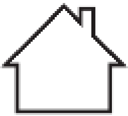
Low Temperature Golden Gate® Diamond ATR System

User Manual

2I-10590 Issue 11

List of Safety Symbols

This manual should be consulted when any of the Safety Symbols below appear, to understand the nature of the Hazards and any actions to be taken to avoid them.

Safety Symbol	ISO 7000 symbol meaning
	**CAUTION!**
	**WARNING – Hot Surface** (Reference ISO 7010-W017)
	**For indoor use only** (Reference IEC 60417-5957)
	**WARNING – Low Temperature/Freezing conditions** (Reference ISO 7010-W010)

Caution! For indoors use only. Do not subject to dripping or splashing. Not for use in wet locations.

CAUTION! Only use the configured temperature controller supplied with this accessory.

CAUTION! This user instruction manual should be read in conjunction with the temperature controller and Golden Gate^tm manuals provided separately with the respective Specac accessory.

Low Temperature Golden Gate® Diamond ATR

User Manual

2I-10590 Issue 11

Low Temperature Golden Gate® Diamond ATR Models GS10590 / GS10592

1. Introduction

Thank you for purchasing a Specac Product.

The Low Temperature Golden Gate single reflection diamond ATR top plate is designed for use on the Golden Gate ATR optical base unit. It is used to study liquid or solid samples at temperatures from −150°C to 80°C. Liquid samples are analysed by simply covering the diamond ATR crystal, and the standard Golden Gate clamp bridge is used to provide pressure for solid samples, providing excellent sample contact.

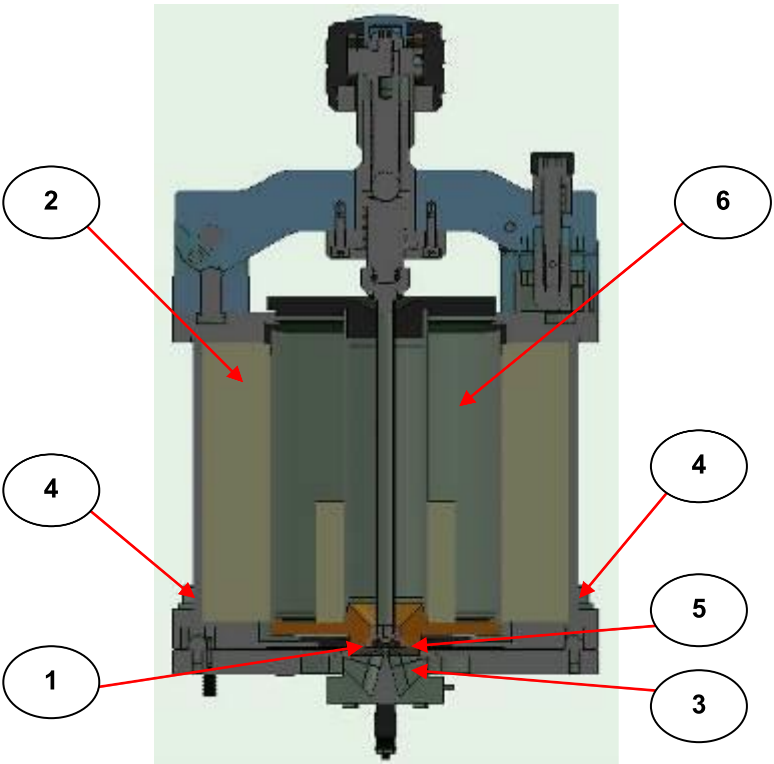


Fig 1. Sectional Front View of Low Temperature ATR Golden Gate Top Plate

The analysis area of the top plate is a heatable diamond crystal bonded into a tungsten carbide support disk (**1**), surrounded by a cooling jacket dewar assembly (**2**). The dewar assembly is clamped to the heated diamond ATR top plate (**3**) by four clamping screws (**4**). There is a heat transfer gasket (**5**) between the diamond/ tungsten carbide disk (**1**) and the centre part of the dewar (**2**). (Please see Figs 1 and 2.)

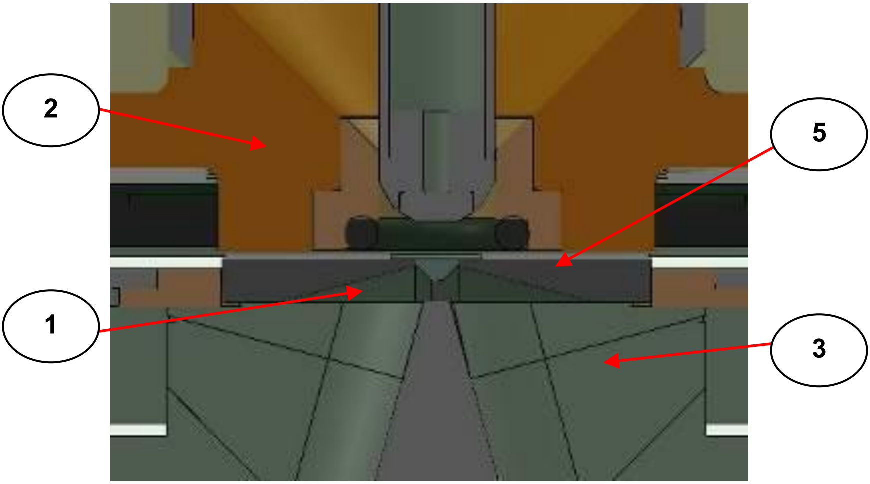


Fig 2. Diamond/Tungsten Carbide Puck and Gasket detail area of Low Temperature Golden Gate Top Plate

Using liquid nitrogen as an example refrigerant in the dewar chamber (**6**) and from control of power to the heaters, temperatures below ambient can be achieved. For temperatures above ambient, no refrigerant is required and the heaters for the diamond crystal are used alone. Power to the heaters is provided by a dedicated 4000 Series Temperature Controller. A separate instruction manual is provided for operation of the Temperature Controller with the Low Temperature Golden Gate ATR top plate.

2. Packing and Checklist

On receipt of your Low Temperature Golden Gate ATR accessory please check that the following have been provided.

