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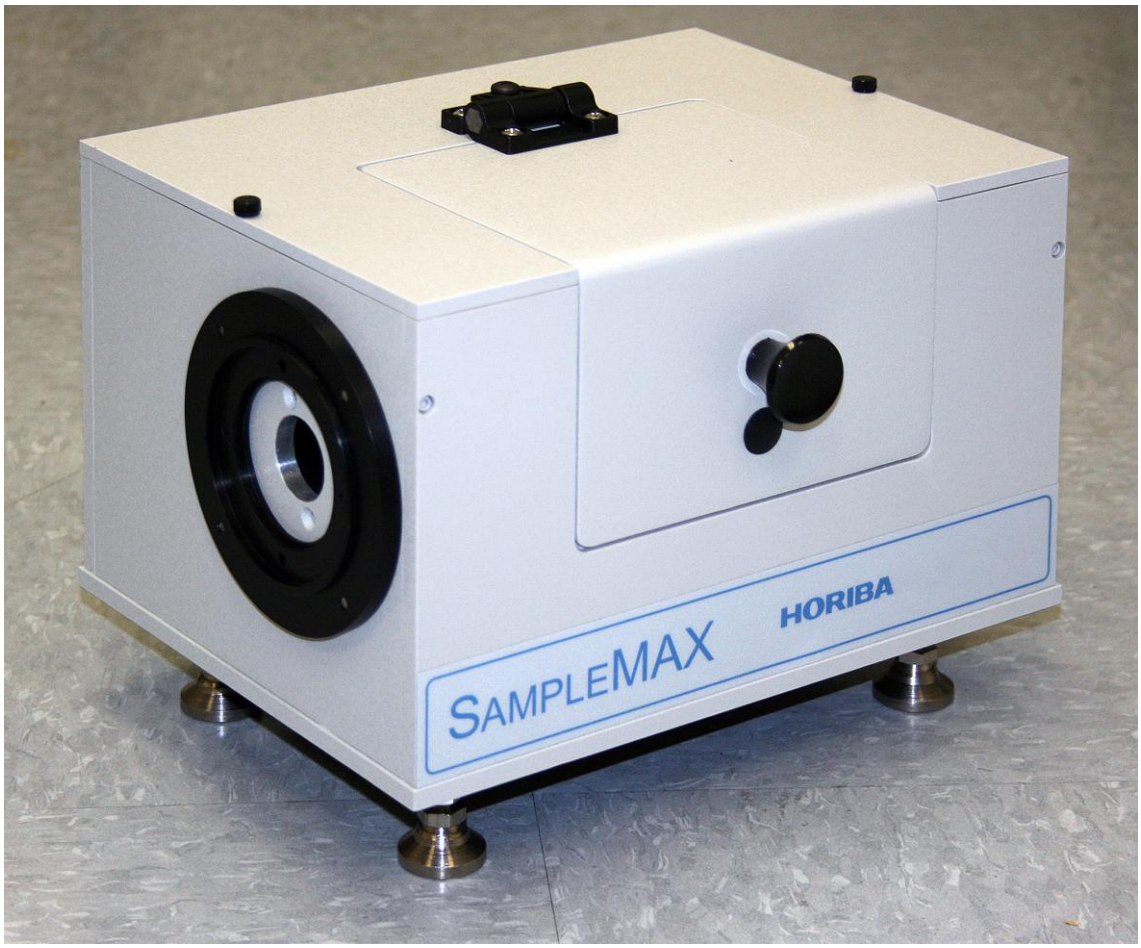
HORIBA Scientific

# SampleMax
# Universal Sample Compartment

## Operation Manual
## Part number J81036 rev. C


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# SampleMax
# Universal Sample Compartment




## Operation Manual

Rev. C
www.HORIBA.com

JOBIN YVON Technology

HORIBA Scientific



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November 2012

Part number J81036



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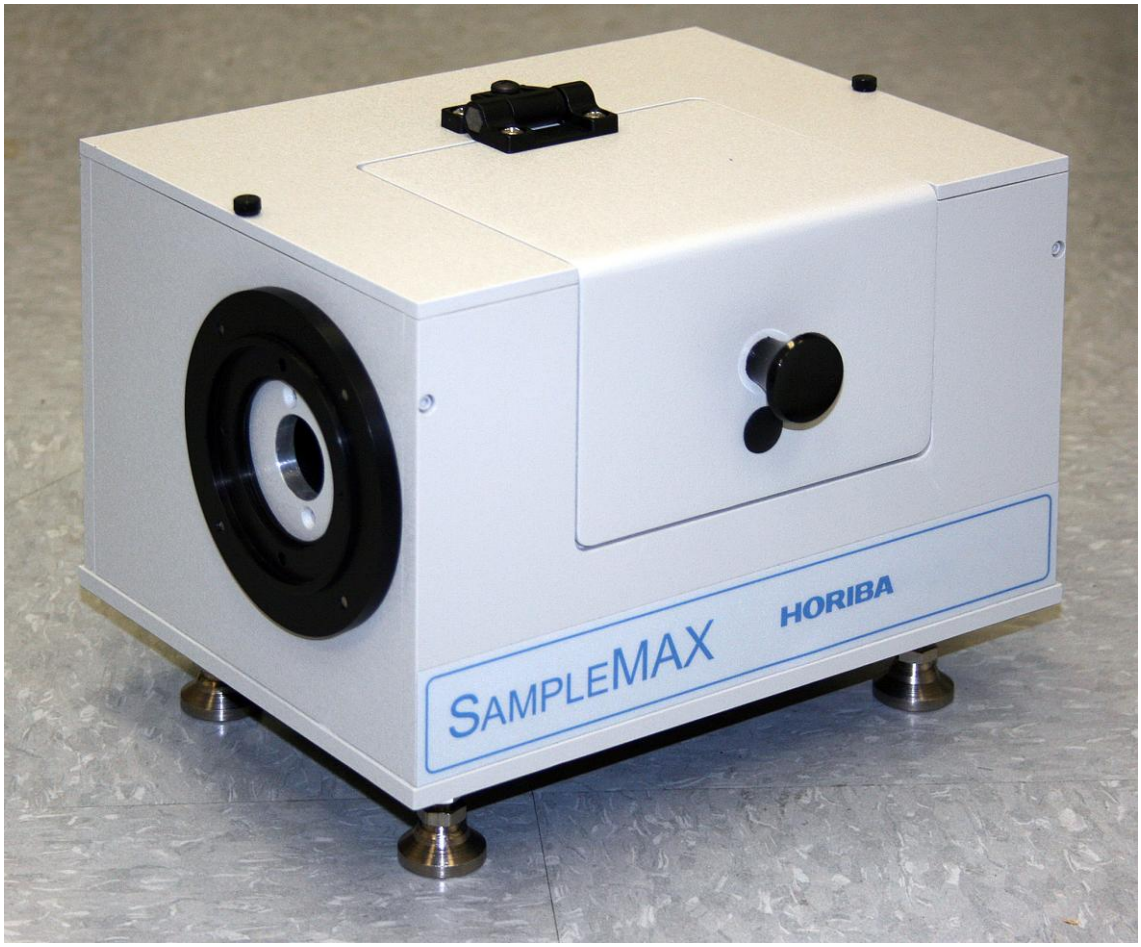

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# 1: Introduction

## About the SampleMax




The SampleMax (part number ASC-xx) is a universal sample compartment designed for a variety of sampling needs, including absorption, reflection, fluorescence, and photoluminescence measurements. Designed to hold thin films, liquids, and solid samples, the SampleMax easily attaches to HORIBA light sources, accessories, and spectrometers. The SampleMax attaches directly to the entrance and exit slits of the TRIAX and iHR series of spectrometers.

There are two models of SampleMax. The ASC-VIS uses glass lenses, and is designed for spectroscopy in the visible and near-infrared bands. The ASC-UV has quartz lenses, and may be used for ultraviolet, visible, and IR measurements.



***Note:*** *Keep this and the other reference manuals near the system.*



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# Chapter overview


| | |
|---|---|
| **1: Introduction** | Safety information |
| **2: Requirements & Installation** | How to set up and use the SampleMax. |
| **3: Experiments** | Several major kinds of experiments in which to use the SampleMax |
| **4: Optional Accessories** | A list of accessories and information about them |
| **5: Service Information** | Service policy, return authorizations, and warranty |
| **6: Adapters** | Connecting adapters for various instruments |
| **7: Index** | |




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# Disclaimer

By setting up or starting to use any HORIBA Instruments Incorporated product, you are accepting the following terms:

You are responsible for understanding the information contained in this document. You should not rely on this information as absolute or all-encompassing; there may be local issues (in your environment) not addressed in this document that you may need to address, and there may be issues or procedures discussed that may not apply to your situation.

If you do not follow the instructions or procedures contained in this document, you are responsible for yourself and your actions and all resulting consequences. If you rely on the information contained in this document, you are responsible for:

- Adhering to safety procedures
- Following all precautions
- Referring to additional safety documentation, such as Material Safety Data Sheets (MSDS), when advised

As a condition of purchase, you agree to use safe operating procedures in the use of all products supplied by HORIBA Instruments Incorporated, including those specified in the MSDS provided with any chemicals and all warning and cautionary notices, and to use all safety devices and guards when operating equipment. You agree to indemnify and hold HORIBA Instruments Incorporated harmless from any liability or obligation arising from your use or misuse of any such products, including, without limitation, to persons injured directly or indirectly in connection with your use or operation of the products. The foregoing indemnification shall in no event be deemed to have expanded HORIBA Instruments Incorporated's liability for the products.

HORIBA Instruments Incorporated products are not intended for any general cosmetic, drug, food, or household application, but may be used for analytical measurements or research in these fields. A condition of HORIBA Instruments Incorporated's acceptance of a purchase order is that only qualified individuals, trained and familiar with procedures suitable for the products ordered, will handle them. Training and maintenance procedures may be purchased from HORIBA Instruments Incorporated at an additional cost. HORIBA Instruments Incorporated cannot be held responsible for actions your employer or contractor may take without proper training.

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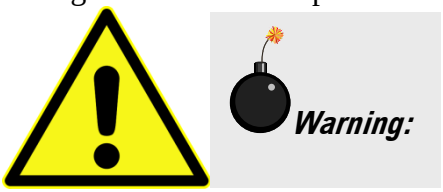
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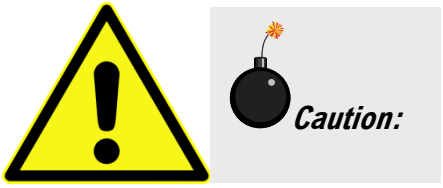
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# Safety summary

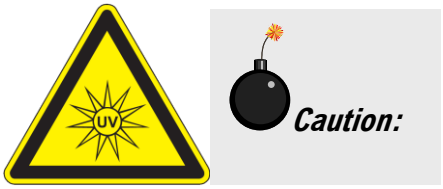
The following general safety precautions must be observed during all phases of operation of this instrument. Failure to comply with these precautions or with specific warnings elsewhere in this manual violates safety standards of design, manufacture and intended use of instrument. HORIBA Instruments Incorporated assumes no liability for the customer's failure to comply with these requirements. Certain symbols are used throughout the text for special conditions when operating the instruments:



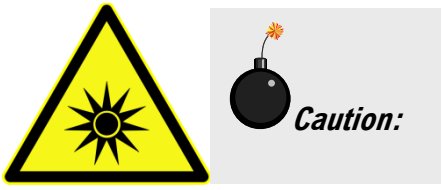

A WARNING notice denotes a hazard. It calls attention to an operating procedure, practice, or similar that, if incorrectly performed or adhered to, could result in personal injury or death. Do not proceed beyond a WARNING notice until the indicated conditions are fully understood and met. HORIBA Instruments Incorporated is not responsible for damage arising out of improper use of the equipment.



