Short Guide to the Tuning Fork Vibro Rheometer: RV-10000A

"How you will become able to measure what once appeared impossible to measure"

Laboratory Project
A&D Company, Limited
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Contents

Introduction

Thank you very much for your interest in the RV-10000A!

The tuning fork vibro method is A&D's own technology. Since the release of the original SV (and then SV-A) viscometers, it has contributed to thousands of researchers in realizing kinds of measurements that they once thought impossible. Recently, A&D developed the RV-10000A tuning fork vibro "rheometer" by further evolving these SV/SV-A viscometers. With the added capability to vary the shear rate, it can now closely characterize non-Newtonian fluids in addition to Newtonian fluids.

Yet, as much as the RV-10000A brings about a number of unique advantages, it may be a little difficult to readily absorb the way the instrument works, especially if you are well accustomed to the conventional, rotational types of viscometers/rheometers. In this guide we are therefore complementing what is explained in the brochure and also introducing the sorts of applications the RV-10000A can really exert its strengths, using actual data and/or solutions as examples.

For your better understanding of the tuning fork vibro method, please also refer to the Users' Handbook for the SV/SV-A series available separately.

Understanding The Tuning Fork Vibro Method

The measurement system consists of two oscillating sensor plates driven in opposing directions by electromagnetic force, as shown in Figure 1.

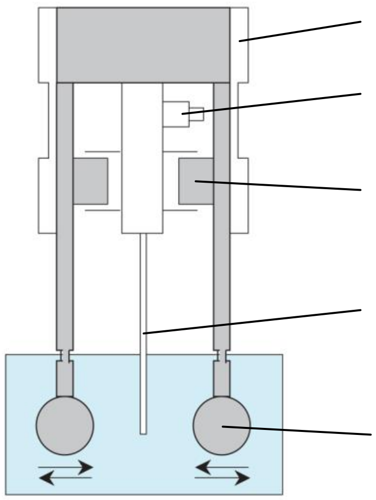


Figure 1. Two sensor plates in a tuning fork arrangement

These two plates are vibrated at their natural (resonant) frequency of 30 Hz within the sample fluid. Further, the electromagnetic driving force is controlled so as to maintain uniform vibration amplitude (displacement).

The more viscous the sample fluid (i.e. the higher the resistance that the plates receive), the greater the driving force required to keep the displacement constant. The viscosity of the sample fluid is calculated based on the amount of electric current the device uses to generate this driving force.

This technique is an application of the electromagnetic equilibrium mechanism employed by high-precision weighing instruments that A&D is renowned for, including microbalances whose scale interval is as small as 1 μg (1/1,000,000 of 1 g).

In principle, the value produced by the tuning fork vibro method as viscous resistance is the product of the "viscosity and density" of the sample fluid (see Note on P.12 for details). However, for sake of simplicity, it is displayed in the [mPa.s] unit of measure, assuming that the density of the sample fluid is 1 g/cm^3.

Understanding Shear Rate In The Tuning Fork Vibro Method

The excursion distance of the sensor plates varies with their vibration amplitude (displacement). When the frequency is fixed, this will result in differences in velocity, effectively varying the shear rate. With the RV-10000A, the amplitude can be altered between the minimum of 0.07 mm (lowest velocity) and the maximum of 1.2 mm (highest velocity) by peak to peak.

In theory, it is possible to make the shear rate variable also by changing the frequency instead of amplitude. However, the sensitivity of the tuning fork vibro sensor increases with sharp resonance phenomena, and changing its frequency from 30 Hz, the resonance frequency of the tuning fork, would invite a reduction of sensitivity. Therefore, the amplitude was made adjustable, rather than the frequency, for the RV-10000A.

Shear rate as an effective value

The classical configuration of two parallel plates with shear rate and shear stress are shown in Figure 2. With this arrangement, shear rate (gradient) is defined and is an independent variable.

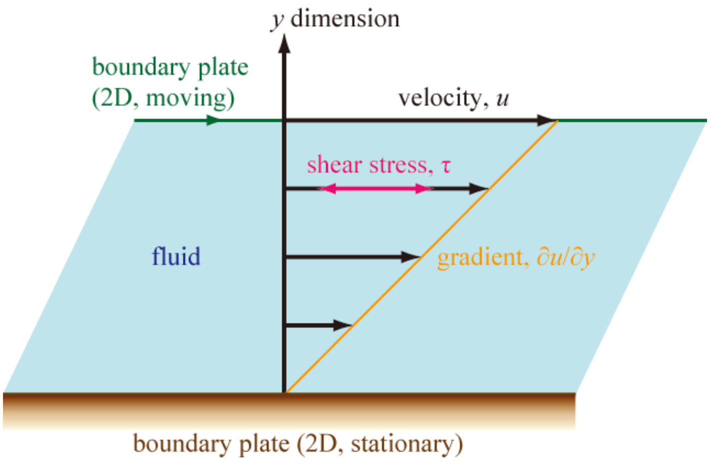


Figure 2. Classic two-plate model illustrating the shear rate and shear stress

Meanwhile, the back-and-forth motion of the tuning fork is a sine wave, with peaks and troughs so that the actual velocity (u) of the moving sensor plates constantly changes. Therefore, the displacement per unit time is expressed by the root mean square (RMS) over one cycle, as shown in Figure 3.

