DHR Series and AR Series

Starch Pasting Cell

Getting Started Guide

Revision E Issued November 2013

Notice

The material contained in this manual, and in the online help for the software used to support this instrument, is believed adequate for the intended use of the instrument. If the instrument or procedures are used for purposes other than those specified herein, confirmation of their suitability must be obtained from TA Instruments. Otherwise, TA Instruments does not guarantee any results and assumes no obligation or liability. TA Instruments also reserves the right to revise this document and to make changes without notice.

TA Instruments may have patents, patent applications, trademarks, copyrights, or other intellectual property covering subject matter in this document. Except as expressly provided in written license agreement from TA Instruments, the furnishing of this document does not give you any license to these patents, trademarks, copyrights, or other intellectual property.

TA Instruments Operating Software, as well as Module, Data Analysis, and Utility Software and their associated manuals and online help, are proprietary and copyrighted by TA Instruments. Purchasers are granted a license to use these software programs on the module and controller with which they were purchased. These programs may not be duplicated by the purchaser without the prior written consent of TA Instruments. Each licensed program shall remain the exclusive property of TA Instruments, and no rights or licenses are granted to the purchaser other than as specified above.

TA Instruments — Waters LLC
159 Lukens Drive
New Castle, DE 19720

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.

MISE EN GARDE: 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.

UL61010A-2-010:2002 Particular requirements for laboratory equipment for the heating of materials + Amendments.

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

Do not attempt to service this cell, as it contains no user-serviceable components.

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.

Instrument Symbols

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

Symbol	Explanation
	This symbol indicates that a hot surface may be present. Take care not to touch this area or allow any material that may melt or burn come in contact with this hot 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. This *Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Table of Contents

Chapter 1:

Introducing the Starch Pasting Cell (SPC)

Overview

This manual is intended as an addendum to the DHR and AR-G2/AR 2000ex/AR 1500ex Rheometer Getting Started Guides.

The Smart Swap™ Starch Pasting Cell (SPC) is designed to measure the viscosity of starch using methods currently in use in the starch industry.

Starch is used as a rheology modifier in many foodstuffs. As a raw material, it is normally supplied in the form of approximately spherical granules, with diameters between about 1 µm and 100 µm. To be used as a rheology modifier, starch must undergo gelatinization, which occurs when the granules are disrupted in aqueous environments, to swell and eventually release the constituent molecules. The temperature at which this process commences will depend on the nature of the starch, but it is usually approximately 60°C. The process is thermally irreversible, and when the temperature is reduced, the system remains gelatinized, although some slow de-gelatinization (retrogradation) may occur.

The precise gelatinization temperature, the time taken for the process to go to completion, and the rheological properties of the gelatinized starch, will depend on the source of the starch, any physical and chemical modifications made, the pH of the aqueous phase, and so on. Test methods have been developed in the starch industry allowing the rapid testing of starches. The general procedure is as follows:

1 Dispersal of a specified quantity of starch in water or buffer solution at low temperature

2 Raising the temperature rapidly to above the gelatinization temperature

3 Holding the temperature for a fixed period

4 Reducing the temperature to below the gelatinization temperature

5 Holding the temperature for a further fixed period

Stirring is carried out during the entire process. The stirring is necessary in the pre-gelatinization stage to prevent the starch particles from sedimenting. In the post-gelatinization stages, it is used to ensure good mixing.

System Components

The SPC consists of four components:

- The *jacket*, which is mounted on the instrument base
- The *cup*, which is removable and inserts into the jacket
- The *cap*, which screws onto the jacket to hold the cup in place
- The *impeller*, which attaches to the instrument spindle

These are shown in the figures below.

