



DISCOVERY DSC Microscope Accessory



Getting Started Guide

Revision A Issued December 2017

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
- TA Instruments Patents
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- 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.


Safety

CAUTION: 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.

MISE EN GARDE : 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.

Accessory Symbols

The following labels are displayed on the accessory for your protection:

Symbol	Explanation
	<p>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.</p> <p>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.</p>

Please heed the warning labels and take the necessary precautions when dealing with those parts of the instrument. The *Discovery DSC Microscope Accessory Getting Started Guide* contains cautions and warnings that must be followed for your own safety.

Electrical Safety

You must unplug the instrument before doing any maintenance or repair work; voltages as high as 120/240 volts AC 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.

Table of Contents

Introduction.....	2
Important: TA Instruments Manual Supplement	2
Notes, Cautions, and Warnings	3
Safety	4
Accessory Symbols	4
Electrical Safety	4
Table of Contents.....	5
Introducing the Microscope Accessory.....	6
Overview	6
Product Description	6
Setting Up the Microscope Accessory	7
Installing the Microscope Accessory	8
Operating the Microscope Accessory.....	12
Preparing the System	12
Operating the Microscope Accessory	12
Using the Polarizer Ring and the Focus Ring	12
Dino-Lite Software	12
Enabling the Microscope Option	13
Collecting Images and Video During a DSC Experiment	16
Extracting Images from a Data File	18
An Application Using the Microscope Accessory	19
Maintaining the Microscope Accessory	20
Replacement Parts	21

Chapter 1:

Introducing the Microscope Accessory

Overview

The DSC Microscope Accessory is used with the TA Instruments Discovery Differential Scanning Calorimeters (DSC 25, 250, 2500). The accessory allows for visual observations of samples during testing in the DSC in the form of static images or video. The microscope camera is active during the experiment to allow for real-time visualization of the material with both static images and video files that can be saved to view after the test is complete. Events that appear in the heat flow data can be confirmed or better interpreted with the coordinating image or video of the physical change that may also occur. The collected visual media can be used to precisely show some of the physical phenomenon only previously theorized due to the lack of concrete visual evidence a heat flow trace alone does not have the capabilities to show.

Product Description

The DSC Microscope Accessory consists of a high resolution, 1.3 MP color, digital microscope camera with an approximate magnification of 50x when focused on a DSC sample pan in the cell. The accessory collects individual static images of up to 1 fps (frames per second) and video at 15 fps. Illumination is provided by several white light LEDs surrounding the lens. The camera also has an adjustable polarizer for viewing highly reflective materials and minimizing the amount of glare on the sample. Silver lids and sapphire discs are included to cover the cell while still allowing a clear view of the sample in the cell.

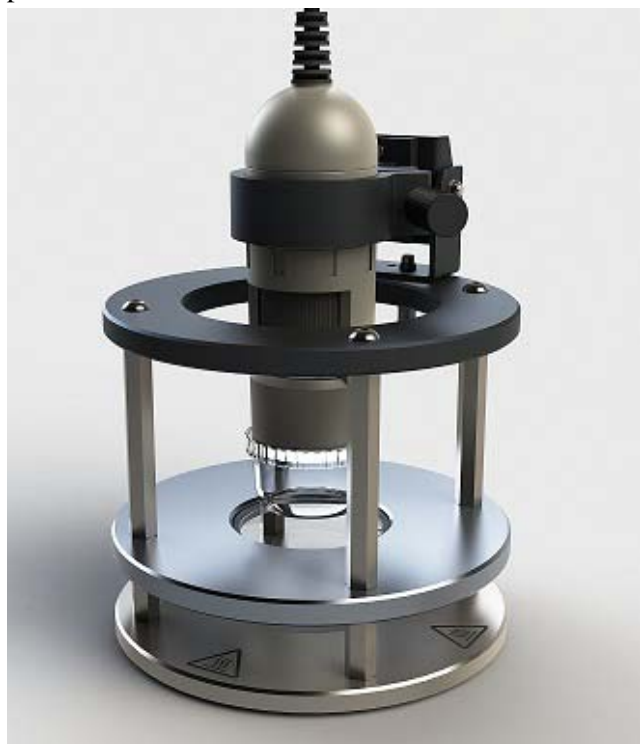


Figure 1 Microscope accessory.

Chapter 2:

Setting Up the Microscope Accessory

This section briefly describes the setup of the Discovery DSC Microscope Accessory and its connection to the Discovery DSC. For more details on setting up the Discovery DSC, consult the Discovery DSC Getting Started Guide and online help.

There are many components provided with the microscope and its accompanying accessory kit which will be used in the setup. Those components are identified in the figure below.

