ARES-G2

Partitioned Plate Accessory

Getting Started Guide

Revision B **Issued November 2013**

Notice

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Introduction

Important: TA Instruments Manual Supplement

Please click the TA Manual Supplement link to access the following important information supplemental to this Getting Started Guide:

- TA Instruments Trademarks
- TA Instruments Patents
- Other Trademarks
- TA Instruments End-User License Agreement
- TA Instruments Offices

Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions. In the body of the manual these may be found in the shaded box on the outside of the page.

NOTE: A NOTE highlights important information about equipment or procedures.

CAUTION: A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.

UNE MISE EN GARDE met l'accent sur une procédure susceptible d'endommager l'équipement ou de causer la perte des données si elle n'est pas correctement suivie.

A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

Un AVERTISSEMENT indique une procédure qui peut être dangereuse pour l'opérateur ou l'environnement si elle n'est pas correctement suivie.

Regulatory Compliance

Safety Standards

For Canada

CAN/CSA-C22.2 No. 61010-1 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements.

CAN/CSA-C22.2 No. 61010-2-010 Particular requirements for laboratory equipment for the heating of materials.

For European Economic Area

(In accordance with Council Directive 2006/95/EC of 12 December 2006 on the harmonization of the laws of Member States relating to electrical equipment designed for use within certain voltage limits.)

EN 61010-1:2001 Safety requirements for electrical equipment for measurement, control, and laboratory use, Part 1: General Requirements + Amendments.

EN 61010-2-010:2003 Particular requirements for laboratory equipment for the heating of materials + Amendments.

For United States

UL61010-1:2004 Electrical Equipment for Laboratory Use; Part 1: General Requirements.

Electromagnetic Compatibility Standards

For Australia and New Zealand

AS/NZS CISPR11:2004 Limits and methods of measurement of electronic disturbance characteristics of industrial, scientific and medical (ISM) radio frequency equipment.

For Canada

ICES-001 Issue 4 June 2006 Interference-Causing Equipment Standard: Industrial, Scientific, and Medical Radio Frequency Generators.

For the European Economic Area

(In accordance with Council Directive 2004/108/EC of 15 December 2004 on the approximation of the laws of the Member States relating to electromagnetic compatibility.)

EN61326-1:2006 Electrical equipment for measurement, control, and laboratory use-EMC requirements-Part 1: General Requirements. Emissions: Meets Class A requirements per CISPR 11. Immunity: Per Table 1 - Basic immunity test requirements.

For the United States

CFR Title 47 Telecommunication Chapter I Federal Communications Commission, Part 15 Radio frequency devices (FCC regulation pertaining to radio frequency emissions).

Safety

WARNING: The operator of this instrument is advised that if the equipment is used in a manner not specified in this manual, the protection provided by the equipment may be impaired.

AVERTISSEMENT: L'utilisateur de cet accessoire est prévenu qu'en cas d'utilisation contraire aux indications du manuel, la protection offerte par l'équipement peut être altérée.

There are several major areas of concern pertaining to personal safety when using the Partitioned Plate Accessory. Please refer to the sections below.

Required Equipment

While operating this accessory, you must wear eye protection that either meets or exceeds ANSI Z87.1 standards. Additionally, wear protective clothing that has been approved for protection against the materials under test and the test temperatures.

Accessory Symbols

The following label is displayed on the APS for your protection:

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide in its entirety. This guide contains important warnings and cautions related to the installation, operation, and safety of the accessory. Ce symbole indique que vous devez lire entièrement ce guide de démarrage. Ce guide contient d'importants avertissements et mises en garde relatifs à l'installation, à l'utilisation et à la sécurité de l'accessoire.
	This symbol indicates that a hot surface may be present. Do not touch this area or allow any material that may melt or burn to come in contact with this surface. Ce symbole indique la présence possible d'une surface chaude. Prenez soin de ne pas toucher cette zone ou de laisser un matériau susceptible de fondre ou de brûler entrer en contact avec cette surface chaude.

Please heed the warning labels and take the necessary precautions when dealing with these areas. The *ARES-G2 Partitioned PLate Accessory Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Thermal Safety Warnings

WARNING: The material used for the partitioned plate is stainless steel. When operated at high temperature, the upper geometry support will heat up. Use protective gloves when removing the geometry ring support for cleaning purposes.

AVERTISSEMENT: Le matériau utilisé pour la plaque cloisonnée est l'acier inoxydable. Utilisé à haute température, le support de géométrie supérieur se réchauffe. Utilisez des gants de protection lorsque vous retirez le support annulaire de la géométrie pour le nettoyer.