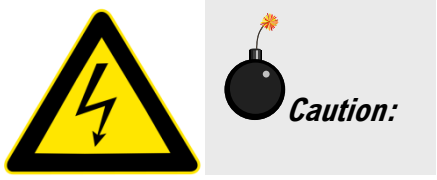

A CAUTION notice denotes a hazard. It calls attention to an operating procedure, practice, or similar that, if incorrectly performed or adhered to, could result in damage to the product. Do not proceed beyond a CAUTION notice until the indicated conditions are fully understood and met. HORIBA Instruments Incorporated is not responsible for damage arising out of improper use of the equipment.




Ultraviolet light! Wear protective goggles, full-face shield, skin-protection clothing, and UV-blocking gloves. Do not stare into light.




Intense ultraviolet, visible, or infrared light! Wear light-protective goggles, full-face shield, skin-protection clothing, and light-blocking gloves. Do not stare into light.



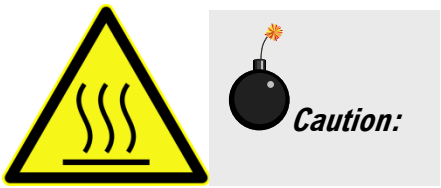

Risk of electric shock! This symbol warns the user that un-insulated voltage within the unit may have sufficient magnitude to cause electric shock.



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This symbol cautions the user that excessive humidity, if present, can damage certain equipment.




Hot! This symbol warns the user that hot equipment may be present, and could create a risk of fire or burns.



Read this manual before using or servicing the instrument.



Wear protective gloves.



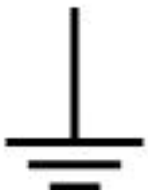
Wear appropriate safety goggles to protect the eyes.



Wear an appropriate face-shield to protect the face.



Disconnect instrument from wall outlet (mains) before servicing.



Earth (ground) terminal; indicates a circuit-common connected to grounded (earthed) chassis.



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Protective earth (ground) terminal.



Alternating current.



On (electrical supply).



Off (electrical supply)



***Note:***

General information is given concerning operation of the equipment.



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# Risks of ultraviolet exposure




***Caution:*** *This instrument is used in conjunction with ultraviolet light. Exposure to these radiations, even reflected or diffused, can result in serious, and sometimes irreversible, eye and skin injuries.*

*Overexposure to ultraviolet rays threatens human health by causing:*

- *Immediate painful sunburn*
- *Skin cancer*
- *Eye damage*
- *Immune-system suppression*
- *Premature aging*

Do not aim the UV light at anyone.
Do not look directly into the light.
Always wear protective goggles, full-face shield and skin protection clothing and gloves when using the light source.

- Light is subdivided into visible light, ranging from 400 nm (violet) to 700 nm (red); longer infrared, “above red” or > 700nm, also called heat; and shorter ultraviolet radiation (UVR), “below violet” or < 400nm. UVR is further subdivided into UV-A or near-UV (320–400 nm), also called black (invisible) light; UV-B or mid-UV (290–320 nm), which is more skin penetrating; and UV-C or far-UV (< 290 nm).

- Health effects of exposure to UV light are familiar to anyone who has had sunburn. However, the UV light level around some UV equipment greatly exceeds the level found in nature. Acute (short-term) effects include redness or ulceration of the skin. At high levels of exposure, these burns can be serious. For chronic exposures, there is also a cumulative risk of harm. This risk depends upon the amount of exposure during your lifetime. The long-term risks for large cumulative exposure include premature aging of the skin, wrinkles and, most seriously, skin cancer and cataract.

- Damage to vision is likely following exposure to high-intensity UV radiation. In adults, more than 99% of UV radiation is absorbed by the anterior structures of the eye. UVR can contribute to the development of age-related cataract, pterygium, photodermatitis, and cancer of the skin around the eye. It may also contribute to age-related macular degeneration. Like the skin, the covering of the eye or the cornea, is epithelial tissue. The danger to the eye is enhanced by the fact that light



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can enter from all angles around the eye and not only in the direction of vision. This is especially true while working in a dark environment, as the pupil is wide open. The lens can also be damaged, but because the cornea acts as a filter, the chances are reduced. This should not lessen the concern over lens damage however, because cataracts are the direct result of lens damage.

Burns to the eyes are usually more painful and serious than a burn to the skin. Make sure your eye protection is appropriate for this work. NORMAL EYEGLASSES OR CONTACTS OFFER VERY LIMITED PROTECTION!






***Caution:*** *UV exposures are not immediately felt. The user may not realize the hazard until it is too late and the damage is done.*

## Training

For the use of UV sources, new users must be trained by another member of the laboratory who, in the opinion of the member of staff in charge of the department, is sufficiently competent to give instruction on the correct procedure. Newly trained users should be overseen for some time by a competent person.



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# Additional risks of xenon lamps





***Warning:*** *Xenon lamps are dangerous. Please read the following precautions.*

Among the dangers associated with xenon lamps are:

- Burns caused by contact with a hot xenon lamp.
- Fire ignited by hot xenon lamp.
- Interaction of other nearby chemicals with intense ultraviolet, visible, or infrared radiation.
- Damage caused to apparatus placed close to the xenon lamp.
- Explosion or mechanical failure of the xenon lamp.

## Visible radiation

Any very bright visible light source will cause a human aversion response: we either blink or turn our head away. Although we may see a retinal afterimage (which can last for several minutes), the aversion response time (about 0.25 seconds) normally protects our vision. This aversion response should be trusted and obeyed. NEVER STARE AT ANY BRIGHT LIGHT-SOURCE FOR AN EXTENDED PERIOD. Overriding the aversion response by forcing yourself to look at a bright light-source may result in permanent injury to the retina. This type of injury can occur during a single prolonged exposure. Excessive exposure to visible light can result in skin and eye damage.

Visible light sources that are not bright enough to cause retinal burns are not necessarily safe to view for an extended period. In fact, any sufficiently bright visible light source viewed for an extended period will eventually cause degradation of both night and color vision. Appropriate protective filters are needed for any light source that causes viewing discomfort when viewed for an extended period of time. For these reasons, prolonged viewing of bright light sources should be limited by the use of appropriate filters.

The blue-light wavelengths (400–500 nm) present a unique hazard to the retina by causing photochemical effects similar to those found in UV-radiation exposure.

## Infrared radiation

Infrared (or heat) radiation is defined as having a wavelength between 780 nm and 1 mm. Specific biological effectiveness “bands” have been defined by the CIE (Commission Internationale de l’Eclairage or International Commission on Illumination) as follows:

• IR-A (near IR) (780–1400 nm)
• IR-B (mid IR) (1400–3000 nm)
• IR-C (far IR) (3000 nm–1 mm)



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The skin and eyes absorb infrared radiation (IR) as heat. Workers normally notice excessive exposure through heat sensation and pain. Infrared radiation in the IR-A that enters the human eye will reach (and can be focused upon) the sensitive cells of the retina. For high irradiance sources in the IR-A, the retina is the part of the eye that is at risk. For sources in the IR-B and IR-C, both the skin and the cornea may be at risk from "flash burns." In addition, the heat deposited in the cornea may be conducted to the lens of the eye. This heating of the lens is believed to be the cause of so called "glassblowers' " cataracts because the heat transfer may cause clouding of the lens.

- Retinal IR Hazards (780 to 1400 nm): possible retinal lesions from acute high irradiance exposures to small dimension sources.
- Lens IR Hazards (1400 to 1900 nm): possible cataract induction from chronic lower irradiance exposures.
- Corneal IR Hazards (1900 nm to 1 mm): possible flashburns from acute high irradiance exposures.

Who is likely to be injured? The user and anyone exposed to the radiation or xenon lamp shards as a result of faulty procedures. Injuries may be slight to severe.



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# 2: Requirements & Installation

## Safety-training requirements

Every user of the SampleMax must know general and specific safety procedures before operating the instrument. For example, proper training includes (but is not limited to):

- Understanding the risks of exposure to ultraviolet, visible, and infrared light, and how to avoid unsafe exposures to these types of radiation
- Safe handling for all chemicals and other samples used in the instrument

Safety-training may be purchased from HORIBA Scientific. Contact your Sales Representative or the Service Department for details.



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# Unpacking and installation

## Introduction

Before use, set up your SampleMax and all accompanying components. The installation of your HORIBA Scientific SampleMax has several separate stages that must be completed in the order below for the system to operate properly.

- Unpacking and equipment inspection
- Installing tilt feet
- Installing the lenses



***Note:*** *Many public carriers will not recognize a claim for concealed damage if it is reported later than 15 days after delivery. In case of a claim, inspection by an agent of the carrier is required. For this reason, the original packing material should be retained as evidence of alleged mishandling or abuse. While HORIBA Instruments Incorporated assumes no responsibility for damage occurring during transit, the company will make every effort to aid and advise.*





***Caution:*** *The SampleMax and accessories are delicate. Mishandling may seriously damage their components. The HORIBA Instruments Incorporated warranty on the SampleMax does not cover damage to the system's optical components that arises as a result of improper handling.*

## Table 1: SampleMax carton contents

| Component | Part number |
|---|---|
| SampleMax | ASC-VIS or ASC-UV |
| Cuvette Holder (included in SampleMax) | J350590 |
| Leveling Feet × 4 | J992100 |
| *SampleMax Operation Manual* | J81036 |

## Directions

### 1 Unpack the SampleMax and place it in its location.

a Carefully open the shipping carton.



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b Remove the foam-injected top piece and any other shipping restraints in the carton.

c With assistance, carefully lift the instrument from the carton, and rest it on the side of the laboratory bench where the system will stay.

d Place the instrument in its permanent location.



***Caution:*** *The SampleMax and accessories are delicate. Mishandling may seriously damage their components. The HORIBA Instruments Incorporated warranty on the SampleMax does not cover damage to the system's optical components that arises as a result of improper handling.*



The first step in using your SampleMax is determining its location in the experiment. If you are unsure about how to fit the sample chamber among the other instruments, consult the *Experiments* section later in this manual for some examples. Because all ports may be used as either entrance or exit ports, you have some flexibility with regard to absorption and reflection experiments. For photoluminescence and fluorescence measurements using a laser, leave the front cover hole exposed to allow the beam to enter the sample chamber. If you have custom modifications made on your SampleMax, consult HORIBA for assistance in configuring your system.

e Inspect for previously hidden damage.
Notify the carrier and HORIBA Scientific if any is found.



***Note:*** *Many public carriers will not recognize a claim for concealed damage if it is reported later than 15 days after delivery. In case of a claim, inspection by an agent of the carrier is required. For this reason, the original packing material should be retained as evidence of alleged mishandling or abuse. While HORIBA Instruments Incorporated assumes no responsibility for damage occurring during transit, the company will make every effort to aid and advise.*

f Check the packing list to verify that all components and accessories are present.

