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## Evaluation of High Shear Viscosity Data from Jet and Concentric Cylinder Viscometers

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With 2 figures and 3 tables

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### Introduction

There is an active interest in the study of fluid flow at high shear. Such investigations give insight into the nature of the liquid state. The results are also significant in practical engineering problems. Unfortunately, the instrumentation for high shear measurements has been slow to develop. This is because of basic problems in apparatus design. Such problems are inherent in the two principal high shear techniques, per se, rotational and capillary viscometers.

One of the earliest and most promising high shear instruments was the so-called jet viscometer developed by *Morris* and *Schnurmann* (1, 2). In principle, the jet instrument is simply a short capillary tube viscometer.

The capillary is thermostatted by a glass jacket and utilizes a variable pressure head to obtain a range of shear rates. The instrument has been used at temperatures and shear rates from 0–100 °C and from 10<sup>3</sup>–10<sup>7</sup> reciprocal seconds (1–9). The jet viscometer is one of the most widely known capillary-type, high shear viscometers.

The second kind of high shear viscometer, the rotational type, has recently reached a state of refinement. One of the most satisfactory models for high shear measurements was developed by *Barber* (10). A number of these viscometers are currently in use in several laboratories.

In the *Barber* viscometer, the test fluid is held by surface tension between concentric

steel cylinders. The inner cylinder is rotated, and the torque transmitted through the test fluid is measured on the outer cylinder. Shear rate is proportional to speed of rotation, and viscosity is a function of torque. Uncertainties in test temperature and shear rate are minimal because of the small clearance between cylinders, below  $1.5 \times 10^{-3}$  inch, and because both cylinders are thermostatted. Results from this type concentric cylinder viscometer have been reported in several publications (10–15). Instrumentally, it covers a viscosity, temperature, and shear rate range similar to the jet viscometer described earlier.

In this laboratory, both types of viscometers have been in use for several years. An opportunity has thus been provided for comparing results from the two viscometric

Absolute viscosities derived from densities and low-shear capillary flow data are shown near the ordinate. They agree with viscosities reported by the API for the same compounds and temperatures. The viscosity of each normal alkane, see fig. 1, is found to be independent of shear, or *Newtonian*, from  $10^2 - > 3 \times 10^5$  seconds<sup>-1</sup>. This conclusion holds for data at a series of temperatures above the melting point of each *n*-alkane.

Concentric cylinder viscometer measurements have also been reported on six low density, low molecular weight polyethylenes (13). For polymers with molecular weight up to about 3500, *Newtonian* flow was observed for shear rates from  $10^2 - 2 \times 10^5$  seconds<sup>-1</sup>, for temperatures from 110–135 °C, and for viscosities to above 4 poises. Small viscosity losses due to shear heating are

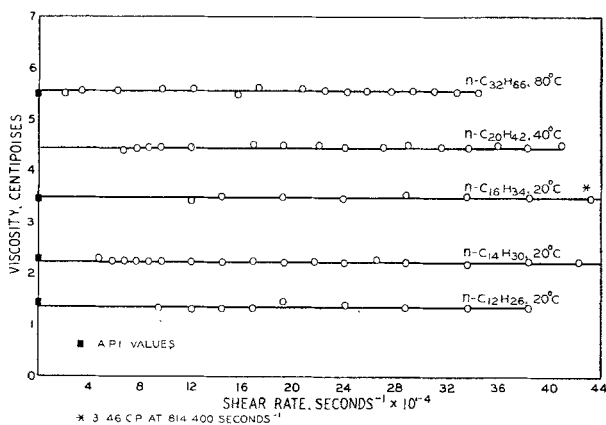


Fig. 1. Rotational viscometer data normal alkanes

techniques. A comparison is useful in ascertaining both the reliability and differences between the two widely different experimental methods. Each method has been extensively used to measure both temporary (1–15) and permanent (4, 6, 11, 14) viscosity losses due to shear. Temporary viscosity changes are due to reversible deformation effects. Permanent viscosity losses are caused by the rupture of large molecules at high shear. The following sections evaluate literature data and new viscosity results derived by the two commonly used high shear viscometers.

