DHR Series

Immobilization Cell

Getting Started Guide

Revision D Issued November 2014

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.

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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
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Notes, Cautions, and Warnings

This manual uses NOTES, CAUTIONS, and WARNINGS to emphasize important and critical instructions.

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 instrument, as it contains no user-serviceable components.

Required Equipment

While operating this instrument, 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 instrument for your protection:

Symbol	Explanation
	This symbol indicates that you should read this Getting Started Guide for important safety information. This guide contains important warnings and cautions related to the installation, operation, and safety of the instrument. 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'instrument.
	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 to 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.
	This symbol indicates that you are advised to consult this manual for instructions. Ce symbole indique que nous vous recommandons de consulter ce manuel pour les instructions.

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.

Cautions and Warnings

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

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.

CAUTION: The Peltier Plate may be damaged by operating the instrument without a flow of water through the Peltier Plate. There is a Peltier overheat protection device that will activate if the device becomes too hot.

MISE EN GARDE: La plaque Peltier peut être endommagée si l'instrument est utilisé sans écoulement d'eau dans la plaque Peltier. Il existe un dispositif de protection contre la surchauffe qui s'active si le dispositif devient trop chaud.

Table of Contents

Chapter 1:

Introducing the Immobilization Cell

Overview

The Immobilization Cell is a Smart Swap™ accessory that can be used to investigate the immobilization kinetics of a coating material as its liquid component is drawn into a substrate by a vacuum. Measurements can be performed under different conditions of temperature, gap, pressure, and rheological deformation.

In the Immobilization Cell, a disk of the substrate under test is held in place by a clamping ring. A vacuum is created below the substrate, and any excess fluid not absorbed by the substrate is captured in a waste flask.

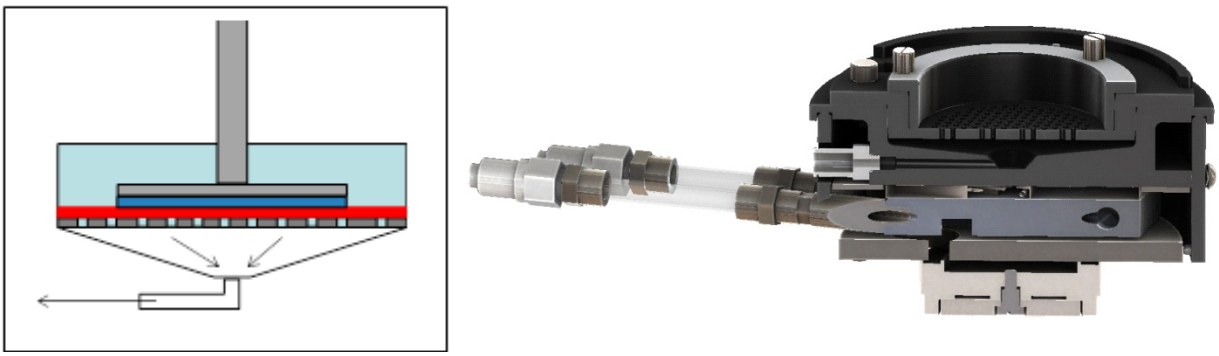

Figure 1

The temperature of the perforated platform is controlled by Peltier elements in the base of the unit. These heat or cool the specially shaped inner jacket, which transfers the temperature to the platform.

Figure 2

The vacuum is controlled using the event step in Trios (see on-line help) to activate a solenoid on the waste management manifold. The manifold also includes a valve that can be used to vent the cell and waste flask at the end of a test, or in conjunction with the gauge on your vacuum pump to meter the pressure in the cell during a test.

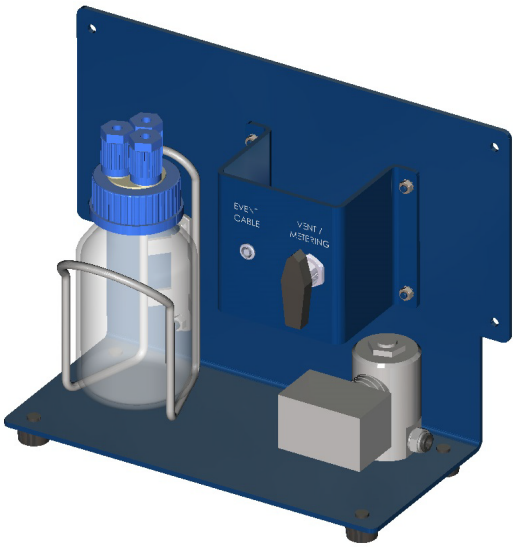

Figure 3

NOTE: A vacuum pump is not supplied as part of the system.