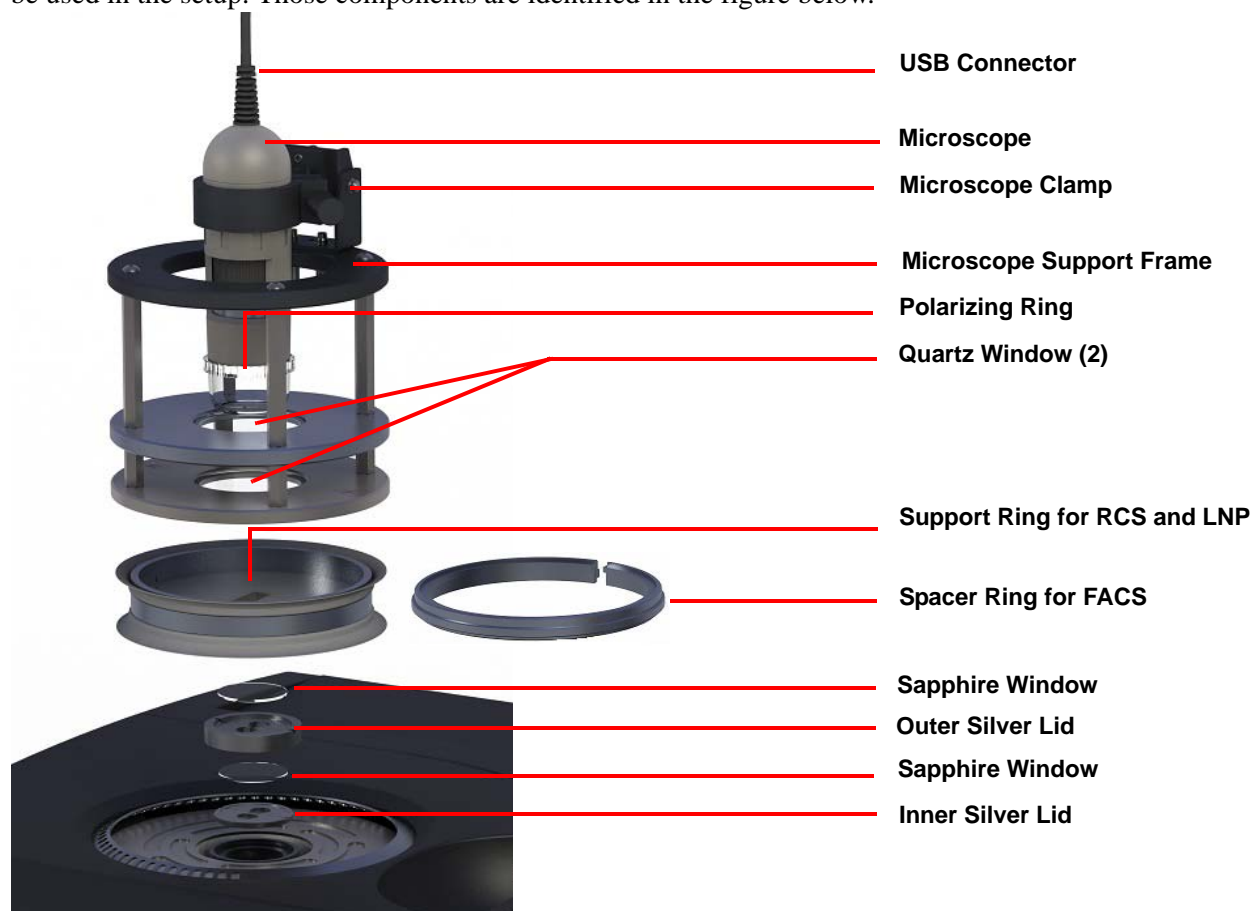


Figure 2 Microscope Accessory Kit components.

Installing the Microscope Accessory

- 1 Install the appropriate cooling accessory on the DSC. The microscope accessory can be used over the full temperature range of all compatible coolers, including the FACS, RCS, and LNP.

NOTE: If the instrument has an Autosampler, open the Lid to prevent it from contacting the microscope accessory. The Autosampler will become disabled when the Microscope Camera option is selected in TRIOS (see [Figure 11](#)) in the next chapter.

- 2 Place the inner silver lid on the cell, and then place a sapphire window on top. Place the outer silver lid on the cell, and then place another sapphire window on top of it. Align the circular openings in the silver lids above the sample and reference calorimeters. See image below.



Figure 3

- 3 If using a FACS, place the spacer ring on the metal grill of the Discovery DSC dress cover.

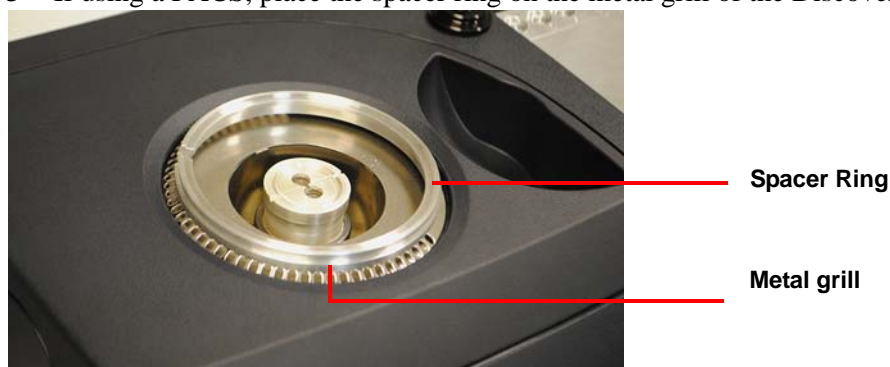


Figure 4 Spacer ring for FACS installed.

