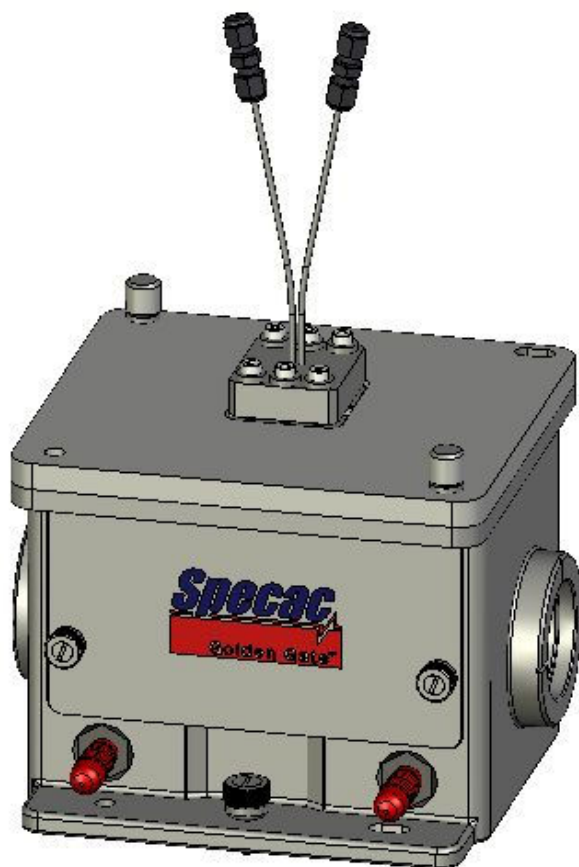


# Supercritical Fluid Golden Gate™ ATR

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## *User Manual*



2I-10586-14



Supercritical Fluid  
Golden Gate™ ATR

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User Manual

# *Supercritical Fluid Golden Gate™ ATR*

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## **SUPERCRITICAL FLUID GOLDEN GATE™ ATR**

### ***USER MANUAL***

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## 1. Introduction

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Thank you for purchasing a Specac Product.

The Supercritical Fluid Golden Gate™ ATR top-plate is used on the Golden Gate™ ATR optical unit to study samples at supercritical fluid temperature and pressure conditions.

The analysis area of the top-plate is an ATR-IR diamond element giving one reflection at an angle of 45°. This high pressure, high temperature top-plate is mounted on the Golden Gate™ optical system which provides a high throughput specification for the diamond ATR crystal and sample interface.

IR spectra of supercritical fluids (SCF's), high pressure gases, and liquids, as well as materials subjected to SCF's, high pressure gases, and liquids, can be measured. The spectral measurements can be obtained from the gas phase through to the supercritical region and to liquid like densities.

This manual provides instruction for use of the Supercritical Fluid (SCF) top-plate itself on the Golden Gate™ ATR optical unit. The instruction manual for the standard Golden Gate™ accessory (GS10500) should be consulted for any other general procedures relating to operation of the Golden Gate™ accessory, for example, installation, mirror and lens alignment and top-plate removal.

Power to the heaters used within the SCF top-plate, is supplied by a dedicated 4000 Series™ temperature controller. A separate manual is provided for operation of the temperature controller with the Supercritical Fluid Golden Gate™ ATR top plate.

## 2. Technical Specifications

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- Maximum high pressure up to 6000 p.s.i.
- Maximum controlled temperature to 300°C.
- Low volume cell (28 $\mu$ l). (The cell volume can be changed to larger volumes as a special, if required. Please contact Specac.)
- Standard 1/16" inlet/outlet fittings for gas/liquid flow.



### **EC Pressure Directive 97/23/EC Rules for use of the SCF Golden Gate™ ATR Accessory**

The above general maximum pressure specification rating of 6000psi is for the study of liquids/gases within this equipment.

EC Pressure Directive 97/23/EC defines group 1 gases as, explosive; extremely flammable; highly inflammable; flammable (where the maximum allowable temperature is above the flashpoint); very toxic; toxic; oxidizing.

If group 1 gases are to be used in the SCF Golden Gate™ ATR accessory, then the maximum operating pressure is reduced to 2900psi (200bars) from 3000psi. Therefore, because the pressure is less than 2900psi and the pressure chamber volume is less than 1 liter, the product falls within Sound Engineering Practice (SEP).

Similarly, for liquids and group 2 gases, because the pressure is less than 7250psi (500bars) and the pressure chamber volume is less than 1 liter, the product falls within Sound Engineering Practice (SEP).

