

Reaction Cell Top Plate For Golden Gate™ ATR

User Manual



List of Safety Symbols

Safety Symbol	Meaning
	General Caution. (Reference ISO 7000-0434B, 2004-01)
	Caution – Hot Surface. Reference IEC 60417-5041, 2002-10)
	Caution – Possibility of Electric Shock
	Indoor Use Only

Electrical Safety



Warning: The temperature controller supplied with the accessory are of Class 1 (earthed) construction and must be connected to an earthed mains socket outlet.



Warning: Disconnect the mains from the temperature controller before cleaning. Clean only with a soft cloth, lightly moistened with water.

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.



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1. Introduction

The Reaction Cell Top Plate P/N GS10507 is one of a series of top-plates that are used on the optical unit of the Golden Gate™ ATR system.

The Reaction Cell Top Plate is essentially a pressure vessel with heating capability that seals over a standard single reflection diamond in a tungsten carbide disc, common to all Golden Gate™ diamond ATR top plates, at the base of the vessel. Reaction materials are contained within the pressure vessel and a beam of infrared light is focused through the diamond ATR element from below the pressure vessel to obtain an infrared spectrum of the reaction materials via the ATR technique.

The Reaction Cell Top Plate, which can be supplied in stainless steel or Hastelloy material to suit the reactions to be monitored, can be pressurised to 2800 psi. and raised in temperature to a maximum of 200°C. The volume of the chamber is 24mls with its standard pressure cap fitted and 22mls with the stirring mechanism pressure cap option fitted (P/N GS10513). (See section (9) page 23 for compatibility of chemicals that can be used in experiments.) The stirring version option of the Reaction Cell Top Plate allows for better mixing of reactant species contained in the reaction cell chamber. Section (8) in this manual explains for the stirring option Reaction Cell Top Plate (P/N GS10513 fitted).

The temperature of the Reaction Cell Top Plate is controlled by use of a dedicated 4000 Series™ controller unit. A cooling water jacket is fitted to the Reaction Cell Top Plate to remove any excess heat away from the base Golden Gate™ optical unit and to minimise heating of the sample compartment.

This specific instruction manual for the Reaction Cell Top Plate is to be used in conjunction with the standard Golden Gate™ manual (GS10500 series) supplied with every Golden Gate™ system and a separate 4000 Series™ Temperature Controller manual.

2. Technical Specifications

- Maximum up to 2800 psi.
- Maximum temperature to 200°C.
- Volume cell chamber (24mls). (The cell volume can be changed as special modification if required. Please contact Specac.)
- Standard 1/8" inlet/outlet Swagelok fittings for gas/liquid flow.



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



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



Caution: Always wear adequate PPE gloves when handling the housings 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.



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



Warning: Risk of burns when operated at high temperatures on accessible parts surrounding assembly.

3. *Unpacking and Checklist*

On receipt of the equipment please check that the following have been supplied:

- Reaction Cell Top Plate for Golden Gate™ ATR (GS10507)
 - Golden Gate Optics Unit (if supplied as complete Golden Gate™ ATR system GS10525)
 - 4000 Series™ Temperature Controller, Power Cable and Manual
 - Torque Wrench (GS10504)
 - Square/Hexagon Drive Adapter
 - 2 Open Ended Spanner 7/16 inch and 3/8 inch AF
 - 3.0mm AF Hexagon Head Bit
 - 6.0mm AF Hexagon Head Bit (Ball End)
 - Allen Key 2mm AF Long Arm
 - 3.0mm AF Hexagon Ball Driver
- 1 Essential Spares Kit of parts (GS10528)

Remove the Reaction Cell Top Plate and/or Golden Gate™ optics unit from their packing and install the Golden Gate™ ATR unit and the Reaction Cell Top Plate into the spectrometer (see Golden Gate™ Manual P/N GS10500 for installation procedure into your appropriate spectrometer).

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Remove the 4000 Series™ temperature controller from its packaging and check. Familiarize yourself with the controller by reading the 4000 Series™ Temperature Controller instruction manual.

For heating of the Reaction Cell Top Plate for Golden Gate™ ATR please follow instructions from the 4000 Series™ Temperature Controller manual.

The various hexagon head bits, spanner and Allen key are used for the different screw fixings found on the Reaction Cell Top Plate.

The 6.0mm hexagon head bit is used with the torque wrench for appropriate torque tightening of the top section M8 screws.