- Low Temperature Golden Gate single reflection diamond ATR top-plate (P/N GS10590).
- Golden Gate Optical unit with choice of ZnSe or KRS-5 lenses and appropriate Benchmark baseplate (if ordered as a complete Golden Gate system (P/N GS10592)).
- 4000 Series Temperature Controller, instruction manual and power cables.
- Flat “transfer load” anvil to sapphire tipped rod assembly.
- Sapphire tipped rod assembly.
- Liquid sample injector consisting of an insulated 16-gauge hypodermic needle and glass syringe.
- Plastic funnel
- Allen key (2mm).
- Allen key (3mm).
- Hex head ball driver (3mm).
- Long T bar type Allen key (5mm).
- Packet of glass filled PTFE gaskets (5).
- Packet of high thermal transfer gaskets (20)
- Spare Viton ‘O’ ring seal.
- An Essential Spares Kit of parts (P/N GS10550)

Carefully remove the equipment from their packaging and proceed to install into your spectrometer system.

3. Safety of Use

WARNING – Low Temperature / Freezing conditions

The Low Temperature Golden Gate ATR system requires the use of liquid nitrogen (LN_2) for operation at sub ambient temperatures. When using refrigerants, ensure all local laboratory safety procedures are followed and wear appropriate personal protective equipment (PPE). Recommended PPE gloves and glasses should be worn at all times when operating this equipment.

Always use a suitable plastic funnel assembly for filling of the dewar with liquid refrigerant to avoid accidental spillage to other areas of the equipment. The funnel supplied (**7**) can be used either for introduction of a liquid refrigerant through the small hole (**8**) or the crescent shaped cut out (**9**) in the dewar cap (**10**). (See Fig 2).

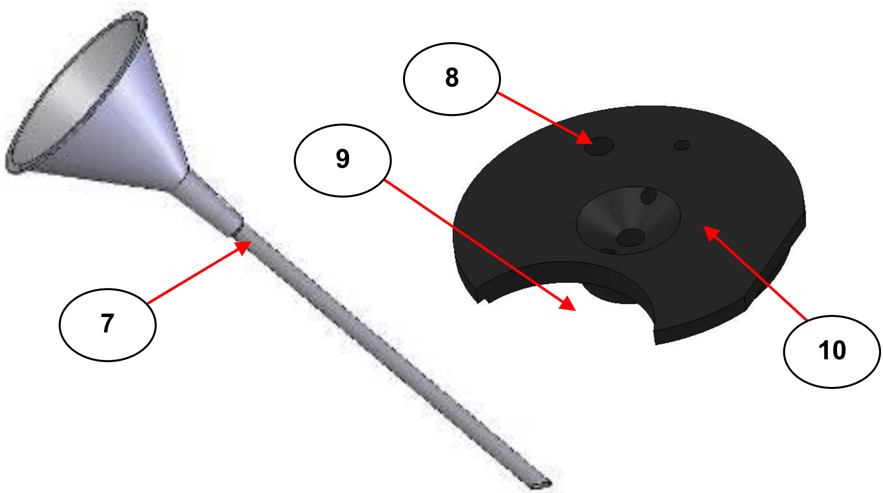


Fig 2. Dewar Cap and Funnel Parts of Low Temperature Golden Gate ATR Accessory

Safety Warnings

Caution: Always follow local laboratory safety protocols and procedures when using potential toxic or flammable substances within this equipment.

Caution: The equipment is intended for use by suitably trained personnel only.

Caution: Always wear adequate PPE gloves when handling the housing if they are to be removed or fitted to or from the spectrometer and when cleaning.

Caution: No user serviceable parts within, contact the manufacturer or approved service agent for advice if the product is not functioning correctly or is visibly damaged.

Warning: Burn Hazard, when operated at high temperatures allow hot surfaces to cool down before handling, risk of burns.

Warning: Heating of materials could lead to the liberation of hazardous substances.

Warning: If the heating of materials could lead to the liberation of hazardous substances or gases, explosion or implosion, then an extraction system and/or a temperature limiting device, relating to the materials and safe temperatures for the materials used, should be installed.

Warning: Any pressure build-up needs to be monitored using a suitable pressure gauge in the pressure line for non-flowing experiments. **You must make sure that any temperature increase throughout any experimentation reaction process does not lead to a pressure rise exceeding the max stated pressure of this product.**

When the experiment is complete, reduce any pressure in the system and allow the reaction chamber to cool before flushing through with a suitable solvent.

Warning! Before use it should be confirmed that the sample to be tested in experimentation is compatible with all of the materials listed. **Specac cannot be held responsible for any damage or breakdown of the equipment that may be caused as a result by chemical attack from unsuitable materials. Whenever the equipment is not being used, ensure it has been thoroughly cleaned and dried before storage.**

Caution: If the equipment is used in a manner not specified within this manual, the protection provided by the equipment may be impaired

Warning: Should potentially hazardous or hazardous materials be used or spilled onto or into the equipment the RESPONSIBLE BODY shall ensure that the appropriate decontamination is carried out and that no decontamination or cleaning agents are used which could cause a HAZARD as a result of a reaction with parts of the equipment or with material contained in it, if in any doubt about the compatibility of decontamination or cleaning agents contact Specac Technical Support for further information.

Only clean with a soft, lightly dampened cloth. Do not use harsh and/or abrasive cleaners on any Specac product. Consult Specac or their authorized agent if in any doubt.

Always check before use that the safety protection provided has not been compromised or impaired. The temperature controller should be made inoperative and secured against unintended operation if in any doubt.

The protection is likely to be impaired if for example if:

- There is visible damage.
- Fails to perform its intended use.
- Has been subjected to adverse storage conditions.
- Has been subjected to severe transport stresses.

End of Lifetime Equipment Use

If any parts have reached their limit of lifetime and need to be replaced, use appropriate WEEE and other local regulations for the safe disposal of electrical equipment and toxic chemicals.

4. Installation

For the Low Temperature Golden Gate ATR system, P/N GS10592 the system is fully assembled before shipping (See Fig.3).