## 2 Attach the feet.

Attach the feet included with the SampleMax and adjust them so that the chamber is level and that the height of the ports matches those of the attached



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instruments. From the top view, screw the feet clockwise to raise the height, and counterclockwise to lower it.

## 3 Insert the lenses.

Place the mounted lenses in the optical rails at the approximately correct location (see the section on *Lens configurations* later in the manual).



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# On configuration of the SampleMax with other components

A diagram of the standard SampleMax as used in an absorption measurement is presented in Figure 1. Because of the versatility of the sample compartment, several other types of measurements are possible, many using other configurations. For example, reflection measurements would have the entrance and exit optical axes at a 90° angle, and the accompanying optical rails and lenses would accommodate this option. The material (glass or quartz) and focal length of the lenses may also be selected to fit the application.

The SampleMax attaches directly to the entrance and exit slits of the TRIAX and iHR series of spectrometers. The SampleMax also attaches to the M-series instruments, as well as to older HORIBA spectrometers, such as the 270M, 1680/81, 340, and HR460. Any of the standard HORIBA accessories and detectors, such as the 100F, 120F, PMT detectors, and the 220NSA slit adapter, will also mount directly to the ports of the sample compartment. For other adapters, see *Chapter 6: Adapters*. There are no electrical parts and few mechanical adjustments in its operation. The optical design allows very little stray light.

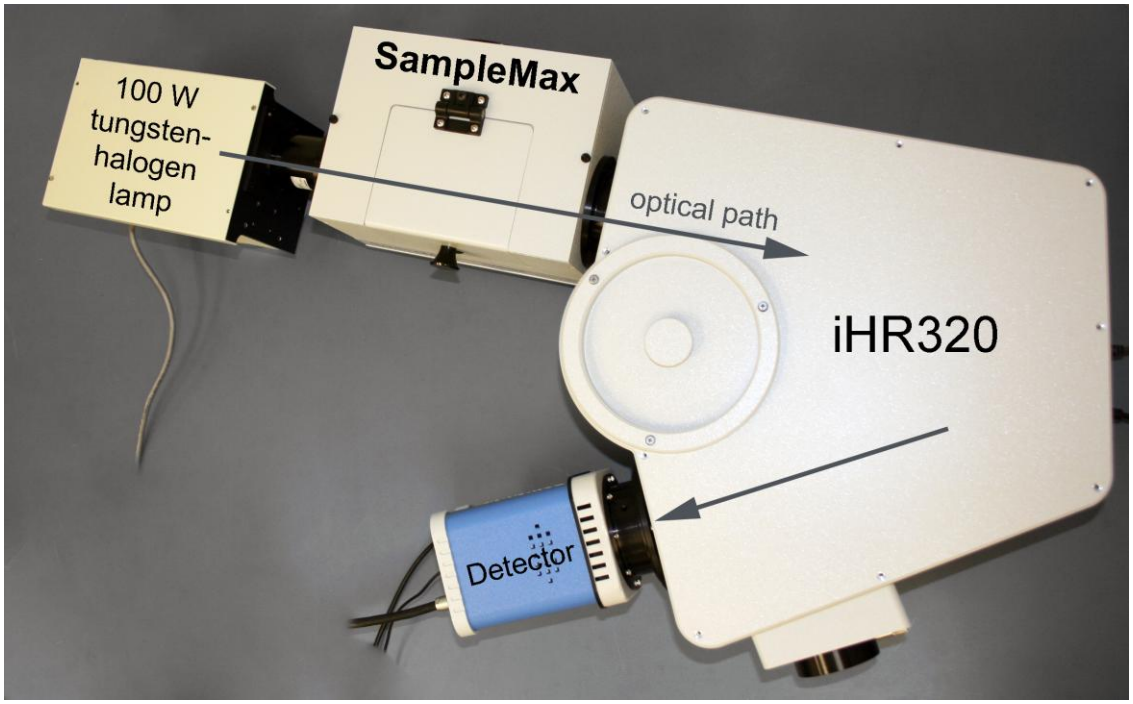



**Figure 1.** SampleMax in an absorption experiment



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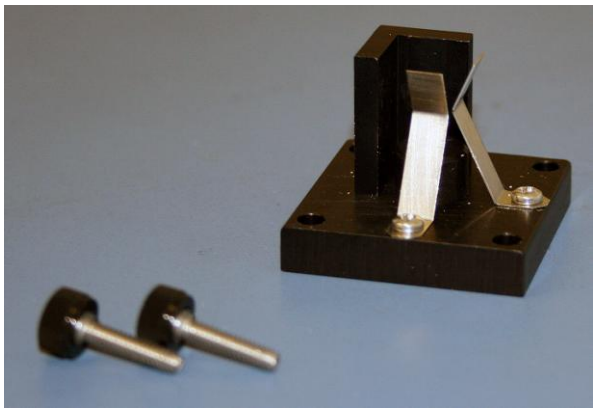


**Figure 2.** Cuvette holder with two thumbscrews

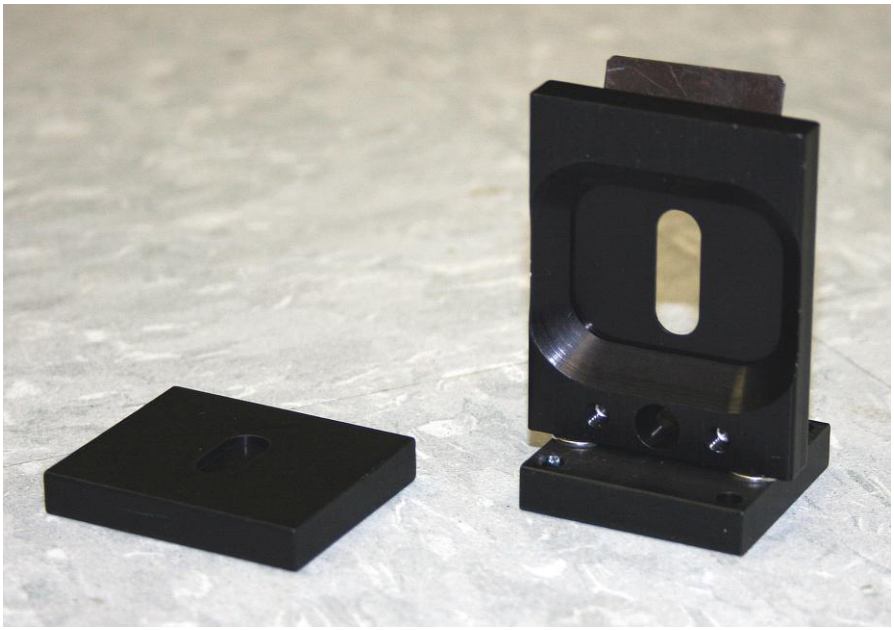


**Figure 3.** Solid-sample holder with block



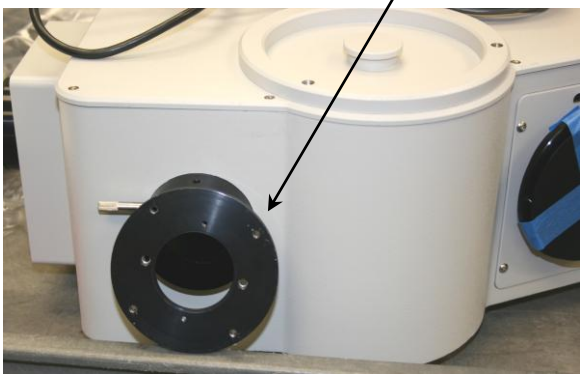
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# Mounting the SampleMax to an instrument

## iHR and MicroHR spectrometers

***Note:*** *These instructions may be used to mount the SampleMax (ASC-VIS or ASC-UV) on front or side entrance slit, or front or side exit slit.*

1. Remove slit cover from iHR entrance slit.
2. Using the M3 screws removed from the slit cover, mount the SampleMax ring (J350386) to the entrance slit.

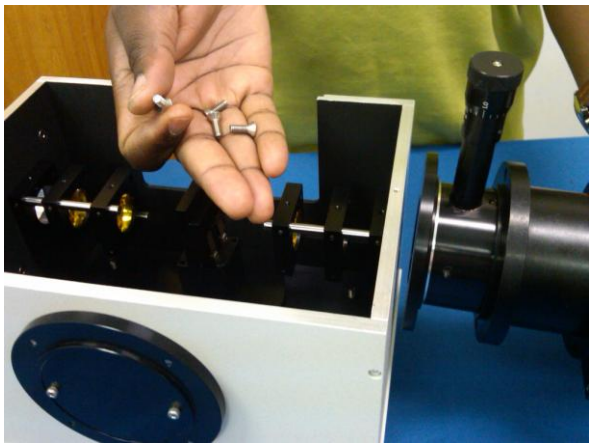


3. Remove the SampleMax sample compartment cover to gain access to the four screws that secure the mounting adapter to the wall of the sample compartment.
4. Place the four 8-32 mounting screws on the front interior wall of the SampleMax, and tighten them to attach to the SampleMax ring.
5. Adjust the height so the SampleMax matches the spectrometer's height.
6. If the SampleMax is set up for a transmission measurement, repeat step 4 to connect the SampleMax to the ASC-1679 slit housing (left



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below) and 1427C detector housing (right below).



**Left:** Attaching the SampleMax to the ASC-1679 slit housing.




**Right:** Interior of LSH housing showing Phillips-head mounting screws (LSH-A270 not shown)




SampleMax attached to iHR



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## M-Series instruments

1. Remove the SampleMax cover to gain access to the four screws that secure the Mounting Adapter to the wall of the SampleMax.
2. Remove the Mounting Adapter.
3. Secure the ASC-1451 M-Series adapter to the M-Series entrance slit using the bolts provided.
4. Using two 6/32″ cap-head screws, secure the Mounting Adapter to the ASC-1451.
5. Using the four screws previously removed, re-secure the Mounting Adapter to the SampleMax.

## TRIAX series

Mounting instructions are similar to the iHR series.



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# Sample placement within the SampleMax

## Introduction

For all types of samples, it is important that the main sample stage be in the correct position. Normally, without the optional external turret rotator, this position is set during the calibration of your system at HORIBA. For a SampleMax with the external turret rotator, ensure the proper position by rotating the turret wheel from the inside until the stage locks. Then, using the appropriate sample holder, mount the sample accordingly.

## Cuvette

For liquid samples in a cuvette, make sure that the entrance and exit faces are clean and clear. The outer faces may be cleaned using methanol and lens tissue. There should be no fingerprints on any surface to be illuminated. (To probe only the sample inside the cuvette, the walls must be transparent to the light.) For this reason, UV measurements require quartz cuvettes, for cuvettes made of ordinary glass absorb ultraviolet radiation. If the cuvette is not 100% transparent to the light and you are making transmission measurements of the sample, you may create a correction that takes this factor into account. This procedure is covered in the *Experiments* section later in the manual.





***Caution:*** *Refer to your Material Safety Data Sheets (MSDS) for hazards regarding the use of methanol.*

To place the cuvette into its sample holder, slide it gently between the two flexible metal clips and the solid retaining pieces on the stage until the bottom of the cuvette is flush with the stage. Be sure that the optical faces are in the proper location for the application (i.e., facing the light paths). For transmission measurements, the walls of the cuvette should be perpendicular to the optical axis and on opposite sides of the cuvette (Figure 4).