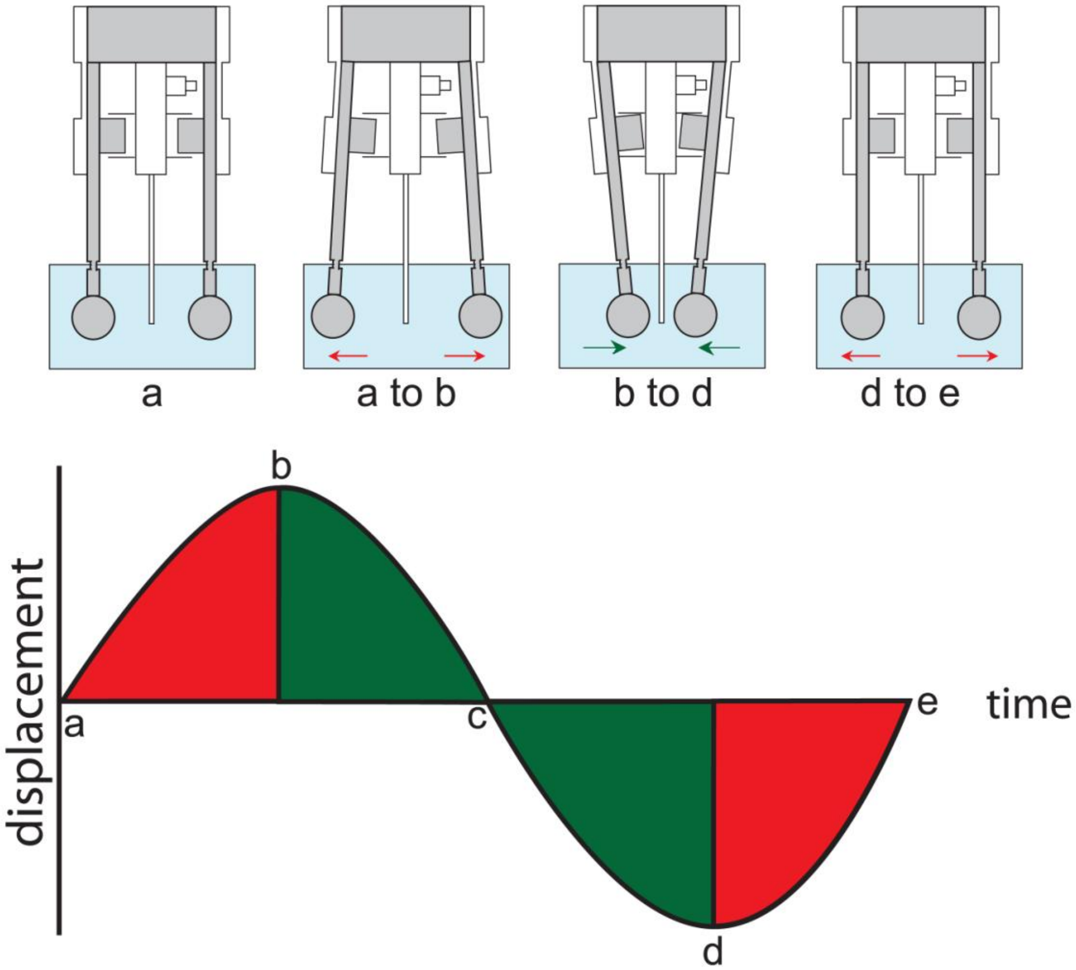


Figure 3 The movement of the tuning fork varies as a sine wave with the rate characterized by the RMS value

Further, in the tuning fork vibro method there is no precise opposite surface to geometrically define shear rate. The higher the viscosity of the sample fluid, the further the shear wave caused by the vibration propagates, making the shear rate lower as a result, as illustrated in Figure 4.

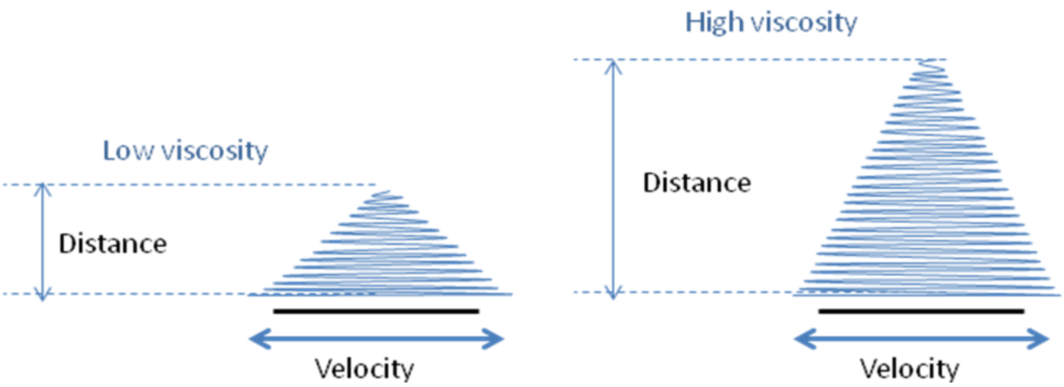


Figure 4. For a given velocity, the more viscous the sample fluid, the lower the shear rate

How the RV-10000A calculates shear rate

With the RV-10000A, shear stress (τ) can be determined by measuring the driving force required to keep the tuning fork in oscillation while the wetted area of the sensor plates is a known value. Shear rate can then be obtained by assigning this shear stress and the measured viscosity value to Newton's law of viscosity (Figure 5).

$$\tau\ [Pa] = \eta\ [Pa.s] \times u\ [S^{-1}]$$

Figure 5. Relationship between shear stress (τ), viscosity (η) and shear rate (u)

Figure 6 shows the shear rates calculated when Newtonian fluids of known viscosities (water and viscosity standards) were measured at different vibration amplitudes.

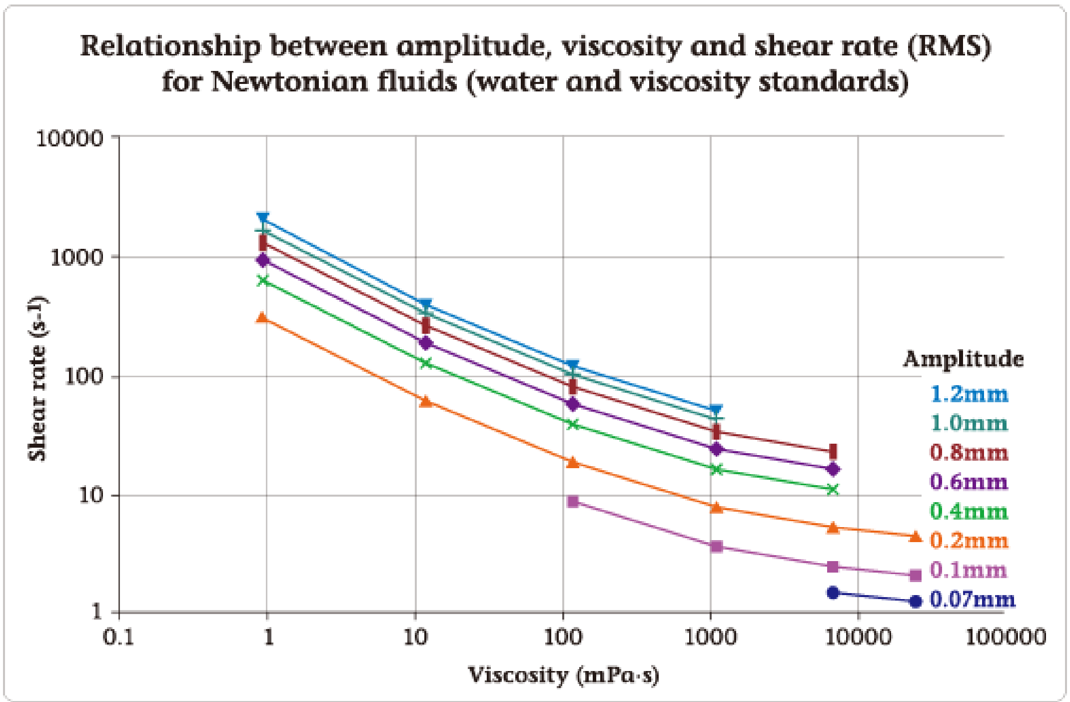


Figure 6. Relationship between amplitude, viscosity and shear rate

In another respect, it can be said that, unlike the rotational method that first determines the distance between two plates to calculate shear rate and viscosity, measurement by the tuning fork vibro method is, as a consequence, based on the actual extent of shear, which is known to vary with the viscosity of the sample fluid.

Measurement range of the RV-10000A

The measurement range is bounded as shown in Figure 7 and Figure 8. Low viscous resistances are difficult to detect with vibration amplitude smaller than 0.2 mm. On the other hand, the larger the vibration amplitude the lower the maximum measurable viscosity as it becomes harder to provide sufficient driving force to maintain the amplitude.