WARNING: Take adequate precautions prior to heating of materials if it can lead to explosion, implosion, or the release of toxic or flammable gases.

AVERTISSEMENT: Prenez des mesures de précaution adéquates avant de chauffer des matériaux, si cela peut entraîner l'explosion, l'implosion ou le dégagement de gaz toxiques ou inflammables.

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Chapter 1:

Introducing the Partitioned Plate

Overview

The partitioned plate test fixture (shown below) is a special geometry for use with the ARES-G2 rheometer. The partitioned plate extends the strain range of rotational rheometers to higher strains in both oscillation and transient tests by delaying the effect of sample fracturing at large deformation.

The partitioned plate geometry consists of an inner plate geometry attached to the torque/force transducer and a stationary outer protection ring, separated from the inner plate by a gap of 0.2 mm. The external ring provides a more rigorous boundary condition at the outer radius of the inner plate, which consequently results in better reproducibility, and which also delays sample fracturing. No sample trimming is required since the torque generated in the outer ring section does not contribute to the calculation of the sample shear stress. The partitioned plate can be combined with any lower plate and cone > 25 mm.

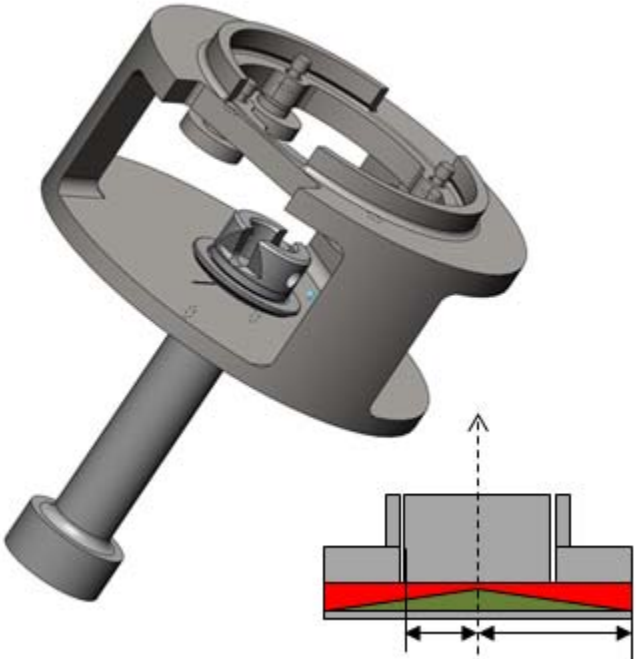


Figure 1 Partitioned plate for ARES-G2 rheometer. The partitioned plate can be combined with any lower plate or cone.

The partitioned plate test fixture requires minimum alignment and can be easily removed for cleaning. The key applications are LAOS (Large Amplitude Oscillatory Shear) and transient (stress growth) measurements on highly viscous elastic fluids such as polymer melts.

Description of the Partitioned Plate

The partitioned plate itself consists of three parts: the center plate shaft (inner plate refers to the lower cross-section surface of the center plate shaft) connected to the torque and normal force transducer; the mounting bracket attached to the transducer base; and the adjustable outer ring cylinder (the outer ring refers to the low cross-section surface of the outer ring cylinder) attached to the mounting bracket. It is only necessary to align the ring with the center plate the first time the partitioned plate is installed on a new ARES-G2 rheometer.

