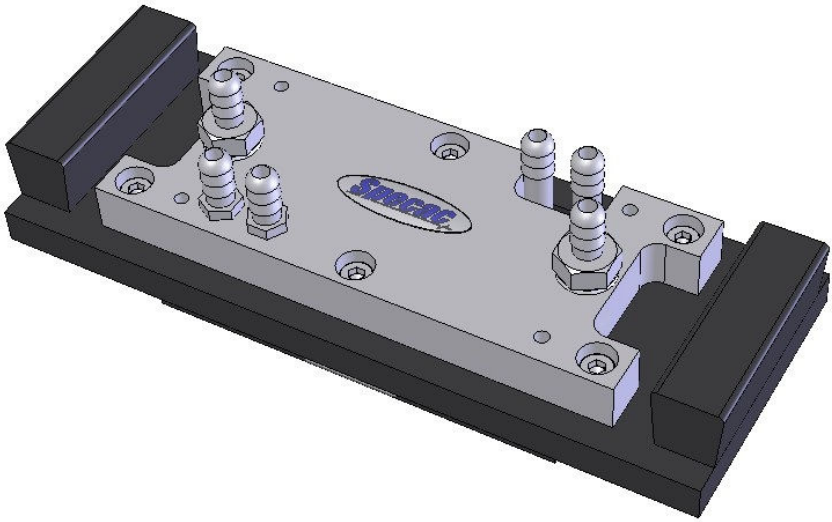
Gateway™ ATR Thermostabilised Flow Through Top Plate

User Manual



2I-11118-4

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User Manual

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Gateway™ ATR Thermostabilised Flow Through Top Plate P/N GS11118

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1. Introduction

Thank you for purchasing a Specac Product.

This user instruction manual for the Gateway™ ATR Thermostabilised Flow Through Top Plate P/N GS11118 is to be used in conjunction with the user instruction manual provided for the Gateway™ 6 Reflection ATR Accessory against P/N GS11165 (2I-11165-4). Understanding of the Gateway™ ATR Accessory itself helps in usage of any alternative ATR Top Plates that are compatible with this ATR system.

The Gateway™ ATR Thermostabilised Flow Through Top Plate P/N GS11118 is an alternative Top Plate that can be used on the optical unit (P/N GS11170) of the Gateway™ ATR Accessory. The Gateway™ ATR Accessory supplied as standard for a kit of parts under P/N GS11165, provides for an optical unit (GS11170), a trough top plate assembly fitted with a 45° ZnSe crystal (GS11166), a flat top plate assembly fitted with a 45° ZnSe crystal (GS11133) and a clamp assembly (GS11171). (See user instruction manual 2I-11165-4).

Note: ***Any*** *Gateway™ ATR Top Plate assembly can also be used on older Benchmark™ ATR optical units, P/N's GS11160, GS11110 and GS11180.*

Using the Gateway™ Thermostabilised ATR Flow Through Top Plate in conjunction with any of the above optical unit systems, a liquid sample can be flowed or held static over a 6 Reflection event ATR crystal to be studied over a temperature range from ambient to 130°C. Chemical interactions for the mixing of sample species in a flowing environment may also be monitored. (e.g. a solid sample can be deposited onto the ATR crystal and a fluid can be flowed over the sample to observe any changes spectroscopically.)

A liquid sample is introduced to the ATR crystal analysis area via connection of tubing to the inlet and outlet sample flow ports on the top section of the Gateway™ ATR Thermostabilised Flow Through Top Plate. The liquid sample flows through a cavity with a volume of 550 microlitres, when covering over the entire top surface area of the

crystal. The liquid sample can be kept flowing using a suitable flow tubing (clear silicone rubber – as supplied) combined with a peristaltic liquid pumping system, or held static over the crystal when the pump is turned off.

Surrounding the sample flow and crystal area of this Top Plate assembly are water flow cavities to carry a thermostabilised fluid to study the sample at elevated temperatures up to 130°C. Suitable flow tubing is connected to the thermocirculating flow ports on the Top Plate assembly to allow for introduction of a heated fluid. A flow of heated fluid (water or oil) is provided via a thermocirculating system such as that offered by Specac P/N GS11127.

The Gateway™ ATR Thermostabilised Flow Through Top Plate has been designed with the capability to remove the ATR crystal for thorough cleaning or using a replacement ATR crystal material in the Top Plate if desired. Any of the crystal materials usable with the Gateway™ ATR system – ZnSe, germanium or silicon – can be placed into the Gateway™ ATR Thermostabilised Flow Through Top Plate, although Germanium loses its spectral IR transmission capability with a progressive rise in temperature, becoming completely opaque at temperatures of circa 100°C and above.

