〈912〉 VISCOSITY—ROTATIONAL METHODS

The principle of the method is to measure the force (torque) acting on a rotor when it rotates at a constant angular velocity or rotational speed in a liquid. Rotational rheometers/viscometers are used for measuring the viscosity of fluids, both Newtonian and non-Newtonian. The following procedures are used to determine the viscosity of Newtonian fluids or the apparent viscosity of non-Newtonian fluids. The calculated viscosity of Newtonian fluids should be the same (within experimental error), regardless of the rate of shear (or rotational speed). Given the dependence of viscosity on temperature, the temperature of the substance being measured should be controlled to within ±0.1°, unless otherwise specified in the individual monograph. [NOTE—For additional information, see *Rheometry* 〈1911〉.]

- **METHOD I. SPINDLE VISCOMETERS**

Apparatus: In the spindle viscometer, the apparent viscosity is determined by rotating a cylinder- or disk (or disc)-shaped spindle, as shown in *Figure 1* and *Figure 2*, respectively, immersed in a large volume of liquid.

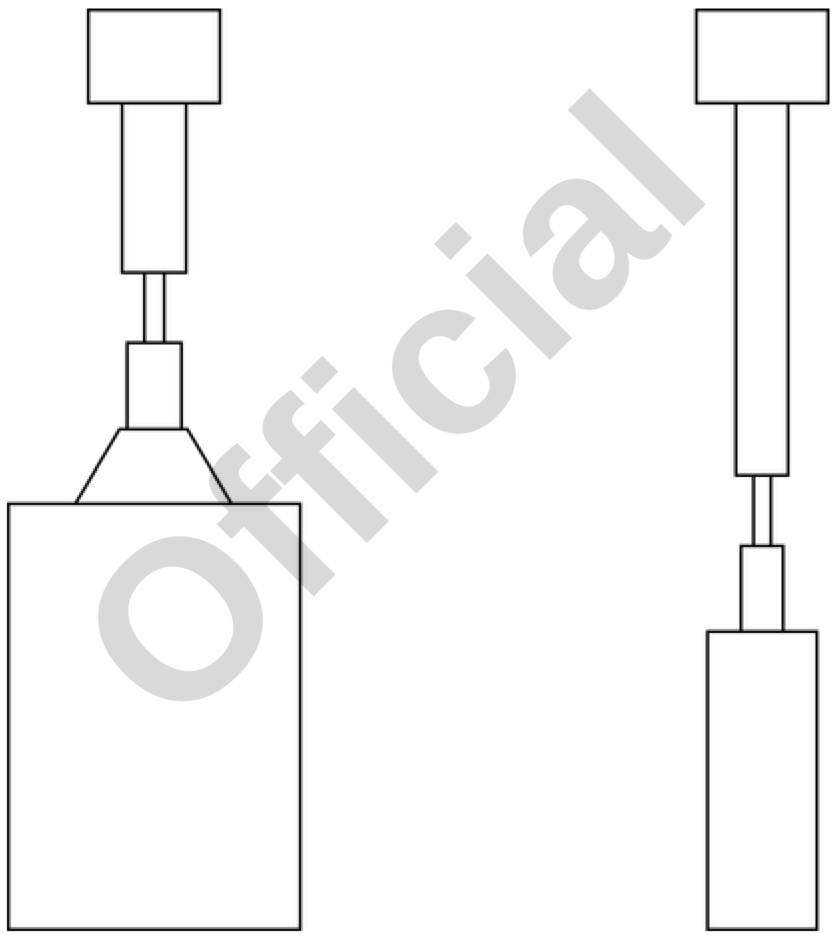

Figure 1. Cylinder-shaped spindles.