## Results

The most effective comparison of high shear viscometers may be made from data on pure compounds. Fig. 1 shows concentric cylinder viscometer data on five normal alkanes from *n*-dodecane to *n*-dotriacontane.

found for cylinder clearances above  $3 \times 10^{-4}$  inch. *Newtonian* results at high shear have also been found for a series of bulk polyisobutenes with molecular weights to several thousand (14). The observation of *Newtonian* flow for these systems is real. Prominent non-*Newtonian* flow has been observed with the concentric cylinder viscometer for other polymeric systems and for higher molecular weight polyethylenes and polyisobutenes at relatively low rates of shear (10–14).

For comparison, jet viscometer data are available on tetradecane, one of the compounds in fig. 1. High shear measurements have been reported at four temperatures from 6.4–35.3 °C (7). These data may be compared in table 1 with results from the concentric cylinder viscometer. The capillary shear rate used, which is of course variable in a capillary viscometer, is the maximum value, that calculated at the wall. The rotational shear

rate is essentially constant across the narrow annulus. The maximum temperature increase at high shear in the concentric cylinder viscometer is near 1 °C (10–15). For tetradecane, this corresponds to a viscosity reduction of about 2%. Since instrumental uncertainty is of similar magnitude, concentric cylinder data are *Newtonian* within experimental error from  $10^2 - > 10^5$  seconds<sup>-1</sup>. In contrast, jet viscometer data show prominent non-*Newtonian* flow for tetradecane. These viscosity losses, it is claimed (3, 6), are not thermal effects as the temperature gradients in the jet capillary are not more than 0.05 °C.

Table 1  
Viscosities of Tetradecane

| Test Temp., °C | Low Shear Viscosity Centipoises <sup>*)</sup> | % Viscosity Loss Due to Shear<br>Shear Rate Seconds <sup>-1</sup> | Rotational Viscometer | Jet Viscometer |
|----------------|---|---|-----------------------|----------------|
| 20.0           | 2.335   | 281,800   | 3.2                   | 23.6           |
| 35.0           | 1.734   | 223,800   | 4.2                   | 24.3**)        |

\*) Values from API Project 44.

\*\*\*) Measured at 35.3 °C.

Viscosity data on castor oil are also available by the two high shear techniques. As originally presented (3), jet viscometer data on castor oil at 100 °C showed a pronounced viscosity minimum with increasing shear rate near  $10^5$  seconds<sup>-1</sup>. These data were subsequently modified to include a large kinetic energy correction. These jet data, as republished, show a large and regular decrease in viscosity with increasing shear (3, 5, 6, 7, 9). For castor oil with a low shear viscosity of 22 centipoises at 100 °C, a temporary viscosity loss of 24.4% is reported at 234,700 reciprocal seconds (5, 6, 7, 9). In contrast are concentric cylinder data on castor oil. For tests at 80 °C and 100 °C on an oil with low shear viscosities of 33.4 and 17.7 centipoises, respectively, a maximum viscosity loss of 3.5% is found at the identical shear rate of 234,700 seconds<sup>-1</sup>.

Differences between the two high shear methods are also apparent from data on petroleum base oils. These comparisons are made from tables 2 and 3. More extensive concentric cylinder data are available on a variety of petroleum fluids (10, 15). The largest viscosity losses previously found in oils at high shear may be due in part to shear heating (15).

Table 2  
Jet Viscometer Data on Petroleum Oils<sup>\*)</sup>

| Petroleum Fluid           | Low Shear Viscosity Centipoises | Test Temp., °C | Shear Rate, Seconds <sup>-1</sup> | Per Cent Viscosity Reduction |
|---------------------------|---------------------------------|----------------|-----------------------------------|------------------------------|
| Light Mineral Oil "A"     | 5.59                            | 13.6           | 134,900                           | 20.6                         |
| Straight Mineral Oil "G"  | 14.4<br>37.7                    | 100.0<br>75.0  | 282,000<br>146,000                | 26.8<br>14.7                 |
| A-30 Transformer Oil      | 21                              | 20.0           | 228,000                           | 18.0                         |
| Medicinal Liquid Paraffin | 28.6                            | 56.1           | 182,300                           | 17.1                         |

\*) Data from Morris and Schnurmann (7).