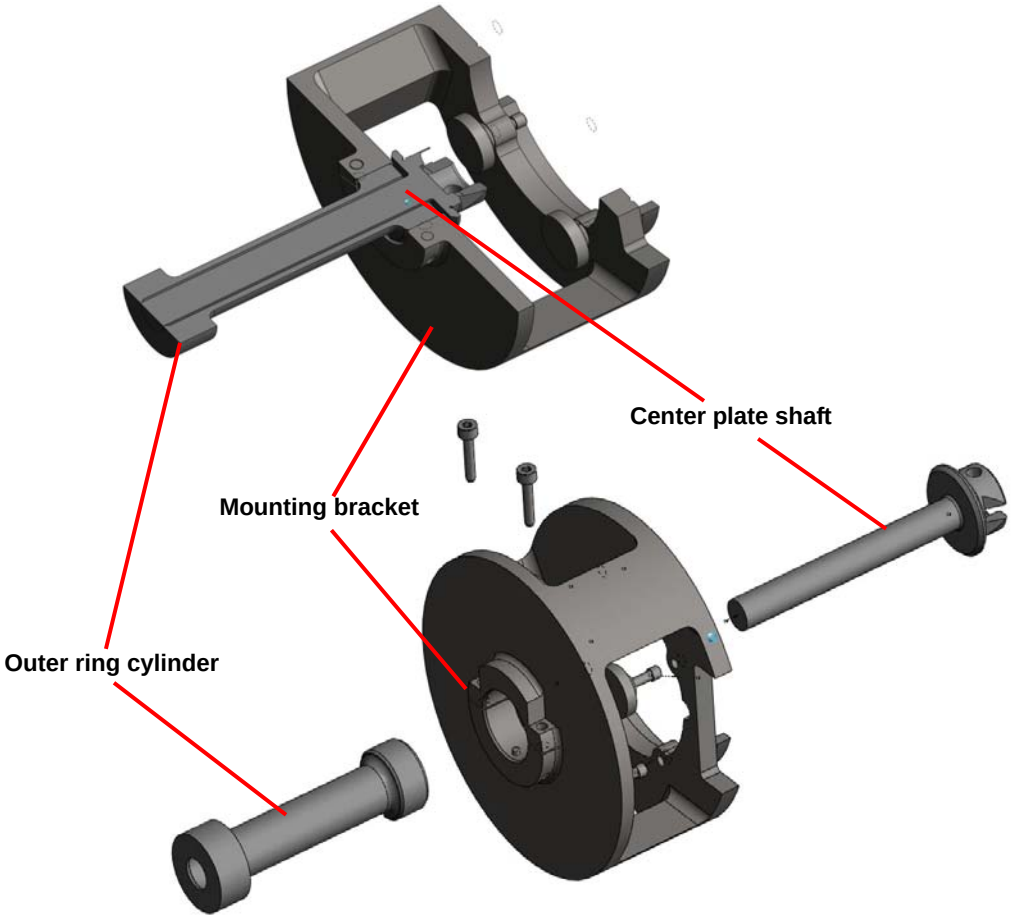


Figure 2 Section view and expanded view of the partitioned plate. The partitioned plate consists of three parts, the center plate shaft, the outer ring cylinder and the mounting bracket.

The section view in figure 2 shows the test fixture when assembled. The center plate shaft (plate diameter 10 mm) in the middle is to be attached to the torque transducer, and fits into the hollow outer ring cylinder. The outer ring cylinder slides up and down to allow alignment of the ring surface with the plate surface. The ring cylinder is secured in position with two M4 screws. It is crucial to have enough clearance between inner shaft and outer cylinder such that the torque reading at the center plate shaft is not affected.

The lower geometry can be any plate or cone geometry. The kit includes a 25 mm cone with a lip to avoid the sample dripping into the test chamber.

Figure 3 Lower cone with lip.

Application for LAOS Measurements

A key application of the partitioned plate is the performance of large amplitude oscillation shear (LAOS) experiments on highly viscous elastic fluids such as polymer melts. Most polymer melts show extensive secondary flow effects when transitioning from the linear to the non-linear regime. Secondary flow induces severe sample fracturing which starts at the sample rim and slowly propagates to the center of the plate. The partitioned plate cannot eliminate this effect, but delays the effect on the measurement as long as the fracturing has not propagated to the center plate.

Application for Transient Viscosity and Normal Force Measurements

Another major application of the partitioned plate is non-linear transient and flow testing. The partitioned plate geometry provides more consistent steady state test results at high strain. Note that in cone plate geometry, the normal thrust measurement is a result of the first and second normal stress difference.

Chapter 2:

Installing the Partitioned Plate

This chapter briefly describes the setup of the Partitioned Plate on the ARES-G2.

