The Selector™ Diffuse Reflectance Accessory

User Manual



2I-19900 Issue 6

The Selector™ Diffuse Reflectance Accessory User Manual

The Selector™ Diffuse Reflectance Accessory P/N GS19900

USER MANUAL- CONTENTS

1. Introduction	3
2. UNPACKING AND CHECKLIST	
3. SAFETY IN USE OF THE SELECTOR™ ACCESSORY	6
4. OPTICAL LAYOUT AND INSTALLATION OF THE SELECTOR™	7
L TO R SELECTOR™ ACCESSORY OPTICAL LAYOUT	7
R TO L SELECTOR™ ACCESSORY OPTICAL LAYOUT	10
INSTALLATION OF THE SELECTOR™ ACCESSORY	13
5. ALIGNMENT OF THE SELECTOR™ ACCESSORY	16
SAMPLE INTRODUCTION INTO THE SAMPLING CUP	16
FINE ALIGNMENT - FINAL OUTPUT MIRROR ADJUSTMENT	18
OUTPUT ELLIPSOID MIRROR E2 ADJUSTMENT	21
MICROMETER SCREW ADJUSTMENT	22
6. SAMPLE ANALYSIS	
SAMPLE CUP OPTIONS	
11MM DIAMETER STANDARD SAMPLE CUP	
4MM DIAMETER MICRO SAMPLE CUP	
DIABRAZE PAD AND DIABRAZE PAD MOUNT HOLDER	
TILTED (TOTAL REFLECTANCE) SAMPLE CUP	
PROCEDURE FOR SAMPLE MEASUREMENTS	
USING A SAMPLE CUP OPTION	
USING A DIABRAZE PAD OPTION	
7. CARE AND MAINTENANCE	
8. LEGEND FOR THE SELECTOR™ ACCESSORY	
9. SPARE PARTS FOR THE SELECTOR™	
10 SELECTOR™ BASEPLATE INSTALLATION GUIDE	34

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1. Introduction

Thank you for purchasing a Specac product.

Infrared radiant light when reflected from a solid surface can be either diffuse, specular or both (total reflectance) in nature.

Diffuse reflectance is based upon the collection of radiation that has been **diffusely scattered** from the sample. From illumination by infrared radiation of a solid sample surface that can deemed non linear or irregular (e.g. powdered, granular or fabric/textile type samples), although there will be components of some specularly reflected light from the surface, the majority of light to collect for measurement will be diffusely scattered.

The Selector™ accessory P/N GS19000 uses an optimized off-axis optics configuration which selectively collects and maximizes the diffusely reflected components of light, whilst minimizing the specular component. The off-axis design also allows for the expansion of use of the Selector optics with alternative specialized baseplates such as the Environmental Chamber (P/N GS19930).

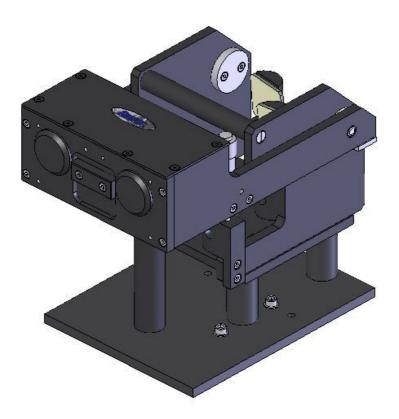
The Selector™ accessory is provided with a range of sampling cups for appropriate sample study which include a standard 11mm diameter cup, a micro 4mm diameter cup and a tilted cup. The tilted cup can be used for the collection of **total reflectance** (diffuse and specular components of light) spectra, if desired. In addition, there are also abrasive sample mounts for use with 12mm diabraze pads. The diabraze material is rubbed against the surface of an intractable solid to deposit a small amount of the solid on the abrasive pad surface. The diabraze pad, on the sample mount, is then placed into the sampling position post of the Selector™ accessory.

The design of Selector™ accessory for the arrangement and infrared light beam passage sequence for its optical mirror components means that it is a **beam direction dependant accessory** for correct installation into a spectrometer system. A left to right or right to left beam direction (source to detector) as it passes through the

User Manual

spectrometer sample compartment means a Selector™ accessory will **only** fit on to its appropriate beam direction dependant baseplate. The Selector™ accessory can be installed into different spectrometer systems by change of an appropriate baseplate, but the spectrometer systems themselves **must** have the same common left to right or right to left beam direction.

If you are in doubt as to whether your particular build of Selector™ accessory may be suitable for use and installation into a specific spectrometer system, please contact Specac for advice.



Selector™ Accessory (in Right to Left Beam Build Configuration)

2. Unpacking and Checklist

On receipt of your Selector[™] accessory please check that the following have been supplied: (The Selector[™] accessory will be supplied in its own carry case.)

- 1 Selector™ optical unit assembly in left to right or right to left beam direction configuration.
- 1 Selector™ baseplate fitted with 4 support posts in left to right or right to left beam direction configuration (for your make and model of spectrometer)
- 1 Sample holder post (already fitted to the Selector[™] baseplate on some versions – simply screws into position).
- 1 Micro sample cup (4mm dia x 2.5mm deep).
- 2 Standard sample cups (11mm dia x 2.5mm deep).
- 1 Tilted cup. (For total reflectance measurement.)
- 2 Abrasive sampler (diabraze pad) mounts.
- 1 Packet of self-adhesive diabraze pads (20pads 12mm dia).
- 1 Extended 'T' bar Allen key.
- 1 Allen Key 1.5mm A/F
- 1 Allen Key 2.0mm A/F
- 1 "Tommy Bar" (metal rod) for mirror adjustment
- Baseplate fixing screws and washers (where necessary).

Carefully remove the Selector™ optical unit assembly, its baseplate and associated components from the carry case in preparation for installation into the spectrometer.

3. Safety in Use of the Selector Accessory

There are no specific precautions to be taken when the optical unit assembly of the Selector™ accessory is being used on its own standard Selector™ baseplate and sampling cups or diabraze sample pads, other than those associated safety procedures you may need to adopt for a specific sample type. (e.g. if the sample is toxic or hazardous, etc), as the sample for analysis is exposed to the local environment.

Warning!



If you are to use the optical unit assembly of the Selector™ accessory on the baseplate of the complimentary Environmental Chamber accessory (P/N GS19930), along with any considerations that may be required for safe handling of the sample itself, the recommended operating safety procedures found from the Environmental Chamber instruction manual, must be followed.

With the combination of Selector™ optical unit affixed to the Environmental Chamber accessory, an appropriate sample can be measured for diffusely reflected scatter of light at elevated temperatures (up to 800°C) and pressures (up to 500psi). The sampling post position of a standard Selector™ baseplate assembly that accepts the individual sampling cups and diabraze sample pads of the standard Selector™ accessory is effectively replaced by an environmentally controllable chamber, whereby the sample for analysis is placed into an enclosed heatable cup and covered by a pressure certified window cap assembly. As standard the window material for the pressure certified window cap assembly is zinc selenide (ZnSe).

Further information for the Environmental Chamber accessory is found within its own instruction manual.

