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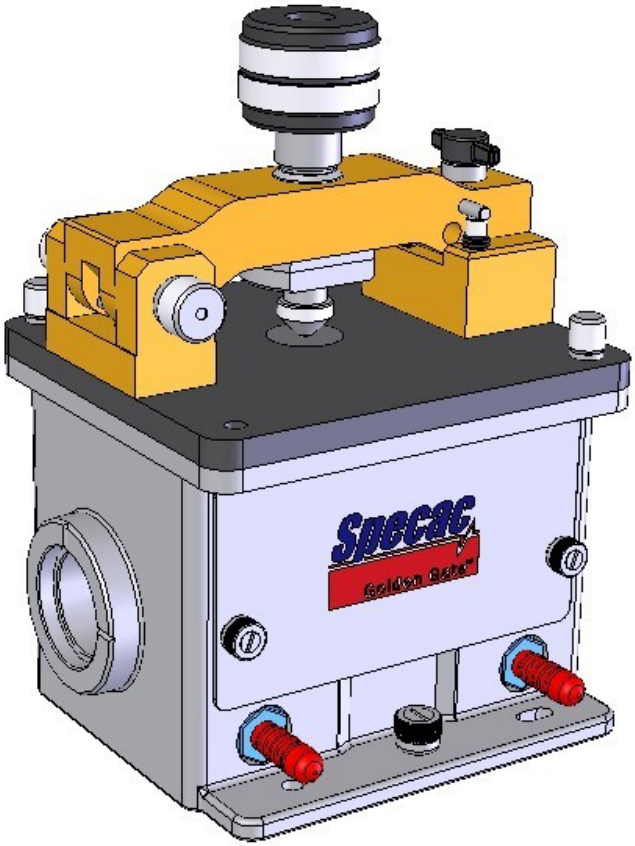
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Golden Gate™ Single Reflection Diamond ATR System

User Manual



2I-10500 Issue 19

Golden Gate™ Single Reflection Diamond ATR System

User Manual

2I-10500 Issue 18

Golden Gate™ Single Reflection Diamond ATR System (GS10500 Series)

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1 Introduction

Thank you for purchasing a Specac Product.

The Golden Gate™ Single Reflection Diamond ATR system (GS10500 Series) is an extremely versatile sampling accessory to allow for many different sampling options in the quantitative and qualitative analysis of solids, liquids, pastes and microsamples by FTIR spectroscopy.

A complete Golden Gate™ ATR accessory system consists of an ATR crystal top plate assembly fixed over an optical beam condensing unit. This complete accessory is mounted into an infrared spectrometer using a Benchmark™ baseplate.

The angular incidence of light at the ATR crystal sample interface is nominally at 45°. Although liquid samples by their nature in general make excellent sample contact to the ATR crystal being used for any spectral analyses, most of the Golden Gate™ ATR top plate options available have a special bridge and clamping mechanism that provides for excellent and reproducible contact between a solid sample and the single reflection ATR crystal. The different top plate options that have a bridge and clamping mechanism can utilise a choice of different compression anvil types to provide the best possible contact and sample containment over the ATR crystal for an extremely diverse range of solid sample forms.

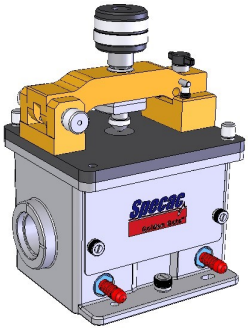
In sampling of solid sample types, when pressure is applied to force the sample into contact against the ATR crystal, any anvil being employed does not rotate against the sample. This ensures that heat is not generated by friction in the force application, as heat could change the sample. It also ensures that the sample is not displaced from the correct sampling position for consistent measurement.

The optical unit of the Golden Gate™ ATR consists of a combination of mirrors and beam condensing lenses designed to achieve optimal infrared radiant energy throughput. The beam condensing lenses used in the optical unit are a choice of ZnSe or KRS-5 material. These lenses are distinctly separate from the single crystal diamond ATR

crystal that is used in the top plate assembly (in all but the germanium crystal top plate option), where the sample to be studied makes contact. Use of a specific set of lenses in the optical unit determines the spectral throughput range achievable for the Golden Gate™ ATR accessory, but the structural and chemical integrity of the ATR crystal is maintained. KRS-5 lenses allow for a slightly wider spectral transmission range for Mid IR studies to circa 350cm-1 rather than ZnSe lenses which fully absorb at circa 550cm-1. However, an anti-reflection coating on ZnSe lenses allows for a better overall light energy throughput than the uncoated KRS-5 lens options, over the typical “fingerprint” region of the IR spectrum.

The choice of top plates available to be mounted on an optical unit for a Golden Gate™ accessory system includes:

- Diamond crystal top plate P/N GS10563.
- Germanium crystal top plate P/N GS10566.
- Heated diamond crystal top plate P/N GS10540.
- High temperature diamond crystal top plate P/N GS10640.
- Wire holder diamond crystal top plate P/N GS10565.
- Microspecular reflectance top plate P/N GS10514.
- Reaction cell diamond crystal top plate P/N GS10507.
- Low temperature diamond crystal top plate P/N GS10590.
- Supercritical fluid diamond crystal top plate P/N GS10585.



Golden Gate Accessory GS10500 Series

2. Safety Considerations



With use of any spectroscopic accessory that involves 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 is possible you should follow a procedure for safe handling and containment of the type of sample to be used.

With respect to safety in operation specifically for the Golden Gate™ ATR accessory, a single crystal daimond or germanium crystal material for the ATR crystal is used in the top plate assembly where a sample makes contact. There is a choice of either ZnSe or KRS-5 material for the lens assemblies in the optical unit.

Out of these four different crystal material types, KRS-5 and then ZnSe are the most potentially hazardous materials with respect to a toxicity risk in use and handling. Both diamond and germanium crystal materials can be considered relatively safe to use, although germanium may be harmful to the body if it is ingested in significant quantity. The general rule when working with **any** crystal material (and sample) is to always wear gloves and safety gear (e.g. safety spectacles) when handling to obviate the risk of contact with the skin. Specifically in operation of a Golden Gate™ ATR accessory there is a minimal risk associated with contact to the KRS-5 or ZnSe materials used for the lens assemblies as these are safely contained within the optical unit and should not require being handled after an initial alignment and optimum throughput has been obtained at installation.

Crystal material safety data sheet information for each of the material types can be consulted for safe handling. A copy of each of these datasheets can be found in this instruction manual in the **Notes On Cleaning** Section found on pages 50 to 53.

3. Unpacking and Checklist

The configuration of Golden Gate™ ATR accessory ordered from a specific part number will determine the items to check on delivery. Please refer to Section 4 of this manual for the specific top plate option and additional items that are supplied for the accessory combination.

All Golden Gate™ ATR accessory systems will include the following:

- 1 Golden Gate™ optical unit with beam condensing lens assemblies (ZnSe or KRS-5 lens material options).
- 1 Benchmark™ baseplate for your model of FTIR spectrometer.
- 1 Ball driver 3.0 mm.
- 1 Allen key 2.0 mm.
- 1 Purge bellows (GS10707).
- 1 Essential Spares Kit for the particular Golden Gate™ ATR accessory.
- 1 Quick Start Guide for the Golden Gate™ ATR accessory.
- 1 Benchmark™ baseplate installation guide instruction manual.

Carefully lift your Golden Gate™ top plate and optical unit from the carry case and place onto a flat bench. Installation of the top plate (**1**) is achieved by positioning it on to the optical unit (**2**) and securing by turning the two thumb screws (**3**) clockwise.

Important: *The top plate must be fitted the correct way around. When fitted correctly the sides of the top plate will be flush with the sides of the optical transfer unit.*

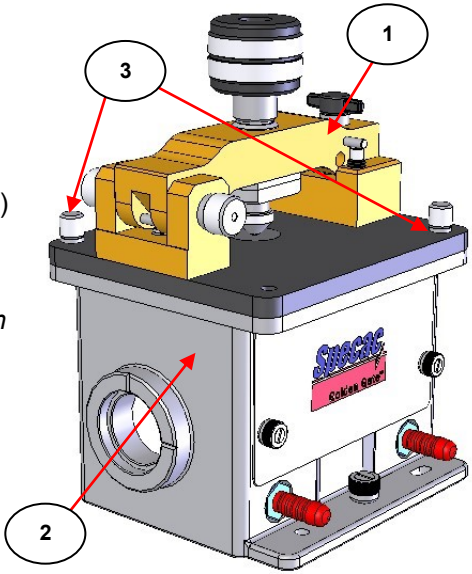


Fig 1. Fitting of Golden Gate™ Top Plate to Optical Unit

4. Golden Gate™ ATR System Top Plates

There are a variety of top plates for the Golden Gate™ Single Reflection ATR system. The top plates can be ordered as part of a complete Golden Gate™ ATR accessory, or separately as upgrades.

Diamond Crystal ATR Top Plate (P/N GS10563) from P/N's GS10500-Z or GS10500-K Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ Diamond ATR Top Plate (GS10563).
- 1 Sapphire anvil (GS10531).
- 1 Pellet anvil (GS10532).
- 1 Volatiles cover (GS10503).
- 1 Essential Spares Kit of parts (GS10550). (When ordered as accessory).

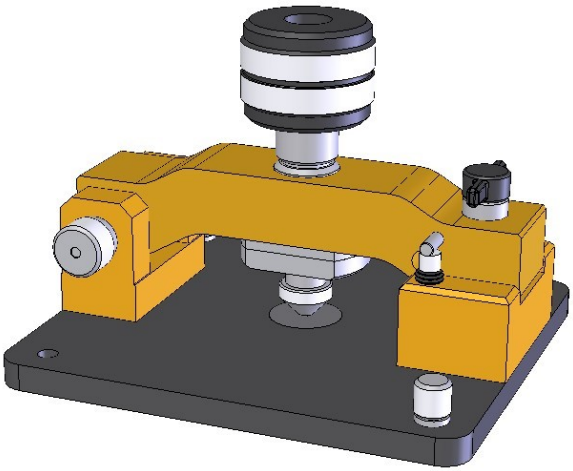


Fig 2. Golden Gate™ Diamond ATR Top Plate GS10563

Germanium Crystal ATR Top Plate (GS10566) from P/N GS10516 Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ Germanium ATR Top Plate (GS10566).
- 1 large stainless steel anvil (GS10567).
- 1 Volatiles cover (GS10503).
- 1 Essential Spares Kit of parts (GS10550). (When ordered as accessory).

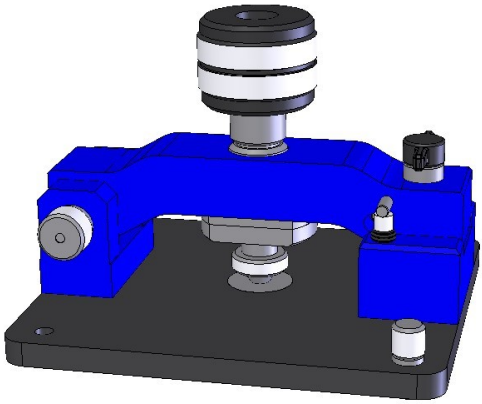


Fig 3. Golden Gate™ Germanium ATR Top Plate GS10565

Important! - Note for Use of Germanium Crystal

The germanium crystal does not have the same hardness and chemical resistance as a diamond and should be treated accordingly. Be careful not to put a point loading on the crystal, particularly with hard or abrasive samples. With samples such as rubber and soft polymers covering the crystal, normal anvil pressures may be used.

Check on the chemical resistance before potentially damaging materials are brought into contact with the crystal. Thin films on plastic substrates can be analysed, but take care if the substrate is metallic. Test for suitability by applying pressure gradually from the clamp anvil mechanism, taking scans to see if the spectra are acceptable.

Heated Diamond ATR Top Plate (GS10540) from P/N's GS10542-Z or GS10542-K Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ Heated Diamond ATR Top Plate (GS10540) to 200°C.
- 1 4000 Series Temperature Controller for the heated top plate.
- 1 Sapphire anvil (GS10531).
- 1 Pellet anvil (GS10532).
- 1 Volatiles cover (GS10503).
- 1 Essential Spares Kit of parts (GS10550). (When ordered as accessory).
- 1 Instruction manual for 4000 Series Temperature Controller operation.