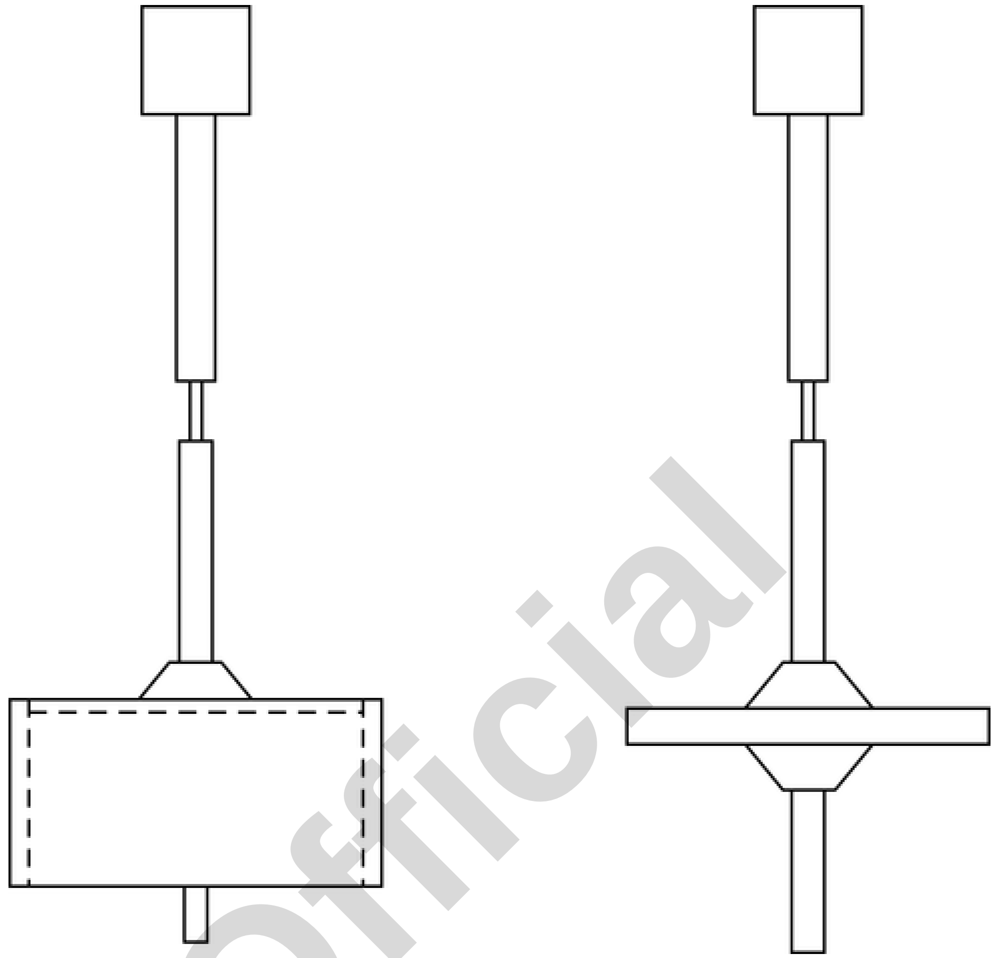

Figure 2. Disk-shaped spindles.

An absolute viscosity cannot be calculated due to the large gap between the spindle and the container wall, or the geometry of the spindle. The torque to maintain a given angular velocity does give a measure of the liquid resistance to flow but is often described as an apparent viscosity.

Other spindle type viscometers may be used provided that the accuracy and precision is NLT that obtained with the viscometers described in this chapter.

Procedure: When viscosity measurement is performed in a beaker or cup, as the shear rate is unknown, in order to enable reproducibility among labs that measure viscosity using different instrumentation, these parameters must be reported along with the measured viscosity:

1. Size and geometry of spindle
2. Angular velocity or rotational speed of the spindle
3. Temperature of the test substance

The spindle should be immersed to the recommended depth maintaining at least 1 cm clearance from the bottom and side of the container.

The preparation of the test specimen, including its temperature equilibration, is specified in each individual monograph. Follow the instrument manufacturer's recommendations regarding sample loading, spindle selection, and viscometer operation.

Calibration check: Check the calibration of a particular viscometer configuration at the test temperature using one or more fluids of known viscosities (Newtonian viscosity standards). The viscosity values of the calibration standards should bracket the expected viscosity value of the sample liquid. [Note—To help verify the linearity of the apparatus, it is suggested to perform measurements of a Newtonian viscosity standard at multiple rotational speeds at the test temperature.]

A viscometer is deemed to be calibrated if the measured apparent viscosities are within ±5% of the stated values.

Generally, calibration, operation, and cleaning of the viscometer should be performed according to the recommendations of the instrument manufacturer.