As a standard offering for P/N GS11118, a 45° angle ZnSe crystal (P/N GS11145) is fitted. The ATR crystal options are:

P/N GS11145 - Gateway™ ZnSe crystal, 45° angle.
P/N GS11146 - Gateway™ Silicon crystal, 45° angle.
P/N GS11147 - Gateway™ Germanium crystal, 45° angle.

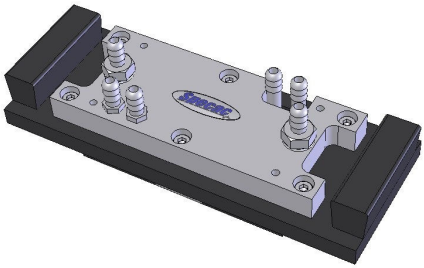


Fig 1. Gateway™ ATR Thermostabilised Flow Through Top Plate

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.*

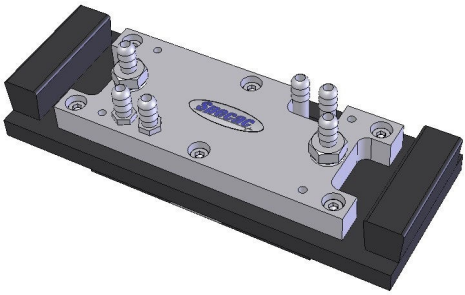
Both Ge and Si crystal materials can be considered relatively safe to use, although Ge may be harmful to the body if it is ingested in significant quantity. The general rule when working with **any** crystal material (and sample) **is to always wear gloves and safety gear** (e.g. safety spectacles) when 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 the Gateway™ ATR instruction manual (2I-11165-4) in the **Notes on Cleaning** Section found on pages 42, 43 and 44.

3. Unpacking and Checklist

The Gateway™ ATR Thermostabilised Flow Through Top Plate is supplied packed in a black plastic carry case. The carry case contains the following:

- Thermostabilised Flow Through ATR Top Plate with Zinc Selenide crystal (or as specified from germanium and silicon options)



- ATR spectrum of the crystal as fitted to the Top Plate assembly to show its background throughput.
- Clear silicone tubing for sample flow connection, 1/4” OD, 1/8” ID.
- Combination (open ended and ring) spanner 3/8” A/F.
- Allen Key (2.5mm A/F)

Remove the Gateway™ ATR Thermostabilised Flow Through Top Plate from the portable carry case and unwrap it from the protective polythene cover.

4. Alignment of the Gateway™ ATR Thermostabilised Flow Through Top Plate

The Gateway™ ATR Thermostabilised Flow Through Top Plate is to be installed onto the optical unit (GS11170) of the Gateway™ ATR Accessory for operation in the study of flowing or static liquids at varying temperature conditions over the range from ambient to 130°C maximum. The Thermostabilised Flow Through Top Plate should be optimised for its energy light throughput from a particular alignment of the mirrors on the optical when installed for use.

If the Gateway™ ATR Accessory (as the Kit GS11165) is available for use please follow the instruction manual supplied (2I-11165-4) for the alignment procedure to optimize the light energy throughput of the ATR system on your particular spectrometer using either the Trough Top Plate (GS11166) or Flat Top Plate (GS11133) assembly as supplied. The input and output mirror settings for their rotation and tilt angles will be established on the optical unit (GS11170) from the alignment procedure using either of these two Top Plates.

When subsequently placing the Thermostabilised Flow Through Top Plate onto the optical unit as a replacement for either the Trough (GS11166) or Flat (GS11133) Top Plate assembly, a signal throughput will be registered, but it is possible the mirrors in the optical unit may require slight re-adjustment for an optimum throughput to be established in use of this alternative Flow Through Top Plate. Therefore, follow the alignment procedure with the Thermostabilised Flow Through Top Plate in position to obtain the best throughput energy signal for the spectrometers system.

Note: *If just the Gateway™ optical unit (GS11170) and a Thermostabilised Flow Through Top Plate are available to use, unless the optical unit has been pre-aligned to register some energy throughput with a Top Plate in position, then it may be difficult to gain an optimum alignment using the FTIR spectrometer systems energy source and detection system.*

A rough pre-alignment is to be established with the optical unit and Thermostabilised Flow Through Top Plate in position using a visible light source such as the Laser Alignment Accessory (P/N GS24500).

Fitting the Gateway™ ATR Thermostabilised Flow Through Top Plate

The Gateway™ ATR Thermostabilised Flow Through Top Plate fits onto the top of the Gateway optical unit via alignment of the round hole (**1**) and slot hole (**2**) on the underside of the Top Plate over the two fixing location pins (**3**) on the optical unit. (See **Fig 2** and **Fig 3**.)

