

Diffuse/Specular Reflectance Manual

Introduction

The Diffuse/Specular Reflectance is an easy to use, both diffuse reflection and specular reflection accessory. Its advantages include high throughput, constant optical path with all mirrors positioned above the sampling area. This is a significant design feature which protects all the optical components of the accessory against spills and corrosion often caused by KBr/sample mixes. It also makes cleanup much easier, as accidental spills can be simply removed out of the bottom of the accessory. Sample introduction is performed using an integral precision slide.

NOTE: This accessory has two functions. One is testing the Diffuse Reflection surface; the other is testing the Specular Reflection surface

Part I

As the diffuse reflection accessory

Installation

Place the accessory in the sample compartment of the spectrometer and secure it to the base plate with a captive screw.

The accessory has been aligned and tested to ensure that it performs to specifications and no additional alignment is necessary. In order to optimize the signal throughput in your instrument only small adjustments will be required.

The alignment procedure is as follows:

1. Loose the screws marked by red circle, and, open the lid of the accessory (Fig1).
2. Place the alignment mirror in one of the sample positions and move the mirror into the appropriate position in the beam (Fig2&3).
3. In the TSTB mode (Fig4), check the signal throughput of the spectrometer with the accessory in place.
4. Gently vacillate the mirror on top of the accessory to the back and forth to get the higher infrared signal.(Open the top cover of the accessory, refer to Fig5 .)
5. Repeat step 3 until there is no further increase in infrared signal.