The 3.0mm hexagon head bit is used with the torque wrench for appropriate torque tightening of the bottom section M4 screws.

The open-ended spanners are used for tightening of the inlet and outlet flow pipe Swagelock Fittings.

4. Understanding the Reaction Cell Top Plate

On initial receipt of the Reaction Cell Top Plate it will be provided assembled and ready for connection to its supplies for reactants, cooling water and electrical power for heating. The reaction chamber itself will already be sealed for use at the top and bottom sections of the Reaction Cell Top Plate

Top Section of Reaction Cell Top Plate (See Figures 1, 2 and 3)

The top section of the Reaction Cell Top Plate has a pressure plate (1) secured to the reaction chamber. Through this pressure plate pass the 1/8" O.D. inlet and an outlet flow supply tubes for reactants (2) and a temperature monitoring thermocouple (3). The inlet flow tube emerges inside the reaction chamber very close to the base of the chamber near to the diamond surface, with the outlet tube emerging just inside the top of the pressure plate (1). With this arrangement operation by flow of reactants can induce a mixing effect within the chamber.

Alternatively, the reactants can be introduced to the chamber via the inlet flow tube and then the tube can be closed by an inline valve to allow the reaction to occur in a non-flowing environment.

The Reaction Cell Top Plate is to be used with the top section pressure plate (1) secured into position over the reaction chamber using the eight M8 socket head screws (4). (See Figure 2.) Sealing between the top section pressure plate (1) and reaction chamber is achieved using a Perfluoroelastomer O-ring (5).

Note: *On receipt the eight M8 screws (4) will have been screwed tight to a torque setting of 120 cNm.*

Sealing of the reaction chamber by the top section pressure plate allows for pressures up to 2800 psi inside the reaction cell. (See **Pressure Directive Rules - page 4**). The monitoring thermocouple tip (3) must be kept immersed in the reactants all the time during operation. The Reaction Cell Top Plate **must not be used without any reactants** as the thermocouple will be measuring an air temperature in the vessel and overheating may occur. A thermal fuse

that cuts power to the accessory is fitted as a safety precaution should this ever occur.

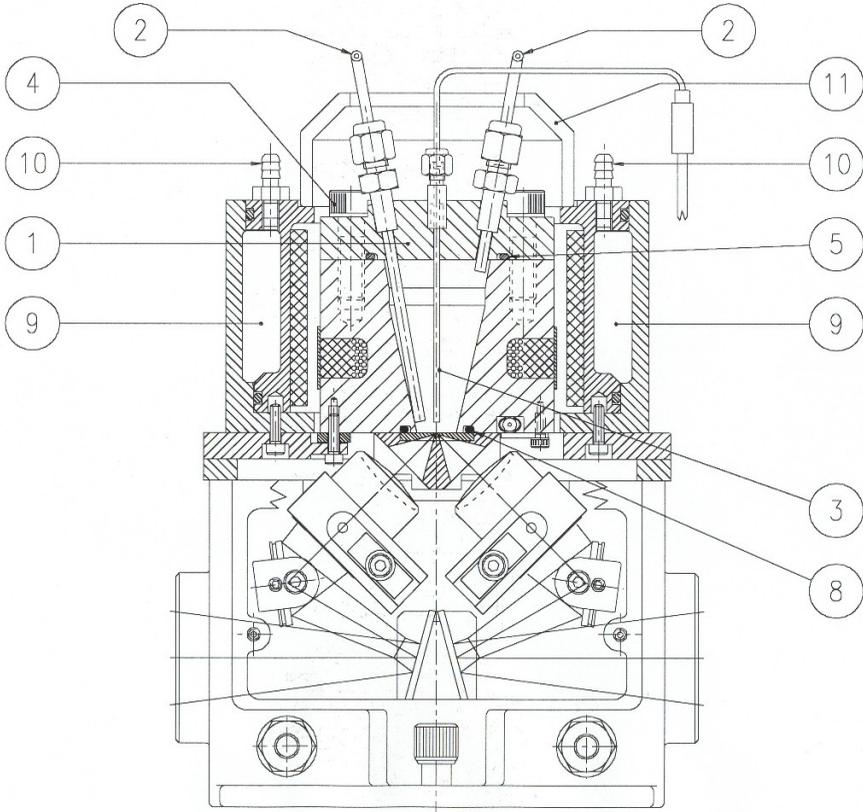


Figure 1. Cut away front view of Reaction Cell Top Plate on Golden Gate™ optical unit

