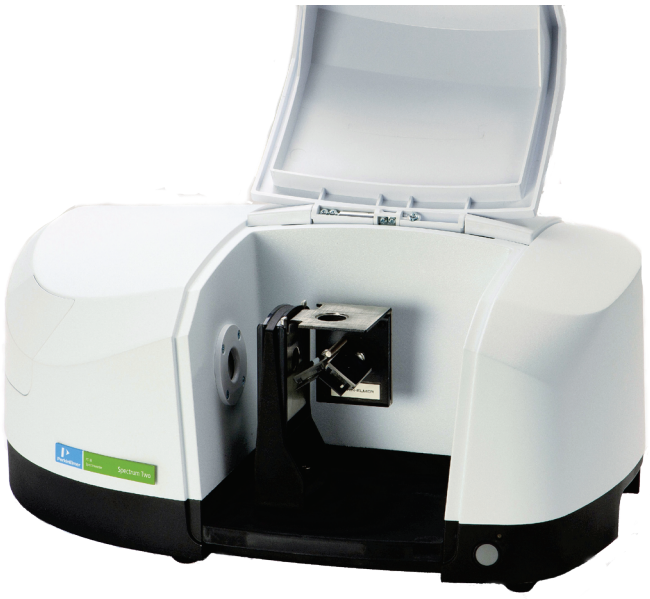
SPECTRUM TWO FIXED ANGLE SPECULAR REFLECTANCE ACCESSORY



User's Guide

PerkinElmer®

Release History

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Introduction

Specular reflectance is reflectance in one direction from a surface, as opposed to diffuse reflectance, which is reflectance in all directions. A fixed angle specular reflectance accessory is a simple device, consisting of a sample position and two mirrors. One mirror focuses the IR beam at the sample; the other collects the reflected radiation. The spectrum produced can be an absorption spectrum of a thin layer on a reflective surface.

With a specular reflectance accessory you can obtain a spectrum of any flat, reflecting surface on plate or sheet material. This spectrum will usually be ratioed to a reference spectrum, which is obtained with a flat mirror in the sample position.

You can use the Fixed Angle Specular Reflectance (FASR) Accessory with a variety of samples. Three sizes of aperture masks are included, for use with small samples or for studying a small area of a larger sample. Sample size is limited only by the size of the spectrophotometer sample compartment. A standard cell slide, which fits most spectrophotometers, mounts the accessory in the sample compartment.

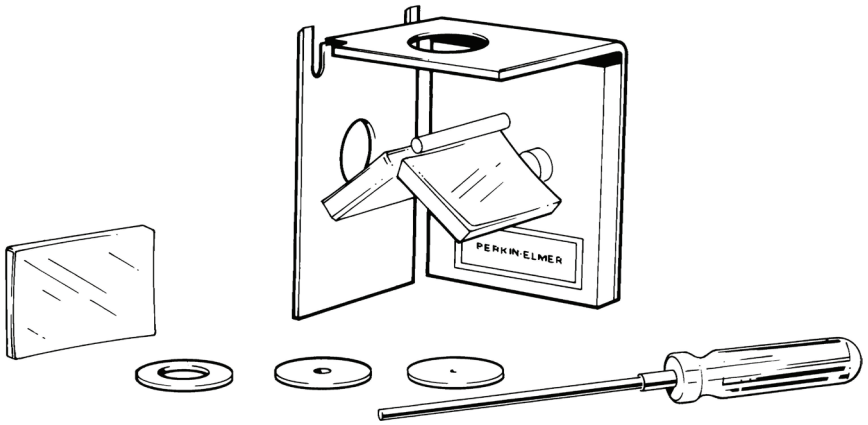

Figure 1 The Fixed Angle Specular Reflectance Accessory

Figure 2 is an optical diagram of the specular reflectance accessory. The entire optical system has 3-point (kinematic) mounting for stability and ease of alignment. There are two spherical mirrors. A bolt mounted above the mirrors blocks stray light. Radiation from the source will be incident on the sample at a mean angle of about 16° from the perpendicular.

A reference mirror is provided with the accessory, for use in the sample position during alignment, and for obtaining reference spectra.