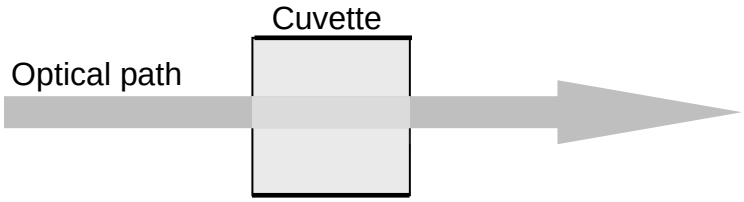



**Figure 4.** Top view of optical path through cuvette for transmission measurements

For fluorescence experiments, the optical walls should be adjacent at right angles to each other and also perpendicular to the optical axes (Figure 5).



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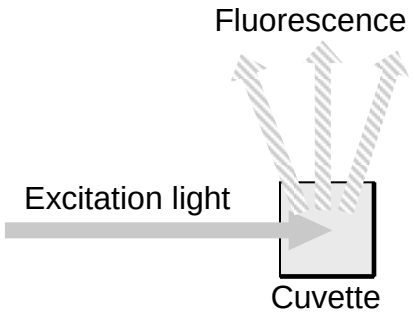



**Figure 5.** Top view of optical path through cuvette for fluorescence measurements

## Solid-Sample Holder

The solid-sample holder has an aluminum base with a spring-loaded flexible metal clip to hold the sample in place. Place the solid sample between the black plate and the holding piece so that the maximum amount of the sample is visible through the hole in the aluminum portion (Figure 3).

When the lenses are aligned and light is provided to the system, check that the sample is in a position of maximum illumination. If you have the optional X-Z stage, you may change the micrometer position instead of moving the sample within the holder.

Included with the holder is also a square metal block with an oval depression on one side. The metal block covers the hole on the aluminum piece and is better for holding the sample for reflectance and fluorescence measurements. Not using the block allows light transmission through the hole in the sample holder, and is recommended for transmission measurements (Figure 6).

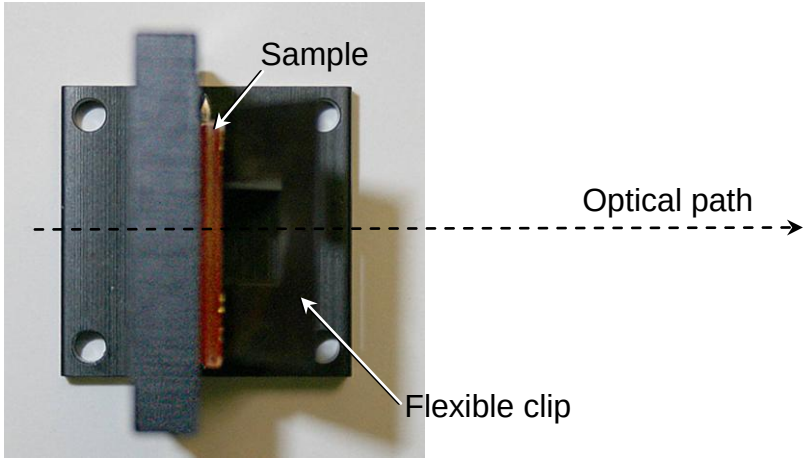



**Figure 6.** Top view of absorption light-path with solid-sample holder

For reflectance, using the block, place the sample-holder so that the sample is 45° with respect to the optical axes (Figure 7). The front of the sample-holder (i.e., the end without the spring-loaded piece) should face the light source. If the metallic holding-



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clip faces the entrance, you may see unwanted scattered light off of it. Tighten the sample-holder to the SampleMax using a small 4-40 screw to ensure a stable position.

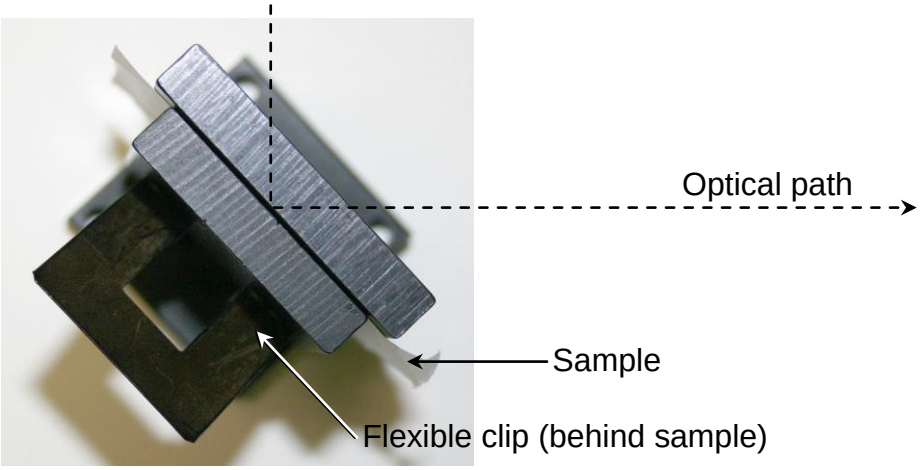



**Figure 7.** Top view of reflection light-path with solid-sample holder



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# Lens configurations

You can set up your lens configuration in various ways. Here are some examples of common applications within the SampleMax.

## Single-lens configuration (for absorption and transmission)

This configuration refers to a single lens both before and after the sample, used in the setups shown in Figures 1 and 12. Depending on the application, alignment using the standard SampleMax package typically involves moving two lenses, one before the sample and one afterward, along the optical axis. When the sample is in place in the appropriate holder and the input light source has been introduced, proper adjustment of the lenses should provide the best possible throughput and sample illumination for this particular configuration. Figure 8 below shows a representation of a well-aligned SampleMax for an absorption experiment using a cuvette holder.

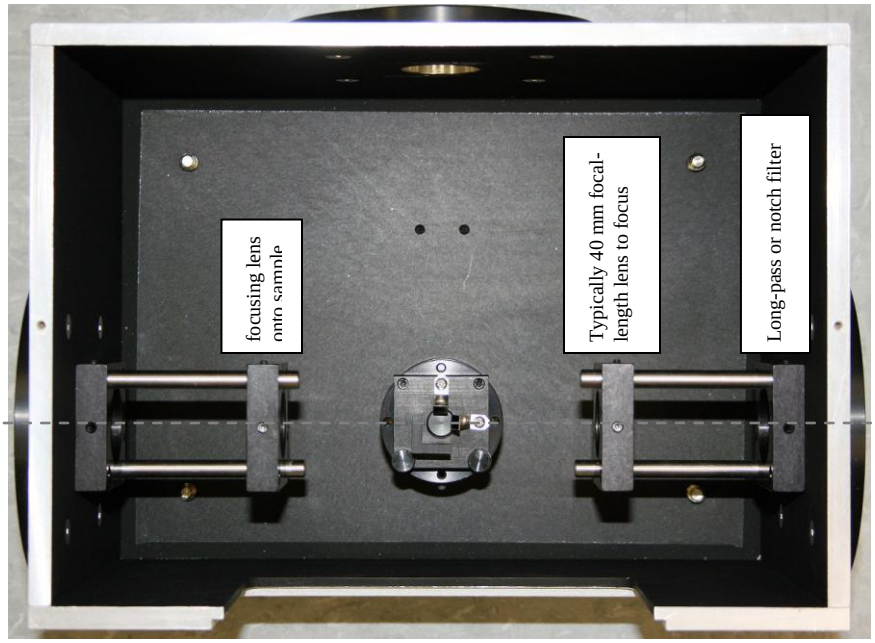



**Figure 8.** SampleMax single-lens configuration for absorption and transmission

For many applications, focus the light at the sample, in order to maximize the intensity of the light on the sample and avoid light-clipping on the sample holder. If the input light is collimated, as in a laser source, place this lens on the optical rail at a distance from the sample equal to its focal length. Using a business card to observe the beam-size at the focus, move the lens until the beam is smallest at the location of the sample. It probably helps to remove the sample from the compartment for ease of alignment. When you have found the optimal location for this lens, tighten the set screw to the optical rail to lock its position.



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If the input light diverges (as in a point source), positioning the lens to focus the light at the sample is a bit more complicated. See the section on *Optical Considerations* for a more detailed discussion of lens optics. The goal of minimum spot-size at the sample remains the same, however. Without any further knowledge of optics, you may simply move the lens until this condition is met. Again, alignment generally works best without the sample in the sample compartment. For collimated light at the sample, place the lens at a distance from the point source equal to the lens' focal length.

After focusing at the sample, the beam begins to diverge, but most applications require focusing at the output of the sample compartment. Using the second lens at the appropriate distance from the sample, the collected light should be focused at the output port. As with focusing, it is not necessary to know the optical equations involved, but such information is presented in the *Optical Considerations* section. It is sufficient to move the lens along the optical rails until the focused light is visible at the output using a business card. For 180° configurations (i.e., transmission measurements), removing a relatively opaque sample should help in exit alignment. For 90° experiments (e.g., reflectance measurements), using a mirror or other relatively high reflector in place of the sample should similarly aid output alignment. For collimated light at the sample, simply place this second lens at a distance from the exit target (e.g., the entrance slit of the monochromator) equal to the lens' focal length.

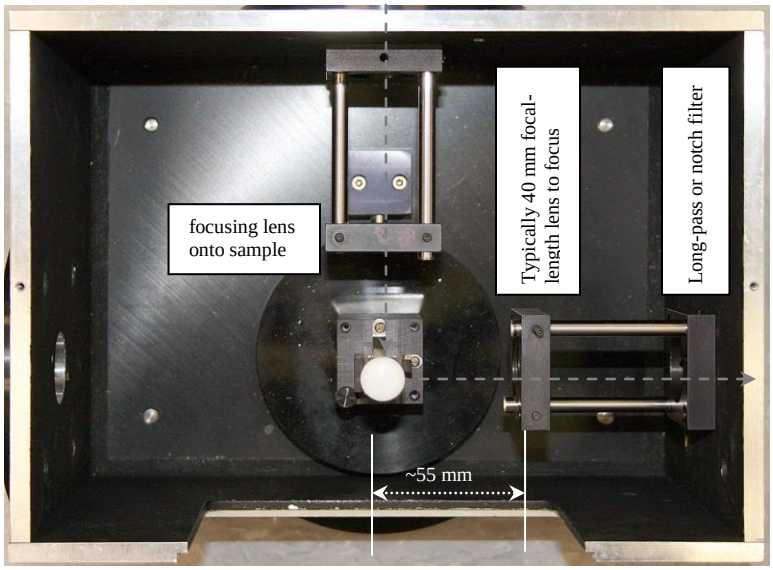



**Figure 9.** SampleMax single-lens configuration for laser-excited photoluminescence of liquids, or Raman spectroscopy

## Dual-lens configuration (for fluorescence, photoluminescence, and Raman)

This configuration refers to any setup with two lenses on at least one optical rail. For the maximum throughput and matching of the *f*/#, use three lenses, one before the sample and two afterward. Figure 10 presents a photograph of the SampleMax used in a common dual-lens configuration.



