Technical Article

A High-Performance,

Fully Self-Contained, Rotational Rheometer

Routine viscosity measurements of materials are important in most industrial processes such as mixing and pumping, spraying and coating, extrusion, laydown, and leveling. Typically, a Dynamic Shear Rheometer is employed to measure the flow characteristics of most non-Newtonian systems; however, these instruments can be complicated and relatively expensive to purchase and maintain. They can require an operator with an advanced degree. A lower-cost, simple-to operate, rotational Rheometer can provide a similar flow curve profile of these

Figure 1

non-Newtonian materials if designed and produced to be application specific, incorporating a self contained temperature control system, and interfaced with a user-friendly yet powerful Windows-based software package. This article describes the MERLIN VR self-contained, rotational Rheometer (*Figure 1*), which is capable of performing routine rheological tests from quick, single-point checks to complete viscosity flow profiles, and yield stress determinations without the need for complicated test method setup, laboratory facilities such as compressed air, and external fluid circulator and/or water connection for temperature control.

The MERLIN VR is well suited for investigating the mixing, stirring, and pumping behavior of coatings, emulsions, and dispersions, as well as for performing conventional flow and viscosity profile experiments. The innovative design incorporates a Peltier temperature control system that allows isothermal, step, and / or ramp temperature profiles.

The DIN standard sample measuring systems of cone and plate, parallel plate, and bob and cup (*Figure 2*), coupled with a wide shear rate and torque range (*Table 1*), provide a measurable viscosity range from 1 to 1E08 cP. This is accomplished by using the DIN measuring system over the specified torque and / or angular velocity ranges of the MERLIN Rheometer and then combining the results.

Table 1.

Technical specifications for MERLIN VR Rheometer

Angular velocity	**0.01–200 rad/sec**
Shear rate range	**Measuring system dependent**
Torque range	**0.001–20 mNm**
Shear stress range	**Measuring system dependent**
Temperature range	**–10 °C to 120 °C**
Speeds	**20,000 discrete steps**
Measuring systems	**Cone and plate, parallel plate, and concentric cylinders**
Dimensions	**180 × 520 × 340 mm**

Figure 2

The enhanced performance specifications of the Rheometer enable the testing of a wide range of materials, including paints, coatings, inks, surfactants, polymer solutions, foods, pharmaceuticals, cosmetics, biochemicals, asphalt, molten polymers, adhesives, sealants, and petrochemicals.

- High Performance Integrated Rotational Rheometer
- Wide range of measuring systems
- Powerful Windows based software
- Built in Peltier Temperature Controller

Rheometer performance

To demonstrate the performance of the instrument with some standard measuring systems—1) 30-mm-diam parallel plate, 2) 30-mm/5° cone and plate, and 3) 25-mm-diam bob and cup—a nominal 1000-cP silicone oil was evaluated at 25 °C. The parallel plate experiment was performed with a 0.5-mm gap, while the 5° cone and plate had a 0.150-mm truncation. Steady shear experiments were performed using the Windows™-based MICRA user interface software (Microsoft, Redmond, WA) from high to low shear rate.

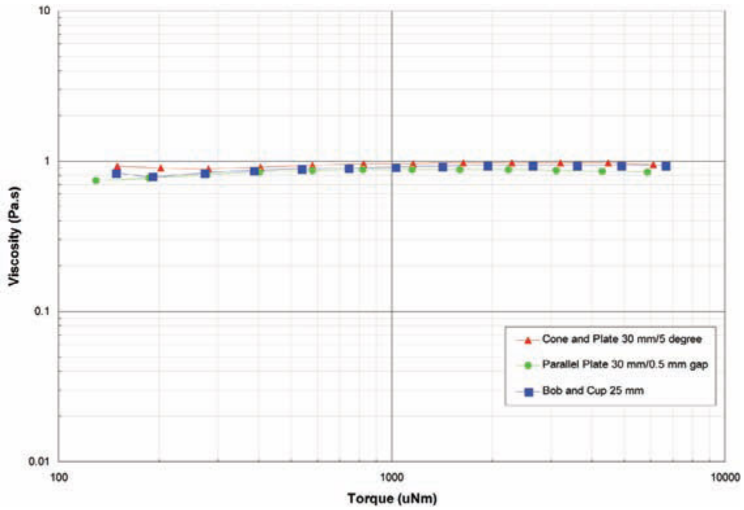

Figure 4 Comparison of viscosity measurement for 1000 cP oil at 25 °C using three geometry setups: cone and plate, 30 mm; parallel plate, 30 mm; and bob and cup, 25 mm.

