

Variable Temperature Cell Holders



User Manual



2I-21525-21

Variable Temperature Cell Holders

User Manual

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1. Introduction

Thank you for purchasing a Specac product.

The Variable Temperature (VT) Cell/Cuvette Holders P/N GS21525 (Cell Holder) and P/N GS21530 (Cuvette Holder) have been designed to allow for the study of liquid and solid samples, via transmission spectroscopy ranging from the Far UV through to the Far IR over a wide temperature range (-190°C to +250°C).

The desired sample test temperature is achieved by regulating the temperature of the heating elements inserted into the cell holder assembly with or without applying different refrigerants (see Table 1) while the chamber vacuum environment is maintained. The low pressure of a surrounding vacuum environment minimizes the heat losses of the sample in the liquid/solid cell holder and therefore more reproducible and stable temperatures can be achieved for a variety of applications and experimental conditions.

Note: *Liquid sample cell assemblies are limited to a temperature range between -70°C and +250°C due to the limitations of materials used in their construction.*

The VT Cell Holder Types.

1. The VT Cell Holder P/N GS21525 is provided with a transmission type cell holder assembly that accommodates a variety of Specac sample cells. The list of sample cell types recommended for use within the GS21525 are as follows:

P/N GS05910 Series high pressure, heatable, sealed, **flow** liquid cells.

P/N GS20510 Series heatable, demountable, **static** liquid cells.

P/N GS20580 Series heatable, demountable, **flow** liquid cells.

P/N GS20610 Solids holder.

The outer vacuum jacket has two window ports arranged at a 180° angle orientation to allow for transmission of light through the

sample cell accommodated within. A pair of NaCl windows, P/N GS20800, are provided as standard for use in the outer vacuum jacket of the VT Cell.

By choosing different window materials for both the internal liquid cells and the vacuum jacket body of the VT Cell holder P/N GS21525, samples analyses spectroscopically can be achieved over a wide frequency range. from the UV to the far IR spectrum.

2. The VT Cuvette Holder P/N GS21530 is provided with a cuvette holder assembly that accommodates standard rectangular quartz glass cuvettes up to 10mm in pathlength. (The cuvette itself is not supplied by Specac.) The outer vacuum jacket of the VT cell holder has four window ports arranged at 90° angle orientation to each other, thus allowing for transmission experimentation where light would pass through from source to detector in a straight line at a 180° angle orientation, or at a 90° angle from a light scattering technique. (e.g. Raman or Fluorescence/UV Spectroscopy).

Two pairs of Spectrosil B (UV Quartz) windows P/N GS20898 are provided as standard for use in the four-window port outer vacuum jacket.

For correct temperature control operation of the VT Cuvette Holder P/N GS21530 it requires a vacuum is created that surrounds the cuvette in the cuvette holder/dewar assembly. Therefore, any cuvettes to be used in this system must be stoppered to prevent the sample contained within being lost to the vacuum environment.

Note: Separate instruction manuals have been written for the operation and use of the various sample cells types as listed above. These instruction manuals should be consulted prior to use within the VT Cell holder P/N GS21525 or GS21530 accessory.

Power to either version of VT Cell/Cuvette Holders (P/N GS21525 or P/N GS21530) is provided by a dedicated 4000 Series™ temperature controller. A separate instruction manual (supplied) has been written for operation of the controller and so the controller manual should also

be consulted along with this instruction manual for operation of the VT Cell/Cuvette Holders.

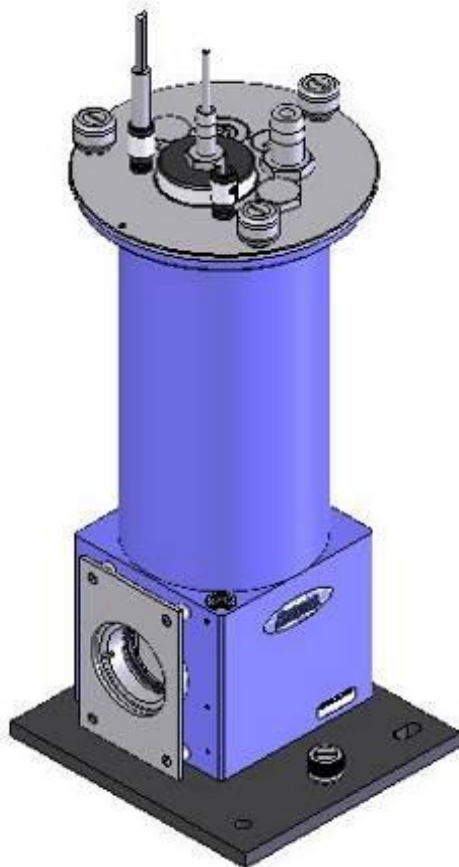


Fig 1. VT Cell Holder Type P/N GS21525

2. Checklist

When you receive your version of VT Cell Holder you should check the following parts have been supplied.

For VT Cell Holder P/N GS21525

1. Dewar/Cell Holder Assembly.
2. Copper/Constantan thermocouple.
3. 2 Window Port Vacuum Jacket Assembly with 3" x 2" mount and Benchmark™ baseplate fixing.
4. Pair of NaCl Windows for Vacuum Jacket.
5. Window Key.
6. Mushroom Rods (large and small pistons).
7. Drop Disc Block and Rod.
8. Bung.
9. Allen keys (5/64" and 3.0 mm sizes).
10. Power Cable Assembly.
11. Benchmark™ baseplate.
12. 4000 Series™ Temperature Controller and manual.
13. Benchmark™ baseplate installation instruction manual.
14. Essential Spares Kit P/N GS21526

For VT Cuvette Holder P/N GS21530

1. Dewar/Cuvette Holder Assembly.
2. Copper/Constantan thermocouple.
3. 4 Window Port Vacuum Jacket Assembly with 3" x 2" mount and Benchmark™ baseplate fixing.
4. 2 Pairs of Spectrosil B (UV Quartz) Windows for Vacuum Jacket.
5. Pair of blank (metal) windows for Vacuum Jacket.
6. Window Key.
7. Mushroom Rods (large and small pistons).
8. Drop Disc Block and Rod.
9. Bung.
10. Allen keys (5/64" and 3.0 mm sizes).
11. Power Cable Assembly.
12. Benchmark™ baseplate.
13. 4000 Series™ Temperature Controller and manual
14. Benchmark™ baseplate installation instruction manual.
15. Essential Spares Kit P/N GS21526.