4. Optical Layout/Installation of the Selector™

The Selector™ accessory is a beam direction dependent accessory whereby infrared light radiation passes from the source to the detector of a spectrometer through the sample compartment area from a left to right (**L** to **R**), or right to left (**R** to **L**) orientation, as viewed from the front or above the spectrometer.

L to R Selector™ Accessory Optical Layout

Fig 1 shows an L to R beam Selector™ optical unit (1) when fitted to its corresponding L to R Selector™ baseplate assembly (2) and in the **Operating Position**. In this case for Fig 1, the combination of the parts as shown forms the Selector™ accessory that can be used in a Perkin Elmer Spectrum One spectrometer.

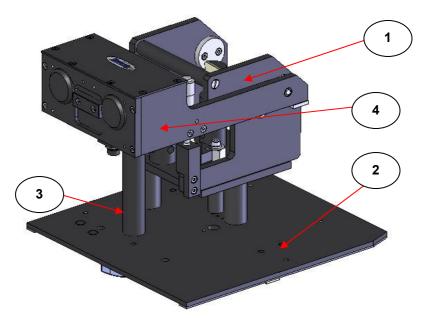


Fig 1. L to R Beam Selector™ Accessory Fitted to an appropriate L to R Beam Baseplate (e.g. for Perkin Elmer Spectrum One)

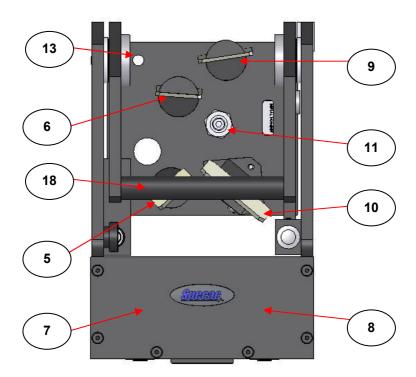


Fig 2. Top View of L to R Beam Selector™ Optical Unit

Fig 2 shows a top view of an **L** to **R** SelectorTM optical unit alone and Fig 3 shows the same **L** to **R** SelectorTM optical unit but from the underside. From the Figs 2 and 3, the light beam sequence for passage of light through the SelectorTM optical unit for an **L** to **R** direction is as follows:-

The light beam from the source is directed to a fixed angled mirror (5) on its mount post and onto another fixed angle mirror (6) on its mount post. The light beam is then projected to an **input** ellipsoid mirror E1 (7) which then focuses the light onto the surface area of a sample held in a cup that is positioned on top of the sample post (3) – see Fig 1. (In Fig 2 the E1 mirror (7) has been indicated, but it is positioned underneath the top cover of the ellipsoid mirrors arm assembly (4) – see Fig 1.) It is easier to see E1 (7) from the underside view of Fig 3.

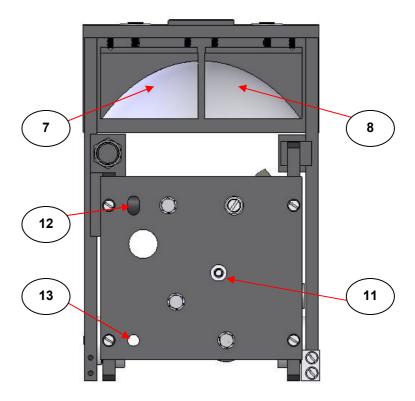


Fig 3. Underside View of L to R Selector™ Optical Unit

At the sample surface (e.g. a powder sample contained in a sample cup) the incoming light from E1 (7) will be scattered and it is the diffusely reflected component of this scattered light that is collected by the output ellipsoid mirror E2 (8).

Note: The "off axis" design for both E1 (7) and E2 (8) mirrors being forward and on the same side of the sampling surface area means that when a flat, horizontal sample surface cup is used, then only diffusely reflected light will be collected by the E2 (8) mirror. Any specularly reflected component of incident light is lost to the system.

After the scattered light has been collected by the E2 (8) mirror, the light is focused towards a fixed mirror (9) on its sample mount which then deflects the light to a **movable** (for rotation and tilt) mirror (10) on its mount. The light beam can then be deflected precisely to the detector of the system.

In summary, the beam sequence for an L to R Selector™ as indicated for the mirrors from Figs 2 and 3 is:-

Source
$$-(5) - (6) - (7) -$$
Sample $-(8) - (9) - (10) -$ Detector.

R to L Selector™ Accessory Optical Layout

Fig 4 shows an **R** to **L** beam Selector[™] optical unit (1) when fitted to its corresponding **R** to **L** Selector[™] baseplate assembly (2) and in the **Operating Position**. In this case for Fig 4, the combination of the parts as shown forms the Selector[™] accessory that can be used in a variety of Nicolet Spectrometers (Nexus, Avatar, iS10, etc).

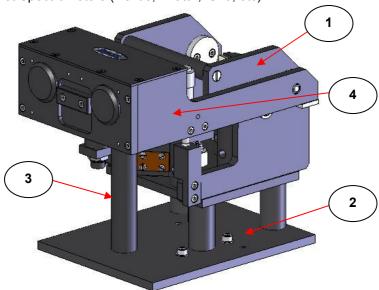


Fig 4. R to L Beam Selector™ Accessory Fitted to an Appropriate R to L Beam Baseplate (e.g. for Nicolet iS10)

Accordingly, similar to an L to R Selector[™], Fig 5 shows a top view of an **R to L** Selector[™] optical unit alone and Fig 6 shows the same **R to L** Selector[™] optical unit but from the underside.

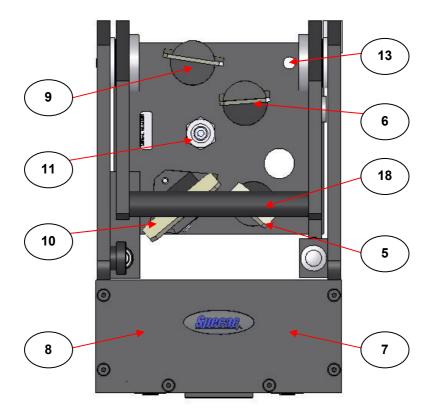


Fig 5. Top View of R to L Beam Selector™ Optical Unit

From the Figs 5 and 6, the light beam sequence for passage of light through the Selector™ optical unit for an **R to L** direction is as follows:-

The light beam from the source is directed to a fixed angled mirror (5) on its mount post and onto another fixed angle mirror (6) on its mount post. The light beam is then projected to an **input** ellipsoid mirror E1 (7) which then focuses the light onto the surface area of a sample held

User Manual

in a cup that is positioned on top of the sample post (3) – see Fig 4. (In Fig 5 the E1 mirror (7) has been indicated, but it is positioned underneath the top cover of the ellipsoid mirrors arm assembly (4) – see Fig 4.) It is easier to see E1 (7) from the underside view of Fig 6.

