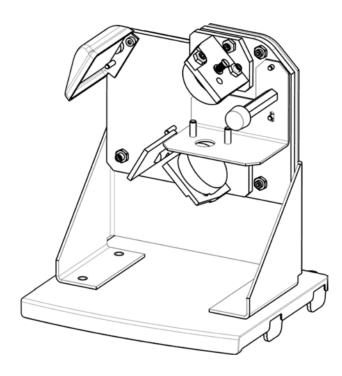
# SPECTRUM TWO BEAM CONDENSER ACCESSORY



**User's Guide** 



#### **Release History**

Part Number	Release	Publication Date
L1050227	С	September 2017

Any comments about the documentation for this product should be addressed to:

User Assistance PerkinElmer, Inc. 710 Bridgeport Avenue Shelton, CT 06484

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## Introduction

The Spectrum Two beam condenser accessory enables you to analyze smaller quantities of material than can be sampled by conventional transmittance methods, where most of the beam will miss the sample and degrade the quality of the spectrum. The beam condenser reduces the beam diameter to approximately one-fifth of its normal size, allowing you to collect good-quality spectra on microgram or sub-microgram amounts of sample.

The beam condenser is both versatile and easy to use. The accessory is pre-mounted on a base plate for easy installation in the Spectrum Two FT-IR spectrometer sample compartment. The removable sample holder collar in the accessory accepts the following:

- Aperture discs for supporting and masking small samples (included with the accessory).
- KBr micropellets for analyzing sub-microgram to microgram amounts of sample.
- Third-party etched slit apertures for fibers or other elongated samples.
- A diamond anvil cell for pressing out and measuring thin films of crushable materials.

The configuration of the optical system (Figure 1), combined with the choice of focusing mirrors, maximizes the infrared light transmission through small apertures. When the accessory is correctly aligned, this high transmission gives a signal-to-noise ratio of typically 50% of full beam performance using a 2.5 mm aperture, requiring minimal signal averaging.

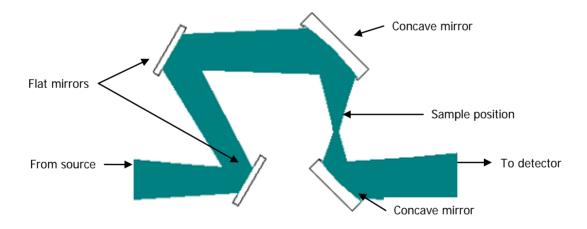
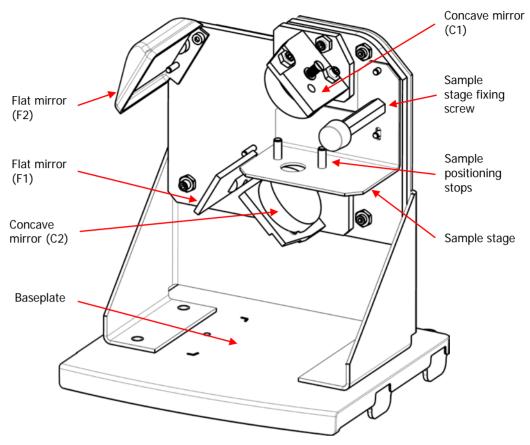


Figure 1 Optical diagram of the beam condenser accessory



The main parts of the beam condenser accessory are shown in Figure 2 below.

Figure 2 Main components of the beam condenser accessory

# Unpacking the Accessory

As soon as you receive your beam condenser accessory, make sure that it includes the parts listed below. If anything is missing or damaged, contact your local PerkinElmer office.

Part Number	Description	Quantity
L1600121	Beam condenser accessory	1
L1601764	Sample holder collar	1
L1601788	Aperture disc (0.5 mm)	1
L1601789	Aperture disc (1.0 mm)	1
L1601790	Aperture disc (1.5 mm)	1
L1601791	Aperture disc (2.0 mm)	1
L1601792	Aperture disc (2.5 mm)	1
09200328	Hexagonal key (2.5 mm)	1
L1605061	L1605061 Alignment aid	
L1050242	Spectrum Two Manuals CD	1

# Initial Installation of the Accessory

The beam condenser accessory is pre-mounted on a baseplate for quick installation into the sample compartment of the spectrometer.