Fig 1



Fig 2&3

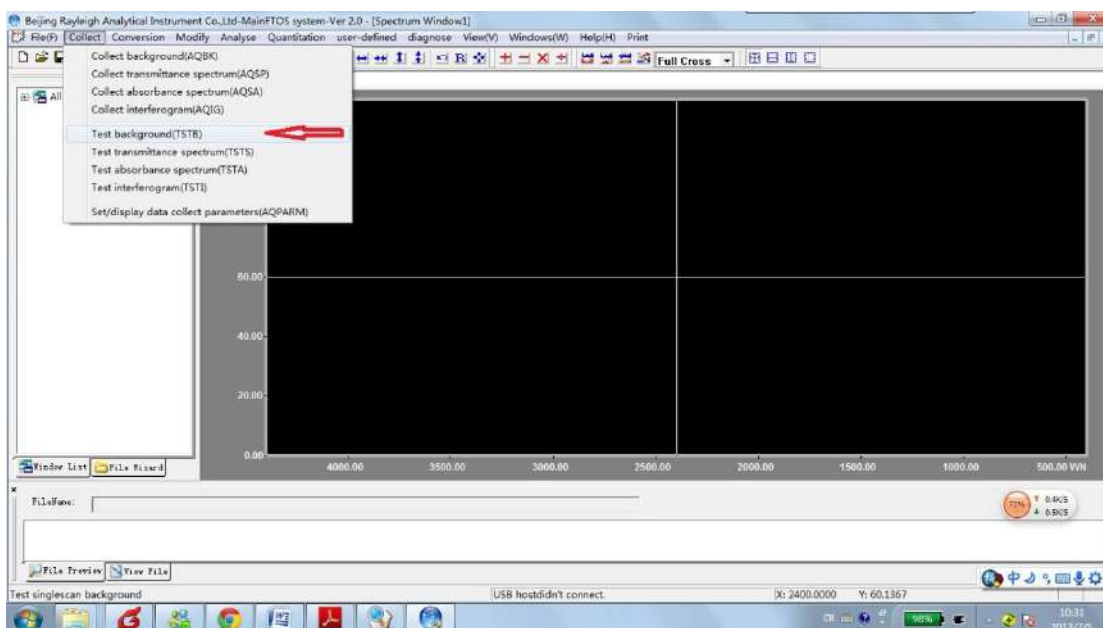


Fig4

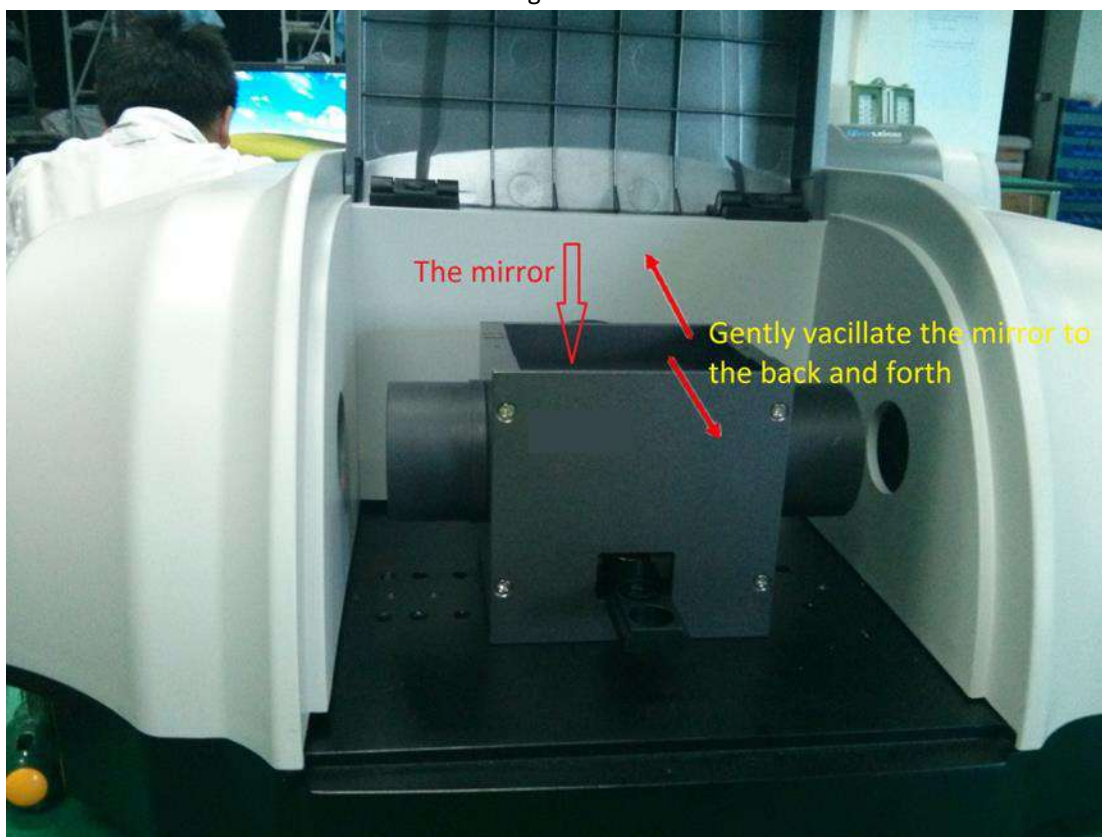


Fig5

Performance Verification

With the accessory removed from the sample compartment, collect a background spectrum. Place the accessory in the sample compartment. Place the alignment mirror in the beam position. With the accessory aligned to the previous procedure, collect a transmission spectrum using the same collection parameters as used to collect the background spectrum. The minimum transmission of the accessory should be 30% at wavenumber 2400cm^{-1} .

Preparing the Sample

The sample to be analyzed is often diluted in a transmitting matrix when making mid-infrared measurements. Place the sample in the mortar and grind finely using the pestle. Add KBr powder to the sample. With the pestle, mix the sample with the KBr powder so that the sample particles are small and evenly dispersed. The sample should be diluted to 1% to 5%. The optimum amount of dilution will depend on the sample to be analyzed but the percent transmission of the strongest band in the resulting spectrum should ideally be in the range from 10% to 50%. If possible start with a 5% dilution and if the resulting bands are too intense then re-dilute the sample.

CAUTION: KBr readily absorbs moisture. To reduce the effect of moisture in the sample spectrum the following guidelines should be used. Always keep the cap on the KBr bottle when not in use. If possible store the KBr in a dry place. Fill the reference cup (neat KBr) and the sample cup (sample diluted in KBr) at the same time. Any water that has been absorbed in the reference and sample will tend to ratio out in the final spectrum.

