# DHR Series and AR Series

# **UV Curing Accessories**



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.

#### **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 instrument 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 light guide UV Curing Accessory. For all detailed information regarding safety, please refer to the  $OmniCure^{TM}$  User's Guide.

#### **Electrical Safety**

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 VAC are present in this system.

WARNING: High voltages are present in this instrument. Maintenance and repair of internal parts must be performed only by TA Instruments or other qualified service personnel.

AVERTISSEMENT: Présence de tensions élevées dans cet instrument. La maintenance et la réparation des pièces internes doivent être effectuées uniquement par TA Instruments ou tout autre personnel d'entretien qualifié.

#### Radiation Danger

WARNING: Do not expose eyes and skin to ultraviolet light. Never look into light emitting end of the light guide the beam from the light guide, or a reflection of the beam. The light could severely damage the cornea and retina of the eye. When using the UV Accessory, wear the protective eye glasses provided or other suitable eye protection, and clothing to protect exposed skin. Lower the upper fixture cover or UV cover before operating the accessory.

AVERTISSEMENT: N'exposez pas les yeux et la peau au rayonnement ultraviolet. Ne regardez jamais dans l'extrémité électroluminescente du faisceau du conduit de lumière à partir du conduit de lumière ou d'une réflexion du faisceau. Le rayonnement pourrait endommager gravement la cornée et la rétine de votre œil. Lorsque vous utilisez l'accessoire UV, portez les lunettes de protection fournies ou tout autre protection oculaire appropriée, ainsi que des vêtements pour protéger la peau exposée lorsque vous utilisez l'accessoire de séchage UV. Abaissez le couvercle de l'installation supérieure ou le couvercle UV avant d'utiliser l'accessoire.

#### Additional Safety Warnings

WARNING: For detailed information regarding the safe use of the OmniCure 2000, see the *OmniCure Series 2000 User's Guide*.

AVERTISSEMENT: Pour des renseignements détaillés sur l'utilisation sécurisée d'OmniCure 2000, consultez le quide de l'utilisateur *OmniCure Series 2000*.

#### Warranty Information

WARNING: Please take care when using your unit to protect it from misuse or mishandling. TA Instruments offers no warranty after the initial installation of the UV-Curing Accessory, on the Light Guide, or on the Mercury Lamp.

AVERTISSEMENT: Pour protéger l'appareil d'une mauvaise utilisation ou manipulation, faites attention lorsque vous l'utilisez. TA Instruments n'offre aucune garantie après la première installation de l'accessoire de séchage UV sur le double conduit de lumière ou sur la lampe à mercure.

#### **Instrument Symbols**

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

Symbol	Explanation
	This symbol, on the front of the Curing Accessory, warns that ultraviolet light or blue light is present during operation of this instrument. DO NOT EXPOSE EYES OR SKIN TO ULTRAVIOLET OR BLUE LIGHT. Always wear the protective glasses provided or other suitable eye protection when operating the equipment.
	Ce symbole affiché à l'avant de l'accessoire de séchage avertit de la présence d'un rayonnement ultraviolet ou d'une lumière bleue pendant l'utilisation de cet instrument. N'EXPOSEZ PAS LES YEUX ET LA PEAU AUX RAYONNE-MENTS ULTRAVIOLET OU À LA LUMIÈRE BLEUE. Portez toujours les lunettes de protection fournies ou une autre protection oculaire appropriée lors de l'utilisation de cet équipement.

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. This manual is intended to be used in conjunction with the *DHR Getting Started Guide* and the *AR-G2/AR 2000ex/AR 1500ex Getting Started Guide*. For full details on the operation of the rheometer and safety information, please refer to that manual. Both manuals contain cautions and warnings that must be followed for your own safety.

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# Chapter 1:

# Introducing the UV Curing Accessory

### Overview

Ultraviolet (UV) light is usually defined as electromagnetic radiation in the range from 1 to 400 nm. Adhesives, surface coatings and similar materials have been formulated to cure by irradiation with UV light, usually with a wavelength of greater than 300 nm. UV-curing adhesives and coatings have several advantages over alternative materials. They cure very rapidly, usually in a matter of seconds, are solvent free, and are more energy efficient than heat or infrared drying systems. They also have high adhesive strengths on almost all substrates. Blue light, with a wavelength around 450 nm, is most frequently used for curing highly filled dental restoratives.