3. Safety Warnings

List of Safety Symbols

Safety Symbol	Meaning
	Caution (Reference ISO 7000-0434B, 2004-01)
	Caution – Hot Surface (Reference IEC 60417-5041, 2002-10)
	Caution – Low Temperature / Freezing Conditions (Reference ISO 7000-W010, 2010-05)
	Caution – Possibility of Electric Shock
	Indoor Use Only

General Safety

It is important to read any associated safety information before use of any Specac accessory and/or the 4000 Series temperature controller.



Caution! *This user instruction manual should be read in conjunction with the manual provided with the intended Specac accessory for which this controller was supplied.*



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

Electrical Safety



Caution! *If the inlet fuse requires replacement, replace only with the correct fuse type as indicated on the rating label of the product and in section 3 Specification of this user manual.*

Whenever it is likely that the safety protection has been impaired the controller shall be made inoperative and be secured against unintended operation. The protection is likely to be impaired if for example the controller:

- Shows visible signs of damage.
- Fails to perform the intended measurements.
- Has been subjected to prolonged storage under unfavorable conditions.
- Has been subjected to severe transport stresses.



Warning: This equipment is intended to be connected to an earthed mains socket outlet. Protection provided by this equipment will be impaired if the earth connection is not present.



Warning: Only use the mains cord set supplied with the controller. If an alternative cord set is required, replace it only with an equivalent type of the same rating which is acceptable for use in the country for which the controller is to be used.



Warning: Do not operate the appliance with a broken cord or plug, or if the appliance malfunctions, or is dropped or damaged in anyway.



Warning: Do not use this equipment with any accessory it has not been specifically configured to operate. Contact Specac Technical Support or our sales team for further information regarding the compatibility of the controller with other devices and 3rd party accessories.



Warning: Do not position the equipment so that it is difficult to access the disconnecting device (Mains plug and/or appliance coupler) when in use. Only use a main outlet socket that is accessible during operation.



Warning: No user serviceable parts within. Contact Specac and/or your local Specac authorised agent for all service and repair requirements.



Warning: Disconnect the device from the mains supply before cleaning. 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.



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



Warning: Do not block or cover the ventilation holes on the rear, and sides of the controller. It is recommended to have at least 100mm spacing around the sides and rear of the product from other objects or electrical equipment and to be placed on a stable, flat surface.



Warning: Never allow liquid or solid objects to fall into the ventilation openings. If any liquid or solid object falls inside the temperature controller, unplug the temperature controller immediately and have it checked by Specac before operating it further. Never push objects of any kind into the temperature controller through the ventilation openings as they may come in contact with dangerous voltage points or short-out parts that could result in a fire or electric shock.

Safety



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.



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.



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



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.

Safety Warnings – use of refrigerants.



Warning: Risk of Frostbite, cold burns.

When using refrigerants with this accessory, wear appropriate safety clothing (PPE) such as cryo gloves, face shield, use insulated containers and avoid skin contact.

Always follow local laboratory safety practices and procedures.



Warning: Asphyxiation (LN₂ vapor) and Explosion.

Use in a well-ventilated area or with fume hood, use an oxygen monitor if applicable.

Use an appropriate Oxygen Depletion Calculator to calculate the volume of released gas vs the volume of the working area.



Warning: DO NOT USE test samples that are potentially explosive under varying temperature conditions in this device. Check material data sheets.



Warning: When using refrigerants DO NOT block the vent tube located in the Dewar bung (part 44) this must be open to allow refrigerant vapour to boil off.

4. Explanation of VT Cell Types and Their Configuration for Operation

Note: *The following instructions are applicable for operation of both versions of VT Holders P/N's GS21525 and GS21530. For explanation in the main, diagrams have been used representing the VT Cell Holder P/N GS21525 with the refrigerant dewar/cell holder and 2 window port vacuum jacket assembly.*

Vacuum Requirement for Operation

When operating the VT Cell, it must be connected to a vacuum system. For low temperature work using liquid nitrogen as a refrigerant the best nitrogen hold time is achieved operating at a vacuum of less than 0.05 Torr. (0.067 mbar). Although the whole accessory will operate at higher pressures, the hold time for refrigerants being used in the dewar will be reduced. Specac recommends that the VT Cell accessory should be evacuated even when used at temperatures above ambient to facilitate for precise temperature control.

Specac provides a Vacuum Pump Kit P/N GS03640 with an appropriate vacuum pump, vacuum hoses and connection fitting parts for specific use with the VT Cell accessories P/N's GS21525 and GS21530 to create the vacuum conditions necessary for operation.

Main Assemblies of the VT Cell Holders

The VT Cell consists of two main parts: a refrigerant dewar/cell holder assembly and a vacuum jacket assembly

Dewar/Cell Holder and Vacuum Jacket Assembly (See Fig. 2.)

In operation, a specific sample cell type for the VT Cell P/N GS21525 or cuvette for the VT Cell P/N GS21530 is placed into the dewar/cell holder assembly (1). This combined assembly of parts is then placed into the vacuum jacket assembly (2), which will already have had a set of jacket windows installed into position. (See Windows for VT Cell

Vacuum Jacket - page 10). The dewar cell holder assembly attains a fixed/sealed position in the vacuum jacket guided by a locating pin hole (3) and tightening of three thumb screws (4). The pin hole (3) is aligned with the vacuum jacket pin (5). The fixed positioning ensures that the correct alignment in the optical beam from the spectrometer system is achieved every time. When assembled, the whole VT Cell accessory (vacuum jacket, refrigerant chamber and sample cell) is mounted in a spectrometer by means of a Specac Benchmark™ type baseplate via the adapter mounting plate (6), affixed to the underside of the VT Cell vacuum jacket.