Installing the Partitioned Plate

1 Raise the stage and remove any installed geometry.

2 Remove the standard large thumbscrew from the anvil and replace it with the small thumbscrew included in the kit.

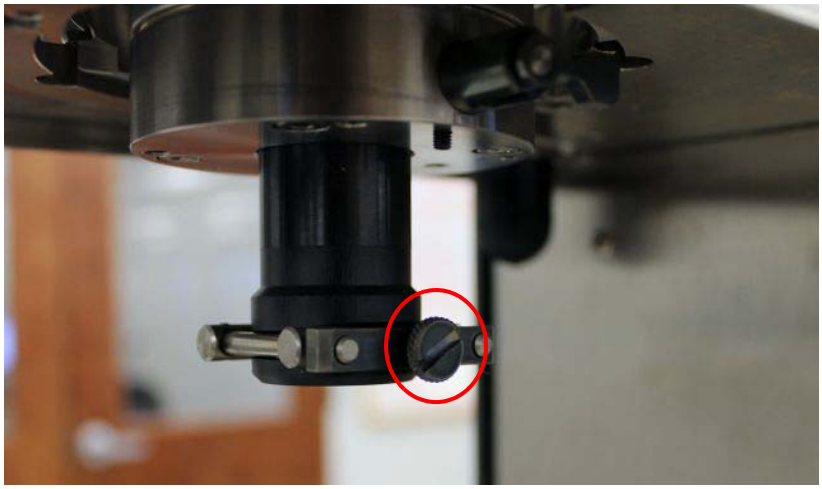


Figure 4 Anvil with a small thumbscrew.

3 Insert the 10 mm plate geometry and secure the geometry in place; hand-tighten the thumbscrew. Before installing, make sure there is no dirt or dust on the geometry location surface. Note that the 10 mm plate cannot be used together with the PRT sensor.

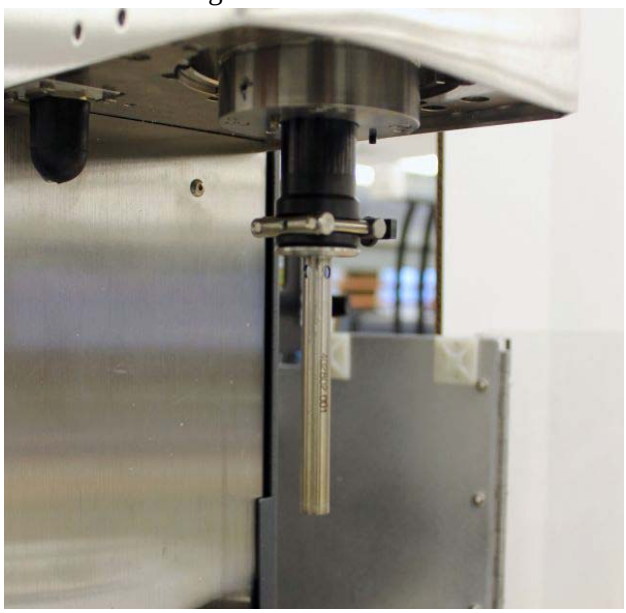


Figure 5 Center plate geometry installed.

4 Obtain the mounting bracket next and slide it over the plate shaft and anvil. The geometry has to be oriented with the cut-out to the right. Turn it such that it goes past the transducer anvil and position it at the transducer base. Hand-tighten the four captive thumbscrews.

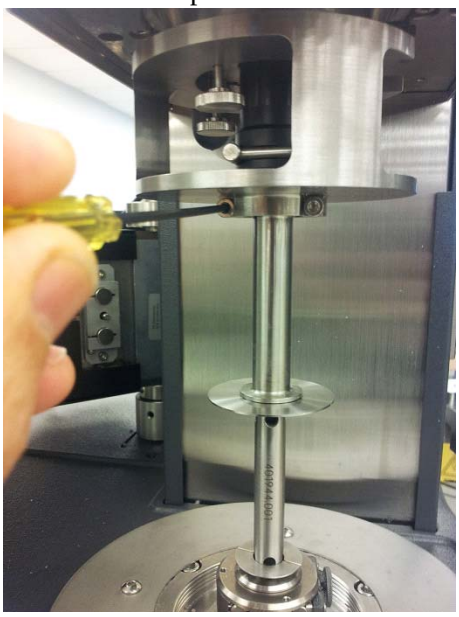
Figure 6 Installation of the mounting bracket.

5 Obtain the outer ring cylinder; slide it over the center plate shaft into the mounting bracket. Make sure the ring cylinder is oriented such that the pin on the bracket aligns with the slit in cylinder. Secure with the two M4 screws, the center plate emerging slightly.

Figure 7 Installing the outer ring cylinder.

6 Install a 25 mm (or larger) bottom plate. Manually bring the partitioned plate down until the upper and lower plates are in contact. Apply a small axial force (50 g).

7 Loosen the two M4 screws on the mounting bracket. Press the outer ring slightly and evenly by hand onto the bottom plate to align the ring and plate. Retighten the two clamping screws evenly. Raise the head with the partitioned plate geometry and check the alignment with a mirror. The plate has to be centered. Check the torque reading. The torque transducer must not show any hysteresis when deflecting the center shaft plate in clockwise and counter-clockwise direction.



