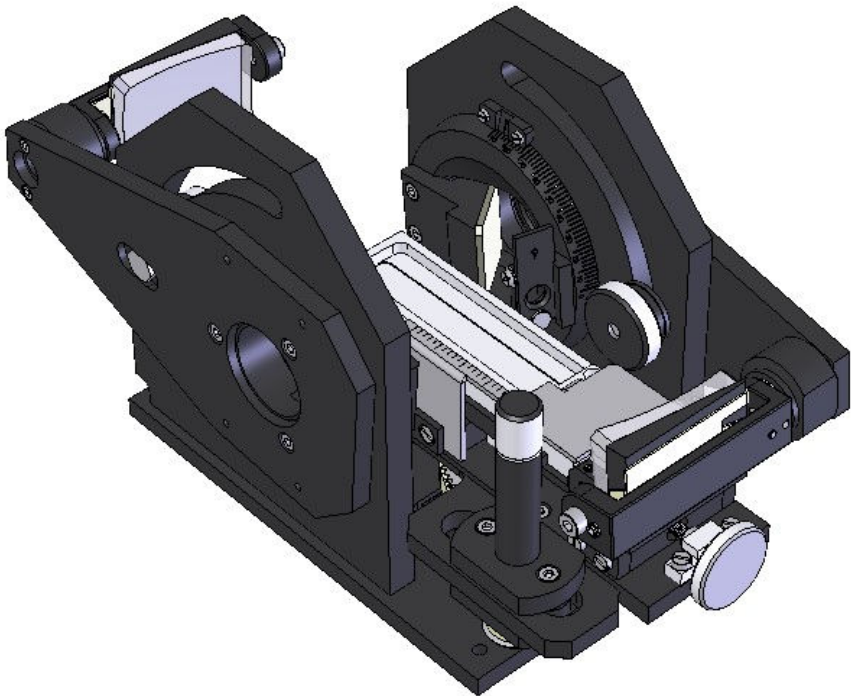




Monolayer Grazing Angle Specular Reflectance Accessory

User Manual



2I-19650-8

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Specular Reflectance Accessory

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Monolayer Grazing Angle Specular Reflectance Accessory P/N GS19650

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1. Introduction

Thank you for buying a Specac product. We trust it will provide you with invaluable and excellent service in use.

Specular reflectance is a non-destructive method for surface measurements using a mirror-like reflection from the shiny or semi-matt surface of a sample. Specular reflectance occurs when the reflected angle of infrared radiation equals the angle of incidence. The amount of light reflected depends on the angle of incidence, the refractive index, surface roughness and absorption properties of the sample.

The Monolayer Grazing Angle Specular Reflectance Accessory P/N GS19650 is a dual-purpose sampling device designed to provide the means of in-situ FTIR monolayer investigation of films at an air/liquid interface and variable grazing incidence angle measurements of thin film coatings on solid reflective surfaces. The Accessory can be simply and easily converted from liquid surface sampling applications to solid surface sampling measurements by use of an appropriate sampling platform.

The beam of radiation is projected to the sampling area via two mirrors (**M1** and **M2**) mounted on a movable “butterfly” arm. (**See Figure 1 on page 5**). One mirror (**M1**) has a plane (flat) surface and is fixed at the hub rotational point of the arm. It moves in direct relation with rotation of the arm from a minimum of an 8° angle setting to a maximum of an 85° angle setting for the arms position – all angles being measured against the perpendicular plane to the sampling surface. The second mirror (**M2**) is fitted at the end of the arm and is adjustable for tilt and rotation of the mirror surface and has a specific radius of curvature to focus the light beam to the sampling area. There is a similar “butterfly” arm/mirror assembly (mirrors **M3** and **M4**) to collect the specularly reflected light and project it to the detector. The range of incident angles of specularly reflected light that can be measured with this accessory corresponds to the 8° to 85° angles with no stray light, on most spectrometer systems. (**See baseplate installation (section 12) for usable range with a specific spectrometer.**) Aperture stops are

provided to redefine the beam profile, for prime position on samples at or near monolayer grazing angles (usually between 80° to 85° angles), to enhance the performance.

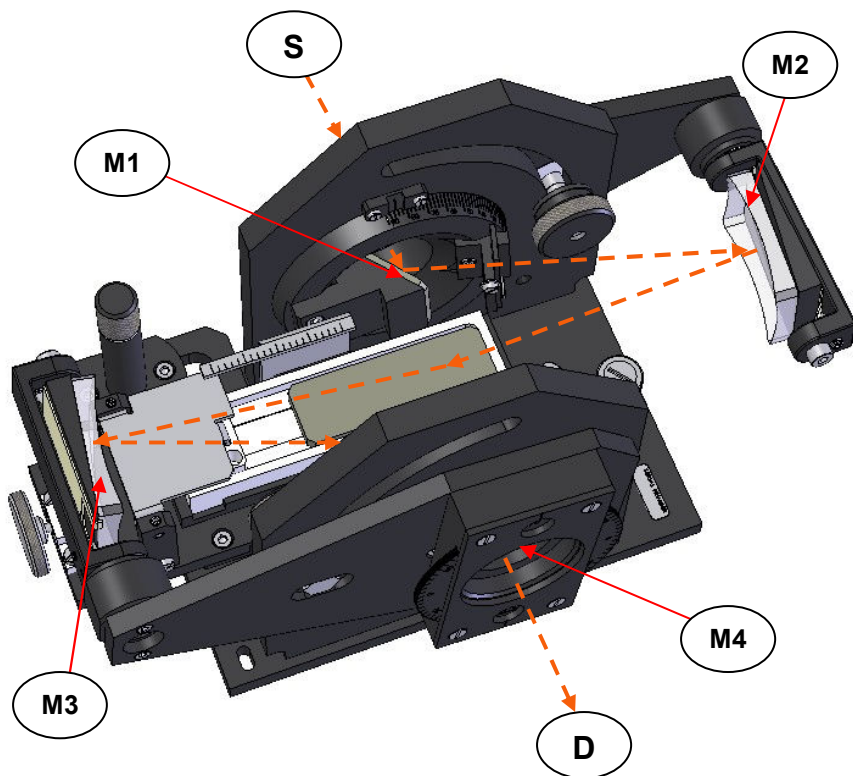


Fig 1. IR light beam sequence through the Monolayer Grazing Angle Accessory from a left to right beam direction (source (S) to detector (D) in a spectrometer sample compartment) is S, M1, M2, sample/reference, M3, M4, D.

Using the **liquid sampling platform assembly**, a variety of monolayer systems including surfactants, proteins, detergents, oils, polymers and phospholipids in biological membranes can be studied in their native environment for information such as solvation, molecular orientation,

configuration and phase transition. The liquid sampling trough part of the platform assembly, fabricated in PTFE, is used to contain the sample of interest. A film stretching device for the trough and an angular graduated rotatable polarizer mount are provided a standard to aid in these types of studies.

For variable incidence angle studies of solid samples, reflective surfaces, such as those found on fragile silicon wafers, can be analysed for optimised coating efficiency and reflectivity. For solid surface sampling an alternative **solid sampling platform assembly** (flat stage) is used within the Monolayer Grazing Angle Accessory in place of the PTFE trough and liquid platform assembly.

An important point about the sampling area requirement for the Monolayer Grazing Angle Accessory is that it is chiefly dependent on the incident angle of the beam of light being projected to the sample surface. Whether it is a liquid sample or a solid surface, as the angle gets shallower (i.e. towards the 85° angle maximum for monolayer experiments), the spread of light across the surface gets larger as the elliptical shaped “pool of light” gets longer. You therefore need a longer and thinner sample size area to make use of the available light, to maximise a sample signal.

At monolayer grazing angles the amount of specularly reflected light can be very low in intensity and hence Specac would normally recommend that a sensitive detection system such as an MCT detector is used for measurement.

2. Principles of Operation for Specular Reflectance Measurement

General Principle

For any spectroscopic measurement, in order to find out any spectral information for an unknown sample, it is usually a requirement to collect “reference” information to compare against the findings for the unknown. The set up and initial alignment of the accessory itself to try and optimise for the best throughput of light available is important to provide sufficient light energy for the measurement involved.

In the instances of use of the Monolayer Grazing Angle Specular Reflectance Accessory P/N GS19650, light itself is specularly reflected off the surface of a suitable sample species. The light presented to the sample at a specific angle in a particular plane of orientation is reflected off towards the detector with some information relating to interaction with the sample species, at the same incident angle and plane of orientation when it leaves the sample, if the sample itself is sufficiently smooth and does not scatter the light too much.

Sampling with the Monolayer Grazing Angle Accessory

With respect to the Monolayer Grazing Angle Accessory for sampling there are two sampling platform assemblies provided to use. One is a liquid sampling platform and the other is a solid sampling platform. For either sampling platform when in position, to obtain an alignment reference throughput measurement the procedure is to use the flat reference mirror provided. The reference mirror is placed within the PTFE trough part of the liquid sampling platform or centrally on top of the solid sampling platform and both butterfly arms that carry the mirrors **M1**, **M2**, **M3** and **M4** are set at an angular value of 45°. For an optimum throughput measurement, the top reflecting surface of the reference mirror is ideally at a **central focal spot** of the incoming beam of light at the 45° angle and the top reflecting surface of the mirror also matches, as best as can be gauged, **the beam height** of the spectrometer. (The **beam height** is the dimension from the

spectrometer floor to the centre line of the light beam as it passes through the sample compartment from a left to right or right to left direction from source to detector and if there was nothing within the sample compartment to deflect or stop the light passage.)

The height of the top reflecting surface of the reference mirror or for any sample itself (to optimize the position for a correct focal point of incoming light), when used on either of the two sampling platforms is adjusted by the geared thumbwheel **height adjustment mechanism** that both the liquid and solid sampling platforms are fitted to for their operation.

Alignment and Sample Positioning

The object of a correct alignment is to create the conditions from the Monolayer Grazing Angle Accessory's multiple settings for angles of incidence from the butterfly arms and different sample sizes and thicknesses etc, such that the focal spot of light at the sample surface can be consistently achieved. If the focal spot position for a consistent height and therefore surface level of the sample is compared to the **central hub** of a wheel, then the angle of incident light that is being directed to the focal spot is a **radial spoke** of the wheel. As the angle of incident light changes with alteration of the angular setting of the butterfly arm the focal spot of any incident light angle should always be concentrated to the "central hub" position and, in effect, correctly and centrally on the top surface spot of the reflecting mirror. A correct height of the sampling surface is therefore very important for consistent sampling when comparing between a background and reference or unknown sample.

Background Spectral Collection

In use of the Monolayer Grazing Angle Accessory, when wishing to take a background spectrum the reference mirror provided and used for the alignment procedure can act as the background sample for both liquid and solid samples. However, if you are looking to study a thin film as a surface layer (e.g. oil on water) using the liquid sampling platform, then the reference "sample" as the background conditions could be just the water itself instead of the reference mirror for background light throughput conditions. Nevertheless, whatever type of

sample is chosen to use as the background conditions for spectral collection, it is important that the reflecting surface height and analytical conditions will be very similar to those when an **actual sample to measure for its surface layer** is placed into position to collect the spectral data.

Sample Thicknesses and Spectral Measurement

If a spectral measurement for a background setting at a particular angular setting of the butterfly arms, e.g. 45°, has been taken, then when changing over from the background “sample” to measure a reference sample or your unknown sample, **the angular settings of the butterfly arms should remain the same** to obtain the best chance for direct measurement of the sample under similar conditions to the background measurement. However, if there is a significant change in the sample surface height from the background conditions, (e.g. a thicker film sample than the reference mirror is to be measured), then a surface height adjustment alone should be made from appropriate adjustment by raising or lowering as necessary the complete sampling platform assembly via the Monolayer Grazing Angle Accessory **height adjustment mechanism**. Monitoring an energy level for the light throughput when the sample is in place using the spectrometers own detection system can be carried out whilst the surface height is altered to establish a peak optimum energy throughput value.

Sample Area Coverage

The Monolayer Grazing Angle Accessory optics gives approximately 1:1 imaging of the instrument beam spot onto the sample. However, the circular beam will end up as an elliptical illuminated area on the sample because of the angle of incidence. The width of the elliptical spot is just the size of the instrument beam or the size of the aperture stop (if smaller). The length of the elliptical spot is the beam diameter divided by the cosine of the angle of incidence. In an example of 10mm diameter for the focal spot size of an FTIR beam spectrometer system and the angles of 30° and 45°, the length of the elliptical pool of light for $\text{Cosine}(30^\circ) = 11.5\text{mm}$ and for $\text{Cosine}(45^\circ) = 14.1\text{mm}$.

These are only approximate sizes, so the sample should be significantly larger if possible to capture as much of the sampling surface to measure. Note that the beam size will vary in many instruments for a change in the resolution setting.

Suitable Angle of Incidence to Use

A “suitable angle of incidence to use” is a loose term, but it pertains to a particular angle of incidence that can be set on the butterfly arms related to the sample type for a surface layer to study. If the sample is relatively thick (another loose term) but of the order of a few microns in thickness and consists of certain laminate layers each of say 10’s of microns in uniform thickness, then incident angles over range of circa 30° to 60° will penetrate to a certain depth into the sample and it may be possible by varying the angle of incidence to discriminate between particular laminates as the surface layers. A suggested way to measure such sample types would be to set sequentially in 5° intervals over the range of 30° to 60° on the butterfly arms for successive spectral collections of the sample having already taken separate background spectra measurement over the same range of angles too, to subtract for the correct comparison conditions (background from sample).

However, when using very thin films (monolayers) like oil on water, the surface layer of the oil film may be of the order of 0.1 microns or a few Angstroms in thickness and so a “shallower” range of incidence angles are needed to be set on the butterfly arms of the Monolayer Grazing Angle Accessory towards the 80° to 85° angle maximum settings. At these **shallow** incident angles, the incident light could be considered as “skating” across the surface of the very thin layer, but there is usually sufficient interaction to register (measure) for a thin layer of a film sample on the surface of say an aqueous solution for these sample types.

At shallow incident angles the set of four aperture stops provided with 1.0mm, 2.0mm, 4.0mm and 6.0mm diameter apertures can help to redefine the beam profile, for prime position on samples at or near monolayer grazing angles (usually between 80° to 85° angles), to enhance the performance.

3. Safety Considerations



With use of any spectroscopic accessory that can involve the study of a wide range of chemical samples, the associated risk in handling may mostly be attributed to the specific sample type to be handled itself. As far as it possible you should follow a procedure for safe handling and containment of the type of sample to be used.

With respect to safety of use specifically to the Monolayer Grazing Angle Specular Reflectance Accessory, the main risk is likely to be from the types of sample that may be analysed by the specular reflectance technique.

No specific hazardous optical materials or components etc are used in construction of the accessory itself. The user should be aware that either by use of the liquid or solid sampling platform assemblies, the sample to be studied will be exposed to the surrounding environment for local temperature and pressure considerations. Therefore, if the sample is toxic or injurious to health, then it must be considered if it can be handled safely using this accessory.

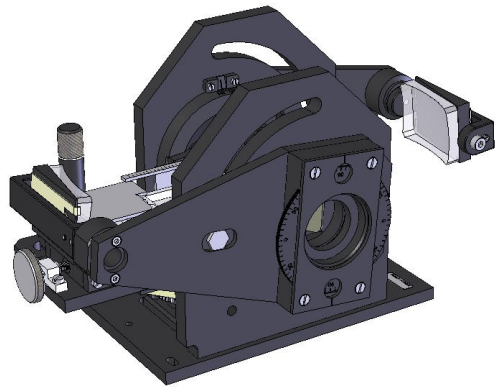
The general rule when working with **any** sample type **is to always wear gloves and safety gear** (e.g. safety spectacles) when handling to obviate the risk of contact with the skin and to avoid contamination of any sample itself.