- **Method II. Concentric Cylinder Rheometers**

Apparatus: In the concentric cylinder rheometer, the apparent viscosity is determined by placing the liquid in the gap between the inner cylinder and the outer cylinder. Both controlled-stress and controlled-rate rotational rheometers are available commercially in configurations with absolute geometries (e.g., very small annular gaps between concentric cylinders) that allow calculation of apparent viscosities for non-Newtonian fluids. Controlled shear stress rheometers measure the shear rates resulting from the application of a given force or torque (the stress). Controlled shear rate rheometers measure the shear stress (from the torque on the rotor axis) resulting from a given shear rate (or rotational speed). Concentric cylinder rotational rheometers are sometimes referred to as cup-and-bob rheometers. These rheometers involve an additional design consideration depending on whether the outer cylinder (the cup) or the inner cylinder (the bob) rotates. Rotating-cup rheometers are called Couette systems, while rotating-bob rheometers are called Searle systems, as shown in *Figure 3* and *Figure 4*, respectively.

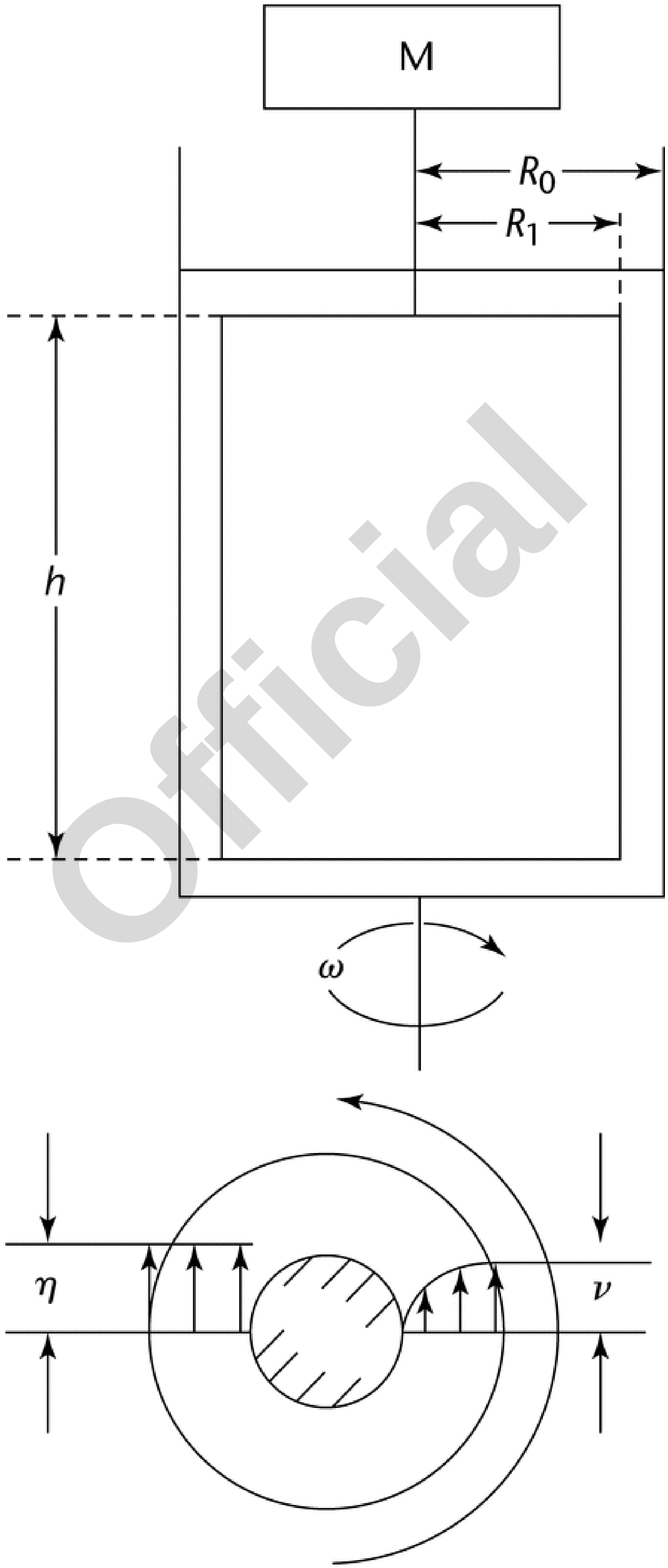

Figure 3. Couette concentric cylinder system for rotational rheometry.