To remove the current accessory and then install the beam condenser accessory:

1. Open the sample compartment cover of the Spectrum Two (if fitted), raise it to the vertical position and then lift it upwards, clear of the instrument (Figure 3).



### Figure 3 Removing the sample compartment cover

 Pull the baseplate of the current sample accessory towards you, and slide the accessory out of the sample compartment (Figure 4).
 Store it in a safe place for future use.



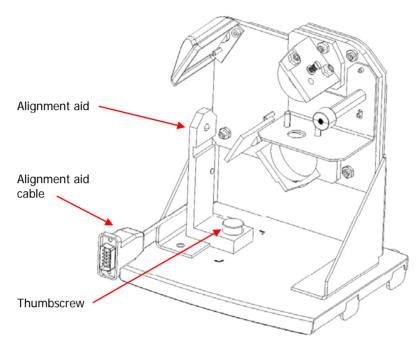
### Figure 4 Removing the sampling accessory

- Start the Spectrum software, log on and connect to the instrument. Refer to the Spectrum Two User's Guide (P/N L1050228) for more information.
- 4. Select **Monitor** from the Measurement menu in the Spectrum software and record the energy throughput of the open beam.

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- 5. Click to stop monitoring.
- 6. Install the alignment aid on to the beam condenser accessory by positioning it over the hole in the baseplate and tightening the thumbscrew firmly (see Figure 5).

Push the alignment aid against the bracket on the accessory to position it correctly before tightening the thumbscrew. Make sure the cable is not trapped behind the alignment aid.



### Figure 5 Beam condenser accessory with alignment aid in position

- 7. Slide the beam condenser accessory baseplate into the sample compartment.
- 8. Push the accessory in firmly to ensure that the connector on the rear of the baseplate connects properly with the spectrometer socket.
- Plug the alignment aid cable into the EXT DETECTOR socket on the back of the Spectrum Two spectrometer.

An LED on the alignment aid will activate.

When the accessory is correctly installed in the sample compartment, the Spectrum software detects it, and the Setup Instrument Basic (Figure 6) and Setup Instrument BeamPath (Figure 7) tabs are updated to show that the beam condenser accessory is in position.

The Accessory toolbar displays the beam condenser icon

Setup Instrument Auto-Name	Setup Instrument Data Collection	Setup Instrument BeamPath	Setup Instrument Advanced	Setup Instrument Basic
Actions Restore Defaults Load and Save	Wavenumber Ordinate Units %T	Start (cm-1)      Rei        4000      4		Scan Type Sample v ccumulations 4 Scans v
	Accessory	Condensr		

### Figure 6 Setup Instrument Basic tab with beam condenser accessory

Setup Instrument Auto-Name	Setup Instrument Data Collection	Setup Instrument BeamPath	Setup Instrument Advanced	Setup Instrument Basic
<b>—</b>		Setting	Value	
		Source	MIR (8000 - 30) cm-1	
	1	Beamsplitter	OptKBr (7800 - 400) cm-1	
		Detector	MIR TGS (15000 - 370) cm	-1
		Window	KBr	
		Optimum Scan	(7800 - 450) cm-1	
		·		
*				
		Setting	Value	1
	7 \	🏀 J-Stop Imag	8.94	
		Please remove a	ny externally fitted J-Stop card	l.
		J-Stop Wavenu	4000	
		Desiccant chan	1760	
		Instrument serv	. 320	
		Accessory	Condensr	

Figure 7 Setup Instrument BeamPath tab with condenser icon (circled)

The accessory is now installed in the sample compartment, and is ready to be aligned.