4. Checklist of Contents

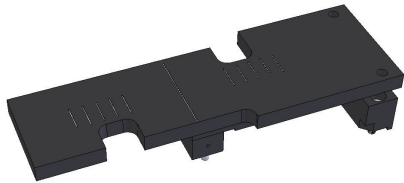
The Monolayer Grazing Angle Specular Reflectance Accessory is provided in its own specific packaging.

On delivery from new please check that the following parts items have been supplied.

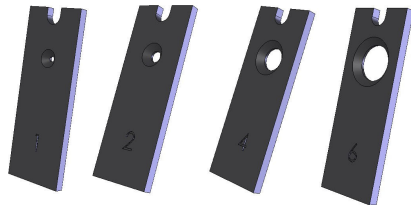
1x Monolayer Grazing Angle Accessory with the PTFE trough and liquid sampling platform assembly fitted in position.



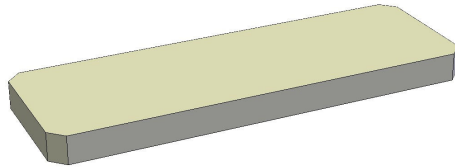
1x Flat (metal) solid sampling platform assembly.



1x Set of four aperture stops.
1, 2, 4 and 6mm diameter apertures.



1x Aluminised glass
reference mirror.



- 1x Allen key 2.0mm A/F (short arm)
- 1x Allen key 2.5mm A/F (long arm)
- 1x Allen key 3.0mm A/F (short arm)

1x Monolayer grazing angle baseplate or fittings (if needed) to mount the Accessory in a specific spectrometer. (Please refer to the Monolayer baseplate installation and configuration guide at Section 12) of this instruction manual for your spectrometer to identify the appropriate baseplate or Monolayer Accessory received.)

1x Instruction manual for the Monolayer Grazing Angle Accessory.

Carefully remove your Monolayer Grazing Angle Accessory and associated parts from the packaging in readiness for use. The mirror parts will be covered in protective lens tissue, so be careful when removing the tissue covers in preparation of usage.

Serial Number of the Monolayer Accessory

The Monolayer Grazing Angle Accessory P/N GS19650 will be provided and marked with a serial number. This is found on a label on the underside of the Accessory and on the carry case.

It may be useful to take a note of the serial number to mark below as this will be needed for identification of the Accessory should you wish to contact Specac.

Monolayer Grazing Angle Accessory Serial No.

5. Installation of the Monolayer Grazing Angle Accessory

The Monolayer Grazing Angle Accessory has a beam height dimension of 63.5mm measured from the underside of the Accessory's own support base to the centre line position of the angled input and output mirrors (**M1** and **M4**) as fixed to the butterfly arms (**1**).

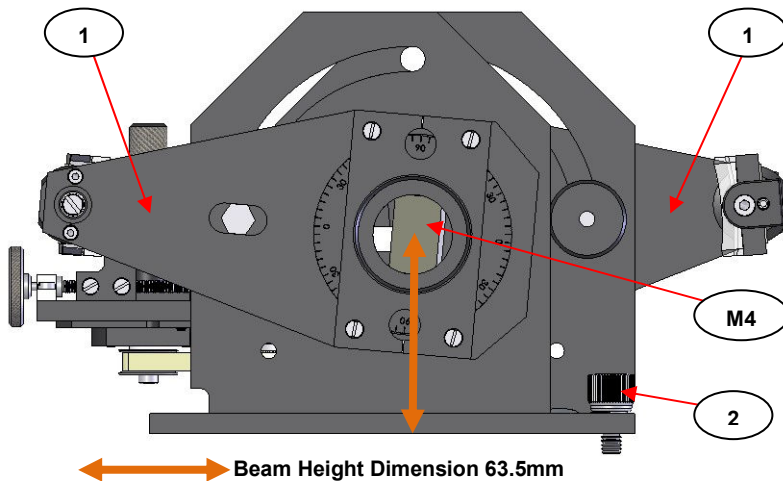


Fig 2. Beam Height Dimension from Monolayer Accessory Support Base to Centre Line Position of M1 and M4 Fixed Mirrors

Depending on the beam height of the spectrometer, the Monolayer Accessory is installed using a specific mounting baseplate or by location studs/pins fitted directly to the accessories own support base. For some spectrometers that require use of a specific baseplate mount to install the Monolayer Accessory, it is necessary to install the specific mounting baseplate into the spectrometer and then attach the Monolayer Accessory to this mount. The Monolayer Accessory is normally secured to its baseplate or the spectrometer floor using the fixing thumbscrew (**2**). For your specific spectrometer please see the installation instructions in Section 12), pages 48 to 64 of this manual.

6. Configuring the Monolayer Grazing Angle Accessory for Liquid or Solid Sampling

When setting up the Monolayer Grazing Angle Accessory prior to use, it is easier to change the sampling platforms before installation in the spectrometer. It is also easier to fit a polarizer to the polarizer mount (22) and to set the polarizing angle (if required). (See pages 30 to 33).

As supplied new, the Monolayer Grazing Angle Accessory is fitted with the PTFE trough (3) on the liquid sampling platform assembly (4) for the analysis of liquid type samples. (E.g. oil film dispersions on water). If solid type samples are to be analysed, then the liquid sampling platform assembly (4) needs to be replaced with the solid sampling platform assembly (11).

The Liquid Sampling Platform Assembly (4)

The liquid sampling platform assembly (4) consists of the following parts as shown in Figs 3, 4, and 5..

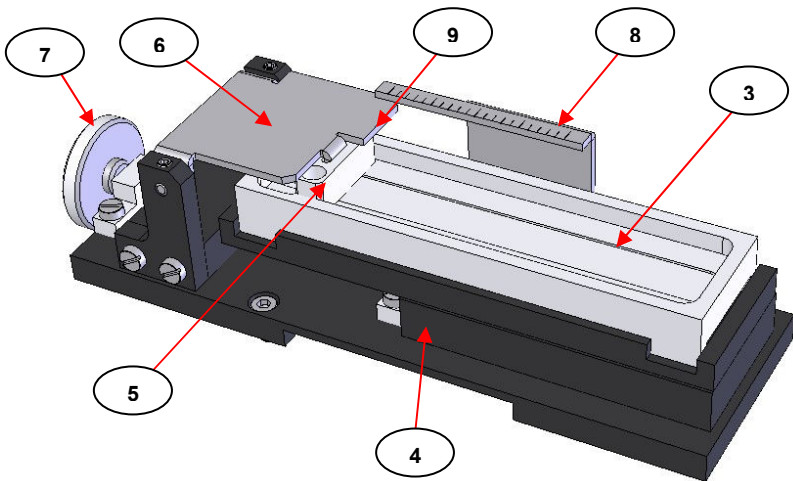


Fig 3. PTFE Trough and Liquid Sampling Platform Assembly

Fig 3. shows that the PTFE trough (3) rests in a recess on top of the main liquid sampling platform assembly (4) and is positioned correctly when the “wider” end of the trough (3) reservoir is near to the tensioning bar (5) adjustment mechanism. (See Fig 4.).

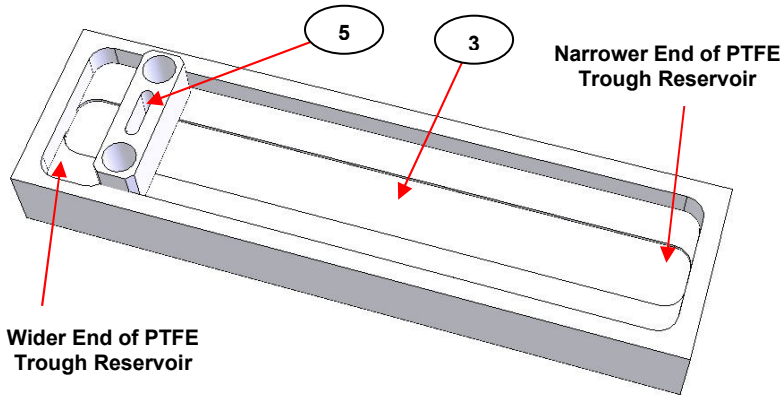


Fig 4. PTFE Trough and Tensioning Bar

The **wider end** of the PTFE trough (3) accepts a small PTFE tensioning bar (5) which is used to alter the surface area and tension of a thin film, such as an oil sample, that would float on top of an aqueous (water based) solvent or solution when placed in the trough (3). The tensioning bar (5) is held in position in the trough (3) by the small tab piece on a hinged plate (6); the tab piece locates into the central slot of the tensioning bar (5). The tensioning bar (5) can be moved laterally, forwards or backwards within the length of the trough (3), by rotation of the thumbscrew (7) to change the relative thickness of any liquid film sample from contraction of the film surface area (film thickness increases), or extension of the film surface area (film thickness decreases). (The hinged plate (6) and the thumbscrew (7) constitute the parts involved in the tensioning bar (5) adjustment mechanism).

Turning the thumbscrew (7) clockwise will **pull** against the tensioning bar (5) via the hinged plate (6) to **extend** the surface area of any floating thin film to make the film **thinner**. Turning the thumbscrew (7) anti-clockwise will **push** against the tensioning bar via the hinged plate

(6) to **contract** the surface area of any floating film to make the film **thicker**. In both events, moving the tensioning bar (5) from a fixed start position when a suitable liquid sample has been placed in the PTFE trough (3) will affect the surface tension characteristics of any floating film and the thickness change can be measured spectroscopically from a particular incident angle set on the monolayer grazing angle accessory butterfly arms (1).

Fixed to the liquid platform assembly (4) is a small graduated scale (8). A different position for the tensioning bar (5) if or when moved in the PTFE trough (3) by rotation of the thumbscrew (7) can be recorded against the division markers on the graduated scale (8) from the front edge (9) of the hinged plate (6).

Note: *It is important that for any movement and repositioning of the tensioning bar (5) in the trough (3) that the hinged plate (6) and its leading edge (9) **pass under** the graduated scale (8). If the hinged plate (6) rises to pass over the top of the graduated scale (8) during any movement, damage may occur to the mechanism.*

For the containment of liquid samples, the dimensions of the PTFE trough (3) reservoir are 95mm long, 22mm wide and 5mm deep. However, because of the fitting of the tensioning bar (5), when at a fully **extended** position for a **thinner** surface film thickness (i.e. when the tensioning bar (5) is pulled towards the wider end of the trough (3) reservoir), the maximum surface length of any liquid sample is limited to 85mm. (The width dimension at 22mm is unaltered.) When the tensioning bar (5) is in a fully **contracted** position for a **thicker** surface film thickness, pushed towards the narrower end of the trough (3) reservoir via maximum anticlockwise rotation of the thumbscrew (7), the surface length of any liquid sample is changed to 40mm.

Between these surface length extremities and change of measurable surface area for a liquid sample, there is no change in the depth of any liquid within the reservoir and hence importantly, **no change in the surface level of the sample** because of the trough (3) reservoir design which allows for flow of the liquid sample **underneath** the tensioning bar (5) as it travels laterally forwards and backwards within the trough (3) reservoir.

Focus Position of Incident Angles of Light on the Sample

With respect to the importance of **a consistent surface level** for successive measurements (i.e. between samples or between a reference and a sample measurement), when a beam of focused IR light at any incident angle from 8° through to 85° is directed to the surface of a liquid sample within the trough (3) reservoir, if the liquid sample surface is consistent with a depth of 5mm for the trough (3) reservoir maximum depth, then the **centre spot** of the incident light will strike the sample surface level at a position circa 20mm in length from the **narrower end** of the trough (3) reservoir.

Note: *This is the **midpoint** position of the 40mm sampling length when the tensioning bar (5) has been set for a fully **contracted** position from maximum rotation of the thumbscrew (7).*

This centre spot sampling position at a 20mm length from the narrower end of the trough (3) reservoir for a focused beam of incident light should ideally **always be in the same position** for a correct optical geometry of beam passage through the Monolayer Grazing Angle Accessory irrespective of whether the tensioning bar (5) is at a maximum or minimum setting allowing for sample surface lengths between 85mm and 40mm.

Apart from consistent depth filling of a liquid sample within the trough (3) reservoir, another crucial factor for achieving a consistent surface level for correct positioning of the centre spot of incident light is the **height position of the complete liquid sampling platform assembly (4) itself**. If the liquid sample depth in the trough (3) reservoir is different between sampling (the volume of liquid sample is different for whatever reason), then the difference in surface level that results between samples can be compensated for by adjustment to the height itself of the liquid sampling platform assembly (4).

The overall height position of the liquid (4) and solid (11) sample platform assemblies when fitted is adjusted by the **height adjustment mechanism (12)** of the main Monolayer Grazing Angle Accessory unit. (Please see Section 7), pages 33 to 34 for explanation on how to make a surface height adjustment).

Fixings for the Liquid Sampling Platform Assembly

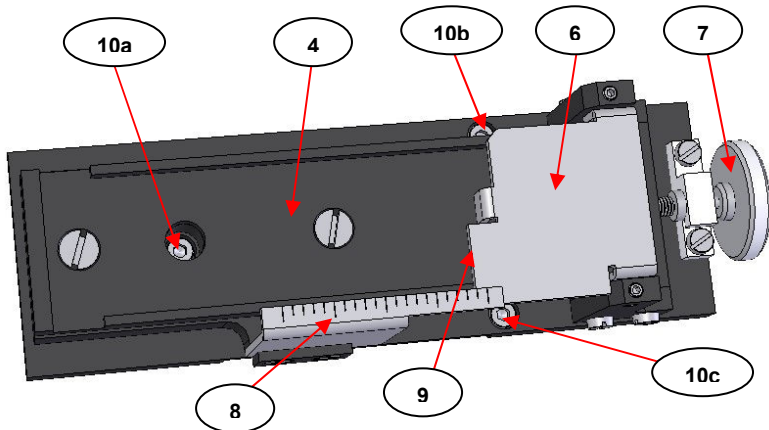


Fig 5. Fixing Screws of Liquid Sampling Platform Assembly

Fig 5. shows the liquid sampling platform assembly (4) after removal of the PTFE trough (3) and tensioning bar (5). The three M3 x 6mm cap head screws (10a, 10b and 10c) are indicated and these are used to affix the complete liquid sampling platform assembly (4) to the **height adjustment mechanism** (12) of the main Monolayer Accessory unit.

Note: *To remove the trough (3) and tensioning bar (5) parts from the liquid sampling platform assembly (4), the hinge plate (6) must be withdrawn to its fully extended position by maximum clockwise rotation of the thumbscrew (7) such that its front edge (9) passes clear and free from underneath the graduated scale (8) and can be hinged upwards and away.*

To remove the liquid sampling platform assembly (4) for use of the solid sampling platform assembly (11), the three screws (10a, 10b and 10c) are undone (screwed anticlockwise) using the 2.5mm Allen key supplied. (See **Fig 6.**) When the liquid sampling platform assembly (4) has been removed, make sure that the fixing screws (10a, 10b and 10c) are kept safely for re-use when necessary.

Note: The PTFE trough (3) **must** be removed from the liquid sampling platform (4) to gain access to the screw (10a) for removal or fitting of the liquid sampling platform assembly (4). The two screws (10b) and (10c) are accessible even with the trough (3) fitted in position. (See Fig 6.)