Table 3  
Rotational Viscometer Data at 38 °C on Petroleum Oils

| Petroleum Fluid | Low Shear Viscosity, Centipoises | Per Cent Viscosity Reduction at $3.00 \times 10^5$ Seconds <sup>-1</sup> |
|-----------------|----------------------------------|--|
| A White Oil     | 13.72                            | 1.6  |
| A Pale Oil      | 19.53                            | 2.7  |
| SAE 10 Base Oil | 36.03                            | 2.3  |
| SAE 20 Base Oil | 73.59                            | 4.7  |
| SAE 30 Base Oil | 120.6                            | 4.9  |

Tables 2 and 3 give data on similar oils over a comparable viscosity and shear rate range. The jet viscometer gives viscosity decreases which are consistently larger than the virtually *Newtonian* results found by the concentric cylinder viscometer. Cylinder clearances which were used varied from  $1-3 \times 10^{-4}$  inch. Certain high molecular weight oils measured in the rotational viscometer show larger non-*Newtonian* effects than those reported in table 3.

The jet shows, however, consistently larger effects than the concentric cylinder and other high shear viscometers (15, 18–20). Data on petroleum fluids thus show the same trend as results reported on the tetradecane and castor oil.

High shear viscometers generally reveal non-*Newtonian* flow in high polymer systems. Fig. 2 shows high shear viscosity data on two methacrylate polymer solutions. The systems are similar to modern multigrade motor oils. Fig. 2 shows non-*Newtonian* flow in a plot of reduced viscosity, i. e., high shear value divided by low shear capillary viscosity versus rate of shear. The point of interest is the viscosity increase found by the jet visco-

meter above  $5 \times 10^4$  seconds<sup>-1</sup>. The jet results in fig. 2 were obtained in this laboratory. An approximate value, not entered in fig. 2, was calculated for the kinetic energy correction. This correction reduces but does not eliminate the minimum in fig. 2. This conclusion is also true for certain previously reported jet data (3, 5). Other factors may contribute to the apparent upturn in viscosity at high shear. A likely cause, particularly for polymer systems, is elastic energy. This contribution has been extensively studied by Gaskins and Philippoff (16, 17). Results indicate that considerable elastic as well as kinetic energy can be dissipated at the capillary exit.

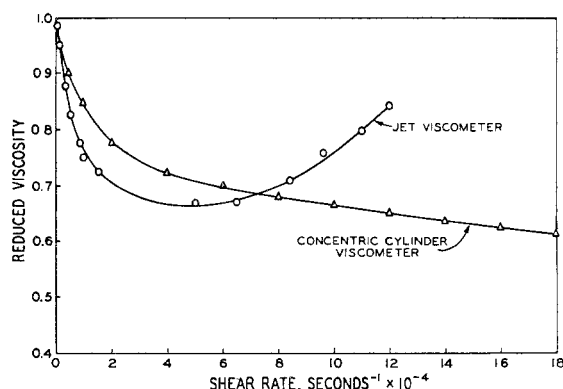


Fig. 2. Methacrylate polymer solutions

Viscosity minima with increasing shear are generally not found by concentric cylinder and other types of rotational instruments (10–15, 18). Viscosity anomalies are only occasionally found in data from capillary viscometers other than of the jet type (17, 19–24). Where they occur, elasticity phenomena are a possible contributing cause (24, 25).