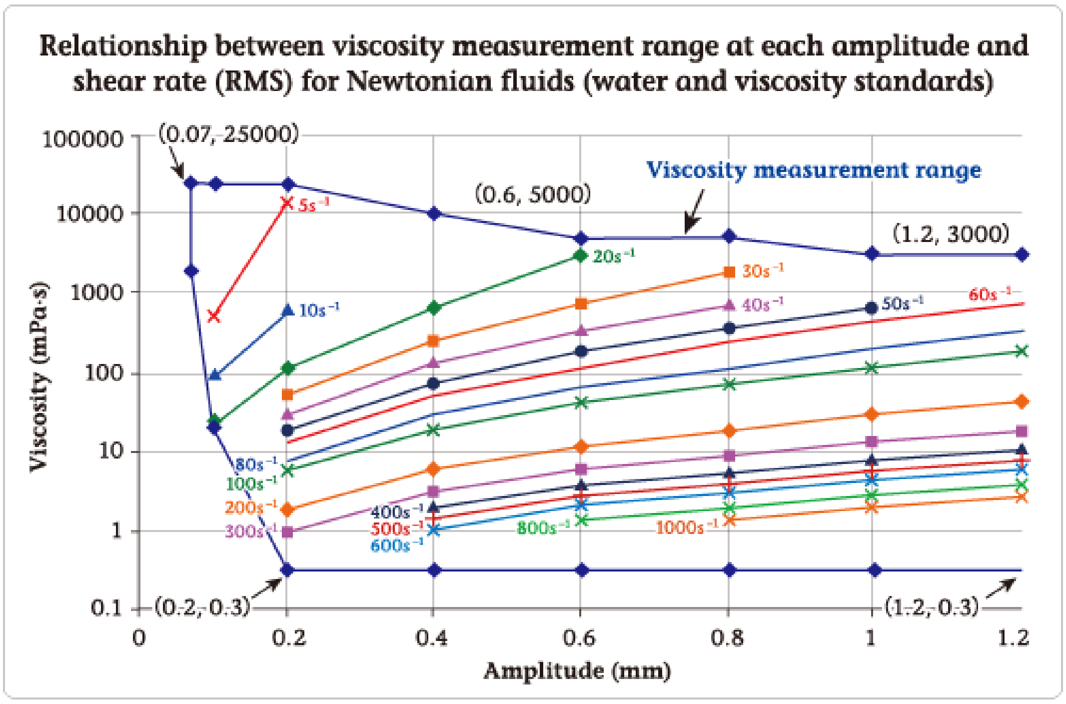


Figure 7. Viscosity measurement range at each amplitude with shear rates (RMS) for Newtonian fluids (water and viscosity standards)

Measurable viscosity range	0.07 mm ≤ amplitude < 0.1 mm	2,000 to 25,000 mPa･s
	0.1 mm ≤ amplitude < 0.2 mm	20 to 25,000 mPa･s
	Amplitude = 0.2 mm	0.3 to 25,000 mPa･s
	0.2 mm < amplitude ≤ 0.4 mm	0.3 to 12,000 mPa･s
	0.4 mm < amplitude ≤ 0.8 mm	0.3 to 5,000 mPa･s
	0.8 mm < amplitude ≤ 1.2 mm	0.3 to 3,000 mPa･s

Figure 8. Measurable viscosity range for each amplitude setting

Strengths Of The Tuning Fork Vibro Rheometer

If you are expecting that a new device should produce exactly the same values for any measurement sample as rotational types, the RV-10000A probably isn't for you. (When the measurement method—especially how shear is caused—is different, the resulting values will also be different for non-Newtonian fluids.) However, if you are looking for a way to somehow quantify significant differences that you couldn't possibly see with rotational types, the RV-10000A may finally come to your rescue.

For example, you may have these difficulties and frustrations with a rotational viscometer/rheometer…

1. Measurement results are unstable and not repeatable when testing low viscosity (e.g. below 100 mPa.s) samples
2. A large sample quantity has to be consumed for each measurement (the B-type viscometers typically require approx. 500 mL for low viscosity measurements)
3. Increasing/decreasing processes of viscosity cannot be followed in a continuous manner (spindles need to be replaced)
4. The relationship between the temperature and viscosity of a sample fluid cannot be measured precisely
5. Measurement cannot be performed without breaking samples' original physical states or properties
6. Samples stick to and gather around the spindle, slurries scrape the surface of the spindle, etc.

Moreover, many researchers feel that the data taken with rotational types often don't represent how the samples are actually sensed by humans. Nevertheless, the rotational method and the data it produced have long been treated as de-facto standards, though there shouldn't be such restriction on viscometry in the first place. Depending on the nature of the sample and the purpose of your research, you'll be able to measure what you really want to measure if you choose a different, more appropriate instrument.

Now, let's take a look at the main advantages that the RV-10000A can bring to you.

Advantage 1: Quick stability and high repeatability with low viscosity fluids

The RV-10000A takes only 20 seconds to stabilize (and thereafter displays viscosity variation in real time). It also achieves such high repeatability as 1% of the measured value (by standard deviation) for its entire measurement range, with the required sample quantity being as small as 10 mL. These remain true even when the sample viscosity falls below 100, 10 or 1 mPa.s!

Advantage 2: Wide continuous measurement range (max. 0.3 to 25,000 mPa.s)

The RV-10000A can uninterruptedly measure from very low to high viscosity with a single viscosity detection system (i.e. no replacement of parts required), which realizes high versatility and allows measurement of various types of fluids. With a succession of shear rate changes, it can comprehensively capture a fluid's flow characteristics.

Advantage 3: Accurate, concurrent measurements of temperature and viscosity

With the temperature sensor (0 to 160 ˚C range) located right between its tuning fork tines, the RV-10000A measures the sample's temperature and viscosity changes precisely and simultaneously. It is also possible to control the sample temperature (between 0 and 100 ˚C) using a commercially available temperature control tank.

Advantage 4: Little interference to sample fluids by the measurement system

The low frequency and low amplitude of the tuning fork causes only minute displacement in the sample fluid and keeps changes to its temperature and physical structure to a minimum, ensuring stable measurements over a long period of time. Samples won't gather around or scrape the sensor parts, either.

Advantage 5: Automatic shear rate changes and graphing data

The dedicated controller of the RV-10000A can automatically change the vibration amplitude (i.e. shear rate) and take data in the manner you programmed (useful preset programs are also available). Further, it shows and graphs measured values in real time so that viscosity changes as a function of time, temperature and/or shear rate become plainly visible.

Advantage 6: Easy maintenance and calibration

All you have to do is wipe off the sample residue with alcohol after each measurement to clean the sensor unit and keep it in good condition. You can also calibrate the RV-10000A (at either one point or two points) using viscosity standards easily by yourself, without having to consider the so-called geometry factors. In addition, for a viscosity range around 1 mPa.s, a highly useful, "simplified calibration function" is available, which uses purified water and requires a simple one-key operation for quick and automatic calibration.

