are used to measure the distance through which the sphere has fallen. The viscosity is calculated from Stokes' law, after applying the Faxén correction for the wall-effect.

A useful survey[4] of viscometric techniques for use with polymeric liquids summarises the range of viscosities over which various methods are useful; and suggests that other methods, such as capillary extrusion, tensile creep and parallel-plate plastometry, might be more suitable for liquids of very high viscosity. Tensile creep experiments have been carried out on plastic propellant,[5] but not on its liquid ingredients. Other desiderata for a suitable method are listed,[4] including ease of thermostatting, availability of a wide range of shear stresses and shear rates, and applicability to small samples. These considerations support the choice of parallel-plate plastometry in the work now reported. The size of sample required is small (the whole apparatus may easily be inserted in a laboratory oven or cold chamber) and about a one-hundredfold range of load is easily available, giving, as will be shown later, about a 10^4-fold range of shear rate.

The availability of a wide range of shear-rate is important when dealing with polymers, which are usually non-Newtonian, ie their viscosity is not independent of shear-rate. It has been shown[6] that the mechanical properties of plastic propellant are highly dependent on strain-rate, so it is important to consider the effect of this parameter on the rheological properties of the ingredients. The routine falling-sphere method, in contrast with the parallel-plate plastometer, does not so readily enable the strain-rate to be varied over a wide range.

The theory and application of the parallel-plate plastometer have been discussed previously:[7,8,9] if a cylindrical sample of volume V is compressed by a constant load W between parallel plates, then a plot of $1/h^4$, where h is the plate separation at time t, against t will give a curve which after the elastic and delayed elastic deformations have taken place becomes linear. From the slope of the linear portion of the curve, the viscosity may be determined from a modified form of Stefan's equation:

$$\frac{3V^2}{8\pi}\left(\frac{1}{h^4} - \frac{1}{h_o^4}\right) = \frac{Wt}{\eta} \qquad (1)$$

The assumptions on which this analysis is based are as follows:

a) the material is incompressible;
b) the viscosity of the material is independent of shear-rate over the range of rates employed during a single test;
c) no body force acts on the material;
d) the motion is slow;
e) there is no slip at the surface of the plates;

f) the plate separation is so small compared to the radius that the velocity component in the perpendicular direction is negligible compared with that in the radial direction.

These assumptions are commented on below.

Assumption (a) is satisfied to a first-degree approximation by a void-free liquid or dispersion of solid particles in a liquid. Assumption (b) is approximately correct within experimental error, as a linear plot of $1/h^4$ vs t is found while the strain-rate is varying over the relatively small range occurring during a single experiment. Assumption (c) is reasonable, as the body forces are of the order of a thousandth of the applied forces. Assumption (d) enables terms in the square of the velocity vector to be neglected in the Navier-Stokes equation. Assumption (e) has been shown[1] to be correct for plastic propellant, and so it is likely that the unfilled liquid medium also will adhere without slippage. Assumption (f) was said[7] to be met if the ratio of radius to height of the compressed specimen was greater than 10: we find no inconsistencies with the ratio ranging from as small as 6 up to 12 for Vistanex and up to 10 for PSB. The USB experiments had ratios in the range 5 to 9. With plastic propellant the range was wider still,[2] from about 2 or 3 up to 18.

It is reported,[7] and confirmed in the work reported below, that viscosities below 1 kPa s at low strain-rates cannot readily be measured with the parallel plate plastometer: such materials slump out of position before they can be tested (ie assumption (c) does not apply).

Another limitation is that when high rates are used, a polymeric sample reacts elastically at the initial compression, causing a disturbance to the record of height against time.[10] With our materials, this phenomenon become noticeable at viscosities lower than a few kPa s.

2 EXPERIMENTAL

The apparatus used was mechanically simple, being essentially a wide circular brass base-plate, on a threaded co-axial support, enabling it to be raised or lowered by rotation about its axis. The whole apparatus stood on a levelling platform fitted with three adjustable screw legs. Above the base-plate there was another horizontal circular brass plate, 51 mm diameter, fixed squarely to, and supported by, a vertical rod (9.5 mm diameter) passing through a low-friction bearing and provided with a concentric circular weight-pan, on which circular slotted weights could be placed. At the top of the rod was rigidly fixed an inverted stirrup, fitting through the jaws of a bomb-release mechanism, which supported the rod and upper plate until the mechanism was activated and the jaws sprang apart, when the upper plate with the rod and weights was free to fall. A cylindrical sample of the material to be tested was placed centrally on the lower plate, which was then raised until the top of the sample just touched the upper plate. Special care was taken to minimise slumping with the less viscous materials. The experiment was started by activating the bomb-release mechanism.