The curing process can be monitored using oscillatory rheology. TA Instruments has developed Curing Accessories, designed to be used with the DHR-3, DHR-2, and AR-G2/AR2000ex Rheometers. Most curable materials start as low to medium viscosity fluids. Upon being irradiated, they undergo either partial or full curing, usually in a matter of seconds. To follow this process rapid data acquisition is needed, and TA Instruments has developed fast sampling software for use with the Curing Accessories.

### **Product Description**

#### General

The Curing Accessory consists of two fixtures. The *lower fixture* is attached to the rheometer through a standard Smart Swap<sup>TM</sup> fitting (see the main rheometer manual for details). The *upper fixture* is used for temperature control, if required, and uses the Electrically Heated Plate (EHP) upper. The fixture mounts on the rheometer head.

The uncured sample is loaded between two 20-mm diameter plates. The lower 20-mm plate is mounted on the Curing Accessory's lower fixture, and may be either quartz or, for temperatures below 50°C, polymethyl methacrylate (acrylic).

The upper plate connects to the rheometer shaft in the usual manner (see the main rheometer manual for details). The sample is irradiated by UV or visible light that passes through the lower disc. The light irradiance is set using TRIOS Software. The curing process is monitored using oscillatory rheology in fast sampling mode.

#### **Light Source Options**

Light is usually described by its irradiance and wavelength. Irradiance is defined as the rate at which light energy is transferred per unit area, and, therefore, has units of power per square meter (typically mW.cm-2).

**NOTE**: This quantity is often called intensity, but this can lead to confusion with radiant intensity, which has different units.

Three options are available for the light source: Two *fixed* wavelength light emitting diode (LED) arrays (365 and 455 nm) or a *variable* wavelength lamp connected to the lower fixture via a 5-mm light guide. Alternatively, you may supply your own light source, but in this case, software control of the device will not be possible.

The light source for the variable wavelength light guide option is the OmniCure<sup>TM</sup> S2000 system. This system uses a high-pressure mercury lamp capable of producing a light irradiance at the sample of 300 mW.cm<sup>-2</sup>. Selectable band pass filters can used to customize the wavelength (320-500 nm is fitted as standard).



Figure 1 Omnicure.

## **Specifications**

**Table 1: Fixed Wavelength LED Option** 

	UV	Blue
Wavelength	365 nm	455 nm
Maximum sample irradiance	350 mW.cm-2 *+	350 mW.cm-2 *
Temperature range	Ambient Ambient to 150°C with upper EHP 5 to 150°C with EHP upper and vortex -50 to 150°C with EHP upper and GCA	

#### Table 2: Variable Wavelength Omnicure<sup>TM</sup> 2000 Light Guide Option

Wavelength	320-500 nm standard
Maximum sample irradiance	300 mW.cm-2 *
Temperature range	Ambient Ambient to 150 °C with upper EHP 5 to 150 °C with EHP upper and vortex -50 to 150 °C with EHP upper and GCA

<sup>\*</sup> Depends on radiometer sensitivity and spectral distribution of the source.

<sup>&</sup>lt;sup>+</sup>Systems with serial numbers ending with 09VA, 09VB, 10VA, 10VB, 11VA, and 11VB are limited to 150 mW.cm-2.

# Chapter 2:

## Installing the System

### Installation

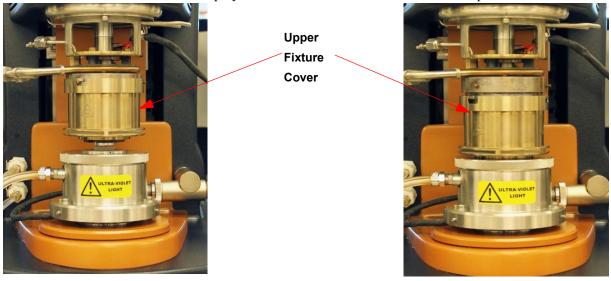
Installing the Curing Accessory for use with the DHR and AR Rheometer involves the installation of the following components, some of which are optional:

- Upper fixture
- LED lower fixture
- Light guide lower fixture
- Upper plate
- OmniCure<sup>TM</sup> light source

#### **Installing the Upper Fixture (Optional)**

Optional temperature control is provided by the upper fixture, which is the same as the Electrically Heated Plate (EHP) upper fixture. The fixture mounts on the head of the rheometer. Follow the installation instructions found in *DHR Getting Started Guide* or the *AR-G2 / AR2000ex / AR1500ex Getting Started Guide* to install the upper fixture.

