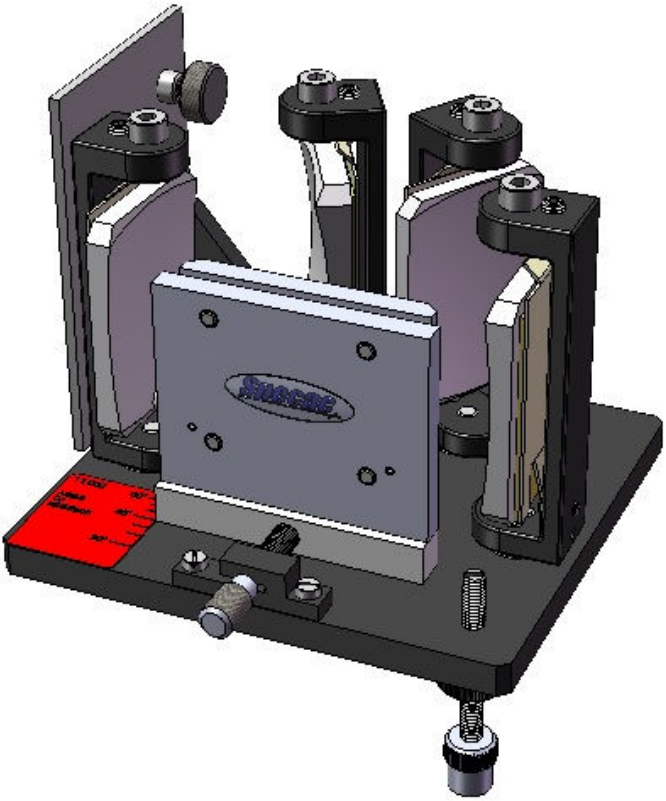
25 Reflection Variable Angle ATR

User Manual



2I-11000 Issue 6

25 Reflection Variable Angle ATR

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2I-11000 Issue 6

25 Reflection Variable Angle ATR P/N GS11000

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1. Introduction

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

The 25 Reflection Variable Angle ATR accessory P/N GS11000 is a vertically mounted ATR crystal accessory capable of being operated for an ATR crystal incidence angle of between 30º to 60º, allowing for the collection of an ATR IR spectrum of a wide range of solid and liquid sample types under ambient (room) temperature and atmospheric pressure conditions.

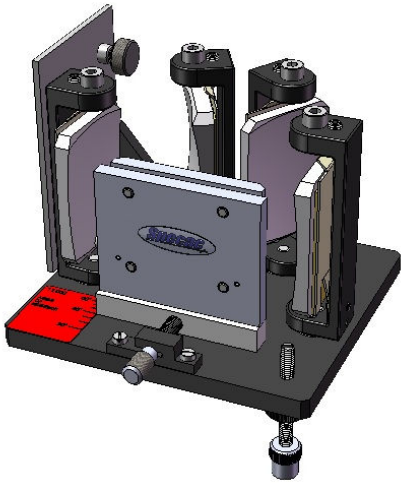
The angle of incidence for the ATR crystal is quickly and easily selected by rotation of a knurled adjustment screw on a translation stage. This moves the sample holder and ATR crystal assembly to the correct position for sampling when the front surface edge of the translation stage is aligned with the required angle setting mark as indicated on the graduated plate.

The ATR crystal used is a trapezium shape of nominally 52mm length (tip to tip), 20mm width and 2mm thick with 45° angled faces. A sample to measure for its ATR IR spectrum contacts ***both*** the long surface areas of the ATR crystal. Because of the crystal shape, one of the ATR crystal surface areas is longer than the other and so for 25 ATR reflection events at the sample/crystal interface, there will be 13 reflections from the longer surface at a 45° incident angle and 12 at the shorter surface length for the same 45° incident angle. Although the sample holder and crystal assembly can be positioned between the angles of 30º to 60º, it is only when the holder and crystal assembly are correctly aligned at the 45º angle, that 25 internal reflections are obtained within the ATR crystal. At all other angles, the true number of reflection events within the crystal is not precisely known.

The 25 Reflection ATR accessory is supplied with a zinc selenide (ZnSe) crystal P/N GS11014 as standard, but a variety of crystal materials such as KRS-5, germanium (Ge) and silicon (Si) are available to extend the sample handling and study capabilities of the accessory.

The IR beam is directed through and collected from the ATR crystal as supported in its crystal mount holder by four mirrors on the accessory which are adjustable for both their rotation and tilt. The accessory is supplied with a standard 3” x 2” mounting plate that allows it to be positioned in an IR spectrometers sample compartment using the instruments own 3” x 2” mounting baseplate. In addition, the 25 reflection ATR accessory is supplied with a support foot that is used at one corner of the base. The foot is adjustable for height to provide mechanical stability of the accessory whilst in the sample compartment. A thumb nut locks the foot in position.

As standard under P/N GS11000, the 25 Reflection ATR accessory is provided with the solids sample holder P/N GS11001 for mounting of the ZnSe crystal P/N GS11014 and the study of solid sample types. (e.g. thin films, plastics sheets etc.) Additional ATR crystal holders such as the paste holder P/N GS11002 and the liquids holder P/N GS11003 are available to extend the sampling capabilities of the accessory for ATR IR spectral collection of suitable liquid and mobile sample species.



25 Reflection Variable Angle ATR Accessory

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 25 Reflection ATR accessory, this uses different crystal materials for the ATR crystal holder assemblies where a sample is bought into contact for analytical spectroscopic study. As standard, Zinc Selenide (ZnSe), Thallium Bromoiodide (KRS-5), Germanium (Ge) and Silicon (Si) are the four crystal materials of choice that can be used.