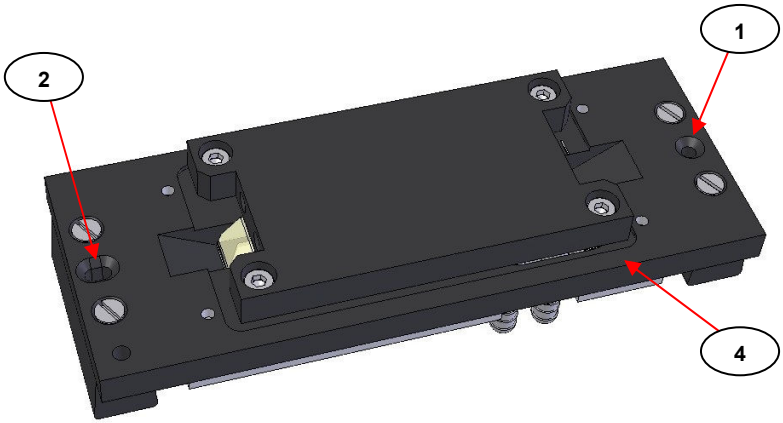


Fig 2. Round and Slot Location Holes on Underside of Gateway™ ATR Flow Through Top Plate

The Gateway™ ATR Thermostabilised Flow Through Top Plate is fitted and positioned the correct way around on the optical unit when the round hole (**1**) is placed over the **right side** location pin (**3R**) and the slot hole (**2**) is placed over the **left side** location pin (**3L**) as viewed from the front of the optical unit. The main body plate (**4**) of the Top Plate should be sitting flush and even all the way around its edge onto the top surface of the optical unit when properly located.

Note: *To maintain a correct optical alignment for consistency of optical throughput energy of the Gateway™ ATR Thermostabilised Flow Through Top Plate, it should always be placed onto the optical unit the same way around after removal for cleaning or crystal replacement etc, with the round (**1**) and slot (**2**) holes fitting over their respective fixing location pins (**3R**) and (**3L**).*

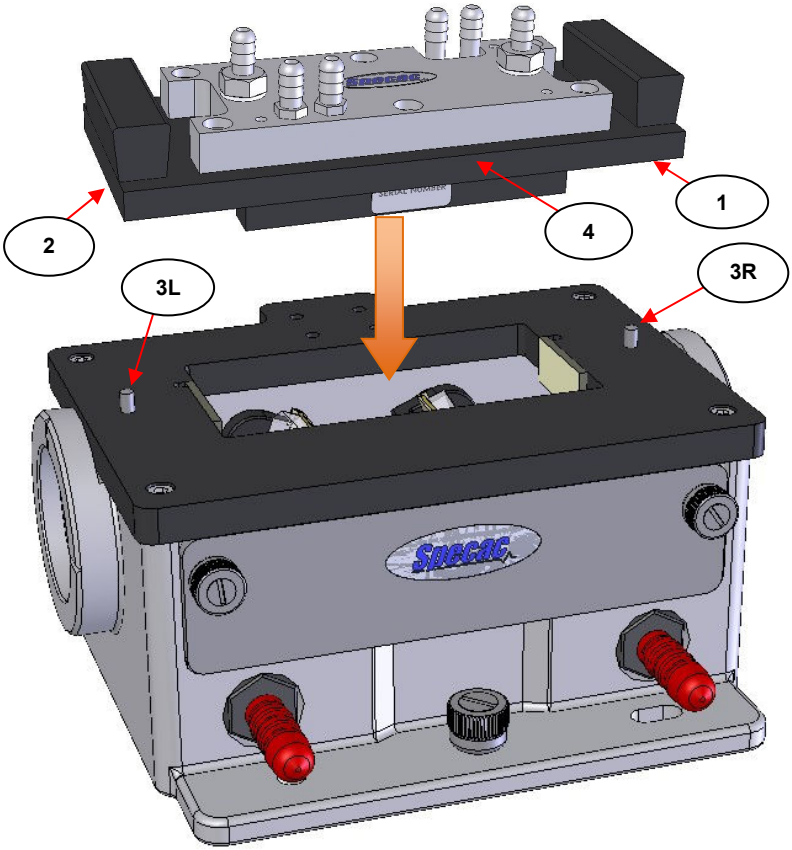


Fig 3. Fitting of Gateway™ ATR Thermostabilised Flow Through Top Plate to the Gateway™ Optical Unit

5. Introducing the Liquid Sample

When the Gateway™ ATR Thermostabilised Flow Through Top Plate has been correctly installed onto the optical unit and aligned, then operation for collection of a background spectrum and then a sample spectrum can begin. (Please see Spectral Collection Using the Gateway™ ATR Accessory from the instruction manual 2I-11165-4).