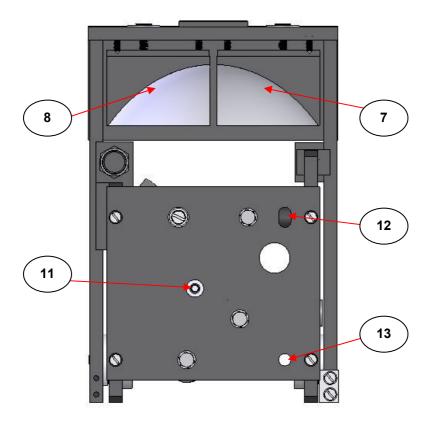


Fig 6. Underside View of R to L Selector™ Optical Unit

At the sample surface (e.g. a powder sample contained in a sample cup) the incoming light from E1 (7) will be scattered and it is the diffusely reflected component of this scattered light that is collected by the output ellipsoid mirror E2 (8).

Note: The "off axis" design for both E1 (7) and E2 (8) mirrors being forward and on the same side of the sampling surface area means that when a flat, horizontal sample surface cup is used, then only diffusely reflected light will be collected by the E2 (8) mirror. Any specularly reflected component of incident light is lost to the system.

After the scattered light has been collected by the E2 (8) mirror, the light is focused towards a fixed mirror (9) on its sample mount which then deflects the light to a **movable** (for rotation and tilt) mirror (10) on its mount. The light beam can then be deflected precisely to the detector of the system.

In summary, the beam sequence for an **R** to **L** Selector[™] as indicated for the mirrors from Figs 5 and 6 is:-

Source
$$-(5) - (6) - (7) - Sample - (8) - (9) - (10) - Detector.$$

Installation of the Selector™ Accessory

The Selector™ optical unit (1) is mounted onto a baseplate (2) to provide high stability when installed into the spectrometer. Initially, the spectrometers own sample mount and/or sample compartment (if this is part of the spectrometer's design) will have to be removed before the Selector™ accessory can be installed. The L to R or R to L Selector™ optical unit (1) is supplied with an appropriate baseplate (2) and the necessary fixing screws specifically for the spectrometer into which it is to be installed and used.

Some spectrometer systems (such as Mattson, Bruker and Nicolet 500/700 Series instruments) require the baseplate (2) to be installed into the sample compartment before the Selector[™] optical unit (1) is attached to the baseplate (2). For other spectrometers, the Selector[™] optical unit (1) can be mounted on the baseplate (2) prior to installation in the sample compartment.

For installation of the Selector[™] baseplate (2) itself into a specific spectrometer (with or without the Selector[™] optical unit (1) fitted), please see the Installation Guide in Section 10) of this manual.

Attachment of the Selector™ Optical Unit (1) to the Baseplate (2)

It can be seen by comparison of the build of Selector[™] accessory that an **R to L** beam direction version is a reversed "mirror" image of an **L to R** beam direction version for positioning of the mirror components. On the Selector[™] optical unit base (for both **L to R** and **R to L** versions) there is a central fixing M4 x 25mm captive screw (11). There is also a slot hole (12) and circular hole (13) on the Selector[™] optical unit base. (See Figs 3 and 6.) From the combination of fixing screw (11) and location holes (12) and (13), an **L to R** or **R to L** Selector[™] optical unit is fitted to its appropriate baseplate. Similar to the mirrors, the fixing hole positions on the Selector[™] optical unit base and central fixing screw for attachment to the specific **L to R** or **R to L** baseplate are also in a reversed mirror image configuration.

For attachment of the Selector[™] optical unit (1) to its baseplate (2) carefully lift the Selector[™] optical unit by the lifting handle (18) - see Figs 2 and 5 - and position over the baseplate (2) – see Figs 7 and 8.

Note: It is best to have the Selector™ ellipsoid mirrors arm assembly (4 in the down/closed (Operating Position) such that you can see the locating holes (12 and 13) and fixing screw (11) of the Selector™ optical unit, to match them up to the baseplate posts.

Align the slot hole (12) with the front support post (15) stud and the circular hole (13) with the rear support post (16) stud of the baseplate (2) and push the optical unit (1) onto the baseplate such that the optical unit is also supported on the third post (17) of the baseplate.

Note: The locating holes and post studs will be on the left hand side of the optical unit base for an L to R beam direction accessory and on the right side of the optical unit base for an R to L beam direction accessory. You cannot fit an L to R beam direction Selector™ optical unit to an R to L beam direction baseplate (or vice versa). The sample post (3) should be to the front of the baseplate near to and underneath the ellipsoid mirrors (7, 8).

Using the extended 'T' bar Allen key supplied, carefully screw the captive fixing screw (11) into the middle pillar (14) on the baseplate.

Gently tighten to secure the Selector[™] optical unit (1) to the baseplate (2), but do not overtighten the fixing screw (11). The optical unit base underside should not actually touch the top of the middle pillar (14).

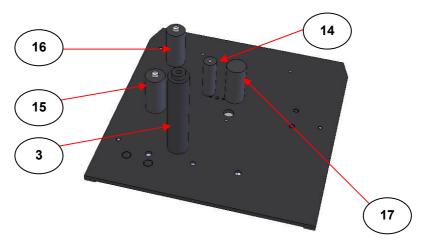


Fig 7. L to R Selector™ Baseplate (Perkin Elmer Spectrum One)

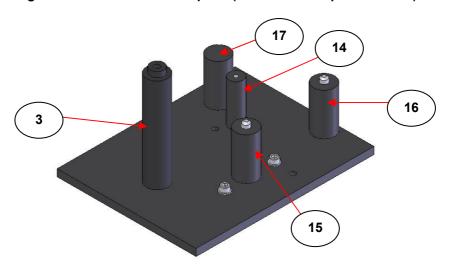


Fig 8. R to L Selector™ Baseplate (Nicolet Avatar, Nexus, iS10)

5. Alignment of the Selector™ Accessory

The Selector™ diffuse reflectance accessory has been pre-aligned before leaving Specac and will only require minor adjustment to some of the parts to maximize the light beam throughput when the accessory is installed in your spectrometer.

Note: Usually, only the output ellipsoidal mirror E2 (8) and final output mirror (10) that are adjustable may need to be altered for their settings to obtain an optimum light throughput on your spectrometer. The mirrors (8) and (10) will be on the left side of the optical unit (1) looking from above for a spectrometer with an R to L beam direction and on the right side for a spectrometer with an L to R beam direction.

Specifically, if you have a Selector™ accessory for use in a Perkin Elmer Spectrum 2000 (GX) spectrometer, the initial input (**5**) and final output (**10**) mirrors on the optical unit (**1**) are **both** adjustable for rotation and tilt. This is because the Spectrum 2000 (GX) spectrometer can be used with an **L** to **R** or **R** to **L** beam direction depending on the position of the sampling compartment and detector system to the light source" unit. Although the input (**5**) and output (**10**) mirror assemblies look similar, there is a difference in the fixed angled face of the mirrors on their adjustable mounts. Hence, you will have been provided with a specific L to R or R to L beam direction Selector™ optical unit (**1**) that will only affix to the appropriate L to R or R to L baseplate (**2**) for your configuration of Perkin Elmer Spectrum 2000 (GX) spectrometer.