Caution: *Out of these four different crystal types, ZnSe and KRS-5 are the most potentially hazardous materials with respect to toxicity risk in use and handling.*

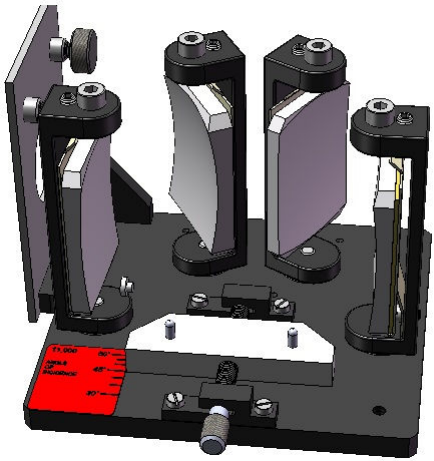
Both Ge and Si crystal materials can be considered relatively safe to use, although both materials may be harmful to the body if ingested in significant quantity. The general rule when working with **any** crystal material (and sample) **is to always wear gloves and safety gear** (e.g. safety spectacles)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

Provided with each ATR crystal material supplied, there is a safety datasheet 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 User Instruction Manual in the **Notes On Cleaning** Section found on pages 24 to 28.

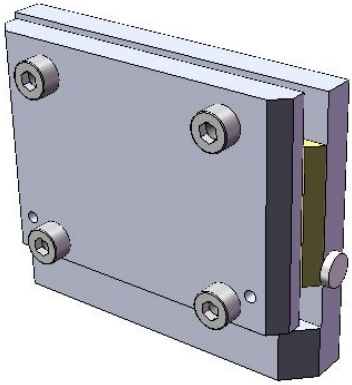
3. Unpacking and Checklist

The 25 Reflection Variable Angle ATR is supplied in its own packing case. Please check on receipt that the following items are included.

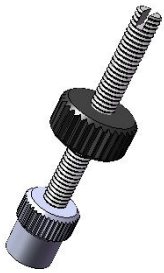
- 1 ATR unit optical assembly.



- 1 Solids sample holder P/N GS11001 with a ZnSe crystal P/N GS11014 fitted and mounted for use.



- 1 Adjustable support foot and locking nut.



- 4 Plastic mirror cover protectors.
- 1 Allen key 2.0mm A/F short arm
- 1 Allen key 2.5mm A/F short arm
- 1 User Instruction Manual.
- 1 IR background spectral throughput trace from initial alignment at Specac.

Carefully lift the 25 Reflection ATR unit from the case.

Take the support foot and screw it into the base of the accessory from the underside. (See **Fig 1.** - page 9.)

Carefully remove the protective plastic covers from the mirrors taking care not to touch the mirrors with your fingers. Wherever possible, wear safety gloves to protect yourself and the equipment.

4. Installation and Alignment

The supplied 25 Reflection ATR accessory will have been preliminarily aligned from new at Specac for an optimum energy level throughput to obtain the reference background IR spectral trace as provided. Appropriate rotation and tilt adjustments for positioning of the 4 mirrors on the accessory will have been made using the supplied ZnSe crystal in the solid sample holder. The ZnSe crystal in the solid sample holder assembly will have been set at the 45° incident angle setting for positioning of the translation stages front edge as indicated from the 45° mark on the graduated scale.

When installing the 25 Reflection ATR accessory as new and ***for the first time in your IR spectrometer***, it will be necessary to finely align the mirrors on the accessory to best match for an optimum energy light throughput level on the new IR spectrometer system. The ZnSe crystal as fitted into the solids sample holder for a complete assembly (***with no sample***), has been supplied to best replicate the conditions of factory procedures for initial installation and alignment purposes into a new spectrometer. The translation stage for positioning of the supplied ATR crystal in the solids sample holder assembly must also be set for the 45° angle position to replicate measurement conditions.

Important: *Until an optimized level of the light energy throughput has been established from a first fit installation of the 25 Reflection ATR accessory into a new spectrometer system,* ***DO NOT*** *dis-assemble the factory supplied ZnSe ATR crystal in the solids holder assembly.*

Installation Set Up

Take the 25 Reflection ATR accessory as prepared from the unpacking and checklist stage (Section 3 of this User Instruction Manual), from fitting of the support foot (**1**). (Screw the support foot (**1**) into the retaining hole from the underside of the ATR accessory unit.)

Insert the 3” x 2” slide mount back plate (**2**) into the IR spectrometers own 3” x 2” slide mount sample support holder and clamp the 25

Reflection ATR accessory unit platform into position by tightening of the 3" x 2" slide mounts locking screw (**3**). (Turn screw clockwise.)

Note: *The IR spectrometers own 3" x 2" slide mount plate should be positioned at the focal point of the beam in the sample compartment to provide the optimum throughput.*

The diagram of the 25 Reflection ATR accessory at Fig 8. (page 31) shows the IR beam passage from source to detector through the sample compartment for a **left to right** spectrometer system whereby mirror M1 is the primary input and mirror M4 the final output. As the accessory is of a symmetrical optical arrangement, it will work just as well in a **right to left** beam spectrometer system whereby mirror M4 becomes the primary input and M1 the final output. However, it is important that the 3" x 2" slide mount arrangement is always positioned at the focal point of the spectrometer, whatever the beam direction. Adjust the support foot (**1**) so that it rests on the floor of the sample compartment, just enough to give stability to the ATR unit. The support foot locking nut (**4**) is tightened to the underside of the base (**5**) to secure the sample foot (**1**) in position.

