RHEOLOGY APPLICATIONS NOTE

AR-1000 Temperature Control

FEATURES/BENEFITS

Since viscoelastic material properties such as viscosity change significantly with temperature, it is important to accurately control temperature during rheological measurements. There are three key considerations which must be weighed when choosing the best method of temperature control. These three considerations are: temperature range covered, heating/ cooling rates available, and the temperature accuracy and precision achieved. TA Instruments offers a broad range of temperature control options for use on the AR-1000, including fluid jackets, two Peltier plate systems, and extended temperature modules (ETM). These options allow the operator to choose those that best meet his specific needs.

SPECIFICS

When comparing rheometer temperature control options, the following must be considered:

- *Temperature Range Covered*. Although some material evaluations require only ambient temperature measure ment, it is more common that behavior is evaluated over a range of temperatures representing production, storage, and/or end-use. Depending on the material, this range can extend from below 0ºC (e.g. foods) to above 350ºC (e.g. polymers). Thus, the rheometer temperature control system used should be capable of covering at least the range of immediate interest.

- *Heating/Cooling Rates Available*. In addition to evaluating a material over a range of temperatures, it is often important to be able to study the material under both dynamic (changing) and isothermal conditions. Particularly when simulating processing conditions, the ability to rapidly change temperature may be required. Even in isothermal experiments, the ability to rapidly change temperature and achieve equilibration of the sample and measurement geometry is desirable.

- *Temperature Accuracy and Precision*. The viscosity of a Newtonian fluid decreases exponentially with temperature according to the relationship $\eta = Ae^{B/T}$ where η is viscosity, T is the absolute temperature and A and B are constants of the liquid. Furthermore, for Newtonian fluids, the greater the viscosity the stronger the temperature dependence. Hence, to obtain viscosity values with an accuracy of ±1%, temperature must be controlled to better than ±0.3°C. In addition, the temperature should be measured in or near the sample material to ensure against discrepancies due to gradients.

On the following page, Table 1 provides an overview comparison of the temperature control options offered by TA Instruments for the AR-1000 Rheometer. Additional details on each option are covered in later sections.

TABLE 1

	Fluid Jacket	Peltier Plate	ETM
Temperature Range	-25 to 150°C	-10 to 99.9°C (standard) -20 to 180°C (extended)*	-100 to 400°C
Heating/Cooling Rates	up to 2°C/minute	up to 50°C/minute	up to 120°C/min
Temperature Precision	±0.1°C	±0.1°C	±0.1°C

* Temperature range of the Peltier plate can be extended to -40°C by use of refrigerated circulator.

Fluid Jacket

In this arrangement, fluid from an external bath is circulated through a jacket which surrounds the sample and test geometry (Figure 1). The actual sample temperature attained is measured and controlled by one of two options:

1. Temperature is controlled at the circulation bath and not in the fluid jacket near the sample. In addition, there is no readout of sample temperature on the controller (computer). This is the least accurate option, and generally not recommended.
2. An integrated system where a Pt100 sensor inserted in the sample cup provides a sample temperature not only for readout to the computer, but also for control of the circulator bath. This arrangement provides excellent temperature accuracy and precision, and is recommended for fluid jacket operations.

Because of the large mass of the jacket and circulating fluid, rapid temperature change is not possible. Therefore, the fluid jacket is used only for temperature control when the concentric and double concentric cylinder geometries are required.

Figure 1. **FLUID JACKET**

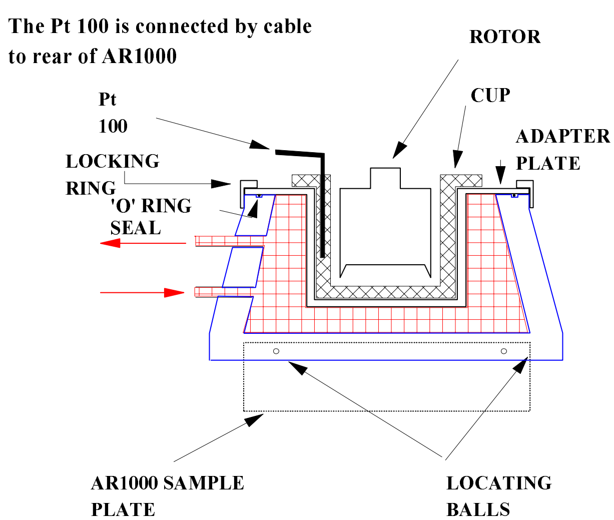
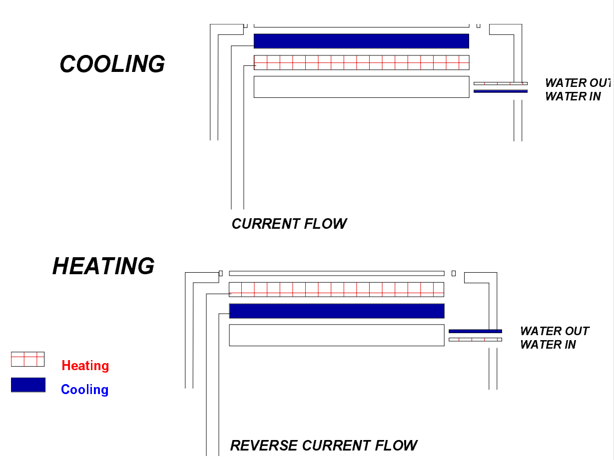


Figure 2. **PELTIER PLATE**



The fluid jacket can be used with the blank sample plate (if Peltier plate is not required).