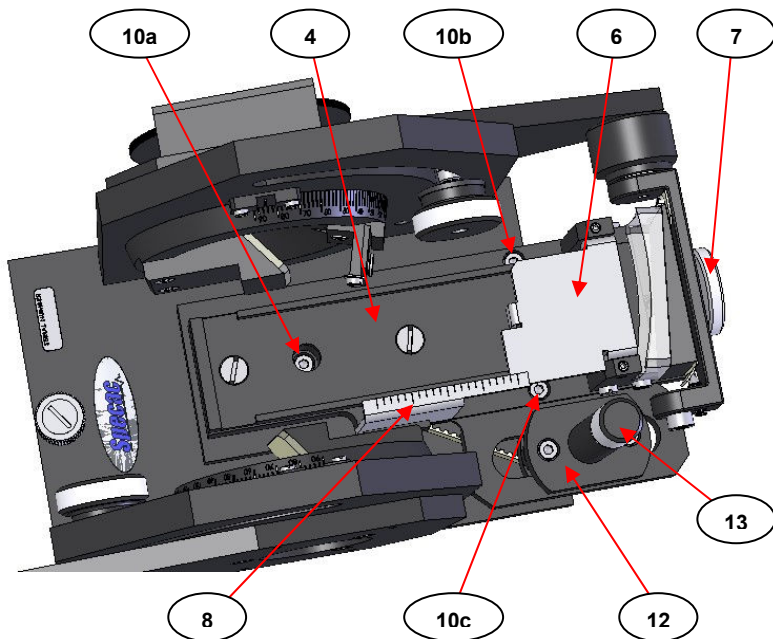


Fig 6. Liquid Sampling Platform Assembly as Fitted in the Monolayer Grazing Angle Accessory



Warning! When changing over the sampling platform assemblies be careful not to damage the mirrors (**M1**, **M2**, **M3** and **M4**) on the butterfly arms (**1**). It may be necessary to have both butterfly arms (**1**) set to an angle of 45° to gain safe working access to the fixing screws of the sampling platform assemblies. (See Section 7, on Adjustment of the Butterfly Arms, pages 28 to 29).

Fig 7. shows the screw fixing positions for (10a, 10b and 10c) on the height adjustment mechanism (12) for the liquid sampling platform assembly (4) when the liquid sampling platform assembly (4) has been removed from the Monolayer Grazing Angle Accessory.

Note: *The height adjustment mechanism (12) alters a platform surface level position by rotation of the thumbscrew post (13). (See Section 7), and pages 33 to 34 for more explanation).*

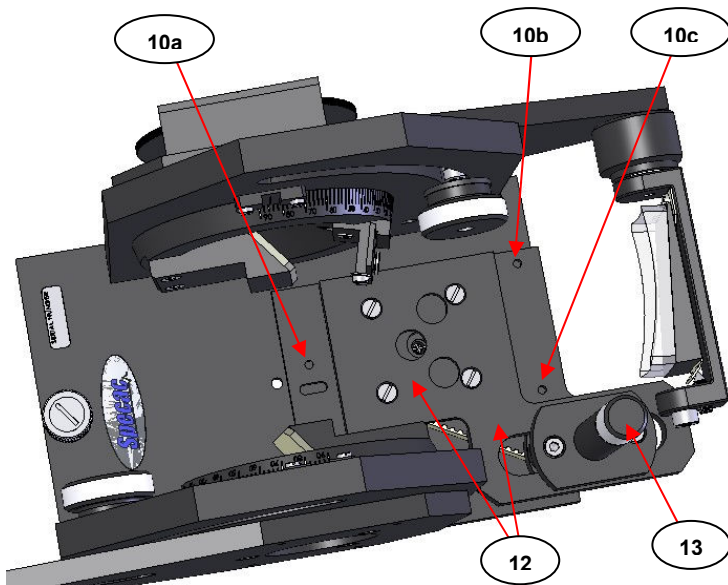


Fig 7. Screw Fixing Positions for the Liquid Sampling Platform Assembly on the Height Adjustment Mechanism

There is a horizontal slot next to the screw hole fixing (10a) for the liquid sampling platform assembly (4) in the height adjustment mechanism (12). This slot is used for location of the dowel pin fixing (14) on the solid sampling platform assembly (11) and so care must be exercised in precise fitting of the two different sampling platform assemblies. (See Fixings for the Solid Sampling Platform Assembly page 22.)

The Solid Sampling Platform Assembly (11)

The solid sampling platform assembly (11) is shown in **Fig 8.** as both topside and underside views.

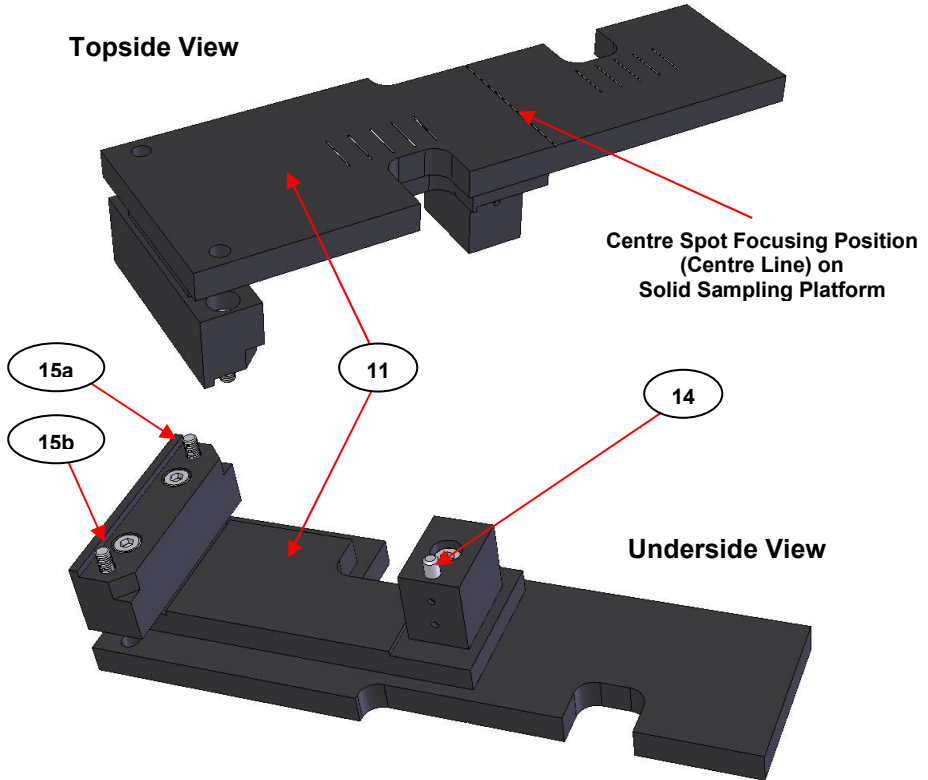


Fig 8. Solid Sampling Platform Assembly

Any smooth, flat solid sample is placed on to the topside surface of the solid sampling platform assembly (11). The maximum length of sample that can be accommodated on the surface of the solids platform (11) is 140mm and the maximum width is 35mm. The **centre spot** of any

incident angle of light to be focused to the sample surface and be specularly reflected for a correct optical geometry of beam passage through the Monolayer Grazing Angle accessory is directed to the **centre line** engraved marking seen on the topside view at **Fig 8**. The centre line is not exactly equidistant at 70mm for the 140mm length of the solid sampling platform (**11**) itself. (It is 80mm from one end and 60mm from the other.) This centre line does though represent the beam passage of light from the source (**S**) to the detector (**D**) as it would pass centrally over the solid sampling platform surface **if the light beam was not deflected** for its passage via the **M1, M2, M3** and **M4** mirrors on the butterfly arms (**1**). Therefore, the centre line can be used as a reference marker to find the centre of any sample for a length dimension to be positioned correctly on the solid sampling platform assembly (**11**). Central sample positioning helps to maximize for sample surface coverage of any focused incident angles of light beams that are created for total light beam passage through the accessory.

Similar to liquid samples with use of the liquid sampling platform assembly (**4**), the height/depth /thickness of a solid sample on the solid sampling platform assembly (**11**) has to be considered in order to provide for a consistent surface level to measure and be coincident with the correct position for the focused **centre spot** of an incident angle of light. Any height adjustment for samples having a significant different in thickness, i.e. more than 1mm, can be compensated for by use of the height adjustment mechanism (**12**). One complete 360° rotation of the thumbscrew post (**13**) of the height adjustment mechanism (**12**) alters the height of a sampling platform assembly (**4**) or (**11**) and hence a sample surface level by 0.5mm. Clockwise rotation of the thumbscrew post (**13**) lowers the platform surface level and anticlockwise rotation of the thumb screw post (**13**) raises the platform surface level.

Fixings for the Solid Sampling Platform Assembly

For fixing of the solid sampling platform assembly (**4**) to the height adjustment mechanism (**12**), there is a single fixing dowel pin (**14**) and two M3 x 12mm cap head (**15a** and **15b**) (captive) screws used as shown for the underside view from **Fig 8**.

Fig 9. is similar to **Fig 7.** and shows the main Monolayer Grazing Angle Accessory components when there is **no** liquid or solid sampling platform fitted to the height adjustment mechanism (**12**), but shows the fixing positions for the dowel pin (**14**) and two screws (**15a** and **15b**) for the solid sampling platform assembly (**11**). Unlike the screw (**10a**) fixing position used for the liquid sampling platform (**4**), the dowel pin (**14**) for the solid sampling platform (**11**) locates into the horizontal slot of the height adjustment mechanism (**12**). The two screws (**15a** and **15b**) of the solid sampling platform assembly (**11**) correspond for a similar fixing as the screws (**10b** and **10c** respectively), used for the liquid sampling platform assembly (**4**).

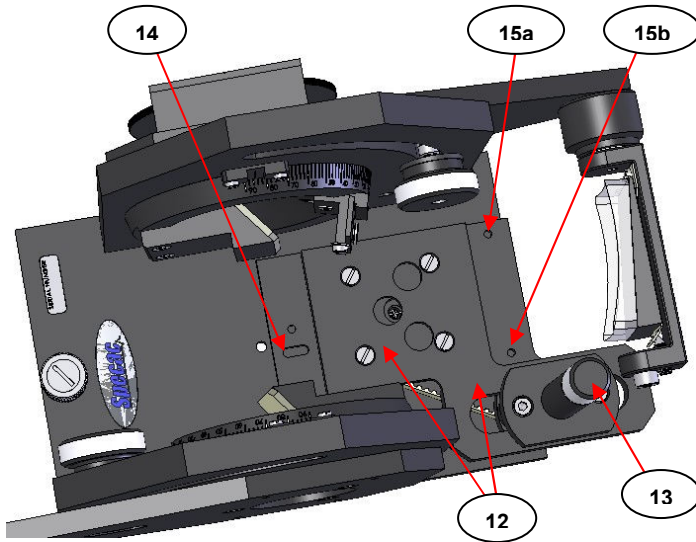


Fig 9. Dowel and Screw Fixing Positions for the Solid Sampling Platform Assembly on the Height Adjustment Mechanism

To remove the solid sampling platform assembly (**11**) for use of the liquid sampling platform assembly (**4**), the two screws (**15a** and **15b**) are undone (screwed anticlockwise) using the 2.5mm Allen key supplied. When the solid sampling platform assembly (**11**) has been removed (pulled clear), the screws (**15a** and **15b**) remain captive in the assembly (**11**) for re-use when necessary.

7. Alignment of the Monolayer Accessory

Before any alignment of the Monolayer Grazing Angle Accessory can be carried out it is necessary to understand the moving parts that require adjustment to obtain an optimum energy throughput level when using the reference mirror (**16**). The parts to adjust involve the butterfly arms (**1**) and the height adjustment mechanism (**12**).

The Butterfly Arms (1)

The Monolayer Grazing Angle Accessory has two very similar butterfly arm (**1**) assemblies. (See **Fig 10.** and **Fig 11.**)

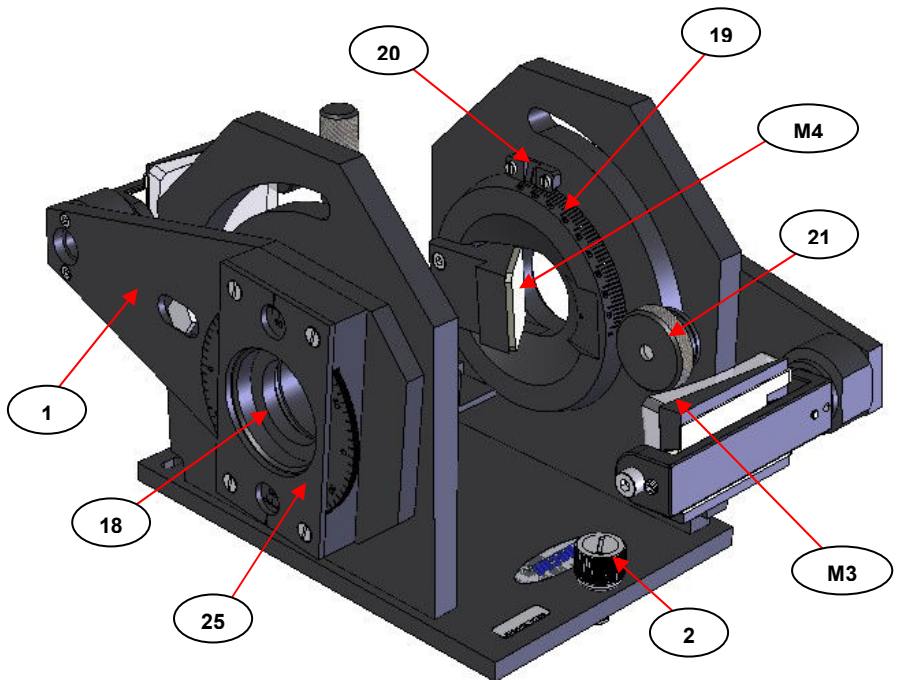


Fig 10. Right Side View of Monolayer Grazing Angle Accessory (Baseplate Fixing Thumbscrew at Rear of Sample Compartment)

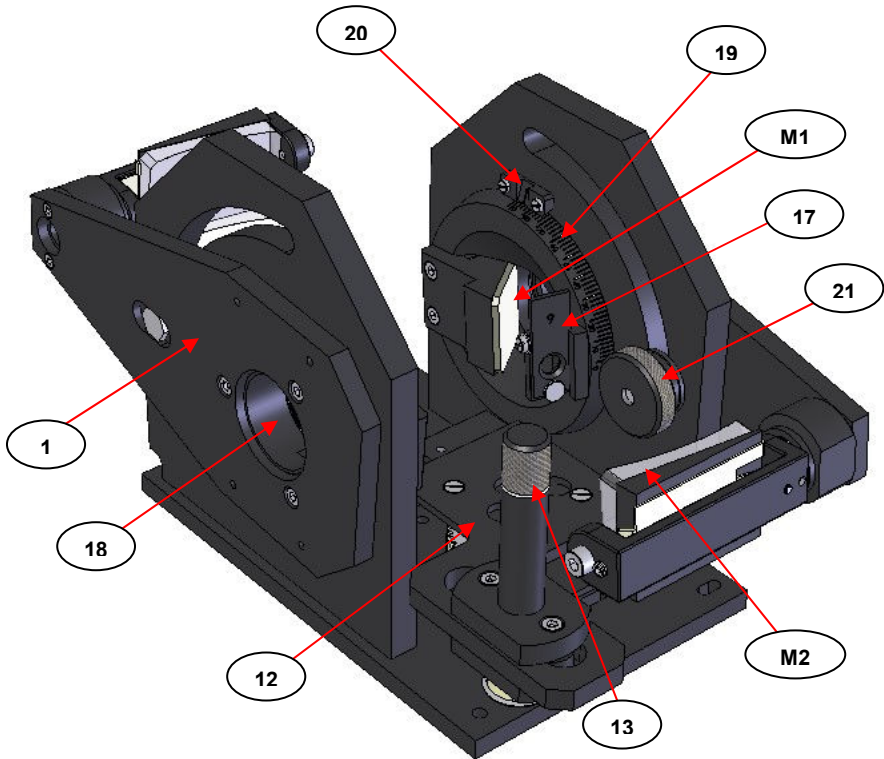


Fig 11. Left Side View of Monolayer Grazing Angle Accessory (Baseplate Fixing Thumbscrew at Rear of Sample Compartment)

Note: *The views for Fig 10. and Fig 11. are specifically for a Monolayer Grazing Angle Accessory that has been built for use in a **Right to Left Beam** direction spectrometer as the light beam travels through the sample compartment from the source to the detector. In comparison to the light beam passage seen traversing through a **Left to Right Beam** direction system from Fig 1., (page 4), the mirrors **M1**, **M2**, **M3** and **M4** have been relabeled for their particular butterfly arm (**1**) fixing to replicate the appropriate beam sequence for a right to left beam direction.*

In respect of **the particular beam direction** that applies to the spectrometer system being used, specifically the aperture stop holder mount (17) component of the Monolayer Grazing Angle Accessory will be fitted to one or other of the butterfly arms (1). In the right to left beam Monolayer Accessory example seen as the view for Fig 11., the aperture holder mount (17) is fixed to the right side butterfly arm (1) as viewed from the front of the Accessory. For a left to right beam built Monolayer Accessory the aperture stop holder mount (17) will be fixed to the left side butterfly arm as viewed from the front of the accessory.