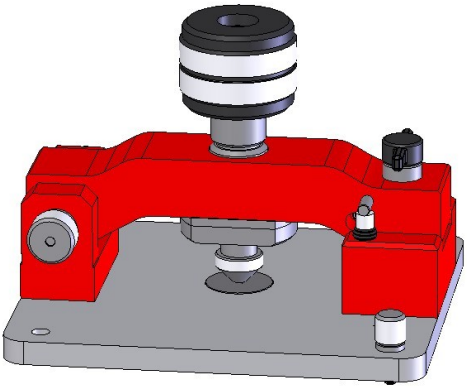


Fig 4. Golden Gate™ Heated Diamond ATR Top Plate GS10540 (to 200°C)

Warning! *The diamond ATR crystal and tungsten carbide support puck in the heated top plate GS10540 has a very small air gap around its circumference edge. This minimizes heat loss to the surrounding top plate and ensures a quick warm up. However, if excessive amounts of solvent are used on the diamond crystal, it is possible for the liquid (or vapour) to leak past the puck into the optical unit below, giving unwanted solvent absorption bands in any experimentation. For this reason, the diamond should only be covered with a minimum of liquid sample or cleaned using a tissue moistened with solvent.*

High Temperature Diamond ATR Top Plate (GS10640) from P/N's GS10642-Z or GS10642-K Accessory Order

Please check on delivery that the following have been included:

- 1 High Temperature Diamond ATR Top Plate (GS10640) to 300°C.
- 1 4000 Series Temperature Controller for the heated top plate.
- 1 Sapphire anvil (GS10531).
- 1 Pellet anvil (GS10532).
- 1 Volatiles cover (GS10503).
- 1 Essential Spares Kit of parts (GS10550). (When ordered as accessory).
- 1 Instruction manual for 4000 Series Temperature Controller operation.

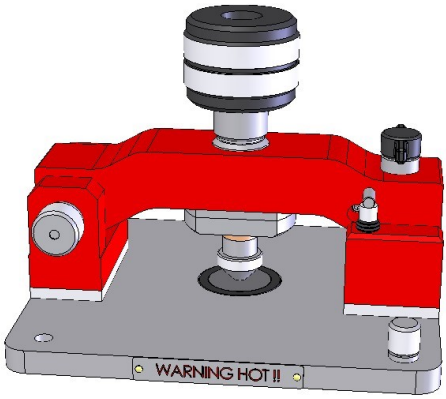


Fig 5. Golden Gate™ High Temperature Diamond ATR Top Plate GS10640 (to 300°C)

Note: *The diamond ATR crystal and tungsten carbide support puck in the high temperature top plate GS10640* ***does not*** *have a small air gap around its circumference edge. The tungsten carbide material is sealed around its circumference edge to PEEK material as a circular barrier which acts as both a thermal insulator and a protection from the ingress of liquid samples and any volatile solvent vapours into the optical unit below. Nevertheless, the diamond should still be cleaned using a tissue moistened with a minimum amount of solvent.*

Microspecular Reflectance Top Plate (GS10514) from P/N's GS10523-Z or GS10523-K Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ microspecular reflectance top plate (GS10514).
- 1 Reference mirror.
- 1 Essential Spares Kit of parts (GS10527). (When ordered as accessory).

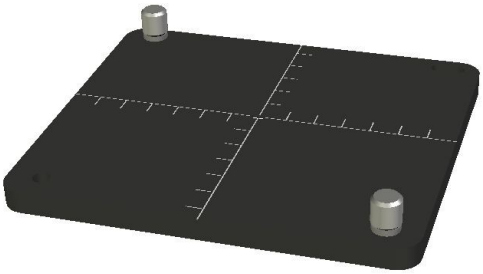


Fig 6. Golden Gate™ Microspecular Reflectance Top Plate GS10514

The microspecular reflectance top plate can be fitted to the Golden Gate™ optical unit to provide a single reflection 45° angle specular reflectance measurement of small solid sample types. The top plate slot aperture is 5mm x 2.5mm and so any sample must be larger than these dimensions when placed over the slot aperture for spectral analysis to prevent the sample from falling into the optical unit below.

Reaction Cell Diamond ATR Top Plate (GS10507) from P/N's GS10525-Z or GS10525-K Accessory Order

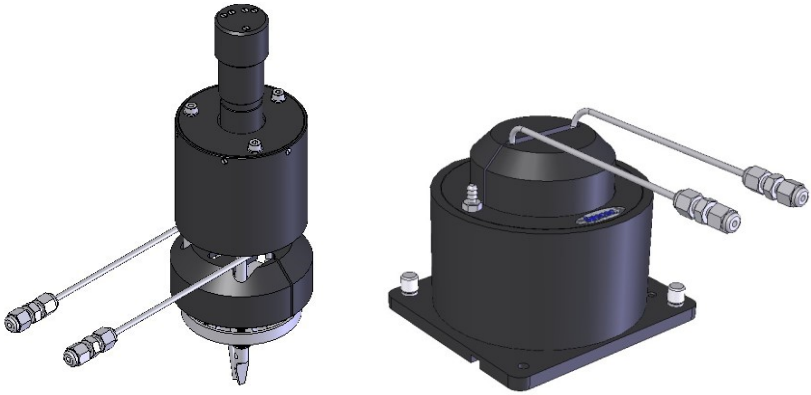
This is a "specialist" Golden Gate™ top plate that incorporates a 45° angle diamond crystal puck at the base of a 24mls volume reaction chamber. It is capable of operating at temperatures up to 200°C and at up to 3000 psi pressure.

A specific temperature controlling system and relevant instruction manuals are provided for this Golden Gate™ top plate option for explanation of operation. A dedicated stirring mechanism (GS10513)

can also be used as an alternative top pressure plate assembly with the Reaction Cell Diamond ATR top plate option if this is ordered.

Please check on delivery that the following have been included:

- 1 Golden Gate™ Reaction Cell Diamond ATR Top Plate (GS10507).
- 1 4000 Series Temperature Controller for the reaction cell top plate.
- 1 Torque Wrench (GS10504).
- 1 Square/Hexagon Drive Adapter.
- 2 Open Ended Spanner 7/16 inch and 3/8 inch AF.
- 1 3.0mm AF Hexagon Head Bit.
- 1 6.0mm AF Hexagon Head Bit (Ball End).
- 1 Allen Key 2mm AF Long Arm.
- 1 3.0mm AF Hexagon Ball Driver.
- 1 Essential Spares Kit of parts (GS10528). (When ordered as accessory).



Stirring Mechanism **Reaction Cell Top Plate**

Fig 7. Reaction Cell Top Plate GS10507 and Pressure Cap Stirring Mechanism GS10513

Low Temperature Diamond 45° ATR Top Plate (GS10590) from P/N's GS10592-Z or GS10592-K Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ Low Temperature Diamond ATR Top Plate (GS10590).
- 1 4000 Series Temperature Controller for the Low Temperature Diamond ATR Top Plate.
- 1 Sapphire tipped solids anvil.
- 1 Liquid sample injector (insulated hypodermic needle and glass syringe).
- 1 Plastic funnel.
- 1 Long T-bar Allen key (5 mm).
- 1 Allen Key (3 mm).
- 1 Glass filled PTFE gaskets (5).
- 1 High thermal transfer gaskets (20).
- 1 Spare Viton O-ring.
- 1 User manual for Low Temperature Golden Gate™ top plate operation.
- 1 Essential Spares Kit of parts (GS10550). (When ordered as accessory).

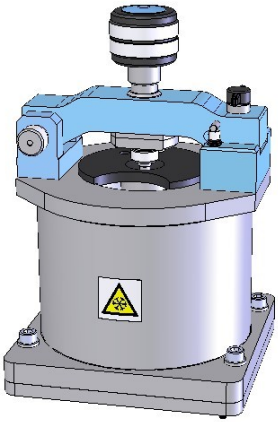


Fig 8. Golden Gate™ Low Temperature ATR Top Plate GS10590

The Golden Gate™ Low Temperature Diamond ATR Top Plate operates over a temperature range of -150°C to + 80°C.

Supercritical Fluid (SCF) Diamond ATR Top Plate (GS10585) from P/N's GS10586-Z or GS10586-K Accessory Order

Please check on delivery that the following have been included:

- 1 Golden Gate™ SCF Diamond ATR Top Plate (GS10585).
- 1 4000 Series Temperature Controller for the SCF Diamond ATR Top Plate.
- 2 Open Ended Spanner 1/4" – 5/16".
- 1 Torque wrench (GS10504).
- 1 Socket drive 2.5 mm A/F.
- 1 Graphite pressure gaskets (10 in packet).
- 1 User manual for the SCF Diamond ATR top plate operation.
- 1 Essential Spares Kit of parts (GS10529). (When ordered as accessory).

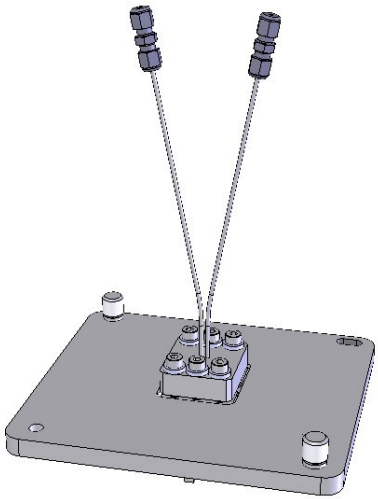


Fig 9. Golden Gate™ Supercritical Fluid ATR Top Plate GS10585

The Golden Gate™ SCF Diamond ATR Top Plate is specified to operate at temperatures up to 300°C and pressures up to 6000 psi.

5. Installation into a Spectrometer

The Golden Gate™ Single Reflection ATR accessory is supported on a Benchmark™ baseplate when installed into a spectrometer. The baseplate has three support pillars (one flat at the back and two with location pins at the front) and a fourth front central pillar into which the fixing thumb screw (**4**) on the optical unit (**2**) is tightened. (See **Fig 10.**)

Note: *It is usually preferable to install the Benchmark™ baseplate in the spectrometer first, before locating the Golden Gate™ ATR accessory.*

Fixing pillars, plates and studs in the Benchmark™ baseplate will vary dependant on the make and model of your spectrometer. For details on how to install your Benchmark™ baseplate and Golden Gate™ ATR accessory into the spectrometer, refer to the Benchmark™ baseplate installation guide instruction manual as supplied.

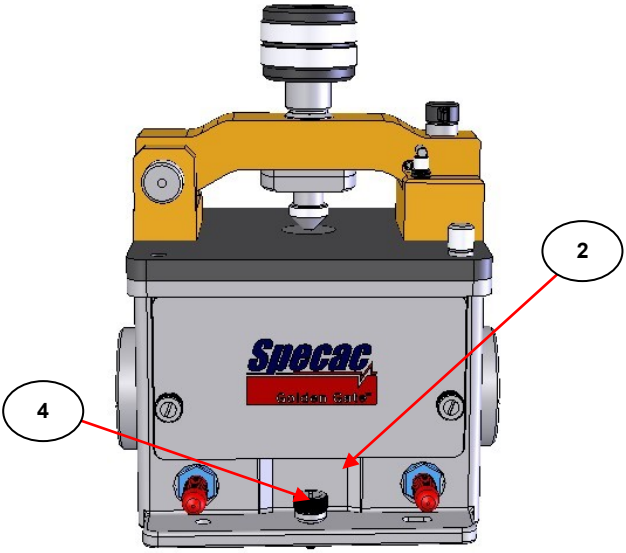


Fig 10. Fixing Thumbscrew on Golden Gate™ Optical Unit.

6. Alignment of Golden Gate™ ATR Accessory

When you have installed the Golden Gate™ Single Reflection ATR accessory into your spectrometer please check the alignment of the accessory. The Golden Gate™ ATR Accessory will be preliminarily aligned when first supplied from Specac. On a local installation for the specific spectrometer to be used, some transmitted energy will be recorded by the spectrometer, but for best results adjusting the accessory optics will maximize the optical throughput.

Note: *The energy (optical) throughput of the Golden Gate ATR accessory when installed varies with the spectrometer design and optical settings It should usually be possible to achieve at least 6% transmission at 1000 cm^{-1} when a spectrum is recorded of the ATR accessory against an open beam background It is important on installation of any accessory into a spectrometer that a maximum throughput is achieved to ensure an optimum analytical performance and signal-to-noise ratio relationship.*

The Golden Gate™ optical unit (**2**) contains six optical components. Two mirrors and one lens make up the input system and an identical set of mirrors and lens form the output system (see **Fig 11**.). The two mirrors (**M1** and **M4**) are fixed while the two mirrors (**M2** and **M3**) have rotation and tilt adjustments. The focusing lenses (**L1** and **L2**) are adjustable for their focus by sliding them within their support mounts. The simplified diagram at **Fig 12**. shows the positioning of these optical components within the optical unit (**2**). A diamond ATR top plate (from GS10563) is shown in position representing the **sample** to complete a beam path. The procedure for alignment of the accessory is the same with use of any other ATR top plate (**sample**) in position on the optical unit, except for the microspecular reflectance top plate GS10514, which requires its own reference mirror to be placed over the sampling aperture slot. In any spectrometer system, the source of light that passes to the detector may be either from a left to right (**L** to **R**) or right to left (**R** to **L**) beam direction through the sample compartment when viewed from above or at the front of the spectrometer system. Hence, for any alignment procedure to be made on the Golden Gate™ ATR

accessory it always best to start with the adjustable **output** mirror in the optical unit (**2**) that is nearest to the detector.