Reaction Cell Top Plate for Golden Gate™ ATR

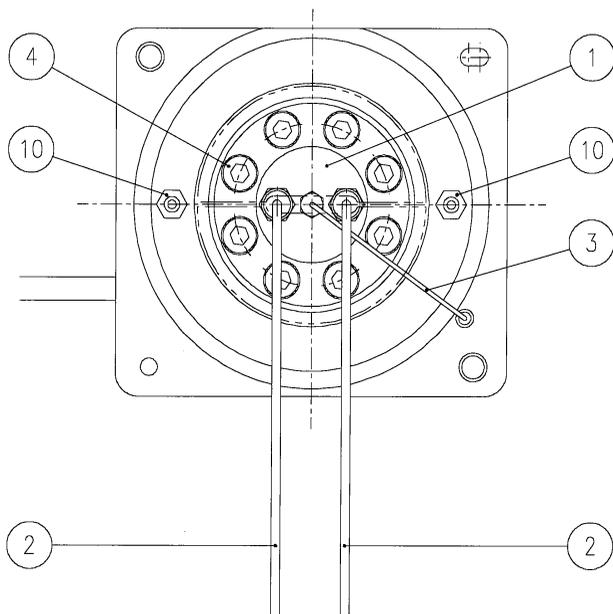


Figure 2. Top view looking at top section of Reaction Cell Top Plate

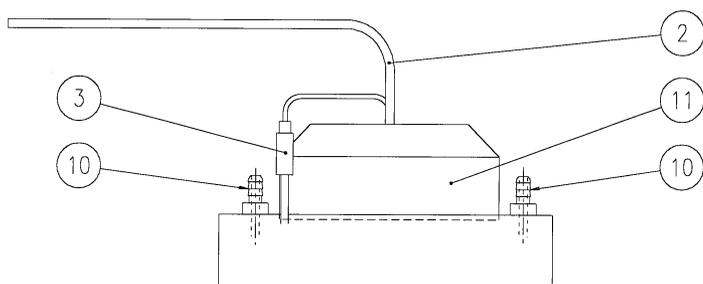


Figure 3. Side view looking at top section of Reaction Cell Top Plate

Bottom Section of Reaction Cell Top Plate (See Figures 1 and 4)

At the base (underside) of the Reaction Cell Top Plate the reaction chamber is secured to the heated diamond assembly plate using six M4 socket head screws (6) that pass through the diamond/tungsten carbide support/clamp bracket (7). Sealing of the base of the reaction chamber to the heated diamond assembly plate is achieved using another, but smaller diameter, Perfluoroelastomer O-ring (8).

Note: *On receipt the six M4 screws (6) will have been screwed tight to a torque setting of 60 cNm.*

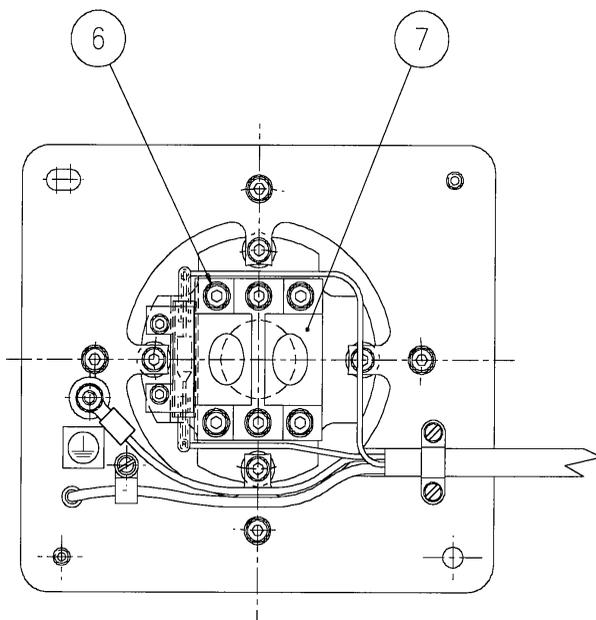


Figure 4. Underside view looking at bottom section of Reaction Cell Top Plate

Water Jacket (See Figures 1, 2 and 3)

Encircling the main body of the Reaction Cell Top Plate is a water jacket (9). For any operation over 50°C temperature, the water jacket must be connected to a flow of cooling water to prevent overheating to the external body of the Reaction Cell and minimise heat transference to the surrounding environment. The water jacket is connected to a cold, water supply using the silicone rubber tubing supplied, pushed over the two fittings (10). Some additional heat shielding is provided in the form of a split cover (11) that is placed over the top section pressure plate (1) when the reaction chamber has been sealed for operation.