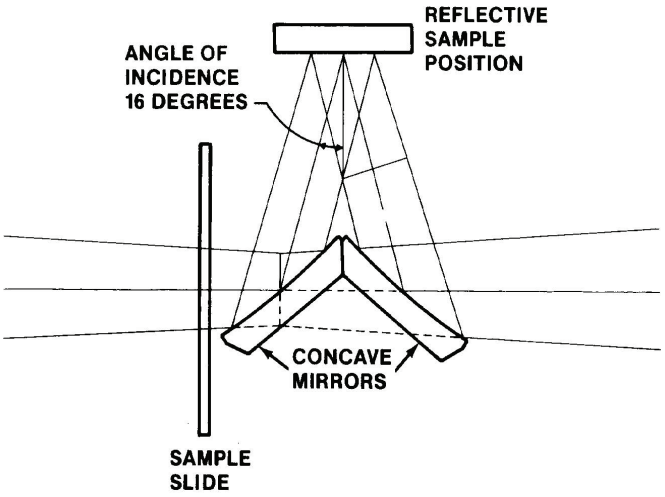

Figure 2 Optical Diagram of the Specular Reflectance Accessory

The accessory can be used to measure thin coatings on metal surfaces, such as drink cans. The spectra shown in Figure 3 are from the inside surface of a soda can.

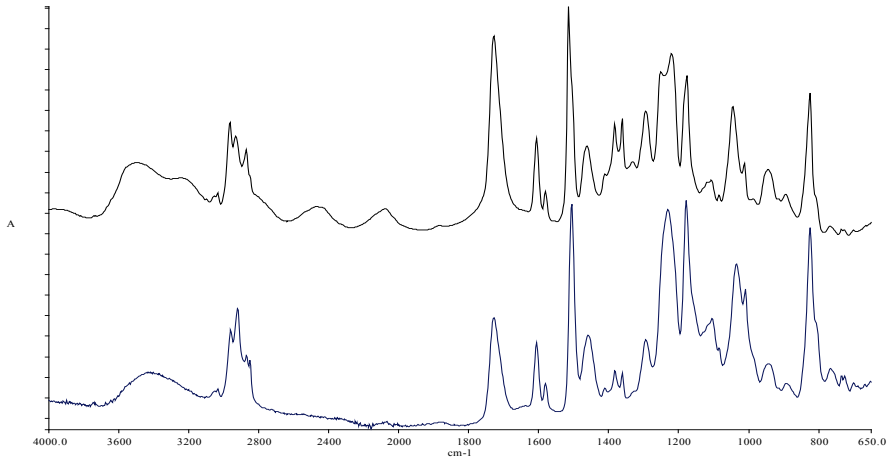

Figure 3 Spectrum of the coating inside a soda can

The upper spectrum was obtained using a specular reflection accessory, the lower spectrum by ATR. There are two significant differences. The upper spectrum shows interference fringes (channel spectra) above 2000 cm^{-1}. This is a thin film and the spectrum is essentially measuring transmission through the film, so fringes are to be expected.

The other difference is in the strong band close to 1200 cm^{-1}. In the upper spectrum this appears to have two peaks, while in the ATR spectrum it is a single band. The double peak in the reflection spectrum occurs because there is reflection from the top surface of the film as well as from the metal substrate. The reflection from the upper surface depends on the refractive index of the film, which can change significantly in the vicinity of strong bands. As the detector sees the sum of reflection from the upper surface and the metal substrate, this leads to distortion of the measured lineshape. It also means that in such reflection spectra the strong bands never reach 0%T.

Specular reflection from a smooth surface does not resemble a normal spectrum with symmetrical absorption bands. Typically, the features have a first-derivative-like shape. This is because the intensity of reflected radiation depends on the refractive index of the material. The refractive index is related to the absorption coefficient and changes in the vicinity of absorption bands. In the mid-IR region, spectra of this type are seen from crystals or non-scattering polymers, provided that they are sufficiently thick that no light returns from within the sample. They are readily seen from carbon-filled polymers, provided that the surface is smooth.

The reflection spectrum can be converted into a normal absorption spectrum by applying a Kramers–Kronig transform as illustrated Figure 4. The upper spectrum is from a block of polymethyl methacrylate and the lower is the result of a Kramers–Kronig transform.