Peltier Plate

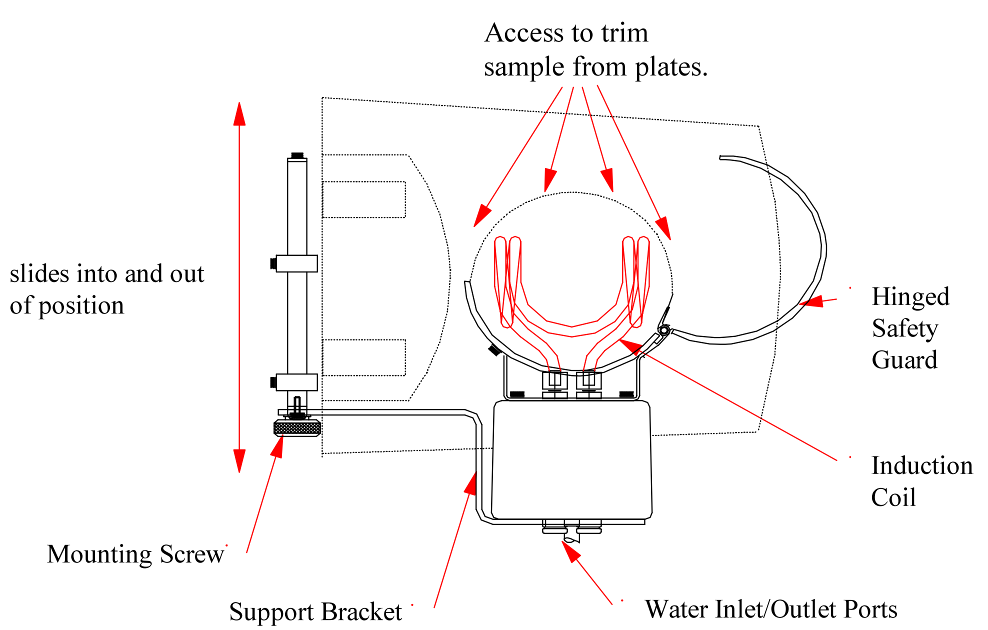
There are two Peltier Plate options for the AR-1000. The Standard Peltier System (SPS) has a top temperature of 99.9°C, while the Extended Peltier System (EPS) has a top temperature of 180°C. They both serve as the lower plate of the cone & plate and parallel plate geometries (Figure 2). The principle of operation is based on the "Peltier effect" which is essentially the reverse of the effect produced by a thermocouple. In the Peltier effect, a current is introduced to create a temperature change at the junction, which is subsequently transferred to the sample. Water continually circulating through the Peltier plate acts as a heat sink or heat source to facilitate this Peltier effect. With narrow gaps, temperature changes from the plate are rapidly conducted through the sample. Hence, the Peltier plate is capable of heating/cooling rheology samples at up to 50°C/minute. Furthermore, the open design of the Peltier plate facilitates sample loading and cleaning.

Extended Temperature Module (ETM)

The ETM is an induction heating system which can be coupled with a liquid nitrogen cooling accessory to cover the broad temperature range (-100 to 400°C). [Two more limited versions of the ETM are also available. These cover low temperatures (-100°C to ambient) and high temperatures (50 to 400°C) respectively.] Heating is achieved in the ETM and its associated high temperature accessories by using special measurement geometries with a central core with a reusable/ disposable aluminum cover (Figure 3). A coil nearly surrounding the measurement geometry generates a high frequency oscillating electromagnetic field which induces a current in the plates. The induced current readily generates heat, and a ceramic heat break on the geometry prevents heat from being conducted up the drive shaft. This is the most efficient form of heating used by any commercial rheometer because the surfaces of the geometry are heated directly and are in direct contact with the sample. Alternative high temperature heaters which use electrically heated air or oil as the heat transfer medium are not as responsive. The ETM can change temperature as rapidly as 2°C/second and is stable to within ± 0.1°C. These high rates of temperature change facilitate simulation of the actual conditions seen by a material during processing. In addition, high temperature samples such as polymer melts can be evaluated with minimal chance for thermal degradation. Furthermore, the atmosphere around the material can be controlled by the use of small quantities of inert gas to eliminate oxidation if required. Controlled cooling is provided in the ETM by small jets of cold nitrogen gas.

The ETM provides easy sample access for trimming, visibility, and cleanability. The ETM can be used with a blank sample plate (if Peltier plate is not required).

Figure 3.



For more information or to place an order, contact:

TA Instruments, Inc.
109 Lukens Drive
New Castle, DE 19720
Telephone: (302) 427-4000
Fax: (302) 427-4001

TA Instruments N.V./S.A.
Gent, Belgium
Telephone: 32-9-220-79-89
Fax: 32-9-220-83-21

TA Instruments, Ltd.
Leatherhead, England
Telephone: 44-1-372-360363
Fax: 44-1-372-360135

TA Instruments S.A.R.L.
Paris, France
Telephone: 33-01-30489460
Fax: 33-01-30489451

TA Instruments GmbH
Alzenau, Germany
Telephone: 49-6023-30044
Fax: 49-6023-30823

TA Instruments Japan K.K.
Tokyo, Japan
Telephone: 813-3450-0981
Fax: 813-3450-1322

Internet: http://www.tainst.com
e-mail: info@tainst.com

TA Instruments
Thermal Analysis & Rheology
A SUBSIDIARY OF WATERS CORPORATION

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