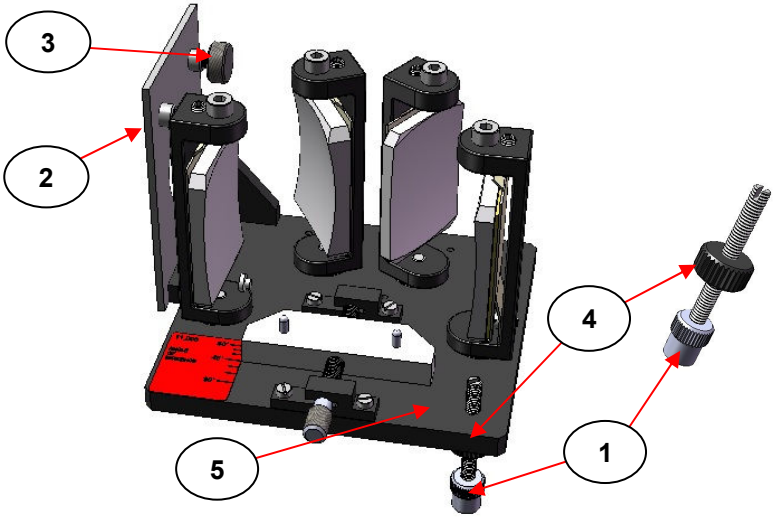


Fig 1. 3" x 2" Mount Plate and Support Foot Fixings

Initial Alignment Procedure

With the 25 Reflection ATR unit securely installed in the sample compartment of the spectrometer, take the supplied ZnSe crystal in the solid sample holder assembly (**6**) and place it into position on the movable translation stage (**7**).

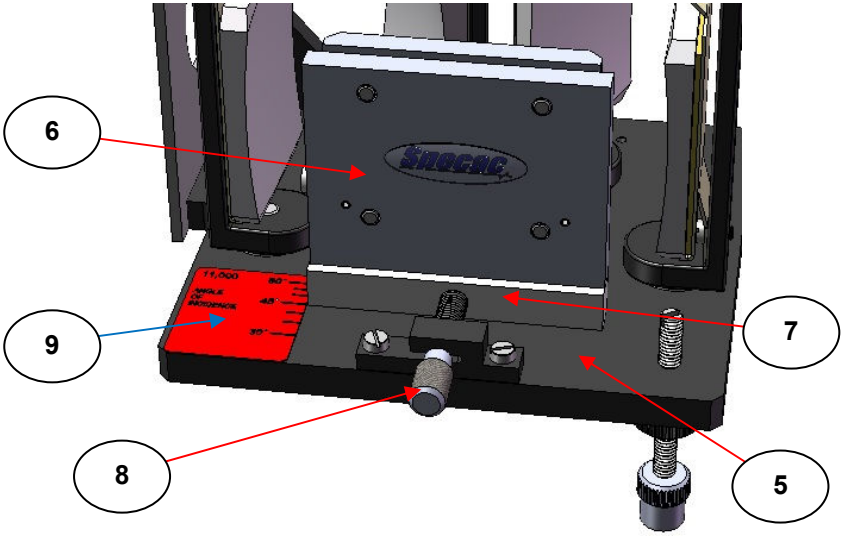


Fig 2. Translation Stage with Fitted ATR Crystal and Solids Holder Assembly

A specific sample holder (**6**) (solids, paste or liquid version) mounts on the movable translation stage (**7**) of the 25 Reflection ATR unit via two fixing pins. The longer edge of the crystal holder assembly (**6**) faces towards the knurled adjustment screw (**8**) at the front of the ATR unit when positioned correctly on the two fixing pins of the translation stage (**7**). The whole sample holder and crystal assembly (**6**) is then moved to a specific incident angle position by rotation of the knurled adjustment screw (**8**). The incident angle is read from the graduated scale plate (**9**) at the front left hand side of the base (**9**). For a correct incident angle of the beam (e.g. 45°) to pass through the crystal, ***align the front edge of the translation stage*** (**7**) to the required angle

setting as indicated on the graduated plate. (See **Fig 8**, page 31 – the diagram shows the solids holder and crystal assembly on the translation stage set at the 45º incident angle).

The four mirrors (**M1**, **M2**, **M3** and **M4**) on the 25 Reflection ATR unit have been factory aligned before despatch of the accessory and positioned so that when the unit is viewed from above, the back surface of the mirrors **M1** and **M4** are aligned with the fiducial (spot) marks (**10**) (see example for **M1**, **Fig 8.),** on the base (**5**).

It should now only be necessary to make minor adjustments for the rotation and tilt setting of the mirrors for the supplied ZnSe crystal and solids holder assembly (**6**) as fitted at the 45° incident angle setting, so that a maximum (optimum) transmission can be obtained for the spectrometer being used.

Fine Mirror Adjustment

When the 25 Reflection ATR unit with the supplied ZnSe crystal in the solids sample assembly (**6**) fitted to the translation stage (**7**) has been set at the 45° incident angle, fine adjustment of the mirrors is carried out to optimize for an IR beam energy throughput level.

On the IR spectrometer system being used, select a mode of operation that registers the IR beam energy level passing through the IR spectrometer sample compartment. With nothing in the sample compartment, an energy level throughput indication (e.g. voltage level, or an arbitrary number value etc), can be considered as a 100% throughput of light energy. When the 25 Reflection ATR accessory with the ZnSe crystal and solids holder assembly (**6**) is installed, there should be a registering of a lower light throughput level at the detector and this acts as the start point level prior to any mirror adjustment. A throughput level to achieve from any alignment adjustment is circa 20% by comparison to the open beam energy level value with the supplied ZnSe crystal assembly (**6**).

Note: *The procedure for alignment that follows is described for a **left to right** beam direction system as shown for **Fig 8**. For a **right to left** beam dircetion system start at mirror **M1** to **M2** etc.*

The mirrors can be ***rotated*** by use of the supplied 2.5mm Allen key in the cap head screw (**11**) at the top of the mirror mount assembly.
The mirrors can be ***tilted*** by use of the supplied 2.0mm Allen key in the grub screw (**12**) at the top of the mirror mount assembly and rotating clockwise or anticlockwise.

Adjust one mirror at a time for its optimum energy level setting, before moving to another mirror to adjust in the sequence.

