DHR Series and AR Series

Peltier Plate



Getting Started Guide



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 <u>TA Manual Supplement</u> 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
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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 2014/35/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States relating to the making available on the market of 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 2014/30/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization 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 |
|---------|--|
| <u></u> | 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.

Cautions and Warnings

WARNING: The material used on the top surface of the Peltier Plate is hard, chrome-plated copper. The material used for the 'skirt' of the standard Peltier is stainless steel, and Nylon for the Dual Stage Peltier. Therefore, use an appropriate cleaning material when cleaning the Peltier Plate.

AVERTISSEMENT: Le matériau utilisé à la surface supérieure de la plaque Peltier est du cuivre chromé rigide. Le matériau utilisé pour la 'jupe' de la plaque Peltier standard est l'acier inoxydable et celui utilisé pour la double platine Peltier est le Nylon. Par conséquent, utilisez l'équipement approprié pour le nettoyage de la plaque Peltier.

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.

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.

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Chapter 1:

Introducing the Peltier Plate

Overview

The standard Peltier Plate (schematic shown in the figure below) is the most widely used temperature control system for the rheometers. It uses the Peltier thermoelectric effect to control the temperature accurately, with rapid heating and cooling. The plate consists of a copper disc, with hard chrome plating on the upper surface. A Pt100 temperature probe is embedded in the disc, in thermal contact with it and close to the surface. Copper is used as the disc material for its very high thermal conductivity, ensuring negligible temperature gradients across the surface of the plate. The hard chrome plating prevents mechanical or chemical damage to the plate.

The basis of the Peltier effect is this: When an electric current passes across the junction between two dissimilar metals or semiconductors, a temperature gradient across the junction results—the temperature will rise on one side and fall on the other. The direction of the current determines on which side the temperature rises.

On the DHR rheometer plates, a set of Peltier elements is placed below and in thermal contact with the copper disc. The desired level of active heating or cooling can be achieved by controlling the direction and magnitude of the current supplied to these elements.

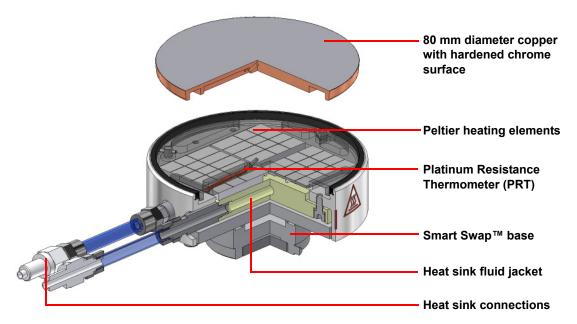


Figure 1 Peltier Plate schematic diagram.

Since a temperature gradient can only be maintained by the input of energy, the Peltier plate requires a heat sink to remove any waste heat. This is provided by a coolant fluid, normally water, passing through a fluid jacket situated below the Peltier elements. If the fluid circulating through the fluid jacket is from a closed reservoir, then its temperature will rise unless it is actively cooled, particularly when the Peltier plate is operating at temperatures far from ambient.

Typical temperature ranges for different cooling fluid configurations are given in the <u>"System Specifications"</u> section.

The flow rate of the fluid through the bath does not need to be high. A flow rate of at 0.5 liters per minute is usually adequate, although this should be increased to 1 liter per minute when working at very low temperatures. If this flow rate is not maintained, the Peltier will lose the ability to provide cooling.

Measuring systems available for use with the Peltier Plate are the cone and plate and parallel plate.

Accessories for the Standard Peltier Plate

Solvent Trap

Many samples are prone to drying due to solvent evaporation during testing. The Solvent Trap cover and Solvent Trap geometry work in concert to create a thermally stable vapor barrier, virtually eliminating any solvent loss during the experiment. The geometry includes a well that contains very low viscosity oil, or even the volatile solvent present in the sample. The Solvent Trap cover includes a blade that is placed into the solvent contained in the well without touching any other part of the upper geometry. The Solvent Trap sits directly on top of the Peltier Plate surface and an insulating centering ring ensures perfect placement for quick and easy sample loading.

Purge Gas Cover

The purge gas environmental cover is a hard-anodized aluminum two-piece split cover with 6-mm diameter compression fittings. An insulating location ring ensures precise and easy location of the cover. This cover is ideal for purging the sample area with nitrogen to prevent condensation during experiments performed below room temperature or with a humidified purge to keep a sample from drying.

Insulating Thermal Cover

The thermal insulation cover is constructed of an anodized aluminum core surrounded by an insulating cover. The aluminum core conducts heat to the upper geometry providing uniform temperature throughout the sample. Recommended for use over a temperature range of -10°C to 90°C, with samples not susceptible to drying such as oils, caulk, epoxy, and asphalt binder. Heat break geometries are recommended for use with the cover.

Peltier Immersion Ring

The Peltier Plate immersion ring allows samples to be measured while fully immersed in a fluid. The immersion ring is compatible with all Peltier Plate models and is easily attached to the top of the Peltier Plate. A rubber ring provides the fluid seal. This option is ideal for studying the properties of hydrogels.