In operation for collection of a background spectrum and then a sample spectrum, ideally the temperature conditions for both spectral acquisitions should be the same. When using a thermocirculating system to provide heat to the Top Plate assembly care must be exercised that the temperature rate rise being provided by the thermocirculating system does not exceed 10°C per minute. Any higher rate rise risks introducing thermal shock to the ATR crystal (**6**) in the Top Plate assembly and causing damage.

When using the Gateway™ ATR Thermostabilised Flow Through Top Plate for liquid sampling it is essential that all the tubing connections are well secured to the sample flow inlet and outlet ports (**5**) and the thermocirculating ports (**6**) and (**7**) of the Top Plate assembly.

Important: *All tubing connected to the ports (**5**), (**6**) and (**7**) on the Top Plate should be tested initially for liquid flow away from installation on to the optical unit. This avoids any accidental spillage of fluid onto the optical unit should the flow tubes not happen to be well secured. The 3/8" spanner supplied can be used to tighten the flow port fittings (**5**) if any leaks occur at these positions.*

Liquid Sample Ports (5) Connection

The clear silicone flow tubing supplied with the Gateway™ ATR Flow Through Top Plate is connected to the sample flow ports (**5**) on the upper thermocirculating plate (**8**). (See **Fig 4**). It does not matter which of the flow ports (**5**) is the **inlet** or **outlet** connection for flow. The clear silicone tubing supplied has dimensions of 1/4" O.D. and 1/8" I.D. and is pushed over the barbed hose type connectors on the flow ports (**5)**.

Tie wraps can be placed around the tubing at the flow port connections to prevent the tubing from separating under a liquid sample flow.

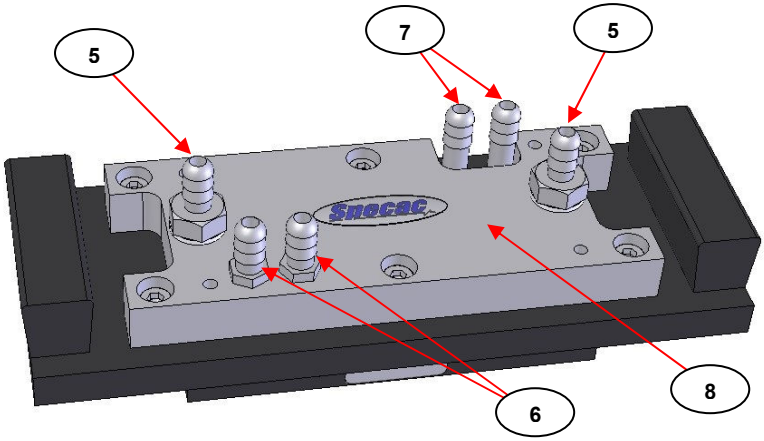


Fig 4. Liquid Sample and Thermocirculating Connection Ports on Gateway™ ATR Thermostabilised Flow Through Top Plate

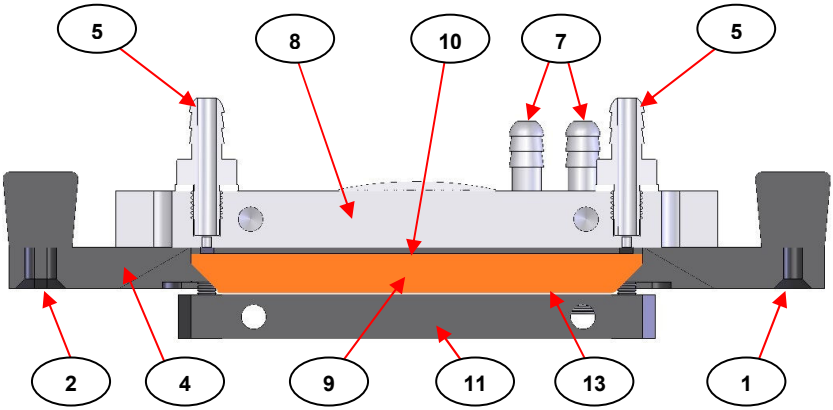


Fig 5. Cross Section Cutaway View Showing Internal Features of the Gateway™ ATR Thermostabilised Flow Through Top Plate

Connect the other ends of the clear silicone tubing that has been attached to the Thermostabilised Flow Through Top Plate sample ports (**5**) to a liquid sample supply and follow the instructions for operation of a liquid sample pumping system if this is being used to circulate the sample flow. The liquid sample passes through the Thermostabilised Flow Through Top Plate across the top surface of the ATR crystal (**9**) contained within, for 6 ATR reflection measurement events. (See **Fig 5.** for a cross section cutaway view of the Thermostabilised Flow Through Top Plate). There is an Isolast gasket (**10**) that seals between the ATR crystal (**9**) top surface and the underside of the upper thermocirculating plate (**8**) that creates the cavity for sample flow with a volume of 550 microlitres.

