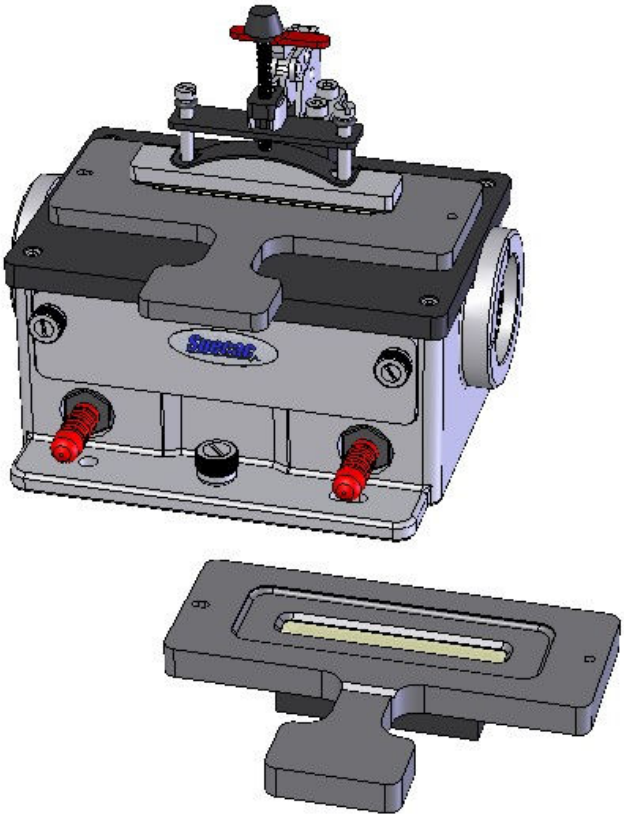
Gateway™ ATR
6 Reflection Horizontal ATR Accessory Kit

User Manual



2I-11165-4

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Gateway™ ATR Accessory Kit P/N GS11165

GATEWAY™ 6 REFLECTION HORIZONTAL ATR ACCESSORY KIT

USER MANUAL

1. Introduction

Thank you for purchasing a Specac Product.

The Gateway™ ATR Accessory Kit P/N GS11165 is a 6 reflection horizontal ATR system consisting of an optical unit (P/N GS11170), a trough top plate assembly with a 45° angle zinc selenide (ZnSe) crystal (P/N GS11166), a flat top plate assembly with a 45° angle ZnSe crystal (P/N GS11133) and a clamp assembly (P/N GS11171). The trough top plate is to be used on the optical unit for the analysis of liquids and pastes and the flat top plate and clamp assembly are used on the optical unit for the analysis of solids and powders. The trough top plate assembly design allows for the easy removal and change of the ATR crystal from ZnSe to germanium (Ge) or silicon (Si) crystal options. The ATR ZnSe crystal is bonded into the flat top plate assembly using an epoxy resin and is not removable, so for any of the crystal options of a flat top plate assembly it is supplied as a complete assembly from Specac.

The Gateway™ ATR Accessory is simple to install and operate within a wide range of FTIR spectrometer systems via use of the Specac Benchmark™ baseplate mounting assembly. Different Benchmark™ baseplates are available for specific fit into a particular make and model of spectrometer system. The Gateway ™ optical unit attaches to the Benchmark ™ baseplate by a single thumbscrew fixing for correct optical beam passage and positioning of the Accessory when installed into the sample compartment of the spectrometer system.

The Gateway™ ATR Accessory offers a relatively high optical light throughput as the result of its unique optical design and the optical unit offers the capability for fitting and use of a polarizer, if required, for specific ATR studies. There is a wide range of interchangeable trough type top plates available that can be used on the optical unit providing for further sampling versatility. These static and flow through and trough top plates allow for different temperatures (up to 200°C for the electrically heated top plate), to be applied for liquid sample measurements and the design of these top plates incorporates removable crystals for ease of cleaning and simple replacement. All top plates are fitted as standard with a 6 reflection ZnSe ATR crystal

with a 45° angle of incidence. The Ge and Si crystal options are available on request for use in these top plates. The ZnSe and Ge crystals have an antireflection coating on their angled faces to help improve the light energy throughput for these high refractive index crystal materials (2.4 and 4.0 respectively).

The range of Gateway ATR top plates that can be used on the optical unit are:-

P/N GS11133 Gateway™ ATR Flat top plate with 45° ZnSe crystal.
P/N GS11134 Gateway™ ATR Flat top plate with 45° Si crystal.
P/N GS11135 Gateway™ ATR Flat top plate with 45° Ge crystal.
P/N GS11166 Gateway™ ATR Trough top plate with 45° ZnSe crystal.
P/N GS11116 Gateway™ ATR Flow Through top plate (550um volume) with 45° ZnSe crystal.
P/N GS11118 Gateway™ ATR Thermostabilised Flow Through top plate (550um volume) with 45° ZnSe crystal.
P/N GS11139 Gateway™ ATR Water Heated Trough top plate with 45° ZnSe crystal.
P/N GS11155 Gateway™ ATR Electrically Heated Trough top plate with 45° ZnSe crystal.

The Gateway™ ATR accessory can be configured for the analysis of all types of samples (for example: solids, films, pastes, liquids, static or flow conditions and heating applications up to 200°C) over a wide spectroscopic range of frequencies using the different top plates and crystal options. As supplied, the Gateway™ ATR Accessory has been factory aligned and will only need minor adjustment after installation for optimization of light throughput and performance in your spectrometer.

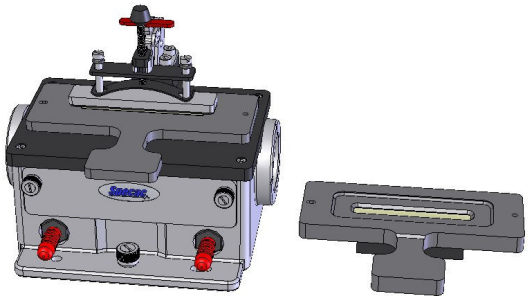


Fig 1. The Gateway™ 6 Reflection ATR Accessory Kit

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 possible you should follow a procedure for safe handling and containment of the type of sample to be used.

With respect to safety of use specifically to the Gateway™ ATR Accessory, this uses different crystal materials for the ATR crystal Trough, Flat and Flow Through Top Plate assemblies where a sample is bought into contact for analytical spectroscopic study. As standard, Zinc Selenide (ZnSe), germanium (Ge) and Silicon (Si) are the three crystal materials of choice that can be used.

Caution: *Out of these three different crystal types, ZnSe is the most potentially hazardous material with respect to toxicity risk in use and handling.*

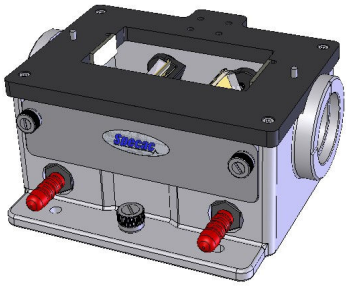
Both Ge and Si crystal materials can be considered relatively safe to use, although Ge may be harmful to the body if it is ingested in significant quantity. The general rule when working with **any** crystal material (and sample) **is to always wear gloves and safety gear** (e.g. safety spectacles) when handling to obviate the risk of contact with the skin.

Provided with each ATR crystal version of top plate assembly is a window material safety data sheet for the crystal material itself that can be consulted for safe handling. A copy of each of these datasheets can also be found in this Gateway™ ATR instruction manual in the **Notes on Cleaning** Section found on pages 42, 43 and 44.

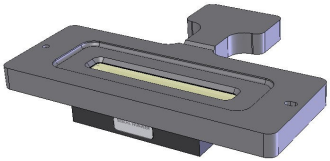
3. Checklist and Contents

The Gateway™ ATR Kit as P/N GS11165 is packed in protective wrappings within its container box. The box contains the following:

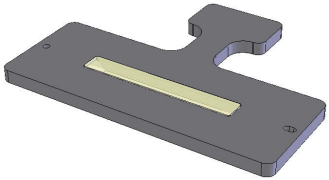
- 1 Gateway™ ATR incompartment optical unit – GS11170.



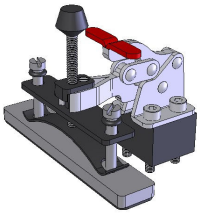
- 1 Trough top plate assembly with 45° ZnSe crystal – GS11166.



- 1 Flat top plate assembly with 45° ZnSe crystal – GS11133.

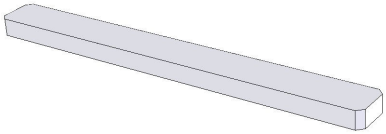


- 1 Clamp assembly – GS11171.



- 1 Pair of flexible purge bellow tubes – GS10707.

- 1 PTFE pad (used with trough plate and clamp).



- 1 Spatula (for spreading pastes or powders)
- 1 Allen key - 2.0 mm A/F.
- 1 Allen key - 2.5 mm A/F.
- 1 Allen key – 3.0 mm A/F.
- 1 User instruction manual for the Gateway™ ATR Kit.

Note: *An appropriate Benchmark™ baseplate for installation of the optical unit into the spectrometer is supplied separately outside the box of the Gateway™ Kit. A Benchmark™ Baseplate installation guide is also provided to follow for fitting of the appropriately supplied baseplate into the spectrometer.*

Carefully remove the items from their packaging and prepare the Gateway™ ATR Accessory for installation, alignment and use.

4. Preparing the Gateway™ ATR Accessory

Before the Gateway™ ATR Accessory is to be installed and optimized for its optical alignment within a particular spectrometer system to be used, it is necessary to fit the clamp assembly (**1**) to the optical unit (**2**) and also to fit either the trough (**3**) or flat (**4**) top plate crystal assembly to the optical unit (**2**).

The Clamp Assembly (GS11171)

The clamp assembly (**1**) is supplied separately to the Gateway™ optical unit (**2**). Please use the following instructions to fit the clamp assembly (**1**) to the Gateway™ optical unit (**2**).

