Silver Gate™ Evolution

User manual

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Silver Gate™ Evolution
Single Reflection ATR System
User Manual

2I-10680 Issue 2

Silver Gate™ Evolution ATR System

SILVER GATE™ EVOLUTION ATR SYSTEM - P/N GS10680 SERIES

USER MANUAL

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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.

The Silver Gate™ Evolution Single Reflection ATR is designed for the quantitative and qualitative analysis of solids, powders, liquids, pastes and gum-like samples. Sufficient sample is required to cover an ATR crystal surface of 7mm diameter.

The Silver Gate™ ATR accessory can be configured for use starting with the optical unit and supplied with or without a bridge and clamp anvil assembly. There is a choice of zinc selenide (ZnSe), germanium (Ge) or silicon (Si) flat ATR crystal top plate assemblies and ZnSe or Ge recessed ATR crystal top plate assemblies to be used on either of the optical unit assemblies (with or without a bridge.) The ATR crystals are glue mounted into their respective ATR crystal top plate assemblies.

A complete Silver Gate™ ATR system consists of an ATR flat or recessed crystal top plate assembly with a ZnSe or Ge crystal at 45° incident angle option fitted to an optical unit **with** a bridge and clamp anvil assembly. This whole assembly is mounted into a spectrometer via a Benchmark™ type baseplate for correct positioning of the accessory to maximize for an optical throughput signal. The ATR crystal top plate assemblies are interchangeable for placing on a common optical unit.

The bridge and clamp anvil assembly can be fitted to the Silver Gate™ optical unit as an option if desired. The simplest Silver Gate™ ATR Accessory configuration is by use of a flat or recessed ATR crystal top plate assembly on the optical unit **without** a bridge and clamp anvil assembly. This combination of parts can be used for the analysis of liquids and pastes.

For sampling of solids and powders, the bridge and clamp anvil assembly is fitted onto the Silver Gate™ optical unit. The bridge part carries a torque limiter screw assembly that provides for a consistent

and reproducible load to an anvil compression head to enable excellent close contact of a solid sample with the ATR crystal.

In operation the entire torque screw limiter assembly knob is rotated until a sufficient load and hence pressure has been applied to the sample. The torque screw limiter assembly knob will continue to rotate when the set load is reached, but the drive connection slips and no more force will be applied. The black colored cover cap with the white “cross” mark on top of the knob acts as the indicator when the set applied load is reached in ceasing to rotate. The flat ATR crystal top plate assemblies are best suited to be used in combination with the bridge and clamp anvil assembly for solid or powder sample types, although if the sample is small enough, the recessed ATR crystal top plate assembly could be used instead.

When using the bridge and clamping mechanism for pressure application, the compression head anvil does not rotate against the sample. This ensures that heat is not generated by friction which may cause changes to the sample, and the sample is not displaced from the correct sampling position.

The transfer optics unit of the Silver Gate™ ATR consists of mirrors and optical components to achieve maximum throughput for each flat or recessed ATR crystal top plate assembly.

As standard a volatiles cover (P/N GS10503) and purge bellows (P/N GS10707) are supplied with the optical unit **without** a bridge assembly. In addition to these parts, a standard stainless steel pressure head anvil (P/N GS10567) and pellet anvil (P/N GS10532) are also provided along with the optical unit **with** a bridge assembly.

2. Unpacking and Checklist

The Silver Gate™ ATR accessory is provided in its own carry case.

Depending on which Silver Gate™ ATR system has been ordered will determine the items to check on delivery.

Please check for the following.

1 Silver Gate™ ATR optical unit **without** bridge and clamp anvil assembly. - This will include:-
1 Volatiles cover P/N GS10503.
1 Pair of purge bellows P/N GS10707.

1 Silver Gate™ ATR optical unit **with** bridge and clamp anvil assembly. This will include:-
1 Volatiles cover P/N GS10503.
1 Pair of purge bellows P/N GS10707.
1 Stainless steel flat anvil P/N GS10567.
1 Pellet anvil P/N GS10532.

With either optical unit assembly:-

1 ATR crystal top plate assembly (choice of ZnSe, Ge or Si flat, or ZnSe, Ge or Si recessed crystal top plate assembly.

(Any additional ATR crystal top plates ordered with the accessory.)

1 Ball driver 3.0mm
1 Allen key 2.0mm
1 Benchmark™ baseplate for your FTIR spectrometer.
1 Instruction manual for Benchmark™ baseplate installation.
1 Essential Spares Kit of parts (P/N GS10685)
Carefully remove your Silver Gate™ ATR accessory and any ATR crystal top plates from the carry case in readiness for use.

3. Installation Via Benchmark™ Baseplate

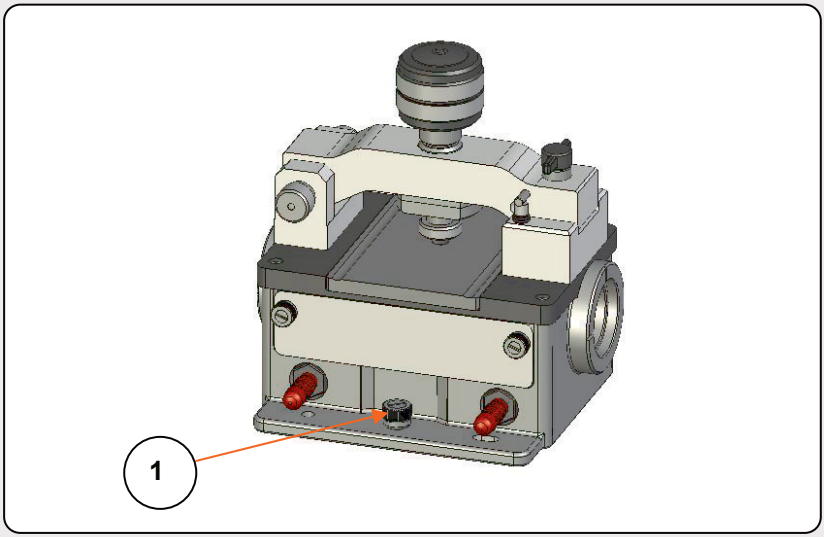

Figure 1. Silver Gate™ ATR Accessory – Optical Unit with Bridge and Clamp Anvil Assembly and Flat ATR Crystal Top Plate Assembly

The Silver Gate™ ATR is supported on a Benchmark™ baseplate when installed into a spectrometer. The Benchmark™ baseplate has three support pillars (one flat support pillar towards the rear and two at the front with location pins) and a fourth front central pillar into which the fixing thumb screw (**1**) of the Silver Gate™ optical unit is tightened.

Note: *It is normally best to install the Benchmark™ baseplate in the spectrometer first before locating the Silver Gate™ ATR accessory.*

Fixing holes and studs in the Benchmark™ baseplate will vary dependant on the make and model of the spectrometer for which the Silver Gate™ is to be installed. For details on how to install your accessory in the spectrometer, refer to the Benchmark™ baseplate Installation Guide/User Manual supplied.

4. Alignment Procedure

When you have installed the Silver Gate™ ATR accessory into your spectrometer on its appropriate Benchmark™ baseplate, you should check for alignment of the accessory. An overall spectral throughput of between 20% and 55% for ZnSe and Ge ATR flat or recessed crystal top plate assemblies should be achievable. The Silver Gate™ ATR will be supplied having a preliminary factory alignment. On installation, some transmitted energy should be recorded on the spectrometers energy/light throughput to detector monitoring system, but it will be necessary to maximize the optical throughput for any specific spectrometer by adjusting the mirrors in the optical unit of the Silver Gate™ accessory. To carry out any alignment procedure one of the Silver Gate™ ATR flat or recessed crystal top plate assemblies **must** be fitted to the optical unit.