* Safety Note on Perfluoroelastomer O-Rings *

The Perfluoroelastomer O-ring seals (5 and 8) used in this accessory are chosen for their extreme chemical resistance. It is their nature that they can suffer from compression set after repeated combined heat and pressure cycling. ***This can give rise to some leakage in extreme cases but can be avoided by making sure that full pressure is not applied before the temperature is raised.***

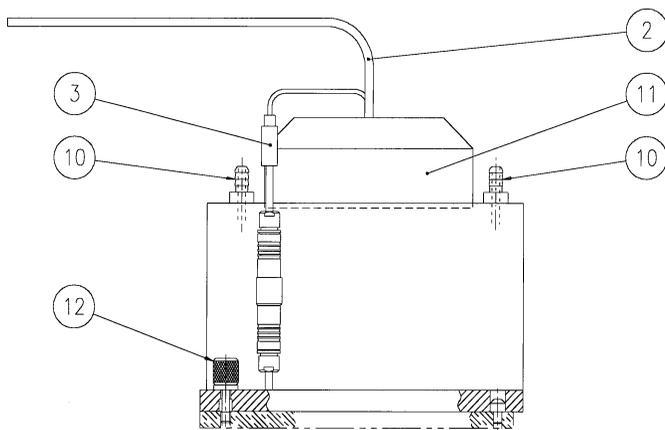


Figure 5. Side view of Reaction Cell Top Plate

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The O-rings should be inspected occasionally and replaced if damaged. The larger diameter O-ring seal in the top section is easily inspected when the top section pressure plate (1) is removed.

To gain access and to remove the bottom smaller diameter O-ring seal The Reaction Cell Top Plate has to be removed from the Golden Gate™ optical unit. (See Figures 4 and 5).

Undo the captive screws (12) and carefully lift off the Reaction Cell away from the Golden Gate™ optical unit.

The Reaction Cell will need to be inverted but make sure the water supply to the cooling water jacket has been sealed off.

Carefully remove the six M4 socket head screws (6) and lift the support/clamp bracket (7) clear. It may be found that the O-ring seal (8) is holding the support/clamp bracket (7) in place and so careful leverage with the tip of a screwdriver blade may be required to release the bracket.

Note: *Check the O-ring seals for evidence of compression set and general condition. Compressed O-rings can often be restored by placing them in an oven at 200°C for 30 minutes. Damaged O-rings must be replaced.*

5. Installation of the Reaction Cell Top Plate

The Reaction Cell Top Plate is fixed to the Golden Gate™ optical unit with the two thumb screws (**12**) (see **Figure 5**). The top plate will only fit to the optical unit in one direction and is correctly fitted when the edges of the top plate are flush with the top edges of the optical unit.

The whole 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 GS10500).

It may be necessary to “fine tune” align the Golden Gate™ optical unit for a maximum throughput of signal with the Reaction Cell Top Plate in position. (Please refer to the Golden Gate™ instruction manual GS10500 to carry out any alignment adjustment via the mirrors and/or lenses in the optical unit of the Golden Gate™).

When installed, make the necessary power connection from the Reaction Cell Top Plate to the 4000 Series™ temperature controller. (See instructions in the 4000 Series™ Temperature Controller manual). Selection of the temperature required for operation of the controller is also found within the temperature controller manual.

6. Operation of the Reaction Cell Top Plate

The Reaction Cell Top Plate and Golden Gate™ optical unit should have already been installed into the spectrometer sample compartment. Optical alignment of the entire accessory should already have been carried out and a satisfactory signal throughput been attained. (Please refer to the installation and alignment procedures from the Golden Gate™ instruction manual P/N GS10500).

In any general procedure for the collection of Infrared data, usually a reference (background) spectrum is required prior to actual sampling. It may be that a reference spectrum is obtained at particular temperature and pressure conditions that will be found during the actual sampling experiment. For the purposes of measuring a reference spectrum at any temperature above ambient up to 200°C, there **MUST** always be some contact of the monitoring thermocouple tip (3) with a reference solvent or solution. A reference spectrum cannot be collected at temperatures above ambient with the thermocouple measuring just the air temperature.