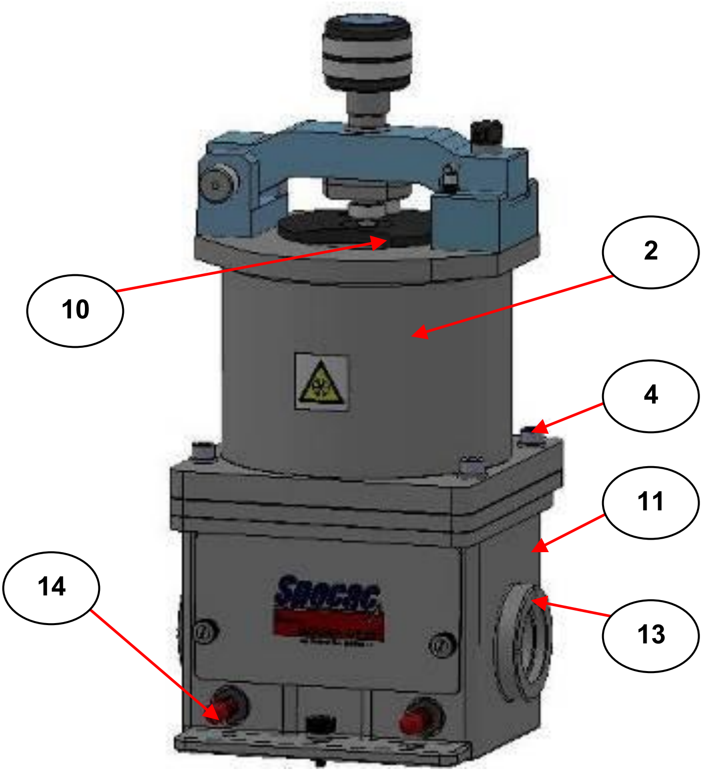


Fig 3. Low Temperature Golden Gate ATR System

Once installed into the host spectrometer, connect the heater and thermocouple cables to the 4000 Series Temperature Controller (Not shown). Refer to the 4000 Series 150W temperature controller instruction manual for further details.

For Low Temperature Golden Gate ATR top plate upgrade (P/N GS10590), the heated top plate is attached to the dewar assembly before shipment. The dewar assembly and will need to be separated from the heated top plate to enable fitment of the Golden Gate optical unit. The dewar assembly is then re-installed to complete the assembly.

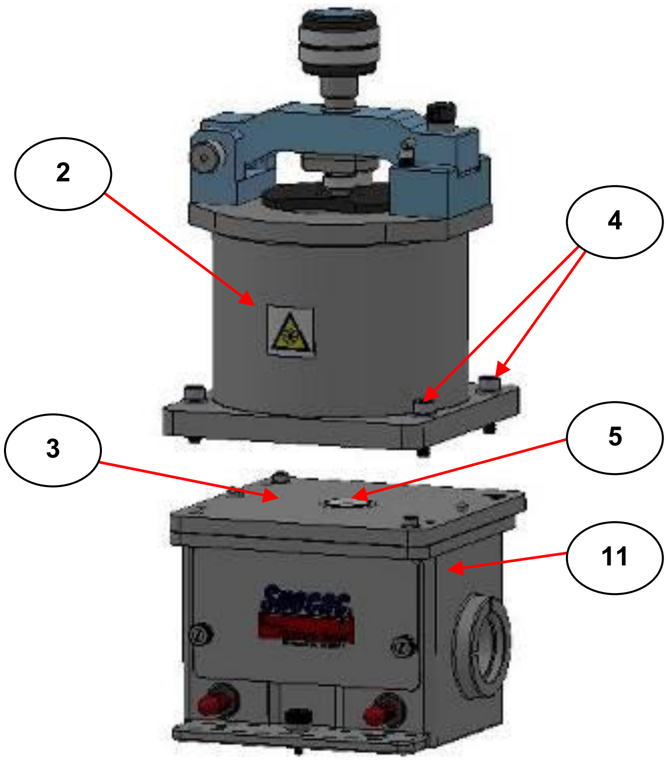


Fig 4. Separation of Low Temperature Golden Gate Dewar Assembly from Heated Top Plate and Optical Unit Assembly

Remove the dewar assembly fixing screws (**4**) (See Fig 4.) using the long T bar type Allen Key (5mm) allowing separation of the dewar from the Golden Gate heated top plate.

The heated top plate assembly (**3**) attaches to the Golden Gate optical unit (**11**) with the two new M4 x 16mm screws (**12**) supplied (See Fig 5.). The new screws replace of the original thumb screws and are shorter in length to allow the fixing of the dewar assembly to the flat stainless steel heated top plate (Refer to P/N GS10500 Golden Gate instruction manual for further details).

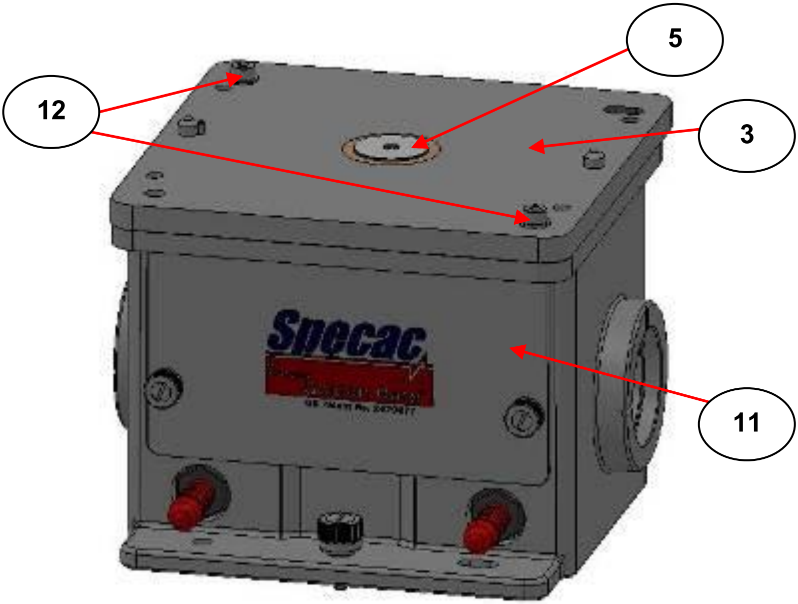


Fig 5. Low Temperature Golden Gate Heated Top Plate and Optical Unit

When the heated top plate assembly (**3**) has been fitted to Golden Gate optical unit (**11**) with the two M4 x 16mm screws (**12**), the cold dewar assembly (**2**) can be re-fitted using the four fixing screws (**4**). When tightening down the dewar fixing screws (**4**) ensure that the two surfaces between the cold dewar assembly (**2**) and heated top plate (**3**) remain parallel and even to ensure the correct thermal contact and sealing of the thermal transfer gasket (**5**) between the dewar assembly (**2**) and the diamond tungsten carbide puck (**1**).