If using an RCS or LN Pump, place the support ring over the cell with the opening aligned so that you can see the sample. The spacer ring is not needed with the RCS or LN Pump

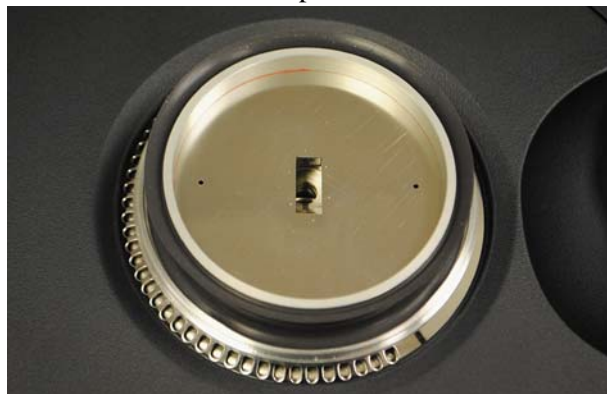


Figure 5 Support ring installed.

NOTE: These active coolers require purging with dry inert gas upon installation to remove residual moisture before the cooler is activated. Moisture in the heat exchanger assembly can lead to condensation on the windows, making image capture impossible.

Install the microscope in the microscope clamp

- 4 Secure the microscope clamp to the microscope support frame from below using two knurled screws found in the kit.

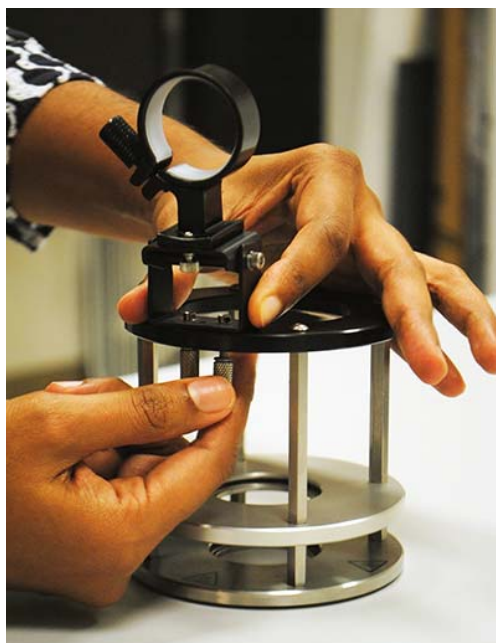


Figure 6 Installing clamp.

- 5 Pass the USB connector up through the bottom of the clamp.



Figure 7 Position the microscope in the clamp.

- 6 Place the body of the microscope within the clamp. Bend the clamp at the hinge so that the microscope is facing downward within the support frame.



Figure 8 Bend the clamp.

- 7 Position the microscope in the clamp so that the focus ring faces toward the left side of the instrument. Secure the clamp to the microscope (above the focus ring) by tightening the thumbscrew.

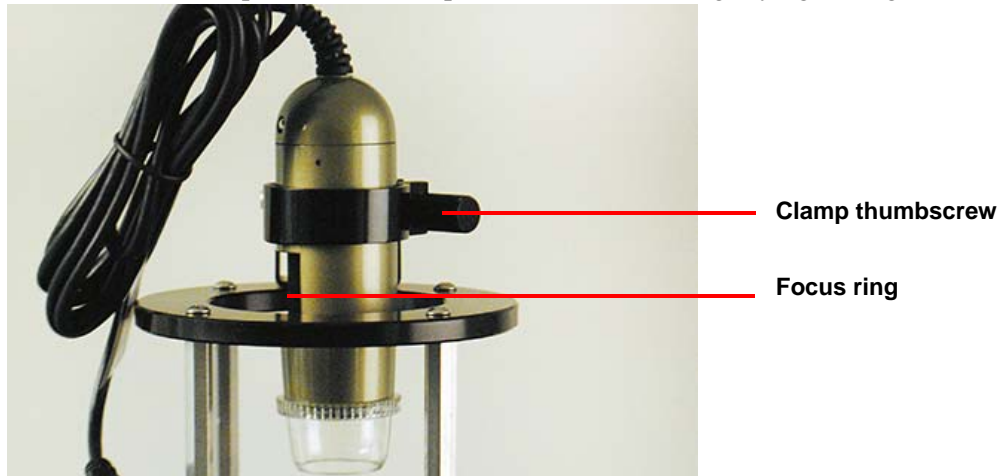


Figure 9 Secure the clamp to the microscope.

- 8 Set the microscope support frame on top of the spacer/support ring.



Figure 10 Frame installed.

- 9 Insert the upper quartz window from the accessory kit into the upper thermal isolation plate of the microscope support frame, in the space beneath the microscope. Then insert the lower quartz window into the lower thermal isolation plate of the microscope support frame.
- 10 Connect the microscope accessory's USB cable to the controller.

Chapter 3:

Operating the Microscope Accessory

This section briefly describes the operation of the Discovery DSC Microscope Accessory.