Connections to the Reaction Cell Top Plate

For operation of the Reaction Cell Top Plate commence connection of the various supplies to the accessory.

- 1) Connect the Reaction Cell Top Plate to the 4000 Series™ controller via the power/thermocouple lead and 6-way plug. (Please refer to the 4000 Series™ Temperature Controller manual). Do not select any operating temperatures yet.

Warning: *Before powering up the controller make sure that the power/ thermocouple lead of the Reaction Cell Top Plate for Golden Gate™ ATR is connected to the back of the 4000 Series™ Temperature Controller. The plug is a push fit and the knurled ring is rotated to click the plug into position.*

- 2) Connect the silicone tubing to the water jacket fittings (**10**) and establish a flow of cold water. (Approximately 1 liter per minute flow is adequate). This must be done if the temperature during the reaction will exceed 50°C.
- 3) Connect the inlet and outlet flow tubes (**2**) to the reactant supply. These tube fittings to the reaction chamber are 1/8" Swagelok as standard.

Depending on your experiment proceed as necessary by:

- A) Establishing a flow of reactants through the reaction chamber.
- B) Raising the temperature to the reaction chamber by setting a temperature on the 4000 Series™ temperature controller.
- C) Regulating any pressure to a non-flow experiment.
- D) Collecting any spectra at pertinent time intervals.

Experimental Procedures

The essential design of the Reaction Cell Top Plate enables the study of a reaction by ATR spectroscopy at pressures up to 2800 psi and temperatures up to 200°C. Although the reaction materials for study can be introduced via the inlet and outlet flow tubes (**2**), it is also possible to fill the reaction chamber with reactant materials with the top section pressure plate (**1**) removed. It is possible that a reaction may not take place until suitable pressure and/or temperature conditions are met, so the reactants can already be introduced to the reaction chamber without the need of the inlet and outlet flow tubes. The experimentation can proceed once the top section pressure plate (**1**) is secured into position, with the reactants contained in the reaction chamber.

The accessory can either monitor a batch reaction taking place in the reaction chamber, (non-flow experiment) using the Swagelok fittings and tubing to supply any pressure, or it can be used to monitor a remote reaction by pumping the reactants continuously through the accessory (flow experiment). Depending on the type of flow experiment, it may be necessary to use a heated transfer line or a well

lagged line between the main reaction vessel and the reaction chamber of the Reaction Cell Top Plate.

Warning: *Any pressure build-up in the reaction chamber 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 2800 psi.***



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.

Cleaning and Storage

At the end of any experiment after flushing through with a suitable solvent it is advisable to remove the top section pressure plate (1). Allow the reaction chamber to dry out (tissues or swabs etc, may be used to speed up the process) unless it is to be used again within a short time. Inspect the top O-ring for any signs of compression and/or damage and re-condition or replace as necessary.

If the Reaction Cell Top Plate is to be stored for a long time, then the water jacket should be emptied of water by inverting the Reaction Cell Top Plate over a sink.

To gain access to the diamond ATR crystal surface, should this require cleaning, then the reaction chamber must be removed from the heated diamond assembly plate by unscrewing the six M4 socket head screws (6) that pass through the diamond/tungsten carbide support/clamp bracket (7) (see Figure 4.).

The diamond crystal and surrounding tungsten carbide support disc are both extremely durable and robust materials so fairly abrasive cleaning materials could be used to clean the diamond surface. However, it is usually sufficient to clean most reactants from the diamond surface using a cloth or tissue dampened with a suitable solvent, such as water, methanol or, maybe, acetone.

7. Operating Parameters

The Reaction Cell Top Plate for the Golden Gate™ ATR System is provided with its own dedicated 4000 Series™ Temperature Controller. A separate manual is supplied for specific operation of the 4000 Series™ Temperature Controller.

For operation of the Reaction Cell 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 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.