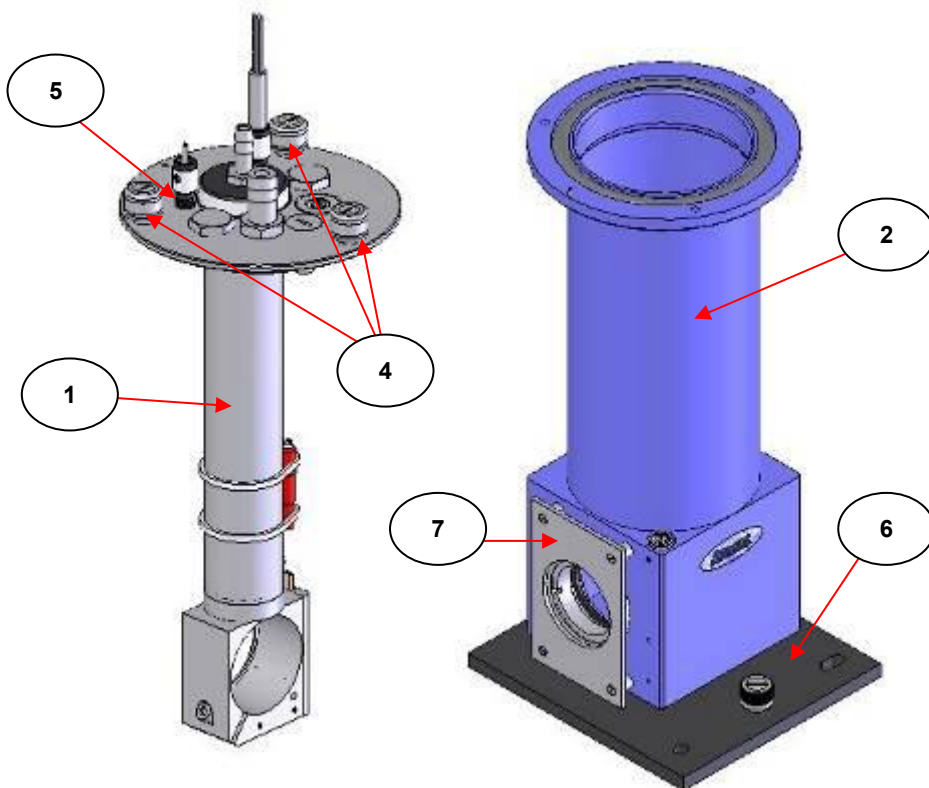


Fig 2. VT Cell GS21525 Dewar and Vacuum Jacket Assemblies

An alternative way to mount the VT Cell accessory is to use a standard 3" x 2" slide plate mount (7) on the vacuum jacket. To make use of the slide plate mount, the Benchmark™ adapter plate (6) must be removed from the vacuum jacket. This is achieved by undoing the two fixing screws (8) on the underside of the vacuum jacket (see Fig 3.)

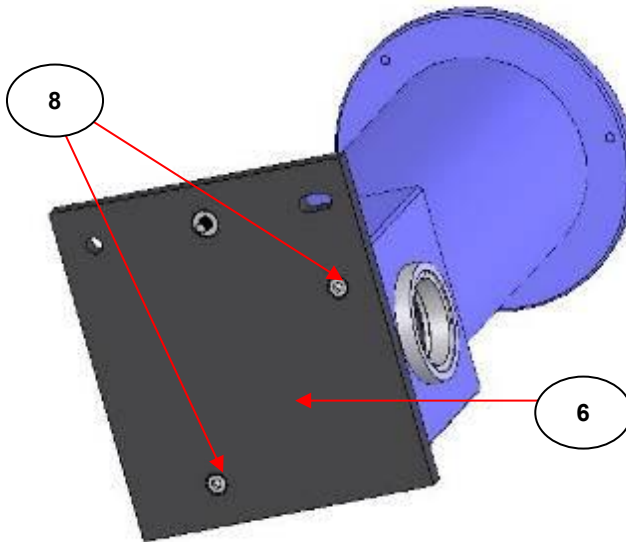
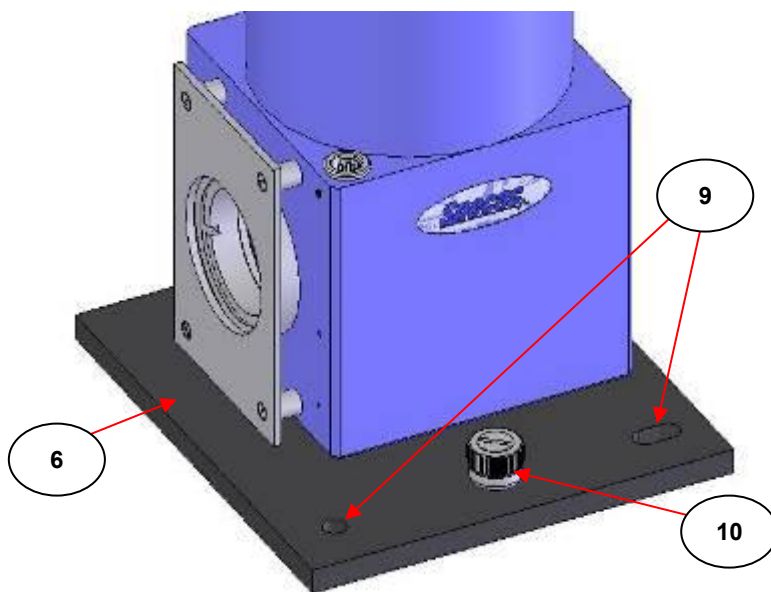


Fig 3. Underside View of GS21525 Vacuum Jacket Showing Benchmark™ Adapter Plate Fixing Screws.

If the VT Cell Accessory is to be installed into the spectrometer using a Benchmark™ baseplate, the two location holes (slot and round – 9) on the adapter plate (6) are placed over the support pillars of the Benchmark™ baseplate and the VT Cell Assembly is then secured to the Benchmark™ baseplate via the middle pillar using the central thumbscrew (10). (See Fig 4.) After installation, the various connections i.e. power cables, thermocouple and vacuum lines are then made to and from the VT Cell Accessory.



**Fig 4. GS21525 Detail of Benchmark™ Adapter Plate
Location Holes and Central Thumbscrew**

Windows for VT Cell Vacuum Jacket

The VT Cell P/N GS21525 vacuum jacket has 2 window aperture ports that accept a wide range of window materials. The standard jacket windows supplied with the VT Cell P/N GS21525 are sodium chloride windows P/N GS20800. These windows are stored in a sealed container with a desiccant. The windows should not be removed from their container until they are ready for installation into the vacuum jacket.