### Discussion

Both the jet and concentric cylinder viscometers operate at shear rates found in machine elements. Therefore, differences observed above for petroleum stocks and for polymer solutions are important in interpreting the performance of modern lubricating oils. An important problem in this regard is the apparent increase in viscosity at high shear found from jet data. Explanations of this anomaly may be offered. In some cases, simply a large kinetic energy correction removes the apparent upturn in viscosity at high shear (3, 6). If an apparent viscosity minimum remains after the kinetic energy

correction, an explanation has been made in terms of *Reynolds'* turbulence (3, 5). The *Reynolds'* numbers involved, however, are generally well below 2000 where turbulence is not expected for either *Newtonian* fluids (26). For example, turbulence, as deduced from an increase in viscosity with an increase in shear, is reported by Schnurmann at *Reynolds'* numbers in the range 0.3–2 (5, 6). Other data do not indicate turbulence at such low *Reynolds'* numbers.

The rotational viscometer operates at low *Reynolds'* numbers. The highest number, about 3.0, is developed for dilute polymer solutions of high molecular weight,  $> 10^6$ . For such solutions, and all other systems tested in the concentric cylinder viscometer, viscosity minima have not been found. In contrast, for comparable systems turbulence is reported in a jet viscometer at *Reynolds'* numbers as low as 0.27 (5, 6).

For the system in fig. 2, the viscosity increase with shear may possibly be explained in terms of molecular relaxation times. The volume of a typical jet capillary is about  $6.3 \times 10^{-6}$  cc. Typical flow times are about fifty seconds for 17 cc. of sample. The residence time in the capillary for a volume element is, therefore, about  $2 \times 10^{-5}$  seconds. This is in the region of relaxation times of polymer molecules in solution (16, 22, 27). Therefore, the point of inflection in fig. 2 may correspond to the shear where molecular orientation is limited by capillary residence time. For stresses imposed for such short duration, the polymer solution will behave elastically rather than viscously. A related effect has been previously called an "elastic" rather than a *Reynolds'* turbulence (28). Several aspects of minima in jet data, including polymer degradation, are in qualitative accord with this conclusion.

The interpretation of viscosity minima by either elastic or *Reynolds'* turbulence cannot explain the apparent increase in jet viscosities reported for mineral oils and for tetradecane (3, 5, 7). This is because mineral oils do not show significant elasticity down to stress times of  $10^{-9}$ – $10^{-11}$  seconds (27). Minima in these cases may be due to inadequate jet corrections. The viscosity variation with pressure is an unlikely cause because of the small and regular magnitude of this variation. Capillary end effects and kinetic energy are possible explanations. In capillaries, kinetic energy causes an apparent increase in viscosity at high shear (21). Moreover, it is difficult to quantitatively

correct this out of jet viscometer data (5, 17), as it is not a true capillary of constant diameter and definite length (1, 2). Other difficulties with this correction have been discussed by *Schnurmann* (5). The essential point is that short capillary viscometers require extensive corrections of several types (17, 29), which tend to make consequent high shear results less certain than comparable concentric cylinder data.

A general problem in high shear viscometry is that of temperature rise within the test fluid. For the instruments discussed here, the temperature uncertainty is purportedly low. The jet viscometer has a gradient of about 0.05 °C (3, 6) and the concentric cylinder viscometer about 1 °C (10, 11). Data comparisons indicate that the jet gives consistently larger temporary viscosity losses than the concentric cylinder viscometer. This is in the direction of larger temperature gradients in the jet. Moreover, per cent viscosity losses in the jet appear to increase with rate of energy input. Excluding the first datum in table 2, the per cent viscosity loss is related to rate of heat generation, i. e., proportional to shear rate times shear stress. It is not clear, however, that difference in temporary viscosity losses may be explained by temperature gradients. Exclusion of other contributions and more complete viscosity-temperature data are needed to ascertain temperature effects in the jet.

*Philippoff* reports that capillary and concentric cylinder data may be superimposed if the shear rate at the capillary wall is used rather than the average capillary-shear rate (21). These are the conditions used here for comparing data. The differences on this basis become larger for viscosity comparisons using the average shear rate in each instrument. It has been pointed out that capillary-shear rates should be corrected when non-Newtonian flow occurs. This correction is predicated on a previous knowledge of viscosity-shear characteristics (16, 17).

