Classification   Products and technology

1) Title   Vibration Rheometer RV-10000

2) Subtitle   Viscosity of various liquids measured by a new method rheometer

3) Keywords   Tuning-fork vibration rheometer, static viscosity

4-1)   Introduction

What follows explains the measurement principles, product specifications and measurement case studies for the world's only tuning-fork vibration rheometer.

The history of measurement of viscosity is quite long, and to date has focused on cup-type, capillary-type, rotational-type or falling-ball- type. For example, for the control of engine oils, a cone-shaped container called a Ford cup is used with a cup-type viscometer. This method of measuring viscosity involved pooling oil in the cone-shaped Ford cup and measuring the rate at which it passed through a narrow hole at the bottom of the cone.

Each of these different types of viscosity measurement has been used to measure specific types of liquid in specific kinds of industry, forming some of the historical background to viscosity measurement. In relation to oils used in the automobile industry, for example, the standards of the American Society for Testing and Materials (ASTM) are generally adopted. However, all of these earlier viscosity measurement methods are generally lacking in versatility, and it has been difficult to measure various types of liquids quantitatively over a wide range of viscosity. This is also obvious from the fact that many viscometers have been standardized not as measurement devices but as testing equipment limited in application to the particular object being tested.

Now a tuning-fork vibration rheometer has been developed through use of a high sensitivity tuning-fork vibration viscometer, which moves two tuning-fork-type oscillators in the middle of the fluid. Using this rheometer, the user is now able to measure liquids with a low viscosity around the level of water to liquids with viscosity as high as a thick honey (around 10000 mPa・s) in quick succession. At the same time, by changing the amount of displacement of the tuning-fork oscillators it is possible to measure viscosity using shear rate as a parameter. By using this tuning-fork vibration rheometer it is now possible to easily measure fluids with strong non-Newtonian characteristics, such as dilatants, Bingham plastic and thixotropic fluids.

With the high sensitivity tuning-fork method, as the energy needed to detect viscosity is extremely low any change in the composition of the fluid is also kept to a minimum when measuring. In the following sections will be reported the principles behind the tuning-fork vibration rheometer, some measurement experiments conducted, and a new physical quantity measured by vibration method: static viscosity.

4-2)   Background to development of the vibration viscometer

The book that has become the bible in the viscosity-related world, "Viscosity (Revised)" was published in November 1958. It was written by Michio Kawata, who at the time was working in the measurement research laboratory of Japan's Agency of Industrial Science and Technology. Sadly, this book is not being

published at present, but in Chapter 6 the author details many theories and potential characteristics of vibration viscometers.

The book details a viscometer device with a vibrating reed which can measure fluids across a wide range of just a few cP to tens of thousands of cP; has a measurement accuracy of 3 to 5%; can perform continuous measurements; is able to measure samples of just 2 to 3cm³, much smaller comparatively to other viscometers available; can measure non-transparent fluids; can obtain viscosity x density figures; and is subject to the influence of elasticity in the case of viscoelastic fluids; with all of these characteristics being principles of the tuning-fork vibration rheometer.

Other than this book, in a supplement to the January 1957 edition of the in-house publication of the Japanese Society of Polymer Sciences "Polymers" there is an article called "Experimental Production of a Vibration Viscometer" by Eiichi Fukada. In this publication, measurement principles, relationship between the wall and the oscillator plate, and actual experimental data such as the gelatinization of polyvinyl chloride, etc., are presented.

These vibration viscometers did not use two oscillators as a tuning-fork, but they show that more than 50 years ago the theory behind the vibration viscometer had been confirmed and development was able to advance to almost commercial viability. When there is only one oscillator being used, the reactive force relative to the drive force needed to power the back-and-forth motion of the oscillator is generated in the axial section supporting the oscillator and interferes with the driving system. Particularly in the low viscosity range when the drive force is low, the effect as a damper of the fluid being measured itself is quite small and there are latent problems such as variability in measurements or not being

able to increase the sensitivity. Therefore, the tuning-fork viscometer using two oscillators with zero reactive force was developed to solve this problem.

5) Measurement principle of the tuning-fork vibration viscometer and static viscosity

With the tuning-fork vibration viscometer, two oscillators are resonated horizontally in a similar fashion to the tuning-forks, and the vibration energy needed to keep the amplitude of the oscillators fixed is compensated by electromagnetic force. In other words, drive force equivalent to the viscosity resistance of the liquid is exerted, and as that drive force is proportional to the viscosity resistance the viscosity value is actually being sought. Also, with vibration viscometers, in principle viscosity x density is being sought as viscosity resistance. This viscosity x density is expressed as "static viscosity"[*1] and is distinguished from dynamic viscosity and kinematic viscosity.