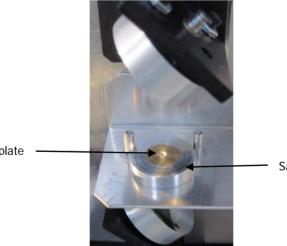
# Aligning the Beam Condenser Accessory

CAUTION	Take care not to damage or smudge the reflective aluminum coating on the mirrors of the accessory. If you do get a fingerprint on a mirror, remove it immediately using a lens tissue and a small amount of a volatile organic solvent.
ATTENTION	Veillez à ne pas endommager ou à tacher le revêtement d'aluminium réfléchissant sur les miroirs de l'accessoire. Si vous obtenez une empreinte digitale sur un miroir, retirez-le immédiatement en utilisant un tissu d'objectif et une petite quantité d'un solvant organique volatile.

Before you collect spectra using your beam condenser accessory, you should align the accessory and ensure that it is operating correctly.

- 1. Install the alignment aid and the beam condenser accessory as described in *Initial* Installation of the Accessory on page 7.
- 2. Select an aperture plate from those provided with the accessory. When aligning the accessory for the first time, we recommend that you use the largest aperture (2.5 mm diameter).
- 3. Install the selected aperture plate into the recess in the sample holder collar.
- 4. Install the sample holder collar on the sample stage, so that it sits inside the hole in the sample stage (Figure 8).

You should now see the beam from the LED very close to the hole in the aperture plate, or possibly around the edge of the hole if the beam is already approximately aligned.



Aperture plate

Sample holder collar

Figure 8 Positioning the aperture plate and sample holder collar in the accessory

5. Use the hexagonal key to adjust the socket screws in the backing plate of the upper concave mirror (designated as C1 in Figure 2) until the beam is centered on the aperture.

Only adjust the alignment screws at this stage, *not* the focusing screw which lies horizontally (Figure 9).

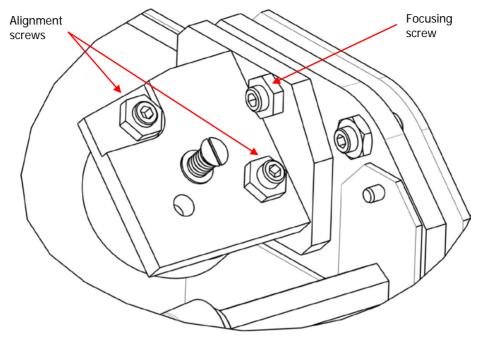


Figure 9 Adjustment screws for mirror C1

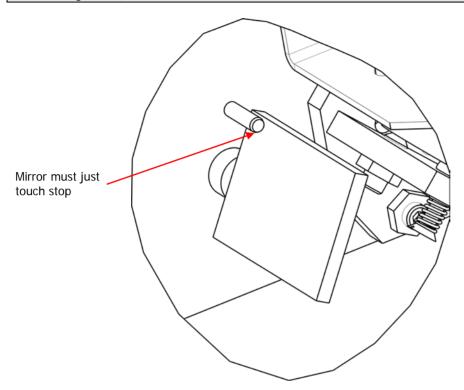
6. Check that a light beam approximately 25 mm in diameter reaches the window on the right side of the spectrometer sample compartment.To see the beam clearly, it may help to hold a small piece of paper in front of the

To see the beam clearly, it may help to hold a small piece of paper in front of the window.

7. If necessary, center the beam on the window using the corresponding alignment screws under the lower concave mirror C2.

As before, do not adjust the horizontal focusing screw.

**NOTE:** When adjusting mirror C2, ensure that you do not knock mirror F1. This mirror, which freely rotates, should just touch the stop near its base when correctly positioned (Figure 10).



#### Figure 10 Correct position of mirror F1

- 8. Remove the alignment aid from the accessory.
- 9. Select **Monitor** from the Measurement menu in the Spectrum software and record the energy throughput of the beam with the accessory installed.
- 10. Use the hexagonal key to adjust the two alignment screws on mirror C1 alternately to achieve the maximum energy throughput.

The energy value should go through a clear maximum with each adjustment. You should be able to obtain an energy value of approximately half that measured for the empty sample compartment (see *Initial Installation of the Accessory* on page 7).