Fields where the RV-10000A will be especially useful

The RV-10000A is unmatched in its ability to measure a diverse array of samples. Irrespective of industry, it can be used by anyone in research and development involving viscosity evaluation of liquids—in particular low-viscosity liquids. Its targets are mainly the following:

► **Research and development of materials**

Polymeric chemistry, electronics materials, inks, film/glass coating materials, battery materials, detergents, cosmetics, fibers, drugs/medicines, rubbers, ceramics, food (mayonnaises, yogurts, refreshing beverages, etc.), and so on

► **Process development for plants**

Thanks to its extremely high sensitivity, measured values are such that they can closely represent humans' actual sensations. It is therefore possible, for example, to study how a hand cream will be felt when smoothed over the skin, or how pleasantly a drink will go down the throat, with hitherto unachievable precision.

Since its sensor plates exert little energy on the sample, the RV-10000A lets you measure such fragile fluids as foams (e.g. fire extinguishant, whipped cream, etc.) without dissipating them or breaking their tiny air bubbles. It also enables measurement of dispersion systems (e.g. suspensions) while they are settling. (Alternatively, the dispersion can be maintained using a microstirrer because the two sensor plates vibrating in opposite phases allow measurement of sample fluid even while it is flowing.)

Besides use in R&D, the quick stability and high repeatability of the RV-10000A also make it a powerful tool for quality control in factories where test samples are taken from the production lines to a laboratory to make pass/fail judgments. To do so, you may simply want to develop a tolerance around a known good sample as a reference and then just use the rheometer as a kind of Hi/Go/Lo comparator.

Note: Theoretical model of the tuning fork vibro technique

When the sensor plates vibrate with a frequency of f, the mechanical impedance R_z received by the sensor plates from the sample fluid can be calculated as:

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

f : vibration frequency (Hz), A : surface area of both sides of the sensor plates
η : viscosity of the sample fluid, ρ : density of the sample fluid

Here, if the force F generated by the electromagnetic drive gives the constant vibration velocity $Ve^{i\omega t}$ to the sensor plates, the equation can also be expressed as:

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

From the equation above, it can be understood that the force from the electromagnetic drive is proportional to the product of viscosity η and density ρ.

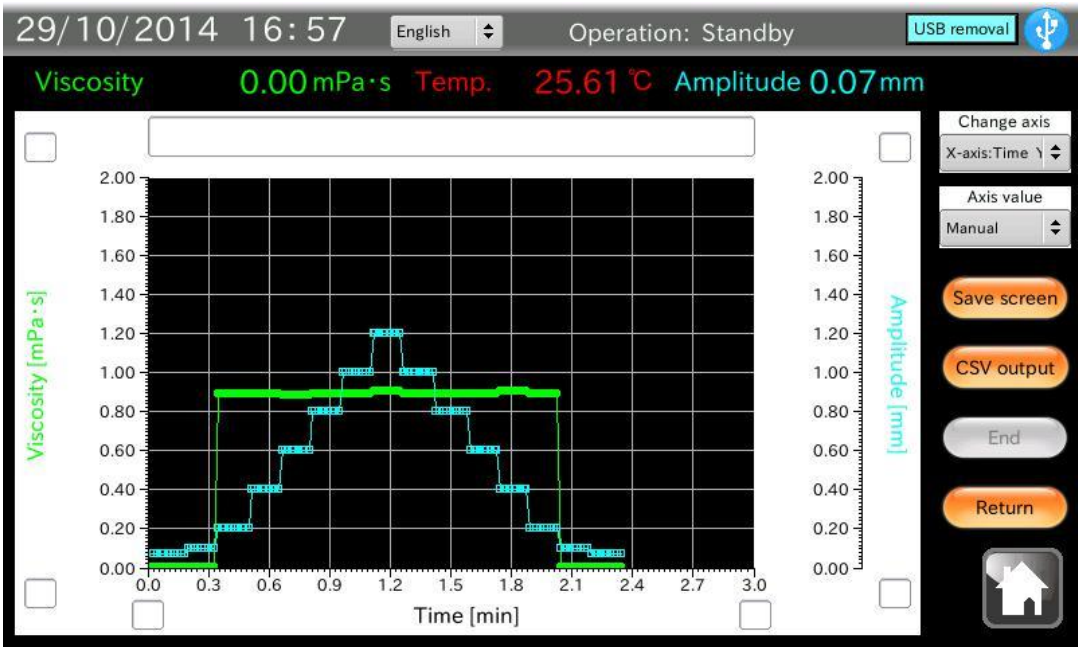
The force F can be obtained using the equation: $F = I \times B \times l$

I: driving current (A), B: magnetic flux density (T), l: coil lengh (m)

Measurement Data Examples

What follow are screen shots of the RV-10000A controller section. As you will see, the X-axis and right Y-axis variables can be switched between Time (elapsed), Temperature, Amplitude and Shear rate. (Time can be indicated by the X-axis only, and the left Y-axis is saved for Viscosity.)