Note: *Care should be taken when adjusting a mirror for tilt. The grub screw (**12**) is wedged against the spring mirror support backplate and if turned too far anticlockwise becomes dislodged from this position. If this happens push the mirror support backplate gently forward and turn the grub screw (**12**) clockwise again until it is wedged correctly again.*

1) To fine tune for optimisation of the energy throughput being registered, start with adjustment of the output mirror **M4**. Rotate and/or tilt this mirror to peak up the signal as registered by the detector of the spectrometer system. If rotation or tilt of the mirror **M4** in one direction peaks for the signal level and then starts to reduce in value, adjust in the opposite direction until an optimum peak value is reached.
2) Now try adjustment of mirror **M3** for rotation and/or tilt and see how this affects the signal. If the signal is increased from any slight adjustment to **M3**, you may need to slightly readjust mirror **M4** again for its rotation and /or tilt to redirect the beam accordingly to the detector.
3) With the ***output side optics*** adjusted accordingly, now check the input side starting at mirror **M1**. Rotate and/or tilt **M1** to maximise the signal throughput being detected.
4) Now check the position of mirror **M2**. Rotate and/or tilt to maximise the signal. Similarly for any adjustment that was made to **M3** for the output optics side, if the signal level overall is increased by adjustment of the ***input mirror*** **M2,** you may need to readjust mirror **M1** slightly.
5) Finally, recheck the ***output optics side again*** starting at mirror **M4**. (See steps 1) and 2)).

Mirror Alignment When Changing The Incident Angle

When the angle of incidence is changed for the position of the sample holder and crystal assembly (**6**) on the translation stage (**7**), it should only be necessary to carry out **rotational positioning** of the mirrors **M2** and **M3** to complete the beam path through the ATR crystal from the source to detector of the spectrometer system.

Mirrors Misaligned

Note: *If the 25 Reflection ATR accessory is placed into a spectrometer and does not register a throughput signal, then it is possible that the accessory is out of alignment. However, it is unlikely that it will be known which mirror or optical component is at fault? Therefore, if ever the accessory is found to be out of alignment due to incorrect positioning of any of the mirrors, then a visible form of light is necessary to follow the procedure for correct alignment* ***prior*** *to introduction of the accessory into the infra-red spectrometer system.*

It is very helpful to have a form of coherent light (laser etc), to trace the light path from the **M1** and **M2** mirrors to obtain a spot of light that is centralised on the ATR crystal angled surface face from the input side.

Place a crystal holder assembly with a fitted crystal (**6**) into position on the translation stage (**7**) and set at the 45° incident angle mark on the gauge indicator (**9**). Set (rotate) Mirror **M1** to its fiduciary mark (**10**) and then shine ***an incoming parallel beam of laser light*** to the centre of mirror **M1**. Then adjust the tilt of mirror **M1** to send a spot of light to the centre of mirror **M2**. Adjust mirror **M2** from its rotation and tilt to direct the spot of light centrally to the ATR crystal angled face as fitted into the crystal holder assembly (**6**).

When a spot of laser light is seen at this position on the ATR crystal face, install the 25 Reflection ATR accessory into the spectrometer, replacing the laser light with the spectrometers own IR beam source. It may only be necessary now to adjust mirror **M3** to continue the IR beam to mirror **M4** and then to the detector to re-establish a signal energy throughput level within the IR spectrometer system.

5. Sampling With The 25 Reflection ATR Accessory

The 25 Reflection ATR accessory as P/N GS11000 is supplied with a solids sample holder P/N GS11001 fitted with a ZnSe ATR crystal P/N GS11014 as standard. A paste holder P/N GS11002 and a liquids holder P/N GS11003 are also available for use.

All the sample holder assemblies (solid, paste and liquid) can be fitted with the choice of ATR crystal options from ZnSe (P/N GS11014), KRS-5 (P/N GS11004), Ge (P/N GS11006) and Si (GS11009).

The choice of an ATR crystal material to use can be dependent upon the wavenumber range of transmitted light to study that the crystal offers, but will mostly be determined as to the nature of the sample itself for chemical tolerance and resistance of the crystal material. (e.g. ZnSe has a pH tolerance range typically between pH4 to pH11 at room temperature conditions – see ZnSe materials datasheet page 29.) If you are in doubt as to whether a sample type will affect the ATR crystal from contact, it may be useful to try a test fragment of the crystal material type first if available.

After the initial installation and optimisation for alignment procedures have been carried out (see Section 4 of this User Instruction Manual), the solid crystal holder assembly (**6**) can be deconstructed for removal of the fitted ZnSe crystal to use for sample mounting and measurement of an ATR spectrum for the sample type.

Sample Holder Types and Their Use

Solids Holder P/N GS11001

The solids holder P/N GS11001 consists of two metal clamp plates (**13** and **14**) being held together by four clamping bolts (**15**). The ATR crystal (**16**) (ZnSe as standard), is sandwiched between the clamp plates for the analysis of ***solid sample types***. (See **Fig 3**.)