Note: *To avoid any accidental damage to certain parts, the clamp assembly (**1**) should be fitted onto the Gateway™ optical unit (**2**) **without** the trough (**3**) or flat (**4**) top plate crystal assembly in position on the optical unit (**2**). Also, be careful when fitting the clamp assembly (**1**) that the mirrors (movable - **5**) and (fixed - **6**) on the optical unit (**2**) are not touched or scratched.*

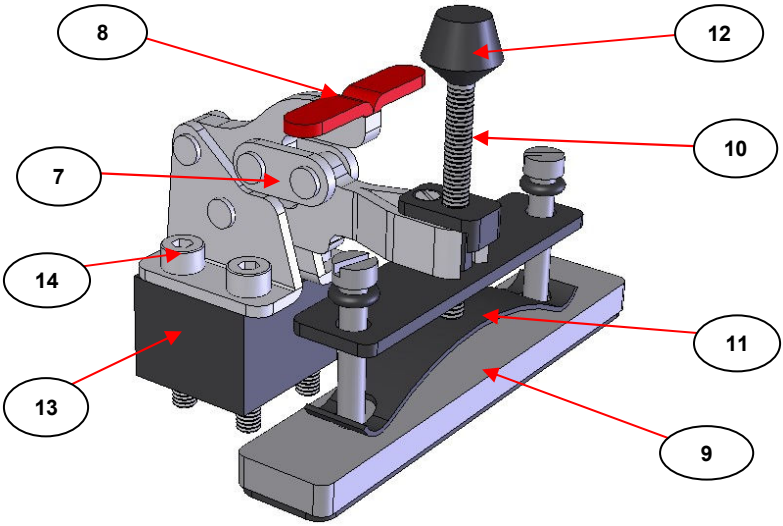


Fig 2. Gateway™ ATR Clamp Assembly

The complete Gateway™ ATR clamp assembly (**1**) consists of a movable (up and down) arm mechanism (**7**) which is locked into its down position when the top T-bar handle (**8**) is pushed fully down and closed. (As shown in **Fig 2.**) The arm mechanism (**7**), that is raised by pulling up on the T-bar handle (**8**) or lowered by pressing down on the T-bar handle (**8**), is connected to a pressure plate and pad assembly (**9**) with use of an adjustable screw (**10**) for tensioning of a spring bar (**11**). The pressure plate and pad assembly (**9**) provides the force against a solid sample (in either solid, powder or film forms) to bring its sampling surface into sufficient contact against the ATR crystal in a flat top plate assembly (**4**) in order to obtain an ATR spectrum for the sample. The screw (**10**) is turned in a clockwise direction by the rubber knob piece (**12**) to exert more force on the spring bar (**11**) and anticlockwise to reduce the force for tensioning of the spring bar (**11**), which in turn adjusts accordingly for a sample force contact to the ATR crystal.

The complete clamp assembly (**1**) is fitted to the optical unit (**2**) via a black coloured clamp support block (**13**) and four M4 x 20mm cap head screws (**14**).

Note: *It is important that the clamp support block (**13**) is fitted the correct way around on the optical unit (**2**) for correct positioning of the clamp assembly (**1**) for its force application and clearance of any fitted trough (**3**) or flat (**4**) top plate assembly. (See **Fig 4**).*

Fixing the Clamp Assembly to the Optical Unit

1) Take the clamp assembly (**1**) and push down on the clamp T-bar handle (**8**) to lower the pressure plate and pad (**9**) for a closed position. This allows for clear access in fixing of the clamp assembly (**1**) through the screw (**14**) holes via the support block (**13**) to the optical unit (**2**).

2) Align the four clamp assembly fixing screws (**14**) with the four holes in clamp assembly arm mechanism (**7**) to pass through these holes first before passage through the screw holes in the support block (**13**) and finally into the corresponding threaded fixing holes on the top surface plate (**15**) of the optical unit (**2**). (See **Fig 3**.)

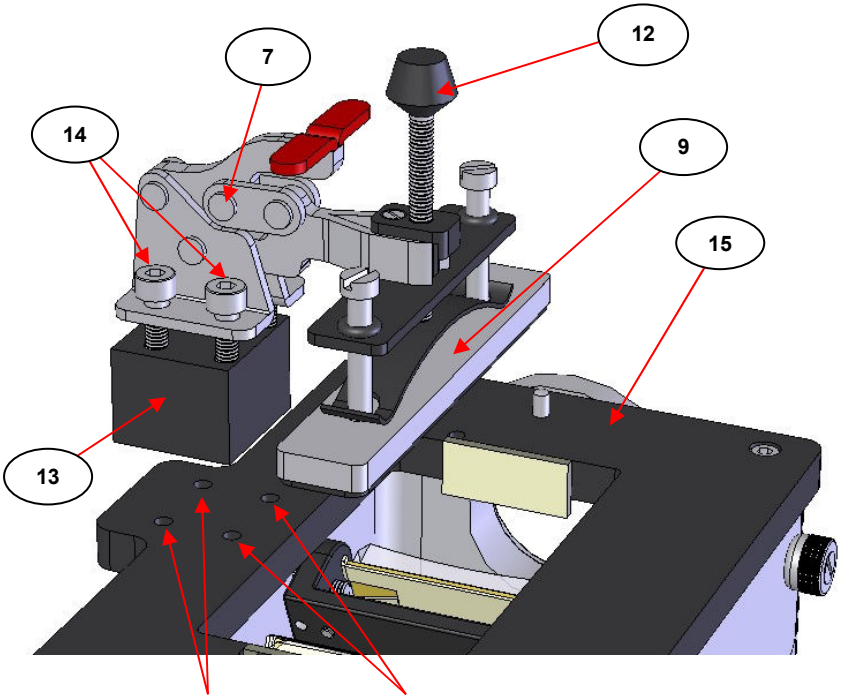


Fig 3. Fixing the Clamp Assembly to the Optical Unit

Note: *The support block (**13**) is orientated correctly as shown in **Fig 3**. when the two screw holes of the four that are closest to the edge of the support block (**13**) are facing towards the pressure plate and pad assembly (**9**) for fitting. (Also see **Fig 4**.)*

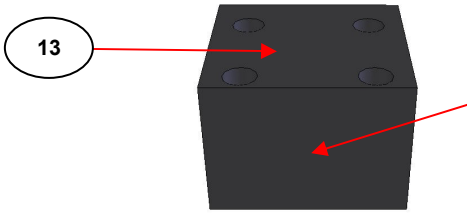


Fig 4. Orientation for Fitting of Support Block (13)

3) Using the larger 3mm A/F Allen key supplied, secure the clamp assembly (**1**) to the Gateway optical unit (**2**) by clockwise rotation for tightening of the four fixing screws (**14**) through the aligned holes of the clamp assembly (**1**) and support block (**13**). (**Do not over tighten**).

Fig 5. shows the clamp assembly (**1**) in its fully closed position with some partial force being applied to the spring bar (**11**) from the tensioning screw (**10**), as correctly fitted to the optical unit (**2**).

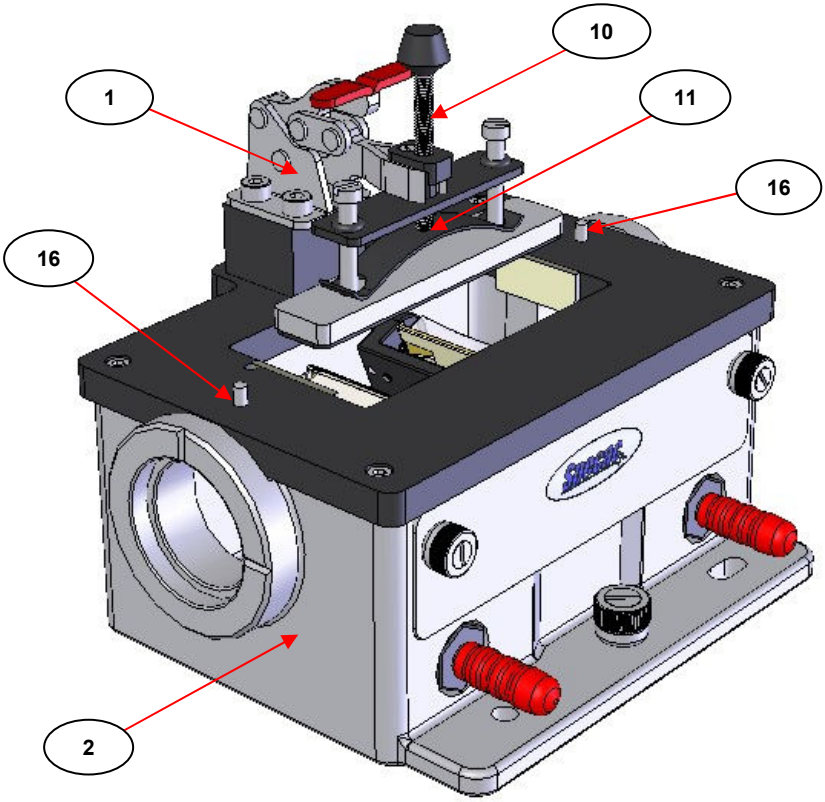


Fig 5. Gateway™ ATR Clamp Assembly Fitted to the Optical Unit

Fitting the Trough or Flat Top Plate Assembly

When the clamp assembly (**1**) has been fitted to the optical unit (**2**), the trough (**3**) or flat (**4**) top plate assembly can also be fitted to the optical unit (**2**) prior to installation and alignment of the Gateway™ ATR Accessory.

Note: *The trough (**3**) or flat (**4**) top plate assembly can be fitted to and removed from the optical unit (**2**) when it has been installed on to its Benchmark™ baseplate within the spectrometer sample compartment, but either top plate assembly **must be fitted** for a necessary optimum alignment of the Gateway ATR accessory to be made*.

On either side of the top surface plate (**15**) of the optical unit (**2**) there are two location pins (**16**). (See **Fig 5**.) At each end of the trough (**3**) and flat (**4**) top plate assemblies there is a round hole and a slot hole that passes all the way through the metalwork of these top plate assemblies. The top plate assemblies are fitted into correct position onto the top surface (**15**) of the optical unit (**2**) from alignment of the round and slot holes of the plates (**3**) and (**4**) with these location pins (**16**). (See **Fig 6**.)