With the tuning-fork vibration model, by calculating the drive force necessary for the oscillators to resonate, as well as the motion equation involving the inertia term, viscosity term and elasticity term, one can understand how the drive force power is proportional to viscosity x density.

To explain the theoretical model behind the vibration viscometer summarily, in the device in Diagram 1 above, if the oscillators are vibrating at a frequency of f, the mechanical impedance (R_z) that the oscillators receive from the fluid is:

$$R_z = A\sqrt{\pi f \eta \rho}$$

with f representing the vibration frequency (Hz), A representing the surface area of both sides of the oscillator plate, η representing the viscosity of the fluid and ρ its density. Here, if we define the force by which the

electromagnetic drive section gives the fixed vibration velocity $Ve^{i\omega t}$ to the oscillator plate as F, we get the following equation:

$$R_z = \frac{F}{Ve^{i\omega t}} = A\sqrt{\pi f \eta \rho}$$

From this graph we can understand how the force induced by the electromagnetic drive is proportional to static viscosity (the product of viscosity η x density ρ)*1.

In actual measurements, the torque generated in the electromagnetic drive section is controlled so as to maintain the fixed vibration amplitude of the oscillators while in the sample fluid. The tuning-fork vibration viscometer draws on the fact that the electrical current required to control that torque is proportional to (viscosity x density), that is, static viscosity.

Photograph 1   Product exterior

6) Development of a tuning-fork typerheometer and actual measurement examples

In applying the technology of the tuning-fork vibration viscometer to the development of the rheometer (RV-10000), while holding the maintenance of high sensitivity as the most important criteria to ensure in the new product, the static viscosity value was sought not by changing the natural vibration frequency, which forms the structural resonance

point of the device, but by incrementally changing the vibration amplitude of the oscillators to adjust the shear rate and then calculating the shear stress from the surface of the oscillators and the torque used to drive them at each shear rate.

Also, as the unit system for static viscosity is not yet determined at the time of writing this report, we nondimensionalize values by assuming the sample's density to be 1.00 g/cm³, equivalent to water, and express the viscosity by mPa・s. In Photograph 1 you can see the exterior of the product. The framework is the same as the viscometer. Also, at present, the shear rate range measurable by the RV-10000 is, when based upon water and JS2000, 0.2 to 1.2 mm by the amplitude of the oscillators, and 10 to 2000 s⁻¹ by shear rate conversion. Because the oscillators on the RV-10000 experience repeated sine-wave vibrations like conventional vibration viscometers do, unlike rotational viscometers, a constant shear rate is not maintained. It is for that reason that the constantly varying shear rate is converted and expressed as an effective value. In other words, one must recognize that the shear rate varies with time. The relationship between shear rate and the measurable range of viscosity for the RV-10000 is shown in Diagram 2.
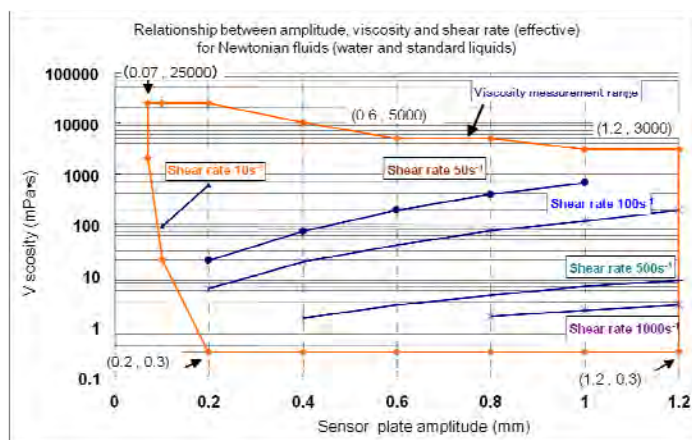

Diagram 2. Relationship between amplitude, viscosity, and shear velocity for Newtonian fluids