NOTE: You should consider installing a vapour trap to protect your vacuum pump and waste management manifold if the vapour extracted from your sample is likely to damage these components.

System Specifications

The tales below contains the technical specifications for the Immobilization Cell.

Table 1: Immobilization Cell Specifications

Item/Area	Specifications
Temperature Range Tap water supply Air cooled circulator (P/N 403209.901) Thermo Cube (P/N 403258.901) at -5°C (80% water/20% alcohol mixture)	 -10°C to 150°C 0°C to 150°C -10°C to 150°C
Ramp Rate	See section below.
Pt100 Internal Resolution	0.01°C
Vacuum	Pressure difference 0–85,000 Pa[1]
Cell Construction	Anodised Aluminum

1. Depends on capability of pump

CAUTION: The Peltier Plate may be damaged by operating the instrument without a flow of water through the Peltier Plate. There is a Peltier overheat protection device that will activate if the device becomes too hot.

MISE EN GARDE: La plaque Peltier peut être endommagée si l'instrument est utilisé sans écoulement d'eau dans la plaque Peltier. Il existe un dispositif de protection contre la surchauffe qui s'active si le dispositif devient trop chaud.

Ramp Rate

The maximum sustainable ramp rate will depend on a number of factors such as the start and end temperature and the temperature/flow rate of circulation fluid. To determine the maximum sustainable heating/cooling rate, perform the following test and analysis:

1 Equilibrate to start temperature.

Perform a time sweep or peak hold test with the temperature set (if possible) to a few degrees in excess of the end temperature. Set the time much longer than you expect; the test can be aborted when the temperature has reached a stable value.

2 Plot a graph of temperature vs. time (min) and take the derivative.

Inspect the derivative curve over your temperature range of interest. The maximum sustainable rate will be the lowest value on the derivative curve.

Chapter 2:

Installing the Immobilization Cell

The Immobilization Cell system consists of a temperature-controlled jacket, a perforated platform and substrate clamping ring. A manifold to control the vacuum and collect waste is connected to the cell. It is used in conjunction with an upper 50mm parallel plate geometry.

To install up the Immobilization Cell, follow these steps:

1 Raise the rheometer head to the top most position.

2 Press the Release button on the control panel, as shown below. A continuous green light indicates that the attachment can be fitted.

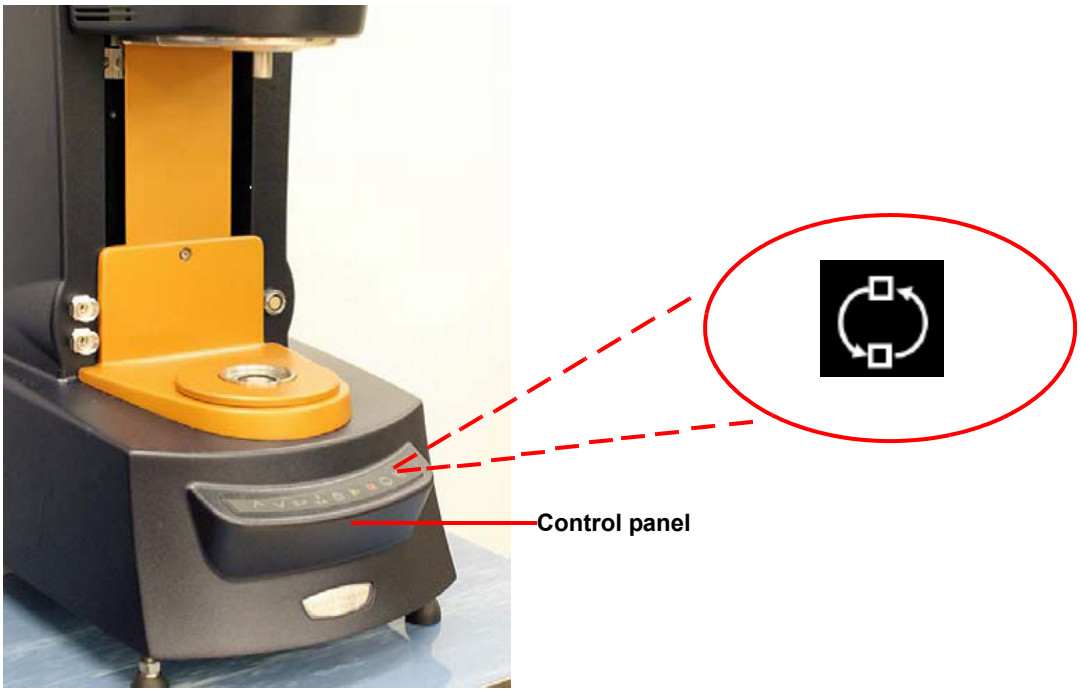

Figure 4 Control panel.