Fitting of the ATR Crystal Top Plate to the Optical Unit

Six different versions of Silver Gate™ ATR crystal top plate assemblies can be fitted to the optical unit. They are:-

ZnSe flat crystal top plate assembly, P/N GS10683-1.
Ge flat crystal top plate assembly, P/N GS10683-2.
Si flat crystal top plate assembly, P/N GS10683-3.
ZnSe recessed crystal top plate assembly, P/N GS10684-1.
Ge recessed crystal top plate assembly, P/N GS10684-2.
Si recessed crystal top plate assembly, P/N GS10684-3.

The flat and recessed ATR crystal top plate assemblies are fitted to the top plate area of the Silver Gate™ optical unit by location holes (**2**) to location pins (**3**) and held in place by magnetic catches (**4**) to counter plate fixings (**5**). (Please see Figures 2, 3, 4 and 5.) Top views of the flat and recessed ATR crystal top plate assemblies using the ZnSe crystal as examples are Figures 2 and 3. (Ge and Si top plates are similar.) The underside view of either the flat or recessed ATR crystal top plate assembly is shown as Figure 4. The top surface (without bridge) on the optical unit where to fit the ATR top plates is shown in Figure 5.

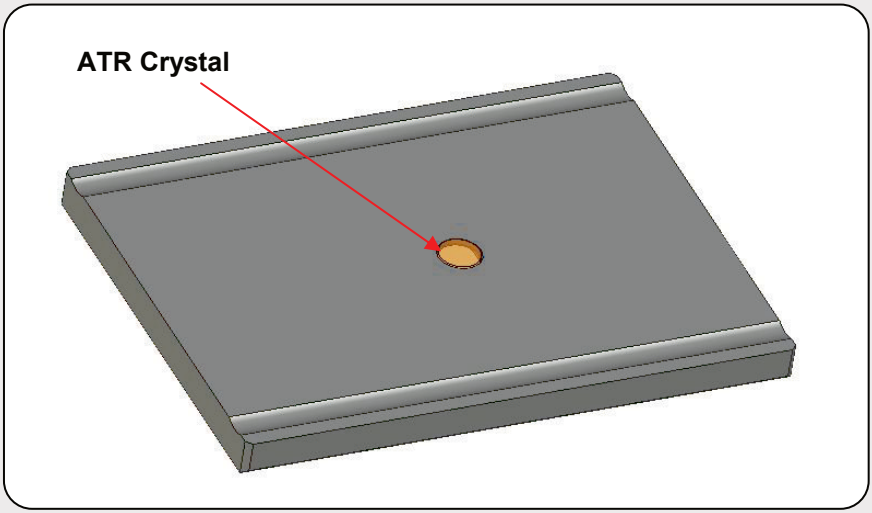

Figure 2. Top view of Silver Gate™ ZnSe Flat ATR Crystal Top Plate Assembly

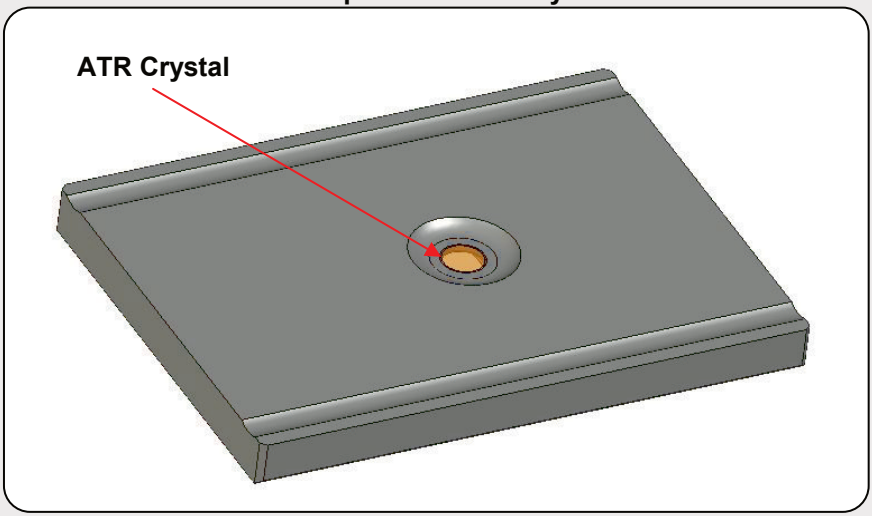

Figure 3. Top view of Silver Gate™ ZnSe Recessed ATR Crystal Top Plate Assembly

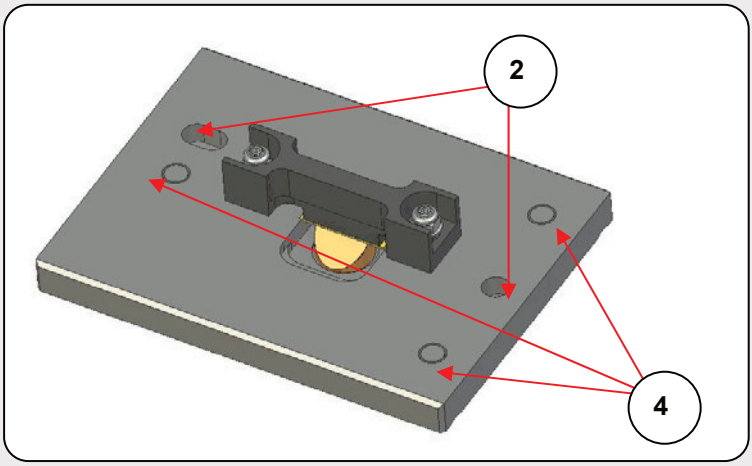

Figure 4. Underside view of Silver Gate™ ZnSe Flat or Recessed ATR Crystal Top Plate Assembly

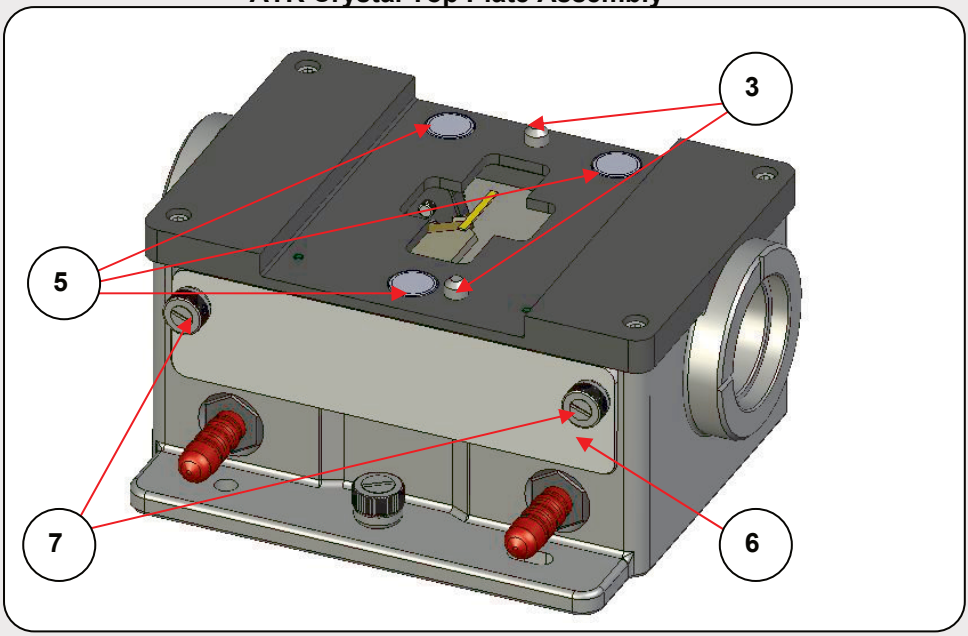

Figure 5. Top view of Silver Gate™ Optical Unit without Bridge

The flat and recessed ATR crystal top plate assemblies will fit into the top surface area of the Silver Gate™ accessory in one orientation only. Align the location holes (**2**) with the location pins (**3**). The location **slot** (**2**) must be towards the front of the Silver Gate™ Accessory where the thumbscrew fixing (**1**) is located. Allow the three magnetic catches (**4**) to engage with their three counter plate fixings (**5**) to hold the ATR crystal top plate securely in position. To remove and swap over with any other ATR crystal top plate assembly, just pull up on the ATR crystal top plate to release the magnetic catches.