### *3. Packing and Checklist*

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On receipt of your Supercritical Fluid Golden Gate™ ATR accessory please check that the following have been provided:

- Supercritical Fluid Golden Gate™ ATR top plate (10585).
- Golden Gate™ Optical unit with choice of ZnSe or KRS-5 lenses and appropriate Benchmark™ baseplate (if ordered as 10586).
- 4000 Series™ Temperature Controller, instruction manual, and power cables.
- Packet of compressible graphite gaskets (10).
- Torque wrench and bit (3.0 mm A/F hexagonal head).
- Allen key, 2.5 mm A/F (long handle).
- Open ended spanner (1/4" to 5/16"). (2 off)
- Hexagon head ball driver
- Allen key 2.0 mm A/F (long handle)

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

## 4. Safety

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**Safety Note:** *High temperatures and pressures are used in operation of the accessory.*

The Supercritical Fluid (SCF) Golden Gate™ ATR top plate is designed to operate at high pressures and temperatures in order to create the conditions for supercritical fluids. The high pressures and temperatures are safely contained within the analysis area of the accessory, but certain precautions must be observed when operating this equipment. **(See Pressure Directive Rules Section 2, page 4.)**

Ensure that any gas/liquid flow connections are tight and do not leak.

It is advisable to operate the accessory in a spectrometer with the sample compartment lid of the spectrometer closed (if possible). If there was to be any immediate loss of high pressure in the system, it would initially be contained within the sample compartment area and the risk of an explosion directly into a laboratory is minimized. If the sample compartment lid of the spectrometer has to be kept open in operation, you should always wear protective safety spectacles whenever in the vicinity of the accessory.

If the sample compartment volume is small (less than 200mm x 200mm x 200mm), then it is possible the sample compartment lid must be kept open to prevent activation of the safety thermal cut out switches as the local environment gets hot. (When the SCF Golden Gate™ top-plate is operating in excess of 100°C). Opening of the lid allows for a flow of air to the local environment to keep the unit from overheating. If the sample compartment lid must be kept closed for safety reasons, to minimize a build up of heat in the sample compartment, Specac recommend that a flow of N2 gas through the sample compartment as a purge should be established.

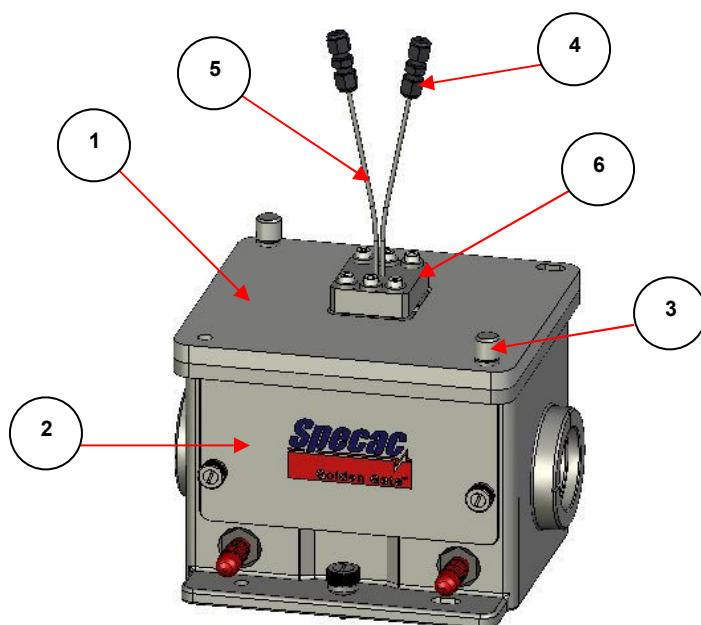
**Warning:** *Specac cannot be held responsible for any event due to misuse of the accessory from over pressurization to the system and its components.*



## 5. Installation

The SCF Golden Gate™ ATR top-plate (1) is fixed to the Golden Gate™ optical unit (2) with the two thumb screws (3). The top plate will only fit to the optical unit in one orientation, when the circular hole and slot on the top plate are in alignment with their locating pins on the optical unit.

**Note:** When fitting the top plate be careful to avoid touching the lenses in the optical unit with the underside of the heating block of the top plate.