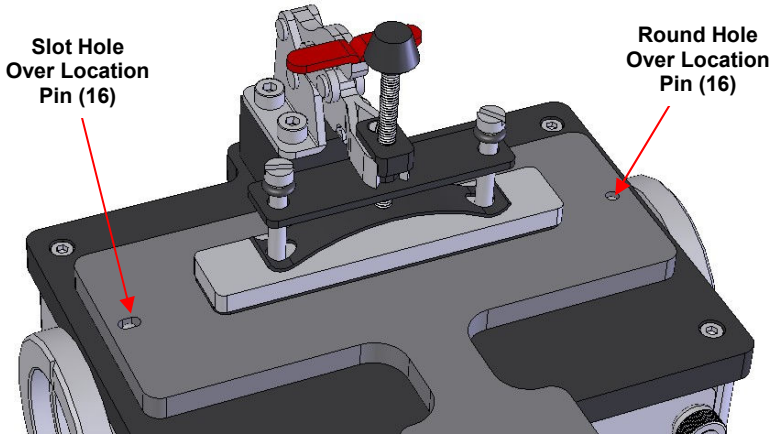


Fig 6. Flat Top Plate and Clamp Assembly Employed as Fitted to the Gateway™ ATR Optical Unit

In fitting of either top plate assembly into position over the location pins (**16**), the pressure plate and pad assembly (**9**) of the clamp assembly (**1**) must be lifted up and away to allow for the clearance in fit. When the trough top plate assembly (**3**) is fitted, the clamp assembly (**1**) for any sample force contact is normally not needed for any liquid sampling so can be kept raised. If the flat top plate assembly (**4**) is being used, once it has been fitted correctly and after any solid sample type has been placed over and to cover the entire ATR crystal surface area, the clamping mechanism is employed to force the sample into suitable contact for an ATR spectral measurement to be made.

Trough (3) and Flat (4) Gateway ATR Top Plates

The trough (**3**) and flat (**4**) top plate assemblies provided with the Gateway™ ATR Accessory have ZnSe ATR crystals (**17**) (P/N GS11145) supplied in place for use. There are further crystal options of Ge (P/N GS11147) and Si (P/N GS11146) available for use in both trough (**3**) and flat (**4**) top plate assemblies. The crystals (**17**) have been sealed into position by epoxy resin gluing for any flat (**4**) top plate assembly, but in any trough (**3**) or **flow through top plate** assembly the crystal (**17**) type options are removable. They are sealed for sample containment over the ATR crystal (**17**) surface within these top plate assemblies from use of specific gaskets made of Isolast or Kalrez material.

The Trough Top Plate Assembly

The ATR crystal (**17**) in the trough (**3**) top plate assembly is held in place within the surrounding plate metalwork (**18**) from a pressure bar (**19**) that pushes against the underside of the crystal (**17**) via a lead pad (**20**) and seals the crystals (**17**) top surface against an Isolast gasket (**21**). A central M4 x 5mm grub screw (**22**) within a base support plate (**23**) is tightened to provide the force to the pressure bar (**19**) for sealing of the whole assembly of parts. The base support plate (**23**) is held in place by four M3 x 12mm cap head screws (**24**). This design for a trough (**3**) top plate assembly allows for removal of the crystal (**17**) for cleaning or for a change of crystal (**17**) completely if damage has been sustained or for use of an optional ATR crystal (**17**) material. (See **Fig 7**. – Underside View.).

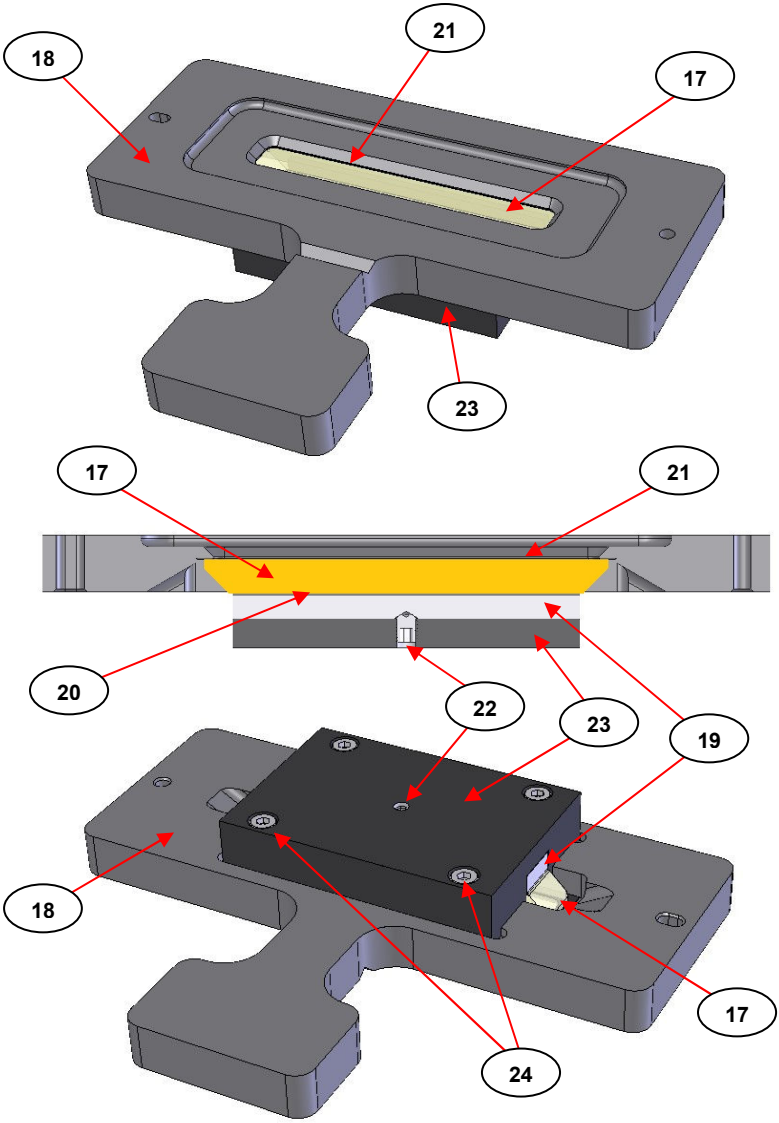


Fig 7. Gateway™ ATR Accessory Trough Top Plate Assembly, Top, Side (Cross Section) and Underside Views

Crystal Removal from the Trough Top Plate Assembly

If the ATR crystal (**17**) from the trough (**3**) top plate assembly needs to be removed for thorough cleaning or has become damaged and needs replacing by a new crystal material of the same type or an alternative crystal material, please use the following procedure.

1) Lay the complete trough (**3**) top plate assembly upside down on a clean flat surface covered with soft tissue. Using the 2mm A/F Allen key supplied, turn anticlockwise to loosen the central M4 x 5mm grub screw (**22**) that pressurizes the crystal assembly of parts, but do not remove it from the base support plate (**23**).

2) Using the 2.5mm A/F Allen key supplied unscrew the four M3 x 12mm cap head screws (**24**) that secure the base support plate (**23**) to the trough top plate metal work (**18**) and remove the support plate (**23**) that is still holding the grub screw (**22**). Access can now be gained to remove the pressure bar (**19**) and the lead pad (**20**) that is in contact with the underside of the crystal (**17**).

3) Carefully remove the pressure bar (**19**) and lead pad (**20**) away from the crystal (**17**).

Important: *Take care not to damage the thin lead sheet of the pad (**20**) between the pressure bar (**19**) and the crystal (**17**), as it will need to be re-used for correct installation of a new crystal (**17**).*

4) Turn the trough (**3**) top plate assembly, still holding the crystal (**17**), the correct way up to gently remove the crystal (**17**) out of its retaining cavity. Be careful that the crystal (**17**) is not being loosely held in place by the top Isolast gasket (**21**) seal and could fall out accidently when the trough (**3**) top plate assembly is turned the correct way up. Usually though, it may be necessary to loosen the crystal (**17**) from the Isolast gasket (**21**) seal by exerting a small pressure on either ends of the crystal from inside the trough area of the top plate metal work (**18**) and pushing down from above. Do not press too hard in case the crystal (**17**) breaks. (ZnSe, in particular, is a hard, but brittle crystal material.)

Tip: *Using gloves and with the fore fingers of both hands covered with soft lens tissue to prevent any scratching to the crystal (**17**) surface, exert light even pressure on both ends of the crystal (**17**) simultaneously from inside the trough.*

With the crystal safely removed, if it needs cleaning please refer to the Notes on Cleaning pages 40 and 41.

Changing the Isolast Gasket of the Trough Top Plate Assembly

The Isolast gasket (**21**) seals around the top of the ATR crystal (**17**) surface and between the trough area of the trough top plate metalwork (**18**). It may be necessary to change the Isolast gasket (**21**) seal if there is leakage of liquid sample away from the crystal surface area, even after tightening of the pressure bar (**19**), or if the gasket (**21**) has become contaminated and/or degraded from use. However, with any removal of the ATR crystal (**17**), it is necessary to check the condition of the Isolast gasket (**21**) before any crystal (**17**) replacement, so the Isolast gasket (**21**) could be changed at this stage if desired.

Note: *The Isolast gasket (**21**) seal usually only needs changing very occasionally if careful use of the trough (**3**) top plate assembly is observed. An Isolast gasket (**21**) replacement from P/N GS11167 will be required.*

To replace the Isolast gasket (**21**) in a trough (**3**) top plate assembly please use the following procedure.

1) Carefully remove the ATR crystal (**17**) as described in the procedure on pages 16 and 17.

2) Remove the Kalrez gasket (**21**) seal from the recess of the trough top plate metal work (**18**). (See **Fig 8**.) Depending on its condition it may come away in one piece or in fragments, but ensure that all parts of the old Isolast gasket (**21**) are removed from the area.

3) Using a solvent such as acetone to moisten a soft, lint free cloth, thoroughly clean the areas of the trough top plate metalwork (**18**) where the Isolast gasket (**21**) had made contact.