It is felt that the concentric cylinder viscometer data are correct because of the (a) uniform, well-defined shear rate; (b) the experimentally determined lack of temperature effects; (c) absence of extensive corrections; e. g., kinetic energy; and (d) measurement times sufficient to rule out relaxation phenomena. The jet viscometer data, on the other hand, are difficult to treat. They typically show too large decreases in viscosity with increasing shear and exhibit viscosity minima.

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### Summary

This work compares and evaluates viscosity data obtained on similar fluids by two widely accepted high shear techniques. Both the jet and concentric cylinder viscometers are useful high shear methods. The major limitation of the jet viscometer is an inability to distinguish quantitatively between energy losses in laminar flow and those due to capillary geometry and experimental conditions. For example, the jet viscometer gives minima in viscosity-shear rate correlations which are difficult to treat. These minima are not found in concentric cylinder viscometer data for the same and similar fluids. The apparent viscosity increase at high shear in the jet may be due to factors other than *Reynold's* turbulence, as previously supposed. This effect may be due to molecular relaxation phenomena in certain cases. The jet viscometer might thus be used to evaluate molecular relaxation and/or other phenomena contributing to this effect.

For a variety of systems, the concentric cylinder viscometer gives significantly smaller temporary viscosity losses due to shear than do the jet viscometer data. These comparisons are made using the maximum jet shear rate at the capillary wall. The differences are, of course, larger if average shear rates are used to compare the data. It is concluded that the jet viscometer results tend to be erroneous. This is possibly due to capillary end effects or problems with kinetic energy corrections.

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## Buchbesprechungen

**Transactions of the Society of Rheology, Vol. V** (Verhandlungen der amerikanischen Gesellschaft für Rheologie, Band 5). Herausgegeben von *E. H. Lee*-Providence/R.I. II, 382 Seiten mit zahlreichen Abbildungen und Tabellen (New York 1961, Interscience Publishers, Inc.). Preis: geb. \$ 10.50.

Der Band enthält die Mehrzahl der auf der 31. Jahrestagung der amerikanischen rheologischen Gesellschaft in Pittsburgh vom 31. 10.–2. 11. 1960 gehaltenen Vorträge.

Die fünf ersten Vorträge stehen im Rahmen eines *Symposiums über die Mechanik der Kontinua*: *M. W. Johnson, jr.* (S. 9–21) berichtet über Variationsprinzip, mit deren Hilfe man stationäre Strömungen nicht-Newtonscher Flüssigkeiten bei vorgegebenen Randbedingungen, sowie die turbulente Strömung Newtonscher Flüssigkeiten beschreiben kann. *J. L. Ericksen* (S. 23–34) formuliert Erhaltungssätze für flüssige Kristalle, die einer verallgemeinerten hydrostatischen Theorie entsprechen. *B. Bernstein* (S. 35–40) diskutiert die Anwendbarkeit von *Nolls* Bestimmtheitsprinzip auf Stoffgleichungen vom sog. „rate“-Typ. *B. D. Coleman* und *W. Noll* (S. 41–46) erweitern die lineare Theorie der Viskoelastizität bei kleinen Scherschwingungen zu einer Theorie zweiter Ordnung und finden einen interessanten Zusammenhang zwischen der Schubspannung und der Normalspannungskomponente in Fließrichtung. *A. J. Ziegenhagen*, *R. B. Bird* und *M. W. Johnson, jr.* (Referat S. 47–49) wenden die obengenannten Variationsmethoden zur Berechnung des nicht-Newtonschen Fließens um eine Kugel an.