**Fig 1 – Front View Of SCF Golden Gate™ ATR Accessory**

The complete SCF Golden Gate Accessory is then mounted on an appropriate Benchmark™ baseplate within the sample compartment of

a Spectrometer. (See installation procedure of the Golden Gate™ optical unit for your Spectrometer from the Golden Gate™ manual 10500).

When installed, make the necessary power connection from the SCF top-plate to the 4000 Series™ temperature controller. (See instructions in the 4000 Series™ temperature controller manual). Selection of the temperature required and operation of the controller is also found within the temperature controller manual.

The high pressure connection (for the sampling fluids) is a 1/16" Swagelok™ fitting (4), connected to stainless steel flow tubes (5), allowing the SCF cell pressure chamber (6) to be coupled to a suitable high pressure pump capable of generating pressures of 5000 to 6000 p.s.i.

**Safety Note:** *It is an important safety consideration (because of the high pressures involved in the use of this accessory) that the compressed volume of fluid is kept to a minimum. Therefore, the pump should be located as close to the SCF cell as possible.*

Continuous monitoring of the pressure within the cell is essential and so the pressurizing system must include a suitable pressure gauge. Because of the low operating volume of the system, pressure can build up very quickly.

## 6. Operation

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When installed into the spectrometer the necessary temperature and pressure conditions can be applied to the SCF top-plate. Apply a temperature, set from the 4000 Series™ temperature controller, and a pressure as specified from your pump/pressurizing system.

**Warning:** *When the SCF top-plate is used for prolonged periods, such as for 1 hour at maximum temperature (300°C), the stainless steel top plate will get hot. When set at 300°C, the outer edges of the top plate may exceed 75°C with the sections of the plate closer to the analysis area being even hotter. Take care not to touch the top plate when the accessory has been used at 300°C for more than 20 minutes.*

After use, allow the accessory to cool to 40°C or below, and the pressure to drop to near atmospheric pressure before flushing through with a cleaning solvent. The accessory should then be dried out completely, if possible, by purging through with dry nitrogen gas.

### Use of Gasket

A graphite gasket (7) is used to seal between the pressure chamber (6) face and the diamond /tungsten carbide puck (8). It is **most important** that a new graphite gasket is used on every occasion that the pressure chamber (6) is removed from the diamond/tungsten carbide puck. During experimentation, pressure and temperature effects will cause the gasket to stick to the clamping surfaces of the pressure chamber and diamond puck. Therefore, the graphite gasket will need replacing each time the top section pressure chamber (6) is removed.

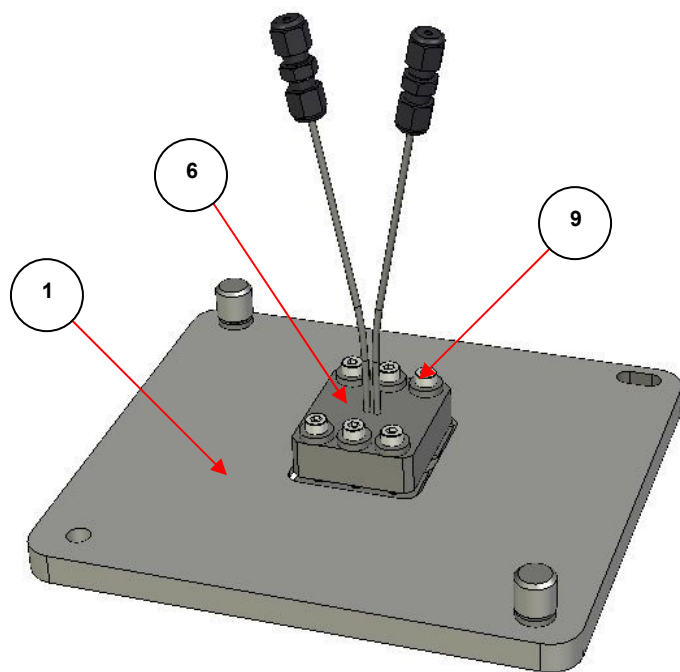
**Note:** *If leaks develop (i.e. pressure cannot be maintained in the system) then the gasket **must** be replaced.*

### Replacing the Gasket

The high pressure, high temperature capability of this accessory depends on the effective use of a compressible graphite material for

the gasket. This material must be compressed evenly to achieve maximum performance. A torque wrench tool is supplied with the SCF Golden Gate™ top-plate to help achieve an even compression to the gasket when it has been changed within the top plate cell chamber. As supplied, the SCF Golden Gate™ top-plate is already fitted with a compressed graphite gasket that has been pressure checked to 6000psi.