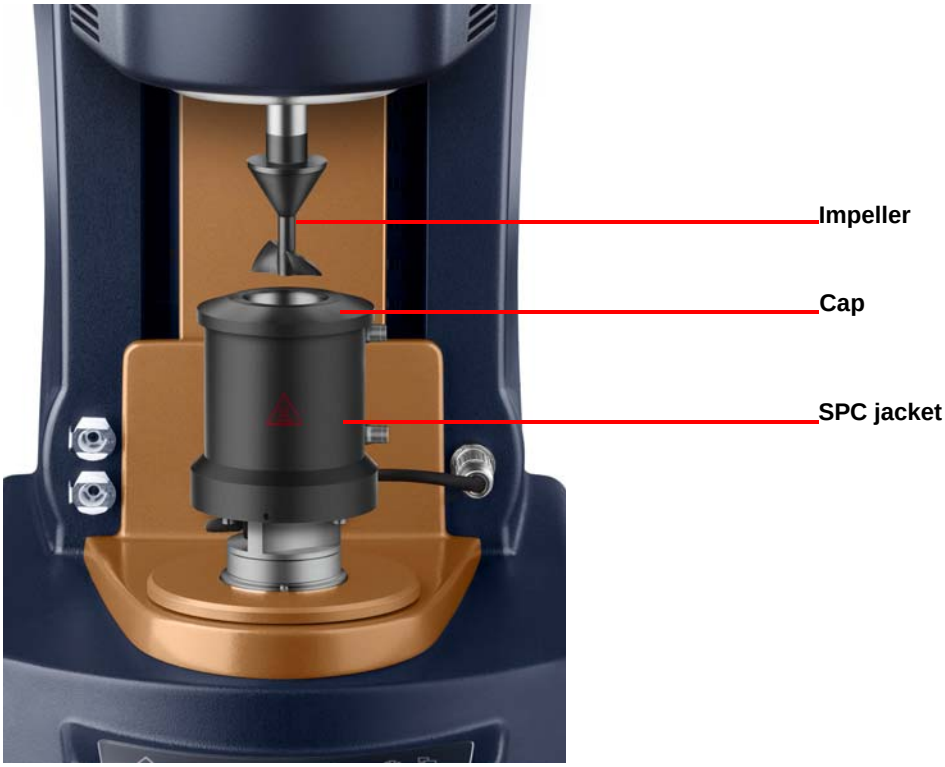

Figure 1 SPC properly mounted on DHR Rheometer.

Figure 2 The cup (left), cap (center), and impeller (right).

Temperature Control

Heating is accomplished through the electrical elements placed within the jacket, concentrically to the cup. The sample is cooled using water carried in a helical conduit in close proximity to the cup outer walls. The cooling water flow is controlled through the Cooling Control Unit (CCU), which is placed upstream of the cup.

Figure 3 Cooling Control Unit (CCU).

Purge air is used to displace the water from the conduit during heating. The temperature is read by a Pt 100 probe in close thermal contact with the cup bottom. The impeller shaft is designed to prevent significant solvent evaporation.

NOTE: If the geometry gap is unreferenced (**"**unknown**"**) or greater than 50,000 microns, the temperature is measured by a probe located in the jacket, rather than by the probe in contact with the cup, as it otherwise would be.

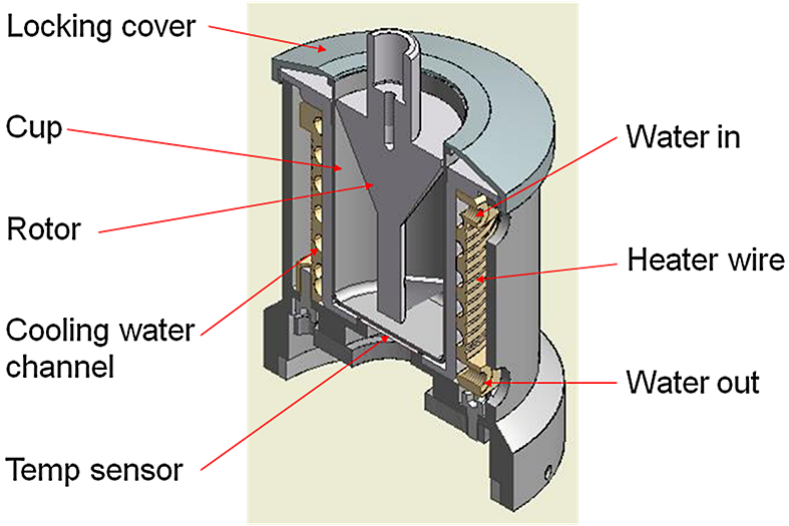

Figure 4 Cross-section of CCU.

Chapter 2:

Installing and Setting Up the SPC System

Follow the instructions in this chapter to install and set up the SPC system.

Installing the Starch Pasting Cell

Follow the instructions below to properly install the SPC on the rheometer. The SPC is a standard Smart Swap™ accessory and is mounted/uninstalled on the rheometer as such.

1 Use the Smart Swap™ connector to mount the fixture on the rheometer.

2 Plug the SPC accessory cable into the rheometer accessory cable port.

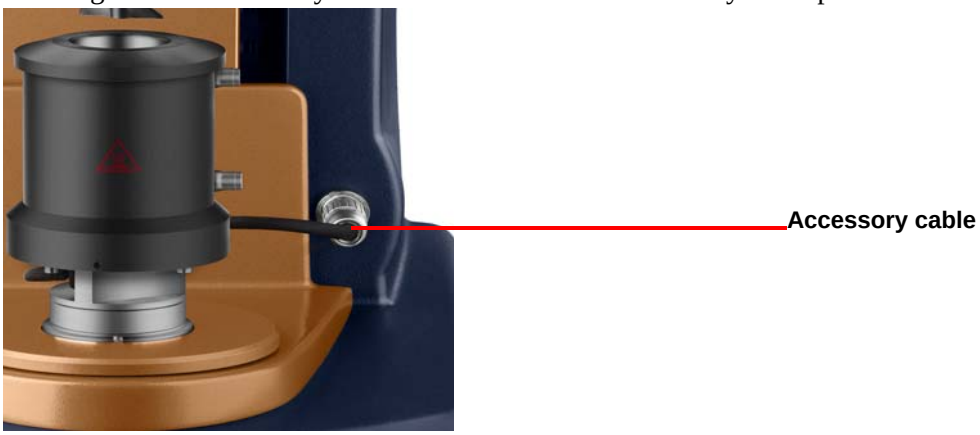

Figure 5 SPC installed on DHR Rheometer.