Caution! Before tightening the dewar assembly to the heated top plate assembly ensure that the correct thermal transfer gasket (**5**) (choice of a white glass filled PTFE or grey graphite gasket) has been placed correctly into position over the diamond/tungsten carbide puck. In addition, ensure the gasket (**5**) does not get dislodged and cover / obscure the diamond crystal while fitting the dewar assembly (**2**) over the heated top plate assembly (**3**).

Additional Information:

When conducting the alignment procedure on the optical components (mirrors and lenses) within the Golden Gate optical unit, ensure that the cold dewar assembly (**2**) and the thermal transfer gasket (**5**) has been tightened/screwed down correctly to the heated top plate assembly (**11**). The diamond/puck assembly (**1**) has been designed to be supported under sprung loaded tension and may move slightly when the cold dewar assembly (**2**) is placed into position. Therefore, optimum light beam throughput is achieved when the cold dewar assembly **(2)** is fixed correctly in the working position over the diamond (**1**). See alignment procedure in the Golden Gate instruction manual P/N GS10500 for further details.

When the Low Temperature Golden Gate ATR Accessory has been supplied under P/N GS10592, a white glass filled PTFE thermal gasket (5) fitted by default.

5. Operation of the Low Temperature Golden Gate ATR Top Plate

Important Note for Operation

When using the Low Temperature Golden Gate ATR accessory at temperatures below the ambient dew point, it is **essential** to use and establish a nitrogen purge within the Golden Gate optical unit (**11**) **before** any refrigerant is poured into the dewar chamber (**6**). This is to prevent condensation or frost forming on the internal optics of the Golden Gate optical unit. The purge can be established by using a spectrometers own purge facility (if available) by using the purge bellows (P/N GS10707) to bridge the gap between the Golden Gate apertures (**13**) and the input and output ports of the Spectrometer. (See Purging the Golden Gate in the P/N GS10500 manual.) Alternatively, with the purge bellows fitted to the aperture ports (**13**), the optical unit (**11**) can be purged via the two, purge port hose connectors (**14**) on the front of the Golden Gate optical unit (**11**) using a dedicated dry nitrogen gas supply. (See Fig 3.)

Dewar Cap (10) Nitrogen Purge Connections

A nitrogen purge that is required to prevent frosting of the optical components in the Golden Gate optical unit (**11**) during low temperature operation is also needed for the central internal area of the dewar assembly (**15**) above the diamond/tungsten carbide puck (**1**) when taking reference background spectra for sub-ambient temperature conditions. See Fig 6.

When the dewar cap (**10**) is placed into position, to cover the dewar chamber (**6**), a supply of nitrogen gas can be introduced through one of the 3.5mm diameter purge gas flow holes (**16**) in the dewar cap (**10**). Similar to the optical unit (**11**) gas purge requirement, a purge gas though the dewar cap (**10**) must be established before introduction of any refrigerant. The inner chamber area (**15**) below the cap (**10**) will be filled with the nitrogen gas and a flow of the gas will minimize any risk of condensation forming and possibly freezing to the surface of the diamond ATR crystal. See Fig 7.

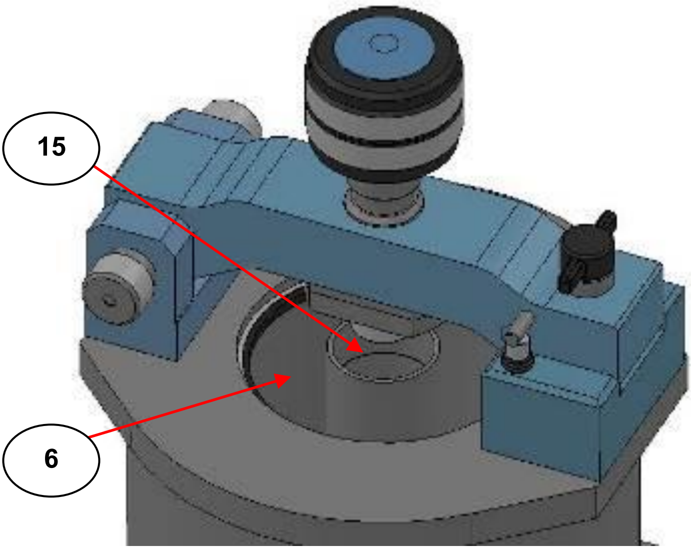


Fig 6. Low Temperature Golden Gate Dewar area with Dewar Cap removed. (Inner and Outer Dewar Chambers)

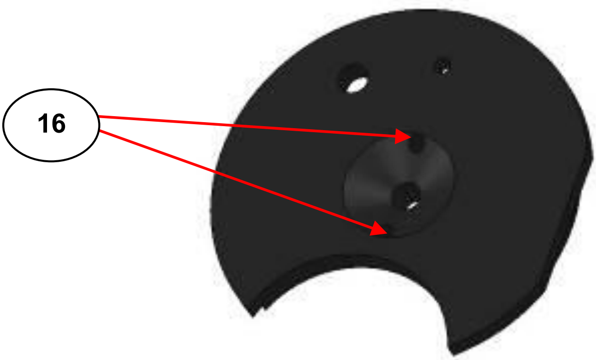


Fig 7. Low Temperature Golden Gate Dewar Cap Purge Holes

CAUTION! Use of alternative refrigerants

The minimum recommended operating temperature is -150°C when used in combination with liquid nitrogen (LN_2) and the graphite high thermal transfer gasket. Other types of refrigerant mixture are listed in the table below.