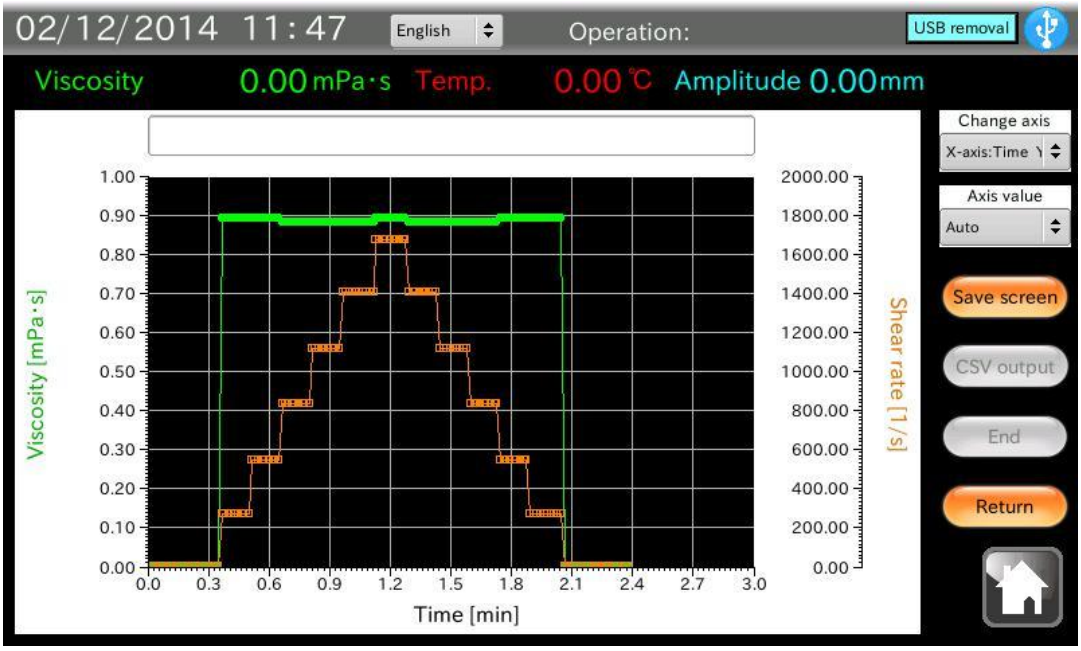
1) Measurement of purified water



Graph 1-1. Relationship between time, amplitude and viscosity of purified water

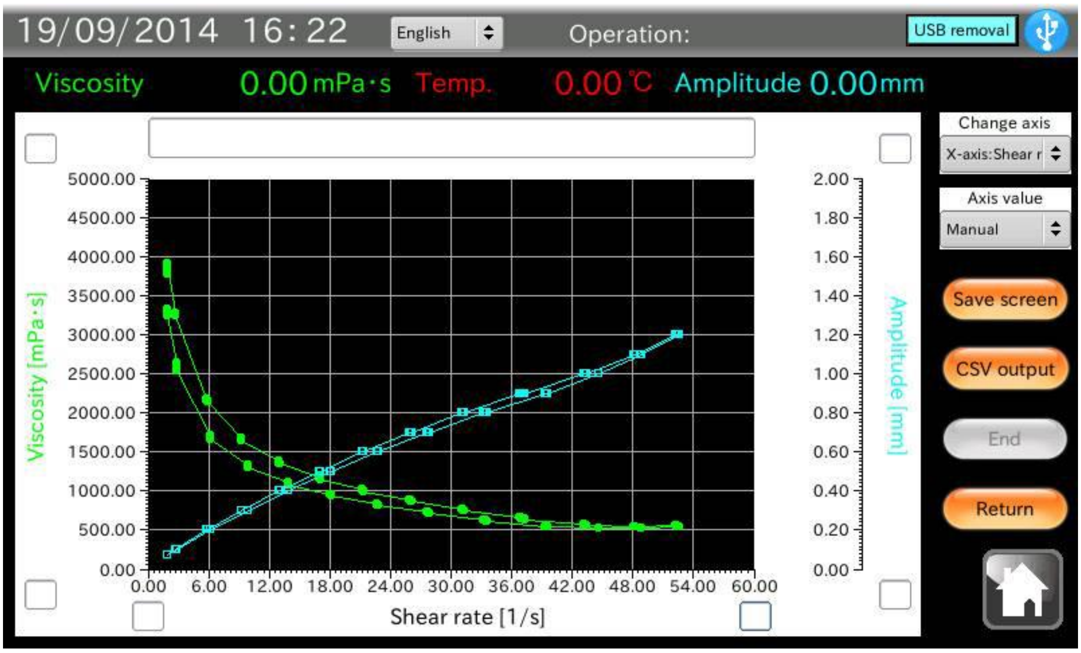
Graph 1-1 depicts viscosity of purified water when the amplitude was automatically increased from 0.07 mm to 1.2 mm in eight steps and then decreased to 0.07 mm in eight steps again (Auto-Quick mode). As water is a Newtonian fluid, the viscosity values are shown to be constant regardless of the amplitude. (No value is recorded where the sample viscosity is out of the measurement range at the given amplitude.)

In Graph 1-2 is the same measurement but the right Y-axis indicates shear rate instead of amplitude. Likewise, you can confirm that the viscosity remains constant despite the differing shear rates.



Graph 1-2. Relationship between time, shear rate and viscosity of purified water

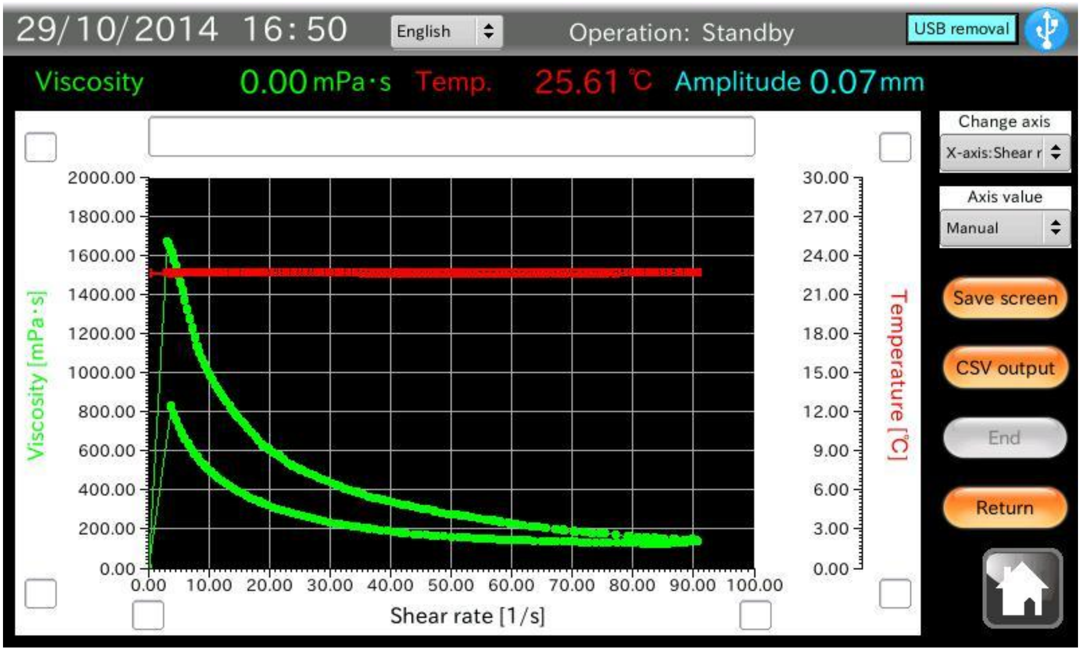
2) Measurement of skin moisturizing cream



Graph 2. Relationship between shear rate, amplitude and viscosity of skin moisturizing cream

Graph 2 shows viscosity measurement for skin moisturizing cream using the same Auto-Quick mode. First, you can readily understand that the shear rate changes in proportion to the amplitude. Secondly, the viscosity is observed to decrease as the shear rate becomes higher, and then increase as the shear rate becomes lower again but without tracing the same path. This "loop" in the graph is called the hysteresis loop, and the larger the area of such a hysteresis loop, the stronger the thixotropic nature of the sample fluid. (Thixotropy is a property of non-Newtonian fluid where viscosity becomes lower when stirred or shaken and it takes time to return to the original state.)