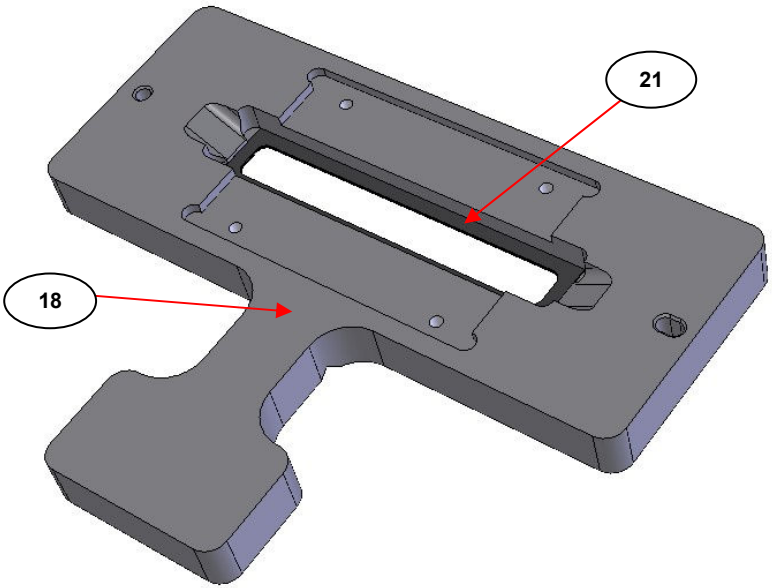


Fig 8. Isolast Gasket in the Trough Top Plate Assembly

4) Place a new Isolast gasket (**21**) seal (from P/N GS11167) in place of the original seal, centrally in the recessed back face of the trough top plate metal work (**18**). (See **Fig 8.**) Ensure that the gasket (**21**) lays flat within and tight to the edges of the recess.

The trough (**3**) top plate assembly is now ready to accept a new replacement or cleaned ATR crystal (**17**) for re-assembly to a working and usable condition.

Re-assembly of the Trough Top Plate Assembly

After an ATR crystal (**17**) has been removed from the trough top plate assembly and the Isolast gasket (**21**) (new replacement or the cleaned original) has been positioned correctly in the trough top plate metalwork (**18**), re-assembly of the trough (**3**) top plate can commence.

Please use the following procedure.

1) Lay the top plate metalwork (**18**) with the Isolast gasket (**21**) fitted, upside down on a clean, flat, soft tissue covered surface as shown at **Fig 8**.

2) Carefully take the ATR crystal (**17**) and gently position it with the longest surface downwards onto the Isolast gasket (**21**) seal. Be very careful to ensure that the crystal (**17**) is centrally positioned in the recess before replacing the lead pad (**20**), pressure bar (**19**) and base support plate (**19**).

3) Carefully position the lead pad (**20**) completely over the shorter underside surface of the crystal (**17**) and then place the pressure bar (**19**) with the central dimpled recess side uppermost on top of the lead pad (**20**). Ensure that these parts are in **exact central alignment** with the ATR crystal (**17**).

4) Take the support plate (**23**) (with the partially loosened grub screw (**22**) in place) and very carefully place it the correct way up over the pressure bar (**19**) and lead pad (**20**) and allow it to locate into the larger rectangular recess on the underside of the trough (**3**) top plate metalwork (**18**). When the crystal (**17**) has been positioned correctly and centrally it will be sitting in the grooved recess of the support plate (**19**) with just the angled faces of the ATR crystal (**17**) showing at each end. (See underside view at **Fig 7**.)

5) Fix the support plate (**23**) to the trough top plate metalwork (**18**) using the four M3 x 12mm screws (**24**) turning them clockwise with the 2.5mm A/F Allen key until tight. At this stage the assembly of crystal (**17**), lead pad (**20**) and pressure bar (**19**) will still be a loosish fit so when tightening the screws (**24**), be careful that these parts do not move out of their central alignment.

6) When the four screws (**24**) are tight, finally use the 2.0mm A/F Allen key to turn the central grub screw (**22**) until it just begins to tighten against the recessed dimple in the pressure bar (**19**). At this stage it should then only be necessary to turn the grub screw a maximum of a further 1/8th turn (45°) clockwise to provide the

correct pressure for holding the ATR crystal (**17**) in position and for its upper surface edge to seal tight against the Isolast gasket (**21**).

Warning! *Do not over tighten the grub screw (**22**) as you risk damage to or breakage of the ATR crystal (**17**).*

Test the trough (**3**) top plate assembly for any fluid leaks past the new seal with a 50% mixture of acetone/water in the trough recess and allow the top plate assembly to stand on some tissues for 30 minutes.

Important: *Do not leave the filled trough (**3**) top plate assembly plate on the Gateway™ optical unit (**2**) when testing. If there is a leak the fluid would fall to contaminate the optical components below.*

If a leak is observed, check for the possible causes (see below). Remove any remaining acetone/water fluid in the trough and dry the assembly completely using soft lens tissues, paying particular care when cleaning around or near to the ATR crystals (**17**) exposed surfaces.

Tip: *Carefully remove any liquid film between the pressure bar (**19**), lead pad (**20**) and the underside of the crystal (**17**).*

Rectify the possible cause of the leak, re-assemble and re-test.

Possible Causes of Leaks

The assembly of parts is not screw tightened sufficiently together.
The trough (**3**) top plate assembly recess (where a fluid is placed), has not been sufficiently cleaned.
The Isolast gasket (**21**) seal is not lying flat and central.
The Isolast gasket (**21**) seal is old/worn and requires replacement.
The ATR crystal (**17**) is defective (e.g. chipped, dirty, warped).
The ATR crystal (**17**) is not symmetrically and centrally located.

The Flat Top Plate Assembly

The flat (**4**) top plate assembly is shown for its top, side (cross section) and underside views in **Fig 9**.

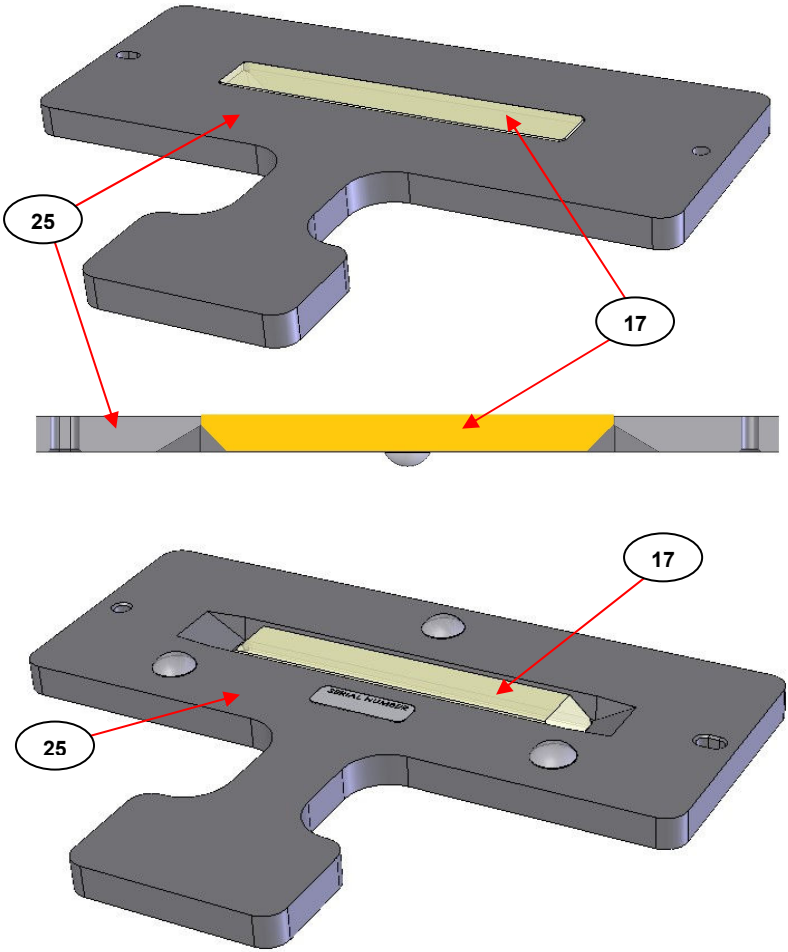


Fig 9. Gateway™ ATR Accessory Flat Top Plate Assembly, Top, Side (Cross Section) and Underside Views

The ATR crystal (**17**) in the flat (**4**) top plate assembly is held in place within its surrounding plate metalwork (**25**) using an epoxy resin glue. From this design it means that the ATR crystal (**17**) is **not removable** for replacement in a flat (**4**) top plate assembly. If damage is sustained to the ATR crystal (**17**), a **complete new flat (4) top plate assembly** needs to be replaced. These complete new flat top plate assemblies with the appropriate ATR crystal (**17**) fitted are provided as P/N's GS11133 (ZnSe – 45°), GS11134 (Si – 45°) and GS11135 (Ge – 45°).

Warning: *Because an epoxy resin is used to bond the ATR crystal (**17**) in the flat (**4**) top plate assembly, chlorinated reagents and other halogen related substituted organic solvents that will dissolve the epoxy resin should be avoided when cleaning the crystal as fitted in to these top plate assemblies. Strongly acidic and basic solutions should also be avoided when using ZnSe crystals (**17**). (See **Notes on Cleaning** pages 40 and 41).*

The PTFE Pad

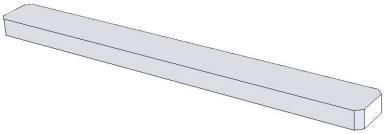


Fig 10. PTFE Pad

The PTFE pad supplied (see **Fig 10**.) is for use with the trough (**3**) top plate assembly with **solid** samples. The pressure required from the clamping assembly (**1**) is transferred to a solid sample via this PTFE pressure pad being placed on top of the sample. Its thickness compensates for the recess depth to the ATR crystal (**17**) surface created by the surrounding top trough plate metalwork (**18**). However, the trough (**3**) top plate assembly is not as efficient as a flat (**4**) top plate assembly for the analysis of very thin films and powders using the clamp assembly (**1**), because of the crystal (**17**) being slightly recessed into the metalwork (**18**). Therefore, the flat (**4**) top plate assembly is recommended for samples of this type to use with the clamp assembly (**1**) alone, **without** the PTFE pad.

5. Installation of the Gateway™ ATR Accessory Using the Benchmark™ Baseplate

The Gateway™ ATR Accessory is base plate mounted. The Gateway™ optical unit (**2**) fixes to the Benchmark™ baseplate via a central fixing thumbscrew (**26**). (See **Fig 11**.) Usually, a spectrometer's own 3" x 2" slide plate sample mount will have to be removed before the Gateway™ ATR Accessory can be installed on the Benchmark™ baseplate.