Note: *When the liquid is flowing, the recommended normal pressure usage is 60 psi maximum. A liquid can be measured in a static mode if the flow system is switched off whilst the liquid is contained within the Flow Through Top Plate cavity.*

Thermocirculating Ports (6) and (7) Connection

To establish different temperatures of operation for measurement of a sample fluid when in contact with the ATR crystal (**9**), a thermocirculating fluid (water or oil) is flowed entirely around the ATR crystal (**9**) by connection of the **upper** thermocirculating plate (**8**) to the lower thermocirculating plate (**11**) using the clear silicone tubing supplied. Either of the connection ports at (**6**) and (**7**) can be used as the inlet or outlet for a thermocirculating fluid to flow around the specific upper (**8**) or lower (**11**) thermocirculating plate, but to provide for a flow of thermocirculating fluid around the entire Top Plate assembly, the **outlet** port (**7**) of the **lower** thermocirculating plate (**11**) **MUST** be connected to the **inlet** port (**6**) of the **upper** thermocirculating plate (**8**).

Note: *Connection of a thermocirculating fluid flow from the lower level plate (**11**) to the upper level plate (**8**) helps to remove any air pockets that may be trapped in the flow system. In addition, appropriate connections to either the upper (**8**) or lower (**11**) thermocirculating sections, mean they could be heated independently of each other if desired.*

The clear silicone tubing supplied has dimensions of 1/4" O.D. and 1/8" I.D. and is pushed over the barbed hose type connectors on the inlet and outlet thermocirculating flow ports (**6**) and (**7**). Tie wraps can be placed around the tubing at the flow port (**6**) and (**7**) connections to prevent the tubing from separating under a thermocirculating liquid sample flow. Connect the other ends of the clear silicone tubing that has been attached to the **inlet** port (**7**) and **outlet** port (**6**) to a thermocirculating pump delivery system and follow the instructions for operation of the system being used for the thermocirculating fluid flow. (An example of such a system supplied by Specac for use is the Thermostatic Bath P/N GS11127).

In operation when running at elevated temperatures for any spectral measurements, establish a flow of thermocirculating fluid around the Top Plate and tubing system and then slowly raise the temperature of the thermocirculating fluid to the desired operating conditions. A gradual increase of temperature to a **flowing sample environment** reduces the risk of thermal shock to the ATR crystal (**9**).

6. ATR Crystal Removal and Replacement

The design of the Gateway™ ATR Thermostabilised Flow Through Top Plate allows for the ATR crystal (**9**) to be removable for easier cleaning or if damage occurs and the crystal needs to be replaced.

Crystal Removal from the Thermostabilised Flow Through Top Plate

If it is necessary to remove the crystal (**9**) for thorough cleaning of the Top Plate assembly of parts or to replace any damaged parts etc, the following procedure should be adopted.

Note*: It is normally easier to work on the Top Plate assembly having disconnected the silicone flow tubing from the inlet and outlet sample flow ports (**5**) and the thermocirculating fluid flow ports (**6**) and (**7**).*

Important: *For safety precautions wear gloves when carrying out the following procedure.*

1) Lay the Top Plate assembly on to a clean workspace area and proceed to remove the four M3 x 16mm cap head screws (**12**) from the lower thermocirculating plate (**11**) on the underside of the Top Plate assembly using the 2.5mm A/F Allen key supplied. Turn the screws (**12**) anticlockwise to remove. (See **Fig 6**.)

Note: *The lower thermocirculating plate (**11**) also acts as the **clamp pressure plate** for sealing of the ATR crystal (**9**) to the Kalrez gasket (**10**) and to the upper thermocirculating plate (**8**).*

2) Between the underside of the lower thermocirculating plate (**11**) and the ATR crystal (**9**) there is a protective lead pad (**13**). Carefully remove the lower thermocirculating plate (**11**) away to gain access to the lead pad (**13**). (See **Fig 7**.) The lead pad (**11**) may be stuck close to the underside of the crystal (**9**) and so it needs to be removed very carefully away from the crystal.