3) Measurement of yogurt

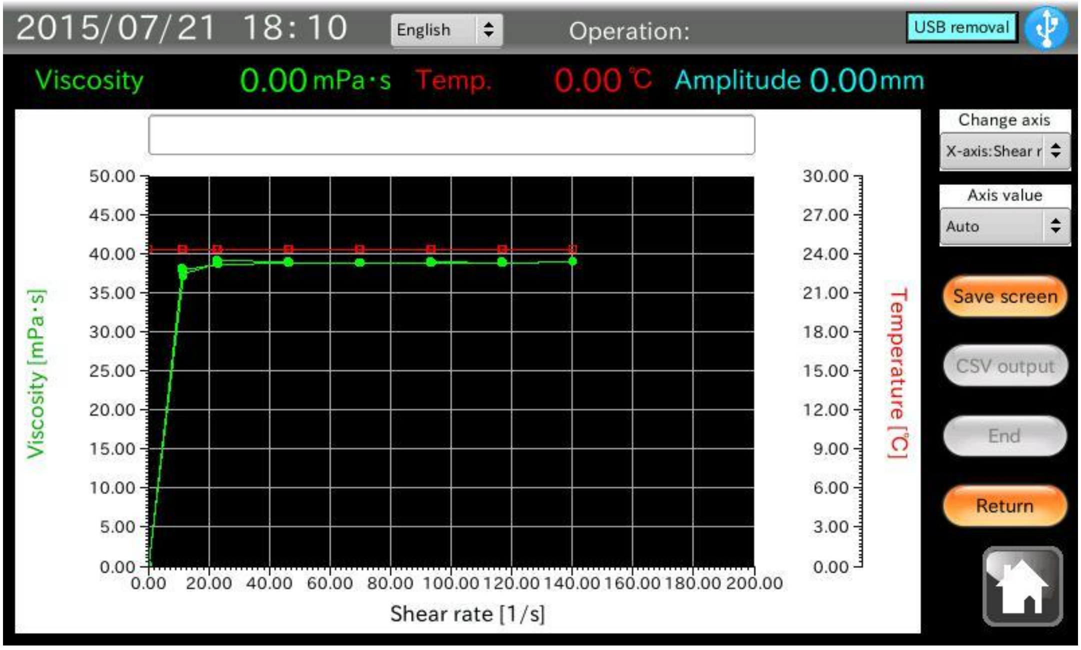


Graph 3. Relationship between shear rate, temperature and viscosity of yogurt

In the measurement of Graph 3, viscosity changes of yogurt were plotted while the amplitude traveled from 0.07 mm to 1.2 mm and back to 0.07mm in scales of 0.01 mm (Auto-Fine mode). The temperature remained constant all the way.

This is another, clearer example of a thixotropic fluid. Perhaps the time-dependent, thixotropic nature of viscosity of yogurt is relatively easy to visualize, if you imagine what happens when you stir up yogurt to mix with sweetener, etc.

4) Measurement of an ink-jet solution

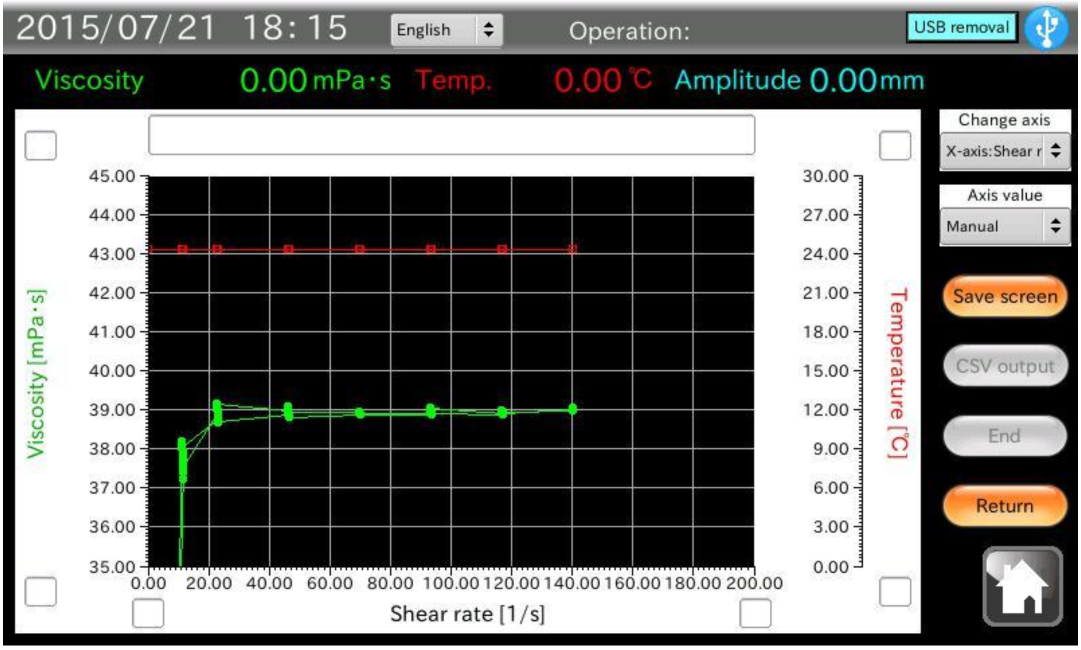


Graph 4-1. Relationship between shear rate, temperature and viscosity of an ink-jet solution

Graph 4-1 is the result of viscosity measurement of an ink-jet solution for printers in Auto-Quick mode carried out for one of A&D's customers. Ideally, the ink-jet solution has to be a Newtonian fluid, and indeed the customer once believed that it was a Newtonian fluid.

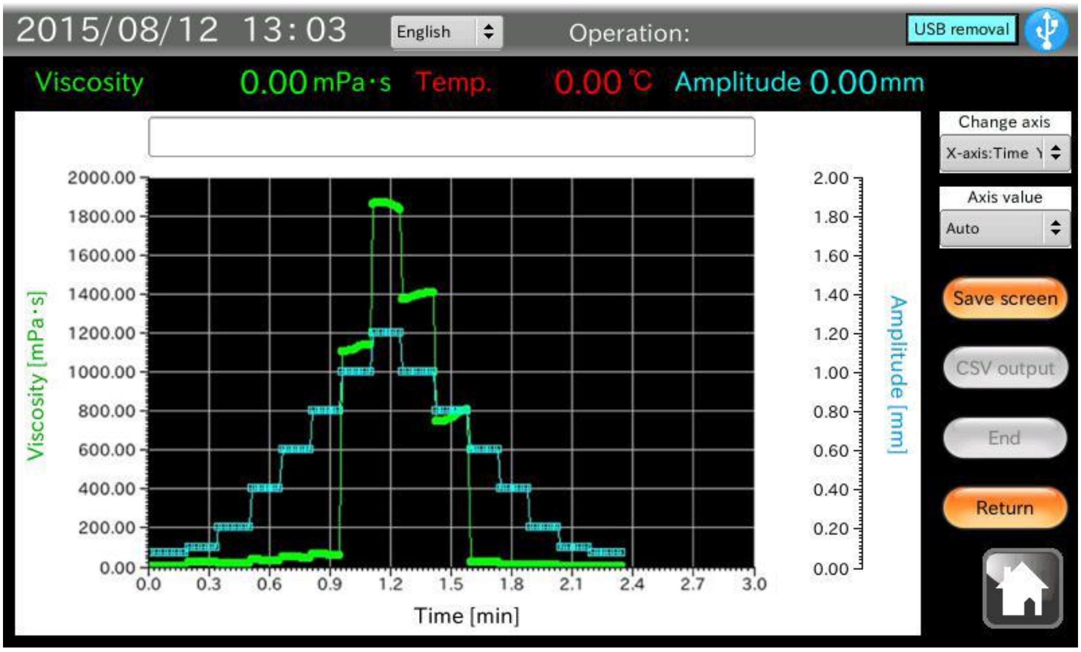
However, if you look at the graph more closely (see the different scaling in Graph 4-2), you will notice that there exists the viscosity's slight dependence on shear rate near the lower end. What this suggests is that although the solution may behave as designed while jetted fast, it may cause some unintended outcomes (such as lumps or unevenness of color) when it arrives on a sheet of paper (and consequently the shear rate diminishes).