Note: *It is best to install the Benchmark™ baseplate into the spectrometer sample compartment first before attaching the Gateway™ ATR Accessory into position.*

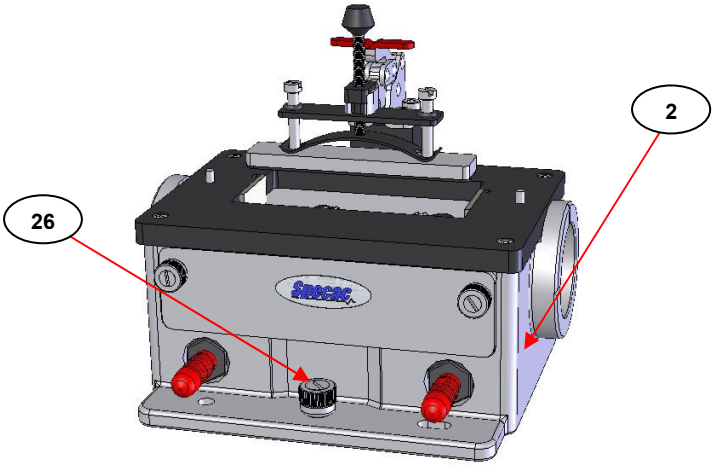


Fig 11. Fixing Thumbscrew of the Gateway™ ATR Optical Unit for Attachment to the Benchmark™ Baseplate

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

6. Alignment of the Gateway™ ATR Accessory

When the Gateway™ ATR clamp assembly (**1**) has been fitted to the optical unit (**2**) and the whole assembly of parts (with or without a crystal top plate assembly (**3** and **4**) already fitted), is installed on the Benchmark™ baseplate in the sample compartment of the spectrometer, the Gateway™ ATR Accessory can be optically aligned for an optimal throughput of light energy for use in this particular spectrometer system.

Note: *For alignment, a crystal top plate assembly (**3**) or (**4**) MUST be fitted correctly to the optical unit (**2**). It is important to check that the top plate assembly is seated evenly to allow for a consistent beam path through the ATR system of optical components.*

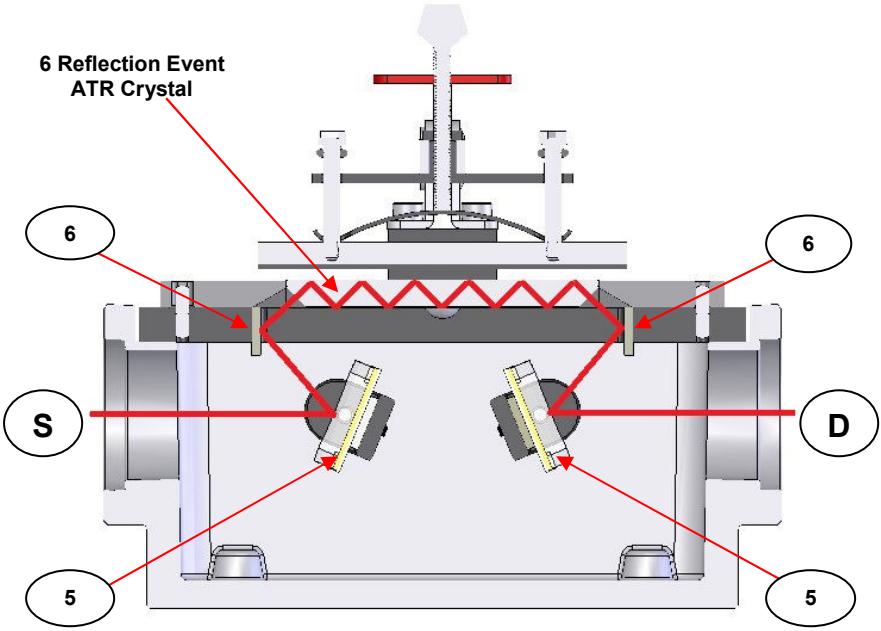


Fig 12. Light Beam Passage in Gateway™ 6 Reflection ATR (Cross Section Cutaway for Internal Optics)

The beam passage of light through the optical components of the Gateway™ ATR Accessory is symmetrical, depending upon whether the light passes from the source (**S**) to the detector (**D**) of the spectrometer in a left to right (**LR**) or right to left (**RL**) beam direction through the sample compartment. For the purposes of explanation, the beam passage sequence for the cross section cutaway image at **Fig 12.** is shown for an (**LR**) beam direction spectrometer system with a flat top plate assembly (**4**) fitted to the optical unit (**2**), as determined from the source (**S**) and detector (**D**) positions.

From the source (**S**), the light strikes the input movable mirror (**5**) (for rotation and tilt) and is directed onto a fixed input mirror (**6**). Light from this fixed input mirror (**6**) nominally passes straight through the 45° angled face of the ATR crystal to set up a sequence of 6 surface reflection events on the top (longer) side of the ATR crystal that is in contact with any liquid or solid sample, as the light traverses through the crystal. The light beam exits the other 45° angled face of the crystal to strike the output fixed mirror (**6**) and then onto the output movable mirror (**5**) before reaching the detector (**D**).

Front Cover Plate Removal

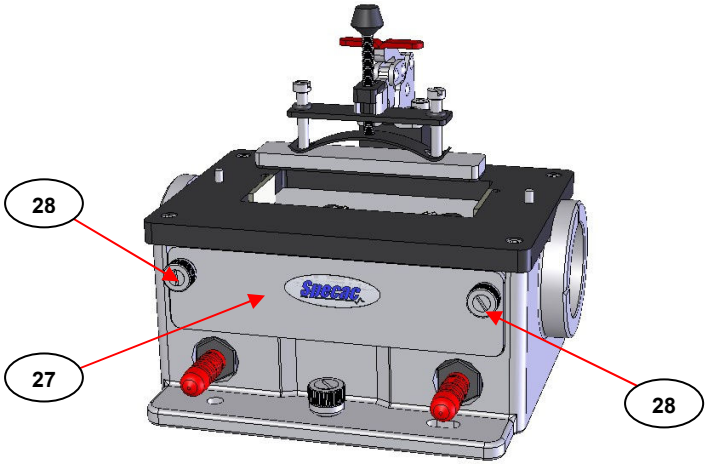


Fig 13. Front Cover Plate on the Optical Unit

To gain access to the movable mirrors (**5**) within the optical unit (**2**) for alignment, the front cover plate (**27**) is removed. There are two fixing thumbscrews (**28**) holding the cover plate (**27**) in position that are loosened by pushing them in and turning anticlockwise (see **Fig 13**.) (The thumbscrews (**28**) will remain captive in the cover plate (**27**).)

Alignment Procedure

With removal of the front cover plate (**27**), the two movable mirrors (**5**) will be accessible as shown from **Fig 14**.

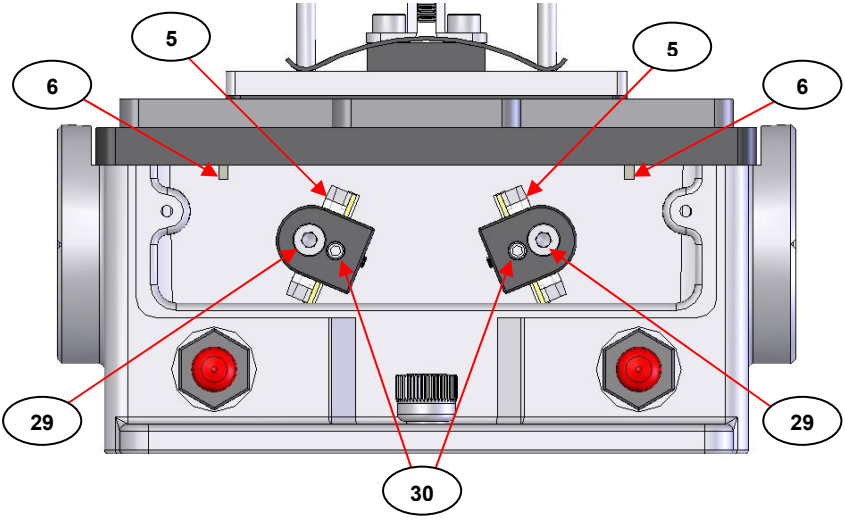


Fig 14. Gateway™ ATR Movable Mirrors for Rotate and Tilt

On the movable mirror (**5**) carriage frame assembly are an M4 x 5mm cap head screw (**29**) and M4 x12mm cone pointed grub screw (**30**).

The mirrors (**5**) can be adjusted for their **rotation** via the M4 x 5mm cap head screw (**29**) and for their **plane of tilt angle** via the M4 x 12mm cone pointed grub screw (**30**) to direct the beam of light accordingly from the input movable mirror (**5**) to the input fixed mirror (**6**) and from the output movable mirror (**5**) to the detector (**D**).

Begin alignment of the Gateway™ ATR Accessory from the following procedure:

1) Establish a beam of light energy level throughput to be monitored from the spectrometers own detection system. The Gateway™ ATR Accessory is aligned in the factory before despatch. On installation, some transmitted signal will be recorded on the spectrometer's energy monitoring system, but it will be necessary to maximize the optical throughput by adjusting the mirrors (**5**) for rotate (**29**) and tilt (**30**) in the Gateway™ ATR Accessory optical unit (**2**) before use.

2) Only small rotation and tilt adjustments should be required. Start first with the **output** movable mirror assembly (**5**) for your beam direction (**LR** or **RL**) spectrometer. Use the 3mm A/F Allen key supplied in the screw (**29**) to **rotate** the mirror (**5**) first. Rotate the mirror (**5**) until an optimum (maximum) signal is registered at the detector (**D**). If you pass through a peak maximum from turning the screw (**29**) in one direction, turn in the opposite direction until a peak maximum is achieved again.

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

3) Having established an optimum throughput from rotation of the output mirror (**5**), now move to its **tilt** adjustment setting using the 2mm A/F Allen key supplied, in the grub screw (**30**). Tilt the mirror until an optimum (maximum) signal is registered at the detector (**D**). If you pass through a peak maximum from turning the grub screw (**30**) in one direction, turn in the opposite direction until a peak maximum is achieved again.