Assembling the Starch Cell

Follow these instructions to assemble the SPC:

1 Raise the instrument head.

2 Insert the cup into the jacket, as shown below.

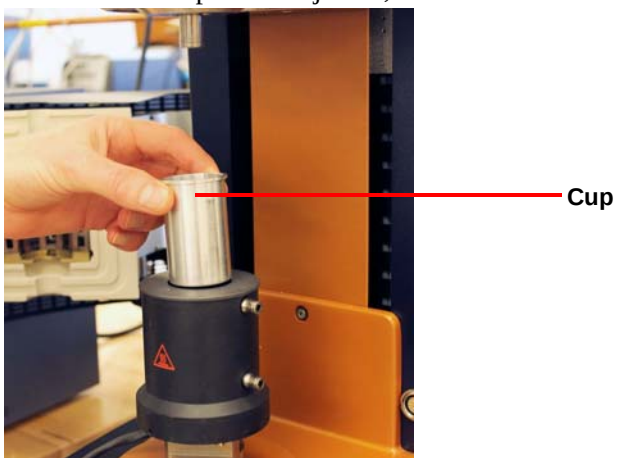

Figure 6 Inserting the cup into the jacket (DHR Series shown).

3 Screw the cap onto the jacket, as shown below.

Figure 7 Fitting the cap onto the jacket (DHR Series shown).

CAUTION: To avoid damaging the threads, do not over tighten. The purpose of the cap is to prevent rotation of the cup and to ensure good thermal contact between the base of the cup and the temperature sensor. Light finger tightness should be sufficient to achieve this.

MISE EN GARDE: Pour éviter d'endommager les filetages, ne serrez pas trop. Le but du bouchon est d'empêcher la rotation de la coupelle et d'assurer un bon contact thermique entre la base de la coupelle et le capteur de température. Un serrage léger à la main devrait suffire pour cela.

Attaching the Impeller

Install the impeller on the rheometer in the same manner you would any standard geometry.

Setting the Geometry Details

For information regarding setting up the SPC geometry in TRIOS software, see TRIOS Help topic *Creating the Starch Pasting Cell Geometry in TRIOS.*

Connecting the Cooling Control Unit (CCU)

The flow of the cooling water and purge air is controlled through a CCU, shown below. This unit may be free standing, or wall mounted using the clearance holes on top of the unit.

Figure 8 Cooling Control Unit (CCU).

Connect the CCU as follows:

1 Connect the air supply to the **GAS IN** port using the 8-mm outer diameter tubing.

2 Connect the water supply to the **LIQUID IN** port using the 6-mm outer diameter tubing. If mains water is used as the supply, then the non-return valve (shown below) should be placed in the line upstream of the CCU.

Important: Note the direction of flow through this valve.

Figure 9 Non-return valve (for use with mains water supply only).

3 Connect the **GAS/LIQUID** outlet port to the upper port on the Starch Cell jacket, using the 4-mm outer diameter tubing with the 4-mm to 6-mm adapter provided.

4 Using the 4-mm outer diameter tubing connect the lower port on the Starch Cell jacket to the drain, if the mains water is the supply, or to the return, if a fluid circulator is used.

5 Connect the **EVENT B** socket on the rear of the AR Rheometer to the **EVENT** socket on the CCU using only the cable.

6 Set the air flow rate to 1 L/min. Note that the reading is taken from the center of the float. To set the flow rate, it may be necessary to raise the temperature of the Starch Cell using the instrument software to ensure continuous flow.

Setting the Cooling Water Parameters

For optimum temperature control, set the parameters for the cooling water from within TRIOS Instrument Control Software.

For more information, see *Setting the Cooling Water Parameters for the AR Starch Pasting Cell* TRIOS Help topic.

Chapter 3:

SPC Use and Maintenance

Running an SPC Procedure

Perform the following steps to load your sample, run the experiment, then remove the sample:

1 Raise the instrument head to the full back off distance.

2 Remove the Starch Cell cap and cup from the jacket. See Chapter 3 for information, if needed.

3 Add the ingredients to the cup, and reassemble the Starch Cell.

4 Send the instrument to the correct geometry gap.

5 Start the procedure. Wait until the experiment is complete.

6 Remove the sample by raising the instrument head to the full backoff distance.

NOTE: If the final viscosity of the sample is high, it may be necessary to release the impeller before raising the head. To do this, hold the impeller shaft firmly, and rotate the drawrod counterclockwise (anticlockwise).

Maintaining the SPC

Bearing Friction Correction

The friction due to the rheometer air bearing is negligible except when very low viscosity samples are used, and no correction is normally necessary when using the Starch Cell. However, if you do wish to introduce a correction for bearing friction, the procedure is available through the instrument control software. See TRIOS software Online Help for details.

Rotational Mapping

Rotational mapping is used on the TA Instruments' AR Series Rheometers to ensure optimal performance for very sensitive samples. It should not normally be necessary with the Starch Cell, but may be used if very precise data are required at low angular velocities. See TRIOS software Online Help for details.

Index

C

I

L

N

P

R

S

T

W