6-1)　Measuring Newtonian fluids

Below is explained the results of measurements of water (ion-exchanged) and JS2000 (a viscosity standard). Water is scientifically speaking a chemically stable substance which can be used as a standard for viscosity measurements, and is defined to have a viscosity of 1.00 mPa・s at 20°C temperature. While temperature variation is relatively small, at around room temperature there is a decrease of about 2% in viscosity for every 1°C temperature increase, demonstrating the necessity of paying attention to temperature management. In Diagrams 3 and 4, under conditions of a constant 25°C temperature, viscosity values are plotted for water and JS2000, changing the oscillation amplitude from 0.2 (0.07) to 1.2 mm by peak to peak (from highest to lowest point). Diagram 3 shows the proportionate relationship between amplitude (shear rate) and viscosity (shear stress) for Newtonian fluids. The JS2000, being a standard fluid for viscosity measurement, also demonstrates good Newtonian properties.
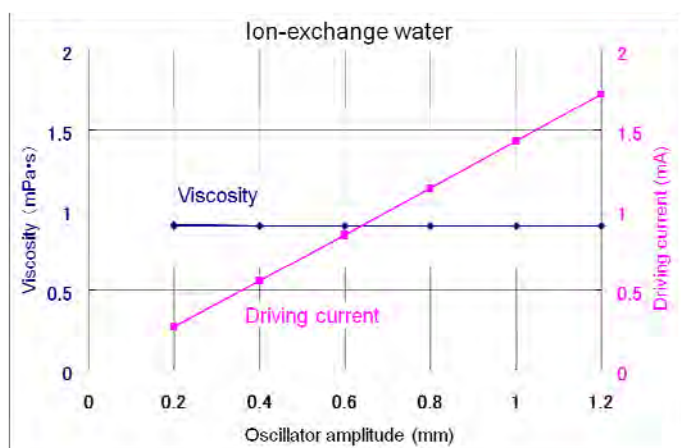
**Diagram 3. Ion exchange water: Amplitude-Viscosity, Driving current**
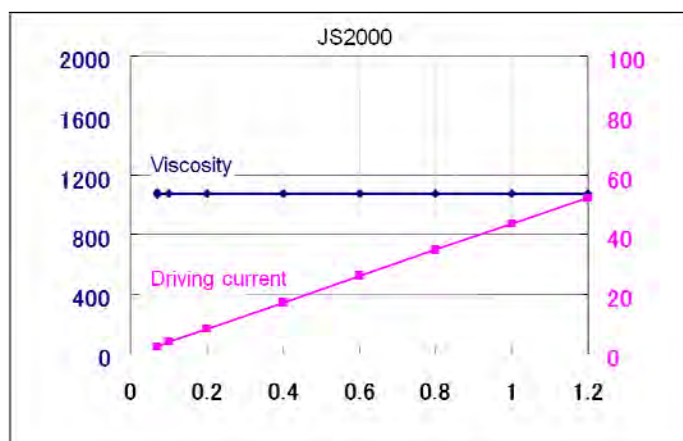
Diagram 4. Standard fluid JS2000: Amplitude-Viscosity, Driving current

6-2)　Bingham fluid (skin moisturizing cream)

The results of measurement experiments conducted on moisturizing cream will now be explained. Diagram 5 shows the results of altering the amplitude of the oscillators by taking measurements at 0.07 / 0.10 / 0.20 / 0.4 / 0.6 / 0.8 / 1.0 / 1.2 mm intervals, approximately Δ0.2 mm each time, starting at the minimum amplitude and increasing to the maximum amplitude, before returning to the minimum again.

The x-axis in Diagram 5 represents time, the y-axis on the left represents viscosity, and the y-axis on the right represents the drive force (current) on the oscillator. Diagram 6 uses the same the data as Diagram 5, but shows the amplitude of the oscillators on the x-axis, while the y-axes show viscosity and the drive current on the oscillators. The Bingham fluid characteristic of viscosity dropping dramatically as the amplitude of the oscillators exceeds a certain point can clearly be seen. Further, the thixotropic nature of the moisturizing cream can be confirmed, as even if the shear rate is reduced the viscosity does not return to its level at the start of the experiment. If the viscosity of a moisturizing cream did not drop after squeezing some into one's