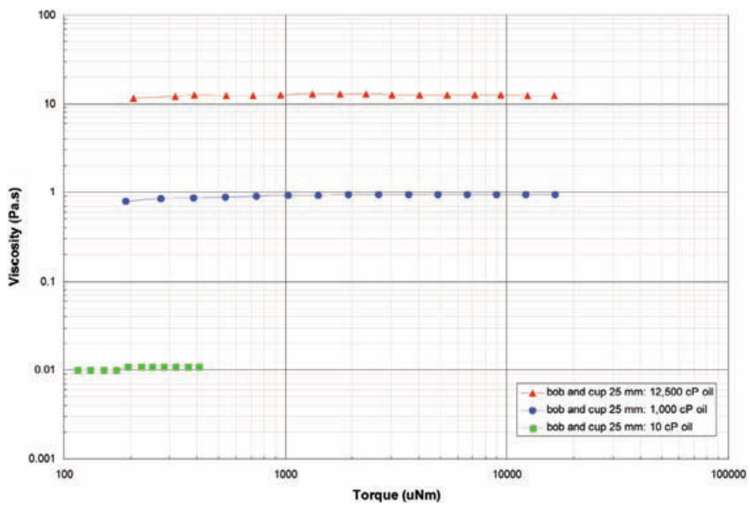

Figure 6 Viscosity as a function of torque for three Newtonian standard oils: 12,500; 1000; and 10 cP at 25 °C using bob and cup, 25 mm.

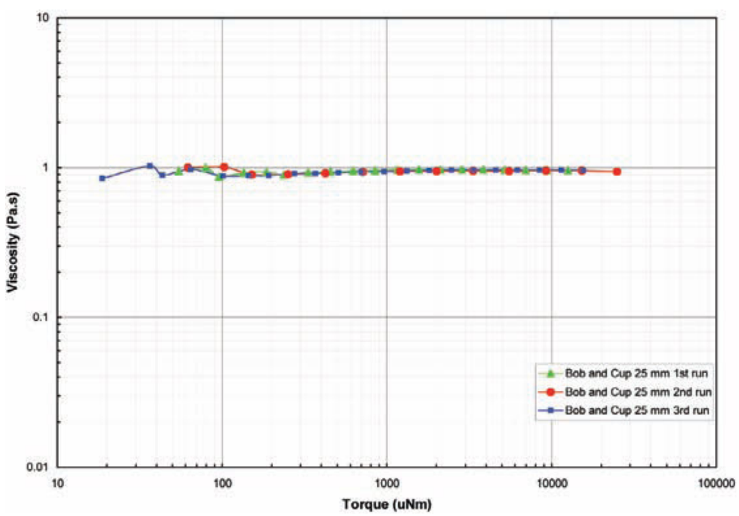

Figure 5 Three independent runs for 1000 cP oil at 25 °C using bob and cup, 25 mm.

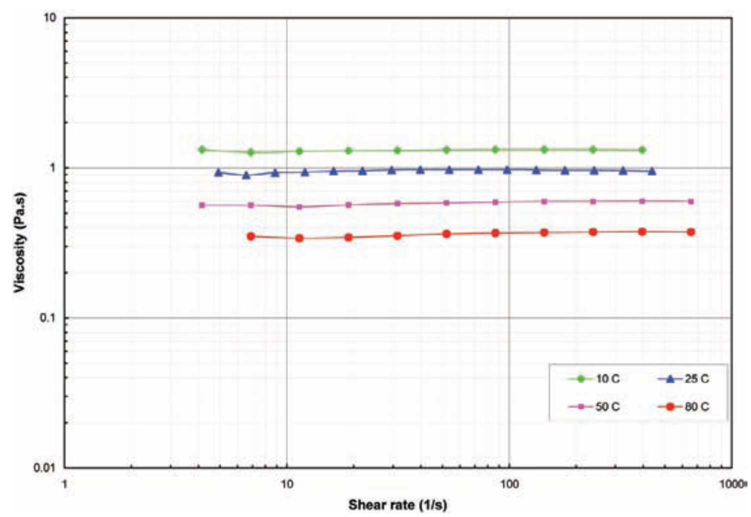

Figure 7 Viscosity as a function of shear rate for 1000 cP oil at various temperatures: 10; 25; 50; and 80 °C using bob and cup, 25 mm.