Note: *If the grub screw (**30**) is turned too far anticlockwise it will dislocate from the back of the spring mount of the mirror (**5**). If this happens, turn the grub screw (**30**) anticlockwise to clear fully from the spring mount. Push the spring mounted mirror (**5**)*

*away from the black anodized mirror mount arm carriage assembly and turn the grub screw (**30**) clockwise until it re-sets again behind the mount.*

4) Having established an optimum signal throughput from both sequential rotation and tilt adjustments of the **output** mirror (**5**), repeat the rotation and tilt adjustment steps 2) and 3), but for the **input** mirror (**5**).

5) When the rotation and tilt adjustments have been made to the input mirror (**5**), it is necessary to return to the **output** mirror (**5**) again for possible very fine re-alignment of this mirror for its rotation and tilt position setting to obtain an overall optimum light throughput. (A "rebalancing" of the incident light input mirror (**5**) settings.)

When steps 2) to 5) have been completed alignment of the Gateway™ ATR Accessory for the particular spectrometer system into which it has been installed will have been achieved.

Note: *The energy throughput varies greatly with spectrometer design and set-up. When all adjustments have been properly carried out and the Accessory is well aligned, a transmission energy in the region of 20-30% when compared to an unobstructed beam of 100% should be achieved with a new ZnSe crystal. This figure will fall to about half when the trough top plate assembly (**3**) is filled with an aqueous solution. When a different top plate is used, it may be necessary to slightly realign the mirrors (**5**) for their settings in the Gateway™ optical unit (**2**).*

Refit the front cover plate (**27**) back into position to prevent the mirror assembly settings from being accidently altered further. After initial alignment for optimal settings of the mirrors (**5**) has been established, the Gateway™ ATR Accessory can be removed and re-installed repeatedly into the same spectrometer for correct positioning without any change in the throughput performance. If there is a change of performance throughput, there may be a problem requiring realignment of the mirrors, although there may be a fault with the ATR crystal material and /or mirror surfaces because of dirt, dust, corrosion or other damage. (See **Notes on Cleaning**, pages 40 and 41.)

7. Features on the Gateway™ Optical Unit

Purging the Optical Unit

If for any ATR experimentation it is necessary to reduce the effects of any residual water vapour or carbon dioxide content in the local atmospheric conditions, a purge gas such as nitrogen (N_2) can be introduced into the Gateway™ ATR optical unit (**2**) via use of the purge port connections (**31**) at the front under the front cover plate (**27**). These purge ports (**31**) are barbed hose type connectors with red plastic cover tube caps. To introduce a flow of a purge gas, the red plastic cover tube is removed and appropriate tubing (e.g. 1/4" O.D, 1/8" I.D. black silicone rubber tubing) is push fitted over the barbed hose connection. Either of the two purge port connections (**31**) can be used as the **inlet** port for any purge gas flow. (See **Fig 15.**)

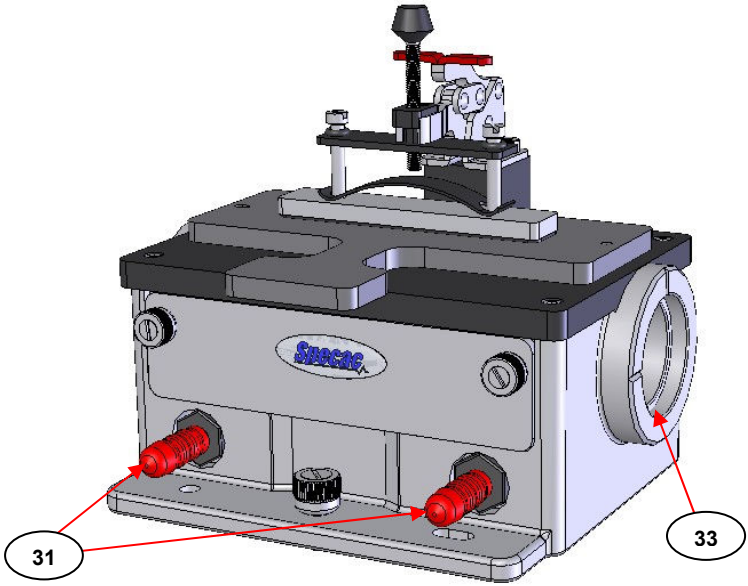


Fig 15. Gateway ATR™ Purge Port Connections and Aperture Ports

To purge the system and maintain a local N_2 gas environment within the optical unit (**2**), it is necessary to enclose the IR beam between the ends of the Gateway™ ATR optical unit (**2**) and the sample compartment of the spectrometer itself. This is done with the flexible purge bellows (**32**) (P/N GS10707 as supplied) when the purge bellows (**32**) have been trimmed appropriately for fit. The bellows (**32**) are fitted over the aperture port rings (**33**) of the Gateway™ optical unit (**2**) for one of their ends and abut tightly against the source (**S**) and detector (**D**) sample compartment bulkhead faces of the spectrometer for the other end. (See **Fig 15** and **Fig 16.**)

Note: *When using infrared spectrometers fitted with infrared transmitting windows at the source (**S**) and detector (**D**) sides to isolate the sample compartment from the rest of the instrument, the Gateway™ ATR Accessory can be purged quickly and independently via use of the purge ports (**31**) and the purge bellows (**32**). If the sample compartment **is not isolated** from the rest of the instrument in this way, it may take a while to purge the entire system using a flow of purge gas into the optical unit (**2**) alone fitted with the purge bellows (**32**).*

Fitting the Purge Bellows (GS10707)

If the gap/distance between the side of the optical unit (**2**) and the spectrometer source (**S**) and detector (**D**) sample compartment bulkhead faces is small, the purge bellows (**32**) can be cut to size for a better compressible fit. It is best to cut all the way around and in between one of the ridged grooves of the purge bellows (**32**) moulding itself, when shortening the length for fit. (See **Fig 16**.)

Tip: *It is recommended that the purge bellows (**32**) are fitted during any use to help stabilize the spectrometer/instrument for background atmospheric conditions even if purging of the optical unit (**2**) via the purge ports (**31**) is not to be carried out.*

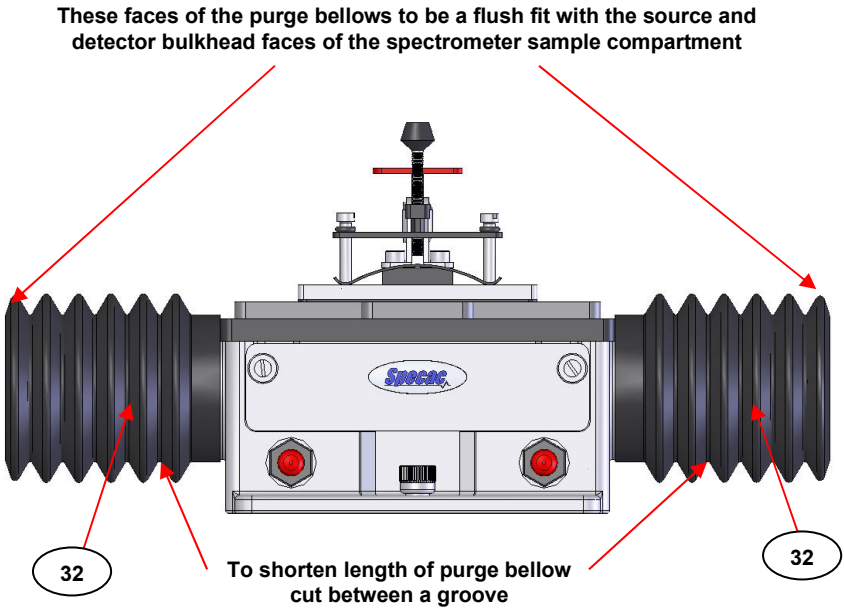


Fig 16. Gateway™ ATR Accessory fitted with Purge Bellows

1) With the Benchmark™ baseplate secured and the Gateway™ ATR Accessory in position, measure the distance between the spectrometer source and detector bulkhead/side walls and the side end faces of the Gateway™ optical unit (**2**). (This is dimension "'X").

2) Using a sharp blade, cut lengths of the flexible purge bellows (**32**) which are equivalent to the measured length ("X") allowing for an additional 10mm for each length to cut. (e.g. X + 10mm.)

3) Unscrew the Gateway™ ATR optical unit fixing thumb screw (**26**) from the Benchmark™ baseplate and remove the Gateway™ ATR Accessory from the sample compartment.

4) Fit the shortened flexible purge bellows (**32**) over both end aperture port rings (**33**) on the Gateway™ ATR optical unit.

5) Replace the Gateway™ ATR Accessory with the fitted purge bellows (**32**) back onto the Benchmark™ baseplate. Ensure that the compressed purge bellows (**32**) are not obstructing the optical beam and retighten the fixing thumb screw (**26**) to secure the Gateway™ ATR Accessory back into position.

Fitting a Polarizer

Specac's FTIR Infrared polarizers (P/N GS12000 Series) can be inserted into the aperture port rings (**33**) on the Gateway™ ATR accessory. The polarizers push-fit in these ring apertures and can be rotated to the desired orientation between 0° and 90° angles for the polarizer grid. Please follow the instructions for fitting of any GS12000 Series polarizer from its own user instruction manual.

Note: *For the actual installation of a polarizer into the beam path from a source (**S**) of light to the sample and then to a detector (**D**), it does not matter if the polarizer is positioned before light has interacted with a sample (between the source and sample) or after interaction (between the sample and detector). The result of discriminating and measurement for a particular plane of polarized light from an angular setting of the polarizing grid deposited on the substrate material, when a polarizer is introduced into the beam passage sequence, is the same. With respect to the aperture ports (**33**) on the Gateway™ optical unit (**2**), either one can be used for installation of a GS12000 Series polarizer.*

8. Sampling Using the Gateway™ Accessory

Important Note for Usage!