When used with the Curing Accessory, the upper fixture must be used with the shield, which should be in the lowered position before any experiments are started. Figure 2 shows the upper fixture installed with the LED lower fixture. The shield is displayed below in both the raised and lowered positions.



**Figure 2** Upper fixture cover: raised position (left) and lowered position (right).

CAUTION: Lower the upper fixture cover BEFORE starting any experiment.

MISE EN GARDE : Abaissez le couvercle de l'installation supérieure AVANT de commencer toute expérience.

CAUTION: Disposable acrylic geometries should not be used above 50°C. The softening and melting points of acrylic are 80 and 130°C, respectively.

MISE EN GARDE: Les géométries acryliques jetables ne doivent pas être utilisées au-delà de 50°C. Les points d'adoucissement et de fusion de l'acrylique sont de 80 et 130°C, respectivement.

#### **Installing the LED Lower Fixture**

Install the LED lower fixture shown below by following the instructions below.



**Figure 3** The LED lower fixture.

- 1 Use the Smart Swap<sup>TM</sup> connector to mount the fixture on the rheometer.
- 2 Connect the flow and return ports on the rear of the rheometer to a source of water, such as a fluid circulator or mains supply.
- 3 Connect the fluid connectors on the fixture to the ports on the front of the rheometer.

**NOTE**: Although there is no direct temperature control on the fixture, steps 2 and 3 are needed because the LED array must be cooled.

CAUTION: To avoid condensation inside the accessory, the temperature of the circulation water should be close to ambient.

MISE EN GARDE: Pour éviter la condensation à l'intérieur de l'accessoire, la température de l'eau de circulation doit être proche de la température ambiante.

The fixture is wired directly to a 'T' connector which plugs into the Smart Swap socket on the rheometer body.

4 Plug the 6-pin female socket on the 'T' connector into the Lemo plug of the upper fixture.

5 Seat the lower plate into the center of the lower fixture.

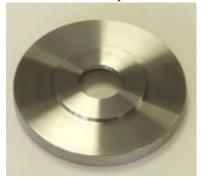


Figure 4 LED lower plate retaining ring.

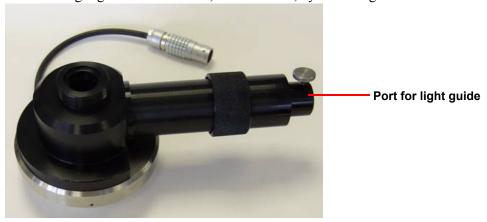
6 Screw the retaining ring, shown in <u>Figure 4</u>, onto the body of the lower fixture to finger tightness. The fully installed LED lower fixture is shown below.



**Figure 5** LED lower fixture mounted on the rheometer.

#### **Installing the Light Guide Lower Fixture**

Install the light guide lower fixture, shown below, by following the instructions below.



**Figure 6** Light guide lower fixture.

- 1 Use the Smart Swap connector to mount the fixture on the rheometer.
- 2 If the accessory is used without temperature control, plug the fixture directly into the socket on the rheometer body.
- 3 If the upper fixture is installed, use the adaptor shown below. Plug the 6-pin female socket on the adapter into the Lemo plug of the upper fixture.

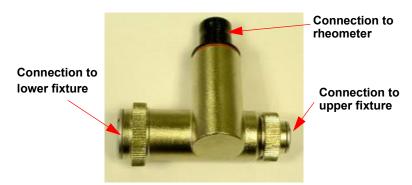


Figure 7 Smart Swap adapter.

- 4 Seat the lower disc into the center of the lower fixture.
- 5 Screw the retaining ring, shown below, onto the body of the lower fixture to finger tightness.