Sample Introduction into the Sampling Cup

Having installed the Selector™ optical unit (1) into the spectrometer via its appropriate baseplate (2), before fine alignment and operation of the Selector™ accessory can be carried out, a representative test sample **must** be placed into position. One of the choice of sampling cups, or a diabraze pad on its support holder that have been provided with the Selector™ accessory can be used for positioning on the top of the sample post (3). (Please refer to Section 6 of this manual to explain about the sampling cups and diabraze pad sampling options.)

Ground spectroscopic grade KBr or KCl powder is recommended for use as a reference material for alignment and background spectra. The powder can be placed into the 11mm diameter sample cup (19) that is mounted on the top of the sample holder post (3). (See Fig 9.)

Tip: It is best to fill the sample cup away from the optical unit (1) and spectrometer in case of accidental sample spillage.

Note: For the purposes of explanation for the alignment procedure an R to L Selector™ accessory is used in the following diagrams. If you have an L to R Selector™ accessory the same steps for alignment are followed but the mirrors (8) and (10) for any adjustments are reversed for their positions as shown on the L to R built Selector™ accessory.

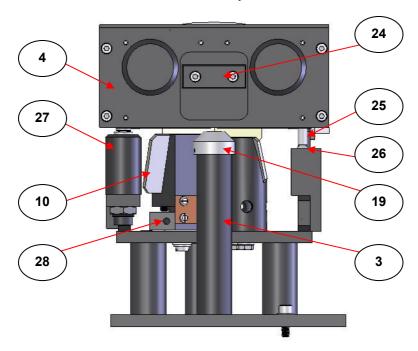


Fig 9. Front View of R to L Selector™ Accessory in Operating Position (Ellipsoid Arm Assembly Down/Closed)

User Manual

Taking the standard 11mm diameter sample cup, fill it with finely ground KBr or KCl powder. When filled, carefully flatten the top surface of the powder with a piece of glass or polished metal, so that is level with the top edge of the sample cup (19).

Raise the ellipsoid mirrors arm assembly (4) by the recessed finger handle (24) on the Selector[™] optical unit (1) to its up/open **Sample Loading Position**. When the ellipsoid mirror arm assembly (4) has been raised upright, it will remain in position. (See Fig 10.)

Place the KBr or KCl filled sample cup (19) carefully onto the top of the sample holder post (3). Try to avoid any spillage of the flattened sample powder when fitting the cup (19) to the post (3).

Lower the ellipsoid mirrors arm assembly (4) back to its down/closed **Operating Position** so that the height adjustment micrometer screw (25) rests on the steel ball stop (26). (See Fig 9.) A shock absorber (27) is fitted to ensure that the ellipsoid mirrors arm assembly (4) comes to rest gently on the steel ball (26) without damaging the micrometer (25).

With the ellipsoid mirrors arm assembly (4) closed the beam sequence through the Selector™ optical unit (1) should be completed and at this stage some energy throughput from diffusely collected light off of the sample surface should be observed by the spectrometers own detection system.

Fine Alignment - Final Output Mirror (10) Adjustment

When the sample cup (19) with sample is in position and there is some energy throughput being measured by the spectrometers detection system, then fine alignment of the optics that are adjustable can be carried out to "peak up" the throughput signal.

Firstly, the final output mirror (**10**) should be adjusted to try and improve the throughput signal. The "Tommy bar" supplied enables rotational adjustment of the final output mirror (**10**). Insert the "Tommy bar" from the front of the Selector™ accessory into the hole in the base (**28**) of the final output mirror mount (**10**). (See Fig 9.) Carefully move the "Tommy bar" horizontally in a left or right direction to improve the throughput energy signal value. If the movement in one direction

decreases the signal strength, then carefully move the "Tommy bar" in the opposite direction until the optimum signal throughput strength is obtained from this mirror adjustment. (The rotational movement is quite fine for a setting between an optimum value and loss of the signal.)

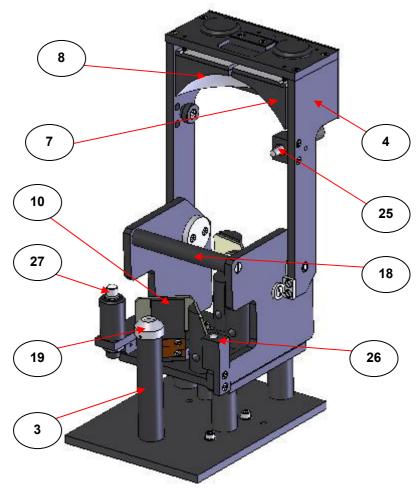


Fig 10. R to L Selector™ Accessory in Sample Load Position (Ellipsoid Mirrors Arm Assembly Up/Open)

User Manual

When mirror (10) has been adjusted for **rotation** to give an optimum peak value, then the **tilt** adjustment of this mirror can be made to try and optimise the light throughput value further. (See Fig 11.- diagram shows a close up of the mirror parts with the ellipsoid mirrors arm assembly (4) in the up/open **Sample Loading Position** for clarity.)

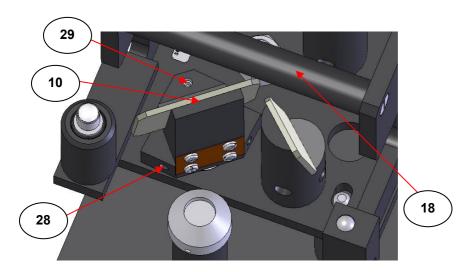


Fig 11. Final Output Mirror (10) for Rotation and Tilt Adjustment

Using the long 2mm Allen key supplied, insert it into the M4 x 10mm tilt adjustment grub screw (29), accessed vertically from the top of the Selector™ optical unit (1) to pass by the lifting carry handle (18). (The ellipsoid mirrors arm assembly (4) will be in the down/closed **Sampling Position**.) Rotate the Allen key clockwise or anticlockwise to turn the grubscrew (29) to obtain an optimum value for the throughput signal.

Note: In adjustment for the tilt angle of the final output mirror (**10**) take care not to block the path of the infrared beam with your fingers when turning the Allen key.

Output Ellipsoid Mirror E2 (8) Adjustment

When the final output mirror (10) has been adjusted for both rotation and tilt to obtain an optimum throughput, the signal throughput may be improved further by adjustment of the output ellipsoid mirror E2 (8).

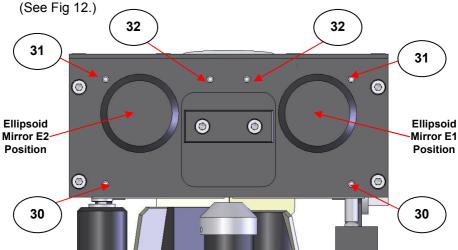


Fig 12. Ellipsoid Mirrors Angle Adjustment Grub Screws

Both the E1 and E2 (7 and 8) mirrors are adjustable for their angular position for projection to and collection of light from the sample surface by rotational adjustment of three M3 X 10mm grub screws. As shown from Fig 12, these are:

Ellipsoid mirror lower diagonal adjustment grub screw (**30**). Ellipsoid corner adjustment grub screw (**31**). Ellipsoid mirror upper diagonal adjustment grub screw (**32**).