Loading the Sample

With the sample prepared in the mortar, place the sample cup in the sample preparation base. Pour the sample from the mortar into the sample holder. The cup may now be filled using the spatula and razor blade. With practice, a perfect repeatable sample surface may be prepared.

When the sample cup is filled, remove the base and holder and place the sample cup into the sample cup slide (Fig6&7).



Fig6

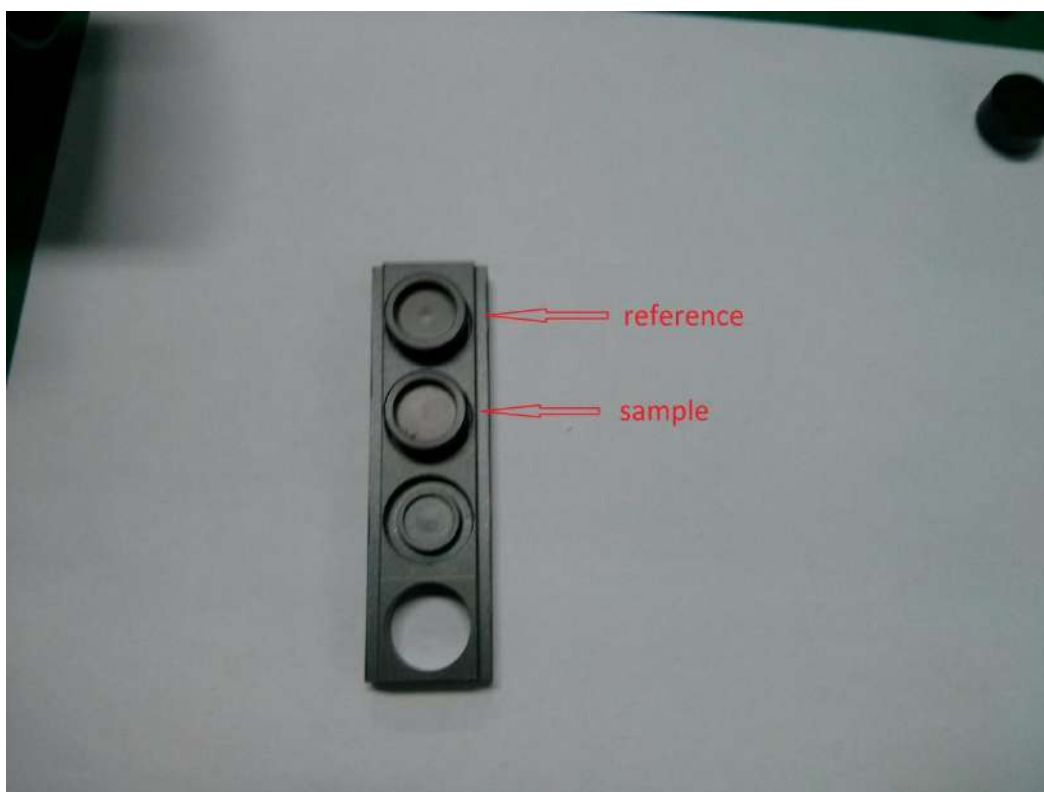


Fig7

Samples may also be analyzed without dilution. For samples that are not powders, the sample may be abraded with a piece of silicon carbide. For this technique a background spectrum is taken of the clean silicon carbide paper prior to abrading the sample.

Using the Accessory

With the two sample cups filled and inserted into the sample cup slide, place the sample cup slide into the accessory. By sliding the sample cup slide all the way in, the rear position is brought into the beam. By sliding the sample cup slide all the way out until it strikes the dowel pin-stop, the front position of the sample cup slide is brought into the beam.

NOTE: Both sample positions must use the same sized cups. There is a height difference between the two types of cups and when changing from one size of cup to another, a focus adjustment of the micrometer will be required.