Alignment

With an ATR crystal top plate assembly in position on the optical unit and when the whole is installed in the spectrometer on an appropriate Benchmark™ baseplate, the Silver Gate™ Accessory can be finely aligned. The optical unit cover plate (**6**) is removed by undoing the cover plate thumb screws (**7**) (see Figure 5.) to gain access to the mirror adjustment screws (**8**) and (**9**).

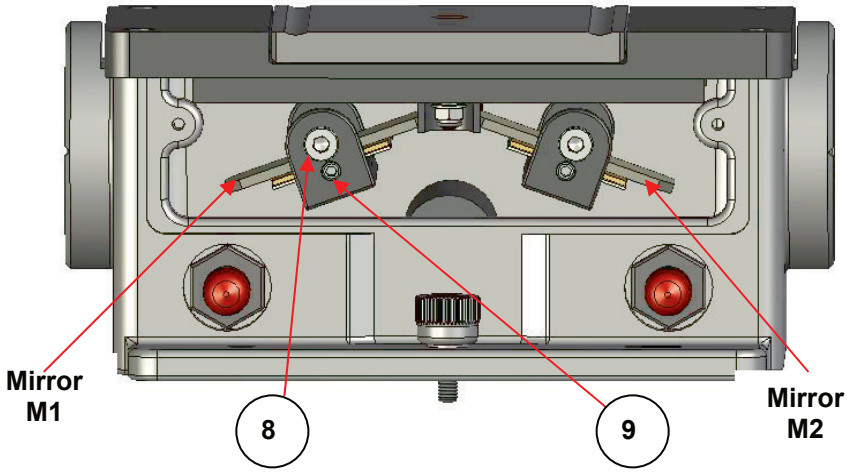

Figure 6. Front view of Silver Gate™ with a Flat ATR Crystal Top Plate and Optical Unit Cover removed to show Mirrors

The mirrors **M1** and **M2** in the optical unit can be adjusted for rotation and tilt. (See Figure 6.) The M4 cap head screw (**8**) allows for rotation of the mirror and the M4 x 12mm grub screw with a cone point (**9**) allows for tilt of the mirror, as fixed to its mirror carriage assembly.

The mirror optical set up is symmetrical so it does not matter which direction the beam passes through the optical unit from source to detector. (Left to right or right to left as seen from the front of the optical unit.) However, to align your Silver Gate™ accessory you need to follow the alignment instructions knowing whether your spectrometer is a left to right or right to left beam (source to detector) configuration.

The instructions below have been written for a **left to right** beam configuration spectrometer.

Tip: *If the energy reading on the instrument monitor goes to zero while making any of the adjustments always bring it back with the same adjustment before proceeding further. It is always best to start alignment from the output mirror (the one nearest the detector). In these instructions it is assumed **M2** is the output mirror. If your output mirror is **M1** follow instructions substituting **M1** with **M2** (and vice versa.)*

1. Using the Ball driver tool in screw (**8**) of mirror **M2** very gently adjust the rotational movement of the mirror to achieve maximum energy.

2. Adjust mirror **M2** tilt position by inserting the smaller Allen key into grubscrew (**9**) and turning to peak the energy.

Important : *Turning the Allen key too far anti-clockwise will cause the grubscrew (**9**) to dislodge from the back of the mirror (**M2**). If this accidentally happens, turn the Allen key a full circle in the same direction to dislodge the screw further and free it. (Do not remove it). Next, push the spring mounted mirror (**M2**) away from the black anodized mirror mount and turn the Allen key clockwise until the screw is re-set against the rear of the spring.*

3. Maximize the energy by adjusting the rotation and tilt movement screws (**8**) and (**9**) respectively of mirror **M1**. (See Figure 6.)

4. Repeat the rotation and tilt adjustments as in steps 2 – 4 until maximum transmission is achieved and then replace the front cover plate (**6**) and tighten the cover plate thumb screws (**7**).

5. If the Benchmark™ baseplate is mounted on a rail or is movable in the spectrometer, move the Silver Gate™ ATR Accessory on the Benchmark™ baseplate to find the best position where the transmission is at a maximum. Now firmly secure the Benchmark™ baseplate in this position.

Note: *In some spectrometers it may be necessary to remove the Silver Gate™ optical unit before securing the baseplate. Where this is the case, ensure that the Benchmark™ baseplate does not move when removing the Silver Gate™ optical unit.*

5. Fitting Accessories to the Silver Gate™ ATR Optical Unit

When the Silver Gate Single Reflection ATR has been installed and aligned in the spectrometer you have the options of fitting a polarizer and/or purging the ATR optical unit.

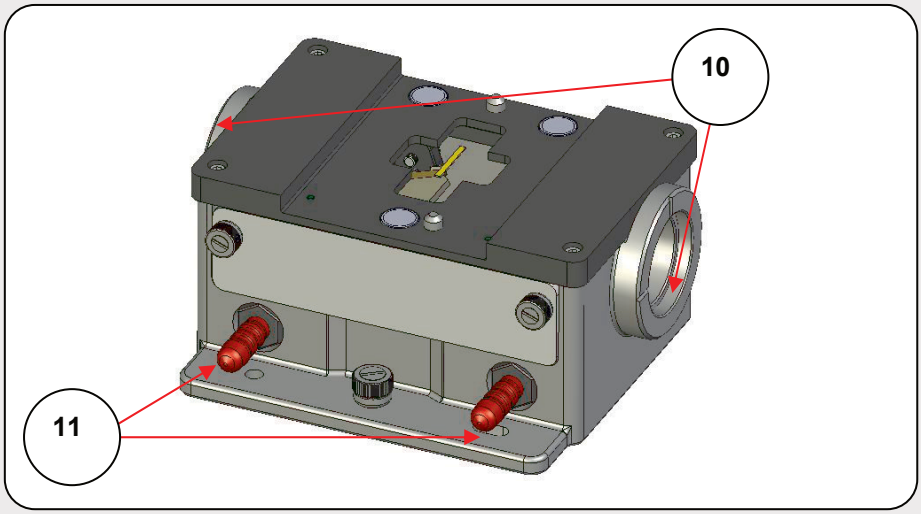

Figure 7. Aperture Ports and Purge Connections on the Silver Gate™ Optical Unit

Fitting a Polarizer

Specac P/N GS12000 Series Polarizers can be fitted to the apertures (**10**) of the optical unit. The polarizers push fit in these apertures and can be rotated to the desired angular orientation. The polarizer will transmit at a maximum (perpendicular component of the polarized light) when it is mounted so that the notch slot in the polarizer holder ring is in the vertical plane. Fitting the polarizer to either aperture of the optical unit will have the same effect.

Note: *Fit a polarizer where required before purging the system.*

Benchmark™ Baseplate Compatible Accessory Polariser Rotator Mount P/N GS12510

In place of the GS12000 Series polariser, a specific Benchmark™ baseplate compatible accessory polariser rotator mount can be fitted to the aperture ports (**10**) of the Silver Gate™ optical unit.

The polarizer mount GS12510 is a rotatable mount that accepts the Specac range of GS57010 Series, 38mm circular aperture, ring mounted polarizers. When a GS50710 Series polarizer has been installed into the GS12510 mount, it can be rotated for a particular angular degree of polarized light by adjustment of an outer rotating ring on the polarizer mount itself.