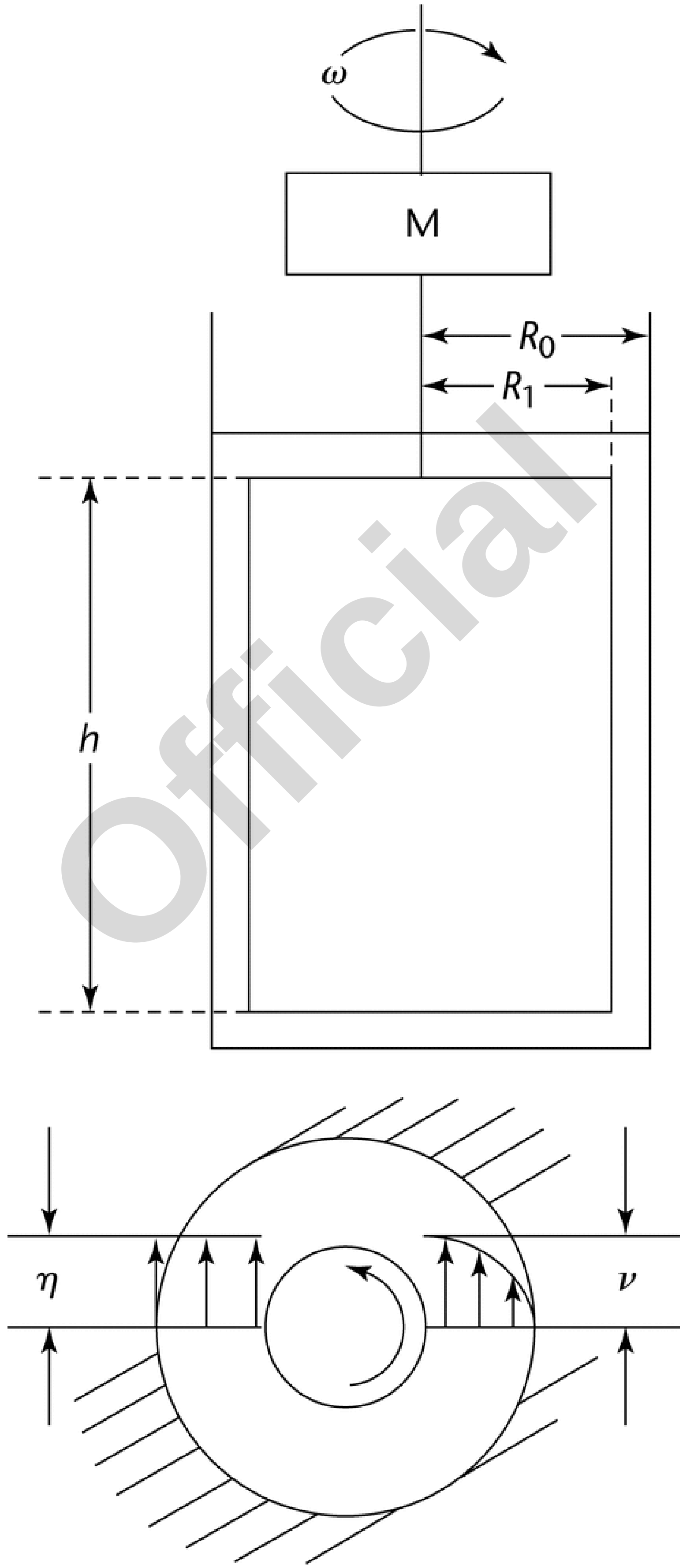

Figure 4. Searle concentric cylinder system for rotational rheometry.

Variables in *Figure 3* and *Figure 4* are defined as:

M = torque acting on the cylinder surface (N · m)
R_0 = radius of the outer cylinder (m)
R_1 = radius of the inner cylinder (m)
h = height of immersion of the inner cylinder in the liquid medium (m)
ω = angular velocity (radians/s)
η = viscosity (Pa · s)
v = velocity (m/s)

Procedure: Place a sufficient quantity of test fluid in the rheometer, and allow the sample to reach thermal equilibrium, as indicated in the individual monograph. Operate the rheometer following the procedure recommended by the instrument manufacturer. For non-Newtonian systems, the monograph indicates the type of rheometer that should be used and the shear rate(s) at which the measurements should be made. Determine apparent viscosities by changing the shear rate (or shear stress, if using a controlled shear stress rheometer) over a range appropriate to the use of the material under test. From a series of such viscosity measurements, the relationship between the shear rate and the shear stress of a non-Newtonian liquid can be obtained.