Slide the sample cup slide to bring the neat KBr sample into the beam. Adjust the mirror (the same procedure with the alignment) to maximize the signal throughput through the accessory. Note that this will not be the same position as the alignment mirror, since the IR beam penetrates into the sample a short distance. Once the signal has been maximized, collect a background spectrum of the neat KBr sample. Move the sample cup slide to the other position and collect a spectrum of the sample. The ratio of these two spectra will produce a spectrum of the sample.

NOTE: After adjust the light path by neat KBr, you could fix the mirror by the screw (Fig8).

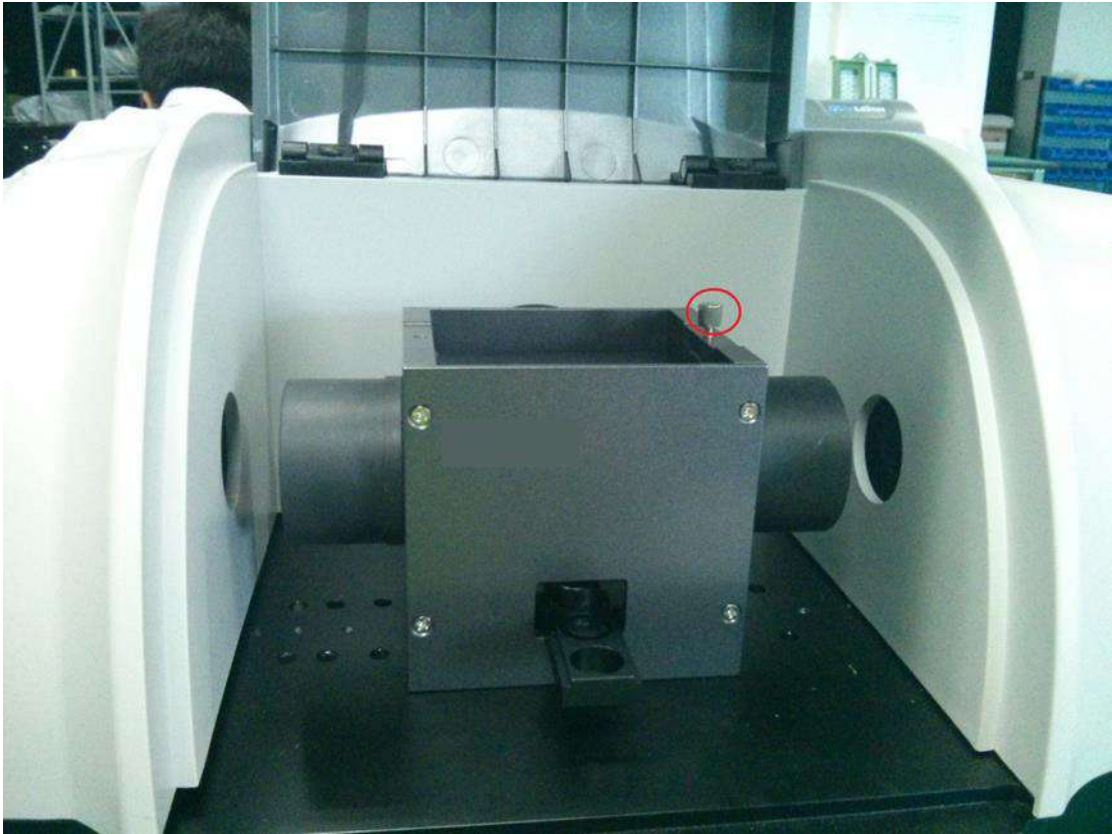


Fig8

Part II

As the Specular Reflection Accessory

Installation

Place the accessory in the sample compartment of the spectrometer and secure it to the base plate with a captive screw.