Figure 8. GS12510 Polarizer Mount

The polarizer mount GS12510 can be used when polarized light experimentation is to be carried out using any Benchmark™ baseplate compatible accessory, particularly if there is limited space within a spectrometer sample compartment. The standard aperture ports of the Benchmark™ baseplate compatible optical units (as item **10** for the Silver Gate™ optical unit), will accept a GS12000 Series polarizer. To rotate this type of polarizer, it must be removed from the aperture port and turned to align with the indicating grooves in the aperture port ring for either parallel or perpendicular polarization and then re-inserted. There is a risk of marking the polarizer substrate from fingerprints etc, whenever placing the polarizer into, or removing it, from the aperture mounting ports. Therefore, if continual changing of the polarizer angular orientation is required, use of an appropriate GS57010 Series polarizer within the GS12510 mount may help to minimize any risk of marking/damage if using a GS12000 Series polarizer that has to be removed from the aperture port and rotated by hand.

Purging the Optical Unit

The Silver Gate™ ATR Accessory is fitted with purge ports (**11**). The purge port connections are fitted with rubber caps. To purge the optical unit the rubber caps are removed and a suitable size of flexible rubber tubing can be pushed over the ¼" hose connection fittings at the purge ports to enable the unit to be filled with a purge gas, e.g. Nitrogen.

To enable the optical unit to be purged it is necessary to fit the flexible purge bellows (**12**) to the optical unit at the aperture ports (**10**). (See Figure 9.) The purge bellows grip fit around the circumference of the aperture ports and bridge any gap between the optical unit and the spectrometer sample compartment bulkheads from the source and to the detector. If the gap/distance between the optical unit and bulkhead is small, the purge bellows can be cut to size. It is best to cut all the way around and in between one of the ridged grooves in the bellow moulding itself.

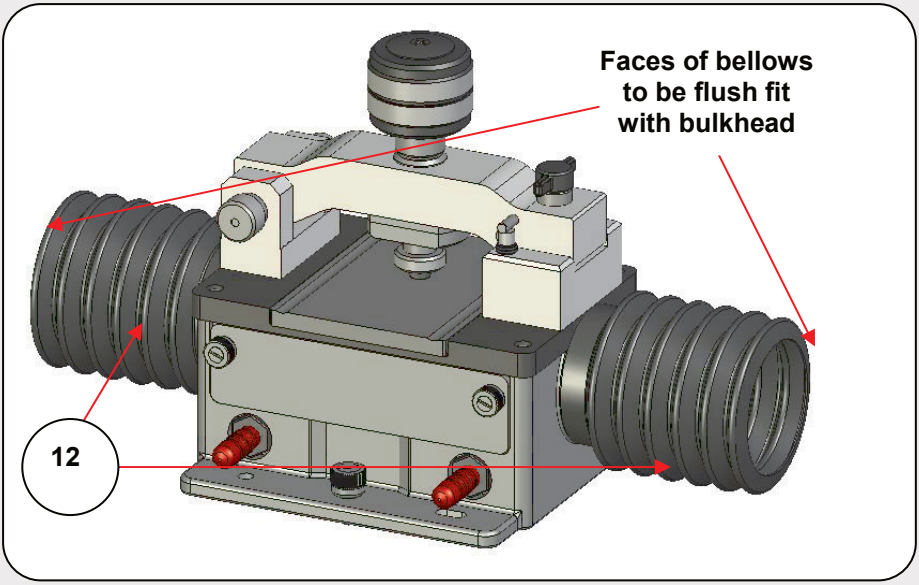

Figure 9. Silver Gate™ ATR Accessory – Optical Unit with Bridge and Clamp Anvil Assembly, Flat ATR Crystal Top Plate Assembly and Purge Bellows

Tip: *It is recommended that purge bellows are fitted during use to help stabilize the spectrometer/instrument background even if purging is not to be carried out.*

Fitting the Purge Bellows P/N GS10707

1. With the Benchmark™ baseplate secured and the Silver Gate™ Accessory in position, measure the distance between the spectrometer source and detector bulkhead/side walls and the flat end faces of the Silver Gate™ Optical Unit. (This is dimension "'X").

2. Using a sharp razor, cut lengths of the flexible purge bellows (**12**) which are equivalent to the measured length ("X") allowing an additional 10mm for each.

Tip : *It is easier to cut the purge tube between the ridged grooves.*

3. Unscrew the Silver Gate™ Optical Unit fixing thumb screw (**1**) and remove the Silver Gate™ ATR Accessory from the sample compartment.

Note: *Fit a polarizer at this stage if required. (GS12000 Series or GS12510 Benchmark™ Compatible Accessory Polarizer Mount)*

4. Fit the flexible purge bellows (**12**) over both end apertures on the optical unit and compress sufficiently to enable the whole assembly to fit into the spectrometer.

5. Refit the Silver Gate™ ATR Accessory with the fitted purge bellows to the Benchmark™ baseplate. Ensure the purge bellows (**12**) are not obstructing the optical beam and then retighten the fixing thumb screw (**1**) to secure the Silver Gate™ ATR Accessory back onto the Benchmark™ baseplate.

6. Anvil Options

Choice of Anvils and their Uses
There are two different compression head anvils (**13**) that can be used with the Silver Gate™ ATR Accessory when fitted with the bridge and clamp anvil assembly. The types of anvil and their uses can be tabulated as follows:-

Anvil	Description	Sample Type / Use
GS10532	Pellet	Polymer beads, softish irregular shaped solids
GS10567	Stainless steel flat	Solids and powders

Pellet Anvil P/N GS10532 (Figure 10.)

This anvil is made of stainless steel and has a concave recess designed to take round shaped samples such as polymer beads. Relatively soft irregular shaped samples can be used with this anvil on any of the Silver Gate™ ATR crystal top plate assemblies.

Figure 10

St/Steel Flat Anvil P/N GS10567 (Figure 11.)

The stainless steel anvil P/N GS10567 should be used generally for all solid and powder samples to bring the sample into good contact with the ZnSe or Ge crystal for any of the ATR crystal top plate assemblies**.**

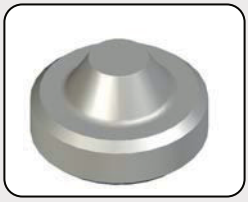

Figure 11

Tip: *When placing an anvil (**13**) into position on the end of the clamp head pressure pin (**14**) on the bridge, push the hollow depression of the anvil on with a rotational movement to ensure it makes a compression fit with the retaining O-ring. Ensure the anvil pressing face is level and parallel to the bridge (**15**). (See Figure 12.)*

7. Sampling Using the Silver Gate™ Accessory

General Principles

When using the Silver Gate™ ATR Accessory, a liquid or solid sample is placed onto the crystal area of one the flat or recessed ATR crystal top plate assemblies to obtain an ATR spectrum for the sample. Particularly for solid samples, the best ATR spectra are produced when the sample is in close contact with the ATR crystal, so it is recommended to use the bridge and clamp anvil assembly (**15**) of the Silver Gate™ ATR Accessory on the optical unit. In this way a load can be applied to the solid sample from an appropriate compression head anvil (**13**) fitted to the clamp head pressure pin (**14**), (see Figure 12), to force the sample into good and close contact with the ZnSe or Ge ATR crystal.

