

Differential Photocalorimeter



Getting Started Guide

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Notices

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Notes, Cautions, and Warnings

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

A NOTE highlights important information about equipment or procedures.



A CAUTION emphasizes a procedure that may damage equipment or cause loss of data if not followed correctly.



A WARNING indicates a procedure that may be hazardous to the operator or to the environment if not followed correctly.

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.

There are several major areas of concern pertaining to personal safety when using the Differential Photocalorimeter (DPC). For all detailed information regarding safety, please refer to the *EFOS Novacure User's Guide Model N2001-A1*.

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.

Radiation Danger



WARNING: NEVER look into the beam from the light guide, or a reflection of the beam. The high degree of ultraviolet radiation can permanently damage the retina of your eye and result in blindness.



WARNING: You should NEVER expose your skin to UV beams. Exposure of skin surface will result in severe burns. ALWAYS wear LATEX gloves when working with ultraviolet beams.

Additional Safety Warnings



WARNING: For all detailed information regarding safety please read the *EFOS Novacure User's Guide Model N2001-A1*.

Chapter 1

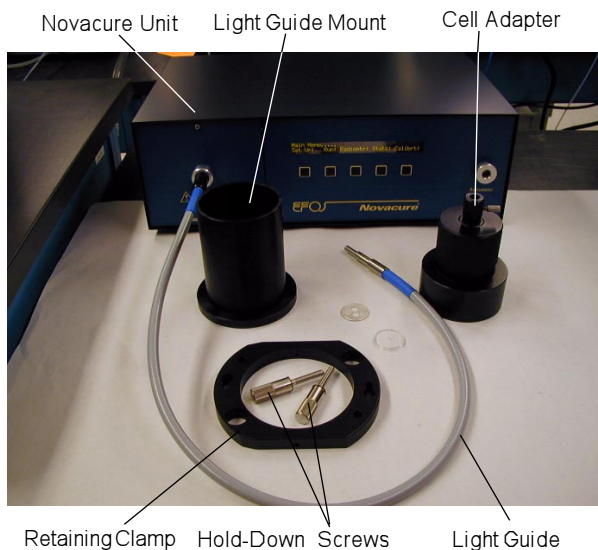
Introducing the DPC

Overview

The Differential Photocalorimeter (DPC) is an accessory used with the DSC 2920 Differential Scanning Calorimeter. This accessory allows samples placed inside the DSC 2920 cell to be irradiated with ultraviolet or visible light. When the samples (usually photopolymers) react to the light, heat is released (i.e., an exothermic reaction occurs). This heat is measured and used to study the relative reactivity and/or kinetics of reaction. The reactions studied are typically rapid and results are obtained in less than 15 minutes.

Product Description

The TA Instruments' DPC accessory is based on the Novacure Model N2001-A1 from Efos Inc. The Novacure contains a high pressure mercury lamp that delivers light over the spectral range 250 to 600 nanometers (nm). A broad-band filter (320 to 500 nm) is provided standard with the instrument. Light is transmitted from the Novacure instrument to the DSC 2920 cell via a one-meter long (5-mm diameter), liquid light guide. The light guide attaches to the cell itself using a special adapter. The figure above shows those system components.



Experiment Considerations

There are four fundamental instrument characteristics that affect the quality of DPC results. Those characteristics are: wavelength range, light intensity at the DSC cell sample and reference positions, baseline noise, and baseline offset. The following paragraphs describe aspects for each which should be considered when planning an experiment using the DPC.

Wavelength Range

The Novacure is a filter photometer instrument. A broad-band filter supplied with the instrument covers 320 to 500 nm and is suitable for most UV and Visible DPC studies. Optional broad-band filters covering the UV (250 to 450 nm) and Visible (400 to 500 nm) regions are available. Narrow band and neutral density filters which mount at the DSC end of the light guide, to further isolate/control specific spectral regions and light intensity, are also options.

Light Intensity

Light intensity can be measured at two points ..at the end of the light guide and at the DSC cell sample and reference platforms. The operator selects the intensity for an experiment on the Novacure unit and then “calibrates” the output at the end of the light guide using the internal radiometer to achieve and maintain that intensity.