Preparing the System

The microscope accessory requires that the laboratory environment has a dew point of 16°C or lower, and use of an active purge of dry inert gas to avoid accumulation of moisture on the windows. An in-line gas dryer or dessicator may be required. TA Instruments recommends the RCS dryer (P/N 200266.001) for the removal of residual moisture from the gas stream.

Before beginning an experiment, allow the cooler to purge to release moisture.

Operating the Microscope Accessory

NOTE: When running a sub-ambient experiment, once equilibrated to the experimental temperature, begin the temperature ramp immediately to minimize the risk of the windows fogging, and avoid staying at a sub-ambient temperature for an extended period of time.

Using the Polarizer Ring and the Focus Ring

The polarizer ring is used to reduce the glare that may occur from the light on the windows, DSC pan, and reflective samples and is adjusted manually on the microscope.

The focus ring is used to focus the microscope image, and is also adjusted manually on the microscope.

Dino-Lite Software

The Dino-Lite software that comes with the microscope hosts a variety of functions, such as the ability to adjust the lighting, take measurements, add text and drawings to the image, etc. View the software's online help for more information.

NOTE: The Dino-Lite software cannot be used in conjunction with TRIOS software; any changes or adjustments made to the microscope camera image in the Dino-Lite software are not carried over to the images collected in TRIOS software.

Enabling the Microscope Option

Before using the microscope accessory, the microscope must be enabled in TRIOS.

- 1 From the **Options** menu, select the instrument (DSC25, DSC250, or DSC2500), and then click **General**. Select **Microscope Camera Enabled**. If an Autosampler is present, enabling the microscope disables the Autosampler. Click **OK**.

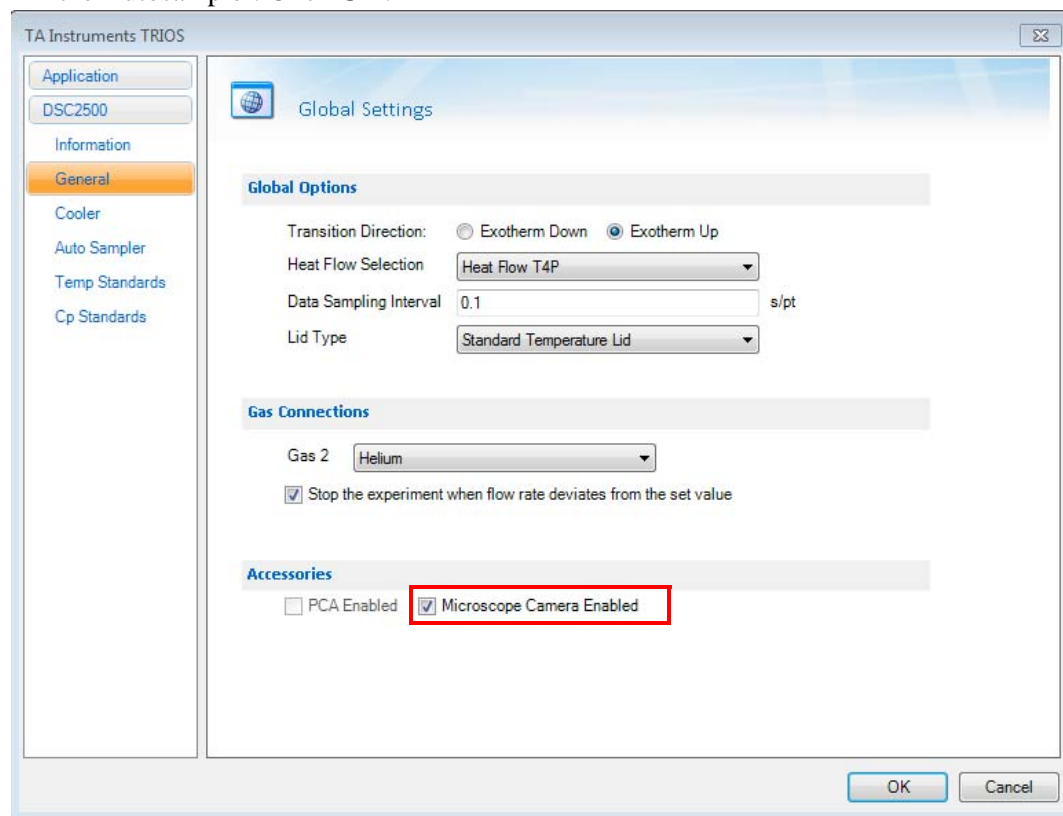


Figure 11 Microscope enabled.