Using the 1.5mm Allen key supplied, adjust the output ellipsoid mirror E2 (8) by insertion into the diagonal grub screws (30 and 32), one at a time, and rotate clockwise or anticlockwise to peak up the throughput energy. Ensure that you reach an optimum throughput value with adjustment of one of the diagonal grub screws (30 or 32) before adjustment of the other.

Note: Usually it will not be necessary to make any adjustment to the corner grub screw (31) to obtain an optimum throughput for the E2 (8) mirror angle position.

Micrometer Screw (25) Adjustment

After adjustment of the final output mirror (10) and the output ellipsoid mirror E2 (8), it may be possible to peak up the throughput energy to a maximum value by adjusting the height of the ellipsoid mirrors (7 and 8) for their focal point from the sample surface in a sample cup (19). (See Fig 13.)

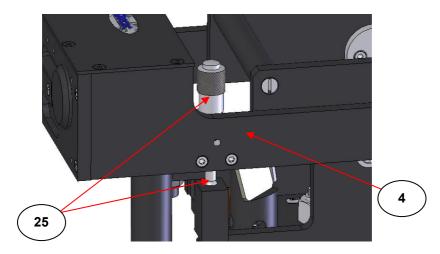


Fig 13. Micrometer Screw for Ellipsoid Mirrors Focus Point

Clockwise rotation of the micrometer screw (25) will raise the ellipsoid mirror arm assembly (4) increasing the distance of the E1 (7) and E2 (8) mirrors from the sample, whilst anticlockwise rotation will lower the ellipsoid mirrors arm assembly (4) and bring them closer to the sample. There is a finite distance whereby continued anticlockwise rotation of the micrometer screw (25) no longer lowers the E1 (7) and E2 (8) mirrors. Downward movement of the ellipsoid mirrors arm assembly (4) is stopped by the shock absorber (27).

After following the sequence of adjustment of final output mirror (10), then ellipsoid E2 mirror (8) and finally ellipsoid focus by adjustment of the micrometer screw (25), an optimum throughput value of light can be obtained.

Note: A typical energy throughput level with use of a KBr powder reference sample for an optimally aligned Selector™ accessory is expected at about a 5% value against an open (unobstructed) light beam source.

If you are struggling to achieve this typical level of light throughput from adjustment of the optical components for the fine alignment procedure (a figure less than 3% throughput is only achievable), it is possible that the **initial input ellipsoid E1 mirror** (7) is not correctly adjusted to send the light source centrally to the sample surface. Hence, adjustment of the E1 mirror (7) may be required by repositioning of its angular setting using the grub screws (30, 31 and 32) to improve the overall signal throughput.

If a lower than expected light throughput is being achieved **after following the fine alignment procedure**, first turn the lower and upper diagonal grub screws ((**30** and **32** - that affect the E1 mirror (**7**)) one at a time to see if the overall throughput of light is improved. (The corner grub screw (**31**) may not need to be adjusted to affect a change in the light throughput, but it can be rotated to see if there is any improvement.) Make any necessary adjustments from these grub screws until a throughput value maximum has been achieved.

If the input ellipsoid mirror (7) has to be adjusted to try and improve the light throughput value, then the procedure for fine alignment starting with the final output mirror (10), then ellipsoid E2 (8) and final possible refocusing of the ellipsoids via the micrometer screw (25) will need to be repeated. Readjustment of these output optical component settings also helps to further improve the throughput value obtainable.

Overall, optimum alignment of the Selector™ accessory is a "balancing effect" of the mirror positions and their settings. If one side of the beam sequence (input of light) is affected, then the output side will need to be corrected accordingly.

6. Sample Analysis

Solid powder or irregular surfaced type samples are analyzed within the Selector™ diffuse reflectance accessory in the following way, either by the sampling cups provided or by the "diabraze" abrasive sample pads fitted to their respective pad holders.

Sample Cup Options

The Selector™ accessory is provided as standard with the following sampling cup options.

11mm Diameter Standard Sample Cup (P/N GS19916)

There are two 11mm diameter x 2.5mm deep standard sample cups (19) provided. One can be used to fill with a reference sample powder for frequent background reference spectral collections (if needed) and the other can be used for solid sample materials (powders etc) to analyse against the background reference material. Interchange between the two sample cups is quick and easy. The underside of the cup (19) simply fits over the top of the baseplate (2) sampling post (3). (See Figs 9, 10.) and can be affixed securely to the sampling post (3) if required by tightening of the small locking grub screw at the cups base.

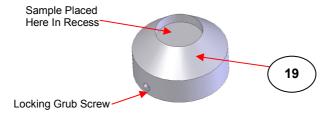


Fig 14. 11mm Diameter Standard Sample Cup

The sample cup (**19**) (see Fig 14.) is filled with a powdered sample in the 11mm diameter x 2.5mm deep recess at the top of the cup. Sufficient sample is normally required to fill the recess completely and the sample surface is usually made level to coincide with the outer

level edge of the recess to the cup. (A blade or smooth edged piece of plastic or glass is run over the surface to make level.) Because the sample cup (19) is easily detachable from the sampling post (3), filling the cup with a sample and the surface preparation can be done preferentially and more controllably, remotely in a safe area away from the spectrometer to minimize any risk of spillage of the sample and contamination of the spectrometer sample compartment area.

4mm Diameter Micro Sample Cup (P/N GS19915)

There is one 4mm diameter x 2.5mm deep micro sample cup (20) provided. (See Fig 15.)

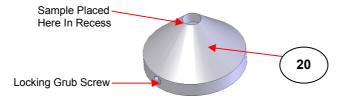


Fig 15. 4mm Diameter Micro Sample Cup

The micro sample cup (**20**) is very similar in operation and usage to the standard sample cup (**19**), but is more suited for the handling of smaller amounts of sample to fill the 4mm diameter and 2.5mm deep recess at the top of the cup.

The Diabraze Pad and Diabraze Pad Mount Holder (P/N's GS19919 and GS19918 Respectively)

As an alternative to use of the sample cups (19) and (20) for powder samples, the Selector™ accessory is provided with some abrasive sampling pads, called diabraze pads (21), and their respective mount holders (22) which allows for the collection of diffusely reflected light from "difficult" samples that have been transferred to the diabraze pad surface.

Note: Twenty diabraze pads (**21**) and two pad mount holders (**22**) are provided with the Selector[™] accessory from new.

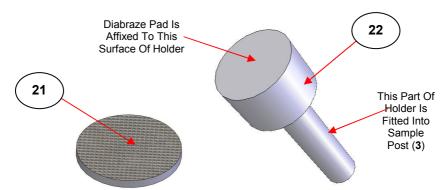


Fig 16. Diabraze Pad and Diabraze Pad Mount Holder

The diabraze pad (21) is 12mm in diameter and consists of small diamond particles embedded in an adhesive backed cloth material as the actual sampling surface. The diabraze pad is used on its own mount holder (22) to be fitted to the sample post (3) of the Selector baseplate (2). For fitting of a diabraze pad (21) to its holder (22), the protective paper is peeled away from the adhesive surface of the diabraze pad (21) and the diabraze pad is then stuck carefully and centrally to the top surface of the mount pad holder (22) (See Fig 16.)