The VT Cell P/N GS21530 vacuum jacket has 4 window aperture ports that accept a wide range of window materials. The standard jacket windows supplied with the VT Cuvette Holder P/N GS21530 are 2 pairs of UV Quartz (Spectrosil B) windows P/N GS20898 for complete sealing of the vacuum jacket at the 4 window aperture ports.

Installation of Vacuum Jacket Windows (See Fig 5.)

Important: Avoid touching the polished faces of the windows on installation.

For both VT Cell types P/N's GS21525 and GS21530, installation of a window material into the vacuum jacket is the same procedure as described below.

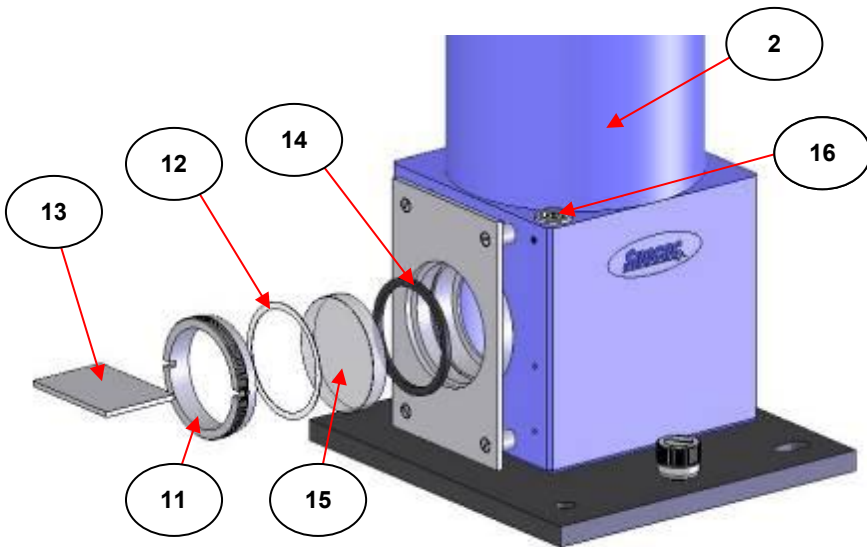


Fig 5. GS21525 Detail for Vacuum Jacket Window Assembly

Hold the vacuum jacket (2) and remove the screw thread retaining rings (11) and PTFE seals (12) from the jacket aperture ports with the window key (13) provided. The window key is a rectangular shape of metal that locates into the slots of the retaining rings (11) enabling them to be loosened or tightened.

Ensure the elastomer seal (14) is in place and insert a window (15) in each aperture.

Replace the PTFE seals (**12**) and screw thread retaining rings (**11**) and tighten sufficiently to establish a vacuum tight seal between the windows on either side of the vacuum jacket.

Note: *To minimize fogging of the windows (if hygroscopic) it is advisable to supply power to the window heaters (24 Volt supply) as soon as possible after the windows are fitted. This can be done by connecting the leads from the temperature controller to the latching socket (**16**) at the base of the vacuum jacket and following the switching on instructions of the controller manual. The heat supplied will keep the temperature of the jacket windows several degrees above ambient. It can also help if a desiccant is left inside the bottom of the vacuum jacket although it is not essential.*

The VT Cell P/N GS21530 vacuum jacket having 4 window aperture ports means that two of the windows that transmit light can be configured for a 180° angle (straight through) orientation or at a 90° orientation (at right angles to each other) depending on the type of spectroscopic experiment to be carried out. When the windows are at a 90° angle orientation to each other, fluorescence and Raman experimentation for scattered light at this angle can be carried out using the VT Cell GS21530 with a 4-sided, clear windowed cuvette.

Please also note that the VT Cell P/N GS21530 vacuum jacket body will accept the VT Cell P/N GS21525 dewar/cell holder assembly, such that with jacket windows installed at two of the four aperture ports in a 180° angle orientation, all of the specific sample cell holders that are compatible for use in P/N GS21525 could be used with this combination of VT Cell accessory parts.

Conversely, the VT Cell P/N GS21525 vacuum jacket body will accept the VT Cell P/N GS21530 dewar/cuvette holder assembly, but because the vacuum jacket assembly only has two window aperture ports, any cuvette sample cells contained within can only be operated for a 180° angle mode of orientation for the light throughput.

VT Cell Dewar / Cell Holder Assembly Top Plate Layout

At the top of the dewar/cell/cuvette holder assembly is a circular plate which carries various connections for vacuum, power cables, thermocouples and liquid delivery tubing. (See **Fig 6.**) There are seven port/connections through the cell holder/refrigerant chamber top plate.

1. Cell Heater Connection (**17**).
2. Controlling Thermocouple Port (**18**).
3. Vacuum Port (**19**).
4. Connector Port 1 for liquid flow through (**20**) - (Inlet port).
5. Connector Port 2 for liquid flow through (**21**) - (Outlet port).
6. Port for additional thermocouple connection (**22**).
7. Access port for electrical connection of the Specac Spectroelectrochemical Cell (**23**).