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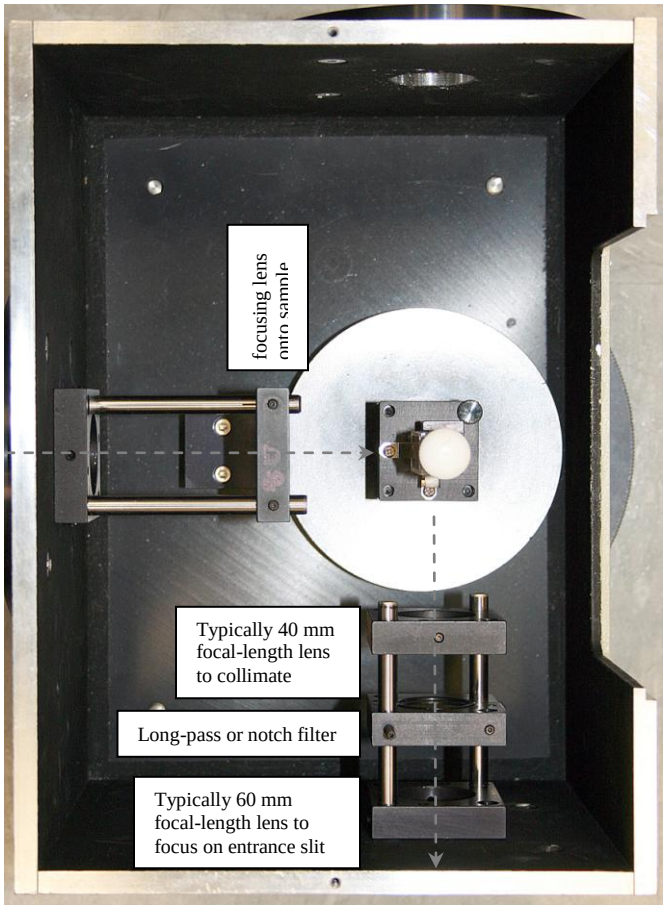



**Figure 10.** SampleMax with dual-lens configuration, assuming a collimated light-source (e.g., laser), for fluorescence of liquids, photoluminescence of solids, or Raman spectroscopy.



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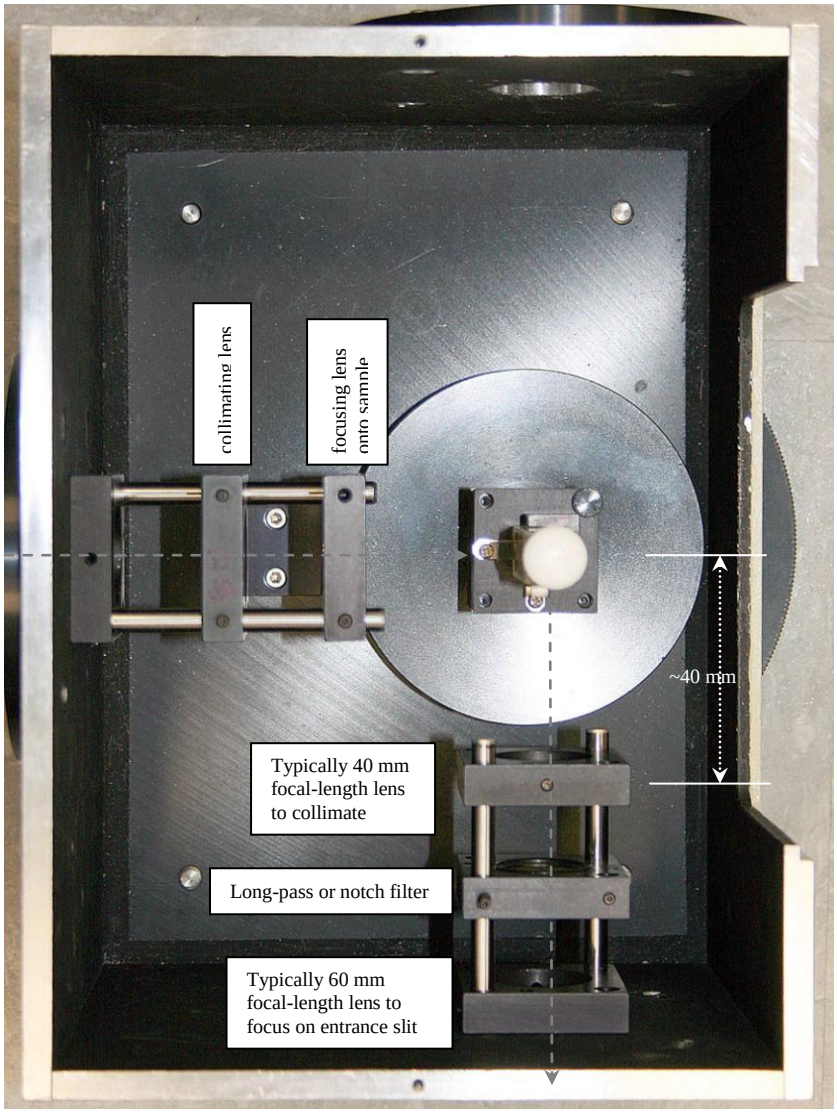



**Figure 11.** SampleMax with dual-lens configuration, assuming a divergent light-source



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# 3: Experiments

## Absorbance/transmttance

Absorption measurements require a light source, SampleMax, spectrometer, detector, and data-acquisition electronics and software. For these measurements, the incident light must pass through the sample chamber without deflection. The two short side ports must be used, with the long side port blocked off to prevent stray light. Connect one port to the input light and the other to the output; it does not matter which port is connected to the input or output. A diagram of an absorption experiment is shown in Figure 1 on page 17.

Because the intensity- and wavelength-dependence of the incident light is different for different configurations, each measurement should be referenced to a scan taken under the same conditions with no sample in place. The ratio of the output light-intensity with the sample in place to the reference intensity is the transmission of the sample, which is frequently expressed as a percentage, %*T*. The absorption is simply the negative logarithm (base 10) of the transmission, or the negative logarithm of the percent transmission plus 2:

$$A = -\log T = 2 - \log (\%T)$$

For liquid measurements using a cuvette, the optical faces must be perpendicular to the incident and transmitted light. For best results, the spot size of the light should be smaller than the width of the cuvette to prevent light scattering. Take reference scans with a clean, empty cuvette (of the same material) in place in the SampleMax.

For solid and thin-film measurements, use the solid-sample holder with the transmissive spring-loading holding piece for maximum transmission. The sample face should be perpendicular to the light, and the spot size should be smaller than the sample-holder hole to prevent light scattering. As with the cuvette, perform reference scans with an empty solid-sample holder in the SampleMax.

To include correction factors for imperfect optical components, use the following equations:

$$\text{Transmittance, } \%T = 100\% \times \frac{\text{Spectrum(corrected)}}{\text{Blank(corrected)}}$$

$$\text{Absorbance, } A = -\log_{10}\left(\frac{\text{Spectrum(corrected)}}{\text{Blank(corrected)}}\right)$$

Two possible orientations exist for absorption experiments. The first, illustrated in Figure 1, has the broadband light source pass through the sample into the spectrometer. The spectrometer, in turn, resolves the light into its wavelength components to be



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detected. The second method, illustrated in Figure 12, has the light source pass through the spectrometer, which serves as a monochromator. The monochromatic light passes through the sample and is then detected. Either method is valid and allows transmission versus wavelength curves to be determined.

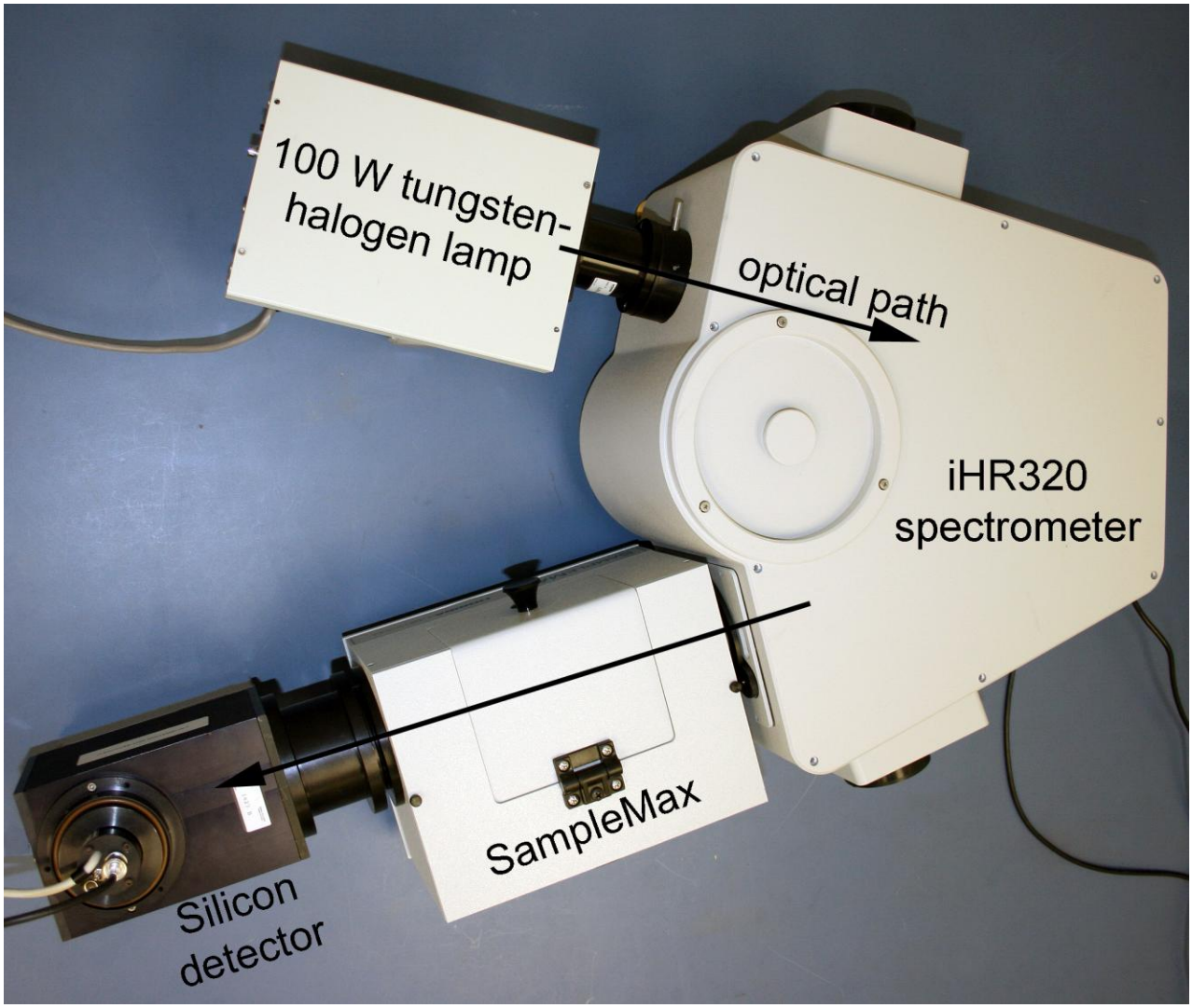



**Figure 12.** Alternate absorption experiment configuration using the SampleMax



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# Reflectance and fluorescence

Reflectance and liquid fluorescence measurements require a 90° angle configuration in which the incident light is perpendicular to the detected light. To attain this configuration, use one short side port and the long side port; the specific ports are not important, but the remaining port should be blocked to prevent stray light. A diagram of a typical reflectance measurement configuration is provided in Figure 13.