Features Of The Silver Gate™ Bridge and Clamp Anvil Assembly

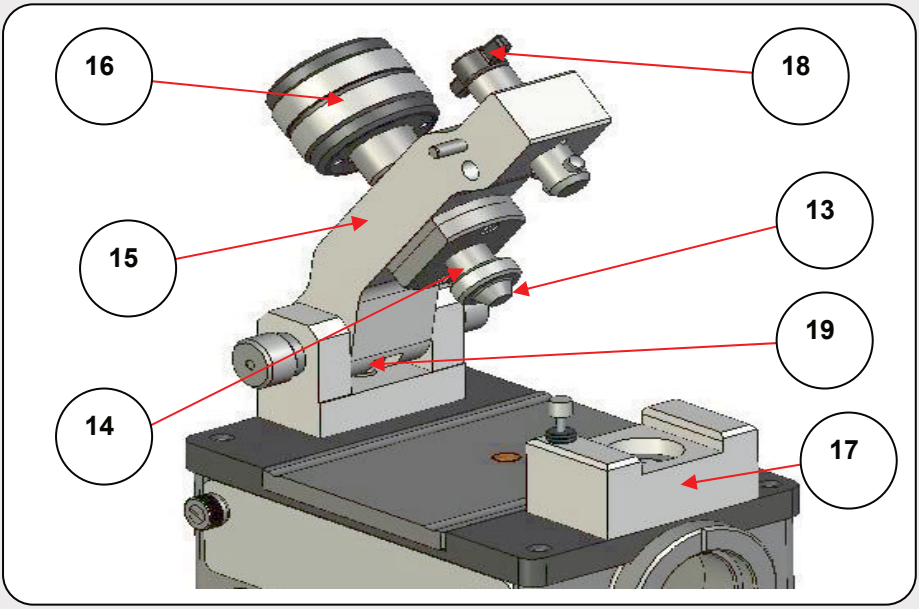

Figure 12. Silver Gate™ Bridge and Clamp Anvil Assembly (Open)

Torque Limiter Screw Assembly

The Silver Gate™ bridge and clamp anvil assembly (**15**) is provided with a special built in torque limiter screw assembly (**16**). For solid sampling, an appropriate anvil (**13**) is fitted to the clamp head pressure pin (**14**). A solid sample is placed upon the ZnSe or Ge ATR crystal to cover it completely and the bridge is closed and locked to the bridge support block (**17**).The bridge is locked shut by pushing down on the T shaped black quick lock/release knob (**18**) and turning it a 90° angle clockwise. (When the bridge is unlocked and open the T shaped locking knob is parallel to the direction of the bridge – see Figure 13.)

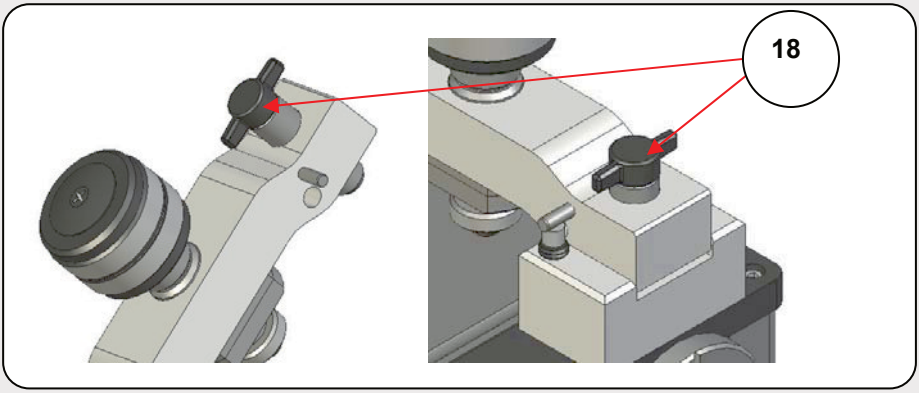

Figure 13. Silver Gate™ direction of T shaped Quick Lock/Release knob (18) when bridge is open (unlocked) and closed (locked)

With an appropriate anvil (**13**) fitted, the torque limiter screw assembly knob (**16**) is turned in a clockwise fashion to provide a pre-determined set pressure (50lbs load spread over the anvil face) to the sample. In operation the entire torque limiter screw assembly knob is rotated until a sufficient load and hence pressure has been applied to the sample. The torque limiter screw assembly knob (**16**) will continue to rotate when the set load is reached, but the black colored cover cap with the white “cross” mark on top of the knob acts as the indicator when the set applied load is reached in ceasing to rotate. (i.e. the white cross stops rotating when the torque/load limit is reached.)

Quick Lock and Release Mechanism

The bridge and clamp anvil assembly (**15**) carries a quick lock/release screw mechanism (**18**). In operation for sampling with **solids** using either the flat or recessed ATR crystal top plate assembly, the bridge must be closed (see Figure 9). The T-shaped quick lock/release knob (**18**) is pushed down and turned clockwise 90º to engage and lock the bridge closed. To release the bridge, the T-shaped knob (**18**) is pushed down and turned anti-clockwise 90º.

Stay Up Device

At the hinge point of the Silver Gate™ bridge and clamp anvil assembly (**15**) there is a spring ball bearing stay up catch device (**19**) that prevents an open bridge from accidentally falling back down to its closed position. The bridge is lifted open to pass beyond the point where the ball bearing spring catch engages. When sampling **liquids** using either the flat or recessed ATR crystal top plate assembly, there is no need to use the bridge and clamping assembly (**15**) with any anvil compression heads (**13**), so the bridge can stay open if desired (see Figure 12.)

Important Note for Usage!

The ZnSe, Ge and Si crystals in both the flat and recessed ATR crystal top plate assemblies have a specific hardness and chemical resistance and should be treated accordingly. Check on the chemical nature of a sample before potentially damaging materials are brought into contact with the ATR crystal.

Be careful not to put a “point load” on the ATR crystal, particularly with hard or abrasive samples. When using samples such as rubber and polymer sheeting there should be no problem and normal anvil pressures may be used. Thin films on plastic substrates should be no problem, but care should be taken if the substrate is metallic.

Apply pressure from the torque knob assembly (**16**) turning it gradually. Take spectra to see if an acceptable load has already been applied for the sample before the maximum load setting has been reached.

Preparing the Silver Gate™ ATR Accessory for Analysis

For the ZnSe, Ge and Si flat and recessed ATR top plate assemblies the active sampling area extends across the whole of the ATR crystal. It is therefore preferable to have enough sample to completely cover the ATR crystal to utilise the maximum energy throughput available for the Silver Gate™ ATR accessory.

The procedure for collecting a sample ATR spectrum requires first obtaining a background spectrum as a reference and then repeating the procedure with a sample in position on the Silver Gate™ ATR accessory.

Collecting a Background Spectrum

1. Choose a ZnSe, Ge or Si flat or recessed ATR crystal top plate assembly to be used on either a Silver Gate™ optical unit with or without a bridge and clamp anvil assembly.

2. Attach the Silver Gate™ optical unit to the installed Benchmark™ baseplate and fit the chosen ATR crystal top plate assembly to the optical unit.

3. If using a Silver Gate™ bridge and clamp anvil assembly (**15**) on the Silver Gate™ optical unit, then choose an appropriate compression head anvil (**13**) (P/N GS10567 or P/N GS10532 - see Figures 10 and 11) to fit to the clamp head pressure pin (**14**).

4. The Silver Gate™ ATR accessory should already be aligned to register an acceptable signal throughput with an ATR crystal top plate assembly in position. Having ensured that the ZnSe, Ge or Si ATR crystal is clean, proceed to collect a background spectrum using any preferred acquisition conditions.

Note: *If using a Silver Gate™ bridge and clamp anvil assembly (**15**) the anvil (**13**) does not need to be in contact with the ATR crystal to record a background spectrum.*

Contamination Check on Anvil

If for any sampling procedure the Silver Gate™ bridge and clamp anvil assembly (**15**) is being used because it is necessary to clamp the stainless steel anvil (**13**) (P/N GS10567) directly onto the ATR crystal, (e.g. to check for contamination of any residual sample that might be left on the anvil surface even if it has been cleaned), care must be taken to ensure that the anvil is level to the ATR crystal. This can be done by rotating the anvil (**13**) slightly around the clamp head pressure pin (**14**) at its O-ring fixing to loosen it for any level movement, before applying a load/pressure from the torque screw assembly.

Warning: *The anvil (**13**) **must** be raised away and not touching the ATR crystal when rotating it about the clamp head pressure pin (**14** as any surface roughness may scratch the crystal. Only a small amount of load/pressure should be needed to check for contamination. It is not advisable to apply the maximum pressure loading.*

Collecting ATR Spectra of Powder Samples

When analyzing for a powder sample it is best to use a flat ATR crystal top plate assembly on a Silver Gate™ ATR accessory fitted with a bridge and clamp anvil assembly (**15**). The best anvil (**13**) to choose would be the stainless steel anvil P/N GS10567. (See Figure 14.)