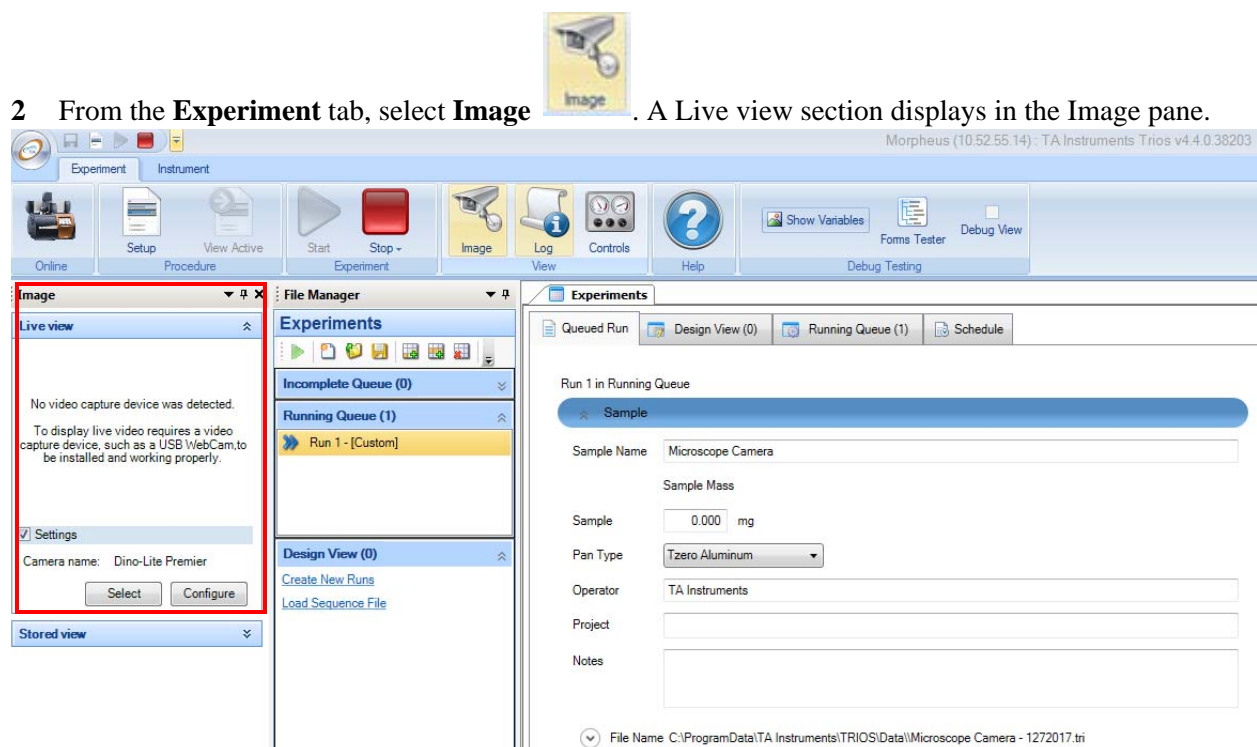


Figure 12 Image pane with Live view.

3 At the bottom of Live view, make sure **Settings** is checked and then click **Select**.

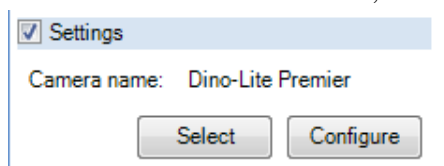


Figure 13 Settings > Select.

4 A “Select video capture device” dialog box displays. Highlight **Dino-Lite Premier** and then click **OK**.

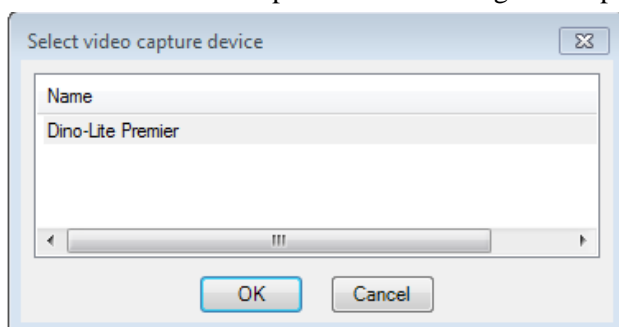


Figure 14 Select video capture device.

- 5 Live view is populated with an image from the microscope. If needed, adjust optical properties by clicking **Configure**. Adjust each property using the slider and click **OK**.

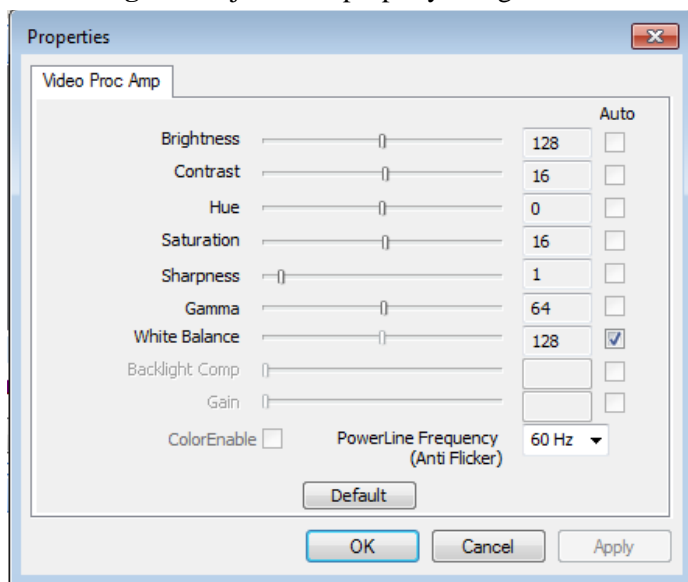


Figure 15 Configure optical properties.