Note: *The lead pad (**13**) will be needed in any re-assembly after cleaning of parts or if replacing with a new crystal (**9**).*

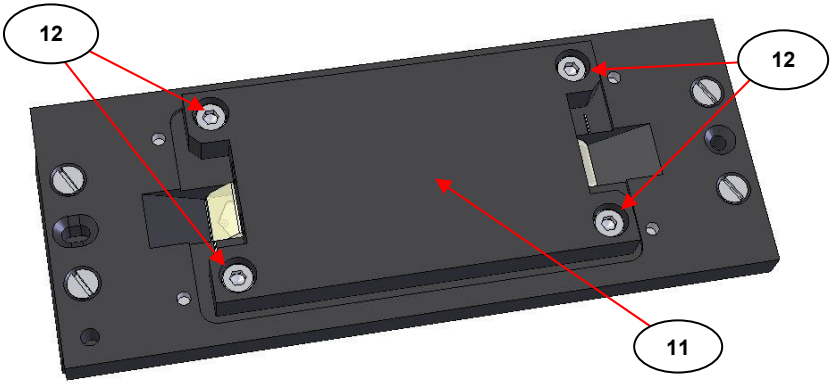


Fig 6. Underside View of Thermostabilised Flow Through Top Plate Assembly

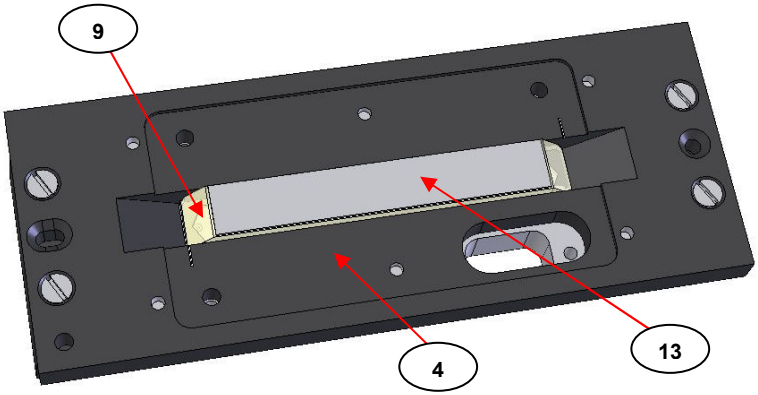


Fig 7. Lead Pad on Underside of ATR Crystal in the Thermostabilised Flow Through Top Plate Assembly

3) If the lead pad (**13**) has adhered to the crystal (**9**), then it may be easier to remove it carefully away from the crystal when the crystal (**9**) itself has been removed from its centralised position in its recess of the main body plate (**4**). Therefore, support the crystal (**9**) and the lead pad (**13**), with one hand and turn over the Top Plate assembly so that the crystal (**9**) can fall out to drop into the palm of your hand. If the crystal (**9**) is also stuck in the main body plate (**4**) because of good sealing to the Isolast gasket (**10**) between the top (sampling) surface of the crystal (**9**) and the upper thermocirculating plate (**8**), then put a soft pad or tissue on the work bench area and lay the Top Plate assembly onto it the same way up as it would be used when fitted to the optical unit. Block one of the sample flow ports (**5**) and connect the other port (**5**) to a clean, dry compressed air line and allow a little pressure into the system. This should dislodge the crystal (**9**) from the body plate (**4**) to fall onto the soft pad or tissue.

When the crystal (**9**) and the lead pad (**13**) have been removed, it may now be easier to separate the lead pad (**13**) from the crystal. If the crystals (**9**) top sampling surface looks OK and can be cleaned carefully, then it may not be necessary to remove the lead pad (**13**) away for further cleaning prior to re-assembly and use. However, if the lead pad (**13**) is to be removed, it may be necessary to immerse the whole crystal (**9**) and lead pad (**13**) assembly in a beaker of water or methanol solvent and apply a gentle “sonication cleaning” of the items. The action of the sonic agitation of the solvent may help to loosen the lead pad (**13**) away from the crystal such that when the items are removed from the solvent they can be easily and safely separated without any damage to the lead pad (**13**) or crystal (**9**) parts.

Clean the ATR crystal (**9**) by following the Notes on Cleaning instructions on page 21.

Changing the Isolast Gasket Seal

When the ATR crystal (**9**) has been removed from the Top Plate assembly, access can be gained to the Isolast gasket seal (**10**) for cleaning or replacement with new if necessary. (See **Fig 8**.)

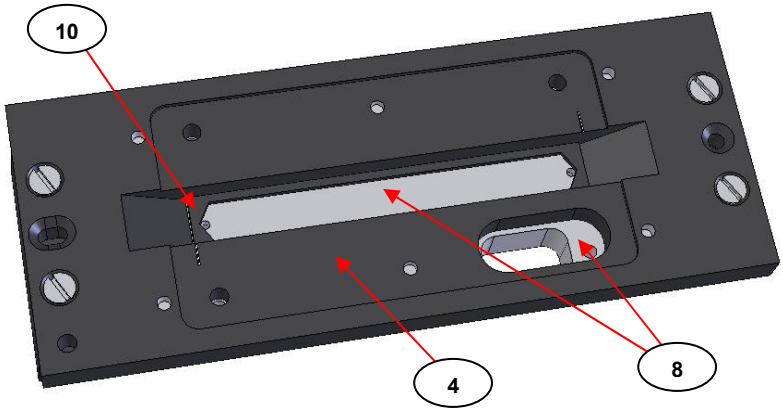


Fig 8. Isolast Gasket Seal in the Gateway™ ATR Thermostabilised Flow Through Top Plate