The customer was surprised to discover this phenomenon, which triggered further study on this sample, and also at how the RV-10000A could precisely measure behavior of such a low-viscosity sample at such low shear rates.



Graph 4-2. Closer look at the viscosity's dependence on the shear rate

5) Measurement of a cornstarch solution



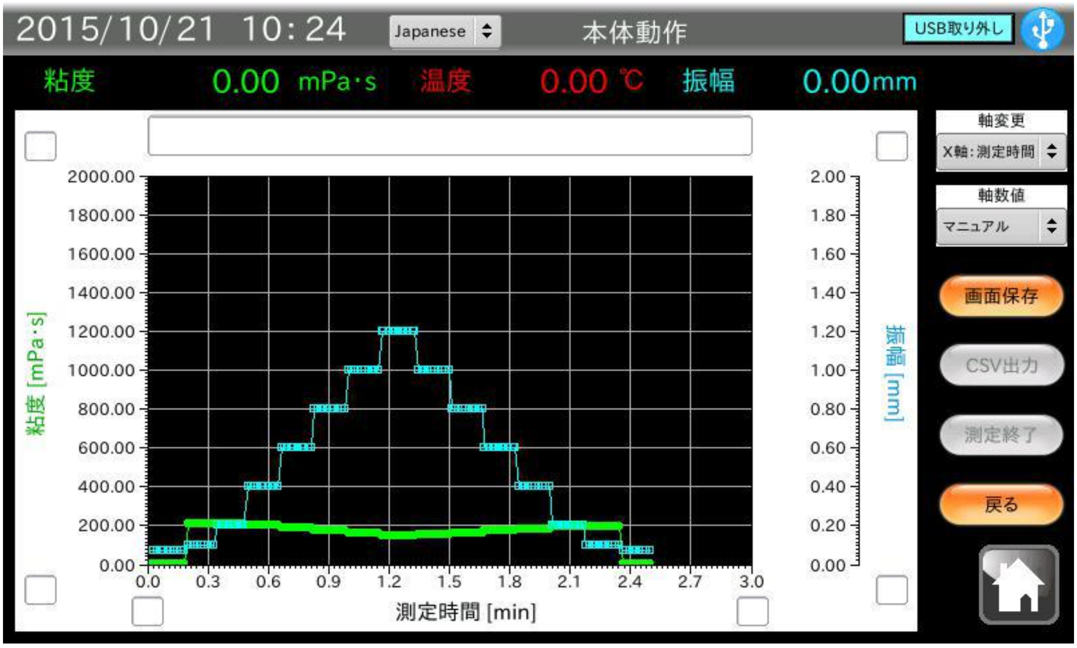
Graph 5. Relationship between time, amplitude and viscosity of a cornstarch solution

Graph 5 shows the viscosity variation of a cornstarch solution in relation to amplitude measured in Auto-Quick mode. This is a good example of a dilatant fluid. The solution is thin when the amplitude is small (low shear rate), but from a certain amplitude point (high shear rate) the viscosity increases rapidly.

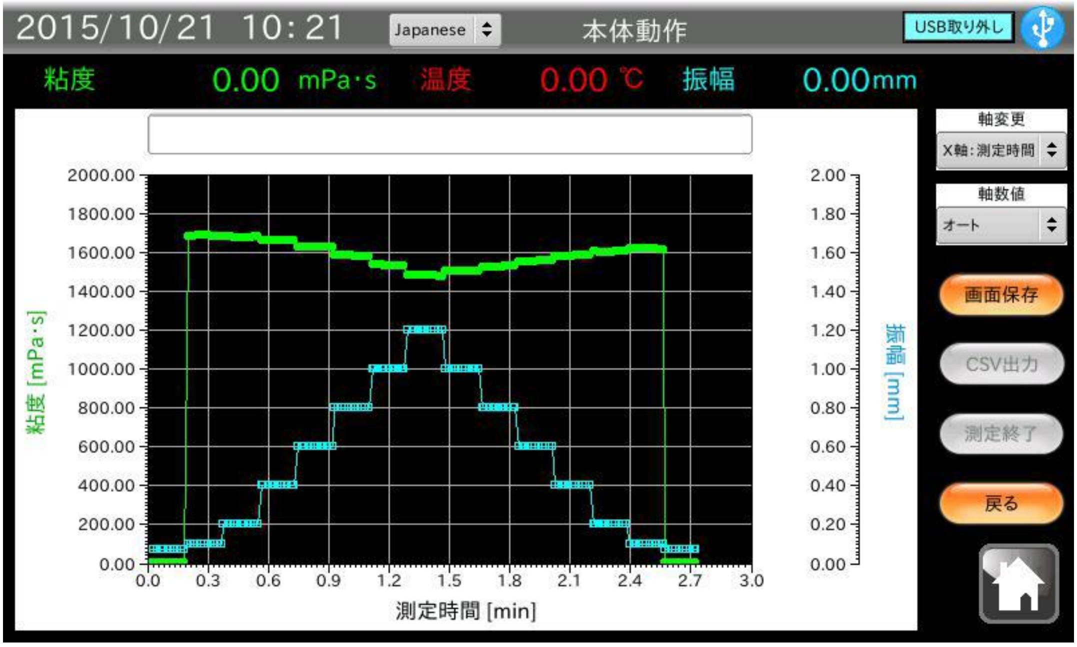
6) Measurement of thickening agents

For recent thickening agents for food for patients with dysphagia (difficulty in swallowing), a strong thickening effect is demanded for a small amount of additive. Graphs 6-1 and 6-2 are the results of measurements of the typical thickening agent, xanthan gum, at 1% and 5% concentrations respectively. Similarly, Graphs 6-3 and 6-4 show the results of measurements of carboxymethylcellulose (CMC) at 1% and 5% concentrations respectively.

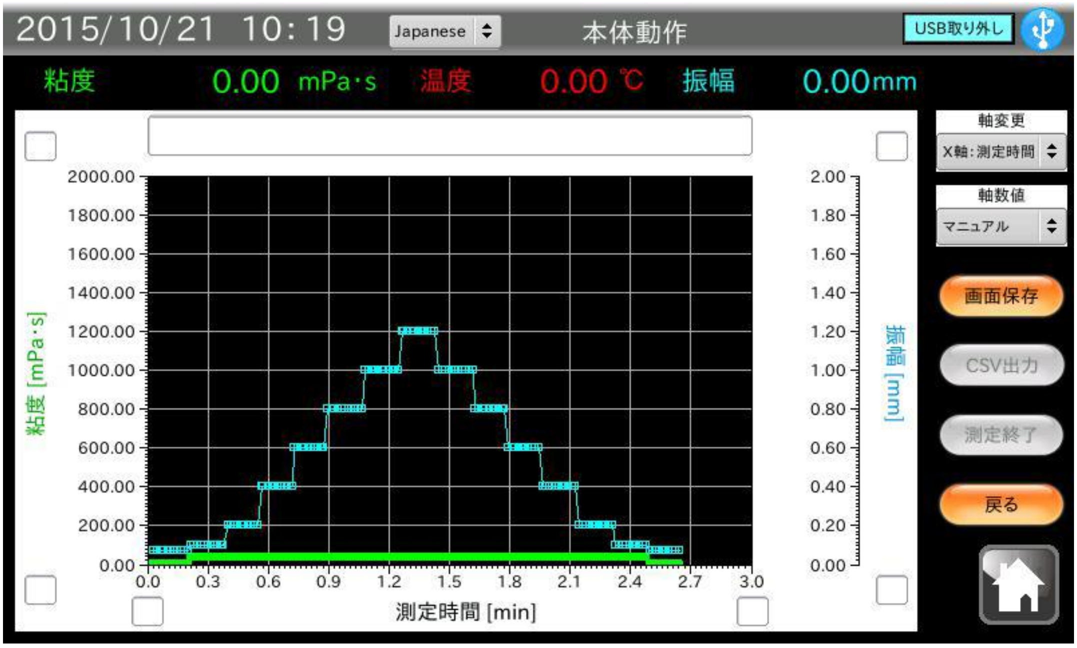
Notice how a large difference in viscosity arises from only a small difference in concentration of xanthan gum or CMC, which points to the extreme importance of volume management when added to food products in the course of nursing care.