NOTE: The release state will only stay active for 10 seconds.

3 Fit the Immobilization Cell, ensuring it is aligned correctly.

Figure 5 Fitting the Immobilization Cell.

4 Connect the power cable and fluid hoses.

Figure 6 Connecting power cable and fluid hoses.

5 When the green status light turns off, the Immobilization Cell is correctly installed.

6 Attach the 50 mm Parallel Plate to the motor shaft.

7 Connect 6 mm pipe from the vacuum port on the side of the Immobilization Cell to one of the three inlets on the waste flask.

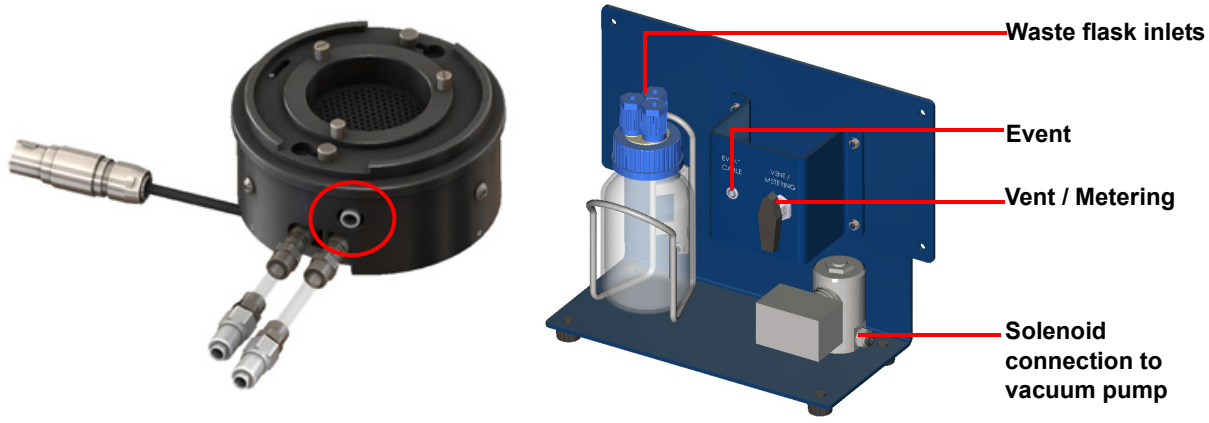

Figure 7 Vacuum port (left) and waste flask manifold (right).