Collecting Images and Video During a DSC Experiment

Use TRIOS software to add images and video to your experiment by adding the Microscope Camera segments to the test method. The maximum collection rate is 1 frame per second. The default setting is 1 image every 30 seconds. The default setting can be edited as needed. Video is captured at a rate of 15 frames per second. This collection rate cannot be edited within TRIOS software.

There are three Microscope Camera method segments that become active when the accessory is connected to TRIOS:

- **Image:** This segment allows you to turn the image collection command on and off during an experiment. The images are stored within the TRIOS data file and can be exported into standard formats such as *.jpg.
- **Video:** This segment allows you to turn the video collection command on and off during an experiment. The video file is not embedded in the TRIOS data file; it is a separate file stored in the same directory as the TRIOS data file. The video file is saved as *.avi.
- **Dynamic Video:** This segment is used to capture video clips only when a heat flow thermal event in the test sample is detected. This helps reduce the size of the video footage by not capturing video during inactive heat flow time periods. The heat flow transition detection trigger initiates when the heat flow rate acceleration is detected to be approximately 0.2 mW/sec^2 . At this point, a video clip is created that contains up to 20 seconds of pre-transition footage, along with footage during the transition. The video file is not embedded in the TRIOS data file; it is a separate file stored in the same directory as the TRIOS data file. The video file is saved as *.avi.

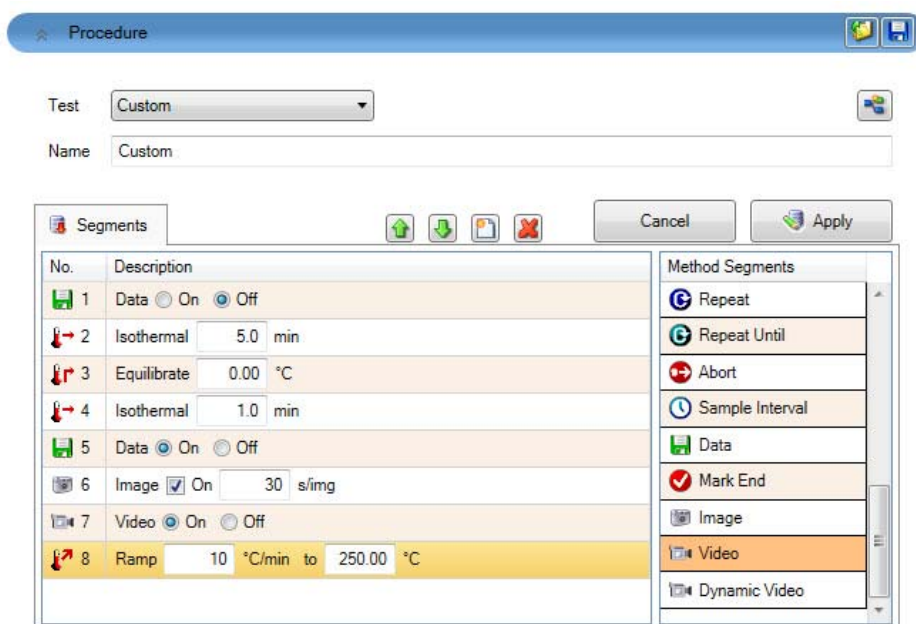


Figure 16 Editing a custom method to add Image and Video segments.

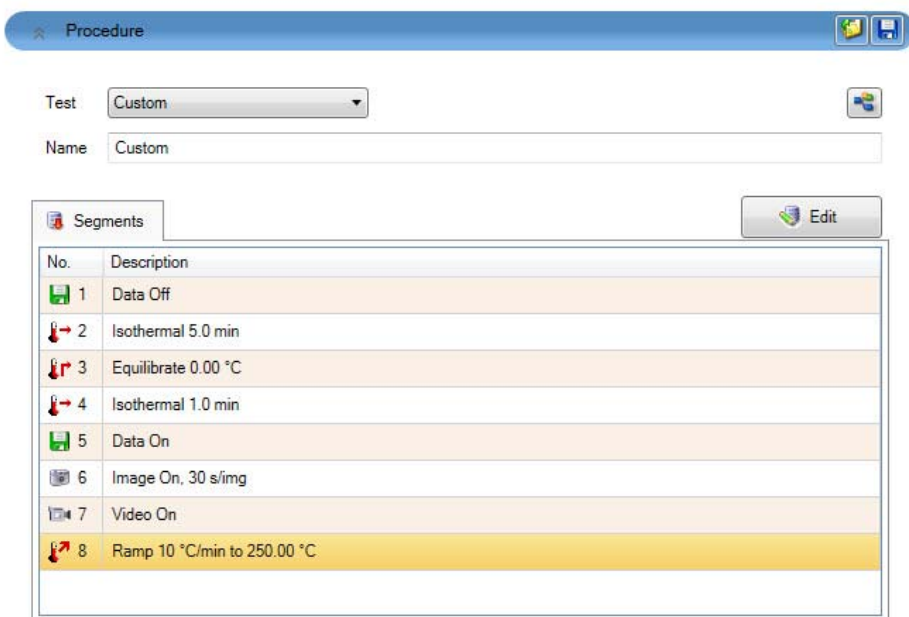


Figure 17 An applied method containing Image and Video segments.