Figure 8 Light guide lower plate retaining ring.

The fully installed variable wavelength lower fixture is shown below.



Figure 9 Fully installed light guide lower fixture.

#### **Installing the Upper Plate**

The upper geometry holders / heat spreaders are similar to the standard components when used with the Electrically Heated Plates (EHP) upper fixture, and the 20-mm diameter plates. The plates may be either reusable or disposable. The EHP disposable upper geometry holder with attached plate is shown below.



Figure 10 Geometry holder for use with the upper fixture.

If the EHP upper is not to be used, either a one-piece reusable 20-mm diameter plate or a disposable system can be used. The 20-mm diameter disposable plate is fixed to a geometry holder by two cap head screws (shown below). A two-piece cover is supplied for ambient use.



Figure 11 Geometry holder for use without the upper fixture.

CAUTION: The reusable plates should only be used if the system shows only very partial cure. For all other cases, use disposable plates.

MISE EN GARDE: Les plaques réutilisables ne doivent être utilisées que si le système affiche remède très partielle. Pour tous les autres cas, utilisez des assiettes jetables.

**NOTE**: The upper geometry holder attaches to the rheometer in the usual way.

WARNING: You must use the geometry holder and standard geometries in conjunction with the two-piece cover (see the figure below). This cover should be in place before any experiment is started.

AVERTISSEMENT: Vous devez utiliser le support de géométrie et les géométries standard conjointement avec le couvercle à deux pièces (voir la figure ci-dessous). Ce couvercle doit être installé avant de commencer toute expérience.



Figure 12 Two-piece cover correctly installed for ambient use.

#### **Installing the Omnicure 2000 Light Source**

For detailed information regarding the installation and safe use of the OmniCure™ 2000, see the *OmniCure™ Series 2000 User's Guide*.

AVERTISSEMENT: Pour des renseignements détaillés sur l'utilisation sécurisée d'OmniCure 2000, consultez le guide de l'utilisateur *OmniCure Series 2000*.

Light is directed from the OmniCure light source to the lower fixture by the one meter light guide supplied.

1 Place the OmniCure unit on the bench to the right of the rheometer when viewed from the front. Ideally the OmniCure unit should on a separate bench to avoid vibrations from the unit influencing rheometric measurements.

2 Connect the 9-pin P3 I/O Signal Port on the rear of the OmniCure unit (circled in the figure below) to the RS232 Auxiliary port on the rear of the rheometer electronics box using the 9-pin cable provided.



Figure 13 Rear of OmniCure.

**NOTE**: If it is intended that the UV Light Guide Accessory be used together with an EHP upper and GCA it is not possible to use the 9-pin cable connection described above. This is because the GSA connects to the single RS232 port on the rheometer. Instead, Event Cable UV LG (PN 533403.901) must be used to connect from Event B on the rear of the rheometer to P1 on the Omnicure. This will allow the Omnicure to be triggered by an event step in TRIOS. However, it is not possible to send an irradiance level in this configuration. This must be set manually on the Omnicure prior to an event. The UV System in the Event step must be set to "none." The UV LED Accessory can be used together with EHP and GCA without this limitation.

3 Insert the 10-mm diameter end of the guide into the port on the front of the OmniCure unit (see the figure above). Push the light guide in until it seats with a second positive "click", as directed in the OmniCure User's Guide.

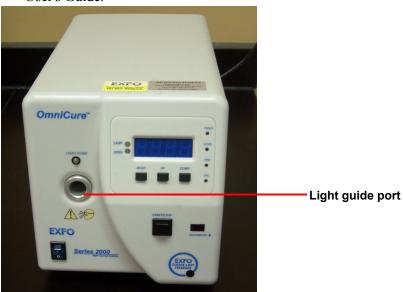


Figure 14 OmniCure.

- 4 Insert the 7-mm diameter end into the port at the end of the arm of the lower fixture. Use the knurled screw to hold it in place. The light guide should <u>not</u> be bent through an angle greater than that stated in the User's guide.
- 5 Ensure that the power switch on the rear of the OmniCure unit is in the OFF position, and connect the unit to the mains supply.