Table of Refrigerant types

Refrigerant Type	Mixed Temperature (a)		Main Hazard
	°C	°K	
Ice Water	0	273	Cold surfaces
Sodium Chloride/Ice (33g salt/81g ice)	-21	252	Cold surfaces
Calcium Chloride/Ice (100g salt/81g ice)	-40	233	Cold surfaces
Chloroform/Liquid Nitrogen (slush)	-64	209	Cold surfaces
[b]Solid CO2/Ether	-78	195	Fire/Explosion
[b]Solid CO2/Acetone	-78	195	Fire/Explosion
[b]Solid CO2/Isopropanol	-78	195	Fire/Explosion
[b]Toluene/Liquid Nitrogen (slush)	-95	178	Fire/Explosion
[b]Pentane/Liquid Nitrogen (slush)	-130	143	Fire/Explosion
Liquid Air (<21% oxygen)	-147	126	Cold surfaces
[b]Isopentane/Liquid Nitrogen (slush)	-160	113	Fire/Explosion
[b]Liquid Oxygen	-182	91	Fire/Explosion
Liquid Nitrogen (LN_2)	-196	77	Cold surfaces, Asphyxiation

(a) These figures are approximate as actual mixture temperatures are affected by impurities.
(b) Appropriate safety risk assessments and laboratory procedures need to be implemented before use of other alternative refrigerants.

For optimum temperature control in operation, you should choose the refrigerant mix closest to the temperature you wish to study. From the examples above, the natural freezing point of LN_2 is -196°C, but the lowest temperature achievable for the system overall is -150°C using this refrigerant with an appropriate grey coloured graphite high thermal transfer gasket (**5**). It will be a matter of experimentation for the best choice of a refrigerant and gasket (**5**) combination, depending upon the minimum and maximum temperatures to be achieved and the temperature degree of control required.

In general, for temperature operation between -100°C and -150°C the high thermal transfer graphite gasket (**5**) **must** be used. This gasket material allows for these low temperatures to be reached but does not have the same degree of temperature control offered by the low thermal transfer white PTFE gasket (**5**).

For temperature operation between ambient and -100°C the low thermal transfer white PTFE gasket (**5**) should be used, if a finer degree of temperature control and thermal stability is required.

Preparation for Use and General Operation

The successful use of the Low Temperature Golden Gate ATR accessory depends on the correct balancing of a specific refrigerants “heat removing” capability against the energy (heat) input from the electrical block heaters (**17**), built into the heated top plate assembly (**3**) at the base of the dewar assembly (**2**). See Fig 8. The type of experiment being carried out also determines a method of use and, most importantly, the choice of thermal transfer gasket (**5**) between the dewar assembly (**2**) and the diamond/tungsten carbide puck (**1**).

Note: *In **any** experiment it is advisable to take a reference background Spectrum (or spectra) at the same **actual** temperature conditions when collecting a spectrum (or spectra) for the sample. This is to obtain the optimum spectral results when the background spectrum is subtracted from the sample spectrum.*

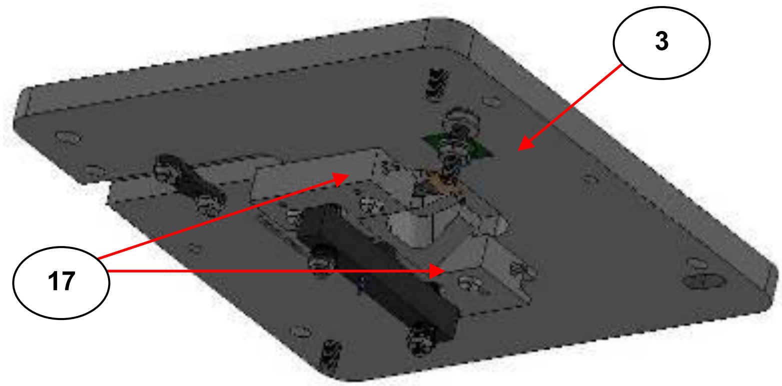


Fig 8. Low Temperature Golden Gate Heated Top Plate Assembly underside view

If it is a requirement to obtain spectra for a sample at two different physical states, solid and liquid, depending upon the freezing point of a liquid sample it may be preferable to use the graphite high thermal transfer gasket (**5**) in position. This gasket material will permit operation down to the minimum temperature obtainable with this accessory to -150ºC. By using this high thermal transfer gasket (**5**) the sample can be quickly frozen using only a small amount of LN2 refrigerant, allowing it to boil off as soon as the desired temperature is reached. This will minimize condensation and icing on the dewar (**2**).

Procedure Using a High Thermal Transfer Gasket

To ensure freezing of the LN2 refrigerants lowest temperature capability (circa -150°C with a graphite high thermal transfer gasket (**5**) being used), the block heaters (**17**) must be switched off by selecting a lower **set** temperature on the controller (e.g. at least 10°C less than the value showing for any **actual** temperature from the LN2 refrigerants cooling action). Therefore, a suggested **set** temperature at the start of experimentation is -150°C on the controller, as the accessory overall will never get colder than this temperature value.

Reference Spectrum Collection

For collection of a background reference spectrum, at room temperature conditions, establish the nitrogen purge gas conditions in the optical unit (**11**) and within the internal sample chamber area (**15**) from connection of the purge gas via a suitable flow pipe or tubing through one of the dewar cap purge gas holes (**16**), before any refrigerant is added.

Introduce some LN2 into the dewar chamber **(6**) using the funnel (**7**) through the funnel hole (**8**) or crescent shaped cut out (**9**) in the dewar cap (**10**). Initially, there will be some fuming of the LN2 as it boils off when contacting with a warmer environment, but by adding small amounts of LN2, after time the **actual** temperature on the controller display will begin to drop fairly, quickly indicating the effect of the LN2 refrigerant action to the sample area for temperature measurement.

When the **actual** temperature has dropped to a value that is to be used for study, stop adding any further LN2 refrigerant. If only small amounts of LN2 have been added, once the remaining residual LN2 has boiled off from the dewar chamber (**6**), and because the heaters (**17**) will still be inactive as the **actual** temperature will be higher than the **set** temperature (-150°C), the accessory will start to warm up naturally from the general ambient surroundings. At this stage when the temperature value becomes relatively stable, a background ATR spectrum can be collected and stored for use against an ATR spectrum taken for a sample at the same temperature and conditions of use.

If the refrigerant effect of the LN2 in combination with a high thermal transfer graphite gasket (**5**) is causing the temperature to be too low in value, (i.e. towards the minimum -150°C value), then the heaters (**17**) may need to be activated to balance the cooling effect and raise the temperature to the value desired. Therefore, if say a temperature of -100°C is required, change the **set** temperature value to -100°C on the controller. If the **actual** temperature is indicating a temperature value lower than -100°C, the heaters (**17**) will activate (the heater light pulses on the controller display) and the temperature will rise.