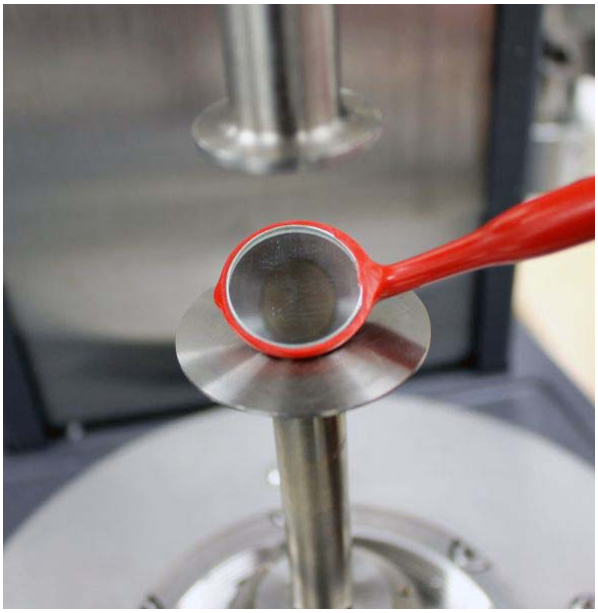


Figure 8 Secure outer ring cylinder after alignment of the plate and ring. Check alignment with a mirror.

8 Remove the bottom plate and install the 25 mm, 0.1 rad cone with lip, included in the set. Zero the geometry.

NOTE: If the partitioned plate is used in conjunction with a lower plate, the gap has to be set before aligning the ring and the plate. Since the diameter of the lower plate is at least 25 mm, the center plate when perfectly aligned cannot measure the compression force at contact.

9 Close the forced air convection oven. The ceramic of the oven will embrace the upper geometry shaft tightly; the lower geometry shaft has to remain free.

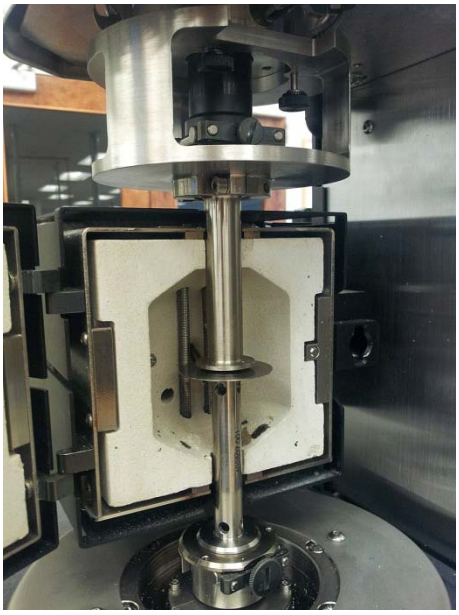


Figure 9 Partitioned plate geometry installed with oven partially closed.

NOTE: If the oven ceramic does not fit around the shaft of the upper geometry, don't force the oven in position – realign the oven.

CAUTION: Closing the oven with force will damage the delicate oven ceramics.

MISE EN GARDE: La fermeture du four avec force endommage les délicates céramiques du four.

Setting up the Partitioned Plate in TRIOS

The partitioned plate is set up in TRIOS Software as a parallel plate or cone-plate geometry. Follow the standard procedure to set up a geometry; see “Configuring a New Geometry” in TRIOS Help.

The specifications for the center plate shaft 402802.001 and the lower 25mm cone with lip 402807.001 are:

- Inertia of the plate shaft: 2.629 µNms^2
- Compliance of the plate shaft: 1.34 mrad/Nm$^{(+)}$
- Inertia of the 25 mm cone with lip: 4.567 µNms^2
- Compliance of the lower plate: 0.705 mrad/N$^{(+)}$
- Nominal diameter of the plate: 10mm.
- Nominal angle of the cone: 0.1 rad
- $^{(+)}$ based on a shear modulus of 7.56E10 Pa for steel

NOTE: It is recommended to use the partitioned plate with torque and normal force transducer set to FRT.

Chapter 3:

Operating and Maintaining the Partitioned Plate

This chapter briefly describes how to align the plate and the outer ring. Note that the alignment of the center plate shaft and the outer ring cylinder surface has to be performed the first time the partitioned plate is installed on a new rheometer.