Note: *R to L beam direction – aperture mount (17) on right arm (1).*
L to R beam direction – aperture mount (17) on left arm (1).

Fig 12. shows a close-up detail of the aperture stop holder mount (17) as fitted to the butterfly arm (1). The 6mm diameter size aperture stop is shown in position in the holder. The circular cut out of the aperture stop is positioned over the locator button of the holder mount (17) and is retained to be held upright in the grooved channel to the side.

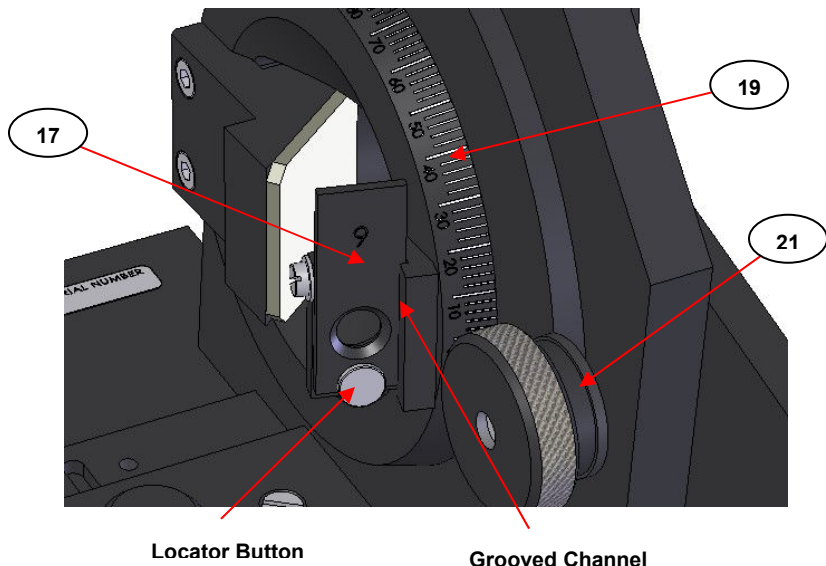


Fig 12. Detail of Aperture Stop Mount Holder on Butterfly Arm

Angle Adjustment of the Butterfly Arm

Each of the butterfly arms (1) are rotatable for an angular range setting of between 8° and 85°. They rotate about an axis point which is the centreline of any IR light beam that passes through the aperture openings (18) to the (M1) (input) fixed mirror on the butterfly arm (1).

Note: As mentioned previously (see page 7), this centerline axis position represents the “central hub of a wheel” with the butterfly arms (1) being the radial spokes of the wheel as the arms (1) with the movable mirrors (M2 and M3) at their ends rotate for their different angular settings. The focal spot of light to the **sample surface level** is ideally directed to this central hub position **for all angles** from angular repositioning of the butterfly arms (1) **only**. No adjustment of the movable mirror (M2 and M3) positions should be necessary for correct focal position of the light at any angle setting for the arms (1) when the mirrors have been aligned at the 45° angle setting from the alignment procedure. (See page 35.)

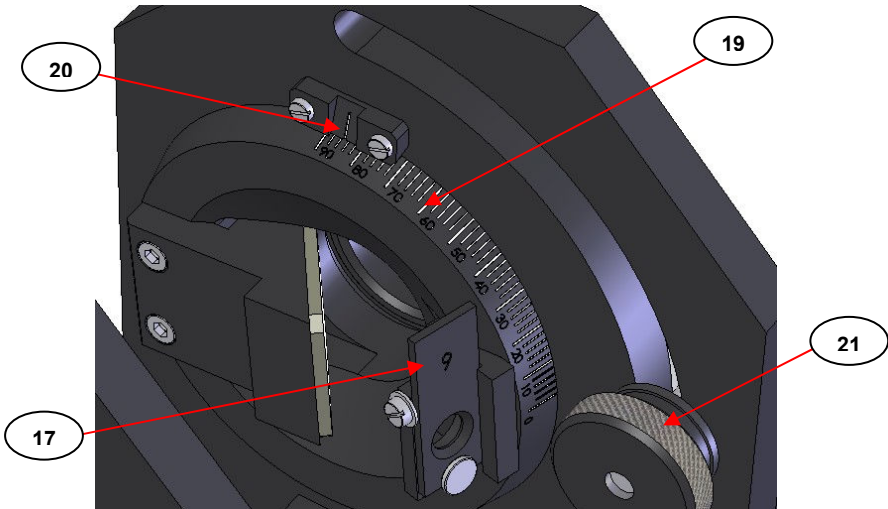


Fig 13. Detail of Angular Division Scale and Reference Mark

On each arm (1) there is an angular division scale (19) from 0° to 90° which is graduated for 2° angle intervals, although it is possible to set the butterfly arm (1) for 1° interval settings at the midpoint between the graduation marks. A specific angle is set for the butterfly arm (1) when the graduated mark on the division scale (19) is aligned with the angle indicator reference mark (20). (See detail at Fig 13. - in the image example shown the butterfly arm (1) has been set for a grazing angle at 85°.) The butterfly arms (1) are adjusted manually from rotation about the central axis point of the aperture openings (18) and are locked for a particular angle setting via use of the locking thumbscrew (21). The thumbscrew (21) is turned clockwise to lock the butterfly arm (1) when at a particular angular setting and anti-clockwise for loosening and to allow rotation of the butterfly arm (1).

Movable Mirror Assembly on the Butterfly Arm

On each butterfly arm (1) there are two mirrors. (Four mirrors in total). The square, flat mirrors identified as (M1) and (M4) are **fixed** to the butterfly arms (1) and rotate accordingly with the butterfly arm (1) for an incident angle setting indicated from the angular division gauge (19) at the reference mark (20).

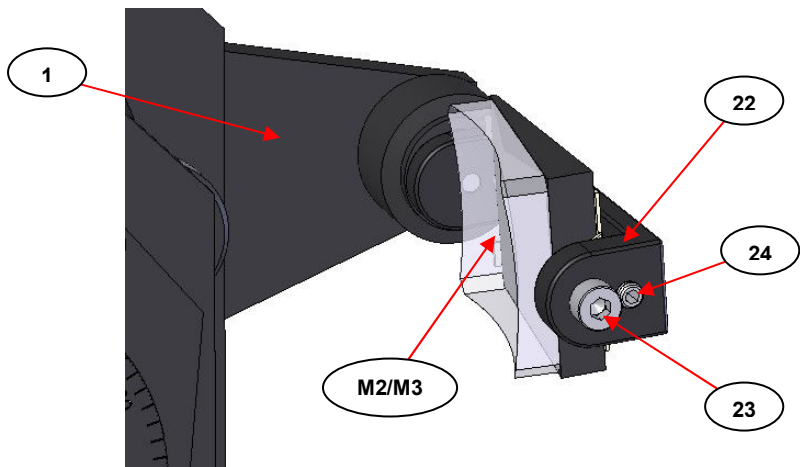


Fig 14. Movable Mirror Assembly on the Butterfly Arm (1)

At the end of each butterfly arm (1) there is a movable mirror carriage assembly (22). The mirrors for this assembly, identified as (M2) and (M3), are rectangular in shape with a concave face for focusing a beam of light to the sample surface centre spot position. The mirrors (M2) and (M3) can be adjusted for their rotation via the M4 x 5mm cap head screw (23) and for their plane of tilt angle via the M4 x 12mm cone pointed grub screw (24) to direct the beam of light accordingly to the centre spot position for the sample. (See Fig 14.)

Note: *Correct alignment for the movable mirrors position has been achieved when the focal spot of light at the sample centre spot is the same at **any incident angle set for the butterfly arms (1) only**. There should be no further rotation or tilt angle changes made to the (M3) and (M4) mirrors for consistent sampling conditions to be maintained.*

The Rotatable Polarizer Mount on the Butterfly Arm

Irrespective of whether the Monolayer Grazing Angle Accessory is to be placed in a left to right or right to left beam direction spectrometer system for operation, as you look at the Monolayer Accessory from the front when installed into a sample compartment, there is a rotatable polarizer mount (25) permanently fitted to the right hand side butterfly arm (1). The polarizer mount (25) is specifically designed to accept Specac's range of P/N GS12000 Series/FTIR Infrared Polarizers, if or when polarized light is a requirement for specular reflectance studies of surface effects.

When sampling with a GS12000 Series polarizer in the polarizer mount (25), light passage from a **right to left** beam direction will pass through the polarizer **first** before reflection off the initial fixed, angled mirror (M1), then on to (M2) and then to the sample surface. However, for any experimentation of light interaction at the sample surface, a polarized beam of light signal from the sample is unaffected, even if the beam direction is from **left to right** and the signal passes through the polarizer as the **last** optical component in the beam path before reaching the detector (D).

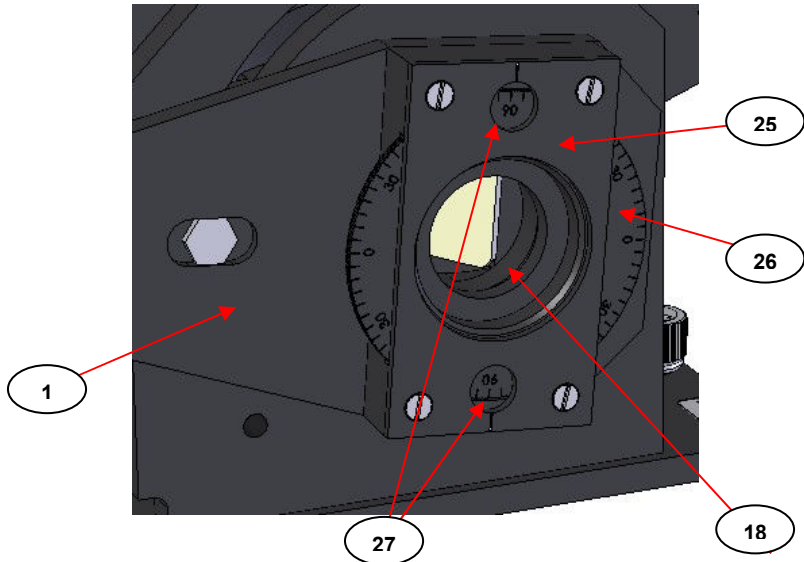


Fig 15. Rotatable Polarizer Mount on the Monolayer Grazing Angle Accessory

Fitting a Polarizer into the Rotatable Polarizer Mount (22)

The GS12000 Series range of Specac polarizers fit directly into the central aperture opening (18) of the polarizer mount (25). (See Fig 15.)

Note: For safe handling and further explanation of the P/N GS12000 Series Polarizers please consult their instruction manual supplied for them - 2I-12000-1.

The GS12000 Series polarizers consist of a grid pattern of 4000 parallel lines per mm photo-etched onto a window substrate material which is supported by a circular metal spring ring clip mount. The outside diameter of the ring mount at circa 34.9mm is an exact fit for the aperture (18) of the polarizer mount (25).

On the spring ring clip mount of the polarizer is a tab piece, which can be adjusted slightly to increase or decrease the circumference of the polarizer ring mount to make the polarizer a tighter or looser fit as required for fit into the polarizer mount (25) aperture (18). However, the tab piece on the polarizer ring mount also determines the direction of the lines of parallelism on the polarizer. With the tab piece of the polarizer ring mount oriented at a 12 O'clock position, the polarizer grid lines run parallel from the 12 O'clock to 6 O'clock positions.

If a GS12000 Series polarizer is to be used for any polarized light measurements using the Monolayer Grazing Angle Accessory the polarizer is fitted via the following procedure.

- 1) Set the right hand butterfly arm (1), that carries the polarizer mount (25), to its maximum horizontal position, i.e. when the 90° angle division on the scale (19) is aligned with the angle indicator reference mark (20). Tighten the locking thumbscrew (21) to secure.
- 2) The polarizer mount (25) carries its own graduated division marked, rotatable ring (26) for an angular setting of the polarizer grid. The ring (26) is marked in 5° angle divisions for setting the angle of the polarizer grid within an estimated 2.5° angular setting if the ring is set midway between a marked division (e.g. 25°, 27.5° and 30° settings). Rotate this ring (26) until the 0° angle graduated marks are at the 3 O'clock and 9 O'clock positions. (As seen for **Fig 15.**)
- 3) Insert the polarizer into the aperture (18) with the tab piece on the polarizer ring mount facing outwards and at the **12 O'clock position** relative to the 0° graduated marks at the 9 O'clock and 3 O'clock positions. The polarizer should be a snug fit (not too tight) to hold in the aperture (18) and be rotated by the ring (26), but its tightness of fit can be altered by adjustment of the tab piece. If the polarizer fit is loose, the tab piece can be prized open a little (do this very carefully to prevent touching the polarizer substrate material) to make a tighter fit when pushed into the aperture (18).

The polarizer is now set for **parallel** lines of polarization running from the 12 O'clock to 6 O'clock positions, with respect to a 90° angle setting being seen through the viewing holes (27) on the polarizer

mount (25). When the butterfly arm (1) is moved and fixed for a specific incident angle of light as indicated by the reference mark (20), although the polarizer in the polarizer mount (25) will rotate to the same degree, the polarization angle of light to the sample remains constant.

For **perpendicular** lines of polarization, the rotatable ring (26) of the polarizer mount (25) should be used to rotate the polarizer by a 90° angle, relative to any angular position that has been set for an incident angle on the butterfly arm (1) to strike at the sample surface, such that the 90° graduated marks are set at the 3 O'clock and 9 O'clock positions of the polarizer mount (25) and the 0° angle division marks are showing in the viewing holes (27).

Note: *Any polarizing grid angle from the range of 0° through to 90° can be set for the polarizer grid against any incident angle over the range of 8° to 85° on most spectrometer systems from appropriate adjustment of the rotatable ring (26) and butterfly arm (1) respectively.*

The Height Adjustment Mechanism (12)

As mentioned in Section 6), there are two different sampling platform assemblies that can be used with the Monolayer Grazing Angle Accessory. For liquid and mobile fluid samples there is a Liquid Sampling Platform Assembly (4) and for solids and film samples there is a Solid Sampling Platform Assembly (11). Each sampling platform is fitted for use directly to the Height Adjustment Mechanism (12) assembly of parts.

For any sampling it is most important to achieve a consistent focal spot of light position for any incident angle of light to be projected at the sample surface. Depending on the thickness of a sample to be measured, and if there are any differences between the reference mirror (16) and the sample thickness itself when placed on the sampling platform surface itself, the actual **sample surface level** for correct focal and spatial light positioning can be achieved from an up or down adjustment using the Height Adjustment Mechanism (12). (See Fig 16.)