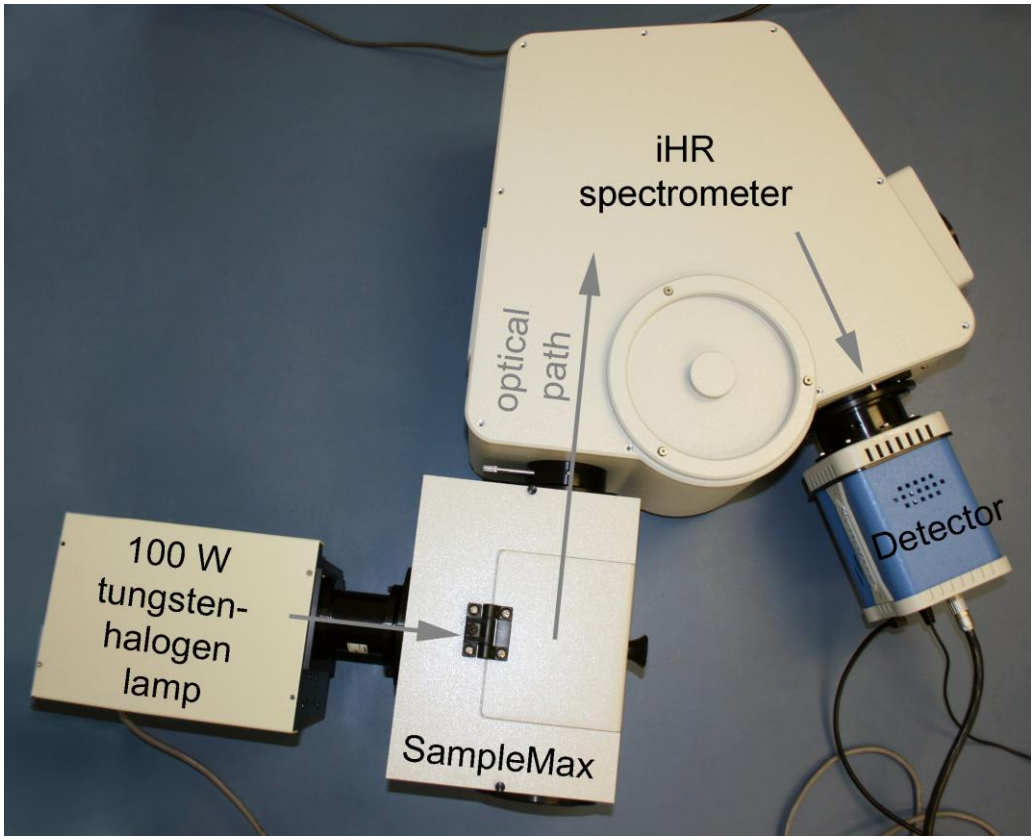



**Figure 13.** Reflection system using the SampleMax

For fluorescence measurements using a cuvette, the optical faces must be perpendicular to the input and output light. The placement of the cuvette is therefore the same as with transmission measurements, but there must be adjacent optical faces for fluorescence experiments.

The excitation source for a fluorescence experiment is usually monochromatic. Often a tunable light source (white light + second monochromator) is mounted on the SampleMax input, in place of the 100 W tungsten-halogen lamp shown in Figure 13.



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# Photoluminescence

Photoluminescence (PL) experiments require a laser, SampleMax, spectrometer, and detector. A typical diagram of these experiments is provided in Figure 14. You may use a single-lens (Figure 9) or dual-lens configuration (Figure 10). Also, you may place an optical chopper between the laser and SampleMax. The ACH-C chopper from HORIBA Scientific may be mounted on the SampleMax using adapter J36540 if you wish.

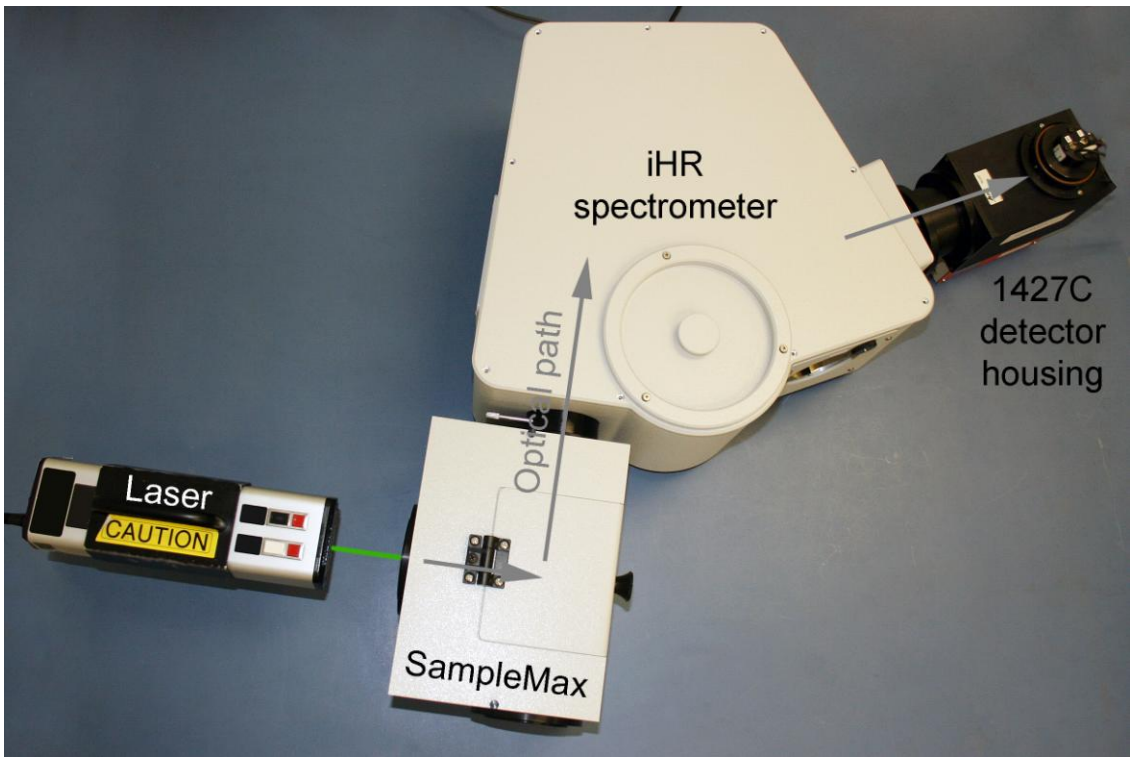



**Figure 14.** Photoluminescence experiment using the SampleMax



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# Optical considerations

For the sake of this discussion, there are two types of lenses: those of **positive focal-length** and those of **negative focal-length**. Positive focal-length lenses can both focus collimated light and collimate diverging light (Figure 15). Negative lenses can collimate converging light and expand collimated light (Figure 16). The lenses in a SampleMax are generally of positive focal length.

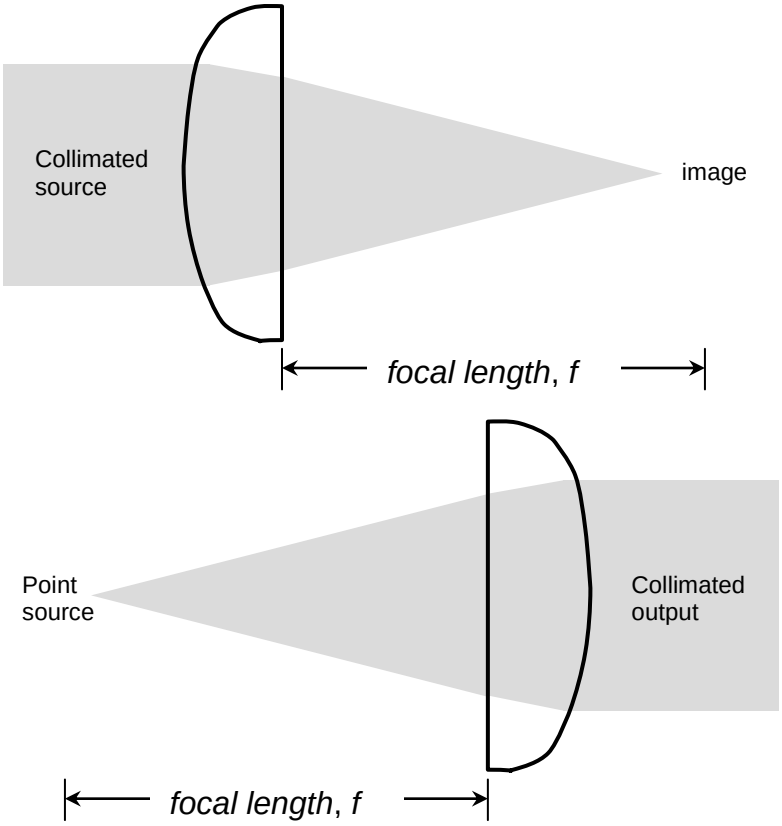



**Figure 15.** Lenses with a positive focal length

Other than the material that comprises the lens, there are two important parameters associated with lenses: the **diameter** and the **focal length**. The diameter refers to the long diameter perpendicular to the optical axis. The focal length is a measure of how tightly a lens focuses. The focal length is given as the distance from the focal point the lens must be to collimate the light (for a diverging source) or focus the light (for a collimated source). A collimated laser incident on a lens with a +60 mm focal length lens will be focused 60 mm beyond the lens.



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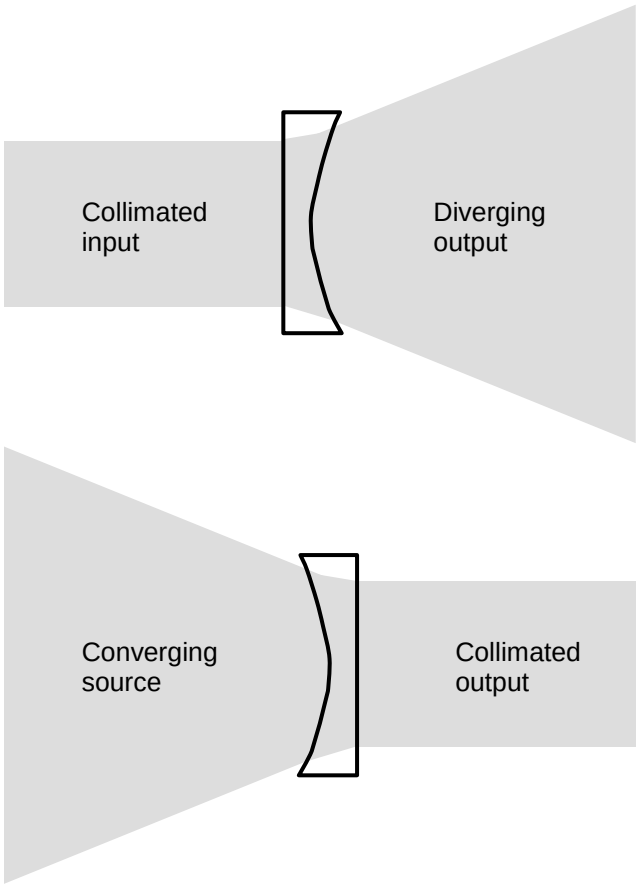



**Figure 16.** Lenses with a negative focal length



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# Basic optics equations

## Single-lens configuration

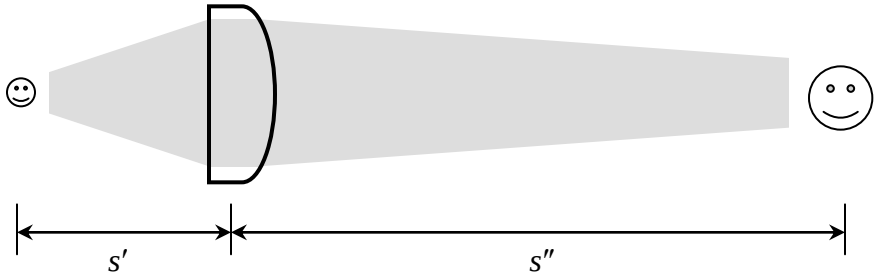



**Figure 17.** Single lens with light source

Referring to Figure 17, the basic lens equation for focusing is:

$$\frac{1}{f} = \frac{1}{s'} + \frac{1}{s''}$$

where $f$ is the focal length of the lens, $s'$ is the distance between the object and the lens, and $s''$ is the distance from the lens to the projected image. A divergent light source 60 mm away from a lens with a 40 mm focal length lens focuses 120 mm away from this lens. For a collimated source, $s'$ is infinite and $f = s''$. Similarly, a lens placed at a distance $f$ from a point source collimates the light. If the lens is placed closer to the light source than the focal length of the lens, $s''$ is negative, and the light does not focus.