AVI file formats can be viewed in open source cross-platform multimedia players such as VLC media player. VLC media player can be downloaded from <https://www.videolan.org/vlc/index.html>.

AVI video format can be converted to other platforms such as MP4, if needed.

Extracting Images from a Data File

- 1 Click the **Results** tab in the File Manager, and then select the spreadsheet view. The camera icon indicates that an image is available.

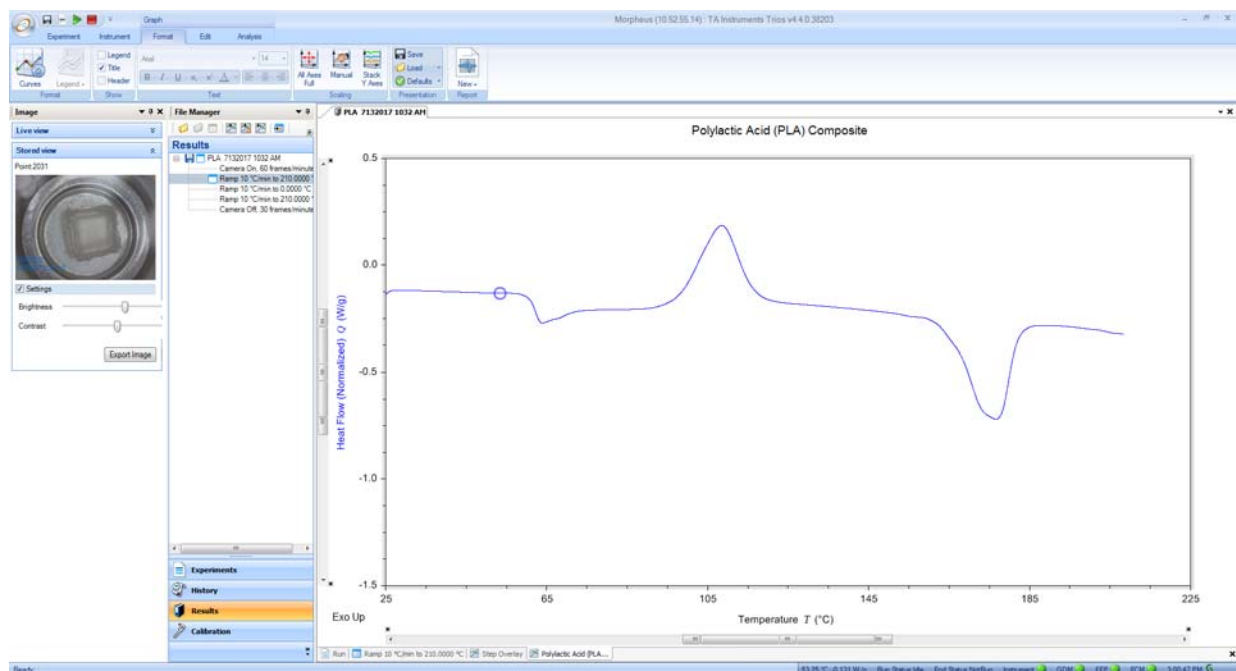


Figure 18 DSC data file with images.