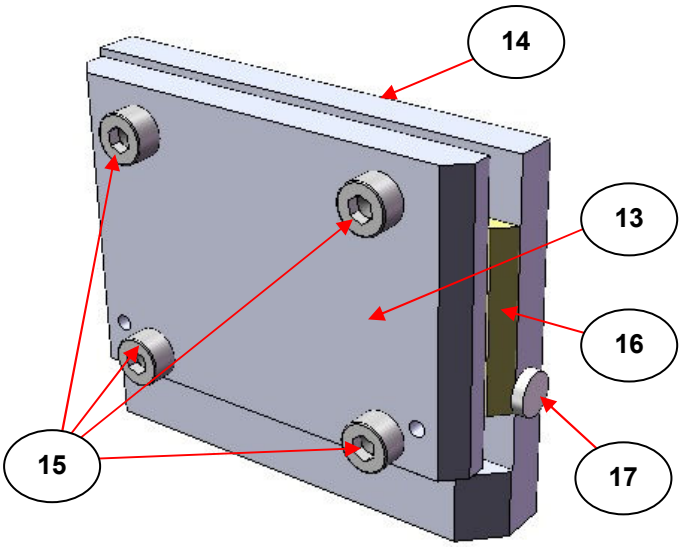


Fig 3. Solids Holder Assembly with ATR Crystal

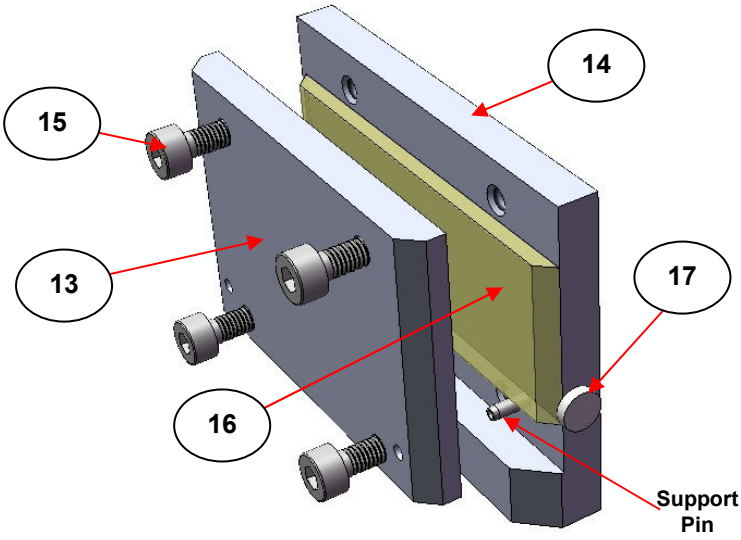


Fig 4. ATR Crystal as Positioned in the Solids Holder

For an assembly of the solids holder, the longer surface face of the trapezium shaped ATR crystal (**16**) is placed against the face of the longer clamp plate (**14**) (to the front of the holder) and the shorter surface face of the ATR crystal is placed against the face of the shorter clamp plate (**13**) (at the back of the holder). For correct positioning, the bottom edge of the ATR crystal sits upon the two support pins with one end (the apex tip) resting against the nylon end stop (**17**). (See **Fig 4**.)

Deconstructing The Solids Holder Assembly

To deconstruct the supplied solids holder assembly with fitted ZnSe crystal (**6**), place the entire assembly on a soft cloth for working with the long clamp plate (**14**) face downwards and carefully undo and remove the four clamping bolts (**15**) (screw anticlockwise) using the 2.5M Allen key supplied.

The shorter clamp plate (**13**) can then be carefully removed away from the ATR crystal (**16**), and the ATR crystal can then in turn be carefully removed away from the longer clamp plate (**14**) surface.

Constructing The Solids Holder Assembly

Construction of the solids holder assembly fitted with an ATR crystal (**6**), can be made in two ways. One way is for obtaining a background reference ATR spectrum (no sample), and the other way is filling the solids holder with a sample between the clamp plates (**13**) and (**14**) and ATR crystal (**16**) surfaces.

Note: *The solids holder with fitted ZnSe ATR crystal assembly (**6**) as supplied is a construction for reference background spectrum collection. (No sample inclusion).*

Construction For Reference Background Spectra

To construct the solids holder with a fitted ATR crystal to obtain any ***background/reference spectra***, the procedure is as follows.

Build the sample holder assembly as a “sandwich” of front (longer) clamp plate (**14**), ATR crystal (**16**) and back (shorter) clamp plate (**13**) components to be tightened together in this sequence.

For correct positioning, the bottom edge of the ATR crystal (**16**) sits upon the two support pins with one end (the apex tip) resting against the nylon end stop (**17**). (See **Fig 4**.)

Insert the four clamping bolts (**15**) through the holes of the shorter clamp plate (**13**) to locate in their respectively aligned threaded screw holes of the longer clamp plate (**14**). Before tightening the bolts securely, ensure that the crystal (**16**) is still positioned correctly in the holder, such that neither of the crystal input/output angled faces are going to be obscured by the rear clamp plate (**13**). (If the crystal (**16**) is resting against the nylon end stop (**17**), then the angled faces should not be obscured when the assembly is made).

Proceed to tighten the 4 clamp bolts (**15**) using the 2.5M Allen key supplied to squeeze all the components together. The ATR crystal (**16**) should be secured for an even and level fit between the inner surfaces of the two clamp plates (**13**) and (**14**).

Important: *Be very careful when tightening the bolts (**15**) when the ATR crystal (**16**) is held secure between the clamp plates (**13**) and (**14**), such that there is a minimal risk to the ATR crystal being damaged from any overtightening.*

Construction For Sample Spectra

To construct the solids holder with a fitted ATR crystal to obtain any ***sample*** spectra, the procedure is as follows.

Build the sample holder assembly as a “sandwich” of front (longer) clamp plate (**14**), sample, ATR crystal (**16**), sample and back (shorter) clamp plate (**13**) components to be tightened together in this sequence. It is easier to build such a sandwich structure of components with the solids holder assembly laying on one side and having the longer clamp plate (**14**) as the bottom component.

When filling the solids holder with a sample, both surfaces of the ATR crystal (**16**) should be in contact with a sample to be subject to the maximum number of reflections events for measurement. (25 reflection events at a 45° incident angle setting for the solids holder assembly (**6**) on the translation stage (**7**).) There also needs to be sufficient sample to cover as much of the two surface areas of the ATR crystal (**16**) as possible, to be subject to the same maximum number of reflection events for the spectral measurement.

Tip: *The best results for sampling may be found if the solid sample as a film, for example, is specifically cut to size to cover the different crystal surface areas, prior to building the sandwich construction of components in the sequence.*

For correct positioning of the sample with the ATR crystal (**16**), the bottom edge of the ATR crystal (**16**) sits upon the two support pins with one end (the apex tip) resting against the nylon end stop (**17**). (See **Fig 4**.) The sample covering both surfaces of the crystal in the construction ***must not be larger than the surface area of the crystal itself.***

Insert the four clamping bolts (**15**) through the holes of clamp plate (**13**) to locate in their respectively aligned threaded screw holes of clamp plate (**14**). Before tightening the bolts, ensure that the crystal (**16**) and sample surfaces are still positioned correctly in the holder, such that neither of the crystal input/output angled faces are going to be obscured by the rear clamp plate (**13**). (If the crystal (**16**) is resting against the nylon end stop (**17**), then the angled faces should not be obscured).