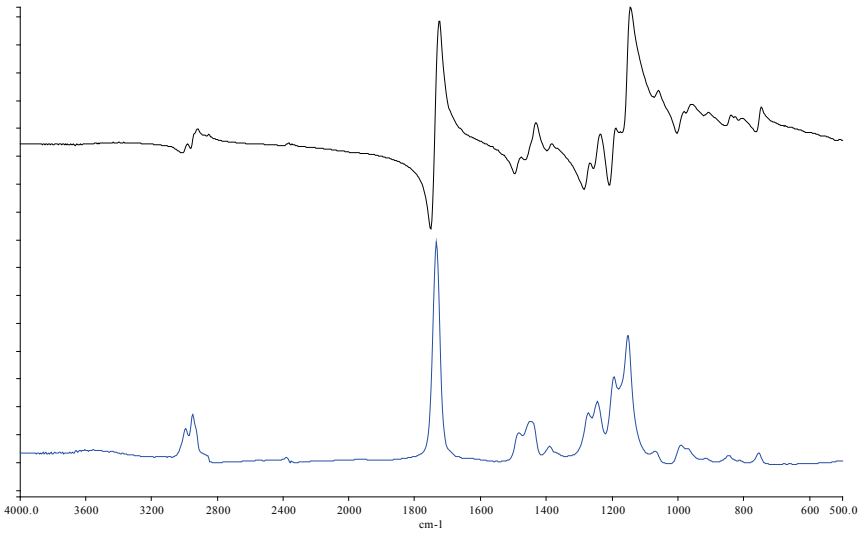

Figure 4 Reflection spectrum and Kramers–Kronig transform

Installation and Alignment

Immediately upon receiving your specular reflectance accessory, check the contents of the shipping container against the parts list below. If anything is missing or damaged, contact your PerkinElmer Customer Care Representative.

Part Number	**Description**	**Quantity**
00071006	Reference mirror	1
01860795	Fixed Angle Specular Reflectance Accessory	1
01861854	Aperture Mask, 2 mm	1
01861861	Aperture Mask, 5 mm	1
01861862	Aperture Mask, l0 mm	1
09907909	Wrench	1

Install and align the specular reflectance accessory as described in *Aligning the Accessory* on page 8.

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.*

Aligning the Accessory

1. Before installing the accessory, place it on a bench top as shown in Figure 5. Look from the right side and locate the following two vertical plates, using Figure 6 for reference:
   - The accessory support bracket is on the right.
   - The optical base plate, on the left, has three 10-32 socket set screws.