Peltier Plate Covers

A variety of Peltier Plate covers are available for applications that can harm the chromium surface of the plate or for samples that exhibit slip during testing. They are available in stainless steel, hard-anodized aluminum and titanium. Crosshatched and sandblasted Peltier covers are used to eliminate sample slippage effects. Covers are compatible with solvent trap.

Peltier Plate Camera Viewer

The camera viewer is used in conjunction with streaming video and image capture software. Real time images can be displayed in the software and an image can be stored with each data point for subsequent viewing during data analysis. The camera viewer is perfect for long experiments with unattended operation as visual inspections of data integrity.

Variants of Standard Peltier Plate

There are three variants of the standard Peltier Plate: Stepped, Stepped Disposable, and Advanced.

Stepped Peltier Plate

The Stepped Peltier Plate has a raised center section with a thread for attaching screw-in plates. This is most commonly used with 25 and 8 mm screw-in plates and Upper Heated Plate (UHP) for asphalt binder tests.

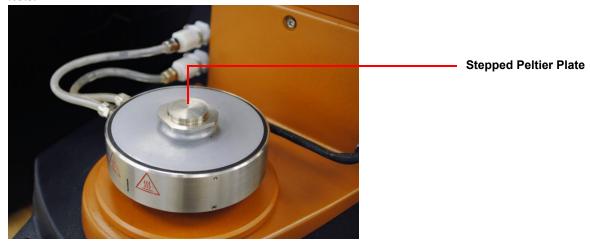


Figure 2 Stepped Peltier Plate.

Stepped Disposable Plate

The Stepped Disposable Plate has a raised center section with a clear hole and locking hex screws to accommodate both disposable plates, and lower Tribo-rheology geometries.

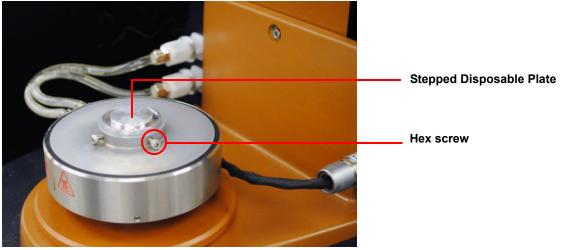


Figure 3 Stepped Disposable Plate.

Advanced Peltier Plate

The Advanced Peltier Plate can be used as a standalone base plate for use with upper geometries up to 48 mm diameter, or it can be used with interchangeable quick-change plates via the use of the bayonet mounting ring. A range of quick-change plates are available in different materials, with steps, different surface finishes (cross-hatched, and sandblasted), or as disposable. These are also compatible with the ARES-G2 APS.

Installing a Quick Change Plate

1 To install a quick-change plate, simply position it on the top raised section of the Advanced Peltier Plate, and lock in place with the bayonet mounting ring ensuring that the quick change plate is correctly centered.

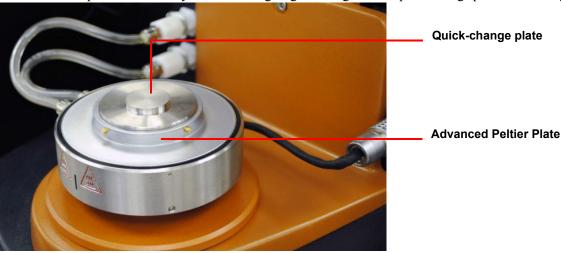


Figure 4 Quick-change plate.

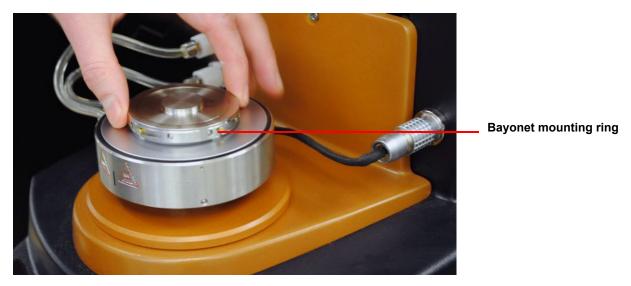


Figure 5 Bayonet mounting ring.

2 Use needle-nose pliers to lock the mounting ring into place. Refer to the image below.



Figure 6 Install the quick change plate.

Un-installing a Quick-Change Plate

To remove a quick-change plate use the needle-nose pliers to unlock the mounting ring. Refer to the image below:



Figure 7 Un-install the quick change plate.

Dual Stage Peltier Plate (DSPP)

The Dual Stage Peltier Plate (DSPP) is a system optimized for low temperature measurements using the standard four element array as a heat sink for a single Peltier element and top plate. This still has the same maximum temperature as the standard plate, but because the lower Peltier elements are the heat sink for the top element rather than a circulating fluid, low temperatures can be reached quickly, and without the use of expensive refrigerated fluid circulators.