Resting on the upper surface of the upper (moving) plate, and supported vertically with little friction was a brass rod (2 mm diameter) supporting a rod-shaped soft-iron core positioned inside the vertical coil assembly of a Schaevitz linear variable differential transformer (Electro Mechanisms Ltd, Slough), which produces an electrical output proportional to the displacement of the core. The output was led to a Sanborn (Hewlett-Packard) model 321 amplifier recorder which when calibrated recorded the plate separation as a function of time. The speed of response of the recorder (minimum full-scale deflection time of 5 ms) was sufficiently high for the fastest experiments described in this report. The duration of experiments varied between 0.2 and 1200 seconds. The available chart speeds were 0.5, 1, 5, 20 and 100 mm/s from which a suitable speed was chosen for each experiment.

In some experiments with very low loads the above apparatus could not be used since the weight of the moving parts was too great. In these cases a lightweight system using the same recording technique was employed, enabling loads as low as 50 g to be used. In a few experiments with loads greater than 5 kg, a 10:1 lever apparatus was used.

Experience has led to the design of a more convenient and precise instrument, in which calibration is simpler, and is provided with temperature control.

In the work described in this report, the temperature was not controlled: all experiments were performed in the laboratory at $21^{\circ} \pm 2^{\circ}$C.

The standard cylindrical specimen, which is 20 mm high and 15 mm diameter, was prepared by moulding the required volume (3.53 cm^3, obtained by weighing, the density being previously determined), in a steel mould lightly lubricated with the surfactant mixture S101 at a pressure of 7 MPa (70 bars) for one minute. The flat surfaces were bounded with discs of non-stick parting paper, which were carefully removed before the sample was placed on the plastometer base-plate.

3 RESULTS

A typical plot of plate-separation versus time, reproduced from the recorder chart, is shown in Fig 1. From each such plot was derived a curve of $1/h^4$ against time, as shown typically in Fig 2, together with a curve relating the radial strain γ (= $\ln(h_o/h)$)[1] with elapsed time, also shown in Fig 2. The slope m of the linear portion of the $1/h^4$ vs t plot enabled the viscosity to be determined by substituting in Eqn 1. For the standard specimen this becomes

$$\eta = \frac{6.57 \times 10^{-3}\ W(kg)}{m(mm^{-4}.s^{-1})}\ \text{kPa s} \qquad (2)$$

The time range of this linear relation was noted, and the strain vs time curve examined over the range, and a mean slope drawn, thus deriving the mean strain-rate, $\bar{\dot{\gamma}}$, at which the viscosity had been measured. The procedure is illustrated in Fig 2.

The results obtained with each liquid, using a range of loads, are summarised in Table 1 (for Vistanex), Table 2 (for PSB) and Table 3 (for USB); Figs 3, 4 and 5 respectively show the variation of viscosity with mean strain-rate for each liquid, together with the falling-sphere determination (described in Appendix A) plotted against the estimated strain-rate, with its range indicated by the horizontal line.

Considering first Fig 3, it is clear that a linear relation between log viscosity and log strain-rate adequately describes the Vistanex results. The least-squares regression is shown, together with the 95 per cent confidence bands ($\pm$ 2 S_η, standard errors of estimate). The regression equation is

$$\eta = 3.95/(\bar{\dot{\gamma}})^{0.41}\ \text{kPa s} \qquad (3a)$$

The power is close to one-half, and indeed a line of this slope (shown pecked) is, within the experimental strain-rate range, contained within the $\pm$ 1S_η band.

This leads to the conclusion that the relation is indistinguishable from the simpler relation

$$\eta = 3.0/\sqrt{\bar{\dot{\gamma}}} \text{ kPa s} \tag{3b}$$

There is thus an empirical relationship between the viscosity and the reciprocal of the square root of the mean strain-rate over a 10^4 range of rate.

It will also be noted that the falling-sphere determination falls within the 95 per cent confidence band, showing that the two methods of determining viscosity are in good agreement.

The data for PSB in Fig 4 have been similarly analysed. Here the regression equation is

$$\eta = 0.41/(\bar{\dot{\gamma}})^{0.49} \text{ kPa s} \tag{4a}$$

with the power very close to one-half. The line representing this equation is barely distinguishable from that for the simpler relation

$$\eta \approx 0.38/\sqrt{\bar{\dot{\gamma}}} \text{ kPa s} \tag{4b}$$

for which the line has been drawn in Fig 4, together with the 95 per cent confidence bands, as before. Again an empirical relationship is found between the viscosity and the square-root of the mean strain-rate, over a 10^2 range of rate.

The falling-sphere determination falls on the edge of the 95 per cent confidence band, showing again that the two methods of measuring viscosity are in agreement.