Another factor to consider involves magnification of the image. In Figure 17, the image of the source is larger than the source itself because $s''$ is longer than $s'$. The magnification ratio can be expressed simply as follows:

$$m = \frac{s''}{s'}$$

For a tighter focus, keep the magnification to a minimum and move the single lens closer to the focal point. For greater collection of light from a diverging source, however, place the lens closer to the source, thereby increasing the magnification at the focal point. Because of the compromises that are sometimes made using a two-lens system, you might consider a four-lens configuration as a superior alternative.



***Note:*** *For a given diameter, lenses with shorter focal lengths present more spherical and spatial aberrations. Larger diameters help reduce these aberrations, but the cost is greater.*



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# Dual-lens configuration

With a single-lens system, the lens collects a diverging light source and focuses or collimates. With a dual-lens system, the first lens collects and collimates the light and the second focuses it. The advantages of such a system are better collection efficiency and throughput, tighter focusing, and added flexibility within a fixed distance between light source and focal point. Single-lens principles apply to each lens when configuring a dual-lens system. To collimate a divergent light source, place the first lens away from the source at a distance equal to its focal length. Then, focus the resulting beam by placing the second lens away from the focal point a distance equal to its focal length. The minimum distance between light source and focal point is equal to the sum of the focal lengths plus half the thickness of the lenses. With two $f$ = +60 mm focal length lenses, one can focus light about 125 mm and up from the focal point. With a single $f$ = +60 mm lens, this distance must be at least 240 mm.

Magnification with a dual lens configuration occurs when the focal lengths of the lenses are different. The equation that determines the magnification is as follows:

$$m = \frac{f_1}{f_2}.$$

In this equation, $f_1$ is the focal length of the first lens and $f_2$ is the focal length of the second lens. If the second lens has a shorter focal length than the first lens, the spot size at the focus is smaller than that of the original source.

# *f*/# matching

The *f*/# of a system can be given by the simple relation:

$$f/\# = \frac{d}{l}$$

where $d$ = the diameter of the optic and $l$ = the distance from the focus to that optic. As Figure 18 shows, a larger *f*/# (with *f*/2 "larger" than *f*/4 because of the inverse relationship) has more divergence in the light beam and is considered "faster", signifying greater throughput. The angle $\theta$ is smaller for the *f*/4 instrument as well. The angle of approach to the focal point equals the angle of divergence from the focal point:

$$\frac{d_a}{l_a} = \frac{d_b}{l_b},$$

where *a* refers to the light path before the entrance slit and *b* refers to light after the slit. Light that enters the monochromator and diverges at an angle greater than $\theta$ "overfills" the optics and leads to scattered light in the monochromator. Light that enters at an angle less than $\theta$ underfills the optics and sacrifices throughput. For this reason, the light focused onto the entrance slit of a monochromator should have the same *f*/# as the monochromator itself. This is known as ***f*/# matching**. Collimated light filling a one inch (25 mm) diameter lens before the monochromator should have a focal length equal



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to 25 mm divided by the *f*/#. For example, the focal length would be 100 mm for an *f*/4 instrument.

***Note:*** *The distance from the optic, not the focal length of the lens, determines the* f/#.

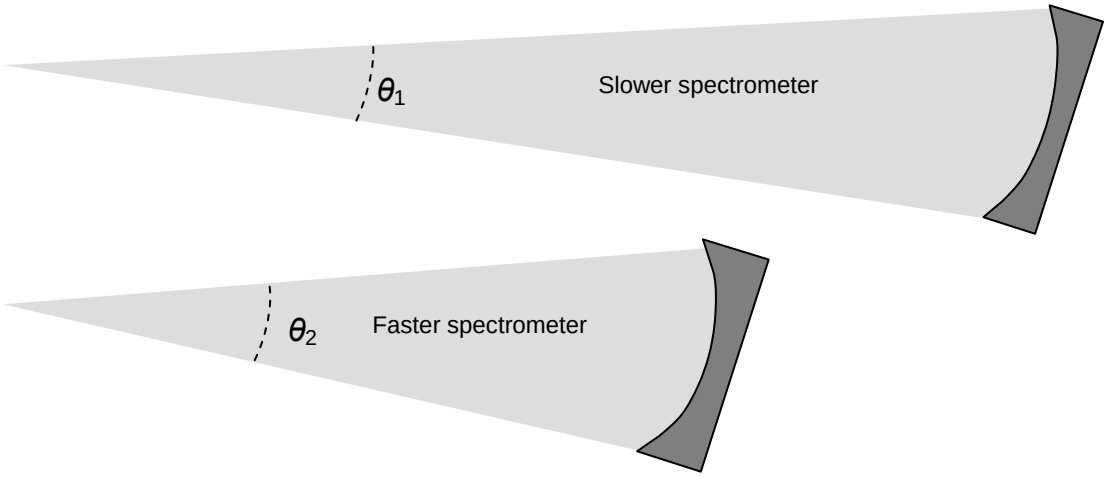



**Figure 18.** Comparison of slower and faster spectrometers from entrance slit to collimating mirror.

For a single-lens system with an *f* = +40 mm lens 80 mm away from the entrance slit, the *f*/# is 25/80 = *f*/3.2. For focused light sources, the same principles apply because it is the angle of focusing or defocusing that determines the *f*/#.



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# 4: Optional Accessories

Several options are available in addition to the basic system. The standard SampleMax includes a cuvette holder (Figure 2) for liquid samples, two optical rails of the user's choosing, and two lenses.

## Table 2: Accessories

| Part number | Description |
|---|---|
| ASC-VIS | SampleMax with glass lenses for measurements in the visible and near-IR regions of the spectrum. Includes two 40 mm focal length lenses, two sets of optical rails and cuvette holder. |
| ASC-UV | SampleMax with quartz lenses for measurements in the ultraviolet, visible, and near-IR regions of the spectrum. Includes two 40 mm focal-length lenses, two sets of optical rails, and cuvette holder. |
| ASC-SSOL | Solid-sample holder (Figure 3) |
| ASC-STUR | External turret wheel allows easy movement of the sample holder into the appropriate position for measurements |
| ASC-ORAIL | Rail mount assembly for third optical axis |
| ASC-SXZ | X-Z stage with micrometer control for the sample holder |
| ASC-VIS30 | Additional glass lens, 30 mm focal length |
| ASC-VIS40 | Additional glass lens, 40 mm focal length |
| ASC-VIS60 | Additional glass lens, 60 mm focal length |
| ASC-VIS100 | Additional glass lens, 100 mm focal length |
| ASC-UV30 | Additional quartz lens, 30 mm focal length |
| ASC-UV40 | Additional quartz lens, 40 mm focal length |
| ASC-UV60 | Additional quartz lens, 60 mm focal length |
| ASC-UV100 | Additional quartz lens, 100 mm focal length |
| J350386 | Mounting adapter for iHR, MHR, or TRIAX slits (three included) |
| J1920 | Quartz cuvette |
| J1925 | Stoppered quartz cuvette |
| J350590 | Cuvette holder |
| J36540 | Adapter for ACH-C chopper, M28 threads |
| LSH-A270 | Adapter for LSH-Series lamp-housings |
| ASC-270 | Adapter to mount to axial entrance of 270M |
| MSA-M28/1679A | Adapter to mount to slits with M28 threads |
| ASC-1451 | Adapter to mount to 1451 slits (M-Series) |
| ASC-1679 | External variable slit with height-limiter |
| 100FLT | Filter holder for 1″ diameter filter, up to 8 mm thickness |
| J1496 | Tiltable filter-holder for 1″ diameter filter (such as notch filter) |
| LPF-CG324 | 324 nm longpass filter, 1″ diameter |
| LPF-CG385 | 324 nm longpass filter, 1″ diameter |
| J50453 | 500 nm longpass filter, 1″ diameter |



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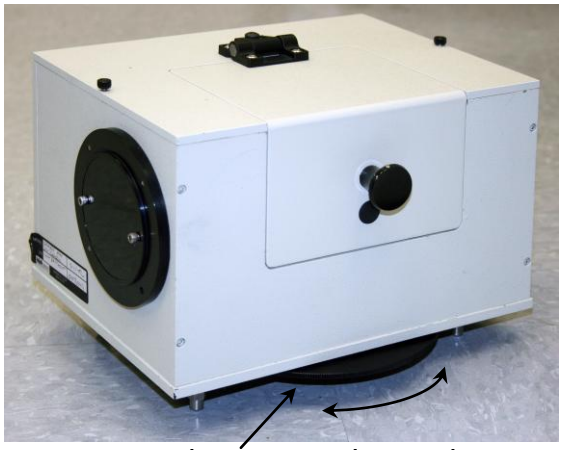
| | |
|---|---|
| J50449 | 550 nm longpass filter, 1″ diameter |
| J50450 | 630 nm longpass filter, 1″ diameter |
| J50451 | 780 nm longpass filter, 1″ diameter |
| J50454 | 830 nm longpass filter, 1″ diameter |
| LPF-D1-0-3MM | 1 µm longpass filter, 1″ diameter |
| J650531 | ZnSe IR lens, 50.8 mm focal length, 1″ diameter; requires J356544 |
| J650533 | ZnSe IR lens, 127 mm focal length, 1″ diameter, requires J356544 |
| J356544 | Kit for mounting 1″ diameter lens on rails |
| ASC-FIBKIT | SMA fiber-optic holder with *xy*-positioning mount; mounts on rail |



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# External Turret Rotator (ASC-STUR)

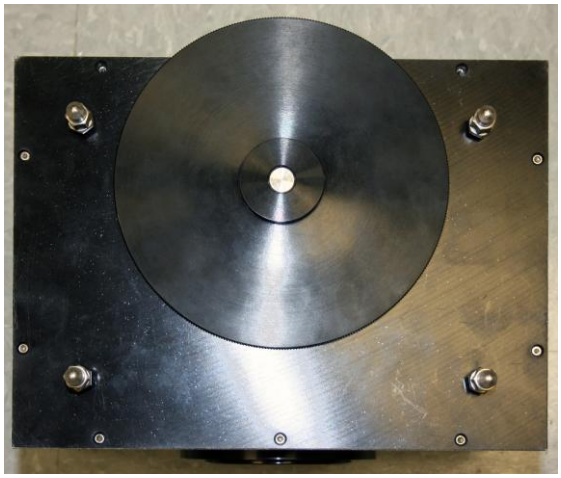
The primary advantage of the external rotator option is the ease with which you may interchange experimental configurations. Without opening the SampleMax, you can switch from reflection to absorption experiments, for example. Grooves in the rotator serve to position the sample, as a metal pin snaps into place in the groove at a number of positions corresponding to potential experimental arrangements.