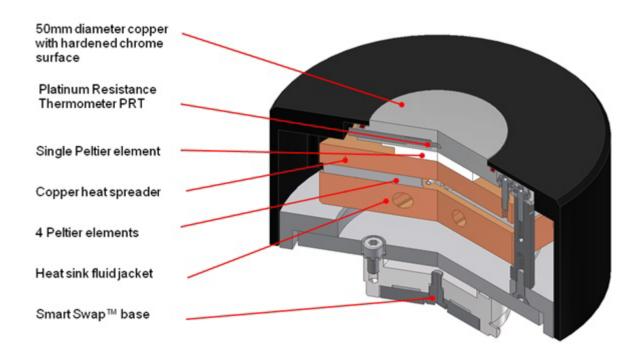


Figure 8 The Dual Stage Peltier Plate.

For optimum performance at low temperatures, use heat break geometries. The gas purge cover (P/N 529612.901) can also be used to reduce frosting. The DSPP kit includes an alignment ring for the gas purge cover, and a spacer ring for use with solvent trap covers.

System Specifications

The table found below contains the technical specifications for the Peltier Plate.

Table 1: Peltier Plate System 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) Appropriate circulating fluid at -20°C | -20°C to 200°C 0°C to 200°C -20°C to 200°C -40°C to 160°C |
|---|--|
| Ramp rate | See section below. |
| Pt100 internal resolution | 0.01°C |

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 2: Dual Stage Peltier Plate System Specifications

| Temperature range Tap water supply at 14°C Air cooled circulator (P/N 403209.901) Thermo Cube (P/N 403258.901) at 5°C Appropriate circulating fluid at 1°C | -40°C to 200°C -20°C to 200°C -40°C to 200°C -45°C to 200°C |
|--|--|
| Ramp rate | See section below. |
| Pt100 internal resolution | 0.01°C |

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.

In the example below, the maximum sustainable cooling rate from 25 to -20° C is -3.87° C/min. So, even though the Peltier plate can step from 25 to -20° C in just over 3 min, the fastest controlled ramp would take almost 12 min (45/3.87).

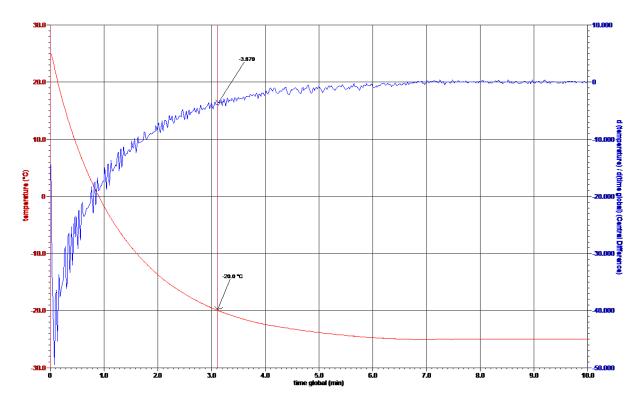


Figure 9 Maximum sustainable cooling rate example.

Chapter 2:

Installing the Peltier Plate

Using the Smart SwapTM

The following sections explain how to attach/detach temperature modules using Smart Swap™. Note that the installation and removal procedures are essentially the same for all modules.

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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.

Installing the Lower Stage

1 Press the **Release** button on the control panel, as seen in the figure below. A continuous green light indicates that the attachment can be fitted.

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

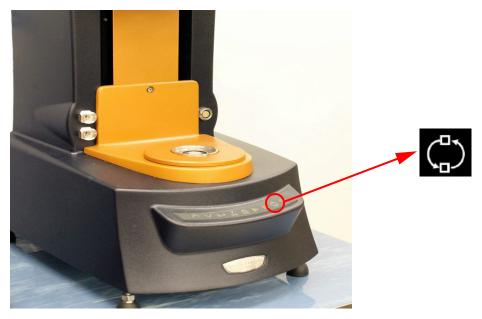


Figure 10 Release button (DHR Series shown).

Fit the attachment as shown in the figure below, ensuring it is aligned correctly. The alignment lug on the lower stage should locate in the slot on the Smart Swap base. To aid in this alignment, there is a mark on the Peltier plate.





Alignment mark and slot

Figure 11 Fitting the attachment (DHR Series shown).

Connect the power cable and, if necessary, the fluid hoses.

Power cable

Fluid hoses

Figure 12 Connecting power cable and fluid hoses (DHR Series shown).

4 When the green status light turns off, the rheometer is ready for use.

Removing the Lower Stage

- 1 Press the **Release** button on the control panel. A flashing green light indicates that the attachment can be unplugged. Refer to <u>step 1</u> in the previous section for **Release** button location.
- 2 Disconnect the power cable and fluid hoses. Refer to <u>Figure 12</u> in the previous section, if needed.
- 3 Press the **Release** button again. A continuous green light indicates that you can remove the attachment.
- 4 Remove the attachment from the rheometer.

NOTE: The release state will stay active for 10 seconds and then revert to locked.