Note: ***Fig 11.*** *shows the optical components for a beam passage in sequence from* ***M1*** *-* ***M2*** *-* ***L1*** *–* ***Sample*** *-* ***L2*** *-* ***M3*** *–* ***M4*** *for an* ***L*** *to* ***R*** *beam spectrometer and* ***M3*** *is the adjustable* ***output*** *mirror. For an* ***R*** *to* ***L*** *beam spectrometer, the beam sequence is reversed and* ***M2*** *becomes the adjustable* ***output*** *mirror.*

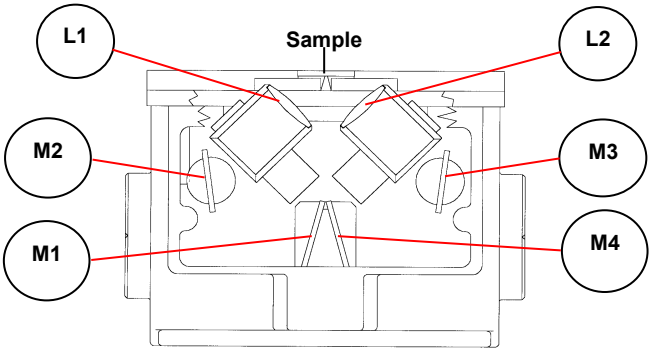


Fig 11. Optical Components in the Golden Gate™ Optical Unit Related to the Beam Passage for L to R or R to L Direction Spectrometers

Alignment of the Golden Gate™ ATR Accessory

The following alignment procedure has been written for the Golden Gate™ ATR accessory when installed into an **L** to **R** beam direction spectrometer. If the Golden Gate™ ATR accessory is installed into a **R** to **L** beam direction spectrometer the input and output mirrors and lens positions are reversed. Prior to installation of the accessory into the spectrometer, set the spectrometer system to read an energy value to measure as a guide during the alignment procedure. An energy level (throughput count number value) or a voltage peak to peak signal level for an unobstructed beam can be used as the reference to compare.

With the Golden Gate™ ATR accessory installed into the sample compartment of the spectrometer, loosen the two thumb screws (**5**)

and remove the front cover (**6**) on the optical unit (**2**) to reveal the optical components for their adjustment (See **Figs 12** and **13**).

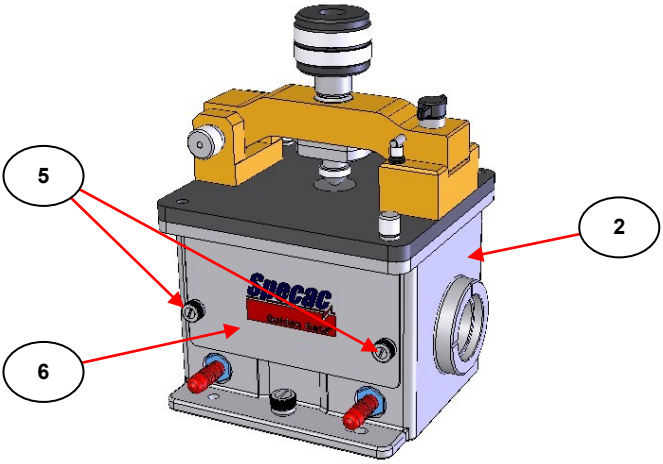
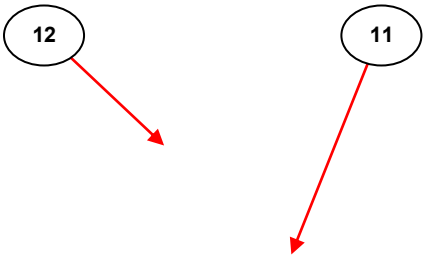


Fig 12. Optics Cover Removal from Golden Gate™ ATR Accessory

Note: The diamond top plate of the Golden Gate™ ATR accessory has also been removed from **Fig 13.** for clarity.



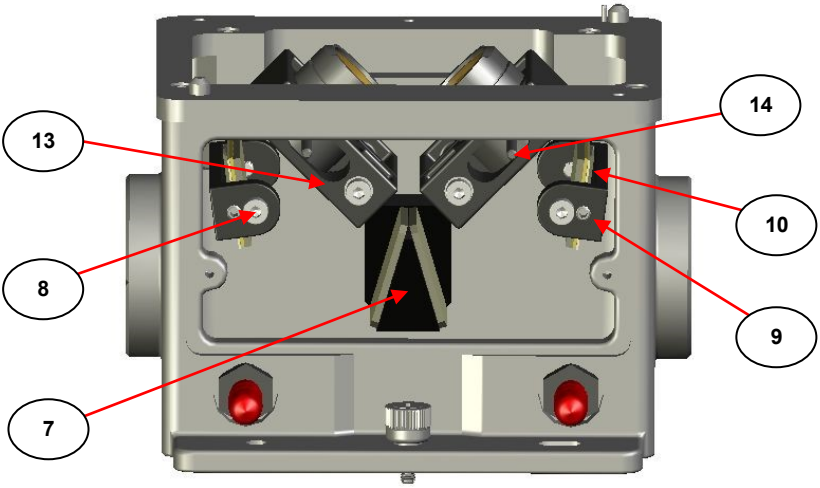


Fig 13. View of Optical Components in the Golden Gate™ ATR System

Usually, from installation of the Benchmark™ baseplate into the sample compartment it remains in a fixed position. The alignment procedure begins from this fixed positioning of the accessory in the sample compartment. However, where a spectrometer system allows the installed Golden Gate™ ATR accessory to be moved either left to right in the sample compartment when fixed to the Benchmark™ baseplate, move the entire assembly of accessory affixed to the baseplate to find the best position where the throughput transmission energy (as indicated by the spectrometer systems energy monitor guide chosen) is at a maximum and firmly secure the baseplate. In some spectrometers it may be necessary to remove the optical unit (**2**) from the Benchmark™ baseplate (loosen fixing thumb screw (**4**)) before securing the baseplate. Where this is the case, ensure that the baseplate does not move when removing the optical unit (**2**).

1. The double mirror mount (**7**) is fixed. The **input** side mirror (**M1**) ensures that the light beam from a source is correctly directed to the adjustable **input** mirror (**M2**). (Similarly mirror (**M4**) is fixed and sends the light beam correctly to the adjustable mirror (**M3**) if these are the **input** optics.)

2. Start by using the 3mm ball driver supplied in the screw (**8**) of the **adjustable output mirror** (**M3**) to very gently adjust the **rotational** movement of the mirror to achieve a maximum energy reading.

Tip: *If the energy reading on the instrument monitor goes to zero while making any adjustment to an optical component, always bring the energy throughput back to a readable value adjusting **the same optical component** before proceeding with adjustment to another.*

3. Next, adjust the mirror (**M3**) **tilt** position by inserting the smaller 2mm size Allen key supplied into the grub screw (**9**) and turning it to achieve a maximum energy reading.

Note: *If the grub screw (**9**) is turned too far anticlockwise it will dislocate from the back of the spring mount of the mirror (**10**). If this happens, turn the grub screw (**9**) anticlockwise to clear fully from the spring mount. Push the spring mounted mirror (**10**) away from the black anodized mirror mount arm and turn the grub screw (**9**) clockwise until it re-sets again behind the mount.*

4. When the mirror (**M3**) has been adjusted for it optimum **rotation** and **tilt** settings, the lens focus on the **output** side is adjusted by slightly loosening the locking screw (**11**) that holds the lens (**L2**) barrel assembly (**12**). A spring washer behind the screw (**11**) ensures there is some friction to hold the lens barrel assembly (**12**) into the outer lens mount (**13**). Hold on to the lens barrel assembly (**12**) by the tiller bar (**14**) whilst loosening the locking screw (**11**) with the 3mm ball driver supplied to prevent the lens barrel (**12**) from sliding down within its mount (**13**). Wriggle the tiller bar (**14**) gently to slide the lens barrel assembly (**12**) up or down in its mount (**13**) between the limits of its travel (tiller bar (**14**) reaching an end stop in the mount slot) to maximize the energy. An optimum throughput is normally achieved when the tiller bar (**14**) is positioned centrally, equidistant from either of its end stop travel positions in the mount (**13**) slot.

5. When lens (**L2**) is in its optimum throughput position, tighten the locking screw (**11**) to secure the lens barrel assembly (**12**) in the lens mount (**13**).

6. Now, maximize the energy throughput on the **input** adjustable mirror (**M2**) by adjusting it for **rotation** and **tilt** in the same way as completed for mirror (**M3**) (see procedure steps 2 and 3).

7. Complete the alignment procedure by adjusting the focus of the **input** lens (**L1**), in the same way as completed for lens (**L2**) (see procedure steps 4 and 5).

8. It may be necessary to repeat the **rotation** and **tilt** adjustments slightly for the output mirror (**M3**) again if any adjustments to the input optical components - mirror (**M2**) and lens (**L1**) - markedly altered the maximum throughput energy achievable, to “rebalance” the system. If there has been no great improvement in optimum throughput from adjustment of the **input** optical components, then the alignment procedure can be considered complete and the front cover plate (**6**) can be placed back onto the optical unit (**2**).

7. Fitting a Polarizer and Purging the Golden Gate™ ATR Accessory

When the Golden Gate™ ATR accessory has been installed and aligned in the spectrometer you have the options of fitting a polarizer and/or purging the ATR accessory.

Fitting a Polarizer

The FTIR Infrared polarizers (GS12000 Series) from Specac can be fitted to the aperture ports (**15**) of the Golden Gate™ ATR optical unit (**2**). (See **Fig 14**.) The polarizers push fit into the aperture port openings and can be rotated to the desired orientation by hand. The polarizer will transmit at a maximum (perpendicular component of the polarized light) when it is mounted such that the metal tab slot in the polarizer ring mount is in the vertical plane. Fitting the polarizer to either aperture port (**15**) of the Golden Gate™ ATR accessory will have the same polarizing effect for measurement of a sample.

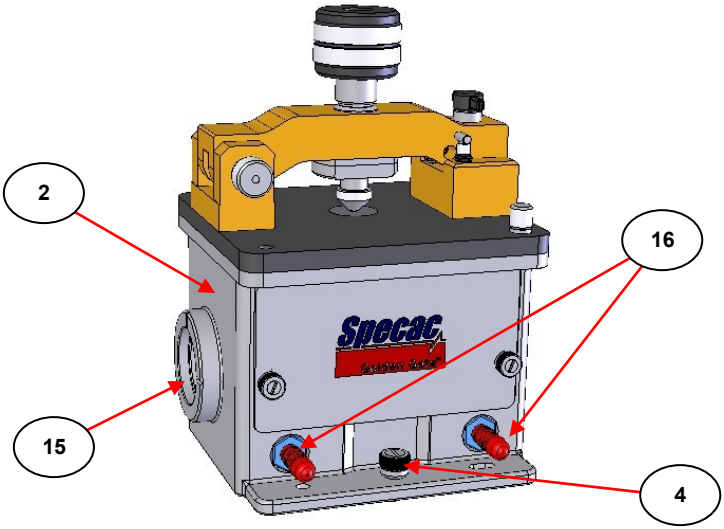


Fig 14. Aperture and Purge Ports of the Golden Gate™ ATR System

There is also an alternative to use of a GS12000 Series polarizer to allow for polarized light measurements. The GS12510 Series Benchmark™ Rotator Mount for 38mm diameter clear aperture GS57010 Series polarizers fits directly over the aperture ports (**15**) of all Benchmark™ baseplate compatible optical units such as that found on the Golden Gate™ ATR accessory. This rotator mount allows for an easier setting of a polarized grid plane of light than that required in use of a GS12000 Series polarizer. (Please ask Specac for details).