The data for USB in Fig 5 have also been analysed in this way, with a regression equation

$$\eta = 1.1/(\bar{\dot{\gamma}})^{0.34} \text{ kPa s} \tag{5a}$$

With the rather scattered results, it is possible that the simpler relation

$$\eta \approx 0.44/\sqrt{\dot{\gamma}} \text{ kPa s} \tag{5b}$$

is adequate, as it falls within the 95 per cent confidence band over the experimental strain-rate range of 10^2. The falling-sphere determination falls just outside this band.

4 DISCUSSION

The high viscosity of liquid polymers is caused by entanglements, and it decreases with increasing shear rate. This pseudoplastic behaviour has been considered,[11,12] and various empirical equations have been discussed.[12] For example, the following equation represents the behaviour of many systems:[12]

$$\eta = \eta_\infty + (\eta_0 - \eta_\infty)/[1 + (\tau\dot{\gamma})^n] \tag{6a}$$

where η is the viscosity at shear rate $\dot{\gamma}$, and η_0 and η_∞ are limiting values at $\dot{\gamma} = 0$ and $\dot{\gamma} = \infty$, respectively. τ is a constant such that at a shear rate τ^{-1} the viscosity assumes the mean value $(\eta_0 + \eta_\infty)/2$.

If η_∞ is zero, or negligible, and at rates of shear such that $(\tau\dot{\gamma})^n \gg 1$, then this becomes

$$\eta = \eta_0/(\tau\dot{\gamma})^n \tag{6b}$$

and a plot of log η against log $(\tau\dot{\gamma})$ is linear, with a slope of $-n$. This is sometimes called the power-law flow equation, which is also of wide application, and is obeyed within experimental scatter by the liquids examined in the present report, with $n \approx 0.5$.

The exponent n shows dependence on molecular weight distribution,[12] and approaches an upper limit of unity for a monodisperse linear polymer. For linear unblended polymers it may be expressed empirically: $n = (\overline{M}_n/\overline{M}_w)^{1/5}$. For blended polymers, as the polyisobutenes examined in the present report may be, the exponent will be lower. The admixture in PSB of low-molecular-weight-plasticiser and surfactants seems to have little effect on the exponent.

5 CONCLUSIONS

Parallel-plate plastometry is a suitable method for measuring the viscosity of liquids above 1 kPa s (10^4 poise). It has been applied successfully to plastic propellant binders over a wide strain-rate range (from 10^{-3} to 10 s^{-1}).

For each of the three liquids tested (polyisobutene, polyisobutene plus surfactants, and polyisobutene plus surfactants plus plasticiser) the viscosity at room temperature is, within experimental error, inversely proportional to the square root of the strain-rate at which it is measured.

6 ACKNOWLEDGEMENTS

Mr G J Spickernell and Mr H C Grant are thanked for the samples of liquids and for the falling-sphere viscosity determinations. They and Mr R W Bryant are also thanked for helpful discussions.

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MATERIALS

1 Polyisobutene

The sample of polyisobutene provided was designated Enjay Vistanex LM-CX 468/AC/17A, drum No 1. It is described simply as 'Vistanex' in this report. It had a reported falling-sphere viscosity of 115 kPa s at 25°C, during which measurement the sphere fell a distance equal to one diameter in about 25×10^{3} seconds. It is assumed that the deformation imposed was, within a factor of two, equal to one diameter, and thus that the strain-rate was 2 to 8×10^{-5} s^{-1}.

It had a temperature coefficient of viscosity, defined as Δ (log η)/Δ (1/T K), of 3000. This coefficient enables the falling-sphere viscosity at 21°C (the temperature of the parallel-plate plastometer measurements with which it is compared in Fig 3) to be derived as 157 kPa s, at a strain-rate 157/115 times slower, ie 1.5 to 6×10^{-5} s^{-1}.

2 Plasticised Mixture

The mixture (batch 20) of plasticiser, surfactants and polyisobutene was designated PSB. It contained 80 parts by weight of polyisobutene, 15 pbw of ethyl oleate and 5 pbw of surfactants. It had a reported falling-sphere viscosity of 59 kPa s at 25°C, during which measurement the sphere had fallen a distance equal to one diameter in about 8×10^{3} s. It is assumed as before that the deformation imposed was, within a factor of two, equal to one diameter, and thus that the strain-rate was 0.6 to 2.4×10^{-4} s^{-1}.

It had a temperature coefficient of viscosity of about 2700, from which the falling-sphere viscosity at 21°C (shown in Fig 4) is derived as 78.4 kPa s, at a strain-rate of 4.5 to 18×10^{-5} s^{-1}.