The resulting steady-state viscosity (Pa s) is plotted versus torque (mNm) in *Figure 4* (torque is plotted on the abscissa rather than shear rate [s–1] to normalize the data for the three experiments). As expected, all three measuring systems show similar data. The ability to use different sample geometries permits the user to expand the capacity and range of the instrument and/or accommodate a wider range of materials and sample sizes/amounts. The bob and cup system is well suited for low viscosity materials and/or low-shear rate measurements since it provides more sample contact surface area and thus more torque signal. Three repeat runs are shown in *Figure 5*, where viscosity (Pa s) is plotted as a function of torque (mNm) for the bob and cup system, demonstrating the performance of the MERLIN Rheometer over the working torque range of the instrument.

The bob and cup geometry was utilized to test several different viscosity standards covering the range 10–10,000 cP at 25 °C. All data are in acceptable ranges of viscosity, as shown in *Figure 6*. Again, the data are plotted versus torque to normalize the abscissa. Viscosity (Pa s) at several set temperatures for the nominal 1000 cP oil is shown in *Figure 7* as a function of shear rate (s–1). The ability to control and/or systematically change the sample test temperature is paramount in making viable rheology and viscosity measurements. The viscometer's built-in Peltier temperature control system supports all measuring systems, including cone and plate, parallel plate, and bob and cup.

The temperature accuracy of the instrument using the bob and cup measuring system is summarized in *Table 2*. It is clear that the temperature was well calibrated throughout the range investigated. A thermal enclosure is available to enhance the performance at higher temperatures.

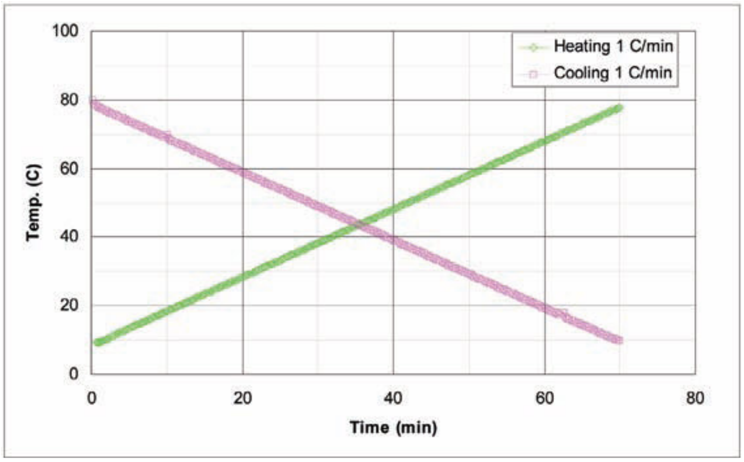

Figure 8 Temperature profi les for heating and cooling between 10 and 80 °C at a rate of 1 °C/min. The sample is 1000 cP oil using bob and cup, 25 mm,

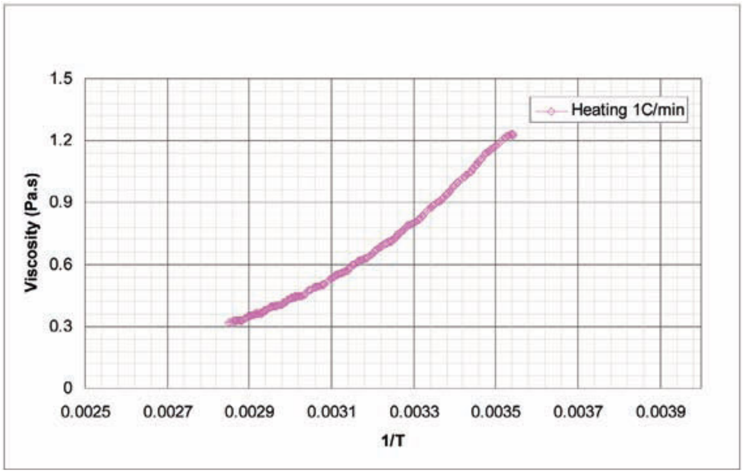

*Figure 9 Viscosity as a function of 1/*T *for heating between 10 and 80 °C at a rate of 1 °C/min. The sample is 1000 cP oil using bob and cup, 25 mm, at a*

An important advantage of the MERLIN VR Rheometer over commercially available viscometers, is the ability to perform user-defined, controlled temperature ramps. This is vitally important for temperature-sensitive materials. The activation energy *Ea* can be estimated from such an experiment. *Figure 8* shows the temperature profiles experiments between 10 and 80 °C, both at a rate of 1 °C/min. A bob and cup geometry was used for this test on 1000 cP oil at a constant shear rate of 10 sec–1. It is clear that the temperature can be controlled well with the Peltier temperature control system.