Purging the System

The Golden Gate™ ATR optical unit (**2**) is fitted with purge ports (**16**). (See **Fig 14.**) The protective rubber sealing covers are removed from the purge ports (**16**) and ¼" O.D silicone tubing can be connected to the barbed hose connections to fill the optical unit (**2**) with a suitable purge gas such as Nitrogen. To allow the system to be purged efficiently it is necessary to fit the flexible purge bellows (**17**) (GS10707) supplied with the accessory. (See **Fig 15.**)

Note: *It is recommended that purge bellows (**17**) are fitted during use to help stabilize the instrument background, even if the Golden Gate™ ATR accessory is not being purged. It is also necessary to fit a polarizer, if required, before any purging of the system.*

Fitting the Purge Bellows (GS10707)

The purge bellows (**17**) push fit at their "flattened, tube ends" over the outside diameter circumference of the aperture ports (**15**) of the Golden Gate™ optical unit (**2**) and bridge any air gap between the optical unit and the side walls of the sample compartment of a spectrometer instrument. As supplied the purge bellows (**17**) may be of an adequate length for a good fit, but if the gap between the optical unit (**2**) and side walls of the sample compartment are small then the purge bellows (**17**) can be cut for a better fit.

1. With the Benchmark™ base-plate secured, and Golden Gate™ ATR accessory installed, measure the approximate lengths between the spectrometer side walls and the flat end of the ATR optical unit (**2**). (This is dimension **'X'** – see **Fig 15.**).

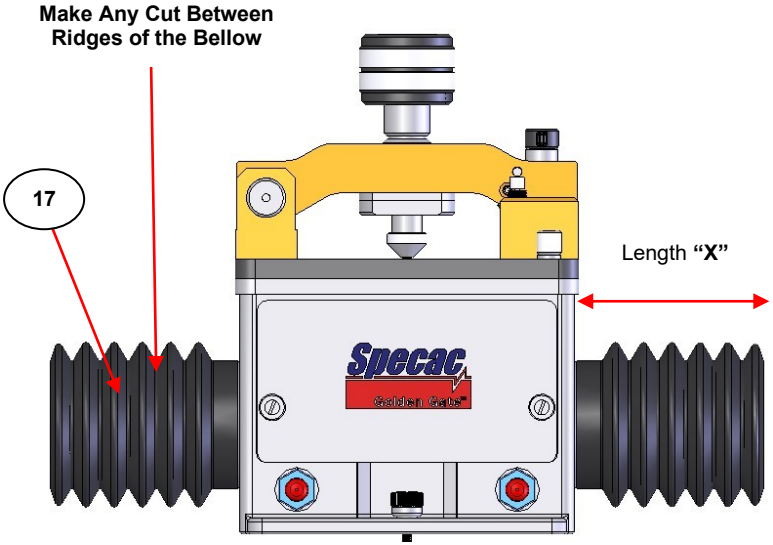


Fig 15. Fitting Purge Bellows to the Golden Gate™ ATR Optical Unit

2. Using a sharp razor, cut lengths of the flexible purge bellow (**17**) tubes which are equivalent to the measured length (**X**) plus an additional 10 mm for each bellow.

Tip: *It is easier to cut the bellow between the hard, edged ridges.*

3. Unscrew the fixing thumb screw (**4**) and remove the Golden Gate™ ATR Accessory from the sample compartment.

Note: *Fit a GS12000 Series polarizer into one of the aperture ports (**15**) and adjust for its correct polarizer grid orientation at this stage if required.*

4. Fit the flexible purge bellows (**17**) over both end apertures as seen at **Fig 15.** and compress sufficiently to enable the whole accessory assembly to fit into the spectrometer.

5. Ensure the bellows are not obstructing the beam and then tighten the accessory fixing thumb screw (**4**) to secure the Golden Gate™ ATR optical unit back onto the Benchmark™ baseplate.

8. Changing Lenses in the Golden Gate™ ATR Optical Unit

The Golden Gate™ ATR accessory is provided with ZnSe or KRS-5 beam condensing lenses as standard in the optical unit (**2**). If you wish to change from one set of lenses to the other they are available as upgrade kits.

GS10552 ZnSe Lens (2) Upgrade Kit.
GS10508 KRS-5 Lens (2) Upgrade Kit.

Removing the Lenses

1. The front cover (**6**) of the optical unit (**2**) is removed by loosening the thumb screws (**5**) to gain access to the optics.

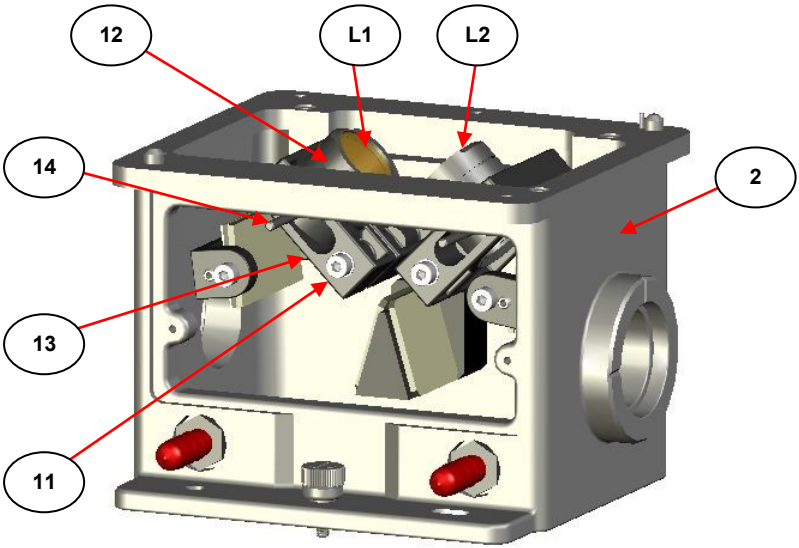


Fig 16. Changing the Lens Assemblies in a Golden Gate™ ATR System

2. Any Golden Gate™ ATR Top Plate being used must be removed from the optical unit (**2**). This is achieved by loosening the two thumb screws (**3**) on the Top Plate and lifting it up and away from the optical unit (**2**). (See **Fig 1**. – page 7).

3. The lens assembly (**12**) in the optical unit (**2**) is adjusted by a tiller bar (**14**) (see **Fig 16**.). Remove the tiller bar (**14**) from its lens barrel assembly (**12**) by unscrewing it anticlockwise.

4. Using gloves, very carefully hold the lens barrel assembly (**12**) at the top (near to the lens material itself) and at the same time loosen the locking screw (**11**) making the lens barrel assembly (**12**) free to move in its outer mount (**13**) casing.

5. Carefully remove both lens assemblies (**12**) by pulling up and out of their mount (**13**) casings, through the top of the optical unit (**2**).

Fitting New Lenses

To fit new lenses the procedure for removal is reversed.

1. Carefully slide the new lens barrel assembly (**12**) the correct way up into its mount (**13**) casing through the top of the Golden Gate™ ATR optical unit (**2**).

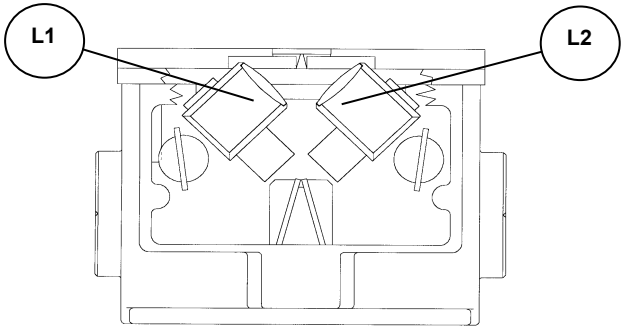


Fig 17. Simplified diagram showing position of lens assemblies L1 and L2 in the Golden Gate™ ATR optical unit. (Note that an ATR Top Plate is also shown in position on top of the optical unit)

Important: *For a correct insertion of the new lens barrel assemblies (**12**), ensure the tiller bar (**14**) screw holes are aligned with the front slots in the mount (**13**) casings.*

2. Tighten the tiller bars (**14**) into their holes in the lens barrels assembly (**12**) (turn clockwise).

3. Adjust the position of the new lens barrel assemblies (**12**) so that the tiller bars (**14**) are approximately midway in their mount (**13**) casings and tighten the locking screw (**11**). See the simple diagram **Fig 17**. to help indicate approximately where the lens should be positioned and how close they will be to the underside of the Golden Gate™ ATR Top Plate when it is in position on the optical unit (**2**).

4. From a replacement of any lens assemblies, the Golden Gate™ ATR accessory will now need realignment. Follow the instructions for alignment at Section 6 of this user instruction manual.

9. Anvils For Golden Gate™ ATR Accessory

Choice of Anvils and their Uses

There are many different compression head anvils that can be used with the Golden Gate™ Single Reflection ATR top plates. The types and their uses are shown in the following table:-

Anvil P/N	Description	Sample Type Use	Top Plate Use
GS10531	Sapphire	Solids and Powders	All diamond plates (*)
GS10532	Pellet	Polymer Beads	All diamond plates (*)
GS10536	Reactive sample	Air Sensitive Samples	All diamond plates (*)
GS10547	Grooved narrow	Fibers/Wires	Diamond wire holder (*)
GS10548	Grooved wide	Fibers/Wires	Diamond wire holder (*)
GS10549	Stainless steel flat	Solids and Powders	All diamond plates (*)
GS10567	Large stainless steel flat	Solids and Powders	Germanium top plate
GS10568	Micro reaction flow cell	Liquids/Supercritical fluids	All diamond plates (*)
GS10569	View thru anvil/bridge assembly	Solids, Powders and Fibers	All top plates(*)

(*) except Top Plates GS10507, GS10585, GS10590.

Sapphire Anvil (GS10531 - Fig 18.)

This anvil can be used with all diamond ATR top plates (see anvil table), for solid or powder samples. The sapphire element on the anvil face is 3.8 mm in diameter.



Fig 18. Sapphire Anvil GS10531

Pellet Anvil (GS10532 - Fig 19.)

This anvil is made of stainless steel and has a concave recess designed for round samples such as polymer pellets. It can be used on all diamond ATR top plates (see anvil table) and, if the sample is relatively soft, with the germanium crystal top plate.

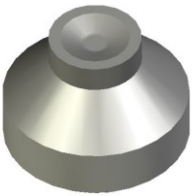


Fig 19. Pellet Anvil GS10532

Grooved Anvils - Narrow (GS10547 – Fig 20a) - Wide (GS10548 – Fig 20b)

These anvils are made of stainless steel and have a groove cut through the pressing face to position fibers or thin wires over the centre of the ATR crystal. These anvils can be used on all diamond ATR plates (see anvil table) and are supplied as standard anvils with the wire holder top plate GS10565.

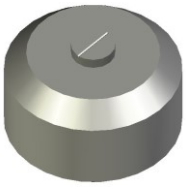


Fig 20a. Narrow Groove Anvil GS10547

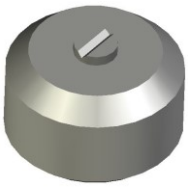


Fig 20b. Wide Groove Anvil GS10548

Stainless Steel Anvils (GS10549 and GS10567 - Figs 21a & 21b)

Either of these stainless steel anvils can be used in place of the sapphire flat anvil GS10531 if desired. GS10549 has a surface pressing face of 4mm diameter. GS10567 has a surface pressing face of 6.5mm diameter. The larger surface stainless steel anvil (GS10567) should always be used with the germanium top plate GS10566. It is the anvil supplied as standard with this top plate.

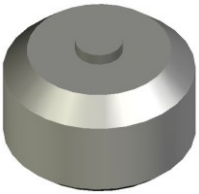


Fig 21a. S/S Anvil GS10549

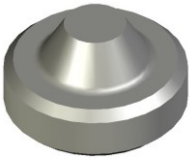


Fig 21b. S/S Anvil GS10567

Reactive Sample Anvil (GS10536 - Fig 22.)

This anvil is effectively a sapphire anvil with an O-ring around the outer edge. When clamped in position an air tight seal is formed around the sample being held in contact against the ATR crystal. Air sensitive samples can be positioned on a top plate in a controlled environment (for example a glove box or fume cupboard). The top plate and anvil can then be taken from the controlled area and placed on the Golden Gate™ optical unit in the spectrometer. This anvil can be used on all diamond ATR top plates (see anvil table) and, if the sample is relatively soft, with the germanium top plate.



Fig 22. Reactive Sample Anvil GS10536

Micro Reaction Flow Cell Anvil (GS10568 - Fig 23.)