hands and rubbing it between them, the cream would have poor stretch and would be hard to apply. Also, after applying it to one's face, the viscosity rising again ensures there are no concerns with it dripping off. Therefore, this product could be thought of as designed to hold these special properties of Bingham fluids. Diagram 7 shows the relationship between the amplitude of the oscillators and viscosity for moisturizing cream in response to a change in temperature. It also demonstrates viscosity's dependence on temperature and the fact that viscosity's tendency to increase under a low shear rate does not change even when temperature changes.
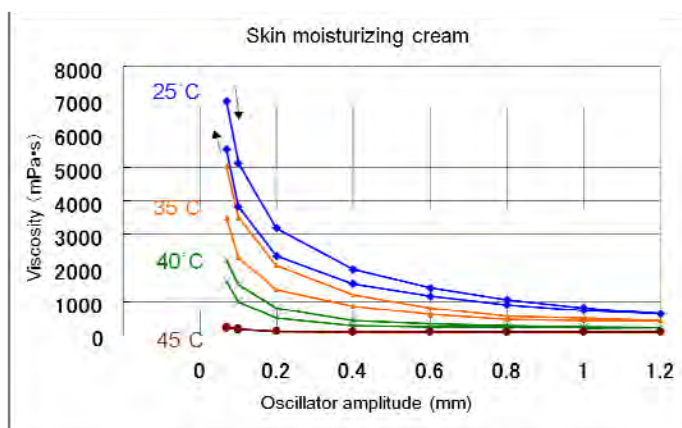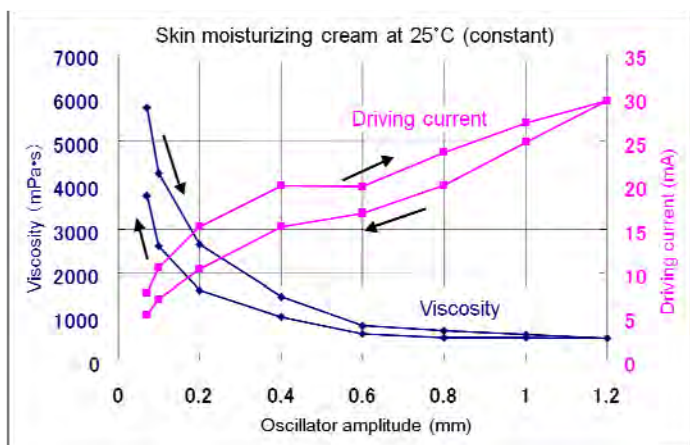

Diagram 7. Moisturizing cream: Amplitude-Viscosity relative to Temperature change

6-3) Dilatant fluid (corn starch watersolution: corn starch 62% + water 38%)

Graphs in Diagrams 8, 9 and 10 show the results of measurements of a cornstarch and water mixture. In such a mixture, when a spoon is rapidly stirred in it there is a strong resistance, yet if the spoon is stirred gently hardly any resistance can be felt at all. In other words, one can see the phenomenon of the viscosity rapidly increasing in response to an increase in shear rate. When this cornstarch mixture was measured, it was found that the viscosity jumped dramatically when the amplitude of the oscillators exceeded 0.8 mm.
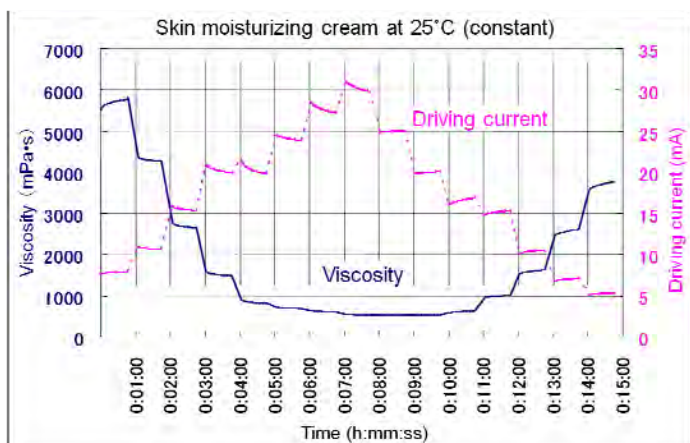
**Diagram 5. Moisturizing cream: Time-Viscosity, Driving current**

Diagram 8 is a similar graph to Diagram 5, showing the viscosity and drive current of the oscillators at a series of different amplitudes. Diagram 9 shows the oscillators' amplitude on the x-axis and viscosity on the y-axis. At the point of the oscillators' amplitude exceeding 0.8 mm, the viscosity with a value previously under 100 mPa・s can be seen to suddenly jump to around 2000 mPa・s.