Note: *It is important for operation at very low temperatures to ensure the LN2 refrigerant level is kept topped up to provide the "balancing effect" against the power from the heaters (**15**).*

Sample Spectrum Collection

When a reference spectrum at a particular low temperature value has been collected the procedure of operation needs to be repeated, but with some subtle changes.

To place a sample into position on the diamond crystal (**1**) after collection of a reference spectrum, the Low Temperature Golden Gate ATR accessory must be allowed to warm up to ambient conditions. When the accessory is near to, or has reached room temperature, dry off any external condensation using tissues. It may be easier to dry the unit thoroughly by disconnecting the 4000 Series Temperature Controller and removing the Low Temperature Golden Gate accessory from the spectrometer and to place it on a workbench.

When cleaned, dried, and re-installed in the spectrometer, at room temperature conditions establish the nitrogen purge gas conditions in the optical unit (**11**) and within the internal sample chamber area (**15**), before any refrigerant is added, a liquid sample is placed in position to cover the diamond of the diamond/tungsten carbide puck (**1**). Included with the accessory is a liquid sample injector consisting of an insulated 16 gauge hypodermic needle and glass syringe that can be used for delivery of a liquid sample at the base of the internal chamber area (**16**) and over the diamond (**1**).

When the liquid sample is in place, re-position the dewar cap (**10**) and you may then wish to re-establish a nitrogen purge flow to the internal chamber (**15**) by use of a nitrogen flow tube through one of the dewar cap holes (**16**). It depends upon the stability of the liquid sample to be analysed (its relative volatility), if this nitrogen purge over the sample may be necessary.

Introduce some LN2 into the dewar chamber **(6**) using the funnel (**7**) through the funnel hole (**8**) or crescent shaped cut out (**9**). Initially, there will be some fuming of the LN2 as it boils off when contacting with a warmer environment, but by adding small amounts of LN2, after time the **actual** temperature on the controller display will begin to drop fairly quickly indicating the effect of the LN2 refrigerant action to the sample area for temperature measurement.

When the **actual** temperature has dropped to a value that has allowed the liquid sample to freeze and solidify, do not add any further LN2 refrigerant. If only small amounts of LN2 have been added, once the remaining residual LN2 has boiled off from the dewar chamber (**6**), and because the heaters (**17**) will still be inactive if the **actual** temperature is higher than the **set** temperature, the accessory will start to warm up naturally from the general ambient surroundings.

The **actual** sample temperature can be monitored from the controller readout as the temperature value rises toward the ambient (room temperature) conditions. ATR spectra can be taken at lower temperature values for a solidified state of the sample and when it has reverted to its liquid state at a higher temperature.

Note: *The above general operation procedure applies equally well to solid samples in place of a liquid sample as has been described.*

Other Examples of Operation

1) Use of Liquid Nitrogen to −80°C

To hold a sample at steady low temperature (down to −80ºC) it is necessary to reduce the rapid cooling effect of the liquid nitrogen (LN2) by using a low thermal transfer gasket (**5**) between the dewar assembly (**2**) and the diamond/tungsten carbide disc (**1**). This gasket (**5**) is the white, glass-filled PTFE, washer shaped disc, supplied and fitted as standard. (See Note page 10.) This gasket allows for a longer lifetime of refrigerant to be maintained in the dewar chamber (**6**) (once the dewar chamber has reached an equilibrium temperature), and to establish a relatively slow temperature fall of the sample. The low thermal transfer capability of this type of gasket also allows for a more controllable and precise way to slowly raise or lower the temperature.

Install the accessory in the Spectrometer, align the optics according to the Golden Gate instruction manual (GS10500) and connect the 4000 Series Temperature Controller. Establish the nitrogen purge to both the optical unit (**11**) and the internal chamber area (**15**) via the dewar cap (**10**).

To ensure that there is a valid background reference at all temperatures of interest, it is useful to store a series of backgrounds at the temperatures at which it will be used. By doing this, it will not be necessary to clean and dry the dewar/diamond interface during any experiments. The easiest way to do this is to input a **set** temperature on the 4000 Series Controller just below the temperature value to be measured and pour small aliquots of liquid nitrogen into the outer part of the dewar (**6**) using the plastic funnel (**7**) supplied, until this temperature is achieved.

Note: *Never fill the dewar chamber (**6**) completely. In general, the level of refrigerant within the dewar chamber should be sufficient to cover the internal foam insulating sleeve within the dewar chamber.*

After reaching the lowest temperature required to measure for a background reference spectrum, allow the Low Temperature Golden Gate accessory to warm up to the next temperature to measure. Change the **set** temperature on the controller to a level just below that needed. Save reference background spectra as the correct temperatures are reached whilst, overall, the temperature is rising back to ambient conditions.

After the reference spectrum has been collected, a liquid or solid sample analysis can be carried out as follows.

Liquid Samples

1. Set a temperature on the 4000 Series Temperature Controller to be a few degrees below the temperature required. Carefully introduce just one or two drops of the liquid sample onto the

diamond (**1**) using the special syringe and hypodermic needle supplied.

2. Cool the dewar (**6**) by adding small amounts of liquid nitrogen from a small vacuum flask using the funnel (**7**) provided to ensure that the liquid nitrogen is directed to the bottom of the outer part of the dewar and does not splash into the inner part of the dewar (**15**). The funnel is inserted into the round hole (**8**) in the dewar cap (**10**) and the level of the liquid nitrogen can be viewed through the crescent cut out section (**9**). Do not fill the level higher than the foam packing sleeve within the outer dewar section (**6**). Monitor the temperature to see that it does not drop too fast. Never fill the dewar (**6**) completely.

3. When the desired temperature level has been set and the sample is still in a liquid, or has changed into a solid state, it is possible to scan it and reference it against the appropriate stored background.