This anvil is made of stainless steel and presents a domed cavity for liquid sample containment over the ATR crystal. It has 1/16” O.D.

stainless steel flow tubes finished with Swagelok unions for introduction of a liquid to flow across the ATR crystal. An O-ring seals the anvil cavity against the ATR crystal and in operation it can be used at pressures up to 1000 psi and temperatures up to 300°C. The internal volume of the cell cavity is 28 microlitres. The micro reaction flow cell can be clamped for use with all diamond ATR plates (see anvil table).

There is also a special “static” (non-flow) version of this anvil type which has Luer ports and PTFE plug fittings in place of the standard 1/16” stainless steel flow pipe fittings. This may be more suited for use with volatile solvents for their measurement and long term contact over the ATR crystal instead of use of the volatiles cover GS10503. (Please contact Specac for more details).

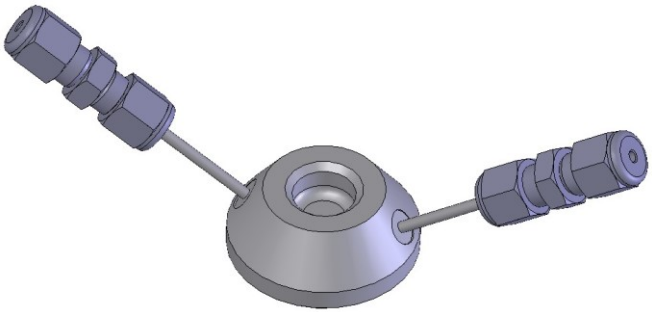


Fig 23. Micro Reaction Flow Cell Anvil GS10568

View Thru Anvil Bridge Assembly (GS10569 - Fig 24.)

This is a standard anvil for viewing of a sample being compressed into contact onto the ATR crystal. The sapphire anvil pressing face doubles as a viewing port as you look down from above into the pressing anvil assembly. The anvil pressing face moves up and down within its own clamp bridge assembly by rotation of the viewing lens assembly. This anvil/bridge assembly combination can be used on all ATR top plates (diamond and germanium ATR crystal options - see anvil table). The clamp head bridge and torque limiter mechanism of the standard ATR top plates must be replaced by the specific view thru anvil bridge

assembly. This is achieved simply by undoing and removing the hinge screw pin from the Golden Gate™ top plate bridge pivot hole, swapping over the standard clamp head bridge assembly with the view-thru anvil bridge assembly and refitting the hinge screw.

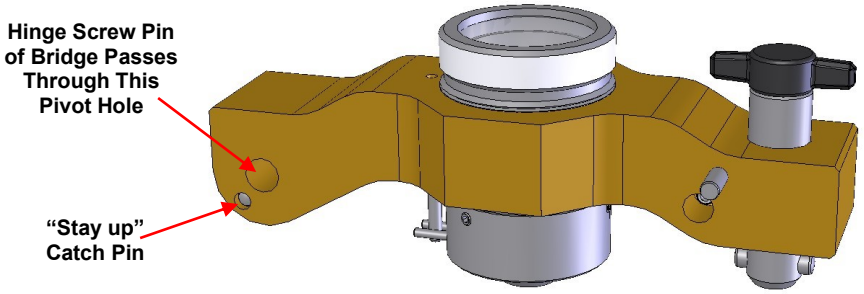


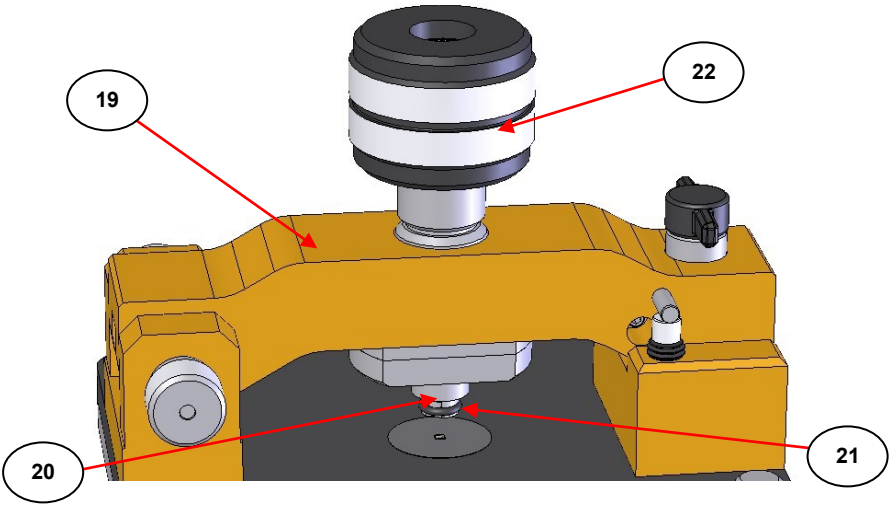
Fig 24. View-Thru Anvil Bridge Assembly GS10569

Note: *When removing the standard bridge mechanism and replacing with the view thru anvil bridge assembly it is important the "stay up" catch pin inside the bridge does not fall out of position. If the catch pin does fall out just slide into back into place.*

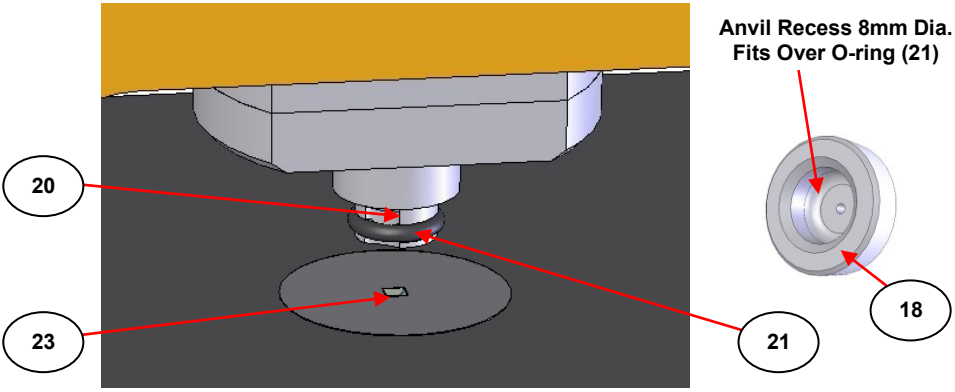
Important! *The View-Thru Anvil Bridge Assembly **must not** be used with the High Temperature Golden Gate™ Top Plate P/N GS10640 because of the sample temperatures that this accessory can generate. (Up to 300°C).*

Fitting of an Anvil

The Golden Gate™ ATR accessory anvil (**18**) options, except the View-Thru Anvil Bridge GS10569 (which has its own bridge (**19**) assembly), are fitted over the clamp head (**20**) via a Viton O-ring (**21**) at the base of the torque limiter screw assembly (**22**). (See **Fig 25.**) There is a recess of 8mm diameter to the underside of the pressing face of any anvil (**18**) option that push fits over the O-ring (**21**) to be held in position. The use of the O-ring (**21**) provides for a self-leveling "float" of the anvil face to adjust to any sample surface it will contact when forcing the sample against the ATR crystal.



General View of Clamp Head Area



Detailed View of Clamp Head Area

Fig 25. Fitting of an Anvil to the Clamp Head of the Torque Limiter Screw on the Golden Gate™ ATR Accessory

Tip: *To fit, apply a small twist to the anvil (**18**) itself whilst pushing it over the O-ring (**21**). Lubricate the O-ring with a light grease if there is resistance to a fit of the anvil over the O-ring.*

10. Sampling Using the Golden Gate™ ATR Accessory

General Principles

When using the Golden Gate™ Single Reflection ATR accessory the sample is placed onto the crystal area (**23**) of the top plate. (See **Fig 25.**) The contact between a solid sample and crystal is improved using the clamp bridge (**19**) and an appropriate compression head anvil (**18**).

For diamond top plates, the diamond crystal (**23**) is brazed into a surrounding support disc of tungsten carbide. The germanium top plate has a germanium crystal glued into a stainless steel support disk. (Specific details for all top plate options can be found in Section 3 of this manual.)

The diamond ATR top plate GS10563 has been used to illustrate for sampling. If using a germanium top plate, the stainless steel flat anvil compression head (GS10567) should be used in place of the sapphire anvil (GS10531) that has been shown for the diamond top plate.

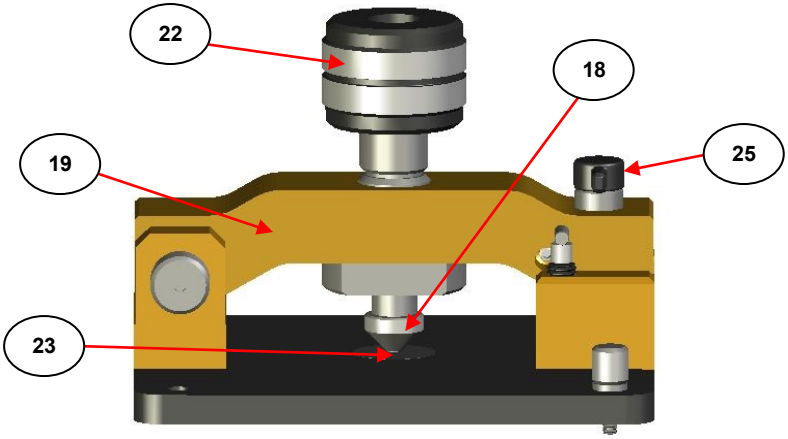


Fig 26. Front View of Diamond ATR Top Plate (Bridge Closed)

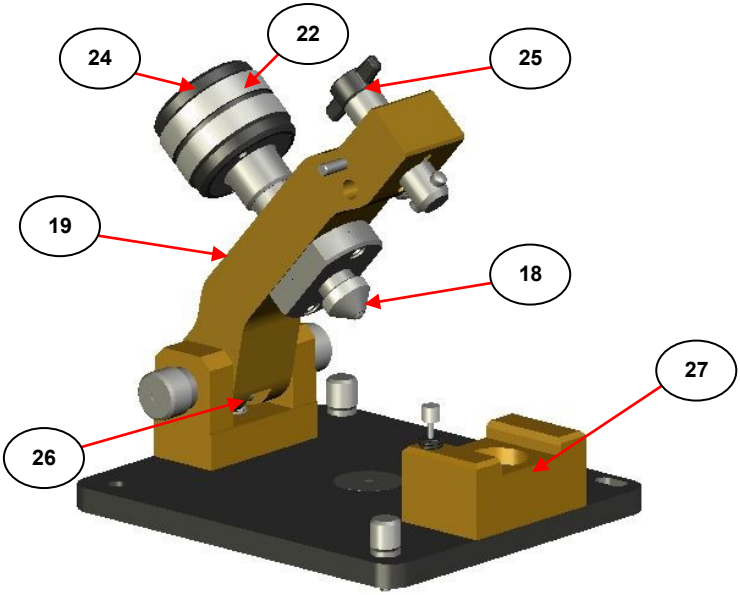


Fig 27. Angled View of Diamond ATR Top Plate (Bridge Open)

Features of the Golden Gate™ ATR Accessory

Torque Limiter Screw Mechanism

The Golden Gate™ ATR top plates with a bridge assembly (**19**) have a built-in torque limiter screw (**22**) mechanism. With an appropriate anvil (**18**) fitted for a solid sample to be compressed against the ATR crystal (**23**) and when the bridge (**19**) is closed, the torque limiter screw (**22**) is turned clockwise to lower the anvil face towards the sample to provide a set load of circa 80lbs spread over the area of the anvil (**18**) face to the sample. When this load has been applied the screw (**22**) can be rotated further, but without increasing the load due to its own slipping clutch mechanism. Consequently, there is no need to use a torque wrench to provide a consistent, reproducible load for routine sampling. However, a torque wrench (GS10504) with Torx head adaptor bit (GS10505) can be supplied separately to provide higher loads up to 160lbs, using the central Torx head screw fixing (**24**) if desired. (See **Fig 28.**). (A complete torque wrench and Torx head bit is GS10509).