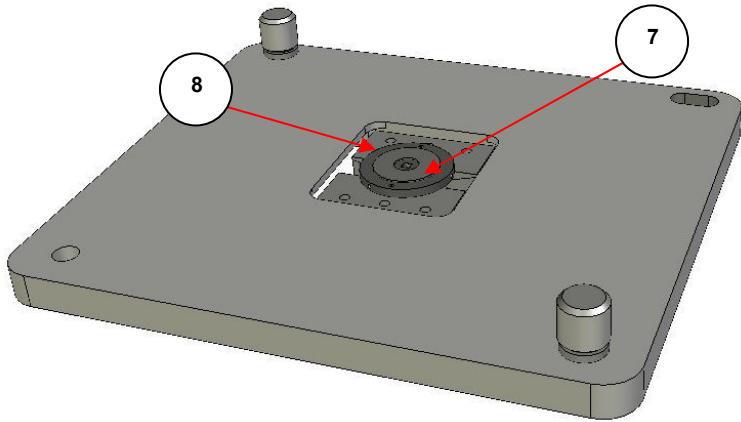
### **Removal of the Gasket**



**Fig 2 – Front View of SCF Golden Gate™ Top-Plate Assembly**

The pressure chamber (6) must be removed from its position to gain access to the gasket (7).

Remove the six clamping screws (M4 x 20mm cap head High Tensile Plated) and special load spreading washers (9) which hold the high pressure chamber (6) in position via the threaded holes of the heating block (11).



**Fig 3 – SCF Golden Gate™ Top-Plate With Pressure Chamber Removed Showing Graphite Gasket Centrally On The Diamond Puck**

When removing the M4 clamping screws it is important that the load is reduced gradually and evenly. **Do not** remove one screw completely at a time as this could result in an uneven load being applied to the diamond/tungsten carbide puck (8). A 3mm A/F Allen key or the torque wrench supplied, with the appropriate Allen key bit can be used to undo the screws (9). If using the supplied torque wrench, select a torque setting greater than 2.5Nm to allow for the M4 screws to be undone. (The M4 screws will have been tightened previously to their required **maximum** 2.5Nm torque setting.) The recommended sequence is to partly slacken a screw at one corner and then do the same to the screw at the opposite corner. Now carry out the same procedure to the other pair of corner screws.

Finally, partly slacken the middle pair of screws until all 6 screws have been loosened. Now, proceed to remove all 6 screws and washers to remove the pressure head assembly and gain access to the gasket (7).

A high clamping force is needed for sealing of the graphite gasket (7) (see Fig 3) between the pressure chamber (6) and diamond/tungsten carbide puck (8). If the temperature has exceeded 60°C during operation, it is possible that the top section pressure chamber (6) surface will be bonded to the graphite gasket (7). In turn the graphite gasket (7) may also be bonded to the diamond/tungsten carbide puck (8).

If the graphite gasket (7) has bonded to both the pressure chamber (6) and diamond puck (8) faces, the diamond puck will be lifted clear of its seating from its two small pin locations (10) on the heating block (11) by removal of the pressure chamber (6). It is therefore **very important**, when the screws (9) have been undone that the action for removal of the pressure chamber (6) is in an upward motion with no twisting to the assembly. Too much of a twisting force might cause damage to the two location pins (10) and it will not be possible to reseat the diamond puck (8) correctly for the next use. (See Fig 4).

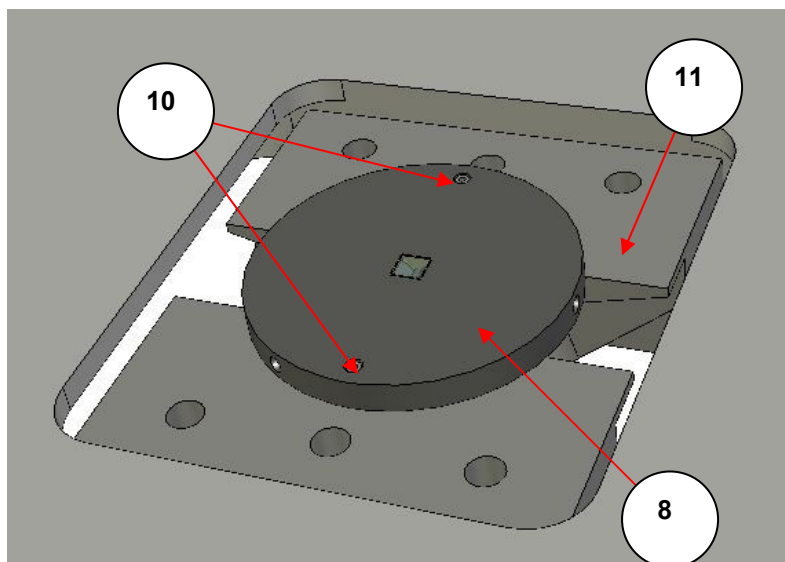


Fig 4 – Close Up View Of Diamond/Tungsten Carbide Puck In SCF Golden Gate™ ATR Top-Plate.