Proceed to tighten the 4 clamp bolts (**15**) using the 2.5M Allen key supplied to squeeze all the components together. The ATR crystal (**16**) with its own layers of sample surfaces should be secured for an even and level fit between the inner surfaces of the two clamp plates (**13**) and (**14**).

Solid samples, films and powders can all be mounted in the solids holder in a similar way.

Important: *Be very careful when tightening the bolts (**15**) when the ATR crystal (**16**) with a sample is held secure between the clamp plates (**13**) and (**14**). It is important to get good contact of the sample with the ATR crystal surfaces to achieve an ATR spectrum, but over-tightening of the components together could cause the crystal to break or become deformed.*

Sampling Procedure

Initially, to take a reference spectrum for the ATR crystal (**16**) being used in the 25 Reflection ATR accessory, mount the crystal in the solids holder ***without*** any sample. Tighten the components together and place the solid holder and crystal assembly onto the movable translation stage (**7**). Adjust the knurled screw (**8**) for the selection of the desired incident angle to set and make any necessary adjustments to the mirrors to peak up the signal. Collect a reference spectrum.

Remove the solids holder and crystal assembly (**6**) from the translation stage (**7**) and proceed to deconstruct the solids holder and crystal assembly (**6**) in readiness to place the sample into position in the holder.

Reconstruct the solids holder with a sample and then place the complete assembly back onto the translation stage (**7**) to take a sample spectrum measurement.

Note: *Be very careful not to move the position of the ATR crystal and solids holder assembly between the reference and sample spectrum collection stages.*

When the analysis and spectral collection of a sample has been achieved, **ALWAYS** remove the sample and crystal from the holder assembly and clean and store it properly for use the next time. Prolonged clamping of the crystal with a sample (particularly if a crystal as soft as KRS-5 has been used), will cause irreparable damage to the crystal and its performance for ATR will be compromised. (See Notes on Cleaning – page 24.)

Paste Holder P/N GS11002

As an alternative to the solids holder P/N GS11001, the paste holder P/N GS11002 can be used in the 25 Reflection ATR accessory for the measurements of pastes or semi-solid types of sample.

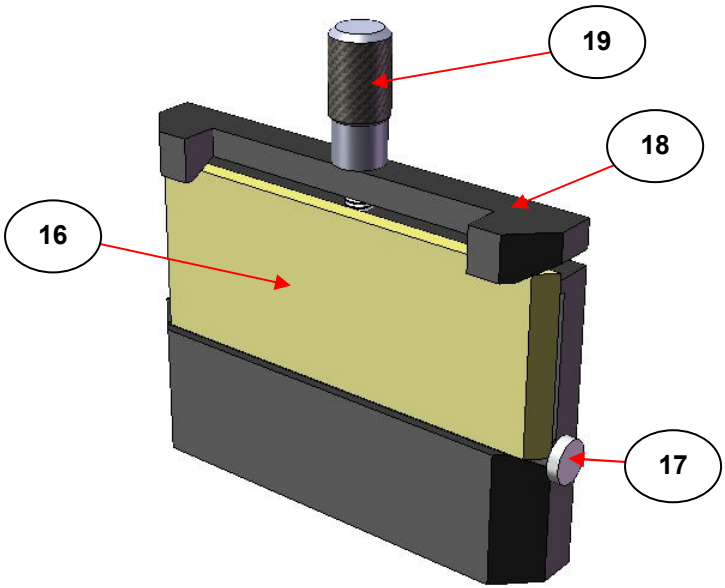


Fig 5. 25 Reflection ATR Paste Holder GS11002

The paste holder is made from Delrin material with the ATR crystal (**16**) being held in place against an upright support plate by a top clamp bar (**18**) and screw knob (**19**). (The longest surface of the trapezium shaped ATR crystal (**16**) rests against the support plate of the holder). Paste samples are applied directly to both sides of the ATR crystal. For sample introduction ensure that the long surface of the crystal (**16**) is coated with the paste first and when the crystal has been placed and clamped into position, carefully apply the paste sample to the shorter crystal surface, but take care not to contaminate the crystal faces where the light beam enters and exits the crystal.

To remove the ATR crystal, the knob (**19**) at the top of the paste holder is unscrewed and the top clamp bar (**18**) is lifted clear. This allows the crystal to be pulled up and out of the holder. Insertion of the ATR crystal (**16**) into the holder (with or without sample) is the reverse procedure. Similarly, as with the solids holder P/N GS11001, the ATR crystal (**16**) is correctly positioned in the paste holder P/N GS11002 when the apex tip of the crystal is touching the nylon end stop (**17**) as seen in **Fig 5**.

The procedure for sampling and instructions for care and handling of any of the ATR crystal material options in the paste holder P/N GS11002 are the same as for use of the solids holder P/N GS11001.

Liquids Holder P/N GS11003

As an alternative to the solids holder P/N GS11001, the liquids holder P/N GS11003 can be used in the 25 Reflection ATR accessory for the measurements of liquid types of sample. (See **Fig 6.**)