• **Method III. Cone-and-Plate Rheometers**

Apparatus: In the cone-and-plate rheometer, the liquid is introduced into the fixed gap between a flat disk or plate and a cone forming a defined angle. The angle of the cone ensures a constant shear rate due to the increase in both the gap and the linear speed as the distance increases from the origin. Viscosity measurement can be performed by rotating the cone or the plate, as shown in *Figure 5* and *Figure 6*, respectively. [Note—Because the volume of sample is small, even a small absolute loss of solvents can cause a large percentage change in viscosity. Such a loss is particularly relevant for volatile solvents but could be significant even for nonvolatile solvents such as water.]

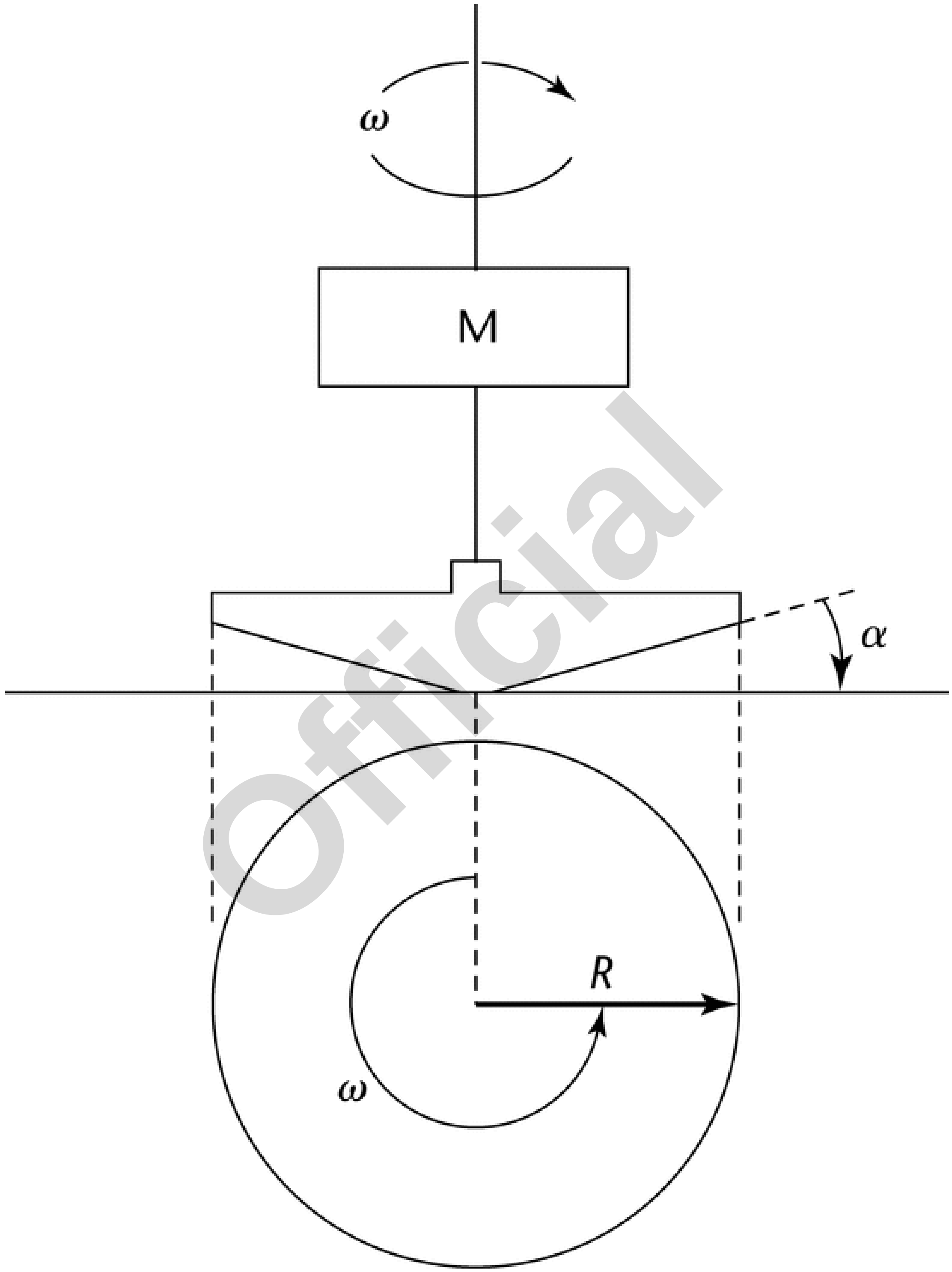

Figure 5. Cone-and-plate rotational rheometer with rotating cone.