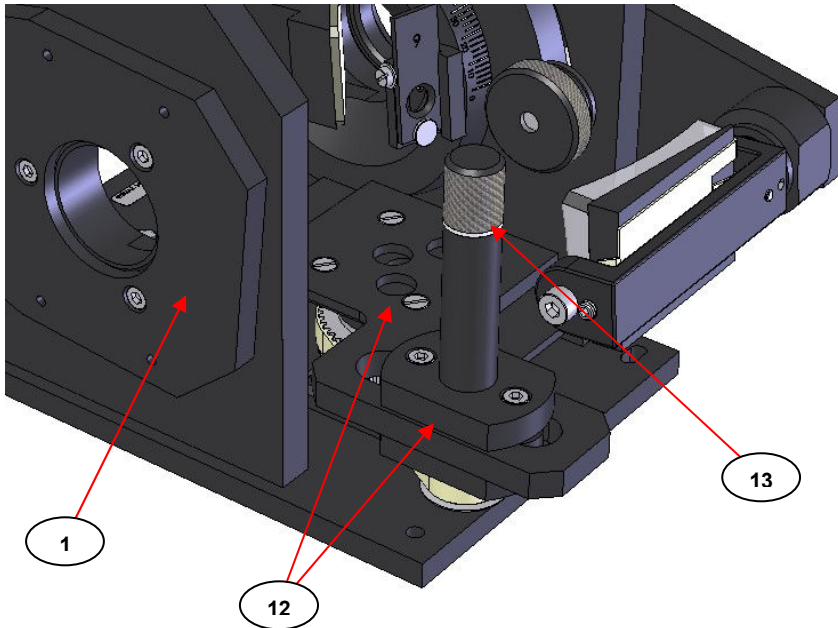


Fig 16. The Height Adjustment Mechanism of the Monolayer Grazing Angle Accessory

Any height adjustment for samples that are significantly different in thickness, i.e. more than 1mm, can be compensated for by use of the height adjustment mechanism (12). A thumbscrew post (13) is connected to two geared wheel cogs and a drive belt assembly of this mechanism. One complete 360° rotation of the thumbscrew post (13) of the height adjustment mechanism (12) alters the height of a sampling platform assembly (4) or (11) and hence a sample surface level by 0.5mm. Clockwise rotation of the thumbscrew post (13) lowers the platform surface level and anticlockwise rotation of the thumb screw post (13) raises the platform surface level.

The Alignment Procedure

The Monolayer Grazing Angle Accessory will have been preliminarily factory aligned, but some degree of fine tuning will be required for use in your specific spectrometer. As supplied from new when packaged, the butterfly arms (1) will be set horizontally for transportation, close to the 90° angle setting for them.

The aluminized surface glass reference mirror (16) supplied is used for alignment and background spectral collection. The signal beam passes through the accessory from either a left to right or right to left direction (source to detector), depending upon which type of spectrometer system being used.

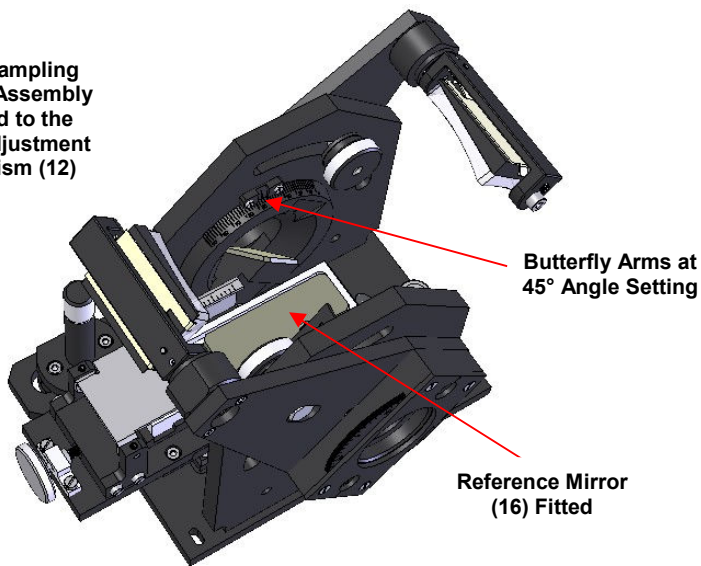
For the purposes in explanation of the alignment procedure to follow, a **left to right beam direction** is used as the example. The beam sequence through the accessory is from: source (S), **M1, aperture stop** (if fitted), **M2, sample/reference mirror, M3, M4, polarizer** (if fitted), detector (D). (See optical beam diagram at Fig 1., page 4).

If a **right to left beam direction** spectrometer system is to be used the sequence changes to: source (S), **polarizer** (if fitted), **M1, aperture stop** (if fitted), **M2, sample/reference mirror, M3, M4, detector** (D).

Alignment Procedure

- 1) The Monolayer Grazing Angle Accessory is installed into the spectrometer system via its appropriate mounting plate system (see details from Section 12), of the particular spectrometer system), with either the liquid sampling platform assembly (4) or the solid sampling platform (11) already fitted into position to the height adjustment mechanism (12). (See Section 6.)
- 2) Loosen the two butterfly arms (1) thumb locking screws (21) and move (turn) each butterfly arm (1) until a reading of a 45° angle setting is registered against the graduated angular scale (19) on each arm (1) at the reference mark (20). Tighten the locking screws (21) to secure both arms (1) in position.

Liquid Sampling
Platform Assembly
(4) Fitted to the
Height Adjustment
Mechanism (12)



Solid Sampling
Platform Assembly
(11) Fitted to the
Height Adjustment
Mechanism (12)

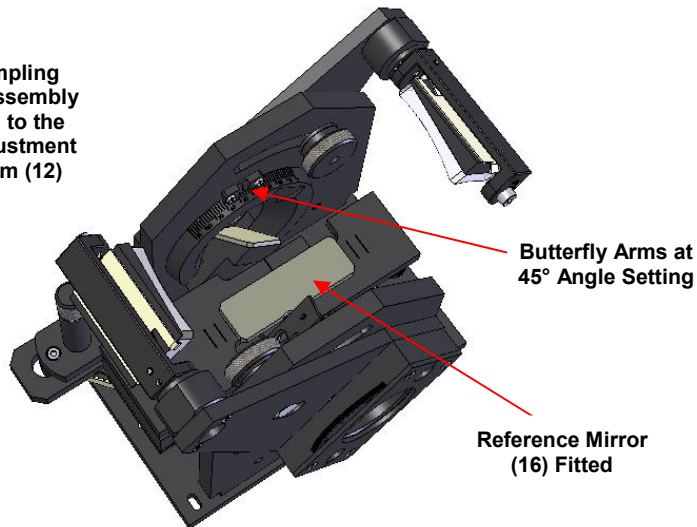


Fig 17. Reference Mirror Fitted for Liquid or Solid Sampling

- 3) Take the reference mirror (**16**) and place it inside the PTFE liquid sampling trough (**3**) of the liquid sampling platform assembly (**4**) or centrally on the engraved section of the flat metal solid sampling platform assembly (**11**), depending on which sampling stage is fitted. (See **Fig 17.**)

Note: *It is extremely important to have the reference mirror (**16**) surface level set at the correct height. When placed in the PTFE liquid sampling trough (**3**) or on the flat metal platform (**11**), the surface level of the mirror should be as near as possible to the spectrometers own optical beam height as this passes through the sample compartment. The optical beam height will vary from spectrometer to spectrometer, but the beam height dimension of 63.5mm as shown for **Fig 2.** on page 14 for the central spot of an aperture (**18**) can be used as a reference to know when the surface level of the reference mirror (**16**) is at its optimum position. The height of the reference mirror surface (**16**) on either sampling platform (**4**) or (**11**) is adjusted via the thumbscrew post (**13**) on the height adjustment mechanism (**12**). Turning the post (**13**) anticlockwise raises the platform height and clockwise lowers the platform height.*

- 4) Adjust the height of the reference mirror for its surface level to be as close as possible to the 63.5mm beam height dimension distance by appropriate up and down movement in turning from a respective anticlockwise or clockwise rotation of the thumbscrew post (**13**). If possible try to approach the optimum height level from one direction only (either an up or down movement) to counteract for any “mechanical slack” that may be found in the height adjustment mechanism (**12**) when there is a change of direction.
- 5) With the two butterfly arms (**1**) set to a 45° incident angle and the reference mirror (**16**) at the correct surface level height, some throughput of signal through the beam path of the accessory should be detected for a signal passing through the spectrometer sample compartment. To optimize an energy throughput signal, the movable mirrors (**M2**) and (**M3**) at the end of the butterfly arms (**1**) will need to be adjusted. (See **Fig 14.** – page 29.)

6) Only small rotation and tilt adjustments should be required. Start first with the movable mirror assembly (**22**) on the light **output** butterfly arm (**1**) that carries mirror (**M3**). Use the 3mm Allen key supplied in the screw (**23**) to **rotate** the mirror (**M3**) first. Rotate the mirror until an optimum (maximum) signal is registered at the detector. If you pass through a peak maximum from turning the screw (**23**) in one direction, turn in the opposite direction until a peak maximum is achieved again.

Note: *Only ever carry out one type of adjustment setting, one step at a time, to establish a peak maximum signal before moving onto another adjustment setting. Mixing rotation and tilt adjustments if a signal peak maximum has not been established from a single adjustment step operation risks **mis-alignment** of the Accessory for an optimum throughput performance.*

7) Having established an optimum throughput from rotation of the **output** mirror (**M3**) now move to its **tilt** adjustment setting using the 2mm Allen key supplied in the grub screw (**24**). Tilt the mirror until an optimum (maximum) signal is registered at the detector. If you pass through a peak maximum from turning the grub screw (**24**) in one direction, turn in the opposite direction until a peak maximum is achieved again.

Note: *If the grub screw (**24**) is turned too far anticlockwise it will dislocate from the back of the spring mount of the mirror (**M3**). If this happens, turn the grub screw (**24**) anticlockwise to clear fully from the spring mount. Push the spring mounted mirror (**M3**) away from the black anodized mirror mount arm carriage assembly (**22**) and turn the grub screw (**24**) clockwise until it re-sets again behind the mount.*

8) Having established an optimum signal throughput from both sequential rotation and tilt adjustments of the output mirror (**M3**), repeat the rotation and tilt adjustment steps 6) and 7), but for the **input** mirror (**M2**).

- 9) When the rotation and tilt adjustments have been made to the input mirror (**M2**), it is necessary to return to the **output** mirror (**M3**) again for possible very fine re-alignment of this mirror for its rotation and tilt position setting to obtain an overall optimum light throughput. (A “rebalancing” of the incident light input mirror (**M2**) settings.)

When steps 2) to 9) have been completed alignment of the Monolayer Grazing Angle Accessory will have been achieved. At an incident angle setting of 45° for the butterfly arms (**1**), with no aperture stops or a polarizer fitted and with the reference mirror (**16**) being used for alignment, a signal throughput of at least 40% transmitted light energy should be achievable when compared to an unobstructed open beam at 100% transmitted light energy on most spectrometer systems.

8. Operation of the Monolayer Grazing Angle Accessory

When the Monolayer Grazing Angle Accessory has been correctly aligned it can be used for the specular reflectance measurement of liquid or solid sample types at their surfaces.

After the alignment stage, the butterfly arms (1) can now be repositioned for the specific angle of incidence required for study and a reference background spectrum can be collected prior to any sample spectral acquisition. (See Section 2), page 7, Background Spectral Collection and page 8, Sample Thicknesses and Spectral Measurement, for more detail.)

Note: *When replacing the reference mirror (16) with a sample, the height of the platform assembly (4) or (11) should be adjusted according to the comparative thickness of the sample. For example, if the sample is a piece of metal thinner than the reference mirror, the solid platform (11) should be raised by turning the thumbscrew post (13) of the height adjustment mechanism (12) anticlockwise until a similar level is achieved for the sample surface height to that of the reference mirror. In trying to keep the reference and sample surface heights as similar as possible, it means that the area and spread of focused radiant light for any given set of incident angles is also as close as possible between reference and sample conditions.*

Liquid Sampling

The Monolayer Grazing Angle Accessory must be fitted with the liquid sampling platform assembly (4) to carry out the study of liquid or mobile fluid samples. For very thin surface films, usually monolayer grazing incident angles over the range from 80° to 85° are selected to be set on the butterfly arms (1).

Note: *A spectrometer with an MCT detector is recommended for use with shallow incident angles over the 80° to 85° range.*

For preparation of a thin film on an aqueous solution and to adjust for the surface tension of such a film to make specular reflectance measurements at monolayer grazing incident angles, the following procedure can be adopted.

After alignment, (see Section 7), pages 35 to 39) and having taken a background spectrum at the incident angle of interest set on both butterfly arms (1), carefully remove the reference mirror (16) from the PTFE liquid trough (3).

Using a dropping pipette, carefully fill the PTFE trough (3) with a liquid (an aqueous solution), close to the top of the trough level, but do not overflow. The surface level of the liquid should be as similar as possible as that presented by the reference mirror (16) when in the PTFE trough (3) for the background measurement step. Filling should be done with the PTFE tensioning bar (5) in position in the middle section of the PTFE liquid trough (3). This position can be gauged if the leading edge (9) of the hinge plate (6) is 3/4's of the way along the graduated scale (8) and circa 50mm distant from the **narrower** internal end of the PTFE trough (3).

Dissolve the sample of interest to cover the aqueous solvent layer as a thin film in a suitable solvent and introduce this solution carefully via a dropping pipette to the aqueous liquid already in the PTFE trough (3).

Allow the sample carrier solvent to evaporate off, so that the sample forms a film on the aqueous layer in the PTFE liquid trough (3).

Proceed with analysis by selecting suitable angles of incidence for monolayer detection. The surface film area can be stretched by turning the thumbscrew (7) clockwise as this pulls on the PTFE tensioning bar (5) to lengthen the film sample and make the film slightly thinner.

Note: *When operating at grazing angles of incidence over the 80° to 85° angular range use of an aperture stop in the holder (17) may be required. Please see Notes on the Aperture Stops, page 43, to explain.*

Cleaning the PTFE Trough (3)

The PTFE liquid trough (3) can be removed from the liquid sampling platform assembly (4) for cleaning. To remove the trough (3) and tensioning bar (5) parts, the hinge plate (6) must be withdrawn to its fully extended position by maximum clockwise rotation of the thumbscrew (7) such that its front edge (9) passes clear and free from underneath the graduated scale (8) and can be hinged upwards and away.

The PTFE liquid trough can be cleaned by soaking in chromic acid solution or refluxed with a suitable solvent to remove all traces of the film sample. It can also be cleaned by an overnight soak in a liquid detergent followed by subsequent washings in a toluene/acetone mixture and then distilled water.

Solid Sampling

The Monolayer Grazing Angle Accessory must be fitted with the solid sampling platform assembly (11) to carry out the study of solid or smooth flat film samples. The full incident angle range from 8° to 85° can be selected for study against an appropriate solid sample type for any surface coverings/coating etc. from installation of the Monolayer Grazing Angle Accessory into most spectrometer systems. Polymer film type samples with different laminate layers may be studied from variation of incident angles and their effective penetration depth for depth profiling of a layer when compared to suitable reference type samples for a known layer thickness. For very thin surface films, usually grazing incident angles over the range from 80° to 85° are selected to be set on the butterfly arms (1).

Note: *A spectrometer with an MCT detector is recommended for use with shallow incident angles over the 80° to 85° range. When operating at these monolayer grazing angles of incidence, use of an aperture stop in the holder (17) may be required. Please see the section Notes on Aperture Stops, page 43, to explain.*

For any solid sampling, after alignment, (see Section 7), pages 35 to 39) and having taken a background spectrum at the incident angle of interest set on both butterfly arms (1), carefully remove the reference mirror (16) from the solid sampling platform (11).

Carefully place the solid sample of interest on the marked area of the solid sampling platform (11). Try to align the centre of the sample for its size with the centreline mark on the surface of the solid sampling platform assembly (11) for maximum potential light coverage of the sample at any angle of incidence to measure. If there is a difference in the relative thickness of the sample to the reference mirror (16), then adjust the height of the sampling platform accordingly with the thumbscrew post (13) of the height adjustment mechanism (12) to try and equalise for the actual sample surface level to measure.