Note: *During the freezing process, the sample may contract and shrink away from the diamond (**1**). To re-establish correct contact the long sapphire anvil (**18**) can be used to apply a small amount of pressure to the frozen sample. The long sapphire anvil (**18**) is put down into the centre part of the dewar (**15**) through the hole (**19**) in the dewar cap (**10**) and the bridge assembly (**20**) of the Low Temperature Golden Gate top plate is closed and locked. A flat anvil (**21**) is fixed to the clamp part (**22**) of the bridge assembly (**20**) and is used to transfer the pressure to the long sapphire anvil (**18**). The torque screw (**23**) is turned clockwise to apply sufficient pressure to the solidified sample. (See Figs 9 and 10, for explanation of these parts.)*

If the frozen sample being analysed is water based, it is possible that too much pressure from the long sapphire anvil (**18**) may cause it to revert to the liquid state. Removing some pressure from the long sapphire anvil (**18**), (turn torque screw (**23**) anticlockwise) should give correct sample contact in readiness for collection of an Infrared ATR spectrum.

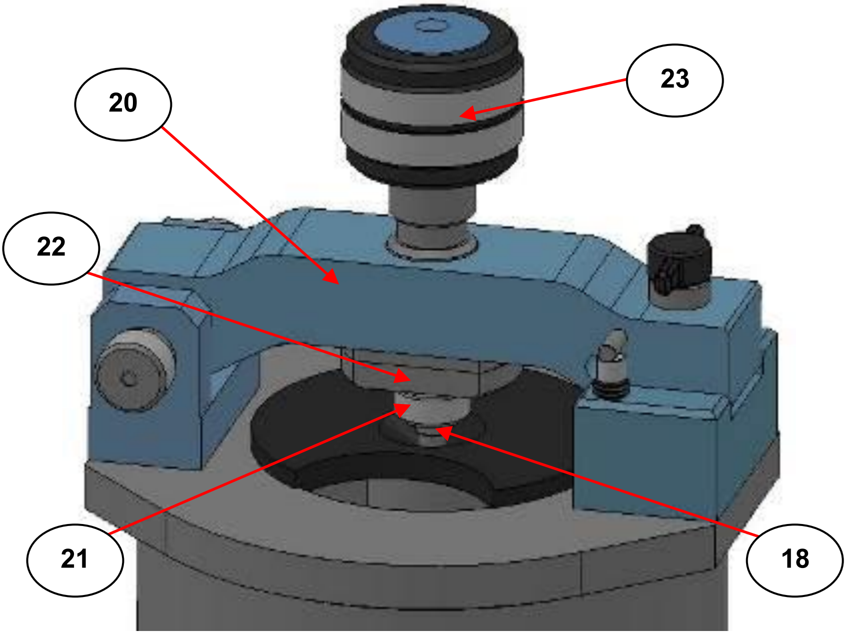


Fig 9. Low Temperature Golden Gate Bridge Assembly with Dewar Cap, Flat Anvil and Sapphire Rod in position

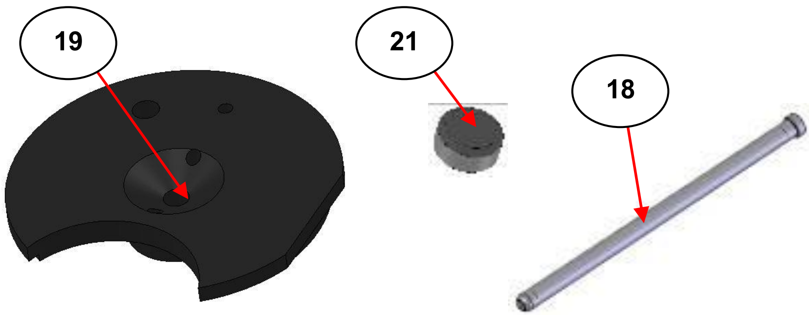


Fig 10. Low Temperature Golden Gate Flat Anvil, Sapphire Rod and Dewar Cap Parts.

4. By allowing the system to slowly warm up, without the addition of any more liquid nitrogen, the solid/liquid phase spectral scan can be repeated as the appropriate **actual** temperature is seen on the 4000 Series Temperature Controller display.

5. After completion of the experiment, use the syringe and hypodermic needle to remove as much sample as possible. Set the 4000 Series Temperature Controller to warm up the top plate to 30 to 40°C. When this temperature has been reached set the 4000 Series Temperature Controller back to room temperature to allow the top plate to cool down.

6. When the accessory is at or near room temperature, dry off any external condensation with tissues. Disconnect the 4000 Series Temperature Controller, remove the accessory from the spectrometer and take it to a workbench.

7. Using the long T bar type Allen Key 5mm loosen the four captive clamp screws (**4**) and remove the complete dewar assembly (**2**) from the Golden Gate heated diamond ATR top plate (**3**). It is now possible to dry off any remaining sample and clean the diamond surface (**1**). The heat transfer gasket (**5**) is situated between the diamond plate (**1**) and the centre part of the dewar (**15**). When the dewar (**2**) is removed this gasket may be found attached to either surface (dewar or diamond plate). The glass filled PTFE type gasket (white) should be in good condition and will be usable for many experiments. Remove it, clean and dry it. The high thermal transfer type graphite gasket (dark grey colour) is more fragile and may be firmly attached to one of its contact surfaces. If it cannot be cleanly peeled off, it will have to be carefully removed with a sharp blade and then discarded. (Be careful not to scratch the dewar contact surface (**2**) with the blade). If the gasket is in good condition it may be cleaned and used again. The gasket will need to be replaced before the next experiment.

   Note: *When replacing the dewar (**2**) make sure the four captive clamp screws (**4**) are tightened evenly and firmly.*

Solid Samples

The ideal solid sample shape for the Low Temperature Golden Gate diamond ATR top-plate is a 5mm diameter disc. The solid sample is dropped down into the centre part of the dewar (**15**) such that it lies correctly on the diamond (**1**). Small pellets (less than 6mm in diameter) and powders can also be analysed by similarly dropping into the centre part of the dewar (**15**) to cover the diamond (**1**).

The long sapphire anvil (**18**) is put into position in the centre of the dewar (**2**) to contact with the top of the sample and can be pressurized from the Golden Gate bridge torque assembly (**23**) to give good sample contact at the desired, selected temperature. The torque screw (**23**) clamp knob is preset to apply a load of circa 80lbs on the sample. When the long sapphire anvil (**18**) is applying this load to the sample and the **set** temperature has been reached you are ready to collect spectral scans of the sample. Similar to the procedure for liquid samples, it is useful to take spectra during cool-down and warm-up to give a double set of results through the possible phase changes.