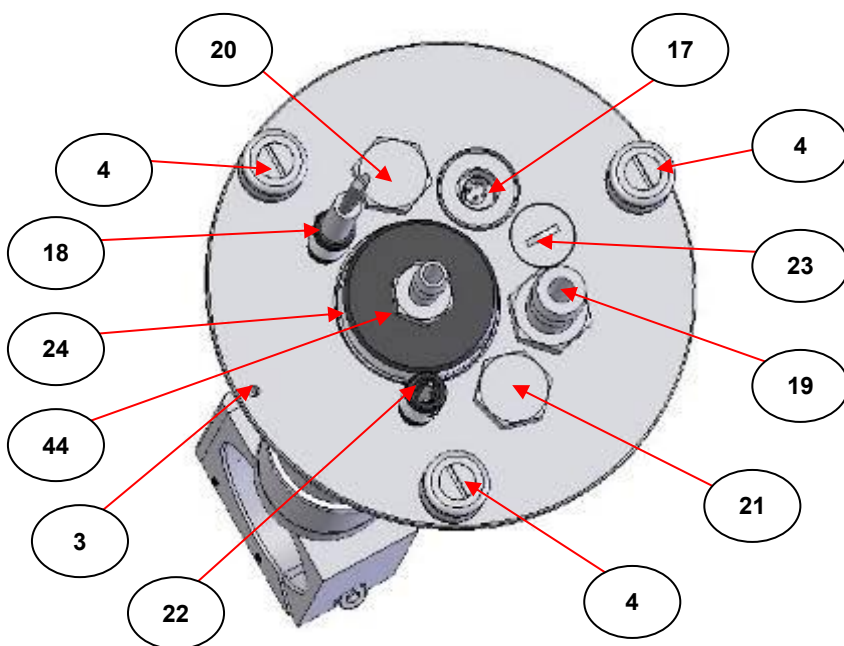


Fig 6. Dewar Assembly Top Pressure Plate Port Configuration

The main off-center circular area of the top plate is the access hole to the dewar assembly refrigerant chamber itself (24). (Figs 6 and 8. show the dewar stopper bung part (44) in position covering the chamber.)

The power cable from the temperature controller for the dewar/cell holder assembly heaters are connected to a four way hermetically sealed socket (17) on this upper circular plate of the dewar assembly.

A copper/constantan (Cu/CuNi) T-type thermocouple (25) is used for control measurement of temperature. It passes through port (18) on the top plate and it is permanently fixed in place with its junction end located in the middle of the two cell holder heaters (26). (See Fig 7.)

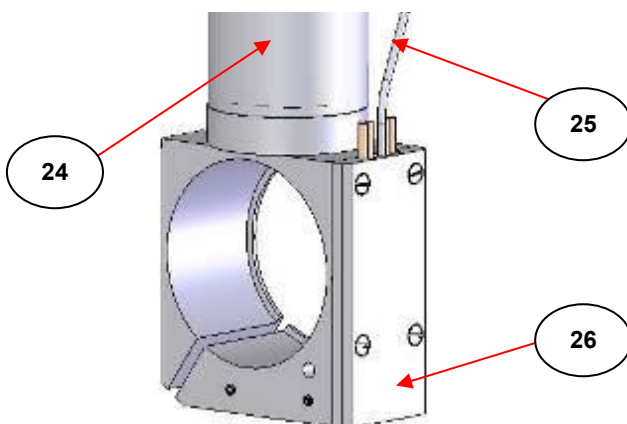


Fig 7. VT Cell GS21525 Cell Holder Detail for Controlling Thermocouple Connection

The controlling thermocouple (25) is thus very near to any sample cell held in the cell holder and in this position the temperature monitored will be very close to the actual sample cell temperature when stability is achieved. However, a more precise cell temperature measurement

position can be achieved. (See section on 'Direct Temperature Monitoring from the Sample Cell' Section 8, Page 37).

The two heaters (**26**) fitted into the cell holder part/area of the dewar assembly are wired in series with each other and when operating at full rating will dissipate about 40 Watts. These heaters enable the user to vary the temperature of the sample cells over a range from -190°C to +250°C.

Note: *Liquid samples used in liquid type cells can be operated between temperatures of -70°C to +250°C. Solid samples used in the solid's holder P/N GS20610 can be operated over the full temperature range capability from -190°C to +250°C.*

A specific choice of refrigerant when placed in the dewar chamber (**24**) determines the actual temperature range capable of being studied in an experiment. Please see an explanation in the 'Refrigerant' section of this manual (Page 22).



Warning: Risk of burns from Heated or Cooled surfaces.

Before removing the dewar/cell holder (1) from the vacuum jacket assembly (2) it must be allowed to return to ambient temperature (~ 15-25 °C), following use. The Temperature Controller should be set to ambient and monitored until this is achieved before any removal of the assembly is attempted.

Vacuum Connection and Blanked Off Ports

The vacuum port (**19**) is for attachment of a vacuum line hose to evacuate the VT Cell Accessory for precise temperature control in operation.

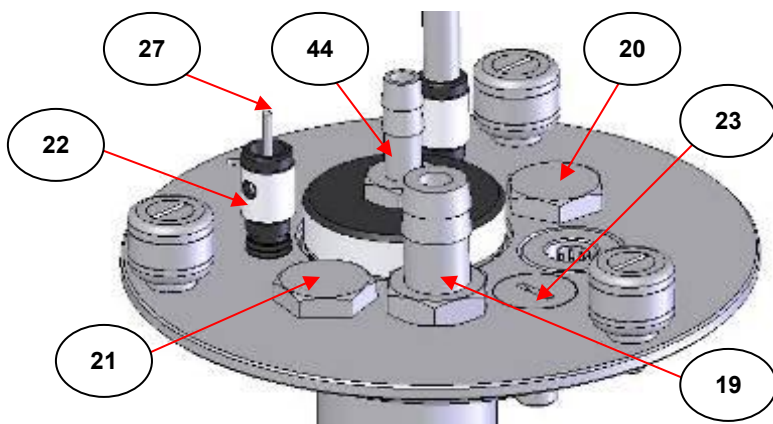


Fig 8. VT Cell Blanked Off Ports When Supplied as Standard

As standard any access port through the top plate that is not utilized during operation of the VT Cell Accessory **must** be plugged to maintain a vacuum. Both versions of VT Cell P/N's GS21525 and GS21530 are supplied as standard with blanking plugs at port positions (20), (21) and (23) and a blanking rod (27) at port position (22). (See Fig 8.)

The type of sample cell for liquids or solids that is to be used within the cell holder part (28 – see Fig 9.) of the dewar assembly will determine which of these port positions may be needed.

- 1) To allow for connectivity of liquid flow to flow cells, the blanking plugs for ports (20) and (21) are removed and the VT Cell Flow Fittings Kit P/N GS20080 is needed to fit to these port positions. (Please see the section on page 21 in this manual for fitting of this kit of parts.)
- 2) To allow for fitting of the Spectroelectrochemical Cell GS20900 Series, the VT Cell Flow fittings Kit P/N GS20080 must be fitted and the blanking plug for port (23) is removed and replaced with the electrical power cable assembly of parts to attach to the Spectroelectrochemical Cell itself. (Please refer to the specific

instruction manual for the Spectroelectrochemical Cell P/N GS20900 for fitting the kit of parts supplied with this cell).