Specifications

Accessory Type P/N's GS10507 and GS10525

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 Reaction Cell Golden Gate™ ATR Top Plate GS10507 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
PPL ₁	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	4.8
ArSt	Automatic Reset (Integral Time Constant)	3.14
rAtE	Rate (Derivative Time Constant)	0.48
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	200
SPLl	Setpoint Lower Limit	0
OPuL	Primary (Heat) Output Upper Power Limit	100
Ct I	Output 1 Cycle Time	4
PhAl	Process High Alarm	200
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	diSA
rP	Setpoint Ramp Rate Value	(No Value)
SP	SP Value	0
SLoc	Set-up Lock Code	10

Reaction Cell Top Plate for Golden Gate™ ATR



8. *Stirring Version of Reaction Cell Top Plate*

The Reaction Cell Top Plate can be offered as an optional stirring version. The standard top section pressure plate (1) is replaced by an alternative pressure plate (13) that incorporates a stirring rod mechanism (14) in addition to the monitoring thermocouple (3) and the inlet and outlet tubes (2). (See Figure 6). The stirring mechanism assists in the mixing of reactants within the reaction chamber and so can allow for a more homogenous mixture to be monitored for any non-flow experimentation within the reaction chamber. Similar to the standard version of Reaction Cell Top Plate, the stirring version can be supplied in stainless steel or hastalloy material.

Stirring Mechanism

Note: *When using the stirring mechanism at any rotation speed this does not affect the maximum temperature (200°C) and pressure (2800 psi) capabilities of the Reaction Cell Top Plate for the Golden Gate™.*

The viscosity of the reactant mixture within the reaction chamber will determine the speed of rotation of the stirrer (14). A lower viscosity (thinner) fluid will allow for faster rotation of the stirrer whereas a higher viscosity (thicker) fluid will tend to suppress the speed of rotation. Because of the specific nature that exists for many types of reactant mixtures that may wish to be analysed within the reaction chamber, Specac cannot recommend which speed of rotation of the stirrer will be ideal for the specific reactant mix. It will be dependent on individual circumstances.

Operation of the Stirring Mechanism

A speed controller unit is supplied with the Stirring Reaction Cell Top Plate to power the motor (15) that turns the stirrer (14) within the reaction chamber. The motor (15) of the stirring mechanism is connected to the speed controller via the positive and negative terminals on the front of the speed controller.

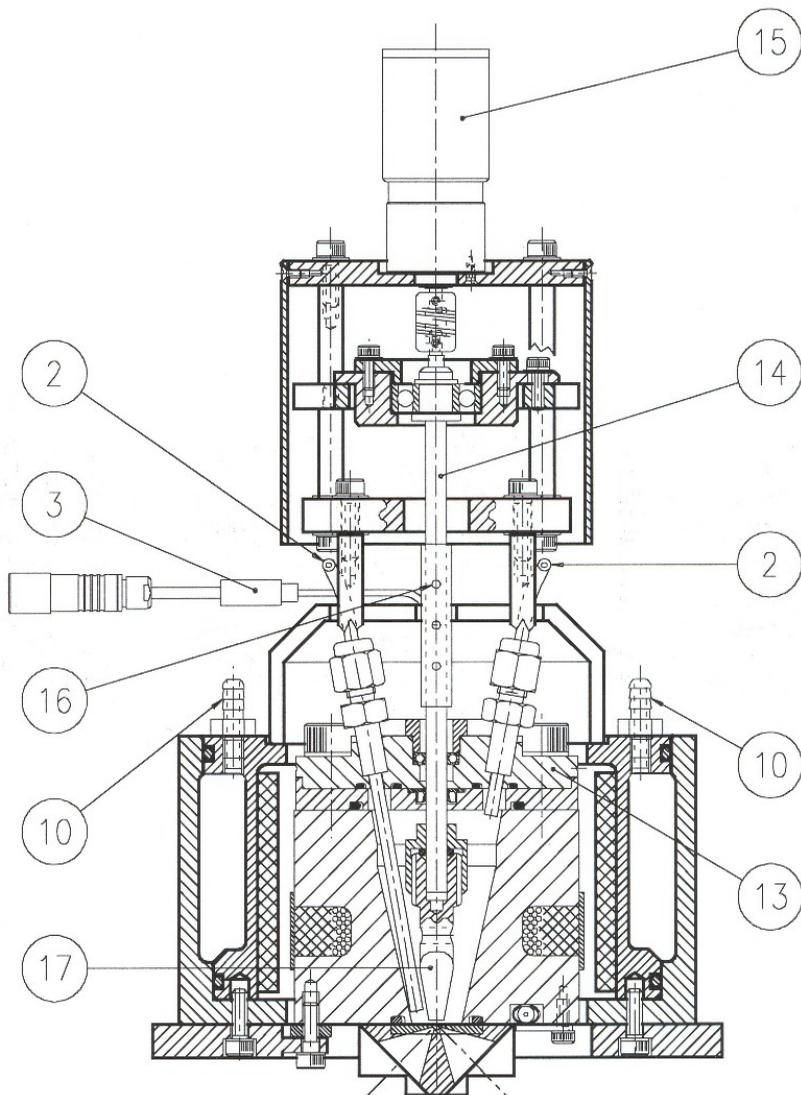


Figure 6. Cut away view of the stirring mechanism top section option on the Reaction Cell Top Plate.