Open the lid and front cover by loose the screws. (Fig9)

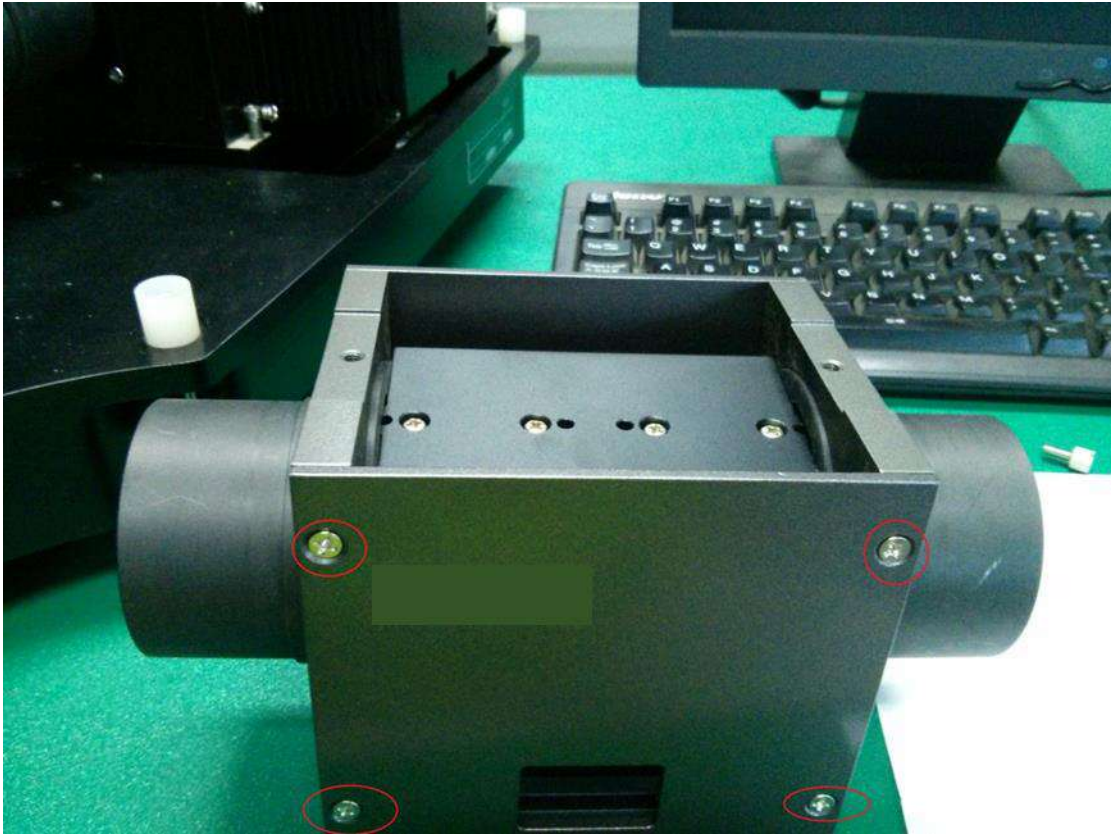


Fig 9

Remove the sampler cup slide holder (Fig10&11&12)