Tip: *Use the spectrometers detection system to monitor the throughput energy level of light at the sample surface as you raise and/or lower the platform from rotation of the thumbscrew post (13). There should be an energy throughput maximum at a similar surface level for the sample when compared to the optimum throughput energy achievable for the reference mirror (16) surface level position.*

Commence analysis at the required angles of incidence as set on the butterfly arms (1).

Notes on the Aperture Stops for their Usage

As incident angles near those of monolayer grazing incidence, i.e. towards shallower angles of 85° maximum as set on the butterfly arms (1), the radiant beam of light becomes elongated and elliptical over the surface of the sample. This can lead to an overfilling of signal for the sample area, but this effect can be minimised by use of an aperture stop.

Four aperture stops of 1, 2, 4 and 6mm diameter are supplied with the Monolayer Grazing Angle Accessory which can be placed in the aperture stop holder (17) to reduce the incoming beam diameter and

give more effective sample coverage if the sample surface itself is small in area. The largest aperture stop should be chosen to conserve as much light energy throughput as possible for a sample measurement and at angles near to the monolayer grazing incidence maximum (85°) **the sample area** should also be as large as possible to provide the maximum potential for light/sample surface interaction.

As mentioned previously (see page 27), in respect of **the particular beam direction** that applies to the spectrometer system being used, specifically the aperture stop holder mount (**17**) component of the Monolayer Grazing Angle Accessory will be fitted to one or other of the butterfly arms (**1**). In the right to left beam Monolayer Accessory example seen as the view for **Fig 11.**, the aperture holder mount (**17**) is fixed to the right side butterfly arm (**1**) as viewed from the front of the Accessory. For a left to right beam built Monolayer Accessory the aperture stop holder mount (**17**) will be fixed to the left side butterfly arm as viewed from the front of the accessory.

Note: *R to L beam direction – aperture mount (**17**) on **right** arm (**1**).*
*L to R beam direction – aperture mount (**17**) on **left** arm (**1**).*

If you should ever require moving the aperture stop holder (**17**) to the other butterfly arm (**1**), simply unscrew the single M2 x 6mm cap head screw fixing with an Allen key. Be careful not to touch any of the mirrors (**M1**, **M2**, **M3** and **M4**) when carrying out this change.

9. Care of the Monolayer Grazing Angle Accessory

The Monolayer Grazing Angle Accessory should provide years of good service if handled carefully and used as instructed.

Specac would recommend that when the Monolayer Accessory is not being used it is stored within the carry case from which it was provided. This will help to keep any dust from settling on the mirrors (**M1**, **M2**, **M3** and **M4**) which would reduce any overall light throughput performance for the Accessory.

If the carry case is to be used for storage purposes, be careful that the movable mirror carriage assemblies (**22**) at the ends of the butterfly arms (**1**) are not disturbed from their settings for use on the next occasion otherwise the Accessory will need to undergo the alignment procedure again for optimum performance of operation.

In general for any sample handling Specac would always recommend that the user wears gloves and suitable safety equipment if the sample itself is in any way hazardous. However, if the mirrors (**M1**, **M2**, **M3**, **M4** and the Reference Mirror (**16**)) become dirty, dusty or marked from accidental spillage of samples or from fingerprints, their surfaces can be cleaned, but ONLY by use of very fine lens tissues and moistened with a suitable solvent such as methanol or acetone.



Warning! *If cleaning a mirror surface, avoid an abrasive rubbing action even when using a soft lens tissue. If the mirror surface becomes scratched, the light throughput performance for the Accessory may be drastically affected.*

For cleaning of the PTFE trough (**3**) between samples, please refer to page 42. If the solid sampling platform assembly (**11**) surface needs cleaning, then appropriate solvents such as water, methanol or acetone can be used with a soft lens tissue.

10. Monolayer Accessory “Bubble Numbers” Part Identification List

- (1) Butterfly arm assembly.
- (2) Fixing thumbscrew to Monolayer baseplate.
- (3) PTFE Liquid Trough.
- (4) Liquid sampling platform assembly.
- (5) PTFE tensioning bar.
- (6) Hinge plate of PTFE tensioning bar (5) adjustment mechanism.
- (7) Thumbscrew of PTFE tensioning bar (5) adjustment mechanism.
- (8) Graduated scale on liquid sampling platform assembly.
- (9) Front edge of hinge plate (6).
- (10) M3 x 6mm cap head fixing screws for liquid sampling platform assembly (4) – (10a, 10b and 10c).
- (11) Solid sampling platform assembly.
- (12) Height adjustment mechanism.
- (13) Thumbscrew post of height adjustment mechanism (12).
- (14) Dowel fixing pin of solid sampling platform assembly (11).
- (15) M3 x 12mm cap head fixing screws for solid sampling platform assembly (11) – (15a and 15b).
- (16) Reference mirror for sampling platform assemblies (4 and 11).
- (17) Aperture stop holder assembly (with aperture stop).
- (18) Aperture opening on butterfly arm assembly (1).
- (19) Butterfly arm angular division scale.
- (20) Butterfly arm angular division scale reference setting mark.
- (21) Locking thumbscrew of butterfly arm (1).
- (22) Movable mirror carriage frame assembly.
- (23) M4 x 5mm cap head screw for movable mirror rotation.
- (24) M4 x 12mm cone point grub screw for movable mirror tilt.
- (25) Rotatable mount for polarizers.
- (26) Rotatable ring of angular division indicator marks on rotatable mount for polarizers.
- (27) Viewing holes for angle setting of polarizer grid on rotatable mount for polarizers.

11. Monolayer Accessory Spare Parts

Complete Monolayer Grazing Angle Accessory

P/N GS19650 Monolayer Grazing Angle Accessory.

(Please specify which spectrometer (make and model number) is to be used when ordering against this part number)

Spare Parts

P/N GS19662 PTFE trough and tensioning bar of the liquid sampling platform assembly.

P/N GS19663 Aperture stops (4) and holder.

12. Monolayer Grazing Angle Accessory and Baseplate Installation into a Spectrometer

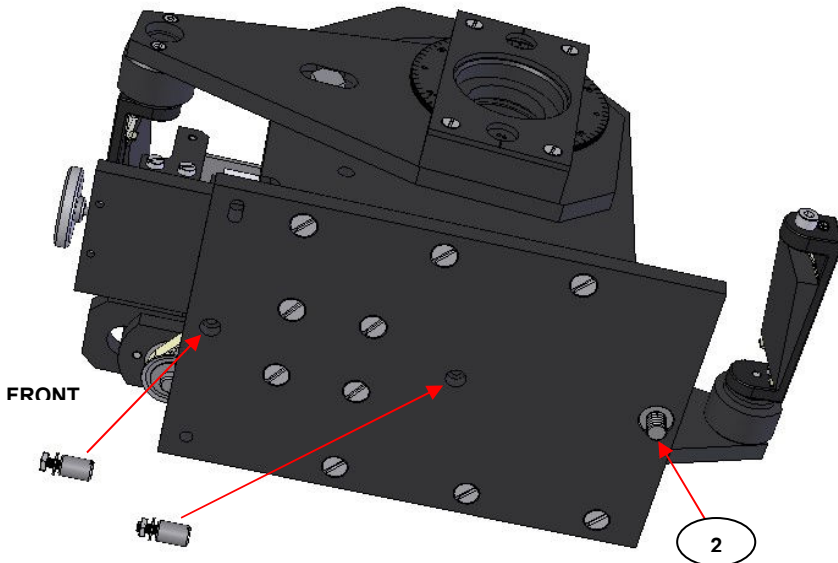
The following is a description of how to fit a Monolayer Grazing Angle Accessory and its specific baseplate (if a separate baseplate is needed) into your appropriate spectrometer system. In most cases it is best to fit the Monolayer Grazing Angle baseplate into the spectrometer sample compartment first and then fix the Monolayer/Grazing Angle Accessory to the installed baseplate. Please locate and follow the description for installation of the Monolayer Grazing Angle Accessory for your specific spectrometer system prior to any alignment that will be needed after correct installation.

Agilent FTS7, 40, 60, 65, 100, 135, 155, 6000

Beam direction: **Right to Left**

Angle range that can be set: **8° to 50°**.

No separate monolayer baseplate required for installation.



Fixing Pins Fitted to Location Holes on Underside Plate

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

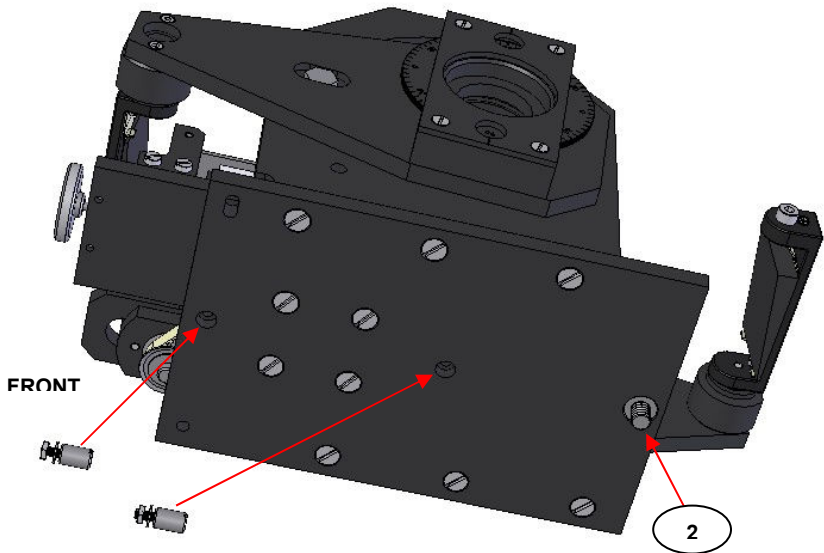
The Monolayer/Grazing Angle accessory is supplied with the necessary two fixing pins attached to the underside of the accessory. (See Diagram). Locate by placing the Monolayer/Grazing Angle Accessory with fixing pins onto the floor baseplate of the spectrometer and secure into the spectrometer floor with the thumb screw (2) of the accessory at the rear of the sample compartment.

Agilent FTS165 175, 185, Excalibur, Scimitar

Beam direction: **Right to Left**

Angle range that can be set: **8° to 85°**.

No separate monolayer baseplate required for installation.



Fixing Pins Fitted to Location Holes on Underside Plate

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

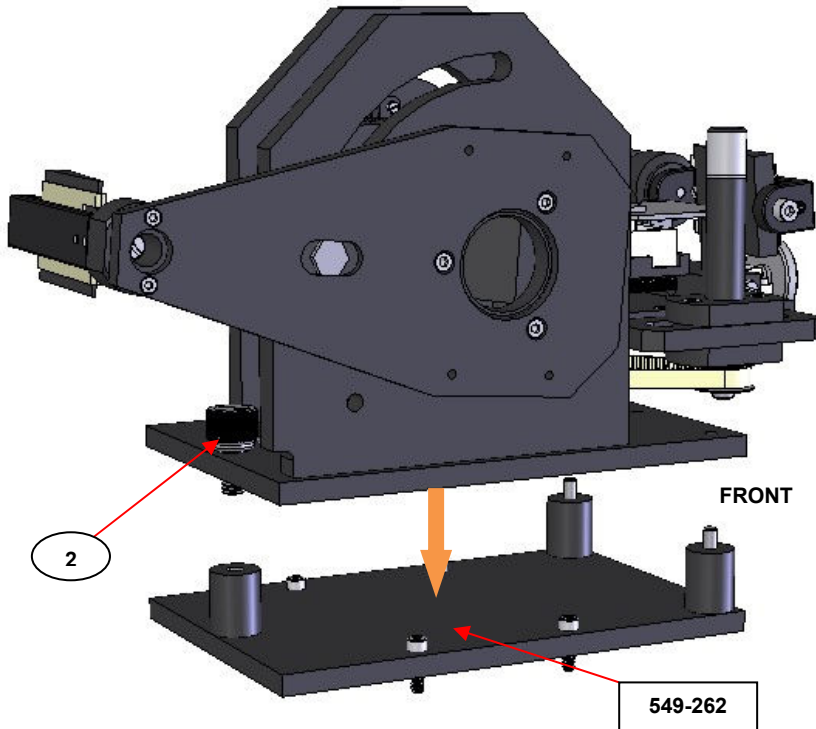
The Monolayer/Grazing Angle accessory is supplied with the necessary two fixing pins attached to the underside of the accessory. (See Diagram). Locate by placing the Monolayer/Grazing Angle Accessory with fixing pins onto the floor baseplate of the spectrometer and secure into the spectrometer floor with the thumb screw (2) of the accessory at the rear of the sample compartment.

Bomem M100 Series (110,120)

Beam direction: **Left to Right.**

Angle Range: **8° to 85° with spectrometer covers removed.**

Baseplate installation required – **baseplate P/N 549-262.**



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

Place the Monolayer/Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment.

Fix the Monolayer/Grazing Angle baseplate to the spectrometer platform with the four 6-32UNC x 1/2" cap head screws supplied.

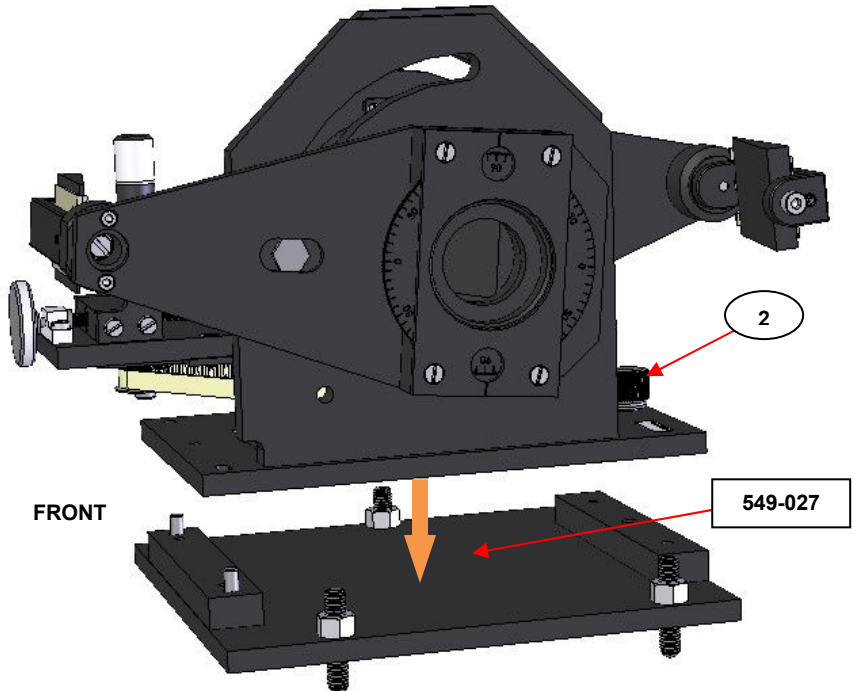
Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single pillar baseplate fixing at the rear of the sample compartment.

Bomem MB100 Series (155,157) Kinematic Mount

Beam direction: **Left to Right.**

Angle Range: **8° to 85° with spectrometer covers removed.**

Baseplate installation required – **baseplate P/N 549-027.**



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

The Monolayer/Grazing Angle baseplate has three adjustable screw feet which locate into the kinematic mounts of the spectrometer. There are two location strips, one with a couple of dowel location pins. Place the baseplate onto the kinematic mounts of the sample compartment with the location strip and two dowels to the front of the compartment. Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single location strip fixing at the rear of the sample compartment.