3 Unplasticised Mixture

The sample of mixture of surfactants and polyisobutene was designated USB. It contained 10 parts by weight of polyisobutene grade B10, and 1 pbw of the surfactant mixture known as S101. It had a reported falling-sphere viscosity of 32.8 kPa s at 25°C, during which measurement the sphere had fallen a distance equal to one diameter in about 1.2×10^{3} s. It is assumed as before that the deformation imposed was, within a factor of two, equal to one diameter, and thus that the strain-rate was 4 to 16 × 10^{-4} s^{-1}. It had a temperature coefficient of viscosity of 3200, from which the falling-sphere viscosity at 21°C is derived as 50.8 kPa s, at a strain-rate of 2.6 to 10 × 10^{-4} s^{-1}.

TABLE 1

Vistanex Viscosity Measurements as a Function of Mean Strain-Rate

Load W, kg	Mean Strain-Rate, $\bar{\dot{\gamma}}$, s^{-1}	Viscosity η, kPa s
0.400	0.55×10^{-3}	86.2
0.400	2.4×10^{-3}	29.3
0.600	1.2×10^{-3}	85.7
0.800	1.5×10^{-3}	43.2
1.000	1.5×10^{-3}	75.7
5.000	2.3×10^{-2}	19.0
5.000	1.1×10^{-2}	49.9
5.000	1.4×10^{-2}	9.6
10.000	5.6	2.5
10.000	6.8	1.6

TABLE 2

PSB Viscosity Measurements as a Function of Mean Strain-Rate

Load W, kg	Mean Strain-Rate, $\bar{\dot{\gamma}}$, s^{-1}	Viscosity η, kPa s
0.050	2.5×10^{-4}	24.7
0.100	6.2×10^{-4}	19.9
0.100	6.8×10^{-4}	12.5
0.075	7.6×10^{-4}	10.0
0.200	1.2×10^{-3}	13.7
0.400	3.9×10^{-3}	4.32
0.300	5.8×10^{-3}	6.57
0.600	9.0×10^{-3}	6.49
0.800	1.7×10^{-2}	2.18

TABLE 3

USB Viscosity Measurements as a Function of Mean Strain-Rate

Load W, kg	Mean Strain-Rate, $\bar{\dot{\gamma}}$, s^{-1}	Viscosity η, kPa s
0.050	1.3×10^{-4}	31.6
0.050	5.9×10^{-4}	20.2
0.100	8.6×10^{-4}	8.14
0.100	9.2×10^{-4}	8.29
0.200	2.1×10^{-3}	8.65
0.200	3.6×10^{-3}	2.25
0.400	3.6×10^{-3}	7.82
0.900	6.9×10^{-3}	7.54
0.900	8.0×10^{-3}	10.5
1.400	1.8×10^{-2}	4.92