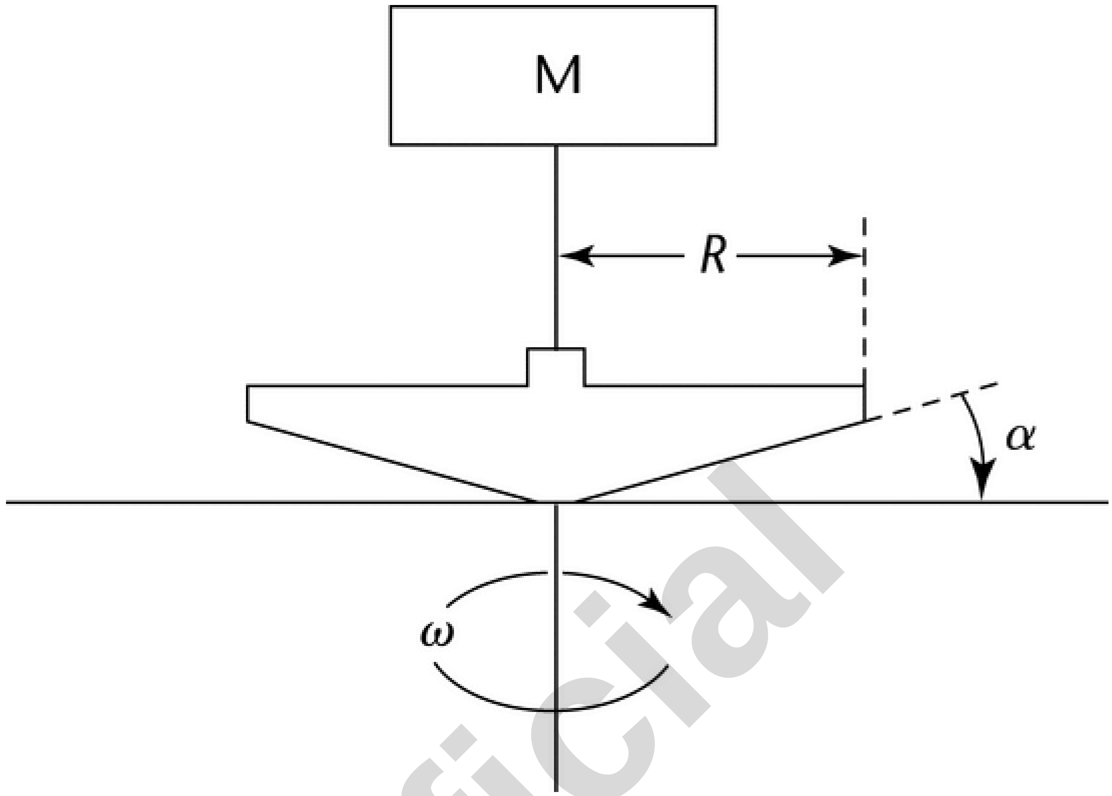

Figure 6. Cone-and-plate rotational rheometer with rotating plate.

Variables in *Figure 5* and *Figure 6* are defined as:

ω = angular velocity (radians/s)
M = torque acting on the flat plate or cone surface (N · m)
α = angle between the flat plate and cone (radians)
R = radius of the cone (m)

Procedure: Proceed as directed for *Method II. Concentric Cylinder Rheometers*.

• **Method IV. Parallel Plate (or Parallel Disk) Rheometers**

Apparatus: Parallel plate rheometers are similar to cone-and-plate rheometers except that the sample to be measured is introduced into the gap between a flat plate or disk (or disc) and another parallel flat plate or disk. Measurements are made typically with the lower plate or disk remaining stationary as the upper plate or disk is rotated at a constant angular velocity, ω (*Figure 7*).