Weitere fünf Vorträge stehen im Rahmen eines *Symposiums über die Rheologie der Suspensionen*: *W. F. Ames* und *V. C. Behn* (S. 53–66) wenden Regressionsmethoden zur Analyse des Konzentrationseinflusses auf das Reibungsgesetz strukturviskoser Suspensionen (Abwasser-Faulschlämme) an und kommen so zu einer Verallgemeinerung der *Ostwald-de Waele*-Formel. *A. F. Gabrysh*, *T. Ree*, *H. Eyring* und *N. McKee* (S. 67–84) untersuchen die Fließeigenschaften von wäßrigen Attapulgit-Suspensionen in Abhängigkeit von der Temperatur, der Konzentration und dem  $pH$ -Wert mit einem Rotationsviskosimeter. Je nach den Versuchsbedingungen beobachtet man Rheopexie oder Thixotropie: Nebeneinander sind sowohl strukturaufbauende als auch abbauende Einflüsse vorhanden. *R. H. Haynes* (S. 85–101) gibt eine Übersicht über die rheologischen Eigenschaften von Blut, insbesondere über die Abhängigkeit der Viskosität vom Schergefälle und dem Kapillardurchmesser.

*D. W. Criddle* und *J. Cortes, jr.* (S. 103–111) untersuchen die Viskosität und das Relaxationsverhalten von Schmierfetten in einem weiten Scherbereich. *J. G. Savins*, *S. G. Wallick* und *W. R. Foster* (Referat S. 113–114) schließlich stellen der „Integrationsmethode“ zur Beschreibung der Fließeigenschaften eine „Differentiationsmethode“ gegenüber, die von größerer Allgemeinheit sein soll.

Anschließend folgen 19 Vorträge, die außerhalb der beiden Symposien gehalten wurden: *H. H. Hull* (S. 115–131) versucht, eine Beziehung zwischen der *Gibbsschen* Freien Energie und der Normalspannung in Gradientenrichtung bei ebener Scherung herzustellen [Ref. kann diese Zusammenhänge nicht einsehen]. *A. B. Metzner*, *W. T. Houghton*, *R. A. Sailor* und *J. L. White* (S. 133–147) berechnen die Normalspannungsdifferenz einer *Weissenberg*-Flüssigkeit (d. h.  $P_{22} = P_{33}$ ) aus der Aufweitung eines aus einem kreisförmigen Rohr frei austretenden Strahls und wenden diese Methode auf verschiedene hochpolymere Lösungen an. *W. Philippoff* (S. 149–162) zeigt mit Hilfe einer neuen Anordnung zur Messung der Strömungsdoppelbrechung, daß hochpolymere Lösungen *Weissenberg*-Flüssigkeiten darstellen, daß hingegen in Suspensionen von starren Teilchen alle drei Normalspannungskomponenten verschieden groß ausfallen. In einer weiteren Untersuchung formuliert *W. Philippoff* (S. 163–191) die Beziehungen zwischen den elastischen Spannungen und der Strömungsdoppelbrechung insbesondere für *Weissenberg*-Flüssigkeiten. *F. D. Deater*, *J. C. Miller* und *W. Philippoff* (S. 193–204) berichten über Strömungsdoppelbrechungsmessungen an Polyäthylenschmelzen und berechnen daraus den reversiblen Dehnungsanteil (recoverable strain). Vergleiche mit Messungen im Rotations- und Kapillarviskosimeter zeigen gute Übereinstimmung. *J. G. Brodnyan* und *E. L. Kelley* (S. 205–220) bestimmen Fließkurven von konzentrierten Polyacrylsäure-Lösungen bei verschiedenen Konzentrationen und Neutralisationsgraden in einem weiten Scherbereich, der sich in vielen Fällen sogar bis in den oberen *Newtonschen* Bereich hinein erstreckt. Außer bezüglich der Relaxationszeit scheinen sich Polyelektrolyte nicht wesentlich von anderen Hochpolymeren zu unterscheiden. *W. H. Fischer*, *W. H. Bauer* und *S. E. Wiberley* (S. 221–235) untersuchen die Fließeigenschaften von Carboxypolymethylen-Wasser-Systemen und finden dabei eine scharfe, sich nach dem Fließen kurzzeitig wieder einstellende Fließgrenze, ausgeprägte Strukturviskosität und höchstens geringe Thixotropie. *E. W. Merrill*, *H. S. Mickleley*, *A. Ram* und *G. Perkinson* (S. 237–246) vergleichen die