The intensity at the cell is a more important experimental parameter because it generally affects the measured rate of reaction. Cell intensity is measured after the light guide is mounted in the cell adapter by placing the adapter over an external radiometer sensor. (Note: An external radiometer is not supplied with the DPC.) Since the external radiometer sensor is essentially at the same distance from the end of the light guide as the DSC cell platforms are when the adapter is mounted on the cell, the intensity measured is representative of what the sample is exposed to during the experiment.

NOTE: The cell adapter has been designed so that the end of the light guide is located the proper distance from the DSC platforms to generate a circle of light large enough to cover the entire sensor area.

Baseline Offset & Noise

As mentioned in the previous section, as light exits the light guide into the DSC cell, the beam gradually spreads out into a circle whose diameter is directly related to the height of the light guide from the sample. The height of the cell adapter, therefore, is designed to provide a circle of light that is just large enough to cover the DSC constantan disk.

NOTE: The adapter does provide the ability to make minor vertical adjustments to accommodate DSC cell to cell variations.

Although the light guide is designed to pass a uniform beam of light, and although the DSC cell constantan disk is designed so that the heat flow characteristics of the sample and reference position are as identical as possible, it is impossible to obtain an absolutely balanced situation. If everything were perfect, the amount of light striking the sample and reference platforms would be identical and the Δ Temperature/differential heat flow measured by the cell, in the absence of a sample or pans, would be zero. In reality, there will be some imbalance and on exposure to light, a heat flow offset will be observed. Rotational adjustments should be made on the cell adapter until this offset (either positive or negative) is minimal.

Likewise, the light intensity delivered through the light guide should ideally be constant. However, the lamp used in the Novacure is a high pressure mercury arc, which has some inherent fluctuations in its output. The DSC cell is sensitive enough to detect those small fluctuations in light intensity as noise in the heat flow baseline. Noise, as expected, increases with the light intensity reaching the cell. There is no adjustment to eliminate this noise. Fortunately, the noise is low relative to the photoinitiated events being studied.

Chapter 2

Setting Up the DPC

This section briefly describes the setup of the Differential Photocalorimeter (DPC) accessory. For more details, consult the DSC 2920 and Novacure Operator's Manuals.

1. Use the event cable to connect the DSC Event port to the Novacure I/O Signal port as shown in the figure below.

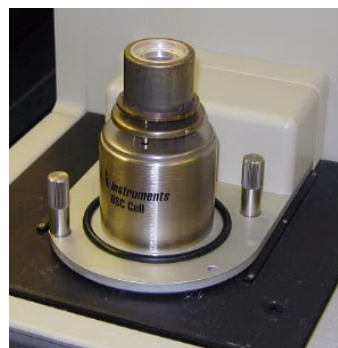


Event Cable

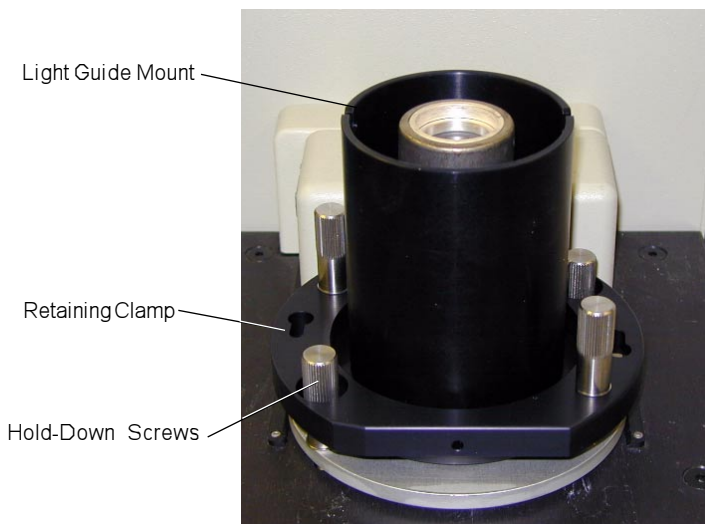
This cable controls the Novacure light source shutter and allows it to be opened and closed using the “Event” method segment in the Thermal Advantage software. “ON” opens the shutter. “OFF” closes the shutter.

NOTE: The Novacure Mode of Operation must be set to “EXTIM” to activate this capability.