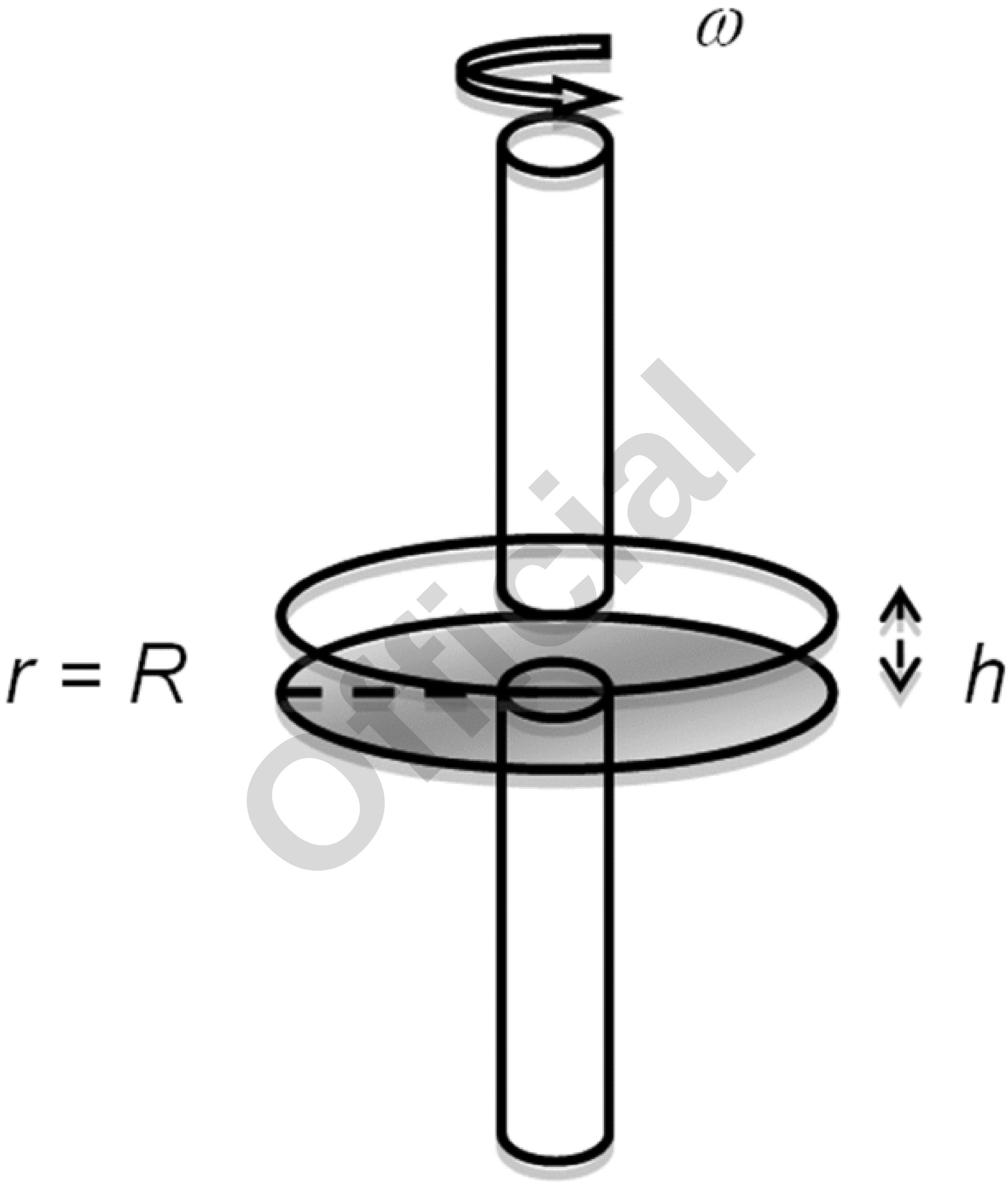

Figure 7. Parallel plate rotational rheometer.

Variables in *Figure 7* are defined as:

ω = angular velocity (radians/s)

R = radius of the plate (m)

h = distance between the two parallel plates (m)

In contrast to the cone-and-plate rheometer, the shear rate between parallel plates increases with distance from the origin of the axis of rotation due to the increasing linear speeds for a given angular velocity with a constant gap. Thus, an average shear rate is obtained. Nonetheless, some advantages of the parallel plate rheometer include ease of sample loading (especially for very viscous liquids and soft semisolids), and suitability for suspensions of particulates. For suspensions, the gap should be set high enough to avoid grinding particles between the plates. Parallel plates have a user-definable gap (within practical limits) and therefore, in the absence of large particles, may be used at narrower gaps. As with cone-and-plate rheometers, evaporative loss of solvent can markedly affect the viscosity of the sample, so adequate precautions need to be taken to minimize solvent loss.

Procedure: Proceed as directed for *Method II. Concentric Cylinder Rheometers*.