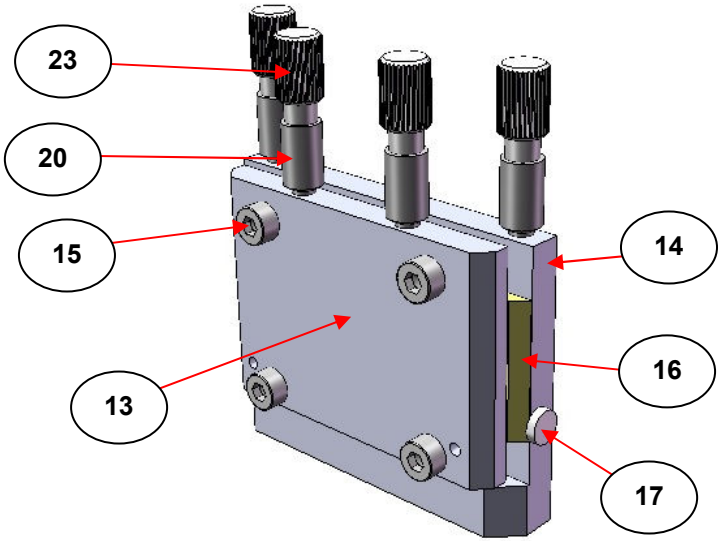


Fig 6. 25 Reflection ATR Liquids Holder GS11003

The liquids holder is very similar in construction to the solids holder, but there are two Luer connection fittings (**20**) at the top of each clamp plate (**13**) and (**14**) to allow for the introduction and flow of a liquid sample to the ATR crystal (**16**) surface. The ATR crystal (**16**) is held away from direct contact to the clamp plates (**13**) and (**14**) inner surfaces by a PTFE gasket (**21**) and (**22**) which also acts as a seal to contain the liquid. There is a long PTFE gasket (**21**) for the longer ATR crystal surface and a shorter PTFE gasket (**22**) for the other surface.

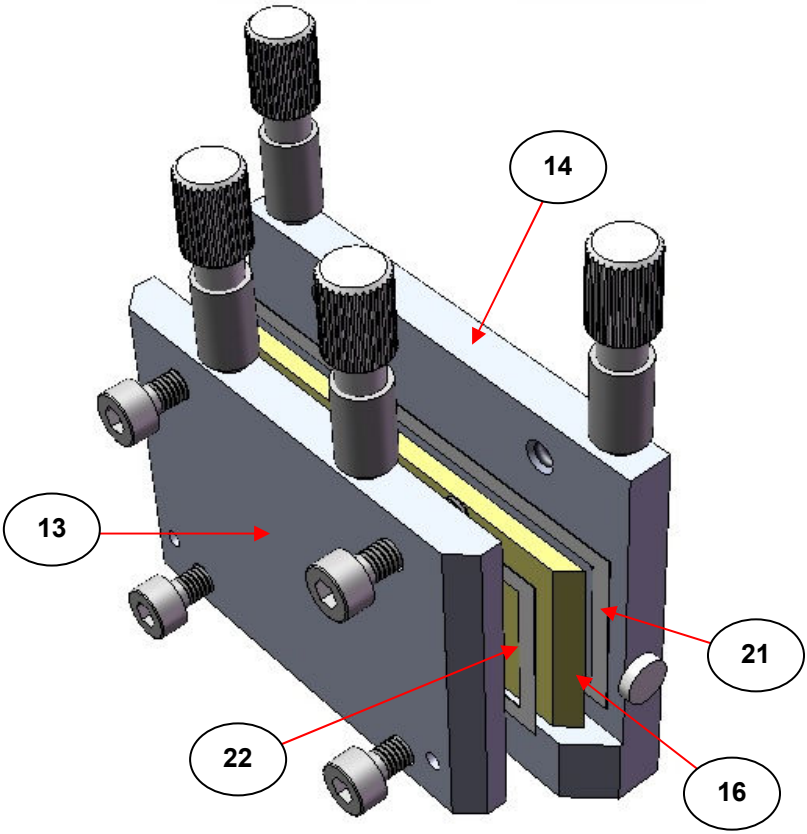


Fig 7. ATR Crystal with Gaskets for Sample Cavities as Positioned in the Liquids Holder

Therefore, in a construction of the liquid sample holder, it consists as a sandwich of front clamp plate (**14**), long PTFE gasket (**21**), crystal (**16**), short PTFE gasket (**22**) and back clamp plate (**13**). (See **Fig 7**.) For a construction of the liquids holder, it is very similar in the way that the solids holder would be built when containing a film sample as the example given. (See pages 16 and 17.) The PTFE gaskets (**21**) and (**22**) as "substitutes" for a film sample, must be fitted carefully to cover over both crystal surfaces around their perimeters, and not to obscure the two fill holes in the clamp plate for introduction of a liquid sample. In actual construction, the longer gasket (**21**), crystal (**16**) and shorter gasket (**22**) "sandwich" will rest on the two protruding support pins at the base of the longer clamp plate (**14**). Similarly, as with the solids holder P/N GS11001, the ATR crystal (**16**) is correctly positioned in the liquids holder P/N GS11002 when the apex tip of the crystal is touching the nylon end stop (**17**) as seen in **Fig 6**.

When filling the liquids holder with a sample remove the PTFE stopper (**23**) from the Luer port (**20**). Introduce the liquid via the Luer port (**20**) connected to enter through the ***lower port hole*** of the clamp plate next to the crystal, so that the second ***upper port hole*** acts as a vent. Sufficient liquid should be introduced to expel the air from the cavity created by the PTFE gaskets (**21** and **22**) between the crystal (**16**) and the respective metal clamp face (**13** and **14**).

Both sides of the crystal (**16**) need to be filled by a liquid sample to attain 25 ATR Refection events at the 45° incident angle setting in positioning of the translation stage (**7**). When filled, close all four Luer ports (**20**) with the PTFE cap plugs (**23**) provided.

Note: *The liquids holder and crystal assembly is not suitable for high pressure liquid system analysis.*

After use, the ATR crystal (**16**) must be removed and cleaned ready for use next time. When replacing the crystal into the liquids holder ensure that the PTFE gaskets (**21** and **22**) which seal the liquid are not damaged and are correctly positioned so that they form a tight seal around the edge of the crystal.

Notes On Cleaning

When cleaning a ZnSe, KRS-5, Ge or Si ATR crystal of the 25 Reflection ATR accessory 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 these four crystal materials ZnSe and KRS-5 are potentially the most hazardous in terms of risk of toxicity from contact with the skin.

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

A useful feature of the 25 Reflection ATR accessory is the capability for removal of the ATR crystal and crystal holder assembly away from the optical unit, such that any sample can be prepared remotely and safely, onto the ATR crystal surface. The resulting crystal holder with fitted ATR crystal and sample assembly can be bought for fitting onto the optical unit whilst installed in the spectrometer.