The ZnSe, Ge and Si crystals in the trough (**3**) or flat (**4**) top plate assemblies for the Gateway™ ATR Accessory have a specific hardness and chemical resistance and should be treated accordingly. The crystals have been sealed into position by epoxy resin gluing for any flat (**4**) top plate assembly, but in any trough (**3**) or flow through top plate assembly the crystal types are removable. They are sealed for sample containment over the ATR crystal surface within these top plate assemblies from use of gaskets made of Isolast or Kalrez material. Check on the chemical nature of a sample before potentially damaging materials are brought into contact with the specific ATR crystal being used for either the flat (**4**) or trough (**3**) top plate assembly and also affecting the epoxy glue or gasket materials respectively.

Be careful not to put a “point load” on the ATR crystal from hard or abrasive solid samples when using the flat (**4**) top plate assembly with the clamp assembly (**1**). When using samples such as rubbers and polymer sheeting there should be no problem and normal clamp assembly (**1**) pressures may be used. The measurement of thin films on plastic substrates should present a minimal risk for crystal damage, but care should be taken if the substrate material is hard and metallic in nature. Similarly, gritty or non-homogenous powder samples for their form should be avoided for pressure clamping against the ATR crystal.

After closing and locking the clamp assembly arm mechanism (**7**) apply any pressure to the tensioning spring bar (**11**) and in turn to the pressure plate and pad (**9**) from gradual turning of the screw (**10**). For some sample types, if there is a risk of damage to the ATR crystal, it is advisable to try and acquire an infrared spectrum to see if an acceptable load has already been applied for sample contact from this gradual approach, rather than application of a maximum tensioning setting to the spring bar (**11**) and pressure plate and pad (**9**) because the screw (**10**) has already be wound fully tight from a maximum allowable clockwise rotation.

Preparing the Gateway™ ATR Accessory for Analysis

For any of the ZnSe, Ge and Si crystal top plate assemblies, the active sampling area extends across the whole of the ATR crystal surface. It is therefore preferable to have enough liquid or solid sample type to completely cover the ATR crystal to utilise the maximum energy throughput available for the Gateway™ ATR Accessory.

For a trough (**3**) or flow through top plate assembly, the top surface of the ATR crystal (**17**) needs only a thin film of liquid, fully covering the crystal surface, to give a good ATR spectrum as the depth of penetration into the sample will only be a few microns. For the ZnSe crystal trough (**3**) top plate assembly as supplied with the Gateway™ ATR Accessory, a plastic Pasteur pipette is the safest way to fill the sample trough and for sucking out most of the sample prior to removal of the top plate assembly and washing clean with a suitable solvent. Oily or greasy samples should be spread onto the crystal surface with a soft brush or the plastic spatula supplied and removed with a suitable solvent and soft lens paper tissue. (See **Notes on Cleaning**, page 40).

For a flat (**4**) top plate assembly in its installation and removal for cleaning between samples, it is necessary that the pressure plate and pad (**9**) of the clamp assembly (**1**) is raised and lowered using the T-bar handle (**8**) of the arm mechanism (**7**). It is also advisable to turn the pressure tensioning screw (**10**) anti-clockwise a few times, reducing any pressure that has been applied to the crystal (**17**) for sampling, before using the clamp T-bar handle (**8**) to release and lift up the pressure plate and pad (**9**).

Note: *The pressure tensioning screw (**10**) is also used to apply a load to the pressure plate and pad (**9**) via the spring bar (**11**) when a solid sample is to be bought into good contact with the ATR crystal (**17**). The Neoprene rubber faced pad on the pressure plate and pad assembly (**9**) allows for good contact of films and powder samples.*

Apply any solid film sample carefully over the ATR crystal (**17**) or cover the crystal (**17**) carefully and to an even depth with a powder sample from use of the plastic spatula supplied. (To clean the flat (**4**) top plate assembly please see **Notes on Cleaning**, page 40).

Spectral Collection Using the Gateway™ Accessory

The procedure for collecting a sample ATR spectrum requires first obtaining a background spectrum as a reference using the Gateway™ ATR Accessory with **no sample** in position on the ATR crystal (**17**) for **any** top plate assembly as fitted to the Gateway™ optical unit (**2**) and then repeating the procedure with a sample in position.

Collecting a Background Spectrum

1. Choose a trough (**3**), flat (**4**) or flow through top plate assembly to be used on the Gateway™ ATR optical unit (**2**) fitted with its clamp arm assembly (**1**).

2. Attach the Gateway™ ATR Accessory to the installed Benchmark™ baseplate and fit the chosen ATR top plate assembly to the optical unit (**2**). (Ensure that the clamp assembly arm mechanism (**7**) is fully raised to allow for correct fitting of the top plate assembly.)

3. The Gateway™ ATR Accessory should already be aligned to register an acceptable signal throughput with a top plate assembly in position. Having ensured that the ZnSe, Ge or Si ATR crystal (**17**) is clean, proceed to collect a background spectrum using any preferred acquisition conditions set on the spectrometer.

Note: *If using a **flat (4) top plate assembly** the clamp assembly pressure plate and pad (**9**) **does not need to be in contact** with the ATR crystal (**17**) to record a background spectrum.*

Collecting ATR Spectra of Powder Samples

When analyzing for a **powder** sample it is best to use the flat (**4**) top plate assembly fitted to the optical unit (**2**) and the clamp assembly (**1**). The procedure for spectral collection is as follows:-

1. Collect a **background spectrum** as described in steps 1 to 3 (page 35).

2. Take the powder sample and spread it very carefully to form a level surface that covers the entire surface area of the ZnSe, Ge or Si ATR crystal (**17**). 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 (**17**) when being pressed.

3. Ensure that the tension of the spring bar (**11**) is loose by having the tensioning screw (**10**) on the clamp assembly (**1**) almost fully raised. Carefully lower the pressure plate and pad (**9**) by pressing down on the T-bar handle (**8**) to lock down the arm mechanism (**7**) in the closed position and to loosely bring the pressure plate and pad (**9**) into contact with the powder sample.

4. Begin turning the tensioning screw knob (**12**) clockwise to exert a force on the spring bar (**11**) and to, in turn, increase the force against the pressure plate and pad (**9**). Continuation of rotation of the tensioning screw (**10**) until it stops turning will provide for a maximum load setting against the sample forcing it against the ATR crystal (**17**).

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the tensioning screw (**10**) 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 tensioning screw (**10**).*

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

6. To remove or change the sample, release the load/pressure on the sample by turning the tensioning screw knob (**12**) anticlockwise to retract the screw (**10**) away from the spring bar (**11**) and the pressure plate and pad (**9**). When the spring bars (**11**) tension is loose, lift up the clamp arm mechanism (**7**) by pulling up on the T-bar handle (**8**) to remove the pressure plate and pad (**9**) away from the sample.

7. Clean the powder sample carefully away from the ATR crystal (**17**) (avoid the possibility of scratching the ATR crystal (**17**) surface, particularly if using ZnSe material) and wipe and clean any powder off the surface of the pressure plate and pad (**9**), if any sample powder has adhered here.

After cleaning (see **Notes On Cleaning** page 40), the Gateway™ 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 the flat (**4**) top plate assembly fitted to the optical unit (**2**) and the clamp assembly (**1**). The procedure for spectral collection is as follows:-

1. Collect a **background spectrum** as described in steps 1 to 3 (page 35).

2. Take the flat, solid sample and place it very carefully such that it covers the entire surface area of the ZnSe, Ge or Si ATR crystal (**17**). Ideally the flat, solid 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 (**17**) when being pressed.

3. Ensure that the tension of the spring bar (**11**) is loose by having the tensioning screw (**10**) on the clamp assembly (**1**) almost fully raised. Carefully lower the pressure plate and pad (**9**) by pressing down on the T-bar handle (**8**) to lock down the arm mechanism (**7**) in the closed position and to loosely bring the pressure plate and pad (**9**) into contact with the flat, solid sample.

4. Begin turning the tensioning screw knob (**12**) clockwise to exert a force on the spring bar (**11**) and to, in turn, increase the force against the pressure plate and pad (**9**). Continuation of rotation of the tensioning screw (**10**) until it stops turning will provide for a maximum load setting against the sample forcing it against the ATR crystal (**17**).

Note: *If the sample may be hard or could produce a point load effect, carry out rotation of the tensioning screw (**10**) 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 tensioning screw (**10**).*

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

6. To remove or change the sample, release the load/pressure on the sample by turning the tensioning screw knob (**12**) anticlockwise to retract the screw (**10**) away from the spring bar (**11**) and the pressure plate and pad (**9**). When the spring bars (**11**) tension is loose, lift up the clamp arm mechanism (**7**) by pulling up on the T-bar handle (**8**) to remove the pressure plate and pad (**9**) away from the sample.

7. Remove the flat, solid sample carefully away from the ATR crystal (**17**), (avoid the possibility of scratching the ATR crystal (**17**) surface, particularly if using ZnSe material) and wipe and clean any sample off the surface of the pressure plate and pad (**9**), if any sample has adhered here.

After cleaning (see **Notes On Cleaning**, page 40), the Gateway™ ATR Accessory is ready to accept a new sample for measurement.

Collecting ATR Spectra of Liquids and Paste Samples

In general, liquid and paste like samples make good, close contact with the ATR crystal (**17**) and it is not necessary to use the clamp assembly (**1**) to obtain a good ATR spectral measurement for these sample types. Hence, when analyzing for a **liquid or paste sample** it is best to use the trough (**3**) top plate assembly fitted to the optical unit (**2**).

If the liquid sample is volatile by nature, Specac recommends use of a volatiles cover (P/N GS11132 – not supplied), to place over the open trough plate area to prevent evaporation of the liquid and to enable an ATR spectral measurement to be obtained. (See **Fig 17**.)