If you want to further optimize the energy reading, adjust the focusing of the concave mirrors as follows:

- 1. Turn the focusing screw on mirror C1 a quarter of a turn clockwise (Figure 9).
- 2. Adjust the two alignment screws on mirror C1 to maximize the energy reading, and compare it with the value recorded in step 10 above.

3. If the energy value has increased, turn the focusing screw another quarter of a turn clockwise and repeat the optimization process until no further increase is measured.

OR

If the energy value has decreased, begin turning the focusing screw counterclockwise by quarter turns and repeat the optimization process until no further increase is measured.

4. Repeat steps 1–3 for mirror C2.

You will find that this mirror is less sensitive to adjustment.

**NOTE:** For best results, the sample compartment cover should be replaced once the alignment is complete.

### Realignment of the accessory

Once correctly aligned, the beam condenser accessory should require very little realignment in future. Small adjustments of the two alignment screws on mirror C1 may be needed to keep the beam centered on the aperture. The focus on mirror C1 may require adjusting using the focusing screw if the sample holder is changed. It is unlikely that mirror C2 will require adjustment. The alignment aid will not be needed unless the alignment is completely lost and no energy is recorded by the Spectrum software. If this occurs, check that the beam has not been blocked and that mirror F1 is in the correct position before attempting realignment.

## Using the Accessory with Spectrum Software

Once the accessory is aligned, you are ready to start collecting spectra.

The Measurement toolbar (Figure 11) displayed by default at the top of the Spectrum software workspace includes the tools you need to collect a spectrum from a sample. You can also select these commands from the Measurement menu.

Sample ID	Description	N 12			Ensure beam path is clear	
Administrator 01	Sample 001 By Ad 😮 🔲 Preview				Press [Scan] to continue	
		Scan Scanalyze	Halt	Background		$\overline{\mathbf{z}}$

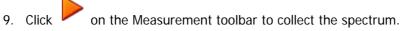
Figure 11 Spectrum Two Measurement toolbar

To perform a scan:

- Ensure that the required scan and instrument parameters have been set.
  When you first install the accessory, the software automatically sets default scan parameters to values that are appropriate for the beam condenser accessory.
   To modify any instrument parameters that are not included on the toolbars, select Instrument from the Setup menu to display the Setup Instrument tabs (Figures 6–7).
- 2. Enter a unique **Sample ID** and **Description** for the sample on the Measurement toolbar.
- 3. Position a suitable blank sample in the accessory, depending on the sample preparation method being used.

For example, use a blank KBr disc of the same thickness as the sample, or an empty diamond anvil cell. If a pure film is to be analysed, simply position the chosen aperture disc in the sample holder collar. Refer to *Preparing Samples* on page 17 for further information.

- 4. Click I on the Measurement toolbar to collect the background spectrum.
- Position the sample in the beam condenser assembly.
  Refer to *Preparing Samples* on page 17 for information on the various sampling accessories available.
- Select Monitor from the Measurement menu, and then select Sample.
  For best results, the maximum transmission in the spectrum should be approximately 90%, which indicates that the background and sample spectra will have similar baselines.
- 7. If required, adjust the focusing of the beam condenser to maximize the energy. Refer to steps 1–4 on page 12.
- 8. Click lostop monitoring.



The spectrum is added to the Samples View. The History tab (accessed by clicking on the sample in the Data Explorer) will contain information about the Accessory and Sampling Type.

The Spectrum on-screen Help describes how to format, process and report your results. To view the Help, select **Contents** from the Help menu.

**NOTE**: For best results, keep the accessory clean and avoid contaminating the surfaces of the mirrors during use. Clean the mirrors as required using a lens tissue and a small amount of a volatile organic solvent. Take care not to scratch the mirrors during cleaning.

## Testing the Beam Condenser Accessory

You can test the performance of your beam condenser accessory by obtaining the spectrum of a thin piece of transparent polymer film. Suitable samples are easy to find; examples are food wrap, adhesive tape or envelope windows.