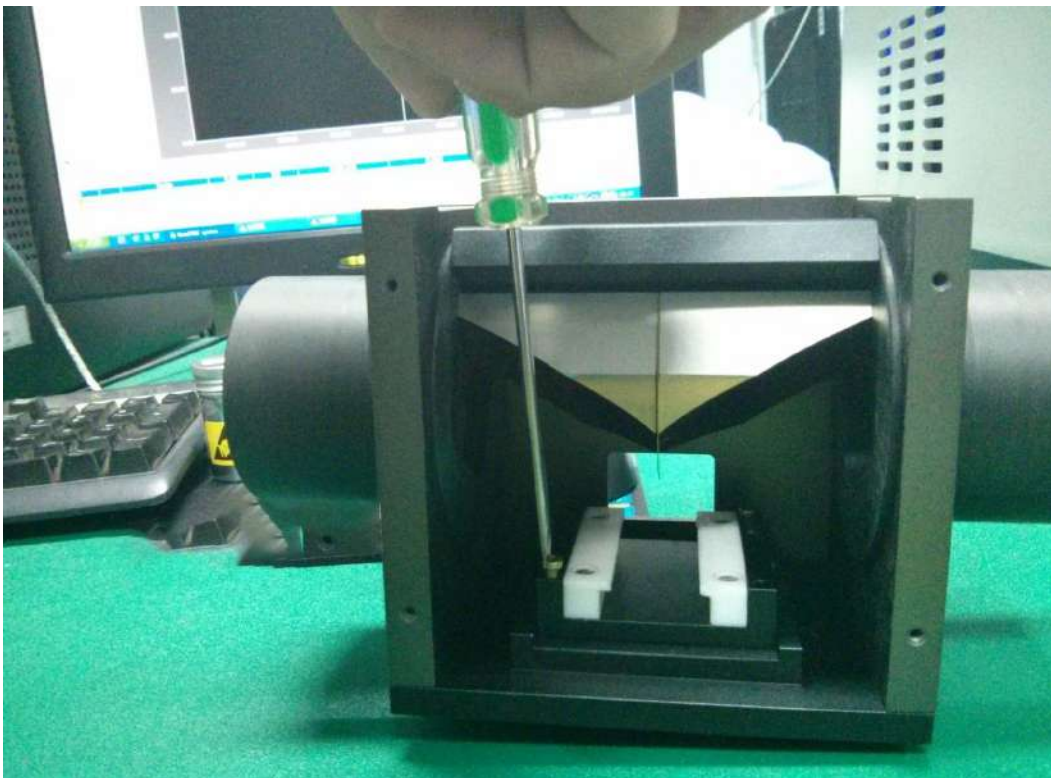


Fig10



Fig 11

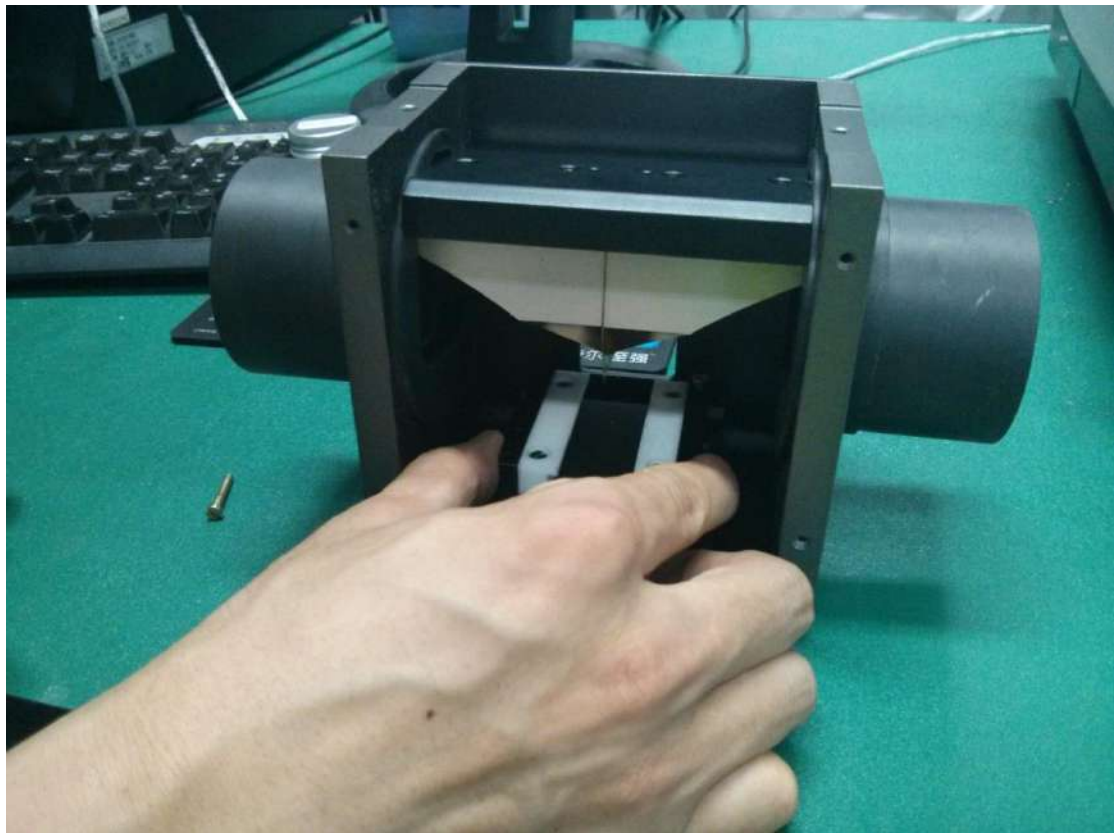


Fig12

Rotate the mirror from up to down, and put the sampler holder on the frame of the accessory (Fig13).

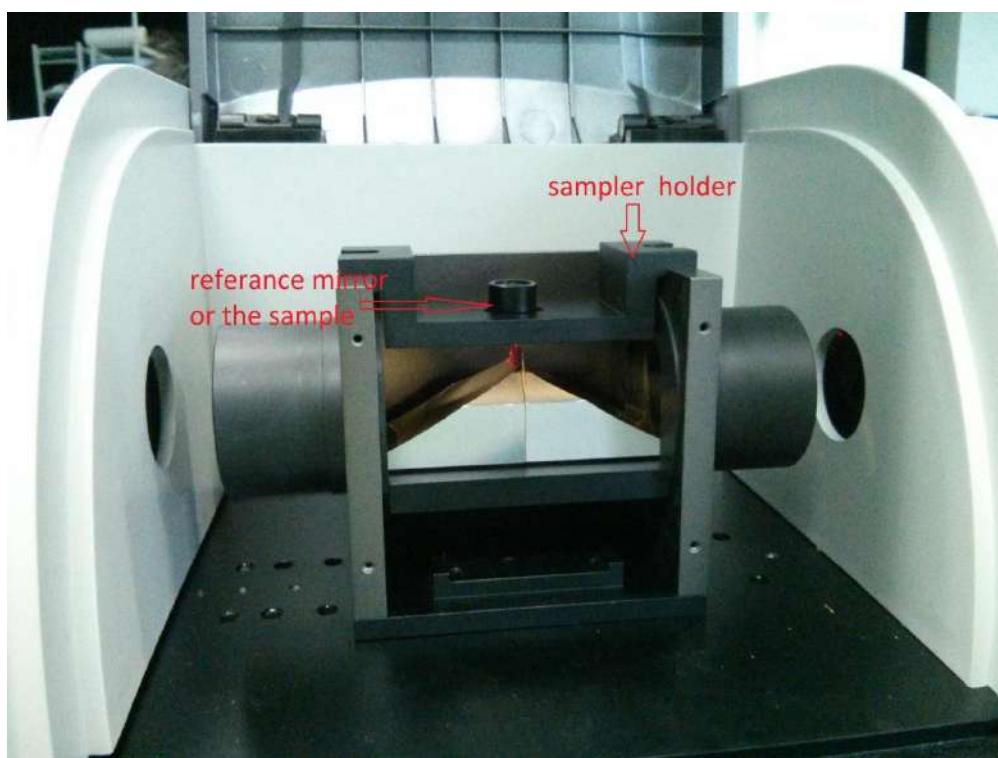


Fig13

The alignment procedure is the same with previous mentioned.

Performance Verification

With the accessory removed from the sample compartment, collect a background spectrum. Place the accessory in the sample compartment. Place the alignment mirror in the beam position. With the accessory aligned to the previous procedure, collect a transmission spectrum using the same collection parameters as used to collect the background spectrum. The minimum transmission of the accessory should be 30% at wavenumber 2400cm^{-1} .

Preparing the Sample

Cut the sampler to a suitable size, and find the flat surface.

Loading the Sample

Put the sample on the sample holder directly

Using the Accessory

After the alignment, collect background (AQBK) putting the mirror into the light path (Fig 13), and then, collect transmittance spectrum (AQSP) putting the sample into the light path (Fig 13).