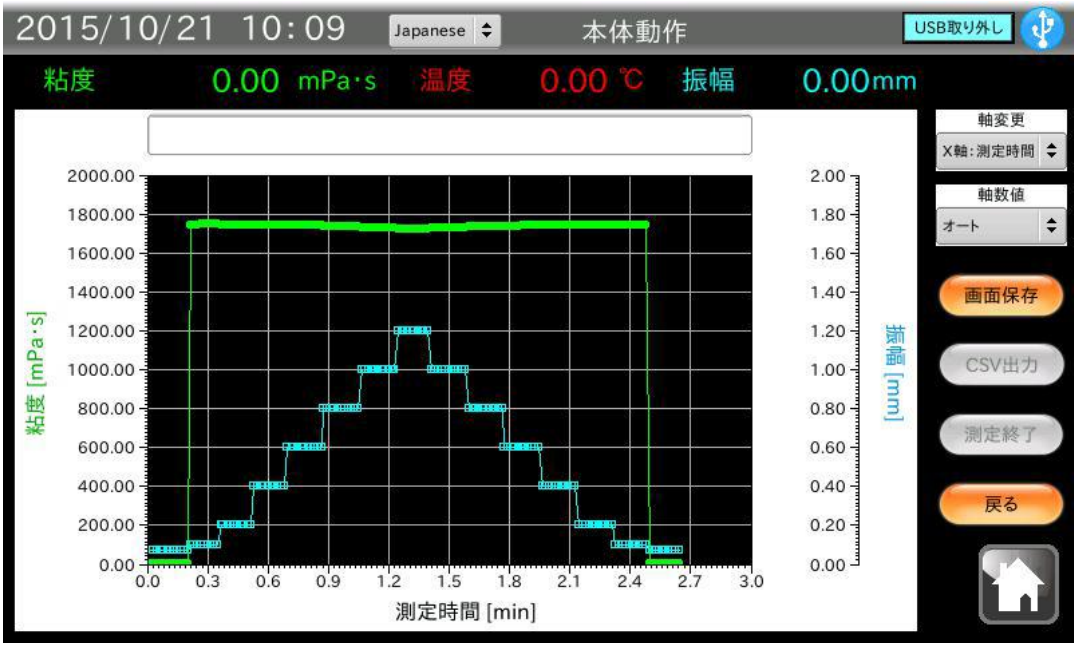
Graph 6-1. Viscosity of xanthan gum at 1% concentration



Graph 6-2. Viscosity of xanthan gum at 5% concentration



Graph 6-3. Viscosity of CMC at 1% concentration



Graph 6-4. Viscosity of CMC at 5% concentration

Additionally, the change in viscosity in relation to change in amplitude is small (especially for CMC), meaning that the change in viscosity in relation to change in the speed at which the food product is swallowed will also be small.

Real-Life Solutions With The Tuning Fork Vibro Rheometer

Finally, let's look at some actual cases in which the RV-10000A (or the RV-10000, the cutdown version) helped customers solve various problems they were facing in research. The RV-10000A is still a new product, and examples such as these should only increase as time goes by. Hopefully, your measurement will be among them in the near future!

In private sector

1. R&D in carbon nanoparticles

   The researchers needed to ascertain that their samples were Newtonian fluids.

   → *It was easily achieved by confirming that the viscosity would remain constant regardless of the sensor plate amplitude.*

2. R&D in the sag resistance and wettability of ink-jet solutions

   Neither a rotational rheometer nor a dynamic viscoelastic measurement device would produce consistent results when shear rates were low.

   → *The RV-10000A provided high reproducibility in measurements at low shear rates. Setting samples was also very easy.*

3. R&D in ceramic coating liquids applied on electronic components

   Repeatability was poor in low viscosity measurements using a rotational viscometer

   → *It became possible to perform measurements of low viscosity samples with high repeatability and further evaluate the dependence on shear rate of the samples.*

4. R&D in lithium-ion batteries

   The sedimentation rates of the samples are high (i.e. particles in suspension settle very quickly), which made the viscosity measurement difficult.

   → *The researchers became able to reproducibly measure their flow properties changing the shear rate while stirring the samples and preventing the sedimentation.*

5. R&D in coating materials

   The researchers were not allowed to use their company's rotational viscometer as their slurry samples would scratch the spindle surfaces.

   → *With the minute vibration, measurements could be performed without causing any damage to the sensor plates.*

6. R&D in food fibers

   The researchers wanted to monitor viscosity variation due to temperature changes.

   → *It became possible to continuously monitor the behavior of a sample varying from low to high viscosities in a wide measurement range.*

7. R&D in plating solutions, resins, etc.

   The engineers suspected that the way they mixed solutions would alter their viscosity characteristics.

   → *The RV-10000 allowed them to see how the sample viscosity would vary with shear rate and further provided stability in low viscosity measurements.*

In universities

1. Study of thickening agents for cosmetics

   With a rotational viscometer, the measurement was unstable and had poor repeatability when the sample was small in quantity and low in viscosity.

   → *The RV-10000A enabled stable low viscosity measurement with a small sample quantity. The researchers were also able to expand their research to the viscosity changes of cosmetic products before and after application to the skin, etc.*

2. Study of the surface polishing agents for glass, metal, etc.

   The researchers wished to measure the dispersion and aggregation movements of abrasive grains in water-like bases with a small sample quantity.

→ *It became possible to examine the movements using a small quantity (10 mL) of low viscosity samples and with the optimal additive concentration.*

3. Study of the liquefaction (softening of saturated soil due to earthquake-induced vibration) of geological strata

   The researchers were interested in how samples would turn soft when differing forces were applied.

   → *The softening processes of wet clayish soils with various moisture contents could now be observed while changing the shear rate acting on them.*

4. Study of food thickeners for people with dysphagia (difficulty in swallowing)

   With a rotational type, the destruction of the sample's molecular structure was large and it was difficult to follow the increasing process of the viscosity.

   → *The destruction of molecular structure was minimized, and stable measurement from sol to gel was realized.*