- 3) To allow for a secondary monitoring thermocouple to be installed and directly attached to a specific liquid sample cell (static or flow) or the solids holder P/N GS20610, the blanking rod (**27**) is removed from port (**22**) and replaced with the monitoring thermocouple assembly P/N GS20200 (**49**). (Please see the section in this manual on 'Direct Temperature Monitoring from the Sample Cell' Section 8, Page 37, for fitting of this thermocouple assembly.)

5. Installation of a Sample Cell into the Dewar/Cell Holder Assembly

The sample cell holder part (area) of the VT Cell Holder Accessories differ between P/N's GS21525 (**28**) and GS21530 (**29**). (See **Fig 9**.) The sample cell holder part is fitted directly to the base of the stainless-steel refrigerant chamber (**24**).

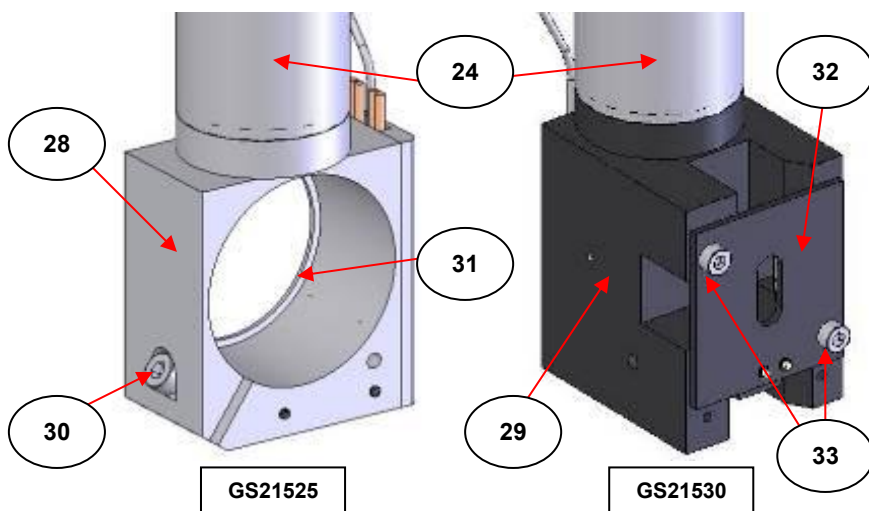


Fig 9. Cell Holder Parts of VT Cells GS21525 and GS21530

The various liquid and solid sample cell holders are fitted into the cell holder part (**28**) of P/N GS21525 and are held in position for good thermal contact by tightening of the clamping screw (**30**).

Note: *The cell holder part (**28**) has a flange ring (**31**) to one side of the cell holder aperture. Sample cells can only be inserted into the aperture of the cell holder from the side opposite to the flange ring (**31**). The flange ring acts as an end stop for correct positioning of a sample cell within the cell holder part (**28**).*

Liquid cuvette sample cells with typical dimensions of 12.5mm long x 12.5mm wide x 41mm high are placed in the cuvette holder part (29) of GS21530. A cuvette can be placed directly into the square slot cavity behind the front clamping plate (32) without the need to remove the clamping plate (32) by its two fixing screws (33). There is a small spring clip behind the clamping plate that pushes against the cuvette and holds it secure in the holder part.

Note: *Because of the vacuum operation environment needed for precise temperature operation of the VT Cell Accessory, any cuvettes being used in P/N GS21530 must be stoppered/capped to prevent loss of the liquid sample to the vacuum.*

Fitting Sample Cells into the Dewar/Cell Holder Assembly of P/N GS21525 (See Figs 10, A, B, C, D, E)

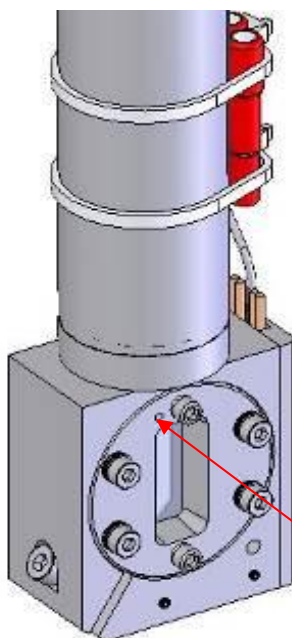
Before the dewar/cell holder assembly (1) is placed into the vacuum jacket (2), a sample cell should be installed into the cell holder part (28) of VT Cell P/N GS21525 and a cuvette cell into the cuvette holder part (29) of VT Cell P/N GS21530.

As mentioned in the introduction a variety of liquid sample cells (static or flow) and a solids holder P/N GS20610 can be used in the VT Cell P/N GS21525. The following procedure is adopted for installation of a sample cell type into the cell holder part (28).

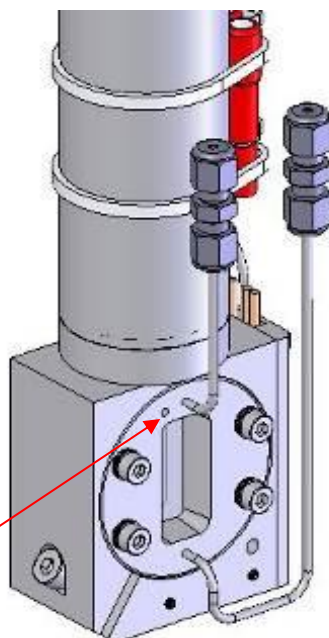
- 4.1. Lightly smear the outside of the sample cell with silicone grease. This will ensure good thermal conductivity when clamped in the cell holder part (28).
- 4.2. Loosen the M4 x 16mm cap head clamp screw (30) of the cell holder part (28) using the 3mm Allen key supplied until the circular hole of the cell holder aperture is large enough to accommodate the sample cell.
- 4.3. The cell holder part (28) will only accept sample cells from one direction. Inside the hole there is a circular flange ring (31) which prevents the sample cell from passing all the way through. Take the sample cell and push it into the cell holder aperture as far as

it will go. Ensure that the front plate of the **liquid sample cells** carrying the monitoring thermocouple well hole (34) is facing out of the aperture hole, i.e. this front plate part of the cell **is not** in contact with the circular flange ring (31). (See **Figs 10A, B, C, D**). The **solids holder P/N GS20610** is different to the liquid cells in that it is inserted into the cell holder part (28) aperture with its monitoring thermocouple well hole connection (34) emerging the same side as the flange ring (31) of the cell holder part (28). (See **Fig 10E**.)