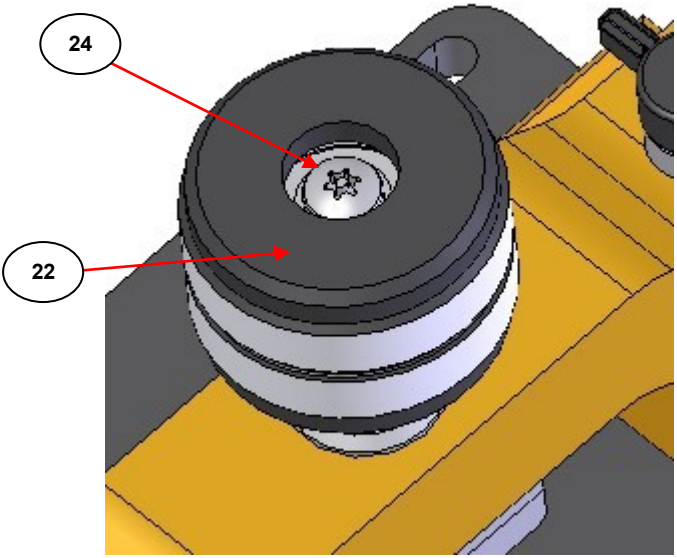


Fig 28. Torque Limiter Screw Showing Torx Head Screw Fixing

Quick Lock and Release Mechanism

The clamp bridge (**19**) has a quick lock and release mechanism (**25**). When pressure needs to be applied to a solid sample via an appropriate fitted anvil (**18**), the bridge must be closed (see **Fig 26.**). The quick lock and release T-knob handle (**25**) is pushed down and turned 90° clockwise to lock the bridge (**19**) closed. To release the bridge the T-knob (**25)** is pushed down and turned 90° anticlockwise.

Important! *When closing the clamp bridge (**19**) ensure that any fitted anvil (**18**) has been retracted so that it will not conflict with the ATR crystal surface or any sample that is in position.*

"Stay Up" Catch

At the hinge point of the Golden Gate™ ATR bridge assembly (**19**) there is a spring/ball bearing "stay up" catch (**26**) that prevents an open

bridge assembly (**19**) from accidentally falling back down to its closed position onto the bridge support (**27**). In addition, when sampling liquids (with the exceptions of the micro reaction flow cell anvil GS10568 or view thru anvil bridge assembly GS10569), there is no need to use the bridge (**19**) with an appropriate anvil (**18**) as the liquid sample will be in adequate contact with the ATR crystal to enable an ATR spectrum to be collected. Hence the stay up catch (**26**) allows the bridge (**19**) to stay open for easier liquid sampling (see **Fig 27.**).

Preparing the Golden Gate™ Accessory for Analysis

For any Golden Gate™ ATR top plate that uses the 2mm x 2mm surface area size diamond ATR crystal, the active sampling area for the concentrated beam of radiant light is about 1.0 mm diameter, in the centre of the diamond crystal surface. Therefore, any samples that are smaller than the 2mm x 2mm surface area should be placed centrally on the diamond for optimum results.

Note: *The germanium ATR crystal in the Golden Gate™ ATR top plate GS10566 is 4mm x 4mm in surface area.*

Collecting a Background and Sample Spectrum

Undo the bridge quick lock T-knob (**25**) and lift the clamp bridge (**19**) up past the stay up catch (**26**). Any anvil (**18**) fitted should not contact the diamond ATR crystal (**23**) to record a background spectrum.

Ensure the diamond is clean and record a background spectrum.

Note: *If it is necessary to clamp the Sapphire Anvil (**18**) (GS10531) directly onto the diamond without a sample, (e.g. to check for contamination) care should be taken to ensure that the anvil is level to the diamond crystal. This can be done by rotating the anvil (**18**) slightly before applying pressure from the torque screw (**22**). Only a small amount of pressure should be needed to check for contamination.* ***It is not advisable to apply the maximum pressure loading without a sample.***

When a background spectrum has been collected, a sample can be placed into position on the ATR crystal to collect a sample spectrum.

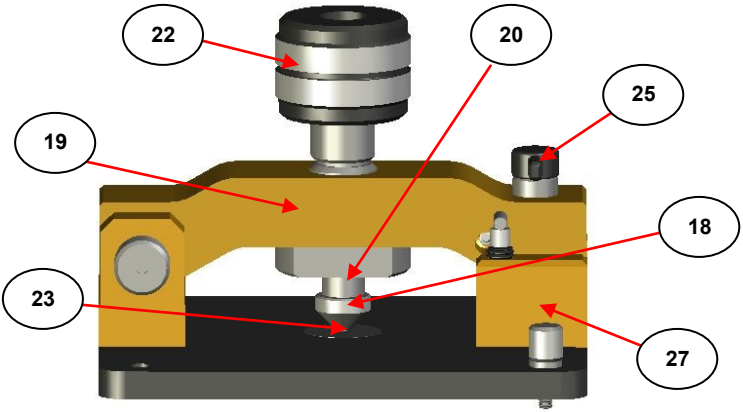


Fig 29. Closed Bridge Golden Gate™ Diamond ATR Top Plate

Collecting ATR Spectra for Different Sample Types

Important: *Golden Gate™ diamond crystals are selected for their freedom from natural structural flaws using optical test methods. It is possible to over-stress the diamond element by applying excessive point loads at the extreme corners. Therefore, if you suspect that your sample may contain exceptionally hard particles, ensure that only a small amount of sample is used and is placed centrally on the diamond.* ***Do not apply maximum loading immediately.*** *Use a torque wrench P/N GS10509 set to 20 cNm (approx. 33 lbs) to apply a load. Acceptable spectra may be obtained without the requirement for the standard torque of 50cNm (approx.80lbs load) or full torque of 100 cNm (approx. 160lbs load).*

Powder Samples

When analyzing for a **powder** sample it is best to use the sapphire anvil GS10531 (**18**) fitted to the clamp head (**20**) assembly. (See **Fig 29.**) The procedure for spectral collection is as follows:

1. Collect a **background spectrum** as described on page 37.

2. Take the powder sample and spread it very carefully to form a level surface that covers the entire surface of the diamond ATR crystal (**23**). Ideally the powder sample should be fine, smooth and homogenous with no hard lumps or inclusions. This will help to avoid any risk of “point load” to the ATR crystal when being pressed.

3. Gently lower the bridge (**19**) past the stay up catch (**26**) and lock it closed using the quick lock T-knob (**25**), ensuring that the anvil (**18**) is initially retracted far enough clear of the sample to be pressed.

4. Begin turning the torque screw knob (**22**) clockwise to lower the anvil (**18**) towards the sample surface. Continue rotation until the central Torx screw (**24**) stops rotating, but the outer black knob continues to turn. At this point the maximum load achievable from the torque limiter screws (**22**) own torque mechanism will be applied to the powder sample forcing it against the ATR crystal.

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the torque knob slowly and apply the load gradually. An acceptable ATR spectrum for the sample may be produced without having to apply a maximum load setting from the torque knob assembly.*

5. Collect and record the ATR spectrum for the sample.

6. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**22**) to retract the anvil (**18**). Unlock the bridge (**19**) by pushing down and turning the quick lock T-knob (**25**) 90° anticlockwise and open the bridge (**19**) up past the stay up catch (**26**).

7. Clean the powder sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface if using the germanium crystal top plate) and wipe and clean any powder off the surface of the sapphire anvil (**18**). If using a diamond ATR top plate there is a minimal risk of damage to the ATR crystal because of the chemical and structural resilience of diamond material.

After cleaning (see **Notes On Cleaning page 48**), the Golden Gate™ ATR Accessory is ready to accept a new sample for measurement.

Flat Solid Samples

When analyzing for a **flat solid sample** it is best to use the sapphire anvil GS10531 (**18**) fitted to the clamp head (**20**) assembly. The procedure for spectral collection is as follows:

1. Collect a **background spectrum** as described on page 37.

2. Take the flat solid sample and place it very carefully for the surface to be measured to be in contact with ATR crystal such that it covers the entire surface of the diamond ATR crystal (**23**). Ideally the flat solid sample should be smooth and homogenous with no hard lumps or inclusions. This will help to avoid any risk of “point load” to the ATR crystal when being pressed.

3. Gently lower the bridge (**19**) past the stay up catch (**26**) and lock it closed using the quick lock T-knob (**25**), ensuring that the anvil (**18**) is initially retracted far enough clear of the sample to be pressed.

4. Begin turning the torque screw knob (**22**) clockwise to lower the anvil (**18**) towards the sample surface. Continue rotation until the central Torx screw (**24**) stops rotating, but the outer black knob continues to turn. At this point the maximum load achievable from the torque limiter screws (**22**) own torque mechanism will be applied to the flat solid sample forcing it against the ATR crystal.

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the torque knob slowly and apply the load gradually. An acceptable ATR spectrum for the sample may be produced without having to apply a maximum load setting from the torque knob assembly.*

5. Collect and record the ATR spectrum for the sample.

6. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**22**) to retract

the anvil (**18**). Unlock the bridge (**19**) by pushing down and turning the quick lock T-knob (**25**) 90° anticlockwise and open the bridge (**19**) up past the stay up catch (**26**).

7. Remove the flat solid sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface if using the germanium crystal top plate) and wipe and clean any sample off the surface of the sapphire anvil (**18**). If using a diamond ATR top plate there is a minimal risk of damage to the ATR crystal because of the chemical and structural resilience of diamond material.

After cleaning (see **Notes On Cleaning page 48**), the Golden Gate™ ATR Accessory is ready to accept a new sample for measurement.

Pellet/Bead Samples

When analyzing for a **pellet bead or irregular shaped sample** it is best to use the pellet anvil GS10532 (**18**) fitted to the clamp head assembly (**20**), to help centralise the sample for contact to the ATR crystal. The procedure for spectral collection is as follows:

1. Collect a **background spectrum** as described on page 37.

2. Take the pellet/bead or irregular shaped sample and place it very carefully such that it covers the entire surface of the diamond ATR crystal. Ideally the pellet/bead sample should be smooth and homogenous with no hard lumps or inclusions. This will help to avoid any risk of “point load” to the ATR crystal when being pressed.

Note: *Pellet/beads of 1.5mm in diameter or less are not suitable for pressing using the concave recessed pellet anvil GS10532 (**18**). Small pellet/beads may have to be analysed by careful manipulation using the sapphire anvil GS10531 (**18**) instead. Beware to avoid any point loading on the ATR crystal with a small pellet/bead sample. For **hard** or **very hard** samples Specac recommend use of diamond ATR crystal top plates only instead of the germanium ATR top plate.*

3. Gently lower the bridge (**19**) past the stay up catch (**26**) and lock it closed using the quick lock T-knob (**25**), ensuring that the anvil (**18**) is initially retracted far enough clear of the sample to be pressed.

4. Begin turning the torque screw knob (**22**) clockwise to lower the anvil (**18**) towards the sample surface. Continue rotation until the central Torx screw (**24**) stops rotating, but the outer black knob continues to turn. At this point the maximum load achievable from the torque limiter screws (**22**) own torque mechanism will be applied to the pellet/bead sample forcing it against the ATR crystal.

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the torque knob slowly and apply the load gradually. An acceptable ATR spectrum for the sample may be produced without having to apply a maximum load setting from the torque knob assembly.*

5. Collect and record the ATR spectrum for the sample.

6. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**22**) to retract the anvil (**18**). Unlock the bridge (**19**) by pushing down and turning the quick lock T-knob (**25**) 90° anticlockwise and open the bridge (**19**) up past the stay up catch (**26**).

7. Remove the pellet/bead sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface if using the germanium crystal top plate) and wipe and clean any sample off the surface of the stainless steel pellet anvil (**18**). If using a diamond ATR top plate there is a minimal risk of damage to the ATR crystal because of the chemical and structural resilience of diamond material.

After cleaning (see **Notes On Cleaning page 48**), the Golden Gate™ ATR Accessory is ready to accept a new sample for measurement.

Fiber Samples

The measurement of fibers or wire type samples can be achieved from utilization of the narrow groove GS10547 and wide groove GS10548 anvils (**18**) for centralisation of the sample over the ATR crystal. If neither of these anvils are available for use with any Golden Gate™ ATR top plate fitted to the optical unit (**2**), then it is best to use the sapphire anvil GS10531 (**18**) fitted to the clamp head assembly (**20**).

Note: *When fitted, some movement of the self-leveling anvil (**18**) pressing surface is allowable to adjust to the sample shape during compression. This movement enables even pressure to be applied across the sample contact area.*

The procedure for spectral collection is as follows:

1. Collect a **background spectrum** as described on page 37.

2. Take the fiber (or fibers) sample and place it very carefully such that it covers the entire surface of the diamond ATR crystal. Lay the fiber centrally across the ATR crystal surface and affix/tape down the fiber ends outside of the ATR crystal surface if necessary to keep the fiber sample in place spanning over the diamond crystal. (If using either the narrow or wide grooved anvil options, then the fiber will be held correctly in position over the ATR crystal.) Ideally the fiber sample should be smooth and homogenous with no hard lumps or inclusions. This will help to avoid any risk of “point load” to the ATR crystal when being pressed.