This construction of parts (21 to 22) can now act as a "reference" sample pad to be placed into the sampling post (3) to obtain a background spectrum from a clean diabraze pad surface from the Selector™ optical unit (1). Once this background spectrum has been collected, you can choose to leave this reference diabraze pad sample assembly as is (for reference background purposes) and make another diabraze pad and holder assembly with the second holder (22) supplied for sampling, or use the abrasive surface of the diabraze pad (21) affixed to its holder (22) by rubbing it against a sample of interest (e.g. hard intractable surface such as stone or plastic etc.) The sample of interest will be transferred to the surface of the diabraze pad (21) and this pad (21)/holder (22) assembly can then be placed into the sample post (3) to collect a diffuse reflectance spectrum of the sample material, compared to the background reference already taken.

Note: This is a once only use of the diabraze pad (21) for sampling.

Tilted (Total Reflectance) Sample Cup (GS19917)

There is one Tilted Cup (23) provided with the Selector™ accessory. (See Fig 17.)

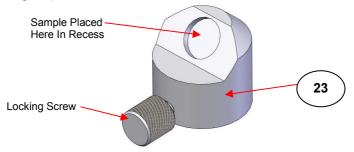


Fig 17. Tilted (Total Reflectance) Sample Cup

The tilted cup (23) allows for the collection of "total reflectance" which is a combination of both diffusely and specularly reflected components of light. The tilted cup (23) is also placed over the sampling post (3) of the Selector™ baseplate (2) for a sample to be measured for total reflectance but the locking screw part of the cup (23) must be facing forward and to the front of the baseplate (2) before it is screwed to secure the cup (23) to the post (3). (See Fig 18.)

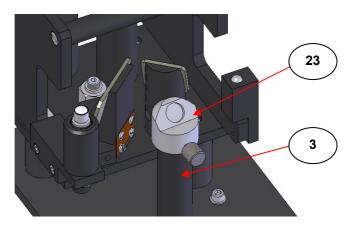


Fig 18. Tilted Sample Cup When Fitted to the Sample Post

User Manual

The sample surface recess is set over at an angle of 45° to the horizontal and as such when the cup (23) is correctly positioned on the sample post (3), light projected from the input ellipsoid E1 mirror (7) to a sample surface set at this angle will be reflected for both its diffuse and specular component to be collected by the output ellipsoid mirror E2 (8).

When using the tilted cup (23), because it collects both diffuse and specular reflectance components of light, a higher energy signal will usually be attainable over use of a standard sample cup (19) for diffuse reflectance measurement alone. In respect of an overall optimum throughput signal from the fine alignment procedure explained in Section 5 (pages 16 to 23 of this manual), the energy signal for a total reflectance measurement may be "peaked up" by slight rotation of the tilted cup (23) on the sample post (3) before the locking screw is secured. The attainment of an energy throughput optimum value helps to orientate the tilted cup (23) for its correct sampling position on the sample post (3).

Procedure For Sample Measurements

Using a Sample Cup Option

When the Selector™ accessory has been optimally aligned for the particular spectrometer system, to collect a spectrum for a sample when using any of the sampling cup options (19, 20 and 23) please follow the steps outlined below.

- A) Record a background reference spectrum using an appropriate reference sample or finely ground KBr or KCl powder in a sample cup option. Fill the sample cup and smooth the sample surface level as described for use of the standard sample cup (19) see page 24.
- B) Lift up the ellipsoid mirror arm assembly (4) by the recessed finger handle (24) to the up/open sample loading position to gain access to the top of the sample post (3).
- C) Carefully place the sample cup with reference powder sample onto the top of the sample post (3). Try to ensure there is no spillage of the sample from the cup.

- D) Lower the ellipsoid mirror arm assembly (4) to the down/closed operating position set by the micrometer screw (25) resting on its ball stop (26).
- E) Collect a spectrum for the reference material.
- F) When the reference spectrum has been collected lift up the ellipsoid mirror arm assembly (4) to gain access to the sample post (3) to remove the sample cup with reference material.

For collection of a **sample spectrum**, repeat the steps A) to F), but replace the reference material with the actual sample material.

Using a Diabraze Pad and Pad Mount Holder Option

When the Selector™ Accessory has been optimally aligned for the particular spectrometer system, to collect a spectrum for a sample when using a diabraze pad (21) with its mount holder (22) please follow the steps outlined below.

- G) Record a background reference spectrum using a clean diabraze pad (21) that has been fitted to the mount holder (22). Ideally the background should be that of the diabraze pad material alone. However, the diabraze pad may give a low throughput signal on some spectrometers. To overcome this, it is recommended that KBr or KCI powder is used on the diabraze pad to improve the background signal.
- H) Lift up the ellipsoid mirror arm assembly (4) by the recessed finger handle (24) to the up/open sample loading position to gain access to the top of the sample post (3).
- Carefully place the diabraze pad (21) on its mount holder (22) into the sample post (3) by simply sliding the stem of the mount holder (22) into the hole at the top of the sample post (3).
- J) Lower the ellipsoid mirror arm assembly (4) to the down/closed operating position set by the micrometer screw (25) resting on its ball stop (26).

User Manual

- K) Collect a spectrum for the reference diabraze pad.
- L) When the reference spectrum has been collected lift up the ellipsoid mirror arm assembly (4) to gain access to the sample post (3) to remove the reference diabraze pad (21) on its mount holder (22).

For collection of a **sample spectrum**, repeat the steps G) to L), but replace the reference diabraze pad (**21**) on a mount holder (**22**) with a diabraze pad (**21**) that has had an appropriate sample transferred to its surface with the actual sample material.

7. Care and Maintenance

When the Selector™ accessory is not in use it can be carefully stored away in its own carry case. This will prevent the potential for any dust or marks accumulating on the mirrors and optical components of the optical unit (1) which would impair the throughput performance of the accessory.

If for any reason finger marks or dirt are found on a mirror surface, the contaminant may be removed by gently and carefully cleaning using an appropriate solvent moistened soft lens tissue **to dab** at the surface of the mirror. (Water, methanol and acetone are suitable solvents to use for cleaning.) **Do not** try rubbing or using an abrading action with the lens tissue against the surface of the mirror as this introduces a risk of creating scratches and indelible marks on the mirror surface which will severely impair the throughput performance of the Selector™ accessory. If you are in any doubt about the condition of a mirror surface and how this can be cleaned safely, please seek advice from Specac or your local Specac representative.

For the sample cups (19, 20 and 23) cleaning, for any sample, but particularly if a salt sample such as KBr or NaCl has been used, Specac recommends that the cups are washed first with cold water to dissolve any residual salt content and are then rinsed with methanol to remove any potential organic residues. Any moisture remaining after this rinsing is removed by finally drying the cup with a tissue.

The diabraze pads (21) are considered to be consumable items and so after sampling, the diabraze pad (21) is removed from its mount holder (22) and can be discarded. The surface of the mount holder (22) can be cleaned using methanol and a tissue to wipe away any residual adhesive to prepare the mount holder in acceptance of a new diabraze pad (21). Replacement diabraze pads are available as a packet of 100 pads under P/N GS19919.