If the pressure chamber (6) and diamond puck (8) have become “fused” together by the gasket (7), the parts can be separated by use of a sharp edged piece of plastic. Insert an edge of the plastic between the chamber and diamond puck at the gap created by the gasket and gently manipulate to prise the parts away from each other.

**Note:** *Specac recommend use of plastic and **not** metal for a separating/ scraping tool as metal may damage the sealing surface.*

Any remains of the used gasket (7) may be scraped off the sealing surfaces of the pressure chamber and diamond puck using the sharp edged plastic tool or sharp edged plastic. As noted previously, **DO NOT** use a metal scraper as this may damage the sealing surfaces.

Clean the last traces off the gasket away with a solvent moistened cloth. (A suitable solvent to use is acetone or methanol).

### **Removal of the Diamond/Tungsten Carbide Puck**

From the design of the SCF Golden Gate™ Top-Plate, the diamond puck (8) is held into position onto the heating block (11) by two location pins (10). (See Fig 4.) There are two corresponding drilled holes in the underside of the diamond puck that align with the location pins.

When the pressure chamber (6) is clamped into position with a gasket (7) in place over the puck (8), the diamond puck is secured in position for operation up to its specified temperature of 300°C.

It is **very important** for repositioning of the puck (8) again onto the heating block (11) during re-assembly (rebuild), that all surfaces are scrupulously clean and free from any imperfections of grit/dirt etc. The puck (8) **must** sit perfectly flat against the heating block support (11) without any “rocking” motion.

**Note:** *Specac cannot be held responsible for any damage or breakage to the diamond puck if it has been repositioned incorrectly after cleaning and changing the graphite gasket.*

## Replacement of the Gasket

To rebuild the pressure chamber assembly, the diamond puck (8) must be repositioned correctly on the heating block (11).

Take a new graphite gasket (7) and carefully position it centrally over the exposed diamond surface of the diamond/tungsten carbide puck (8). (See as Fig 3.) Take the top section pressure chamber (6) and place it carefully over the gasket (7) such that the six screw holes are in alignment with their corresponding screw hole in the stainless steel top plate (1). Using a normal Allen key (supplied), first screw all six clamping screws with their load spreading washers (9) down until the cap heads of the screws are just clear of the top section pressure chamber (6), without applying any clamping force at this time.

Very carefully apply the clamping force to the screws (9) as evenly as possible, tightening the central pair and the diagonally opposite pairs in sequence, until all six screws are just applying some pressure. Now, take the torque wrench set at its lowest value and tighten the clamping screws (9). The clamping pressure must be applied as evenly as possible. Tighten the centre pair of screws (9) just a little, followed by the diagonally opposite pairs in sequence, such that a small amount of pressure is being applied. Continue in the same pattern sequence, using the torque wrench set in increasing torque settings, until the **maximum** torque of 2.5 Nm is achieved.

**Note:** *It is important that a load bearing washer is used with each of the clamping screws (6) to correctly spread the internal force that builds up within the pressure chamber area of the Supercritical Fluid Golden Gate™ and assist in overall sealing. It is also of great importance that the only clamping screws (6) to be used are M4 x 20mm cap head High Tensile Plated screws as provided by Specac, to ensure safe operation for sealing of the accessory pressure head.*

If possible, check the gasket (7) seal for leaks using a solvent, before starting a new experiment.



## *7. Operating Parameters and Specifications*

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The Supercritical Fluid Golden Gate™ ATR Top-Plate is provided with its own dedicated 4000 Series™ Temperature Controller. A separate manual is supplied for operation of the 4000 Series™ Temperature Controller.

For operation of the Supercritical Fluid Golden Gate™ ATR Top-Plate the parameters of the 4000 Series™ Temperature Controller have been factory set as shown on the following page. Not all of 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 particular temperature range certain parameter settings will be altered. You can get back to original factory settings by reprogramming the controller with these original values.