Solvents such as water, methanol and acetone are suitable to use for cleaning purposes with the ATR crystals. 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 crystal, **particularly if using the ZnSe or KRS-5 crystals** as these crystal materials are not as resilient as Ge or Si material in general. Scratches and blemishes to the ATR crystal surface will result in poor light throughput for the ATR technique and an overall degradation in the accessories performance. It should only be necessary to clean away at the surfaces of the ATR crystal that have contacted against the sample itself. If possible, try to avoid any solvent or cleaning solution materials from getting to the angled faces of the ATR crystal. There is a risk that any dried solution components that have been introduced to these surfaces could be deemed an “impurity” against the ATR crystal in any background/reference 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 KRS-5

General

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

Physical Data

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

Stability

Stable.

Toxicology

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

Personal Protection

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

Storage

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

6. "Bubble Numbers" Part Identification List

(**1**) Support foot.
(**2**) 3" x 2" slide mount plate.
(**3**) Locking screw on 3" x 2" slide mount plate.
(**4**) Support foot locking nut.
(**5**) Base of 25 Reflection ATR accessory.
(**6**) ATR crystal in solids holder complete assembly.
(**7**) Movable translation stage for sample holder/crystal assemblies.
(**8**) Knurled adjustment screw for incidence angle setting of translation Stage (**6**).
(**9**) Incident angle indicator scale.
(**10**) Fiducial marks for mirrors M1 and M4 alignment
(**11**) Cap head screw for mirrors **M1**, **M2**, **M3** and **M4** rotation.
(**12**) Grub screw for mirrors **M1**, **M2**, **M3** and **M4** tilt.
(**13**) Solids holder metal clamp plate (short).
(**14**) Solids holder metal clamp plate (long).
(**15**) Solids Holder clamp plate bolt (4 off).
(**16**) 25 Reflection ATR crystal.
(**17**) Nylon button end stop for ATR on solids holder.
(**18**) Paste holder top clamp bar.
(**19**) Paste holder knob for top clamp bar (**18**).
(**20**) Liquid holder Luer port connection.
(**21**) Liquid holder PTFE gasket (long).
(**22**) Liquid holder PTFE gasket (short).
(**23**) Liquid holder PTFE cap plug for Luer port (**20**).

7. Spare Parts for 25 Reflection Variable Angle ATR Accessory

P/N GS11000 25 Reflection ATR Accessory with solids holder and ZnSe ATR crystal.
P/N GS11001 Solids sample holder only (no crystal).
P/N GS11002 Paste sample holder only (no crystal).
P/N GS11003 Liquid sample holder only (no crystal).
P/N GS11004 KRS-5 ATR crystal 52mm x 20mm x 2mm (25 Reflections 45º angle).
P/N GS11006 Germanium ATR crystal 52mm x 20mm x 2mm (25 Reflections 45º angle).
P/N GS11009 Silicon ATR crystal 52mm x 20mm x 2mm (25 Reflections 45º angle).
P/N GS11014 Zinc selenide ATR crystal 52mm x 20mm x 2mm (25 Reflections 45º angle).
P/N GS11008 5 Pairs of PTFE gaskets for P/N GS11003 liquids holder (1 long, 1 short).

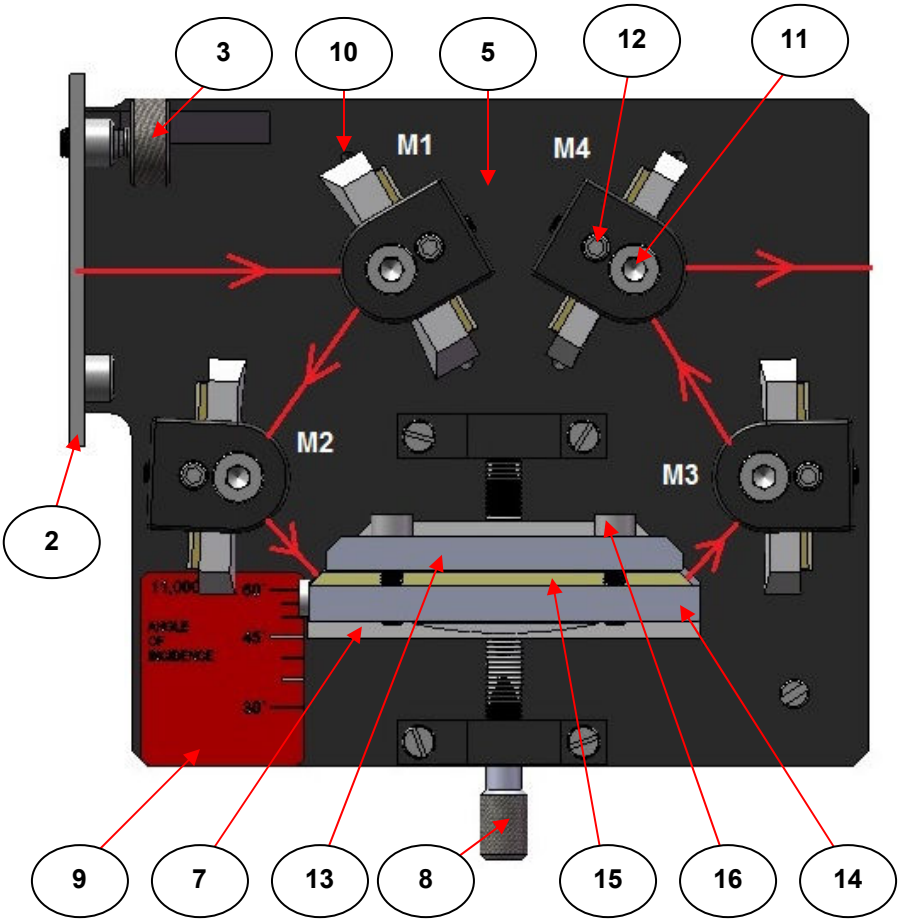


Fig 8. Optical Path Diagram and Parts Identification for the 25 Reflection Variable Angle ATR

Notes For Use Of 25 Reflection ATR Accessory

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