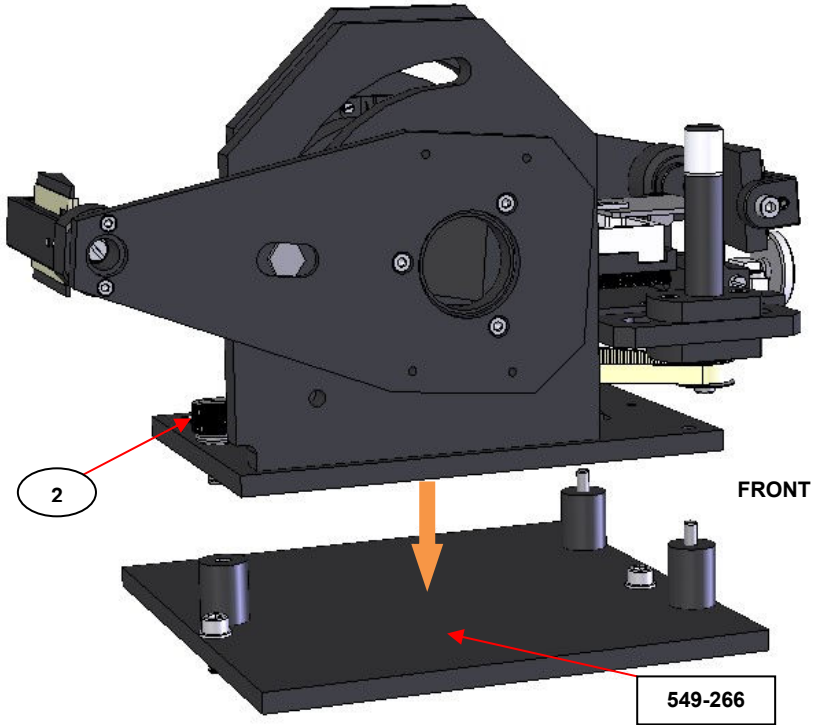
The Monolayer/Grazing Angle accessory may be first fixed to its baseplate and the whole assembly is located directly onto the kinematic mount features of the spectrometer if desired.

Bomem MB100 Series (155,157) Non-Kinematic Mount

Beam direction: **Left to Right.**

Angle Range: **8° to 85° with spectrometer covers removed.**

Baseplate installation required – **baseplate P/N 549-266.**



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

Place the Monolayer/Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment.

Fix the Monolayer/Grazing Angle baseplate to the spectrometer platform with the two 10-32UNC x 1/2" cap head screws supplied.

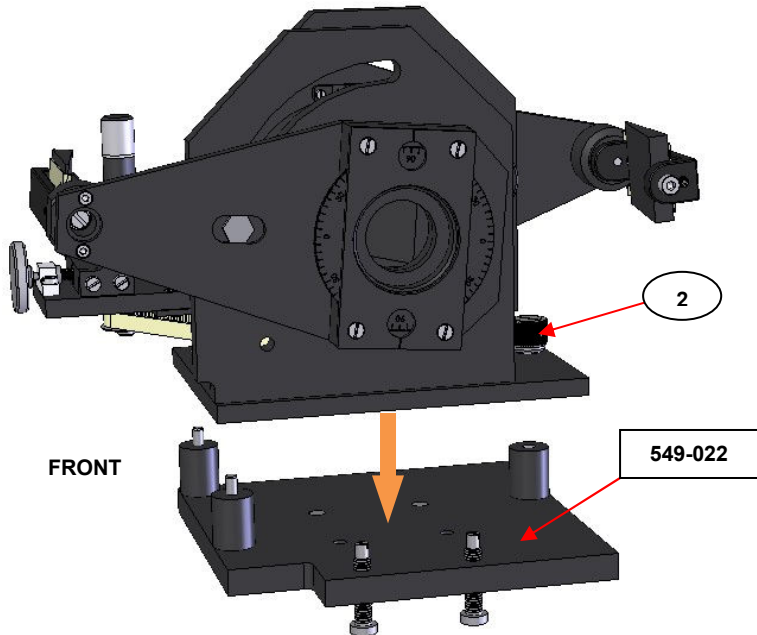
Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single pillar baseplate fixing at the rear of the sample compartment.

Bruker IFS25, 45, 48, 55, 66, 66V, 85, 88, Vector22, Equinox

Beam direction: **Right to Left.**

Angle Range: **8° to 85° Fits with compartment lid open. (8° to 50° and 70° to 85° for IFS88 only.)**

Baseplate installation required – **baseplate P/N 549-022.** (Monolayer accessory base size is unique for fitting to baseplate P/N 549-022).



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment. The two underside fixing screws of the Monolayer accessory baseplate are passed through the larger hole ends of the slots on the Bruker baseplate and the accessory plate is slid from right to left to centralise it on the Bruker plate. The two screws are then turned anticlockwise to tighten the plates together.

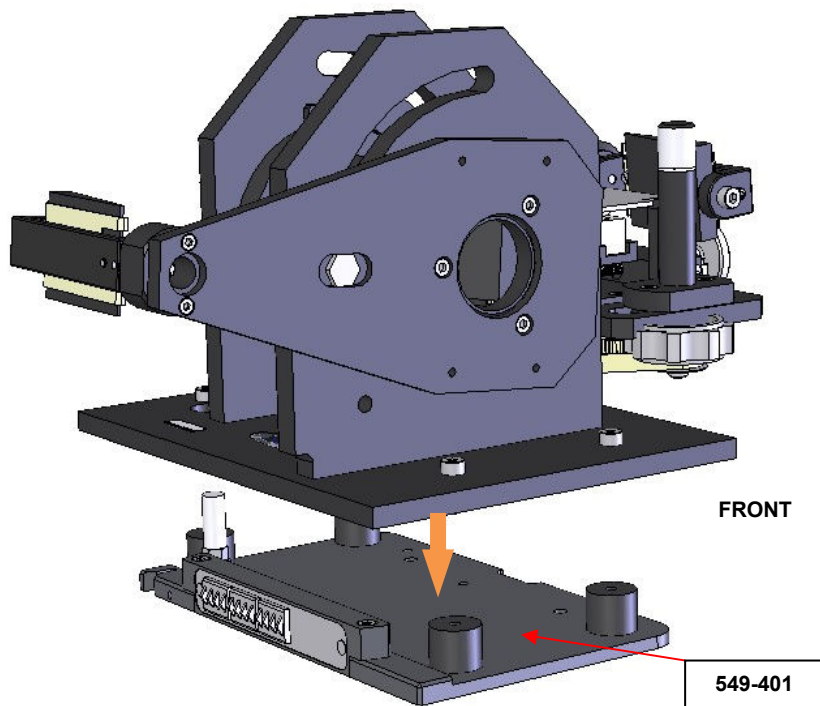
Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Bruker Tensor 27, 37, Quicklock 33 and Vertex 70, 80V

Beam direction: **Right to Left.**

Angle Range: **8° to 85° Fits with compartment lid open. (8° to 60° and 80° to 85° only for Vertex 80V).**

Baseplate installation required – **baseplate P/N 549-401.** (Monolayer accessory base size is unique for fitting to baseplate P/N 549-401).



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

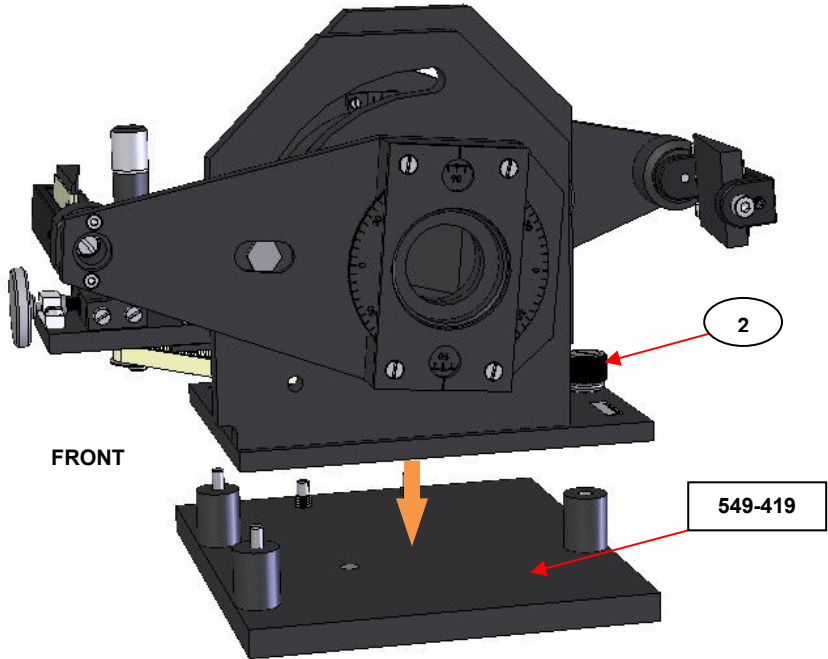
Place the Monolayer Grazing Angle Accessory over the supplied Bruker Quicklock compatible connectivity Monolayer baseplate (as shown in the diagram) and secure the Monolayer Accessory to the baseplate using the four M4 x12mm cap head screws supplied into the support pillars. The whole accessory is orientated correctly in the sample compartment such that the Quicklock baseplate (**549-401**) locates securely by connection to the rear with the electric plug sockets and a push clip at the front.

Bruker IFS113

Beam direction: **Right to Left.**

Angle Range: **8° to 48°.**

Baseplate installation required – **baseplate P/N 549-419.** (Monolayer accessory base size is unique for fitting to baseplate P/N 549-419).



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment.

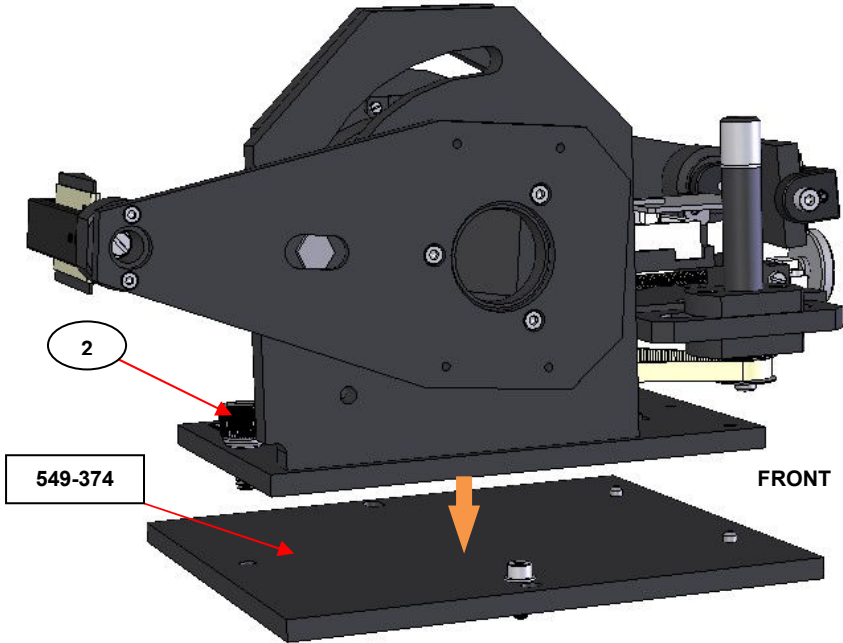
The two underside fixing screws of the accessory baseplate are passed through the larger hole ends of the slots on the Bruker baseplate and the accessory plate is slid from right to left to centralise it on the Bruker plate. The two screws are then turned anticlockwise to tighten the plates together. Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Jasco Instruments 400, 600, 4000, 5000, 7000 Series

Beam direction: **Left to Right.**

Angle Range: **8° to 55° with sample lid compartment open.**

Baseplate installation required – **baseplate P/N 549-374.**



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two location dowels uppermost and to the front of the compartment.

Fix the Monolayer Grazing Angle baseplate to the spectrometer platform with the single M4 x 12mm cap head screw and M4 washer through the smaller of the two holes in the baseplate on the left side.

Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Mattson Galaxy Series 2000, 3000, 4000, 5000, 6000, 7000, RS, Infinity

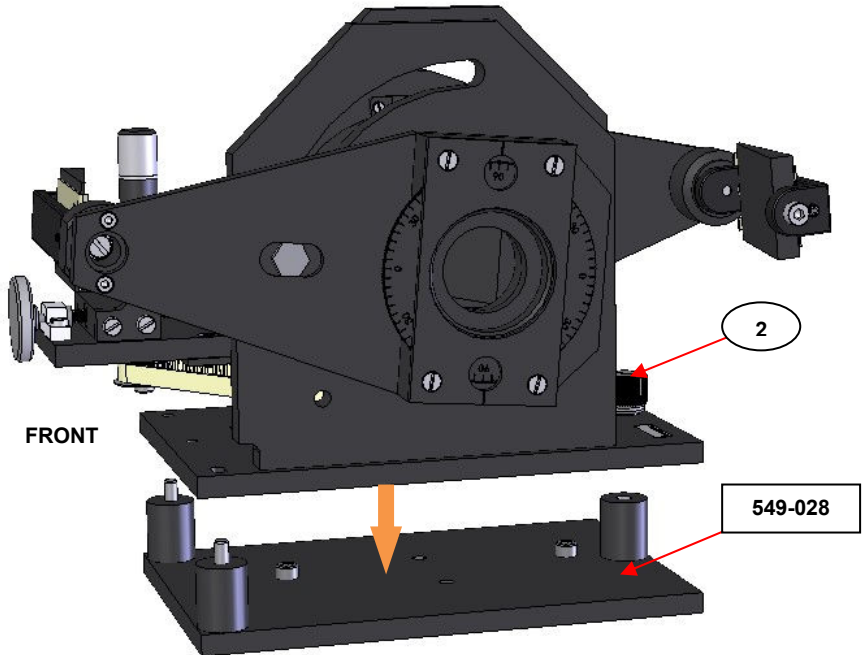
Beam direction: **Right to Left.**

Angle Range: **8° to 65° (3000, 5000, 7000 Series instruments).**

Angle Range: **8° to 75° (2000, 4000, 6000 Series instruments).**

Angle Range: **8° to 85° (RS, Infinity instruments).**

Baseplate installation required – **baseplate P/N 549-028.**



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment and the two dowel fixing pins on the underside of the plate into the floor of the spectrometer.

Fix the Monolayer/Grazing Angle baseplate to the spectrometer platform with the two 8-32UNC x 1/2" cap head screws supplied.

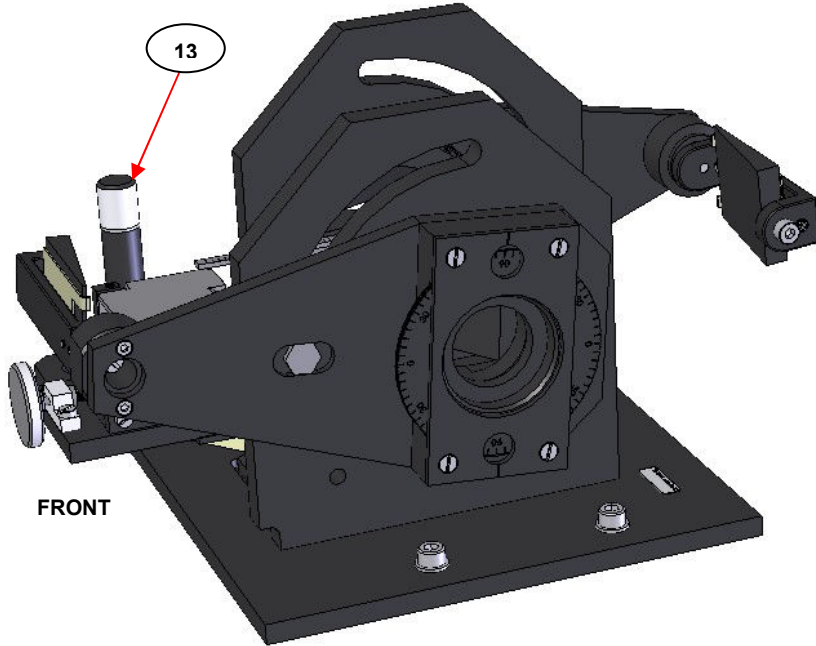
Place the Monolayer/Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Midac (All Midac models)

Beam direction: **Left to Right.**

Angle Range: **8° to 71°.**

No separate monolayer baseplate required for installation.