The rotary dial on the speed controller is a voltage regulator from 0 to 15 volts in 1 volt divisions. Maximum rotation speed of the stirrer is achieved at the 15 volt setting of the dial, but the **actual** rotation of the stirrer is dependent upon the pressure, temperature and reactant viscosity conditions within the reaction chamber. On the stirring mechanism (**14**) there is an external visual indicator dot mark (**16**) that is used to measure the rotation of the stirrer. By counting the number of complete rotations of the stirrer from this dot mark over a set time, the number of revolutions per minute for the stirrer can be calculated against a specific voltage setting on the speed controller dial. With a set, known, standard fluid specific for your experimentation in the reaction chamber at ambient pressure and temperature, the rotation for the stirrer can be calibrated over a range of voltage settings.

Connection of the Stirring Mechanism to the Reaction Chamber

The alternative stirring top section pressure plate (**13**) is secured to the top section of the Reaction Cell Top Plate in the same way as the standard pressure plate with the eight M8 socket head screws (**4**). (See Section 4, page 7 of this manual.)

The stirrer itself (**14**) consists of a sapphire rod that passes through the top section pressure plate (**13**) via a rotatable seal. To the bottom of the rod inside the reaction chamber there is a detachable paddle (**17**), which fits over the end of the sapphire stirrer rod and is clamped in position by its clamp nut. For operation, make sure that the paddle part is secured to the stirring rod, before screwing the top section pressure plate (**13**) to the reaction chamber.

To make the stirrer (**14**) rotate, connect the speed controller to the mains power supply by its power lead and plug. Make sure the voltage regulator dial on the speed controller is initially set at 0 volts (to prevent accidental rotation of the stirrer) and switch on the power from the mains. Turn the dial on the speed controller to a voltage setting and see that the stirrer (**14**) is rotating from the dot mark (**17**). Count the number of rotations of the stirrer and adjust the voltage regulator until a desired speed of rotation is obtained.

Cleaning and Storage

For cleaning and storage of the Stirring Reaction Cell Top Plate please follow the same procedure as for the standard Reaction Cell Top Plate accessory (see **Section 6, page 16 of this manual**).

9. Materials of the Reaction Cell That Are In Contact With Chemicals Used in Experiments

For the Reaction Cell Top Plate Assembly P/N GS10507

Body: Stainless Steel (ISO 316-S13) or Hastelloy (C276).
O-rings: Perfluoroelastomer (Isolast 9509).
Diamond puck: Tungsten Carbide, Diamond & Braze material around diamond.

For the Reaction Cell Stirrer P/N GS10513

Main parts: Stainless Steel (ISO 316-S13) or Hastelloy (C276).
Rotating seal: Turcon T24 (a high, grade PTFE with carbon additive).
Stirrer: Sapphire (rod) and Stainless Steel (ISO 316-S13 or Hastelloy (C276) (paddle).
O-rings: Perfluoroelastomer (Isolast 9509).



Warning! *Before use it should be confirmed that the sample to be tested in experimentation is compatible against 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 of unsuitable materials. Whenever the equipment is not being used ensure it has been thoroughly cleaned and dried before storage.

10. Part Number Identification List

- (1) Top section pressure plate.
- (2) Inlet and outlet flow tubes.
- (3) Thermocouple.
- (4) M8 socket head screws.
- (5) Top section perfluoroelastomer O-ring seal.
- (6) M4 socket head screws.
- (7) Support/clamp bracket
- (8) Bottom section perfluoroelastomer O-ring seal.
- (9) Water jacket.
- (10) Water jacket tubing fittings.
- (11) Heat shield cover.
- (12) Top plate fixing thumb screw.
- (13) Stirring option top section pressure plate.
- (14) Stirring mechanism rod (sapphire).
- (15) Stirring option motor.
- (16) Stirring mechanism rotator dot mark.
- (17) Detachable paddle.

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