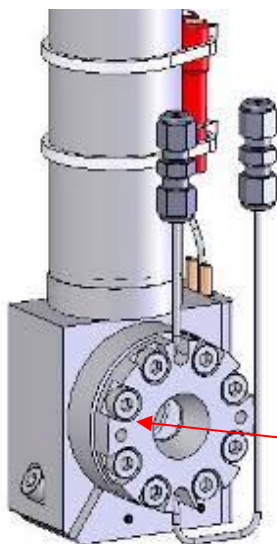
- 4.4** Tighten the clamp screw (30) until the sample cell is firmly held in place and a good thermal contact is made with the cell holder.



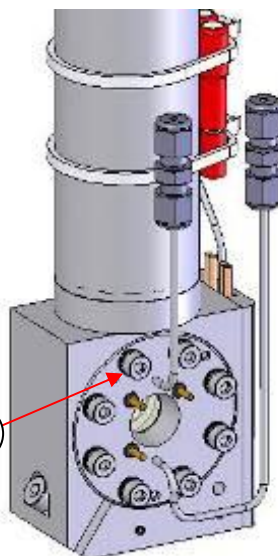
**Fig 10A. GS20500 Series
Liquid Cell Installed**



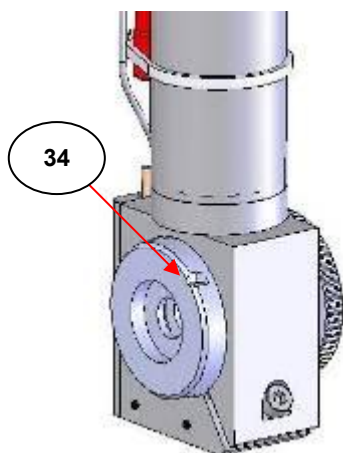
**Fig 10B. GS20560 Series
Flow Liquid Cell Installed**



**Fig 10C. GS05910 Series
High Pressure Liquid
Cell Installed**



**Fig 10D. GS20900 Series
Spectroelectrochemical
Cell Installed**



Important: When using P/N GS20500 or GS20560 Series cells the sample cell should be inserted in the VT Cell holder part (28) such that the aperture of the window (longest side) is vertical. For all the cells the thermocouple well hole (34) should be uppermost in the cell holder such that the beam from a spectrometer is not obstructed if an optional/spare monitoring thermocouple (49) is used (See Section 8, Page 37).

Fig 10E. GS20610 Solids Holder Installed

Installation of Liquid Flow Sample Cells into the VT Dewar/Cell/Cuvette Holder Assembly

If using a **flow** liquid type cell in the VT Cell Holder P/N GS21525, or a **flow** cuvette in the VT Cuvette Holder P/N GS21530, then the VT Cell Flow Fittings Kit P/N GS20080 will need to be fitted to the top pressure plate port positions (20) and (21).

The flow liquid type cells that can be installed in VT Cell Holder P/N GS21525 and require use of the VT Cell Flow Fittings Kit are:

- 1) P/N GS20560/GS20590 Series heatable, sealed and demountable, **flow** liquid cells.
- 2) P/N GS05910 Series high pressure, heatable, sealed, **flow** liquid cells.
- 3) P/N GS20900 Series heatable, demountable, **flow** Spectroelectrochemical liquid cells.

Fitting of the VT Cell Flow Fittings Kit P/N GS20080

Please see **Fig 11.** to identify the parts provided from the VT Cell Flow Fittings Kit P/N GS20080.

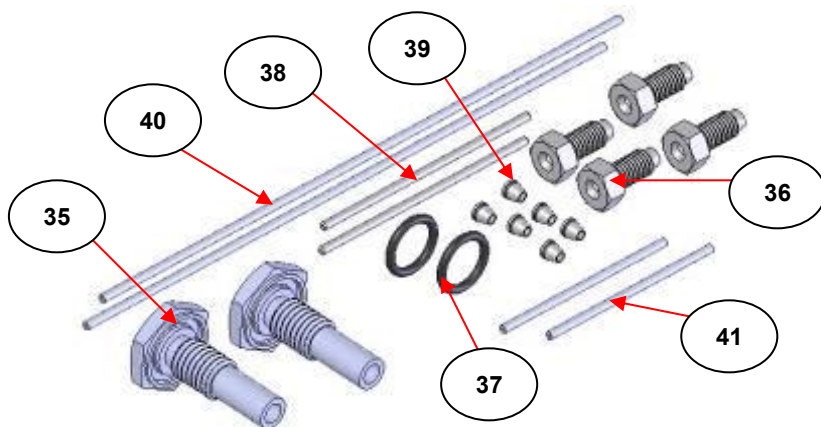


Fig 11. Parts of VT Cell Flow Fittings Kit GS20080

The contents of the VT Cell Fittings Kit are described as follows:

Part **(35)** is a zero dead volume fitting. Two are supplied in the kit.

Part **(36)** is a flow fitting screw nut. Four are supplied in the kit.

Part **(37)** is an O-ring seal for the zero dead volume fitting. Two are supplied in the kit.

Part **(38)** is a 50mm long stainless-steel blanking rod. Two are supplied in the kit.

Part **(39)** is an olive and ferrule set for 1/16" O.D. tubing. Six sets are supplied in the kit.

Part **(40)** is a 130mm long stainless steel 1/16" O.D. flow tube. Two are supplied in the kit.

Part **(41)** is a 40mm long stainless steel 1/16" O.D. flow tube. Two are supplied in the kit.

Included in the kit, but not shown, is a length of PTFE tubing, a 9/16" and 5/8" open ended spanner and two 1/4" and 5/16" open ended spanners (wrenches).