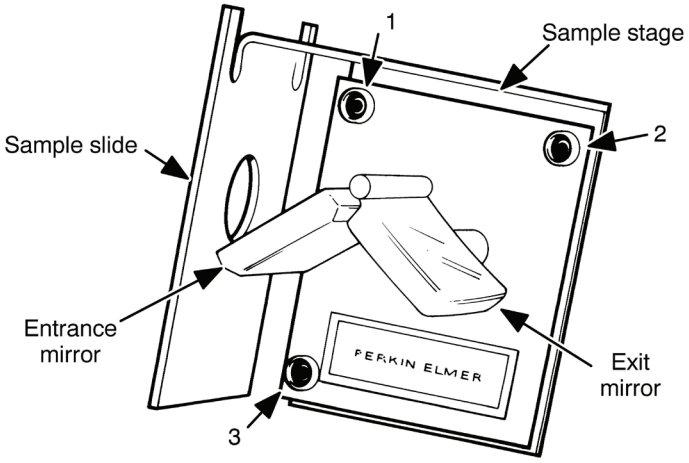

Figure 5 Parts used in alignment

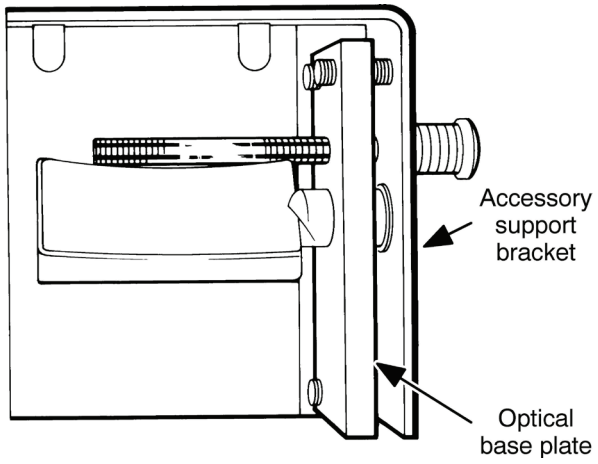

Figure 6 Side view of the FASR Accessory, showing two plates

2. Using the wrench provided, adjust the three 10-32 socket set screws in the optical base (numbered 1, 2, and 3 in Figure 5) so that the space between the optical base and the support bracket is approximately 3/16 inch (5 mm) all around.

3. Ensure that the sample slide is installed in position 2 on the baseplate (Figure 8).

4. Insert the Spectrum Two alignment tool in the first set of holes on the left of the baseplate (position 1).

5. Connect the 15-way connector on the cable of the alignment tool into the EXT DETECTOR port on the rear of the spectrometer.
   The white LED on the baseplate should be lit.

Figure 7 Alignment tool connected to EXT DETECTOR port

6. Insert the 0.5 cm J-stop card into the left slot on the Spectrum Two slide holder.

7. Place the sample slide of the accessory (Figure 5) in right slot.

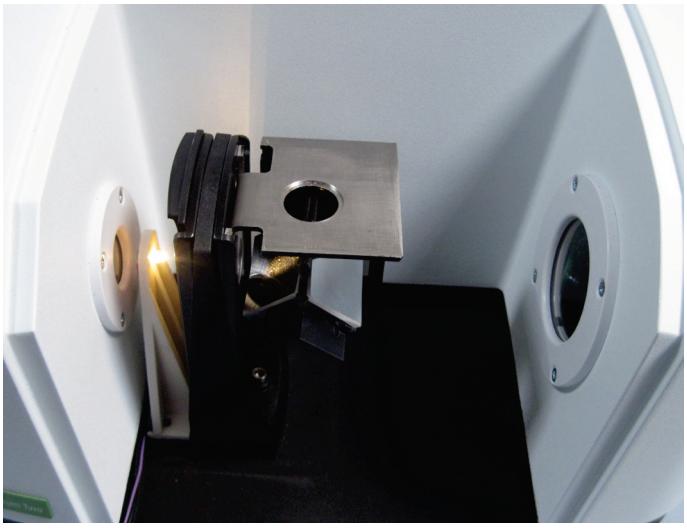

Figure 8 Accessory and alignment tool installed in the Spectrum Two sample compartment

8. Place a piece of paper in position on the sample stage.

9. While looking up from below, rotate the entrance mirror (Figure 5) so that the light from the alignment tool LED passes through the center of the sample aperture.

10. Remove the paper from the sample position.

11. Place the reference mirror in the sample position.

12. Place a piece of paper in front of the window to the right of the sample compartment.

13. Rotate the exit mirror of the accessory and adjust screws 2 and 3 in Figure 5 until the movable part of the beam is centered on the exit window.
    The exit mirror can be used to align the light vertically.
    Socket set screws 2 and 3 align the beam vertically and horizontally, respectively.

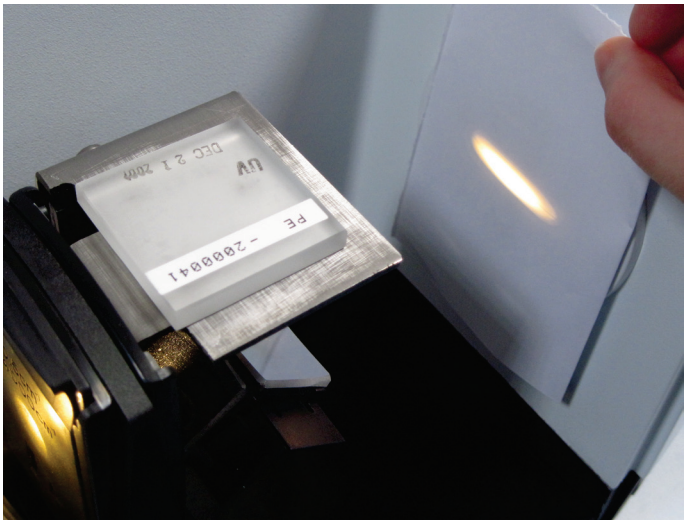

Figure 9 Beam on white paper in front of exit window

14. When the light from the LED appears to be centered in the window, remove the reference mirror, and check that the light is still centered in sample stage.