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

The Monolayer Grazing Angle Accessory is fitted directly to the floor of the spectrometer through two of the four fixing holes in the base by the two 10-32UNC x 1/2" cap head screws supplied. One screw locates through the rear left side hole on the accessory baseplate and the other locates through the front right side hole of the accessory baseplate.

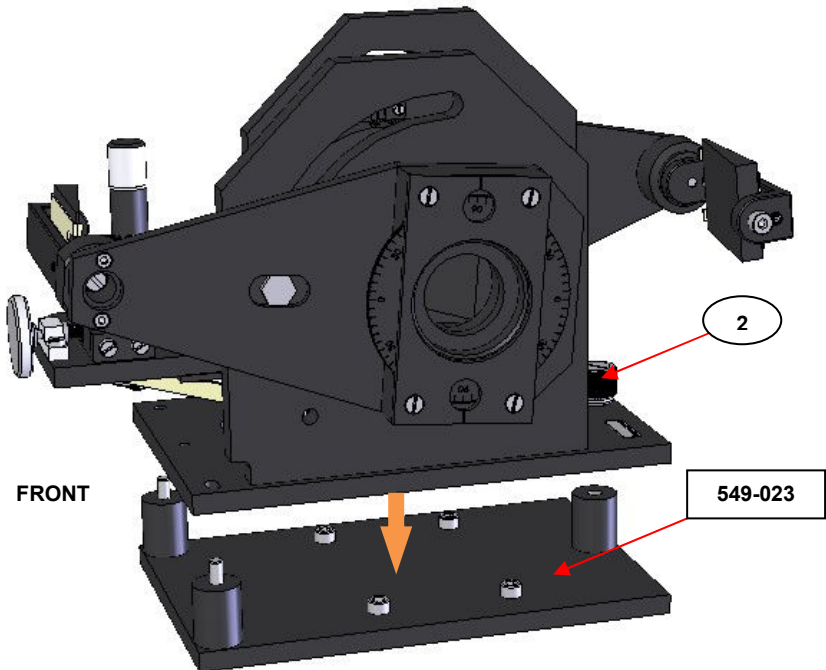
The Monolayer Grazing Angle Accessory is oriented correctly for fit in the sample compartment with the sample platform height adjustment thumb screw post (13) to the front of the spectrometer.

Nicolet 500, 700, 710, 740, 800, 5PC, 5SXC, Magna, Protégé, Avatar, Nexus, 6700, 8700, iS10, iS50

Beam direction: **Right to Left.**

Angle Range: **8° to 85° Fits with compartment lid open.**

Baseplate installation required – **baseplate P/N 549-023.**



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two support pillars and location dowels to the front of the sample compartment. Fix the Monolayer Grazing Angle baseplate to the spectrometer floor platform with the four 8-32UNC x 1/2" cap head screws supplied.

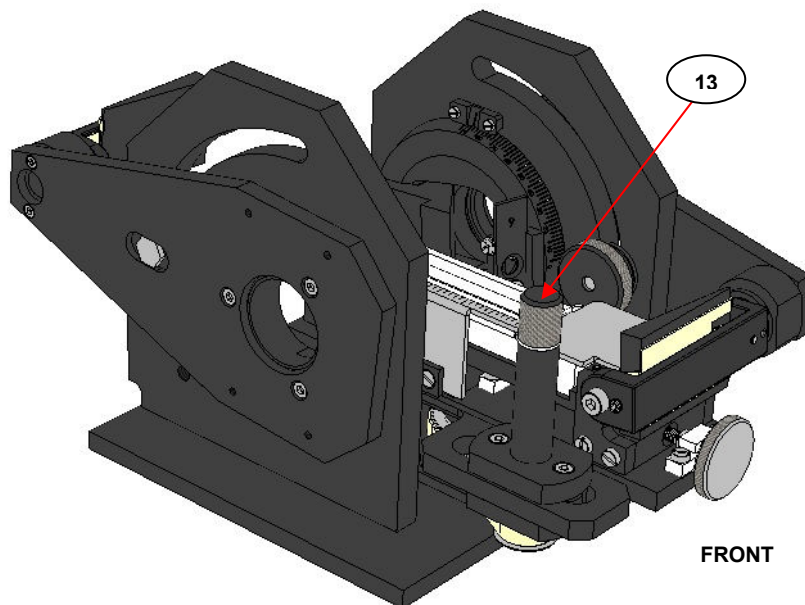
Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Nicolet iS5

Beam direction: **Right to Left.**

Angle Range: **8° to 85°.**

No separate monolayer baseplate required for installation.



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

Align the two small fixing holes on the baseplate of the monolayer accessory with the two location pins on the spectrometer compartment floor. The baseplate is secured into position from "pull down" attraction of the two magnetic discs on the spectrometer compartment floor to the two metallic discs on the underside of the baseplate.

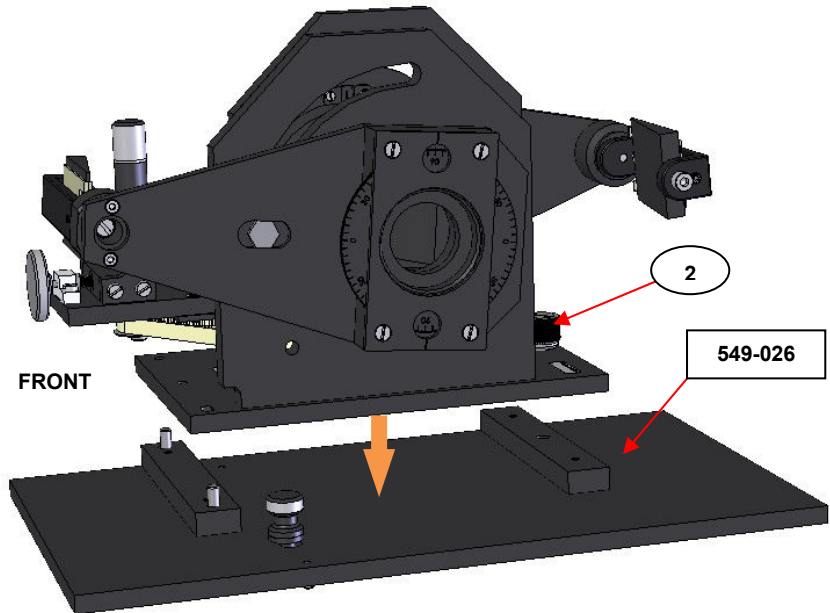
The Monolayer Grazing Angle Accessory is oriented correctly for fit in the sample compartment with the sample platform height adjustment thumb screw post (13) to the front of the spectrometer.

Perkin Elmer PE1700

Beam direction: **Left to Right.**

Angle Range: **8° to 85°.**

Baseplate installation required – **baseplate P/N 549-026.**



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the support strip with location dowels 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.

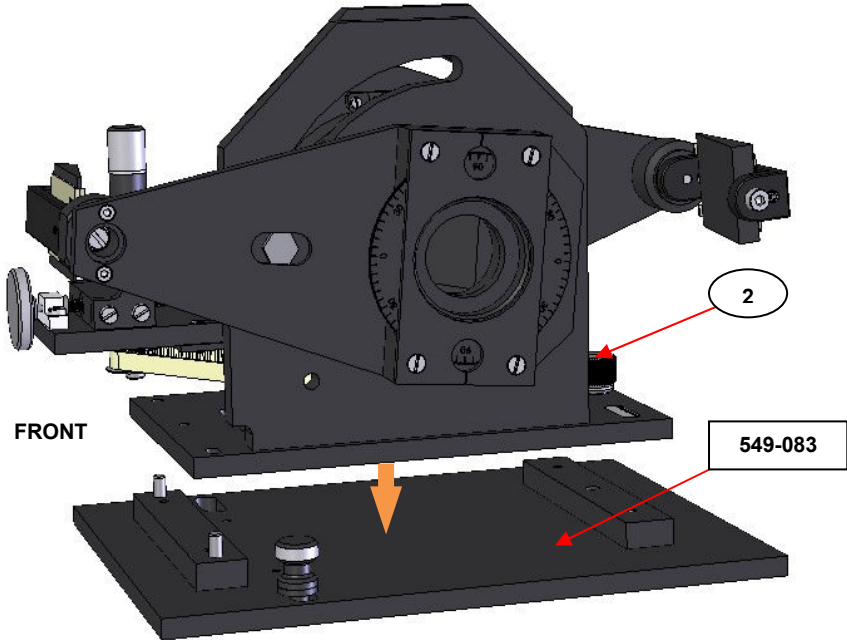
Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb **screw (2)** to the single hole fixing at the rear of the sample compartment.

Perkin Elmer PE2000, Spectrum2000, GX

Beam direction: **Left to Right** or **Right to Left** (Depends on how the spectrometer sample compartment has been fitted).

Angle Range: **8° to 85°**.

Baseplate installation required – **baseplate P/N 549-083**.



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the support strip with location dowels 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.

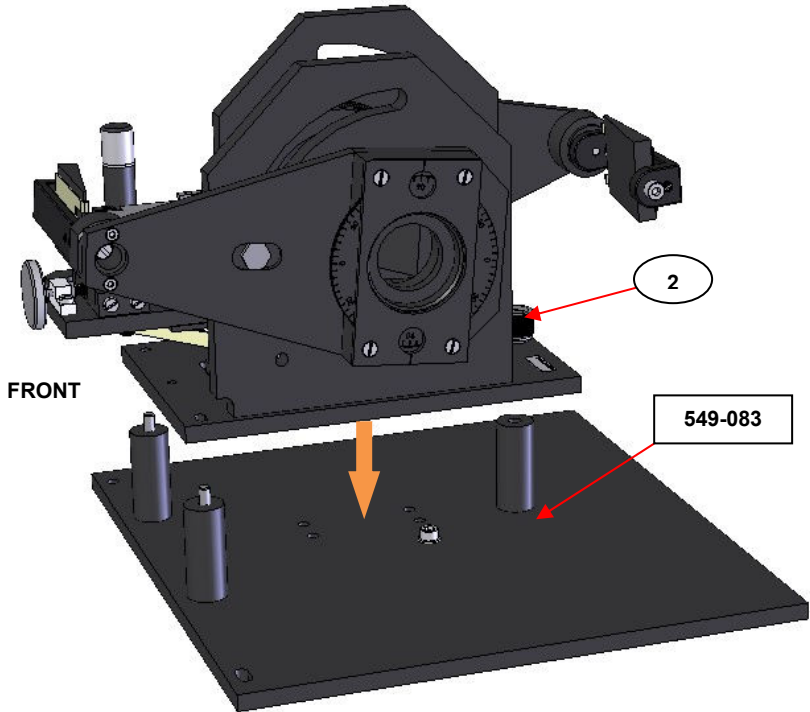
Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Perkin Elmer PE1600 External Bench

Beam direction: **Right to Left.**

Angle Range: **8° to 85° Fits with compartment lid open.**

Baseplate installation required – **baseplate P/N 549-170.**



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

Place the Monolayer Grazing Angle baseplate into the sample compartment with the two support pillars with location dowels to the front of the compartment. Fix the Monolayer Grazing Angle baseplate to the spectrometer platform with the single fixing 8-32UNC x 1" cap head screw supplied.

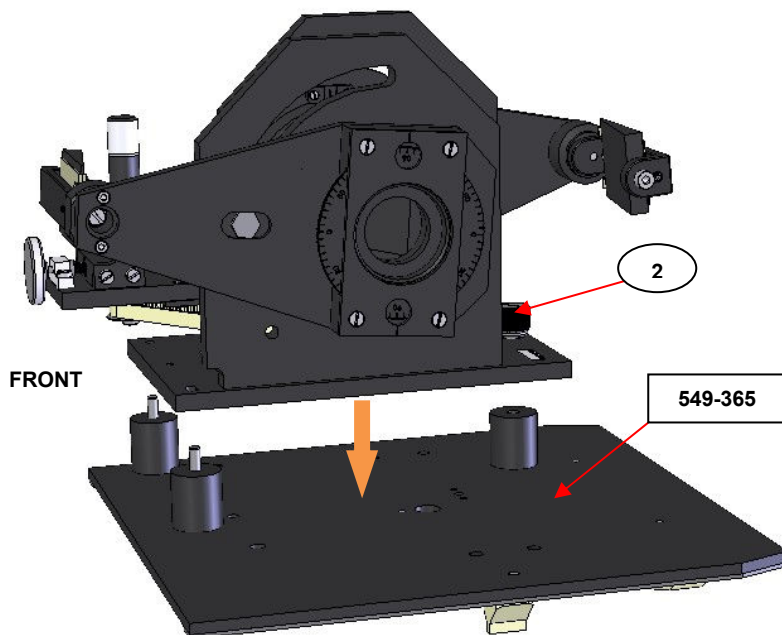
Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Perkin Elmer PE Spectrum One, Spectrum 100, 400, 4000, 8000 (Frontier)

Beam direction: **Left to Right.**

Angle Range: **8° to 85° Fits with compartment lid open.**

Baseplate installation required – **baseplate P/N 549-365.**



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

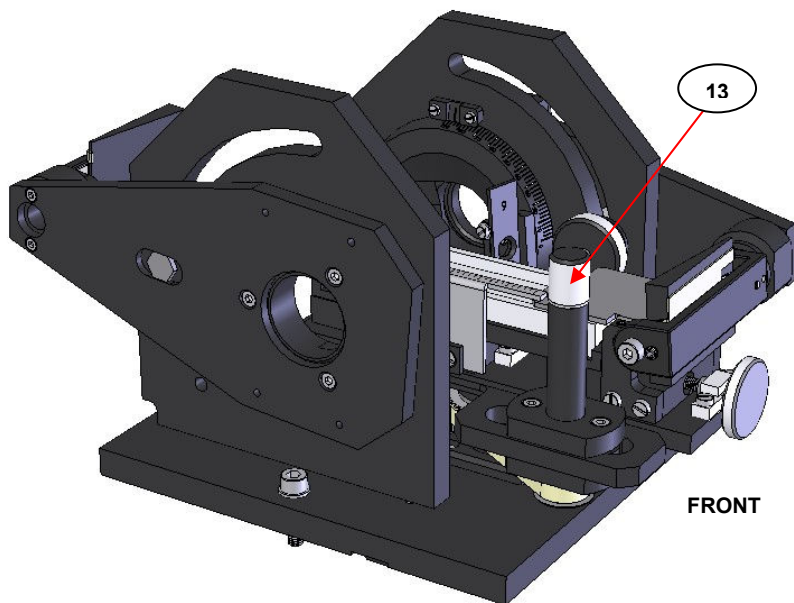
Slide the Monolayer Grazing Angle Accessory baseplate into position along the side edges of the sample compartment with the two support pillars with location dowels to the front of the compartment. The baseplate locates into the spectrometer by engagement of the 15pin connector at the back of the plate 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.

Place the Monolayer Grazing Angle Accessory on the baseplate and secure with the thumb screw (2) to the single hole fixing at the rear of the sample compartment.

Shimadzu 8000, 8100, 8200, 8300, 8500, Prestige 21, IRAffinity, Tracer 100

Beam direction: **Right to Left.**

Angle Range: **8° to 45° (8000 Series, IRAffinity, Tracer 100), 8° to 63° (Prestige 21).** No separate monolayer baseplate required for installation.



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

The Monolayer Grazing Angle Accessory is fitted directly to the floor of the spectrometer by a location strip bar on the underside of the accessories baseplate and a single M5 x 20mm cap head fixing screw through the hole on the left side of the accessory baseplate.

The Monolayer Grazing Angle Accessory is oriented correctly for fit in the sample compartment with the sample platform height adjustment thumb screw post (13) to the front of the spectrometer.

Any Other Spectrometers

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

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email:info@LOT-Oriel.de
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