3. Gently lower the bridge (**19**) past the stay up catch (**26**) and lock it closed using the quick lock T-knob (**25**), ensuring that the anvil (**18**) is initially retracted far enough clear of the sample to be pressed.

4. Begin turning the torque screw knob (**22**) clockwise to lower the anvil (**18**) towards the sample surface. Continue rotation until the central Torx screw (**24**) stops rotating, but the outer black knob continues to turn. At this point the maximum load achievable from the torque limiter screws (**22**) own torque mechanism will be applied to the fiber(s) sample forcing it against the ATR crystal.

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the torque knob slowly and apply the load gradually. An acceptable ATR spectrum for the sample may be produced without having to apply a maximum load setting from the torque knob assembly.*

5. Collect and record the ATR spectrum for the sample.

6. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**22**) to retract the anvil (**18**). Unlock the bridge (**19**) by pushing down and turning the quick lock T-knob (**25**) 90° anticlockwise and open the bridge (**19**) up past the stay up catch (**26**).

7. Remove the fiber sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface if using the germanium crystal top plate) and wipe and clean any sample off the surface of the anvil (**18**) being used. If using a diamond ATR top plate there is a minimal risk of damage to the ATR crystal because of the chemical and structural resilience of diamond material.

After cleaning (see **Notes On Cleaning page 48**), the Golden Gate™ ATR Accessory is ready to accept a new sample for measurement.

Liquids and Paste Samples

In general, liquids and paste like samples make good, close contact with the ATR crystal and it is not necessary to use the bridge (**19**) and clamp head (**20**) with a fitted anvil (**18**). If though the liquid solvent or solution is volatile by nature there are a couple of options that can be employed for prolonged containment in analysis of these liquid sample types over the Golden Gate™ ATR crystal

The volatiles cover GS10503 is provided as standard with the Golden Gate™ ATR top plates GS10563, GS10566, GS10540 and GS10640. The volatiles cover (**28**) is a flat, circular plate which has an

O-ring fitted into a groove at the circumference of the underside surface. (See **Fig 30**.) After a volatile liquid sample has been spotted onto the ATR crystal, the volatiles cover (**28**) is simply placed over the ATR crystal area (**23**) with the O-ring in contact against the diamond/tungsten carbide or germanium/stainless steel puck in a respective diamond or germanium top plate assembly.

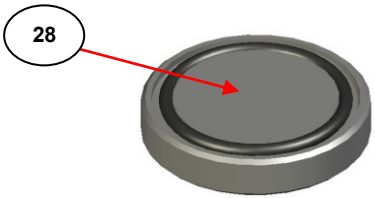


Fig 30. Volatiles Cover GS10503

The O-ring of the volatiles cover (**28**) forms a partial seal to prevent quick evaporation of volatile solvent away from the ATR crystal surface. For additional better sealing of the O-ring, the clamp head (**20**) **without** any anvil (**18**) fitted can be employed from closing the bridge (**19**) and applying a load force by rotation of the torque limiter screw (**22**) to the top surface of the volatiles cover (**28**) to push it closer to the ATR crystal surface area (**23**).

As an alternative to use of the volatiles cover (**28**) for the handling and safe containment of volatile liquids over the ATR crystal area (**23**), the micro reaction flow cell anvil GS10568 (see pages 30 and 31) can be used with appropriate compatible Golden Gate™ ATR top plates.

For analyzing a liquid or a paste:

1. Collect a **background spectrum** as described on page 37.

2. Take the liquid or paste sample and place it very carefully such that it covers the entire surface of the diamond ATR crystal. A dropping pipette can be used to dispense a liquid and a soft bladed spatula can be used to spread a paste over the ATR crystal area (**23**).
3. Place the volatiles cover (**28**) into position over the sample and use

the clamp head (**20**) **without** an anvil (**18**) assembly (**18**) to press against the volatiles cover (**28**) if necessary.

Alternatively, prior to introduction of any liquid sample spotted onto the ATR crystal area (**23**) at step 2), fit a microreaction flow cell anvil GS10568 to the clamp head (**20**) and press this anvil (**18**) assembly for its O-ring to seal over the ATR crystal area (**23**) and introduce a liquid sample into this anvils head cell cavity through the 1/16" O.D flow tubes.

4. Collect and record the ATR spectrum for the sample.

5. To remove a liquid sample, any excess can be removed by sucking up into a dropping pipette and cleaning any residual liquid sample away using soft lens tissues and an appropriate solvent. To remove a paste sample, any excess can be removed by careful use of the soft bladed spatula and cleaning any residual sample away using soft lens tissues and an appropriate solvent. For either a liquid or paste sample be careful when cleaning away from the germanium crystal top plate to avoid risk of damage to the crystal material.

After cleaning (see **Notes On Cleaning page 48**), the Golden Gate™ ATR Accessory is ready to accept a new sample for measurement.



Corrosive Samples

The diamond ATR element in the Golden Gate™ puck assembly is high temperature bonded into tungsten carbide using a metal layer braze (an alloy of 70.5% silver, 26.5% copper and 3% titanium). Tungsten carbide and diamond have exceptional chemical resistance properties; However, Specac cannot guarantee the support disk or brazed bonding material against corrosion from all materials, particularly ***strong mineral acids*** (e.g. phosphoric acid), from prolonged contact under hostile conditions of elevated temperature.

Specac recommend that your sample is confined to the center of the diamond only, if there is a risk of chemical attack upon contact with the other materials of construction. (The diamond puck assembly ***is not indestructible*** and appropriate care in use must be exercised with

potentially aggressive chemicals.) A sample spot size of 2 microns deep with a diameter of 600 microns is sufficient. Specac recommend that a pipette, or similar glass capillary tube, is used to apply small sample spots to the center of the diamond only.

The Golden Gate™ puck assembly has a capability for use with a diverse range of chemically aggressive materials. However, Specac recommend for protection of your accessory, your sample is cleaned off as soon as possible after the analysis is complete.

Pressure on Sample (Load)

The table below gives a guide to recommended loads for different sample types. The optimum load requirements for some typical samples are determined by recording spectra at increasing loads until the desired result is obtained. The optional torque wrench (GS10504) with Torx head adapter bit (GS10505) (complete assembly GS10509) should be used for these torque settings from clockwise rotation of the central Torx screw (**24**). (See **Fig 28**. – page 36.). The Torx head adapter piece of the torque wrench fits in to the Torx screw (**24**) fixing in order to turn the torque limiter screw. Loads from 33 lbs up to 160 lbs (20 cNm to 100 cNm as shown on the torque wrench) can then be applied to the sample.

Type of Sample	**Torque Setting (cNm)**	**Approx. Load (lbs)**
Powders – soft	20 - 60	33 - 100
Powders – hard	40 - 80	67 - 133
Films and Plastic fragments	40 - 80	67 - 133
Polymer pellets – soft	20 - 60	33 - 100
Polymer pellets – hard	60 - 100	100 - 160
Fibers	40 - 80	67 - 133
Liquids and Pastes	Nil	Nil

Notes On Cleaning

When cleaning the diamond or germanium ATR crystal of the Golden Gate™ ATR accessory in preparation for a new sample, it is **very important to take care** to avoid damage to the crystal materials**.** As mentioned in the Safety Considerations (Section 2, page 6), these two crystal materials that are used for sample contact are not particularly hazardous in terms of risk of toxicity if they contact with the skin. There is more risk associated with the ZnSe and most especially the KRS-5 lens materials, should there be a need to clean these items.

Note: ***Always wear gloves to protect yourself and the ATR crystal material from cleaning and sample handling.***

A useful feature of the Golden Gate™ ATR accessory is the capability for removal of the ATR crystal top plate from the optical unit (**2**), such that any sample can be prepared remotely and safely, if desired, onto the ATR crystal surface and then the ATR crystal top plate can be bought back for fitting onto the optical unit (**2**) whilst installed in the spectrometer. Similarly, for cleaning, it may be useful to remove the ATR crystal top plate assembly from the optical unit (**2**) and carry it to a safe area for solvent cleaning and wiping with a tissue and therefore minimise any risk of contamination being carried over to other components of the accessory whilst in situ.

Solvents such as water, methanol and acetone are suitable to use for cleaning purposes. Some sample stains or residues if present around the ATR crystal area (**23**) may require removal using a strong acid or base solution, but if these types of cleaning solvents are employed, do so using sparingly. Quickly clean away any excess solvent using a cloth or tissue and follow by rinsing away with further water to dilute any effect of an acid or base that may remain.

When wiping away any solid or liquid sample, use soft lens tissues to avoid scratches being caused on the surface of the ATR crystal. A diamond crystal will be unaffected, but the germanium crystal material is not as resilient as diamond. Scratches and blemishes to the ATR crystal surface will result in poor light throughput for the ATR technique and an overall degradation in the accessories performance.

In common with general use, it will only be necessary to wipe and clean away at the ATR crystal area (**23**) between samples and possibly the face of any anvil (**18**) if used. If possible try to avoid any solvent or cleaning solution materials from getting to the underside of the crystal area (**23**) and into the optical unit (**2**) and onto the lens and/or mirror components, if the Golden Gate™ ATR top plate has been removed for cleaning. There is a risk that any dried solution residues that have been introduced in this way could be observed as an “impurity” in any “background” spectrum to be collected, and so this contaminant would need to be removed before any further sampling can continue.

Diamond Crystal Considerations

The diamond crystal material in the Golden Gate™ ATR top plates that have this crystal fitted as standard is much more chemically durable than the germanium crystal used in the Golden Gate™ ATR top plate GS10566. Therefore, if a sample is stubbornly resistant to removal from the crystal surface, a more “vigorous” method of cleaning may be carried out for the diamond material than could be achieved with the germanium crystal options. More aggressive solvents and abrasive cleaners could be considered suitable for use to remove the sample, if these are necessary.

In an example of a “cured” and hardened epoxy resin glue that has set permanently over the diamond crystal, the hardened epoxy resin lump may be first “shocked” clear of the diamond crystal by use of a screw driver blade edge, the screwdriver itself being struck carefully with a hammer to impart the force to the blade edge. Any remaining residues from the removed epoxy resin lump may be cleaned with a suitable solvent such as dichloromethane.

This procedure for a stubborn sample to be removed from the ATR crystal surface **could never** be considered for use with the germanium ATR crystal option.

Data Sheet For Diamond

General

Hardest substance known for carbon (C) elemental form.
Can be shaped, cut and polished to form spectral transmission window or crystal for ATR spectroscopy.
Has a highish Refractive Index value and can suffer reflection losses but these can be improved with antireflection optical coatings
Extremely chemically resistant to practically all known materials.
Element symbol: C
Chemical Abstracts Service (CAS) No: 7440-44-0 (Synthetic): 7782-42-5 (Natural)

Physical Data

Appearance: Clear, transparent and generally colourless solid. Has no odour.
Melting point: N/A.
Boiling point: 4827°C.
Vapour pressure: N/A.
Specific gravity: 2.26 g cm^{-3}.
Solubility in water: Insoluble
Hardness: 5700 Kg/mm^2.
Refractive Index: 2.43 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 40,000 to 10 cm-1 (wavenumbers).

Stability

Stable.

Toxicology

Not classified as a dangerous or harmful material according to EC directives.

Personal Protection

Always wear safety spectacles and gloves when handling the window or crystal material.
Allow for adequate ventilation.

Storage

Keep windows or crystal stored in a cool, dry container.

Data Sheet For Germanium

General

Hard and very brittle material, but can be shaped, cut and polished to form spectral transmission window or crystal for ATR spectroscopy.
Because of its high Refractive Index value suffers from large reflection losses but these can be improved with antireflection optical coatings
Is temperature sensitive and loses transmission when heated. (Is optically opaque to IR transmission at 190°C temperature.)
Insoluble in water and alcohols. Soluble in hot sulphuric acid and aqua regia.
Element symbol: Ge
Chemical Abstracts Service (CAS) No: 7440-56-4.

Physical Data

Appearance: Greyish/black, opaque, elemental, metallic solid. Has no odour.
Melting point: 737°C.
Boiling point: 2830°C.
Vapour pressure: 2.66 x 10^{-56} mm Hg at 25°C.
Specific gravity: 5.323 g cm^{-3}.
Solubility in water: Insoluble
Hardness: 780 Kg/mm^2.
Refractive Index: 4.01 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 5,500 to 500 cm-1 (wavenumbers).

Stability

Stable.

Toxicology



Harmful if ingested in large amounts, if inhaled, or if in repeated contact with the skin.

Personal Protection

Always wear safety spectacles and gloves when handling the window or crystal material.
Allow for adequate ventilation.