To remove the solid sample from the diamond/tungsten carbide disc (**1**) and for subsequent cleaning, the complete dewar assembly (**2**) must be removed. The four captive clamp screws (4) are loosened by use of the T bar type Allen Key 5mm. On removal, check to see if the heat transfer gasket (**5**) needs replacing before the next experiment.

General Note: *In a normal laboratory atmosphere, when the accessory is used at very low temperatures, outer parts of the accessory will have moisture or frost on them. Keeping experiments as short as possible will minimize this effect. Always set the temperature to warm up to 30 or 40°C after an experiment and make sure the accessory is clean and dry before storage. Keeping the accessory in a warming cabinet after use is recommended.*

2) Use of Cardice (dry CO2) and Acetone as Refrigerant at -30°C

For operation between temperatures of −30°C through to circa 20°C, cardice (dry C02) and acetone as a refrigerant can be used with a white glass filled PTFE gasket (**5**) in position.

To assist in potential fine control and stability between temperature states over this temperature range it is necessary to change the block heaters (**17**) power by changing the OpuL parameter setting on the 4000 Series temperature controller. (Please see parameter listing in Section 6).

In addition to a change in the heater power setting, the "proportional" (Pb-P), "integral" (ArSt) and "derivative" (rAtE) parameters will need to be adjusted to allow for a smooth delivery of the power such that the change in temperature is gradual, controlled and does not "overshoot" or "undershoot" to the temperature value being set. Please consult the instruction manual supplied for the 4000 Series Temperature Controller to explain how to access the factory set parameter list values for the Low Temperature Golden Gate accessory and how these values/settings can be changed.

For this specific type of experiment using the cardice/acetone refrigerant with the white PTFE low thermal transfer gasket (**5**) over the temperature range from -30°C to 20°C at 5°C steps for temperature measurement, the control parameters are set as follows:

Parameter OpuL (heater power) is set to 35. (35% power).
Parameter Pb-P (proportional) is set to 10.0.
Parameter ArSt (integral) is set to 4.0.
Parameter rAtE (derivative) is set to 0.04.

Note: *The above parameters are changed and used for control with the setpoint ramp rate value parameter rP set to **OFF.***

With practice and experience for different sample types, refrigerant mixtures and temperature range to study, these general parameters and their settings may need to be varied to suit for control accordingly.

6. Operating Parameters for the Low Temperature Golden Gate ATR Top Plate

The Low Temperature Golden Gate ATR system is provided with its own dedicated 4000 series 150W temperature controller. A separate manual is supplied with the controller. For operation of the Low temperature Golden Gate ATR heated top plate the parameters of the 4000 Series temperature Controller have been factory set as shown on the following page. Not all the displayable parameters can be changed but have been listed for reference purposes. If you ever need to change a parameter or autotune the controller for a specific temperature range, certain parameter settings will be altered. You can get back to original factory settings by reprogramming the controller with these original values.

Temperature Controller:

Supply ratings:

Voltage	230V	110V	100V
Frequency	50HZ	60HZ	50/60HZ
Max Power	150W	150W	150W
Fuse Rating	1.6AH	3.15AH	3.15AH
Fuse Type	T	T	T

Refer to the Specac 4000 Series controller manual for further details

Rated operating & Storage Temperatures.
Normal: 5 to 40°C (0% to 80% relative humidity and noncondensing)
Storage: -20 to 55°C (0% to 95% relative humidity and noncondensing)

Altitude: Up to 2000m
Pollution degree: 2

Heated top plate: 27VDC, 3A, 150W

Item weight: 4.6Kg
Dimensions: 142mm (Depth), 155mm (wide), 320mm (high).

Displayable Parameters For Low Temperature Golden Gate ATR Top-plate 10590 with WEST 6100+ (4000 Series™) Controllers

Parameter Display (In Green)	Parameter Name	Parameter Factory Set Value
FiLt	Input Filter Time Constant	3.0
OFFS	Process Variable Offset	0
PP˩˩	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	0.5
ArSt	Automatic Reset (Integral Time Constant)	0.34
rAtE	Rate (Derivative Time Constant)	0.08
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	80
SPLL	Setpoint Lower Limit	-150
OPuL	Primary (Heat) Output Upper Power Limit	100
Ct I	Output 1 Cycle Time	4
PhAl	Process High Alarm	80
AHyl	Alarm 1 Hysteresis	1
PLA2	Process Low Alarm	-150
AHy2	Alarm 2 Hysteresis	1
APt	Auto Pre-Tune enable/disable	diSA
PoEn	Manual Control select enable/disable	diSA
SPr	Setpoint Ramping enable/disable	EnAb
rP	Setpoint Ramp Rate Value	1800
SP	SP Value	0
SLoc	Set-up Lock Code	10

7. Legend - Part Identification of Low Temperature Golden Gate Top Plate

1. Diamond/tungsten carbide support disc.
2. Cooling jacket dewar assembly.
3. Heated diamond ATR top plate assembly.
4. Dewar assembly clamp screw.
5. Heat transfer gasket.
6. Dewar chamber (contains refrigerant).
7. Plastic funnel.
8. Dewar cap funnel hole.
9. “Crescent” area cut out in dewar cap.
10. Dewar cap.
11. Golden Gate optical unit.
12. Heated diamond ATR top plate fixing screw.
13. Golden Gate optical unit aperture port.
14. Purge port hose connections.
15. Central internal area of dewar assembly.
16. Dewar cap purge gas flow hole.
17. Heated diamond ATR top plate block heater.
18. Long sapphire anvil rod.
19. Dewar cap long sapphire anvil rod hole.
20. Low Temperature Golden Gate top plate bridge assembly.
21. Low Temperature Golden Gate flat anvil.
22. Clamp part fixing for flat anvil.
23. Torque knob screw assembly.

8. Spare Parts for Low Temperature Golden Gate Top Plate

P/N GS10593 Glass filled PTFE gaskets (white colour) (Pkt of 5)
P/N GS10594 High thermal transfer graphite gaskets (grey colour) (Pkt of 20)
P/N GS10595 Liquid sample injection device.

Notes

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