FIG. 1

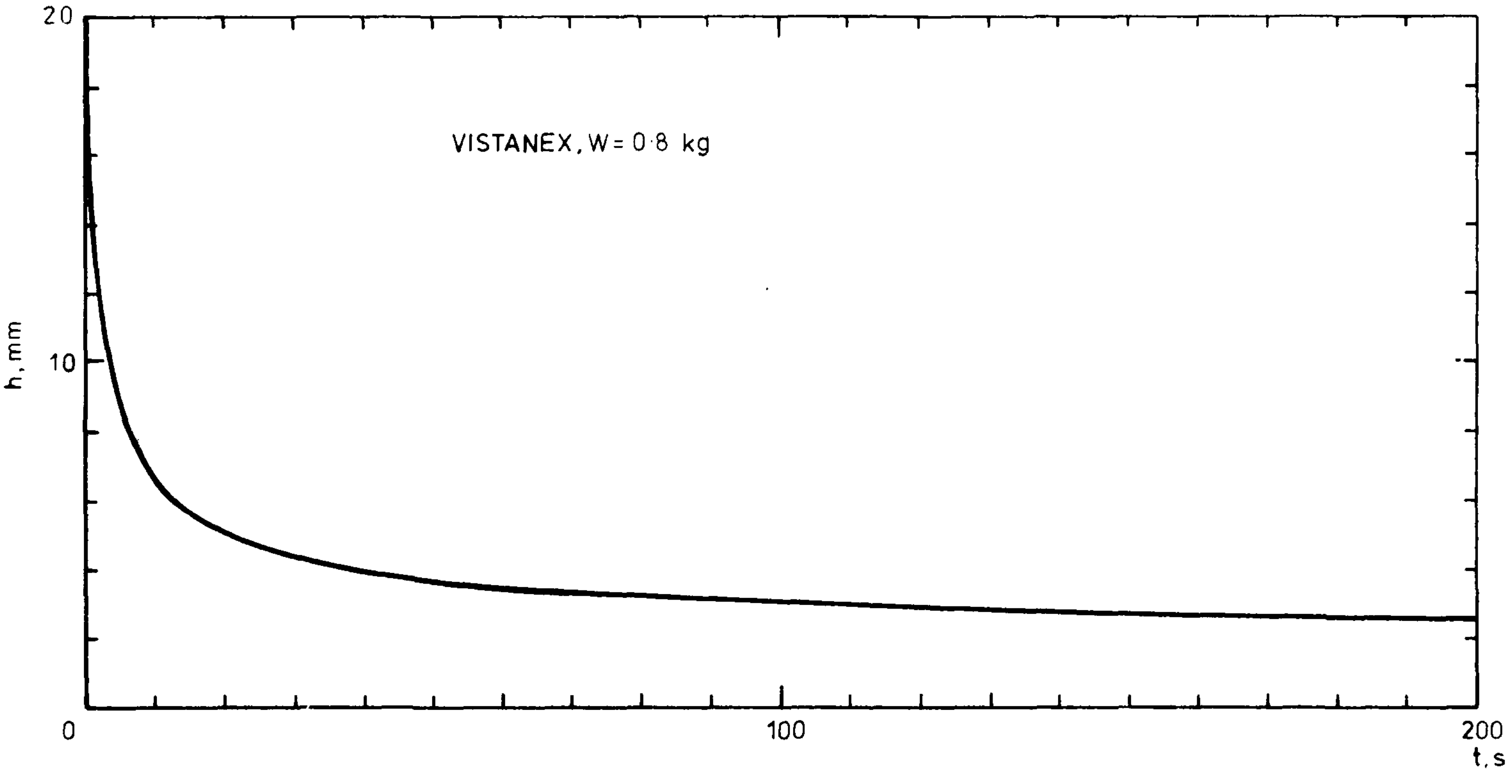

FIG. 1 TYPICAL VARIATION OF SAMPLE HEIGHT DURING COMPRESSION

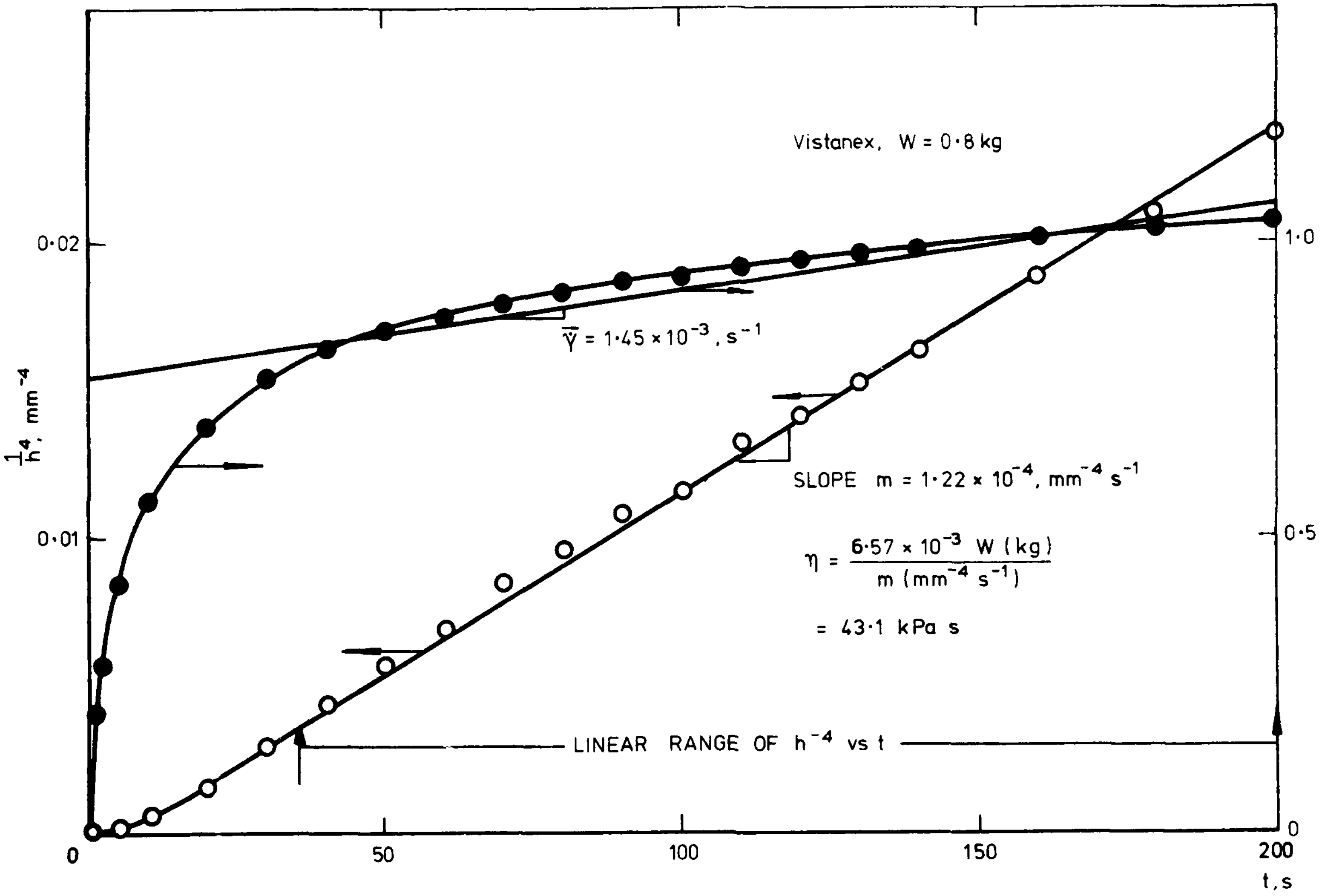

FIG. 2 TYPICAL DERIVATION OF VISCOSITY AND MEAN STRAIN-RATE

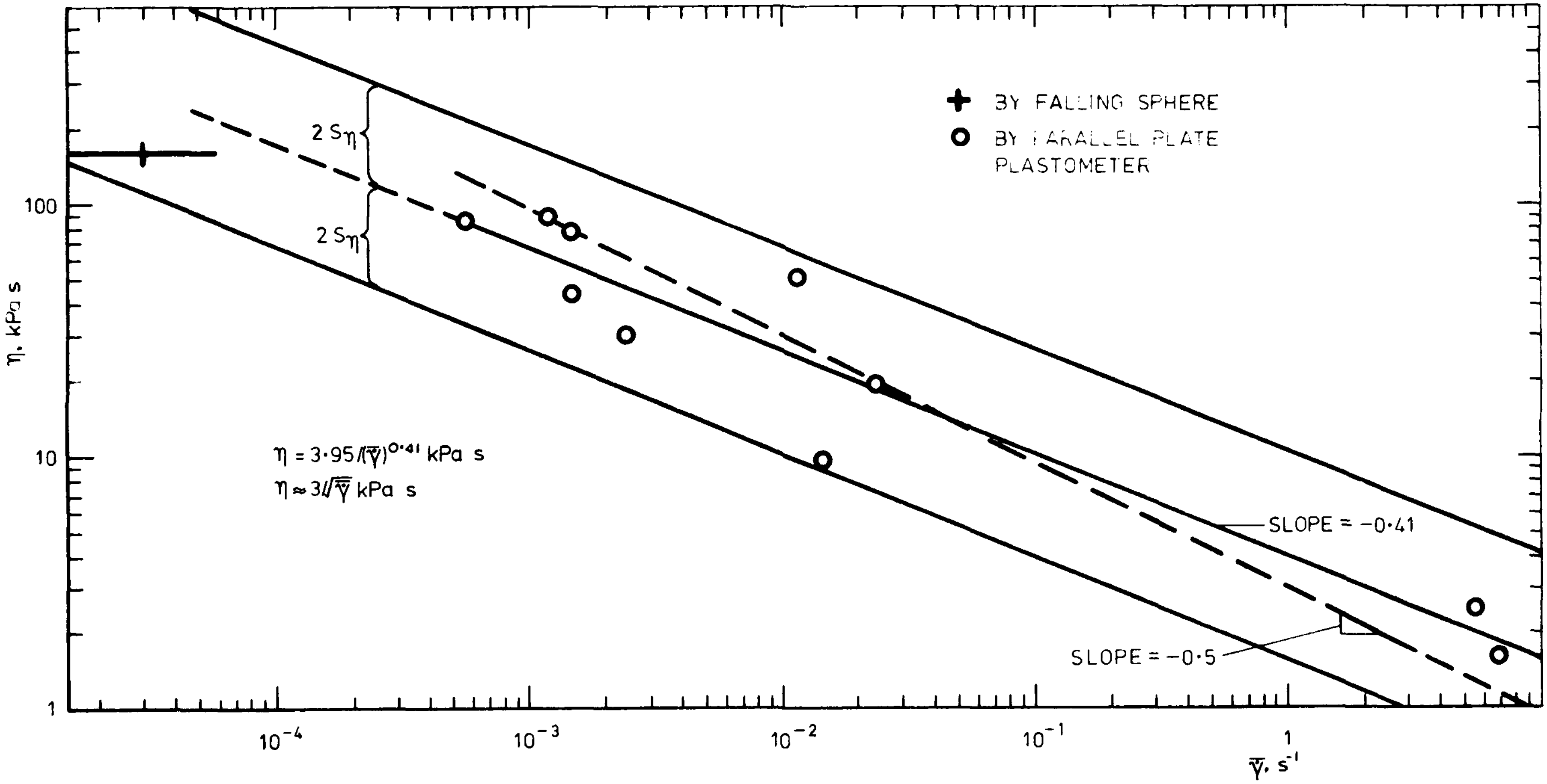

FIG. 3 THE VARIATION OF VISCOSITY WITH STRAIN-RATE OF VISTANEX POLYISOBUTENE

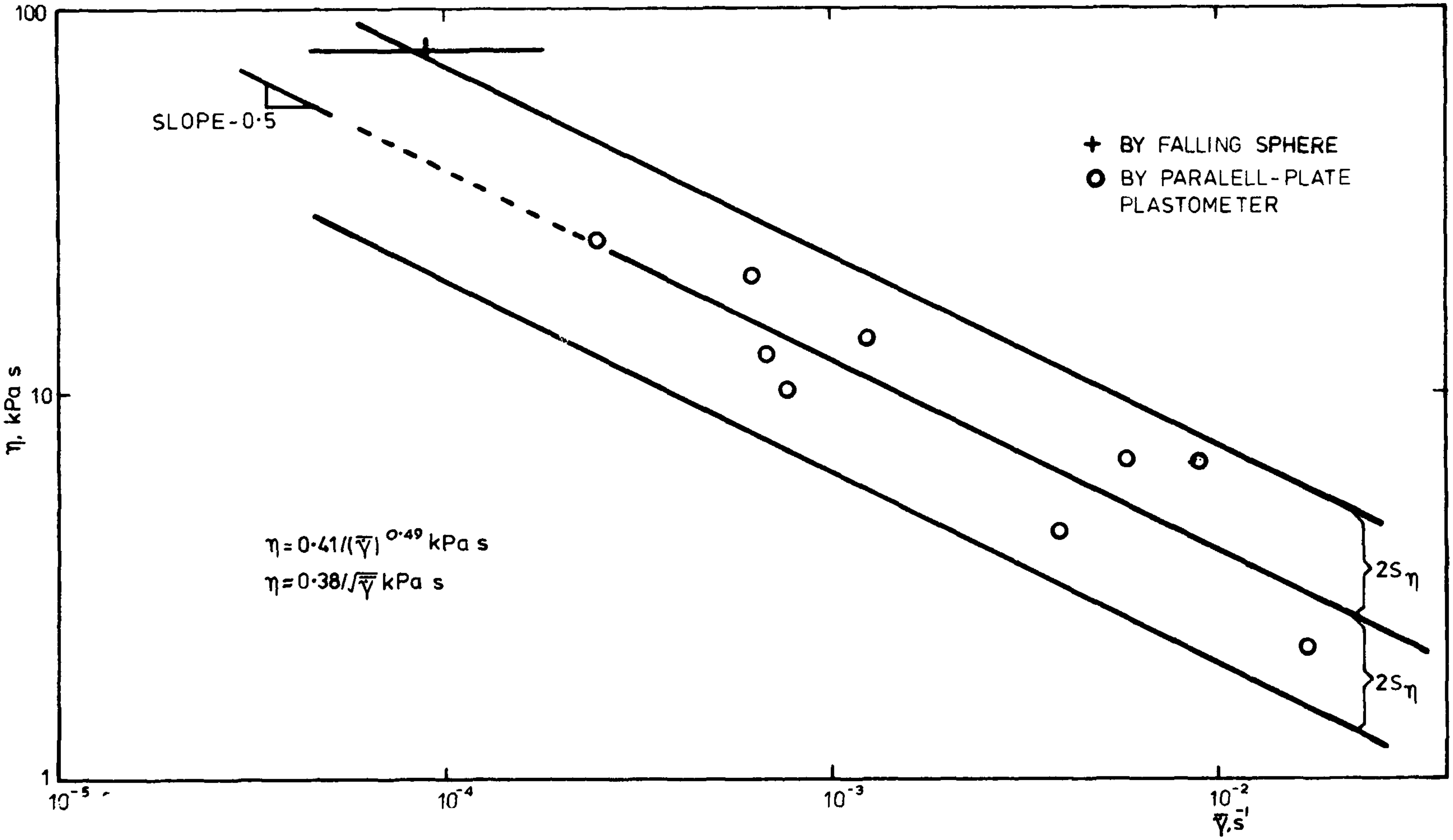

FIG. 4 THE VARIATION OF VISCOSITY WITH STRAIN RATE OF PSB

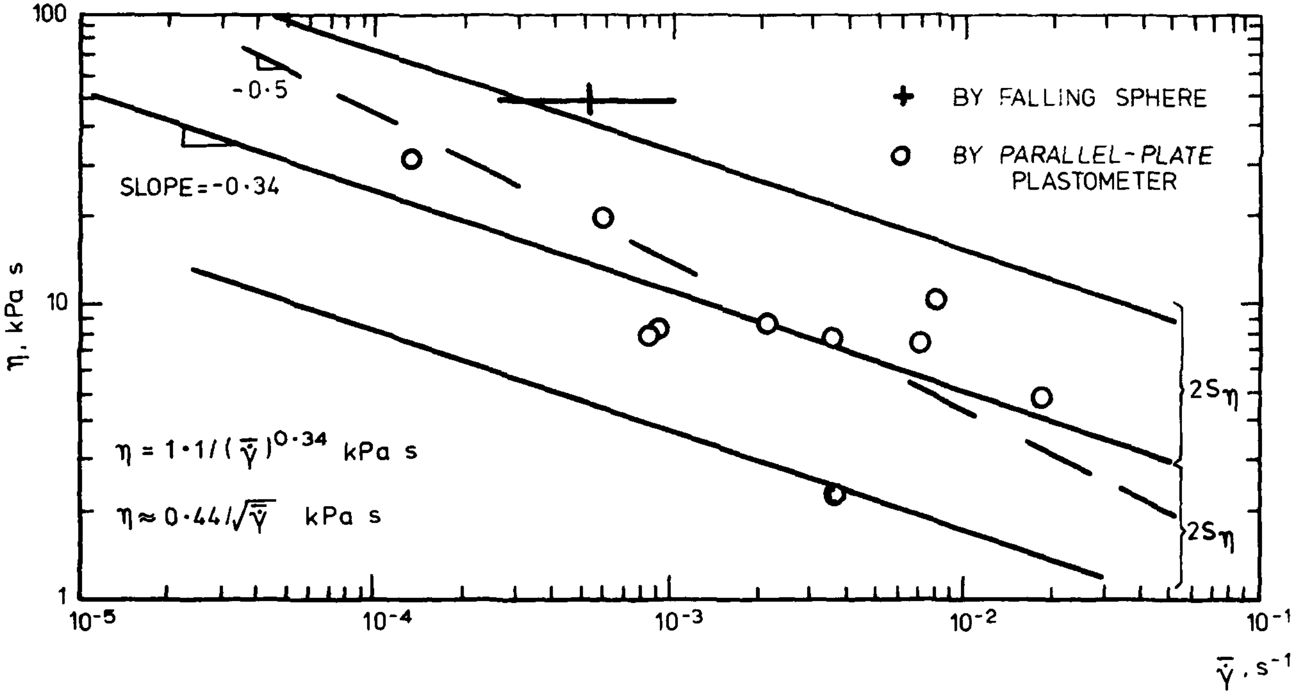