- 1. Cut out a piece of film of sufficient size to cover the aperture in the accessory.
- Collect a background spectrum with the open aperture. Refer to *Using the Accessory with Spectrum Software* on page 14 for further information.
- 3. Place the small piece of film over the aperture.
- 4. Collect and save a sample spectrum.
- 5. Remove the beam condenser accessory from the sample compartment.
- 6. Install the slide holder accessory in the sample compartment.
- 7. Collect a new background spectrum.
- Place a larger piece of sample film over the hole in the slide holder, and hold it in place using adhesive tape if necessary.
   Ensure that the adhesive tape is not within the beampath of the spectrometer.
- 9. Collect and save a second sample spectrum.
- Compare the two sample spectra. They should be virtually identical with very similar signal-to-noise ratios.

**NOTE:** If the alignment has been carried out correctly and the results are still of poor quality, contact your PerkinElmer Service Representative for further advice.

# **Preparing Samples**

## Using aperture discs

The five aperture discs supplied with the beam condenser accessory allow you to measure the transmittance spectrum of thin solids of various diameters by resting the sample on top of the disc.

**NOTE:** When selecting aperture discs to measure samples of different diameters, remember that the amount of light transmitted through the aperture depends on the area of the hole. For example, the transmission through a 0.5 mm aperture will be 25 times less than that through a 2.5 mm aperture.

Other sampling accessories (not included with the beam condenser) are also available.

### Micro KBr discs

The beam condenser accessory is most commonly used with samples prepared as micro KBr discs. The Ultra Micro KBr Die (P/N 01860007) includes 0.5 mm and 1.5 mm sample discs. The 3 mm (P/N 01862292) and 1 mm die sets (P/N 01862295) can also be used with the Quick Press (P/N 01860436).

You will need to run a reference spectrum to which you can ratio your sample spectrum. The reference spectrum can be acquired in either of two ways:

- If the KBr used in your sample spectrum is so dry that the absorption bands from water are not a problem, use an empty sample disc when you run your reference spectrum.
- If you need to use the spectrum of a blank KBr disc as your reference, be sure that this disc is the same thickness as the sample disc. Otherwise, the sample and reference spectra will have different baselines.

The KBr sample discs fit in the sample holder collar in the same way as the aperture plates.

Refer to *Obtaining Infrared Spectra of Solids – Using the KBr Disc Technique* (P/N L1050202) on the *FT-IR Educational Resource CD* (P/N L1050246) for further information on obtaining optimum results using this sampling method.

### Diamond anvil cell

A miniature diamond anvil cell kit (P/N N9302618) is available for samples that can be crushed into a thin film, such as fibers and paint chips. The cell is mounted in the beam condenser accessory as follows:

- 1. Remove the sample holder collar from the sample stage.
- 2. Hold the anvil cell with the tension screws on top and identify the corner of the cell marked with two red spots.
- 3. Push this corner between the sample positioning stops so that both are touching the cell, and its front edge is approximately parallel to the sample plate.

The expected transmission through the cell is approximately 3–4%. Some refocusing of mirror C1 may be needed to optimize the beam energy. If no signal is measured, check that the cell is not obstructed.

### Fiber slits

If you are using the beam condenser for fiber analysis, fiber slits are available for mounting your samples.

Part Number	Description	Quantity	
L1160581	18 µm Fiber Holder	Pack of 5	
L1160582	33 µm Fiber Holder	Pack of 5	

### Third-party apertures

The two sample positioning stops on the sample stage are set apart such that a 1 inch (25 mm) diameter disc will be centered on the optical axis of the condenser when it touches both stops. Etched apertures in mounts approximately 1 inch in diameter are available from both Edmund Optics (for example, P/N 56-281 and 58-541) and Thorlabs (P/N S5R and P5S). Acceptable results can be obtained with the mount placed on the accessory's sample holder collar with the aperture side downwards, so that the aperture is as close as possible to the position of the PerkinElmer aperture discs. Some refocusing may be needed to optimize the spectrum obtained, using the focusing screws on each concave mirror. Refer to steps 1–4 on page 12 for further information.

# **Replacement Parts List**

There are no user replacement parts in the beam condenser accessory. In the event of a failure of any component, please contact your PerkinElmer Service Representative for assistance.

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