1. Collect a background spectrum as described in steps 1 to 4 (page 21).

2. Open the bridge (**15**) and position it past the stay up catch device (**19**). This allows for easier access to the ATR crystal surface area to position the sample correctly.

3. Take the powder sample and spread it very carefully to form a level surface that covers the entire surface of the ZnSe, Ge or Si ATR crystal. 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.

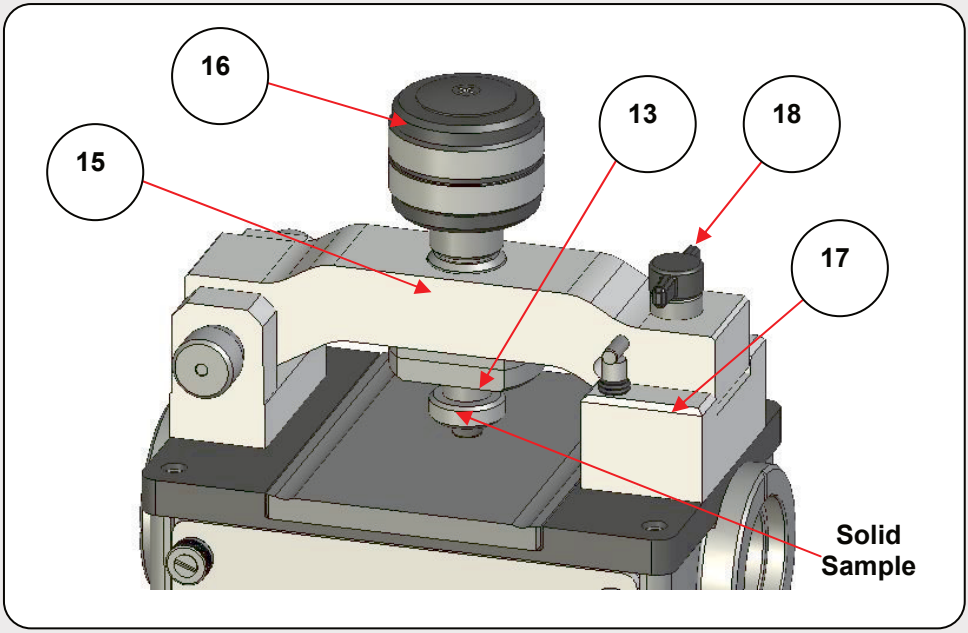

Figure 14. Silver Gate™ ATR Accessory with bridge (15) closed and a powder sample between the anvil (13) and ATR crystal

4. Close the bridge (**15**) and lock it to its support block (**17**) via the quick lock/release screw (**18**).

5. Begin turning the torque knob assembly (**16**) clockwise to lower the stainless steel anvil (**13**) towards the sample surface. Continue rotation until the white cross on the black cover at the top of the torque knob assembly (**16**) ceases to rotate. At this point a maximum load 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.*

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

7. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**16**) to retract the anvil (**13**) before undoing the quick lock/release screw (**18**) and raising the bridge (**15**) up past the stay up catch device (**19**).

8. Clean the powder sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface) and wipe and clean any powder off the surface of the stainless steel anvil (**13**).

After cleaning (see **Notes on Cleaning** page 32), the Silver Gate™ ATR accessory is ready to accept a new sample for measurement.

Collecting ATR Spectra of Flat Solid Samples

When analyzing for a flat solid sample it is best to use a flat ATR crystal top plate assembly on a Silver Gate™ ATR accessory fitted with a bridge and clamp anvil assembly (**15**). The best anvil (**13**) to choose would be the stainless steel anvil P/N GS10567. (See Figure 14.)

1. Collect a background spectrum as described in steps 1 to 4 (page 21).

2. Open the bridge (**15**) and position it past the stay up catch device (**19**). This allows for easier access to the ATR crystal surface area to position the sample correctly.

3. Take the flat solid sample and place it very carefully such that it covers the entire surface of the ZnSe, Ge or Si ATR crystal. 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.

4. Close the bridge (**15**) and lock it to its support block (**17**) via the quick lock/release screw (**18**).

5. Begin turning the torque knob assembly (**16**) clockwise to lower the stainless steel anvil (**13**) towards the sample surface. Continue

rotation until the white cross on the black cover at the top of the torque knob assembly (**16**) ceases to rotate. At this point a maximum load 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.*

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

7. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**16**) to retract the anvil (**13**) before undoing the quick lock/release screw (**18**) and raising the bridge (**15**) up past the stay up catch device (**19**).

8. Remove the flat solid sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface) and clean the ATR crystal and stainless steel anvil (**13**) surfaces.

After cleaning (see **Notes on Cleaning** page 32), the Silver Gate™ ATR accessory is ready to accept a new sample for measurement.

Collecting ATR Spectra of Pellet/Bead Samples

When analyzing for a pellet/bead or irregular shaped sample it is best to use a flat ATR crystal top plate assembly on a Silver Gate™ ATR accessory fitted with a bridge and clamp anvil assembly (**15**). The best anvil (**13**) to choose would be the concave recessed pellet anvil P/N GS10532. (See Figure 14.)

1. Collect a background spectrum as described in steps 1 to 4 (page 21).

2. Open the bridge (**15**) and position it past the stay up catch device (**19**). This allows for easier access to the ATR crystal surface area to position the sample correctly.

3. Take the pellet/bead or irregular shaped sample and place it very carefully such that it covers as much of the surface of the ZnSe, Ge or Si ATR crystal as possible. 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.

4. Close the bridge (**15**) and lock it to its support block (**17**) via the quick lock/release screw (**18**).

5. Begin turning the torque knob assembly (**16**) clockwise to lower the pellet anvil (**13**) towards the sample surface. As the concave, recessed surface of the pellet anvil begins to touch the pellet/bead sample it will help to keep the sample centralised over the ATR crystal.

Note: *Pellet/beads of 1.5mm in diameter or less are not suitable for pressing using the concave recessed pellet anvil (13). Small pellet/beads may have to be analysed by careful manipulation using the flat stainless steel anvil(13) instead. Beware to avoid any point loading on the ATR crystal with a small pellet/bead sample.*

Continue rotation until the white cross on the black cover at the top of the torque knob assembly (**16**) ceases to rotate. At this point a maximum load 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.*

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

7. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**16**) to retract

the anvil (**13**) before undoing the quick lock/release screw (**18**) and raising the bridge (**15**) up past the stay up catch device (**19**).

8. Remove the pellet/bead sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface) and clean the ATR crystal and concave pellet anvil (13) surfaces.

After cleaning (see **Notes on Cleaning** page 32), the Silver Gate™ ATR accessory is ready to accept a new sample for measurement.

Collecting ATR Spectra of Fiber Samples

When analyzing for a fiber sample it is best to use a flat ATR crystal top plate assembly on a Silver Gate™ ATR accessory fitted with a bridge and clamp anvil assembly (**15**). The best anvil (**13**) to choose would be the stainless steel anvil P/N GS10567. (See Figure 14.)

Note: *When fitted, some movement of the stainless steel anvil (**13**) pressing surface level is allowed to adjust to the sample shape during compression. This movement enables even pressure to be applied across the sample contact area.*

1. Collect a background spectrum as described in steps 1 to 4 (page 21).

2. Open the bridge (**15**) and position it past the stay up catch device (**19**). This allows for easier access to the ATR crystal surface area to position the sample correctly.

3. Take the fiber (or fibers) sample and place it very carefully such that it covers as much of the ZnSe, Ge or Si ATR crystal surface as possible. Lay the fiber centrally across the ATR crystal surface and affix/tape down the fiber ends outside of the ATR crystal surface if necessary. 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.