Figure 9 shows the viscosity as a function of 1/*T* for the heating experiment, which can be fit by the Arrhenius model, $\eta = C e^{k/T}$, where the constant k is related to the activation energy by *Ea*, with R the Boltzmann constant. From this experiment, the activation energy obtained from the heating run was 16.9 kJ/mol. Other controlled temperature ramps from 0.1 to over 2 °C/min are also available.

The MERLIN VR is able to measure the dynamic yield stress or yield point of a sample, and the MICRA software supports this experimental mode of testing. Using the 25-mm bob and cup, a commercial ketchup sample was evaluated. *Figure 10* shows the results plotted as shear stress (Pa) versus time (s). The yield stress experiment was performed by applying a 0.1 s–1 shear rate and measuring the resulting stress response. The maximum stress value measured is the yield stress of the sample, approximately 14.7 Pa for this ketchup. This yield stress measuring capability is a significant improvement over commercially available viscometers, which only provide viscosity at a single rotational speed.

Table 2	**Temperature response of the Merlin VR with cup & bob, 25mm**
Instrument setpoint	**Meter Reading**
10 °C	10.1 °C
25 °C	25.1 °C
50 °C	50.0 °C
80 °C	79.9 °C

The Windows based MICRA software also allows for user-defined shear rate sweeps, where the shear rate is increased/decreased step-wise, permitting a complete flow curve profile to be determined. *Figure 11* shows the viscosity versus shear rate for the commercial ketchup sample in the shear rate range 1–1000 s–1. The data reveal that the sample is highly shear thinning (pseudoplastic), and clearly cannot be defined by a single viscosity value as obtained from commercial viscometers.

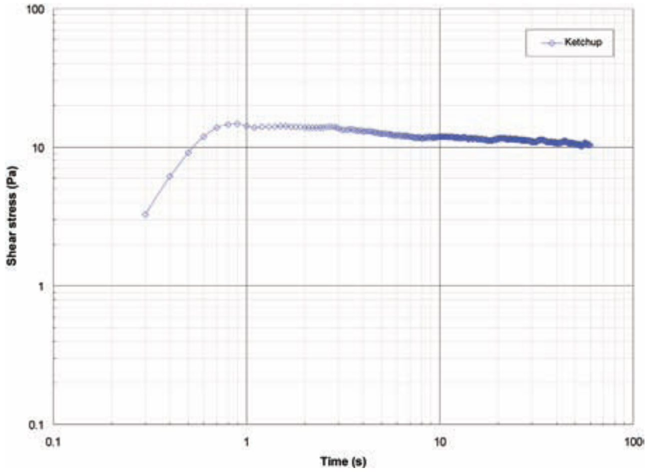

Figure 10 Shear stress as a function of time for a commercial ketchup at 0.1 s–1 using bob and cup, 25 mm, at 25 °C.

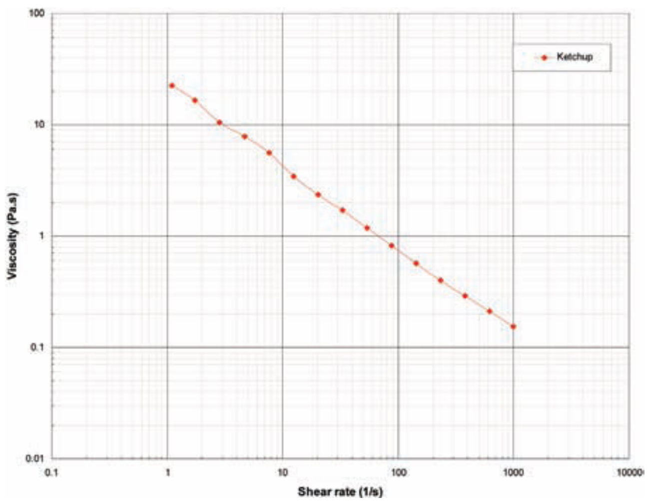

Figure 11 Viscosity as a function of shear rate for a commercial ketchup using bob and cup, 25 mm, at 25 °C.

Conclusion

This article demonstrates that the MERLIN VR Rheometer has the capacity to easily generate precise data in terms of viscosity, flow curves, and yield stress. Its built in Peltier temperature control system and ability to conduct temperature isothermal and ramp experiments distinguishes it from other viscometers / rheometers. In addition, the fact that it is equipped standard with different measuring systems enables the viscometer to accurately and routinely measure the viscosity of a wide range of samples under varied conditions.

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