The Isolast gasket seal (**10**) is supplied already pre-cut to fit and is purchased against P/N GS11150. Should the Isolast gasket (**10**) be worn or damaged after inspection then replace with a new example. If changing for a new gasket (**10**), ensure that all parts of the old gasket are removed from the surface of the upper thermocirculating plate (**8**) to provide a good surface for resealing. It does not matter which surface side of the Isolast gasket (**10**) is placed into contact with either the underside of the upper thermocirculating plate (**8**) or the top surface of the crystal (**9**).

Replacing the Crystal in the Top Plate Assembly

1) Having cleaned all of the parts sufficiently and whether installing a new crystal (**9**) and/or new Isolast gasket (**10**), re-assemble the Gateway™ ATR Thermostabilised Flow Through Top Plate first by aligning correctly and centrally the Isolast gasket seal (**10**) into the underside channel recess of the main body plate (**4**). (See as **Fig 8**.)

Important: *Ensure that the sample port introduction holes (**5**) through the upper thermocirculating plate (**8**) are not blocked or covered by an incorrect positioning of the gasket (**10**).*

2) Insert the ATR crystal (**9**) into the recess channel of the main body plate (**4**) with the longer top surface of the crystal pressing against the installed Isolast gasket (**10**) and the angled faces of the crystal pointing upwards. Ensure that the ends of the crystal (**9**) line up with, or are equidistant from, the alignment marks on either side of the recess channel in the main body plate (**4**) so that the crystal (**9**) is sitting flat in the recess and not resting on an inclined chamfer face.

Note: *If the crystal (**9**) is not placed flat and centrally against the Isolast gasket (**10**) it will break when being pressure sealed from repositioning and screw tightening of the lower thermocirculating (pressure clamp) plate (**11**).*

3) Place the lead pad (**13**) carefully and centrally on the underside face of the crystal (**9**), making sure that it does not obscure the input or output angled faces of the crystal. (See as **Fig 7**.)

4) Carefully replace the lower thermocirculating plate (**11**) over the lead pad (**13**) and crystal (**9**) and refit the four screws (**12**). Ensure that the lead pad (**13**) and crystal (**9**) are not dislodged from their central positioning. (See as **Fig 6**.)

5) Exert a pressure at the middle of the lower thermocirculating plate (**11**) with your thumb and tighten the four screws (**12**) a little at a time, in a rotational sequence, to apply the pressure to the lead pad (**13**), crystal (**9**) and Isolast gasket (**10**) assembly. The objective is to bring the lower thermocirculating plate (**11**) into contact evenly and levelly to the main body plate (**4**) for tightening of the components together to avoid any sample fluid leakage in operation.

Important: ***Do not over-tighten!*** *All of the ATR crystals (**9**) material types, but particularly ZnSe, are brittle crystal materials and uneven pressure or over-tightening may damage the crystal (**9**).*

6) Stand the re-assembled Thermostabilised Flow Through Top Plate on a tissue covering a work surface and test for leaks for any sampling solutions. Do not put it onto the optical unit until tested. Test with a suitable solvent or solution at 70 psi for 30 minutes.

Note: *The recommended normal usage pressure is 60 psi maximum.*

If leaks occur, wipe the Top Plate area with a soft, lint-free tissue and tighten the four screws (**12**) in the lower thermocirculating plate (**11**) by about one sixth of a turn for each screw and re-test.

Possible Causes of Leaks

Assembly not tightened sufficiently.
Lower thermocirculating plate (**11**) and upper thermocirculating plate (**8**) surfaces not sufficiently cleaned.
Isolast gasket seal (**10**) not lying flat.
Isolast gasket seal (**10**) is old/worn and requires replacement.
ATR crystal (**9**) is defective (e.g. chipped, dirty, warped).
ATR crystal (**9**) not symmetrically located.

Cleaning the Gateway™ ATR Thermostabilised Flow Through Top Plate Intact

The procedure for cleaning of the crystal (**9**) with its removal from the Top Plate assembly needs possibly to be carried out only very occasionally. To avoid the need for cleaning the crystal outside of the Top Plate assembly it is important to flush out any sample remaining within the sampling area cavity by cleaning through with a suitable solvent before storage. Clean, distilled water, methanol or acetone are all suitable solvents to flush through for cleaning. After flushing through with a cleaning solvent, any last vestiges of solvent may be sucked out of the cavity by applying a light vacuum line attached to one of the flow port connections (**5**) leaving the other flow port (**5**) open to allow for a flow of air through the Top Plate and to pass over the ATR crystal (**9**).