4. Close the bridge (**15**) and lock it to its support block (**17**) via the quick lock/release screw (**18**).

5. Begin turning the torque knob assembly (**16**) clockwise to lower the stainless steel anvil (**13**) towards the sample surface. Continue rotation until the white cross on the black cover at the top of the torque knob assembly (**16**) ceases to rotate. At this point a maximum load will be applied to the fiber 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.*

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

7. To remove or change the sample, release the load/pressure on the sample first by unscrewing the torque knob assembly (**16**) to retract the anvil (**13**) before undoing the quick lock/release screw (**18**) and raising the bridge (**15**) up past the stay up catch device (**19**).

8. Remove the fiber sample carefully away from the ATR crystal (avoid the possibility of scratching the ATR crystal surface) and clean the ATR crystal and stainless steel anvil (**13**) surfaces.

After cleaning (see **Notes on Cleaning** page 32), the Silver Gate™ ATR accessory is ready to accept a new sample for measurement.

Collecting ATR Spectra of Liquids and Paste Samples

For analyzing a liquid sample it is best to use a recessed ATR crystal top plate assembly on a Silver Gate™ ATR optical unit. It is not necessary to have a bridge and clamp anvil assembly (**15**) fitted when sampling liquids as generally they will make good, close contact with the ATR crystal and do not require any external force from an anvil. (See Figure 15.)

The recessed “trough” area of the recess ATR crystal top plate assembly will contain any mobile fluid to concentrate it over the ATR crystal itself and prevent it from spilling over the ATR crystal top plate

assembly surface. If the liquid is fairly volatile in nature, then the volatiles cover P/N GS10503 supplied (**20**) can be placed over the liquid to contain it during the analysis. (See Figure 16.)

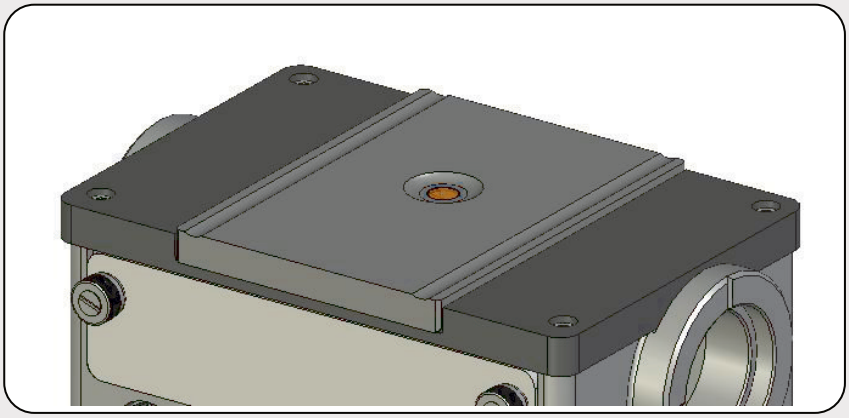

Figure 15. Silver Gate™ ATR Accessory – Optical Unit and Recessed ZnSe ATR Top Plate Assembly without Bridge and Clamp Anvil Assembly

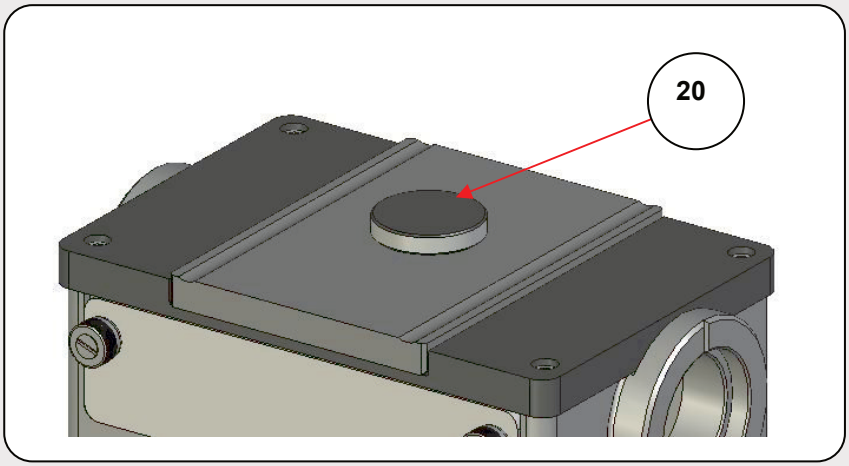

Figure 16. As Figure 15 but with Volatiles Cover (20) over the Recessed ZnSe ATR Top Plate Assembly

For analyzing a paste sample either a flat or recessed ATR crystal top plate assembly could be used on the Silver Gate™ optical unit.

If the Silver Gate™ accessory has a bridge and clamp anvil assembly (**15**) fitted, a liquid sample can be analyzed with the bridge in the open (unlocked) position past the stay up catch device (**19**). Alternatively, the bridge and clamp anvil assembly (**15**) can be removed completely from the bridge hinge support block (**20**) by unscrewing a thumb nut (**21**) for the hinge pin and sliding the hinge pin out of the bridge and support block. (See Figures 17 and 18.)

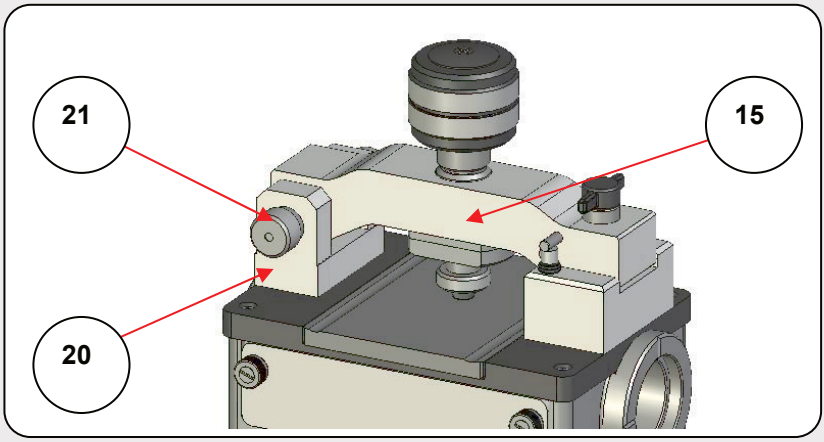

Figure 17. Silver Gate™ Hinge Block (20) and Thumb Nut (21) which is unscrewed for removal of Bridge assembly (15)

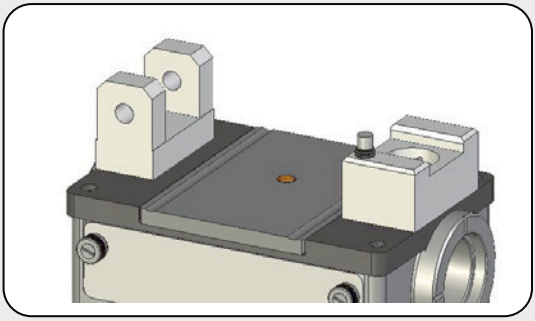

Figure 18. Silver Gate™ ATR Accessory with Bridge (15) removed

Note: *If the Silver Gate™ bridge and clamp anvil assembly (**15**) is removed by loosening and removing the hinge pin, take care that the stay up catch support rod (**22**) that is loose fitted in the bridge and which engages against the stay up catch device (**19**) does not fall out of the bridge and becomes lost (See Figure 19.)*