8 Connect 6 mm tubing from one of the three inlets on the waste flask to the Vent fitting.

9 Connect 6 mm tubing from one of the three inlets on the waste flask to the left side of the solenoid.

10 Connect 6 mm tubing from the solenoid to your vacuum pump.

11 Connect the 4-pin event cable from Event B on the rear of the rheometer to the Event connection.

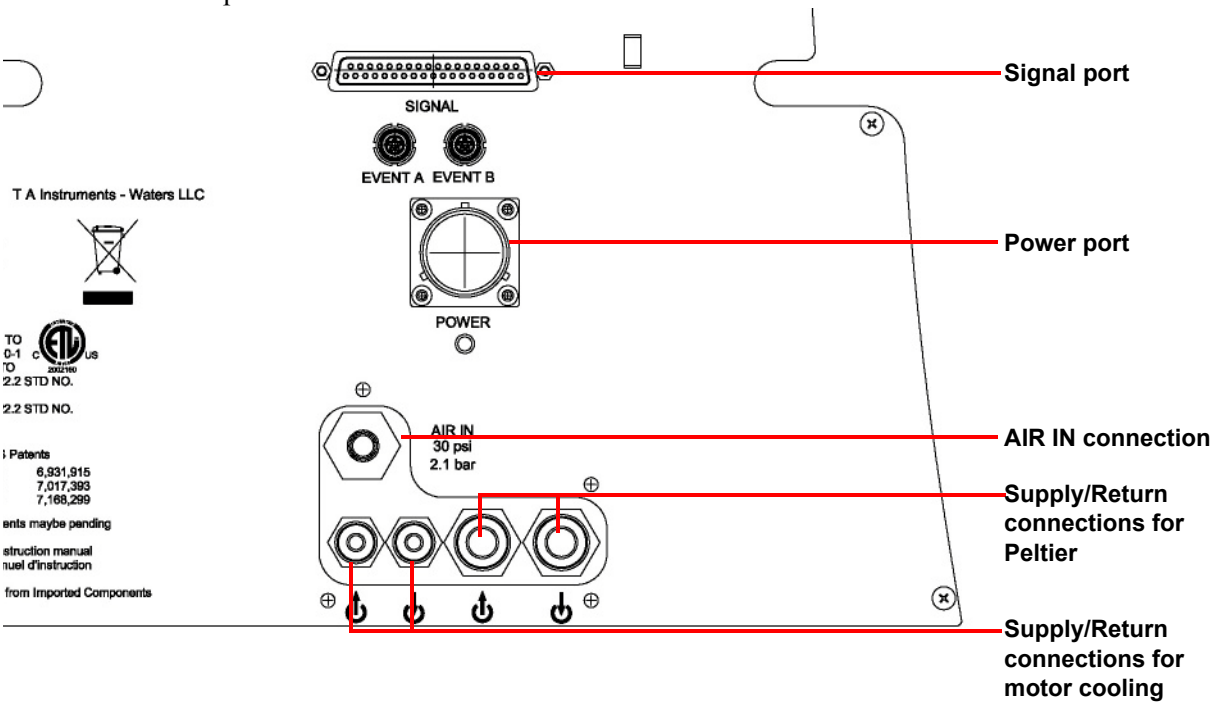

Figure 8 Cable connections on rheometer back panel.

Removing the Immobilization Cell

1 Disconnect the 6 mm tubing from the vacuum port on the Immobilization Cell.

2 Press the Release button on the control panel. A flashing green light indicates that the attachment can be unplugged. Refer to step 2 in the previous section for Release button location.

3 Press the Release button again. A continuous green light indicates that you can remove the Immobilization Cell.

4 Remove the Immobilization Cell from the rheometer.

Chapter 3:

Using the Immobilization Cell

1. Fit the perforated platform into the body of the cell aligning the slots in the platform with the peg and screw heads on the body. Then twist the platform clockwise and clamp in place using the two thumb screws.
2. Cut a 57 mm (2.25") disk of your substrate and place on the platform.
3. Insert the retaining ring and tighten using the 3 thumb screws.
4. In TRIOS, set the zero gap options to Axial force and set a force that overcomes any bow in your sample. This value can be determined by performing an axial compression test, plotting force vs. gap and picking a force above which the gap is invarient. The maximum allowable is 30N.
5. Zero the gap and raise the head to the loading position.
6. Design your experiment.
   - Use event step 2 to turn the vacuum on and off
   - Use axial force control in compression (typically 0N+/-1N)
   - An Oscillation Time Sweep is the preferred type of test step
7. Load the sample.
8. Turn on your vacuum pump.
9. Lower the head to the measurement gap and start the test.
10. Once the test has completed use the vent valve on the waste management manifold to relieve the vacuum in the cell so that the head can be raised.
11. Raise the head and remove the platform, substrate, and retaining ring as once piece for easy cleaning.

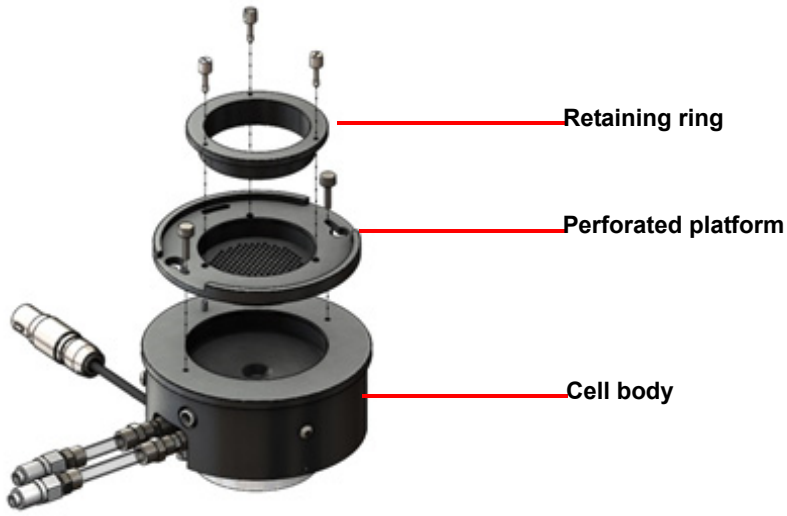

Figure 9