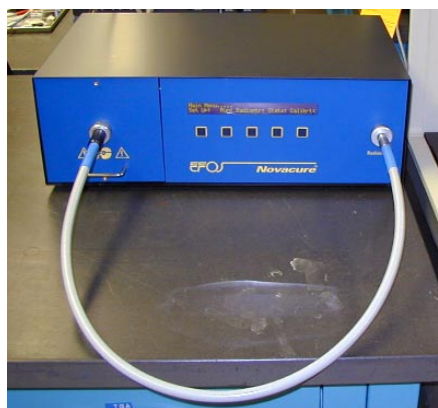
2. Remove the heat shield and silver lid from the DSC 2920 cell, which will leave the cell exposed as seen in the figure to the right.



3. Position the light guide mount around the cell and anchor it to the DSC 2920 base using the retaining clamp and hold-down screws. See the figure here.



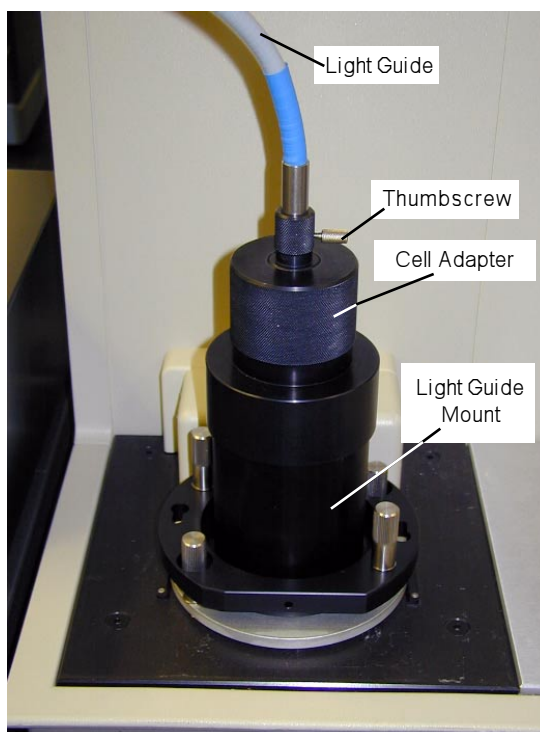
4. Place the modified silver lid in the cell so that the two holes are positioned over the sample and reference positions in the cell. Place the quartz window on top of the silver lid as seen in the figure to the right.



5. Select the desired light intensity at the Novacure source and “calibrate.” Calibration is done with the light guide inserted into the radiometer port as shown in the figure to the left. See the *EFOS Novacure User's Guide Model N2001-A1* for details on calibration.

Please note that the light intensity at the DSC cell sensor will not be the same as that selected and calibrated at the end of the light guide. To determine the relationship between those two intensities requires an external radiometer so that the intensity with the light guide mounted in the cell adapter can be measured. (As a rough guide, 10,000 mW/cm² at the end of the light guide will produce illumination in excess of 150 mW/cm² at the DSC sensor and 1000 mW/cm² will produce illumination in excess of 20 mW/cm² at the DSC sensor.)

6. Insert the cell adapter into the light guide mount to align the light guide over the DSC cell as shown in the figure to the right. There are slots on the mount to ensure reproducible repositioning when the adapter is removed to load a new sample into the DSC.
7. Insert the light guide into the cell adapter.
8. With the light guide mounted over the empty cell, adjust the rotational position of the light guide to minimize offset. This is accomplished by observing change to the Signal A Heat Flow on the TA Instruments



Thermal Analyst controller Signal Display as the light guide is moved. Adjust until this signal is as close to "0" as possible. (This is a very sensitive adjustment; hence actually obtaining "0" will be difficult. Because of the large heat flow signals associated with typical photocalorimetry reactions, offsets of hundreds of microwatts should not be an issue.) Tighten the thumbscrew on the adapter to lock the light guide in place. The light guide and adapter should subsequently be removed and replaced as a single assembly so that offset adjustment remains constant.

(continued)

9. Make sure that the DSC temperature and heat flow response have also been completed using procedures defined in the *DSC 2920 Operator's Manual*. The instrument is now ready to begin conducting DPC experiments.

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