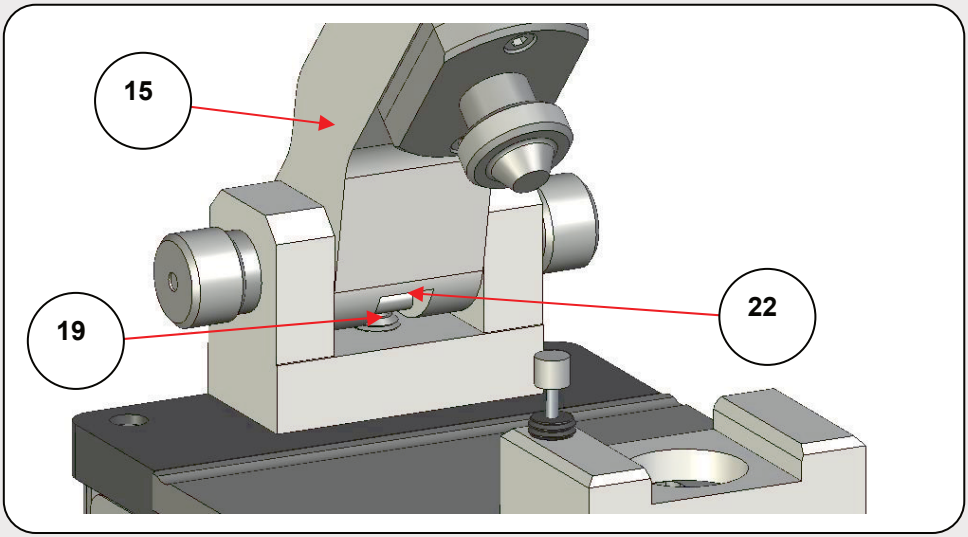

Figure 19. Silver Gate™ Accessory with Bridge (15) up showing detail of the stay up catch (19) and stay up support rod (22)

For analyzing a liquid or a paste:

1. Collect a background spectrum as described in steps 1 to 4 (page 21).

2. If the Silver Gate™ ATR Accessory is fitted with a bridge and clamp anvil assembly (15), open the bridge and position it past the stay up catch device (**19**). This allows for easier access to the ATR crystal surface area to position the sample correctly.

3. Take the liquid or paste sample and place it very carefully such that it covers the entire surface of the ZnSe, Ge or Si ATR crystal. A

dropping pipette can be used to dispense a liquid onto the recessed ATR top plate assembly and a soft bladed spatula can be used to spread a paste over the ATR crystal of either a flat or recessed ATR crystal top plate assembly.

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.

After cleaning (see **Notes on Cleaning** page 32), the Silver Gate™ ATR accessory is ready to accept a new sample for measurement.

Notes on Cleaning

When cleaning either a ZnSe, Ge or Si ATR crystal of the Silver Gate™ ATR crystal top plate assemblies in preparation for a new sample, it is **very important to take care** to avoid damaging the crystal materials. Always wear gloves to protect yourself and the ATR crystal material.

Solvents such as water, methanol and acetone are suitable to use. Avoid the use of halogenated solvents (e.g. dichloromethane, chloroform, etc), as long term contact will result in degradation of the epoxy glue bonding of the ATR crystal in the top plate assembly. Sample solutions that fall within the pH range of pH4 to pH11 are tolerated by ZnSe. Stronger acids and bases will damage ZnSe irreparably.

When wiping away any solid or liquid sample, use very soft lens tissues to avoid scratches being caused on the surface of the ATR crystal. 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.

8. Silver Gate™ ATR Accessory "Bubble Number" Parts Identification List

(**1**) Optical unit fixing thumb screw.
(**2**) Location holes of ATR crystal top plate assemblies.
(**3**) Location pins of top plate area on optical unit.
(**4**) Magnetic catches of ATR crystal top plate assemblies.
(**5**) Counter plate fixings of top plate area on optical unit.
(**6**) Optical unit front cover plate.
(**7**) Optical unit front cover plate thumb screw.
(**8**) Optical unit mirror rotation cap head screw.
(**9**) Optical unit mirror tilt grub screw.
(**10**) Optical unit aperture port.
(**11**) Optical unit purge port tube fitting.
(**12**) Purge bellows P/N GS10707.
(**13**) Anvil (stainless steel P/N GS10567, pellet P/N GS10532).
(**14**) Clamp head pressure pin for anvil.
(**15**) Bridge and clamp anvil assembly.
(**16**) Torque limiter screw assembly/knob.
(**17**) Bridge support block.
(**18**) Quick/lock release knob/screw assembly.
(**19**) Stay up catch device.
(**20**) Hinge support block.
(**21**) Hinge pin thumb nut.
(**22**) Stay up catch device support rod.

9. Silver Gate™ ATR Accessory Spare Parts

P/N GS10681 Silver Gate™ Evolution Optical Unit (includes volatiles cover P/N 10503 and purge Bellows P/N GS10707).
P/N GS10682-1 Silver Gate™ Evolution Top Plate **without** bridge for optical unit.
P/N GS10682-2 Silver Gate™ Evolution Top Plate **with** bridge for optical unit (includes stainless steel anvil P/N GS10567 and pellet anvil P/N GS10532).
P/N GS10683-1 Silver Gate™ Evolution ZnSe flat ATR crystal top plate assembly.
P/N GS10683-2 Silver Gate™ Evolution Ge flat ATR crystal top plate assembly.
P/N GS10683-3 Silver Gate™ Evolution Si flat ATR crystal top plate assembly.
P/N GS10684-1 Silver Gate™ Evolution ZnSe recessed ATR crystal top plate assembly.
P/N GS10684-2 Silver Gate™ Evolution Ge recessed ATR crystal top plate assembly.
P/N GS10684-3 Silver Gate™ Evolution SI recessed ATR crystal top plate assembly.
P/N GS10532 Pellet anvil.
P/N GS10567 Stainless steel anvil.
P/N GS10503 Volatiles cover.
P/N GS10707 Purge bellows.

Silver Gate™ Evolution ATR Crystal Replacement

If damage is sustained to either the Silver Gate™ Evolution ZnSe or Ge ATR crystal of the flat or recessed ATR crystal top plate assemblies, a new replacement top plate assembly can be ordered from Specac, or the damaged plate assembly can be returned to Specac for inspection and to try and effect a repair. In the event of such an occurrence please contact Specac to arrange for return of the item.

10. Silver Gate™ ATR Technical Specification

	ZnSe	Germanium	Silicon
ATR Crystal	ZnSe 45° 7mm diameter	Ge 45° 7mm diameter	Si 45° 7mm diameter
Accessory Transmission Range cm^{-1}	20,000 - 550	5,200 - 650	8333 - 33
Refractive Index at 1000cm^{-1}	2.43	4.0	3.42
ATR Crystal Plate Assembly	ZnSe glued into anodized aluminium top plate	Ge glued into anodized aluminium top plate	Si glued into anodized aluminium top plate
Applied Force From Bridge Anvil	50 lbs	50 lbs	50 lbs
Typical Light Throughput When Aligned	Between 20% and 55% Transmission	Between 20% and 40% Transmission	Between 20% and 45% Transmission
Depth of Penetration	2.0µm (For Sample of Refractive index 1.5 @ 1000cm^{-1})	0.7µm (For Sample of Refractive index 1.5 @ 1000cm^{-1})	0.9µm (For Sample of Refractive index 1.5 @ 1000cm^{-1})

11. Silver Gate™ ATR Serial Numbers

Your Silver Gate™ ATR Accessory will be provided with a serial number for identification of certain individual part assemblies. To help you, please use the grid below to fill in the serial number information of the Silver Gate™ ATR Accessory parts you have received. If you need to contact Specac for any issues regarding your Silver Gate™ ATR accessory it may be necessary to provide the serial number of the item to identify for replacement parts.

Silver Gate™ Evolution Part Number and Description	Serial Number
P/N's GS10681 and GS10682-1 – optical unit and top plate surface without bridge and clamp	
P/N's GS10681 and GS10682-2 - optical unit and top plate surface without bridge and clamp	
P/N GS10683-1 ZnSe flat ATR crystal top plate	
P/N GS10683-2 Ge flat ATR crystal top plate	
P/N GS10683-3 Si flat ATR crystal top plate	
P/N GS10684-1 ZnSe recessed ATR crystal top plate	
P/N GS10684-2 Ge recessed ATR crystal top plate	
P/N GS10684-3 Si recessed ATR crystal top plate	

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