# Chapter 3:

# Operating the Curing Accessory

WARNING: Wear the protective eye glasses provided or other suitable eye protection, and clothing to protect exposed skin when operating the UV Curing Accessory.

AVERTISSEMENT: Portez les lunettes de protection fournies ou toute autre protection oculaire appropriée, ainsi que des vêtements pour protéger la peau exposée lorsque vous utilisez l'accessoire de séchage UV.

Follow the instructions in this chapter to operate the Curing Accessory. Operation, including setting the irradiance and duration of the light illumination, is controlled through TA Instruments TRIOS software. If you have provided your own light source, control of the device through the software will not be possible.

## Calibrating the Light Source

To obtain absolute irradiance of the UV light, you will need to calibrate the light source. For the UV light accessories, a portable radiometer/dosimeter (shown below) is provided to measure the irradiance at the upper plate. This is the irradiance seen by the sample during the curing reaction. The irradiance is a nearly linear function of the power supplied to the LED or Omnicure<sup>TM</sup> 2000. For the blue light LED accessory, a test sheet is provided that gives the irradiance at 100% power. This can be used in the absence of a suitable radiometer.



**Figure 15** Portable radiometer and sensor.

The radiometer does not have equal sensitivity to all wavelengths over its 280 to 400-nm range, and the lamp sources do not provide equal irradiance at all wavelengths. These considerations need to be taken into account when comparing levels of irradiance.

For more information on UV irradiance calibration, see the *DHR/AR Rheometer Temperature Systems/Accessory Calibrations* Help topic in TRIOS software.

### Loading the Sample

Power up the Omnicure unit, if this is to be used. Allow the unit to warm up for approximately four minutes.

The procedures for zeroing the geometry gap, raising and lowering the instrument head, loading and trimming the sample and so on are fully described in the *DHR Getting Started Guide* and the AR-G2 / AR2000ex / AR1500ex Getting Started Guide, and only a brief summary follows.

If the sample you are measuring is relatively low in viscosity, it is recommended that you fit an O- ring over the lower geometry to protect the assembly from sample ingress.

Zero the geometry gap, raise the instrument head, and load the sample. Close the gap to that required and trim the sample. Lower the upper fixture cover or fit the two-piece cover. Zero the normal force at this point, if you are using normal force control during the test.

WARNING: Do not use the start / stop button the OmniCure unit when it is used in conjunction with the rheometer, as this could result in accidental exposure.

AVERTISSEMENT: N'utilisez pas le bouton marche/arrêt de l'appareil Omnicure lorsqu'il est utilisé conjointement avec le rhéomètre, car cela pourrait entraîner une exposition accidentelle.

## Designing and Running a Procedure

Rheological monitoring of curing is usually conducted using oscillatory (dynamic) rheology. The curing process is very rapid and, to capture it fully, fast oscillation is used. The sampling rate for this mode is twice the fundamental oscillation frequency, up to 25 Hz. So, at a fundamental frequency of 25 Hz the sample rate will be 50 points/sec, but at 30 Hz it will be 30 points/sec. The timing and irradiance of the light is set from an EVENT step in the experimental procedure.

For more information, refer to the *Operating the UV Curing Accessory* Help topic in TRIOS software.

## Dismantling the Accessory

#### **Removing the LED Fixture**

Follow these instructions to remove the LED fixture:

- 1 Unplug the water pipes.
- 2 Remove the lower fixture can then be removed from the rheometer using Smart Swap<sup>TM</sup>.
- 3 Remove the upper fixture.

#### **Removing the Light Guide Fixture**

Follow these instructions to remove the variable wavelength light guide fixture:

WARNING: Before dismantling the accessory, ensure that the OmniCure unit is powered down and isolated from the mains supply.

AVERTISSEMENT: Avant de démonter l'accessoire, assurez-vous que l'OmniCure est hors tension et isolé de l'alimentation secteur.

- 1 Loosen the thumbscrew holding the light guide in the port on the lower fixture. Withdraw the guide, holding firmly by the strain relief casing.
- 2 Remove the lower fixture can then be removed from the rheometer using Smart Swap<sup>TM</sup>.
- **3** Remove the upper fixture.