Loading a Sample

Before loading a sample, bring the FCO and the installed geometry up to test temperature. Preheat the oven for several hours in order to allow the instrument to stabilize at temperature. This is very crucial when cone-plate geometry is used. Note that the equilibrium time increases as the test temperature increases.

CAUTION: Do not use the partitioned plate at temperatures above 350°C.

MISE EN GARDE: N'utilisez pas la plaque cloisonnée à des températures supérieures à 350°C.

When operating at high temperature, the best approach is to mold a sample disc of 25 mm. Choose the thickness such, that a maximum of 10 to 20% excess material is squeezed out at geometry gap. There is no need to trim when the lower plate is larger than 25 mm to prevent the excess material from dripping.

NOTE: A nice symmetric rim delays the onset of sample fracturing. Make sure that the sample disc is centered properly during the loading procedure. Use a sample loading conditioning step in order to load the sample in a reproducible way.

Removing and Reinstalling the Outer Ring Assembly

During sample loading and also during test, it is unavoidable that a very small amount of sample is pushed into the gap between the center plate and the outer ring. This material will not interfere with the measurement as long as the transducer is operated in FRT (quasi-infinite stiff) mode. However in order to avoid degradation of the sample in the gap it will be necessary to remove this excess material from time to time. Also at the end of testing, the excess material has to be removed before the test geometry is cooled below the material's Tg ie. Tm to avoid seizing of the center plate geometry.

WARNING: When the materials under test solidifies at low temperature, cooling the test geometry without removing and cleaning the outer ring assembly can cause serious damage to the torque and normal force transducer.

AVERTISSEMENT: Lorsque les matériaux en cours d'essai se solidifient à basse température, refroidir la géométrie d'essai sans retirer et nettoyer l'ensemble de l'anneau externe peut provoquer de graves dégâts au couple et au transducteur de force normale.

In order to remove the outer ring assembly, proceed as follows:

1 Unload the sample and go to the loading position. Remove the test material with the copper scraper.

2 Loosen the 4 captive thumbscrews holding the mounting bracket to the transducer base. Do not loosen the two M4 screws, used to align outer ring cylinder.

3 Carefully remove the mounting bracket with the outer ring assembly. Leave the center plate geometry in place.

4 Use the square brass brush to clean the shaft of the 10 mm plate and the round brush to remove any excess material from the hollow ring cylinder before the material solidifies.

Figure 10 Cleaning of the partitioned plate geometry.

5 When all material is removed, re-mount the outer ring assembly and secure with the four thumbscrews at the transducer base. It is now safe to cool down the test geometry. It is not necessary to reset the gap when resuming testing as long as the center plate has not been removed.

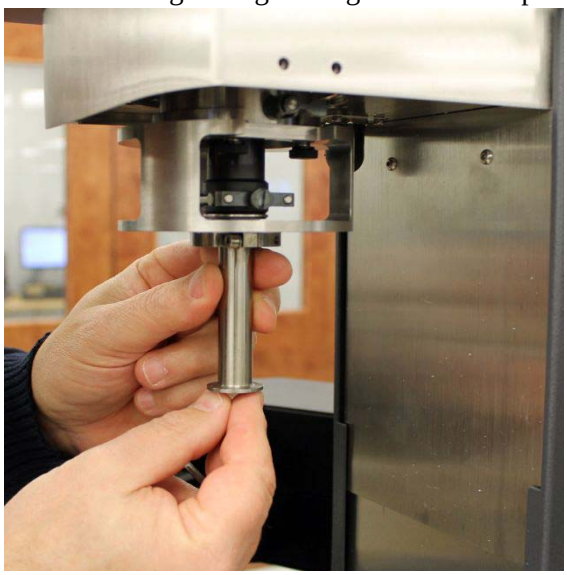


Figure 11 Reinstalling the mounting bracket with the outer ring cylinder.

Cleaning the Partitioned Plate

Polymer samples are tough to clean. Open the gap at test temperature. Use the copper spatula (included in the kit) to remove the sample from the plate at cone immediately after they have been separated. Remove the remaining polymer with a brass brush. Polish the plate surface with a cotton towel.

WARNING: Handling and cleaning hot test geometries can be dangerous. Always wear protective gloves before removing hot test geometries or cleaning hot surfaces.

AVERTISSEMENT: Manipuler et nettoyer les géométries d'essai chaudes peuvent être dangereux. Portez toujours des gants de protection avant de retirer des géométries d'essai chaudes ou de nettoyer des surfaces chaudes.

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