FIG. 5 THE VARIATION OF VISCOSITY WITH STRAIN RATE OF USB

LIST OF SYMBOLS

h	plate separation (mm)
h_o	initial plate separation (mm)
m	slope of linear $1/h^4$ vs t plot (mm^{-4} s^{-1})
n	strain-rate exponent in Eqn 6
$\bar{M}_n$	number-average molecular weight
$\bar{M}_w$	weight-average molecular weight
S_η	Standard error of estimate of viscosity
t	time (s)
V	volume (m^3)
W	load (kg)
γ	radial strain
$\dot{\gamma}$	strain-rate (s^{-1})
$\bar{\dot{\gamma}}$	mean strain-rate (s^{-1})
η	viscosity (kPa s)
η_o	viscosity at $\dot{\gamma} = 0$ (kPa s)
η_∞	viscosity at $\dot{\gamma} = \infty$ (kPa s)
τ	constant in Eqn 6 (s)

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(TEST)

Abstract The viscosities of three liquids used as ingredients of plastic propellant have been measured at room temperature, as a function of strain-rate, by parallel plate plastometry. The liquids were (a) Vistanex LM, a polyisobutene with a falling-sphere viscosity at very low shear-rates of about 100 kPa s (10^6 poise), (b) a mixture of polyisobutene, plasticiser and surfactants known as PSB, used as the binder for many propellant formulations, and (c) an unplasticised mixture of polyisobutene with surfactants, known as USB, similar to another propellant binder.

The strain-rate was varied over a range from 10^{-3} to 10 s^{-1}, and the measured viscosities ranged from 100 to 1 kPa s (10^6 to 10^4 poises).