15. Disconnect the cable from the EXT DETECTOR port and remove the alignment tool from the sample compartment baseplate.

16. Remove the J-stop card from the slide holder.

17. Remove the accessory from the slide holder in the sample compartment.

18. Start Spectrum software.

19. Select **Monitor** from the Measurement menu.
    The Live tab is displayed.

20. Select **Energy**.

21. Record the maximum throughput energy and then click **Halt**.

22. Replace the accessory in the slide holder in the sample compartment.

23. Select **Monitor** from the Measurement menu and then adjust socket set screws 2 and 3 to maximize the energy.

NOTE: Do not touch the entrance or exit mirrors.

Using the Accessory with Spectrum Software

The Measurement bar (Figure 10) displayed by default at the top of the workspace includes the tools you need to collect a spectrum from a sample. You can also select these commands from the Measurement menu.

To perform a scan:

1. Enter a unique **Sample ID** and **Description** for the sample on the Measurement toolbar.

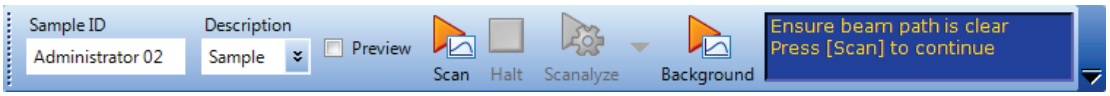

Figure 10 Spectrum Two Measurement bar

2. Place the reference mirror on top of the sample stage and then click  to collect the background spectrum.

   If you intend to use an aperture mask with your sample, place it on the sample stage first.

NOTE: If you want to collect a background that will be added to the Sample View and can be saved separately, then select Background as the **Scan type** on the Setup Instrument Basic tab.

3. Remove the reference mirror and then place your sample on the sample stage.

4. Click  to scan the sample.

   During data collection the spectrum will be displayed on the Live tab in the Viewing Area.

   The completed spectrum is displayed on the Graph tab, and added to the current Samples View in the Data Explorer. The results of the Quality Checks, selected by default on the Setup Instrument Advanced tab, are displayed in the results for the spectrum. If you have the Auto save option selected on the Setup Instrument Data Collection tab, your spectrum will be saved automatically.

If, for any reason, you want to stop scanning your sample, click  .

The Spectrum Help file describes how to format, process and report your results. To open the Help file, select **Contents** from the Help menu.

If you want to change any of the default instrument settings, select **Instrument** from the Setup menu to display the Setup Instrument tabs. The Setup Instrument Basic tab is shown in Figure 11.

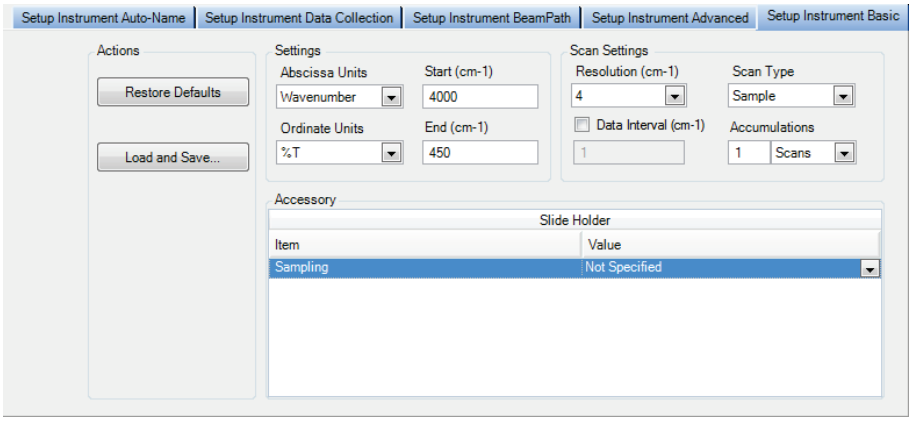

Figure 11 Setup Instrument Basic tab