Note external rotator underneath chassis.



Rotating turret inside SampleMax, with solid-sample holder atop. External rotator is visible underneath chassis.



Underside of SampleMax with external rotator

**Figure 19.** Various views of the External Turret Rotator.



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# X-Z Stage (ASC-SXZ)

Although the standard stage provides centering so that the light input is incident on the sample, an optional X-Z stage (Figure 20) offers far more precise positioning ability. A micrometer consisting of a fixed and rotating part controls each axis position. Maximum displacement in the *x*-direction is 10 mm, and 10 mm in the *z*-direction.

On the fixed part is a scale ranging from 0 to 5, with each unit representing 2 mm of motion. Tick marks represent 0.25 units, or 500 µm. The rotating part has a scale ranging from 0 to 25, with each unit ranging accounting for 0.01 units of the fixed scale, or 20 µm.

You can measure the distance moved across the axis by noting the difference in micrometer settings. For example, if you begin with the fixed scale between 2.25 and 2.50 and the rotating part showing 13 when aligned with the axis on the fixed part of the micrometer, that corresponds to a setting of 2.38. If you finish when the setting is between 4.00 and 4.25 with 22 on the rotating part, your final position is 4.22. Therefore, you have moved 1.84 units, or 3.68 mm.

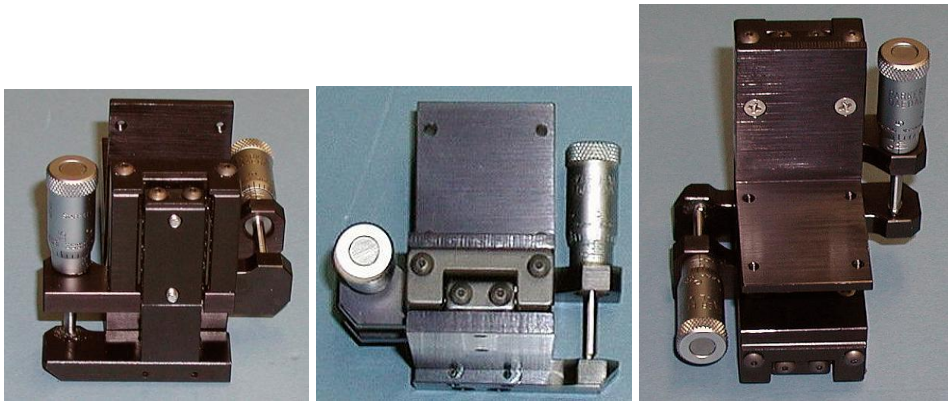
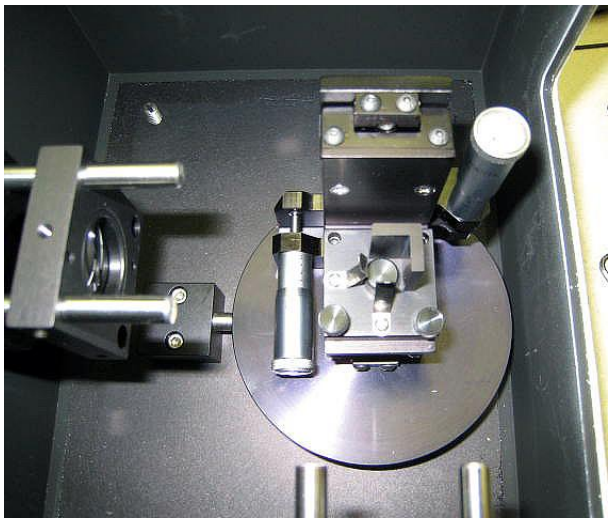


**Figure 20.** Bottom, top, and front views of the ASC-SXZ.

The X-Z stage with precision micrometers enables you to perform mapping measurements. If all other elements in the system remain fixed in position, the micrometers may be adjusted to change the position of the sample so that you can measure the variation in properties of the sample. This type of experiment is known as *mapping*, and is commonly used in photoluminescence of thin films and semiconductor wafers.



ASC-SXZ inside SampleMax, with cuvette holder on top



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# Variable Slit and Height Limiter (ASC-1679)

The ASC-1679 adapter mounts directly onto the SampleMax and places an adjustable slit flush with the port of the sample chamber. The slit may be adjusted from nearly closed to 8 mm wide with calibration marks every 100 µm. The height limiter offers three options: fully closed, 1 mm height, and 15 mm height. Consult Figure 19 to attach the ASC-1679 to the SampleMax.

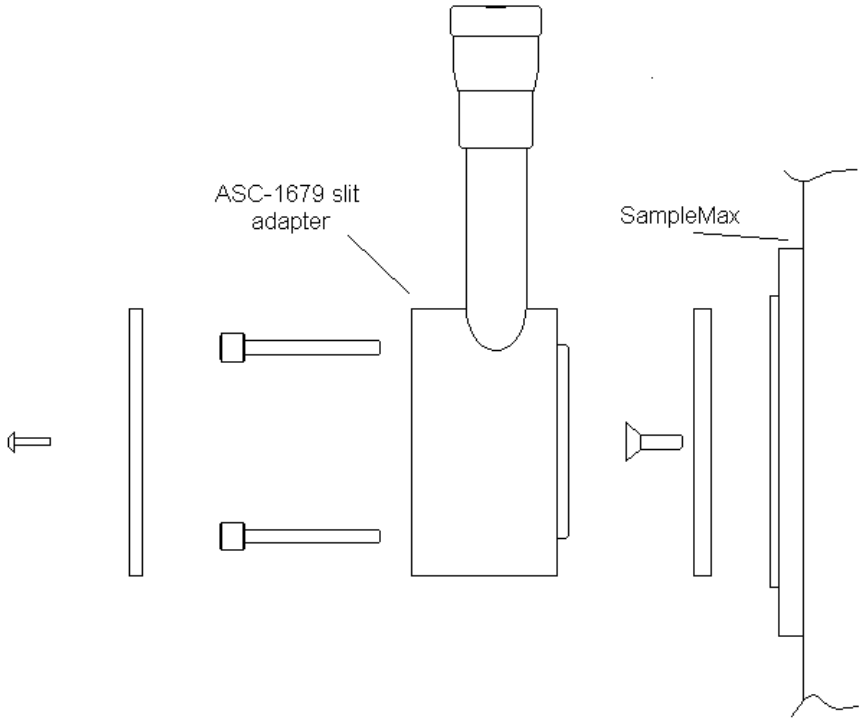



**Figure 21.** ASC-1679 slit adapter attachment diagram



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# 5: Service Information

## Service policy

If you need assistance in resolving a problem with your instrument, contact our Customer Service Department directly, or if outside the United States, through our representative or affiliate covering your location.

Often it is possible to correct, reduce, or localize the problem through discussion with our Customer Service Engineers.

All instruments are covered by warranty. The warranty statement is printed on the inside back cover of this manual. Service for out-of-warranty instruments is also available, for a fee. Contact HORIBA Instruments Incorporated or your local representative for details and cost estimates.

If your problem relates to software, please verify your computer's operation by running any diagnostic routines that were provided with it. Please refer to the software documentation for troubleshooting procedures. If you must call for Technical Support, please be ready to provide the software serial number, as well as the software version and firmware version of any controller or interface options in your system. The software version can be determined by selecting the software name at the right end of the menu bar and clicking on "About." Also knowing the memory type and allocation, and other computer hardware configuration data from the PC's CMOS Setup utility may be useful.

In the United States, customers may contact the Customer Service department directly. From other locations worldwide, contact the representative or affiliate for your location.

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If an instrument or component must be returned, the method described on the following page should be followed to expedite servicing and reduce your downtime.



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# Return authorization

All instruments and components returned to the factory must be accompanied by a Return Authorization Number issued by our Customer Service Department.

To issue a Return Authorization number, we require:

- The model and serial number of the instrument
- A list of items and/or components to be returned
- A description of the problem, including operating settings
- The instrument user's name, mailing address, telephone, and fax numbers
- The shipping address for shipment of the instrument to you after service
- Your Purchase Order number and billing information for non-warranty services
- Our original Sales Order number, if known
- Your Customer Account number, if known
- Any special instructions



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# Warranty

For any item sold by Seller to Buyer or any repair or service, Seller agrees to repair or replace, without charge to Buyer for labor or materials or workmanship of which Seller is notified in writing before the end of the applicable period set forth below, beginning from the date of shipment or completion of service or repair, whichever is applicable:

a. New equipment, product and laboratory apparatus: 1 year with the following exceptions:
   i. Computers and their peripherals
   ii. Glassware and glass products.
b. Repairs, replacements, or parts – the greater of 30 days and the remaining original warranty period for the item that was repaired or replaced.
c. Installation services – 90 days.

The above warranties do not cover components manufactured by others and which are separately warranted by the manufacturer. Seller shall cooperate with Buyer in obtaining the benefits of warranties by manufacturers of such items but assumes no obligations with respect thereto.

All defective items replaced pursuant to the above warranty become the property of Seller.

This warranty shall not apply to any components subjected to misuse due to common negligence, adverse environmental conditions, or accident, nor to any components which are not operated in accordance with the printed instructions in the operations manual. Labor, materials and expenses shall be billed to the Buyer at the rates then in effect for any repairs or replacements not covered by this warranty.

This warranty shall not apply to any HORIBA Instruments Incorporated manufactured components that have been repaired, altered or installed by anyone not authorized by HORIBA Instruments Incorporated in writing.

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The above shall constitute complete fulfillment of all liabilities of Seller, and Seller shall not be liable under any circumstances for special or consequential damages, including without limitation loss of profits or time or personal injury caused.

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# 6: Adapters

## Table 3: List of Adapters

| Part number | Description |
|---|---|
| ASC-270 | Adapter to mount to axial entrance of 270M |
| MSA-M28/1679A | Adapter to mount to slits with M28 threads |
| ASC-1451 | Adapter to mount to 1451 slits (M-Series) |
| LSH-A270 | Adapter for LSH-Series lamp housings |
| J350386 | Mounting adapter for iHR, MHR, or TRIAX slits; three are included with the SampleMax |
| J36540 | Adapter for ACH-C chopper or filter wheel |



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# 7: Index

***Key to the entries:***

Times New Roman font.....subject or keyword
Arial font.....command, menu choice, or data-entry field
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`Courier New font`.....file name or extension


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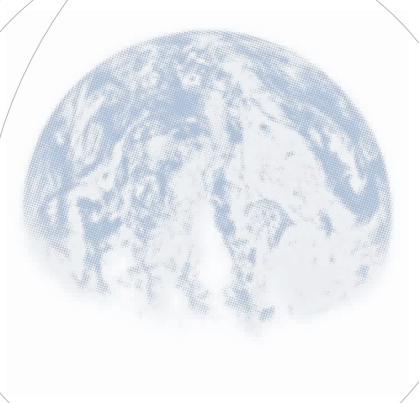
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