### **Specifications**

Accessory Type GS10585 or GS10586

|             |      |      |         |
|-------------|------|------|---------|
| Voltage     | 230V | 110V | 100V    |
| Frequency   | 50HZ | 60HZ | 50/60HZ |
| Max Power   | 150W | 150W | 150W    |
| Fuse Rating | 1.5A | 3A   | 3A      |
| Fuse Type   | T    | T    | T       |

Insulation rating of external circuits (appropriate for single fault condition) = basic insulation and protective (earth) bonding.

Humidity operation range – 20% to 90% relative humidity non-condensing.

### Displayable Parameters For SCF Golden Gate™ GS10586 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 <sub>LL</sub>             | Primary (Heat) Output Power              | 0                           |
| Pb_P                         | Primary Output Proportional Band         | 3.2                         |
| ArSt                         | Automatic Reset (Integral Time Constant) | 1.06                        |
| rAtE                         | Rate (Derivative Time Constant)          | 0.16                        |
| biAS                         | Manual Reset (Bias)                      | 25                          |
| SPuL                         | Setpoint Upper Limit                     | 300                         |
| SPLl                         | Setpoint Lower Limit                     | 0                           |
| OPuL                         | Primary (Heat) Output Upper Power Limit  | 100                         |
| Ct l                         | Output 1 Cycle Time                      | 16                          |
| PhAl                         | Process High Alarm                       | 300                         |
| AHyl                         | Alarm 1 Hysteresis                       | 1                           |
| PLA2                         | Process Low Alarm                        | 0                           |
| 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                                 | 1                           |
| SLoc                         | Set-up Lock Code                         | 10                          |

## 8. Legend

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Below is the list of descriptions that correspond to the parts identified by bubble numbers in the Figures 1 to 4.

- (1) SCF Golden Gate™ ATR top-plate.
- (2) Golden Gate™ optical unit.
- (3) Top-plate fixing thumb screws.
- (4) High pressure 1/16" Swagelok fitting.
- (5) Stainless steel flow tubes 1/16" O.D.
- (6) SCF Golden Gate™ pressure chamber.
- (7) Graphite gasket.
- (8) Diamond/tungsten carbide puck.
- (9) M4 x 20mm clamping screw and load washer.
- (10) Location pin for diamond puck.
- (11) SCG Golden Gate™ heating block.

## 9. Spare Parts

---

|                |                                                  |
|----------------|--------------------------------------------------|
| <b>GS10587</b> | Graphite Gaskets (Pkt of 10).                    |
| <b>GS10577</b> | 3.0 mm A/F hexagonal head bit for Torque Wrench. |
| <b>GS10589</b> | Torque Wrench for SCF Golden Gate™.              |

## Notes

[illegible]

This is to certify that the:

**GOLDEN GATE SUPER CRITICAL & 4000 Series TEMPERATURE CONTROLLER  
10586**

Manufactured by:  
**SPECAC LIMITED**

Conforms with the protection requirements of Council directives 2004/108/EC , relating to the  
EMC DIRECTIVE,

by the application of:

- 1) Testing to the following standard:  
EN-61326:2006/8 EMC (Emissions/Immunity) requirements for Electrical Equipment  
for measurement, control and laboratory use.
- 2) Supported by SPECAC Technical File No. **TF10586**

and also conforms to the general safety requirements of Council Directives 2006/95/EC , relating to  
the LOW VOLTAGE DIRECTIVE,

by the application of:

- 1) EN61010-1:2010, Safety Requirements for Electrical Equipment for  
Measurement, Control and Laboratory use.
- 2) Supported by SPECAC Technical File No. **TF10586**

Conforms with the protection requirements of Council directives 97/23/EC , relating to the  
PRESSURE DIRECTIVE,

by the application of:

- 1) The Pressure Equipment Regulations 1999 amendment 2002.  
The Pressure Systems Safety Regulations 2000.  
The Simple Pressure Vessels (Safety) Regulations 1991.
- 2) Supported by SPECAC Technical File No. **TF10586**  
And adopting (SEP) Sound Engineering Practice as referenced in the above Regulations.

Responsible Person:

**Name:** Mr.G.Poulter  
**Position:** Technical Director  
**Serial No:**  
**Name:**  
**Position:**

**Signature:**



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**conforms to the above**

**Date:** 21st Feb 2013

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**Of:** Specac Ltd.

**Date:**

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