8. Legend (Bubble Part Number) Summary for the Selector™ Accessory

- (1) Selector™ optical unit assembly.
- (2) Selector™ baseplate assembly.
- (3) Sample post (no cup in position).
- (4) Ellipsoid mirror arm assembly.
- (5) First input fixed mirror.
- (6) Second input fixed mirror.
- (7) Input ellipsoid mirror E1.
- (8) Output ellipsoid mirror E2.
- (9) First output fixed mirror.
- (10) Final output adjustable mirror.
- (11) Selector™ optical unit's M4 x 25mm captive screw for fixing to the baseplate assembly.
- (12) Slot location hole for fitting to baseplate.
- (13) Circular location hole for fitting to baseplate.
- (14) Baseplate location pillar for M4 x 25mm captive fixing screw (11).
- (15) Baseplate location pillar for slot location hole (12).
- (16) Baseplate location pillar for circular location hole (13).
- (17) Baseplate support pillar for optical unit (1).
- (18) Carry handle for Selector™ optical unit (1).
- (19) 11mm diameter standard sample cup.
- (20) 4mm diameter micro sample cup.
- (21) Diabraze pad.
- (22) Diabraze pad mount holder.
- (23) Tilted (total reflectance) sample cup.
- (24) Recessed finger handle (to lift and lower ellipsoid mirror arm assembly (4)).
- (25) Height adjustment micrometer screw for ellipsoid mirror arm assembly (4).
- (26) Stainless steel ball stop.
- (27) Shock absorber mechanism for ellipsoid mirror arm assembly (4).
- (28) Location hole for final output adjustable mirror (10) rotation.
- (29) M4 x 10mm grub screw for final output adjustable mirror (10) tilt.
- (30) M3 x 10mm grub screw for ellipsoid mirror adjustment (lower diagonal).
- (31) M3 x 10mm grub screw for ellipsoid mirror adjustment (corner).
- (32) M3 x 10mm grub screw for ellipsoid mirror adjustment (upper diagonal).

9. Spare Parts for the Selector™

P/N GS03600 Pestle and mortar.

P/N GS03610 KBr powder (50g).

P/N GS19915 4mm diameter micro cup.

P/N GS19916 11mm diameter standard sample cup.

P/N GS19917 Tilted cup for total reflectance.

P/N GS19918 Abrasive sample holder mount (packet of 2).

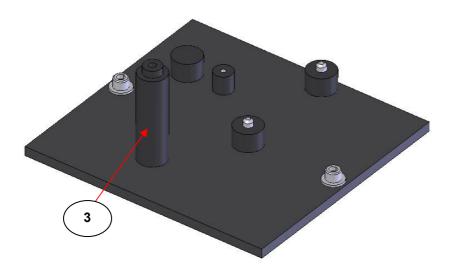
P/N GS19919 Diabraze sample pads (12mm diameter) for Selector (packet of 100).

10. Selector™ Baseplate Installation Guide

Installation of the Selector™ Baseplate into the Spectrometer

The following are descriptions of how to fit the specific L to R or R to L Selector[™] baseplate into your appropriate spectrometer system. In most cases it is best to fit the Selector[™] baseplate (2) into the spectrometer sample compartment first and then fix the Selector[™] diffuse reflectance optical unit (1) to the installed baseplate.

P/N 549-036 - Agilent/Varian/Biorad FTS7, 40, 60, 65, 100, 135, 155, 165, 175, 185, 660, 670, 6000, Excalibur, Scimitar - Beam direction: Right to Left

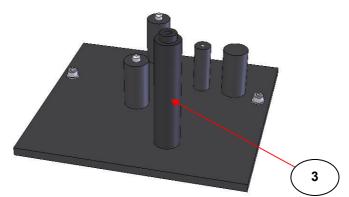


Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector[™] baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head 8-32UNC screws supplied.

Fit the R to L Selector™ optical unit (1) in accordance with the instructions on page 14.



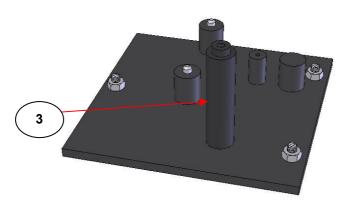
P/N 549-039 - Bomem M100 Series (110, 120) - Beam direction: Left to Right

Remove the spectrometer cover and 3" x 2" sample mount to gain access to the sample compartment.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Fix the Selector™ baseplate to the spectrometer platform with the two 10-32UNC screws supplied.

P/N 549-040 - Bomem MB100 Series (155, 157), MB3000 Beam direction: Left to Right

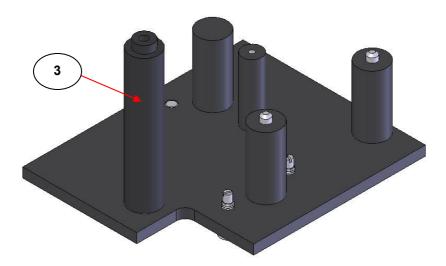


Remove the spectrometer cover and 3° x 2° sample mount to gain access to the sample compartment.

The Selector™ diffuse reflectance accessory may be fixed to its baseplate and the whole assembly is located directly onto the kinematic mount features of the spectrometer.

Fit the L to R Selector™ optical unit (1) in accordance with the instructions on page 14.

P/N 549-037 - Bruker IFS 25, 55, 66, 66V, 85, 88, Vector22, Equinox Beam direction: Right to Left

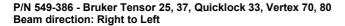


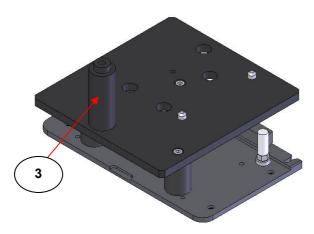
Remove any 3" x 2" mount plate used in the spectrometer from the Bruker baseplate.

Place the Selector[™] baseplate into the sample compartment (on the Bruker baseplate) with the sample holder post (3) to the front.

The two fixing screws of the Selector $^{\text{TM}}$ plate are passed through the larger hole ends of the slots on the Bruker baseplate and the Selector $^{\text{TM}}$ plate is slid from right to left to centralize it on the Bruker plate.

The two screws are then turned anticlockwise to tighten the plates together.

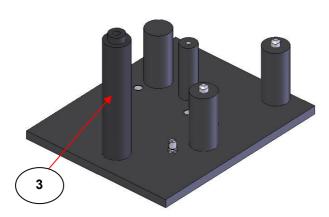




Remove any 3" x 2" mount plate used in the spectrometer from the spectrometer sample compartment.

Place the Selector™ Quicklock baseplate into the sample compartment with the sample holder post (3) to the front of the compartment. The Selector™ baseplate fits securely by connection to the rear with the electric plug sockets and a push clip at the front.