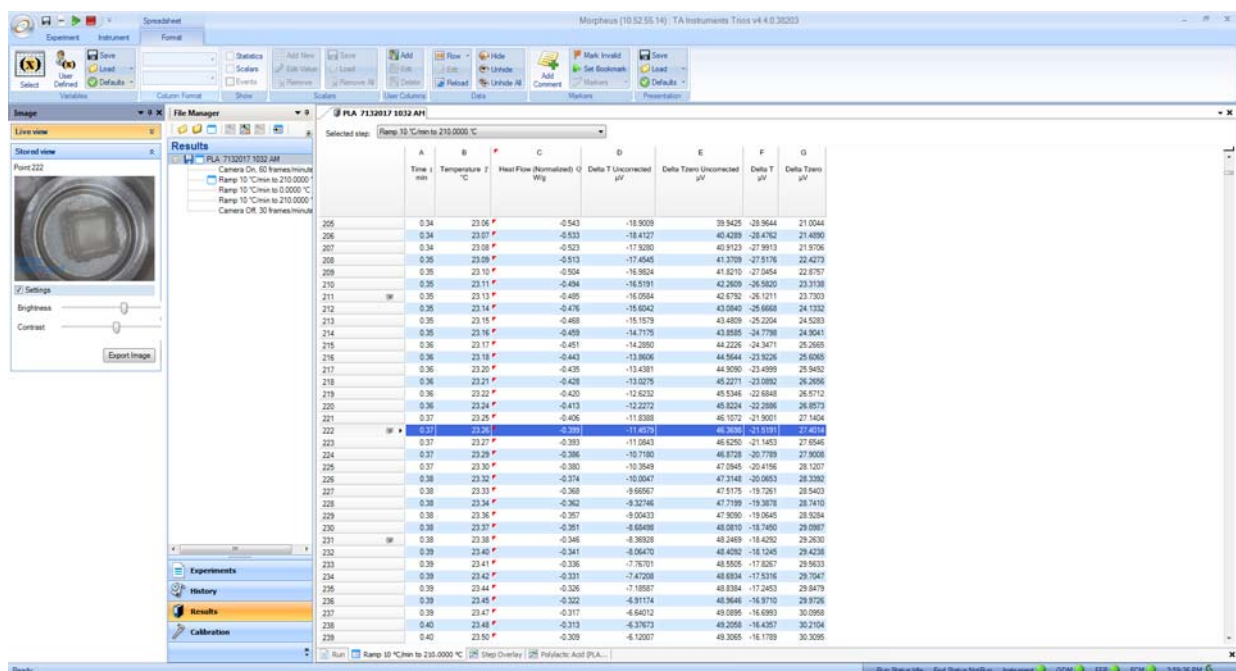


Figure 19 Spreadsheet tab with image icons.

-
- The screenshot shows a context menu for a table row. The menu options are:
- Copy
 - Paste
 - Select Variables...
 - Add Row
 - Append Row
 - Delete Row
 - Add Comment...
 - Mark Invalid
 - Hide
 - Unhide
 - Unhide all
 - Bookmark
 - Export Image** (highlighted)
 - Export All Images...
 - Copy Image
 - Properties...
- The background table has the following visible data:
- | | 48.3483 | 33.6106 | -0.231 |
|------|---------|---------|--------|
| 2046 | 48.3483 | 33.6106 | -0.231 |
| 2047 | 48.3483 | 33.6106 | -0.231 |
| 2048 | 48.3483 | 33.6106 | -0.231 |
| 2049 | 48.3483 | 33.6106 | -0.231 |
| 2050 | 48.3483 | 33.6106 | -0.231 |
| 2051 | 48.3483 | 33.6106 | -0.231 |
| 2052 | 48.3483 | 33.6106 | -0.231 |
| 2053 | 48.3483 | 33.6106 | -0.231 |
| 2054 | 48.3483 | 33.6106 | -0.231 |
| 2055 | 48.3483 | 33.6106 | -0.231 |
| 2056 | 48.3483 | 33.6106 | -0.231 |
| 2057 | 48.3483 | 33.6106 | -0.231 |
| 2058 | 48.3483 | 33.6106 | -0.231 |
| 2059 | 48.3483 | 33.6106 | -0.231 |
| 2060 | 48.3483 | 33.6106 | -0.231 |
| 2061 | 48.3483 | 33.6106 | -0.231 |
| 2062 | 48.3483 | 33.6106 | -0.231 |
| 2063 | 48.3483 | 33.6106 | -0.231 |
| 2064 | 48.3483 | 33.6106 | -0.231 |
| 2065 | 48.3483 | 33.6106 | -0.231 |
| 2066 | 48.3483 | 33.6106 | -0.231 |
| 2067 | 48.3483 | 33.6106 | -0.231 |
| 2068 | 48.3483 | 33.6106 | -0.231 |
| 2069 | 48.3483 | 33.6106 | -0.231 |

An Application Using the Microscope Accessory

Polylactic Acid Composite (PLA)

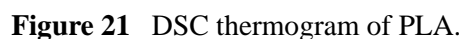




Figure 22 PLA images.

- **A (pre-Tg):** At 50°C, the PLA sample is at a temperature below the glass transition. It is in a rigid amorphous state.
- **B (post-Tg):** At 85°C, the material has passed through the glass transition region and is now in the rubbery amorphous state. Above the Tg, the polymer chains now have translational movement.
- **C (Crystallization Peak):** At 108°C, PLA undergoes crystallization on heating. The material changes from transparent to opaque and is now in a solid, semi-crystalline phase.
- **D (Melt Peak):** At 177°C, PLA undergoes melting. At the peak temperature, the polymer chains are able to move freely.
- **E (Post Melt):** At temperatures above the melt peak, the polymer chains move freely and is in the liquid, amorphous state. As temperature increases, the viscosity decreases.

Maintaining the Microscope Accessory

DSC experiments with the Microscope Accessory require the use of open DSC pans to see the sample. If the sample undergoes a weight loss due to evaporation of solvents, spillage out of the pan, sublimation or decomposition then deposition may occur on the cell, silver lid, and sapphire window. Take precautionary steps to minimize decomposition of the sample in the cell. Refer to the Online Help for instructions on cleaning the DSC cell.

Solvents compatible with silver, constantan, and sapphire may be used to clean the cell and lids. Some materials may alloy to the cell and cannot be cleaned. Contact TA Instruments for technical assistance when needed.

Replacement Parts

Replacement parts for the Microscope Accessory that are available from TA Instruments are listed in the table below; refer to this table when ordering parts.

Table 1: Microscope Replacement Parts

Part Number	Description
202933.002	DIGITAL MICROSCOPE 10X-92X (CAMERA)
203619.001	QUARTZ WINDOW 40MM X 3MM THK
203620.001	SAPPHIRE WINDOW 1.00 DIA X .040 THK
973855.001	LID OUTER SILVER CAMERA
973863.001	LID INNER SILVER CAMERA