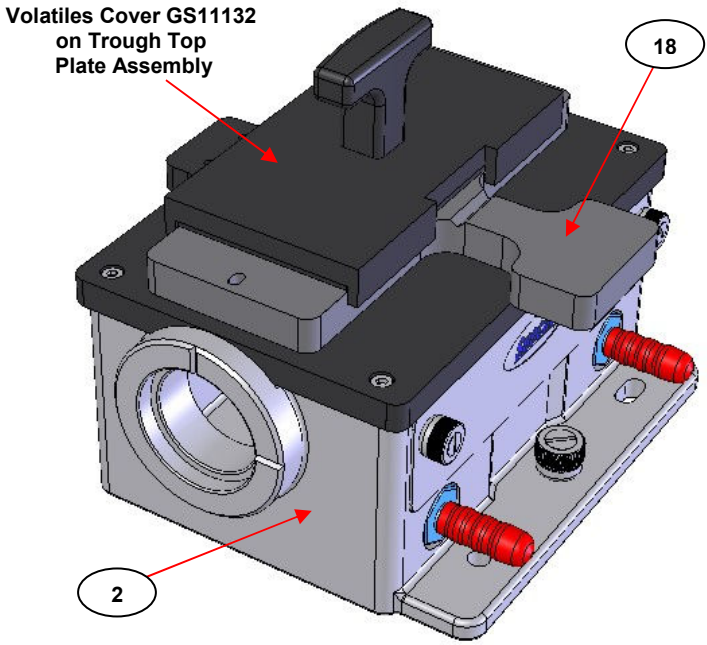


Fig 17. Volatiles Cover for Gateway™ ATR Trough Top Plate Assemblies

Note: *If the volatiles cover is to be used on any trough (**3**) top plate assembly then the clamp assembly (**1**) must be removed from the optical unit (**2**) as shown in **Fig 17**.*

The procedure for a **sample spectral collection** is as follows:-

1. Collect a **background spectrum** as described in steps 1 to 3 (page 35).

2. Take the liquid or paste sample and place it very carefully such that it covers the entire surface area of the ZnSe, Ge or Si ATR crystal (**17**). A dropping pipette can be used to dispense a liquid and the spatula supplied can be used to spread a paste over the ATR crystal (**17**) of the trough (**3**) top plate assembly.

3. Place the volatiles cover into position over the sample if necessary.

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, particularly from the ZnSe crystal (**17**), to avoid risk of damage to this crystal material.

After cleaning (see **Notes On Cleaning**, page 40), the Gateway™ ATR accessory is ready to accept a new sample for measurement.

Notes On Cleaning

When cleaning a ZnSe, Ge or Si crystal (**17**) of the Gateway™ ATR trough (**3**) or flat (**4**) top plate assembly in preparation for a new sample, it is **very important to take care** to avoid damage to the crystal materials**.** As also mentioned in the Safety Considerations (Section 2, page 6), of the three crystal materials ZnSe is potentially the most hazardous in terms of risk of toxicity if it comes into contact with the skin.

Note: ***Always wear gloves to protect yourself and the ATR crystal material.***

A useful feature of the Gateway™ ATR Accessory is the capability for removal of the ATR crystal top plate assembly (**3**) and (**4**) from the optical unit (**2**), such that any sample can be prepared remotely and safely, if desired, onto the ATR crystal (**17**) surface and then the ATR crystal top plate assembly can be bought for fitting onto the optical unit (**2**) whilst installed in the spectrometer. Similarly, for cleaning, it may be useful to remove the trough (**3**) or flat (**4**) 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. Sample solutions that fall within the pH range of pH4 to pH11 are tolerated by the ZnSe crystal material. 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 crystals, **particularly if using ZnSe material as this crystal type is not as resilient as germanium or silicon crystals.** Scratches and blemishes to the ATR crystal (**17**) surface will result in poor light throughput for the ATR technique and an overall degradation in the Accessory performance.

In common and general usage, it will only be necessary to wipe and clean away at the top surface of any Gateway™ ATR crystal in either a trough (**3**) or flat (**4**) top plate assembly between samples. If possible, try to avoid any solvent or cleaning solution materials from getting to the underside of the top plate assemblies. There is a risk that any dried solution components that have been introduced to this underside of the top plate assembly could be seen as an “impurity” against the ATR crystal in any “background” spectrum to be collected, and so this contaminant would need to be removed before any further sampling can continue.

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 4 to 11 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 labelling.

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 Silicon

General

Synonyms: Defoamer S-10.
When powder is fused together, is used as a transmission window material. Very hard, but brittle and relatively inert material. Insoluble in water, resists acids and bases but is attacked by combination of hydrofluoric and nitric acid. Can withstand thermal shock.
Useful for Far IR working in the region 400 to 33cm-1
Molecular formula: Si.
Chemical Abstracts Service (CAS) No: 7440-21-3

Physical Data

Appearance: Grey lustrous solid or grey powder.
Melting point: 1410°C.
Boiling point: 2355°C.
Solubility in water: 0g/100g at 0°C.
Hardness: 1150 Kg/mm^2.
Refractive Index: 3.42 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 8,333 to 33 cm-1 (wavenumbers) - not continuous as absorptions in the Mid IR from circa 1300 to 500 cm-1.

Stability

Stable.
Fine powder is highly flammable. Incompatible with oxidizing agents, bases, carbonates, alkali metals, lead and aluminium oxides, halogens, carbides and formic acid.

Toxicology

Generally regarded as safe.

Personal Protection

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

Storage

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

9. Gateway™ ATR Accessory "Bubble Numbers" Part Identification List

(**1**) Clamp assembly (complete).
(**2**) Optical unit (complete).
(**3**) Trough top plate assembly (complete).
(**4**) Flat top plate assembly (complete).
(**5**) Movable mirror in optical unit (**2**).
(**6**) Fixed mirror in optical unit (**2**).
(**7**) Movable arm mechanism of clamp assembly (**1**).
(**8**) T-bar handle of clamp assembly (**1**).
(**9**) Pressure plate and pad of clamp assembly (**1**).
(**10**) Force tensioning adjustment screw of clamp assembly (**1**).
(**11**) Tensioning spring bar of clamp assembly (**1**).
(**12**) Knob of force tensioning adjustment screw (**10**).
(**13**) Fixing support block of clamp assembly (**1**).
(**14**) M4 x 20mm cap head screw for fixing support block (**13**).
(**15**) Top surface plate of the optical unit (**2**).
(**16**) Fixing location pins for top plate assemblies (**3**) and (**4**).
(**17**) ATR crystal of top plate assemblies (**3**) and (**4**).
(**18**) Metalwork of trough top plate assembly (**3**).
(**19**) Pressure bar of trough top plate assembly (**3**).
(**20**) Lead pad of trough top plate assembly (**3**).
(**21**) Isolast gasket of trough top plate assembly (**3**).
(**22**) M4 x 5mm grub screw of base support plate (**23**).
(**23**) Base support plate of trough top plate assembly (**3**).
(**24**) M3 x 12mm cap head fixing screw of base support plate (**23**).
(**25**) Metalwork of flat top plate assembly (**4**).
(**26**) Fixing thumbscrew of the optical unit (**2**).
(**27**) Front cover plate of the optical unit (**2**).
(**28**) Fixing thumbscrew of the front cover plate (**27**).
(**29**) M4 x 5mm cap head screw of movable mirror (**5**).
(**30**) M4 x 12mm grub screw of movable mirror (**5**).
(**31**) Purge ports on the optical unit (**2**).
(**32**) Purge bellows to fit to optical unit (**2**).
(**33**) Aperture ports on the optical unit (**2**).

10. Gateway™ ATR Accessories Parts List

Gateway™ ATR Optical Unit

P/N GS11170 Gateway™ In-Compartment ATR optical unit.

Gateway™ ATR Top Plates

P/N GS11133 Gateway™ ATR Flat top plate with 45° ZnSe crystal.
P/N GS11134 Gateway™ ATR Flat top plate with 45° Si crystal.
P/N GS11135 Gateway™ ATR Flat top plate with 45° Ge crystal.
P/N GS11166 Gateway™ ATR Trough top plate with 45° ZnSe crystal.
P/N GS11116 Gateway™ ATR Flow Through top plate (550um volume) with 45° ZnSe crystal.
P/N GS11118 Gateway™ ATR Thermostabilised Flow Through top plate (550um volume) with 45° ZnSe crystal.
P/N GS11139 Gateway™ ATR Water Heated Trough top plate with 45° ZnSe crystal.
P/N GS11155 Gateway™ ATR Electrically Heated Trough top plate with 45° ZnSe crystal.

Gateway™ ATR Spares and Consumables

P/N GS11167 Isolast gasket seal for P/N GS11166 only (pkt of 2).
P/N GS11171 Gateway™ ATR clamp assembly.
P/N GS11145 45° ZnSe Crystal for Trough top plates.
P/N GS11146 45° Si Crystal for Trough top plates.
P/N GS11147 45° Ge Crystal for Trough top plates.
P/N GS10707 Pair of Purge Bellows.

Polarizers (GS12000 Series)

P/N GS12000 KRS-5 Polarizer
P/N GS12700 Germanium Polarizer
P/N GS12800 Calcium Fluoride Polarizer
P/N GS12900 Barium Fluoride Polarizer
P/N GS12950 Zinc Selenide Polarizer.

11. Gateway™ ATR Technical Specifications

	ZnSe	Germanium	Silicon
ATR Crystal Active Area	ZnSe 45° 70mm x 10mm.	Ge 45° 70mm x 10mm.	Silicon 45° 70mm x 10mm
Accessory Transmission Range cm^{-1}	7,800 - 550 (AR coated)	5,500 - 600 (AR coated)	8,333 - 33 (non-continuous)
Refractive Index at 1000cm^{-1}	2.43	4.0	3.42
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})
Typical Light Throughput When Aligned	20% to 30% Transmission	20% to 30% Transmission	20% to 30% Transmission
ATR Crystal Plate Assembly	ZnSe sealed with Isolast gasket in Trough assembly. Glued with epoxy resin in Flat assembly	Ge sealed with Isolast gasket in Trough assembly. Glued with epoxy resin in Flat assembly	Si sealed with Isolast gasket in Trough assembly. Glued with epoxy resin in Flat assembly
Gateway ATR Dimensions W x D X H (mm)	160 x 130 x 130	160 x 130 x 130	160 x 130 x 180

12. Gateway™ ATR Serial Numbers

Your Gateway™ 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 Gateway™ ATR Accessory parts you have received. If you need to contact Specac for any issues regarding your Gateway™ ATR Accessory it may be necessary to provide the serial number of the item to identify for replacement parts.

Gateway™ ATR Part Number and Description	Serial Number
P/N GS11170 – optical unit (**2**) **without** the clamp arm assembly (**1**).	
P/N GS11166 – trough (**3**) top plate assembly fitted with 45° ZnSe crystal (**17**).	
P/N GS11133 – flat (**4**) top plate assembly fitted with 45° ZnSe crystal (**17**).	

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