P/N 549-038 - Bruker IFS113V - Beam direction: Right to Left

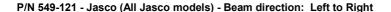


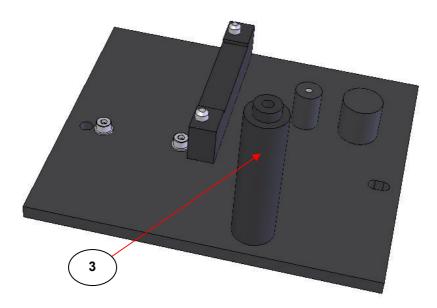
Remove any 3" x 2" mount plate used in the spectrometer from the Bruker baseplate.

Place the Selector™ baseplate into the sample compartment (on the Bruker baseplate) with the sample holder post (3) to the front.

The two fixing screws of the Selector™ plate are passed through the slots of the Bruker plate and the two screws are turned anticlockwise to tighten the plates together.

Fit the R to L Selector™ optical unit (1) in accordance with the instructions on page 14.

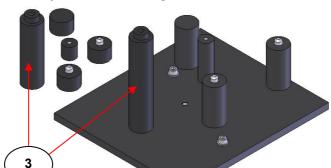




Remove the spectrometer cover and 3" x 2" sample mount to gain access to the sample compartment.

Place the Selector[™] baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head M4 screws supplied.



P/N 549-042 - Mattson Galaxy Series 2000, 3000, 4000, 5000, 6000, 7000, Genesis Series, RS, Infinity - Beam direction: Right to Left

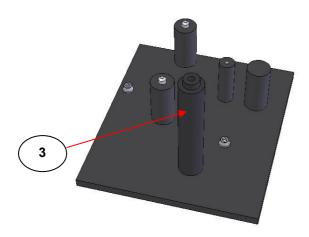
Two sets of pillars are supplied with this baseplate. The set of shorter pillars are used for the Genesis Series spectrometers which have a lower beam height than the other instruments.

Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head 8-32UNC screws supplied. Fit the R to L Selector™ optical unit (1) in accordance with the instructions on page 14.

P/N 549-041 - Mattson Galaxy Series 8000 (New - 3.5" beam height) Beam direction: Left to Right



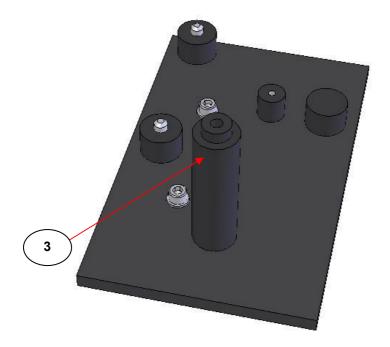
Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head 8-32UNC screws supplied.

Fit the L to R Selector™ optical unit (1) in accordance with the instructions on page 14.

P/N 549-045 - Midac (All Midac models) Beam direction: Left to Right

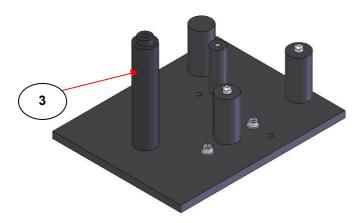


Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head 10-32UNC screws supplied.

P/N 549-031 - Nicolet 500, 700, 710, 740, 800, 5PC, 5SXC, Magna, Protege, Avatar, Nexus, 6700, iS10, iS50 - Beam direction: Right to Left

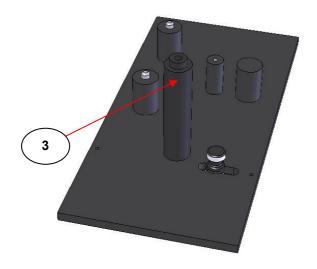


Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

Hold the baseplate in position using the two cap head 8-32UNC screws supplied. Fit the R to L Selector™ optical unit (1) in accordance with the instructions on page 14.

P/N 549-035 - Perkin Elmer PE1700, PE1800, Beam direction: Left to Right



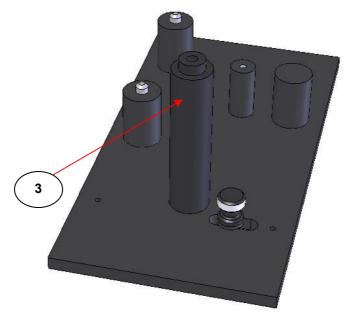
Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

The baseplate is held in position by two location pins into the front support rail of the spectrometer and by tightening of the pull down thumb nut (supplied) to the instruments own fixing screw that passes through the slot at the front of the baseplate.

Fit the L to R Selector™ optical unit (1) in accordance with the instructions on page 14.

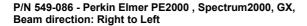
P/N 549-084 - Perkin Elmer PE2000 , Spectrum2000, GX, Beam direction: Left to Right

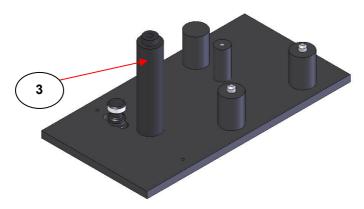


Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector[™] baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

The baseplate is held in position by two location pins into the front support rail of the spectrometer and by tightening of the pull down thumb nut (supplied) to the instruments own fixing screw that passes through the slot at the front of the baseplate.



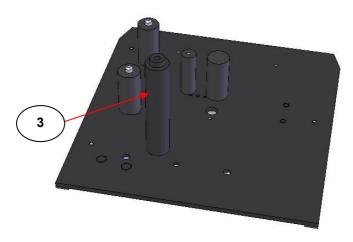


Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment.

The baseplate is held in position by two location pins into the front support rail of the spectrometer and by tightening of the pull down thumb nut (supplied) to the instruments own fixing screw that passes through the slot at the front of the baseplate.

P/N 549-290 - Perkin Elmer PE Spectrum One, Spectrum 100, 400, 4000, 8000 (Frontier) - Beam direction: Left to Right



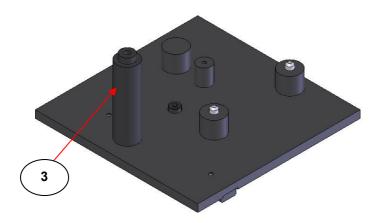
Slide the Selector™ baseplate along the side edges of the sample compartment with the sample holder post (3) to the front of the compartment.

The baseplate locates into the spectrometer by engagement of the 15pin connector at the back of the plate into the spectrometer and by guidance clips on the underside of the plate.

The lilac colored pivoting handle on the underside of the plate is pulled towards you when you need to remove the plate from the spectrometer.

Fit the L to R Selector™ optical unit (1) in accordance with the instructions on page 14.

P/N 549-112 - Shimadzu 8000, 8100, 8200, 8300, 8400, 8500, Prestige 21, IR Affinity, Beam direction: Right to Left



Remove any 3" x 2" mount plate used in the spectrometer.

Place the Selector™ baseplate into the sample compartment with the sample holder post (3) to the front of the compartment. (The Selector™ baseplate has a location strip bar that engages into a slot in the spectrometer sample compartment floor.)

Hold the baseplate in position using the two cap head M5 screws supplied.

Fit the R to L Selector™ optical unit (1) in accordance with the instructions on page 14.

Any Other Spectrometers

For advice concerning spectrometers not included in this manual please consult your nearest Specac representative.

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