In Diagram 10, a graph is displayed for a similar experiment conducted while slightly increasing the proportion of water in the mixture. In the new mixture with just 60% cornstarch, the sudden

**Diagram 6. Moisturizing cream: Amplitude-Viscosity, Driving current**

increase in viscosity due to an increase in shear rate cannot be seen. It was probably the first time that the quick elastic response of dilatant fluid was measured by the physical quantity of viscosity. In these experiments, the slight change to the blend ratio of just 2% producing the completely different result of no rapid increase in viscosity could be considered an extremely fascinating result.
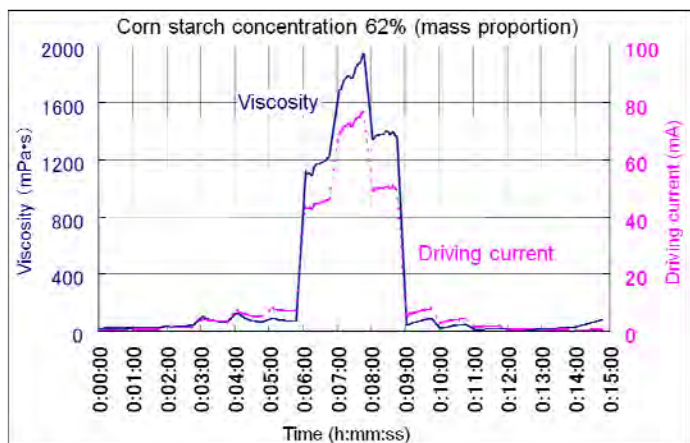
**Diagram 8. Corn starch
(Concentration 62%): Time-Viscosity**
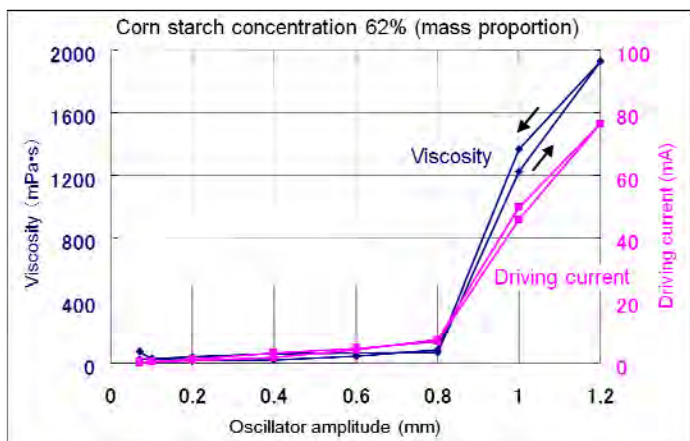
**Diagram 9. Corn starch
(Concentration 62%): Time-Viscosity**
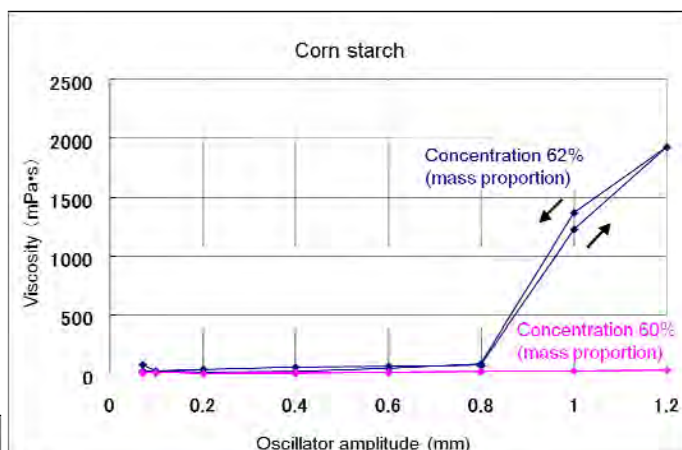
**Diagram 10. Cornstarch: Amplitude-Viscosity
at different concentration**

7) Conclusion and future challenges

Sale of the new RV-10000 rheometer using a tuning-fork vibration method has begun. By applying some of the technology used in A&D's earlier tuning-fork vibration viscometer with a proven sales record, and by altering the amplitude of the oscillators, the shear rate has been made adjustable. By experimenting with several sample fluids with characteristic properties, it was confirmed that by this way of changing shear rate, and accordingly using it as a parameter for measurement, sudden changes in the viscosity or behavior of non-Newtonian fluids could be measured.

In the future, A&D would like to develop an improved product and quantify the characteristics of various low-viscosity fluids, which can be made possible by use of the tuning-fork vibration rheometer, thereby increasing the range of applications where the rheometer can be used.

<References>
*1 『Static viscosity (sv) and vibration-type viscometers』 Society of Instrument and Control Engineers (Incorporated Assoc.), Measurement Division, 24th Sensing Forum

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