Storage

Keep windows or crystal stored in a cool, dry container.

Data Sheet For Zinc Selenide

General

Toxic and hard, yellow coloured crystalline powder when fused together as a solid can be used as a transmission window material or as a crystal material for attenuated total reflectance (ATR) FTIR spectroscopy.
Insoluble in water, but attacked by strong acids and bases. (pH range 5 to 9 tolerant). Organic solvents have no effect.
Fairly brittle as a window material and sensitive to thermal and mechanical shock.
Molecular formula: ZnSe
Chemical Abstracts Service (CAS) No: 1315-09-9.

Physical Data

Appearance: Yellow crystals, granular powder or amber coloured window material.
Melting point: 1515°C at 1.8 atmospheres. (26.5psi)
Solubility in water: 0g/100g at 0°C.
Hardness: 120 Kg/mm^2.
Refractive Index: 2.43 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 20,000 to 500 cm-1 (wavenumbers).

Stability

Stable. Reacts with acids to give highly toxic hydrogen selenide. May be air and moisture sensitive. Incompatible with strong acids, strong bases and strong oxidising agents.

Toxicology





Toxic if small amounts are inhaled or swallowed. In stomach toxic hydrogen selenide (H2Se) is liberated. Skin and eye irritant. Danger of cumulative effects from frequent handling without protection.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material. Allow for good ventilation.

Storage

Keep powder or windows stored in a cool, dry container, with appropriate safety labeling.

Data Sheet For KRS-5

General

Synonyms: Mixture of Thallium Bromide and Thallium Iodide (typically 58% Iodide content). Very toxic red coloured soft crystalline powder when fused together as a solid can be used as a transmission window material or as a crystal material for attenuated total reflectance (ATR) FTIR spectroscopy. Slightly soluble in water, soluble in bases, but not soluble in acids. Not hygroscopic. Organic solvents have no effect.
Soft window material and easily deformed.
Molecular formula: $TlBr_{0.4}I_{0.6}$

Physical Data

Appearance: Red, soft crystals, granular powder or red coloured window material
Melting point: 414°C
Solubility in water: 36g/100g at 0°C.
Hardness: 40 Kg/mm^2.
Refractive Index: 2.38 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 17,000 to 250 cm-1 (wavenumbers).

Stability

Stable.

Toxicology



Very toxic if small amounts are inhaled or swallowed. May be fatal if swallowed. May be absorbed through the skin. Irritant.

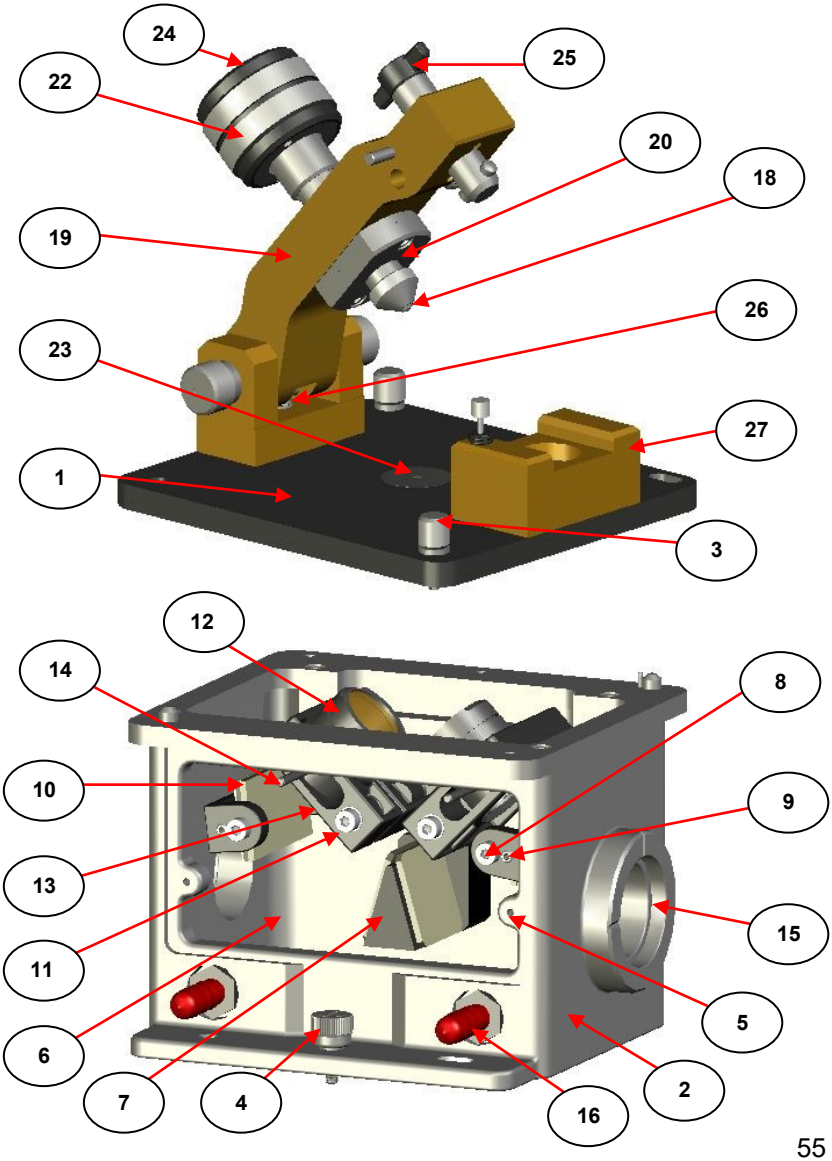
Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material.
Allow for good ventilation. If material is machined, polished or ground, precautions must be taken against inhalation of dust.

Storage

Keep powder or windows stored in a cool, dry container, with appropriate safety labeling.

11. Legend – Bubble Part Number Identification



(**1**) Golden Gate™ ATR top plate assembly.
(**2**) Golden Gate™ optical unit.
(**3**) Fixing thumb screw for top plate to optical unit.
(**4**) Fixing thumb screw for optical unit to Benchmark™ baseplate.
(**5**) Fixing screw for front cover plate of optical unit (position for thumb screw shown in diagram).
(**6**) Front cover plate for optical unit (position for front cover plate shown in diagram).
(**7**) Fixed double mirror mount (**M1** and **M2** mirrors).
(**8**) Rotational adjustment screw for mirror (**M3** and **M4** mirrors).
(**9**) Tilt adjustment grub screw for mirror (**M3** and **M4** mirrors).
(**10**) Spring mounted mirror (**M3** and **M4** mirrors).
(**11**) Locking screw for lens barrel assembly (**L1** and **L2** - ZnSe or KRS-5 lenses).
(**12**) Lens barrel assembly (**L1** and **L2** - ZnSe or KRS-5 lenses).
(**13**) Mount casing for lens barrel assembly.
(**14**) Tiller bar on lens barrel assembly.
(**15**) Aperture port on optical unit.
(**16**) Purge port on optical unit.
(**17**) Purge bellows (not shown).
(**18**) Golden Gate™ ATR anvil (sapphire anvil GS10531 shown).
(**19**) Golden Gate™ ATR bridge assembly.
(**20**) Clamp head of torque limiter screw assembly.
(**21**) Viton O-ring of clamp head (for fixing of anvil).
(**22**) Torque limiter screw assembly.
(**23**) Golden Gate™ ATR crystal sampling area (diamond or germanium crystal options).
(**24**) Torx screw fitting in torque limiter assembly.
(**25**) T-bar handle of the Quick lock and release mechanism of the bridge assembly.
(**26**) “Stay up” ball bearing catch assembly.
(**27**) Bridge assembly support (contains mechanism to lock the bridge closed for solid sampling).
(**28**) Volatiles cover GS10503 (not shown).

12. Spare Parts for the Golden Gate™ ATR Accessory System

For details of upgrade options and spares for your Golden Gate™ ATR accessory, please contact your local Specac office.

Golden Gate™ Top Plate Options

GS10563 Diamond crystal ATR top plate.
GS10566 Germanium crystal ATR top plate.
GS10540 Heated diamond ATR top plate (to 200°C) and temperature controller (specify voltage 110V / 220V).
GS10640 High temperature diamond ATR top plate (to 300°C) and temperature controller (specify voltage 110V / 220V).
GS10514 Microspecular reflection top plate.
GS10507 Reaction cell diamond ATR top plate (to 200°C) and temperature controller (specify voltage 110V / 220V).
GS10590 Low temperature diamond ATR top plate (-150°C to 80°C) and temperature controller (specify voltage 110V / 220V).
GS10585 Supercritical fluid diamond ATR top plate (to 300°C) and temperature controller (specify voltage 110V / 220V).

Spare Lenses

GS10508 KRS-5 Lens Upgrade Kit.
GS10552 ZnSe Lens Upgrade Kit.

Anvil Options

GS10503 Volatiles Cover.
GS10531 Sapphire Anvil.
GS10532 Pellet Anvil.
GS10536 Reactive Sample Anvil.
GS10547 Grooved Anvil – Narrow.
GS10548 Grooved Anvil – Wide.
GS10549 Stainless Steel Flat Anvil.

GS10567 Large Stainless Steel Flat Anvil (for germanium top plate).
GS10568 Microreaction Flow Cell Anvil.
GS10569 View-Thru Anvil Bridge Assembly.

Spares

GS10504 Torque wrench.
GS10505 Torx adapter bit for torque wrench.
GS10509 Torque wrench and Torx adapter bit (combined).
GS10707 Purge Bellows.

13. Technical Specification

ATR Crystal	Diamond Single Crystal Diamond 45° - 2 mm x 2 mm	Germanium Ge 45° 4 mm x 4 mm
Accessory Transmission Range	5000 -700 cm^{-1} (with ZnSe lenses) 6500 - 400cm^{-1} (with KRS-5 lenses)	5000 -700 cm^{-1} (with ZnSe lenses) 5200 - 650cm^{-1} (with KRS-5 lenses))
Refractive Index at 1000 cm^{-1}	2.4	4.0
ATR Plate	Diamond brazed into Tungsten Carbide Disc	Germanium glued into Stainless Steel disc
Active Sampling Area (50% of Transmitted Energy)	0.8 mm diameter	0.8 mm diameter
Maximum Applied Force	100 cNm (torque) 160 lbs or 1.78 Kbar	80 cNm (torque) 128 lbs or 0.36 KBar
Depth of Penetration	2.0µm (For sample of Refractive Index 1.5 @ 1000 cm^{-1})	0.7µm (For sample of Refractive Index 1.5 @ 1000 cm^{-1})

14. Golden Gate™ ATR Serial Numbers

Your Golden Gate™ ATR Accessory will be provided with a serial number for identification of certain individual part assemblies. The serial number takes the form of a letter followed by a five figure number e.g. M12345 and will be found as a label on the equipment.

To help, please use the table below to fill in the serial number information against the part number of the Golden Gate™ ATR accessory and parts you have received. If you need to contact Specac for any issues regarding your Golden Gate™ ATR accessory it may be necessary to provide the serial number of the item to identify for certain specific replacement parts.

Golden Gate™ ATR Accessory Part Number	Serial Number	Date Received
GS10500-Z		
GS10500-K		
GS10516		
GS10523-Z		
GS10523-K		
GS10525-Z		
GS10525-K		
GS10542-Z		
GS10542-K		
GS10586-Z		
GS10586-K		
GS10592-Z		
GS10592-K		
GS10642-Z		
GS10642-K		

Worldwide Distribution

France
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Tel.01 42 08 01 28
Fax 01 42 08 13 65
email: contact@eurolabo.fr

Germany
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Tel: 06151 88060
Fax: 06151 880689
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email:systems-eng@systems-eng.co.jp
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Barcelona
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Fax: 93 675 2405
email: comercial@teknokroma.es

Switzerland
Portmann InstrumentsAG
Biel-Benken
Tel: 061 726 6555
Fax: 061 726 6550
email: info@portmann-instruments.ch
Website:www.portmann-instruments.ch

USA
SPECAC INC.
414 Commerce Drive
Suite 175,
Fort Washington,
PA 19034, USA
Tel: 215 793 4044
Fax: 215 793 4011

United Kingdom
Specac Ltd. - London
Unit 12, Science & Innovation Centre
Halo Business Park
Orpington
Kent BR5 3FQ
Tel:+44 (0) 1689 873134
Registered No. 1008689 England

Brilliant Spectroscopy™

www.specac.com

SPECAC INC.
414 Commerce Drive
Suite 175,
Fort Washington,
PA 19034, USA
Tel: 215 793 4044
Fax: 215 793 4011

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