Test that the crystal (**9**) is suitably clean by taking a spectral measurement before storage of the Top Plate assembly in a clean, dry cabinet, for example the Specacabinet (P/N GS19100), at about 40-50°C.

Notes On Cleaning

When cleaning a ZnSe, Ge or Si crystal (**9**) of the Gateway™ ATR Thermostabilised Flow Through 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 5), 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 any of the ATR crystal top plate assemblies away from the optical unit, such that any sample can be prepared remotely and safely, if desired, onto the ATR crystal (**9**) surface and then the ATR crystal top plate assembly can be bought for fitting onto the optical unit whilst installed in the spectrometer. Similarly for cleaning, it may be useful to remove any Top Plate assembly from the optical unit 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 residues, 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 (**9**) surface will result in poor light throughput for the ATR technique and an overall degradation in the Accessory performance.

In common and general usage of the Gateway™ ATR Thermostabilised Flow Through Top Plate it may only be necessary to flush through with a solvent to clean away at the top surface of any Gateway™ ATR crystal (**9**) within, between samples. If possible, try to avoid any solvent or cleaning solution materials from getting to the underside of the Top Plate assembly. 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. Removal of such a contaminant may require complete removal of the ATR crystal (**9**) from the Thermostabilised Flow Through Top Plate assembly and employment of a suitable cleaning regime.

In storage of the Thermostabilised Flow Through Top Plate assembly, apart from ensuring that the ATR crystal has been cleaned sufficiently, Specac would recommend that the thermocirculating fluid flow tubing is disconnected and any thermocirculating fluid itself (oil or water) is drained out of the upper (**4**) and lower (**8**) thermocirculating flow plates. Before the thermocirculating fluid tubing is disconnected from the Top Plate assembly, connect the open ends of the tubing to a compressed air line to gently blow air through this tubing to remove any fluid contained within the upper (**4**) and (**8**) thermocirculating flow through plates. Dry the Top Plate assembly carefully from any last vestiges of fluid and store accordingly.

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.

7. Gateway™ ATR Thermostabilsed Flow Through Top Plate "Bubble Number" Part Identification List

(**1**) Round location hole on underside of Thermostabilised Flow Through Top Plate.
(**2**) Slot location hole on underside of Thermostabilised Flow Through Top Plate.
(**3**) Fixing location pin on top of Gateway™ ATR optical unit.
(**4**) Main body plate of Thermostabilised Flow Through Top Plate.
(**5**) Sample flow connection port.
(**6**) Thermocirculating fluid connection ports to upper thermocirculating plate (**8**).
(**7**) Thermocirculating fluid connection ports to lower thermocirculating plate (**11**).
(**8**) Upper thermocirculating plate.
(**9**) ATR crystal.
(**10**) Isolast gasket seal.
(**11**) Lower thermocirculating (and pressure clamp) plate.
(**12**) M3 x 16mm cap head screw for lower thermocirculating plate (**11**).
(**13**) Lead pad.

8. Gateway™ ATR Thermostabilised Flow Through Top Plate Spare Parts

Complete Top Plate Assembly

P/N GS11118 - Gateway™ ATR Thermostabilised Flow Through Top Plate with ZnSe crystal, 45° angle.

Replacement Crystals

P/N GS11145 - Gateway™ ATR ZnSe crystal, 45° angle.
P/N GS11146 - Gateway™ ATR Silicon crystal, 45° angle.
P/N GS11147 - Gateway™ ATR Germanium crystal, 45° angle.

Replacement Gasket

P/N GS11150 - Gateway™ ATR Isolast gaskets for 550ul Flow Through Top Plate assemblies. (pkt 3).

9. 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

10. Gateway™ ATR Thermostabilised Flow Through Top Plate Serial Number

Your Gateway™ ATR Thermostabilised Flow Through Top Plate assembly P/N GS11118 will be provided with a serial number for identification of certain individual parts. The serial number is found on a small silver sticker affixed to the underside of the Top Plate assembly.

To help you, please use the grid below to fill in the serial number information of the Gateway™ ATR Thermostabilised Flow Through Top Plate assembly you have received. If you need to contact Specac for any issues, it may be necessary to provide the serial number of the item to identify for replacement parts.

Gateway™ ATR Thermostabilised Flow Through Top Plate Assembly P/N and Description	Serial Number
P/N GS11118 – Gateway™ ATR Thermostabilised Flow Through Top Plate with ZnSe crystal, 45° angle.	

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