To fit the VT Cell Fittings Kit of parts the blanking plugs at ports **(20)** and **(21)** are removed using the 5/8" spanner. (See **Fig 12.**)

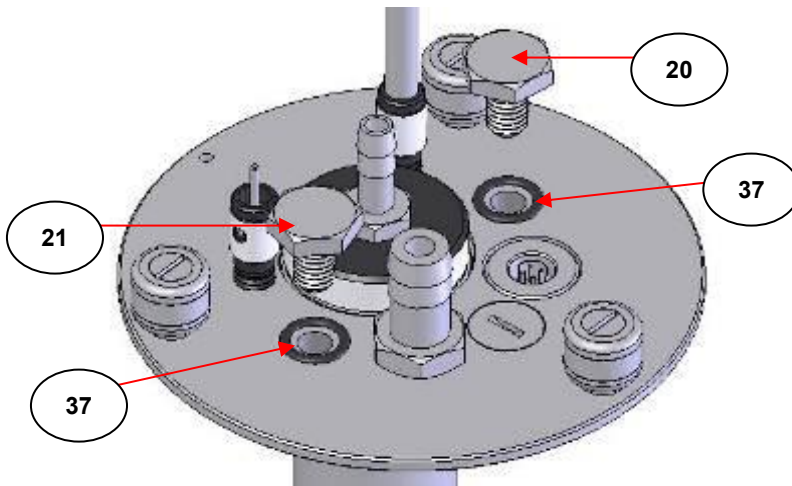


Fig 12. Removal of Blanking Plugs for VT Cell Flow Fitting Kit Use

To fit the VT Cell fittings from the GS20080 kit (as shown in **Fig 11.**) the replacement zero dead volume fittings (**35**) are screwed into the port positions (**20**) and (**21**) onto the top plate via their hexagonal nut head using the same 5/8" spanner. One of the black O-rings supplied (**37** - there are two in the GS20080 kit) is placed between the zero dead volume fitting (**35**) and the top plate to create a vacuum tight seal when the zero dead volume fitting is screwed into position.

Connection of Flow Tubes to the Zero Dead Volume Fittings

When the zero dead volume fittings (**35**) have been fitted to the top plate port positions (**20**) and (**21**), the 1/16" O.D. flow tubes (**40**) and (**41**) can be fitted to allow a liquid to be flowed from the external environment to and from a flow liquid type cell held in the VT Cell holder part (**28**). The 150mm long flow tubes (**40**) are fitted to the underside of the zero dead volume fittings (**35**) to project down for connection to the 1/16" Swagelok™ union fittings on any flow liquid cell installed in the VT Cell holder part area (**28**). The 40mm long flow tubes (**41**) are fitted to the topside of the zero dead volume fittings (**35**) to connect to any other 1/16" O.D. tubing or the PTFE tubing supplied in the kit, for introduction and return of a liquid from/to the external environment.

To seal the flow tubes (**40**) and (**41**) into position in the zero dead volume fitting (**35**), the olive and ferrule sets (**39**) are used.

Take one of the 150mm long tubes (**40**) and pass it through an olive and ferrule set (**39**) and a flow fitting screw nut (**36**) in the direction and sequence of parts as shown from **Fig 13.** such that the ferrule set and flow fitting screw nut assembly is about 10mm along from one end of the tube (**40**).

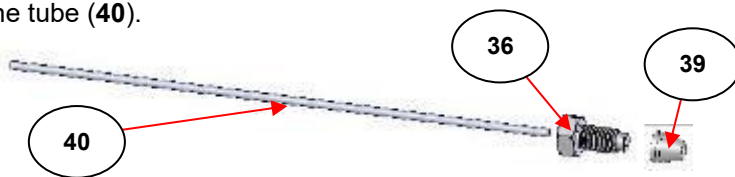
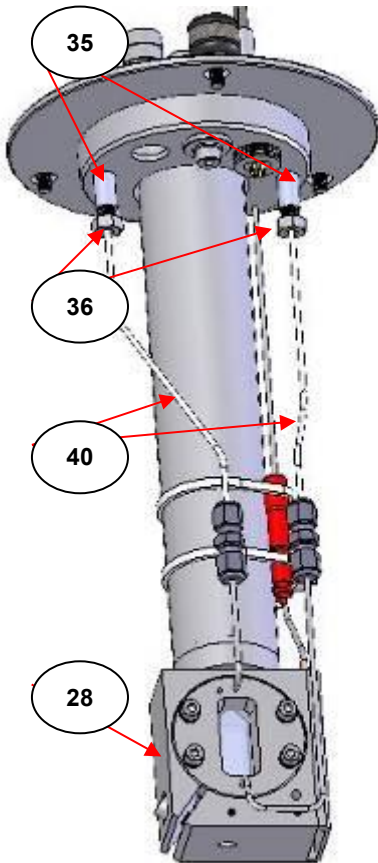


Fig 13. Flow Tube, Flow Fitting Screw Nut and Ferrule Assembly

Then insert this flow tube/flow fitting screw nut/ferrule set assembly into the aperture of the zero dead volume fitting (35) on the underside of the top pressure plate at port position (20), ensuring that the "V" point of the ferrule is pointing into the zero dead volume fitting (35). Screw tighten the flow fitting screw (36) into the zero dead volume fitting (35) using the 5/16" spanner supplied, thus trapping the ferrule set (39) and the flow tube (40). As the screw nut (36) is tightened the ferrule set is sealed/clamped permanently to the flow tube (40).



Repeat this procedure for fitting the other 150mm long flow tube (40) to the zero dead volume fitting (35) on the underside of the top pressure plate at port position (21). Then repeat this procedure for fitting the two 40mm long tubes (41) to the zero dead volume fittings (35) on the top side of the top pressure plate at port positions (20) and (21).

The 150mm long tubes (40) that have now been fitted and are projecting downwards can now be connected to the 1/16" Swagelok™ unions of any flow liquid cell held in the VT Cell holder part (28) by use and sealing of the olive and ferrule sets provided with the Swagelok™ unions on the flow liquid cell. The finished assembly of parts should look similar to **Fig 14**, which shows an example of a P/N GS20560 or GS20590 Series flow liquid cell connected to the fitted kit of parts as seen from the underside of the top pressure plate.

Fig 14. GS20560 Series Flow Cell Connected to GS20080 Flow Kit Fitting a Blanking Rod at the VT Cell Port Positions (20) and (21)

If you wish to install the VT Cell Flow Fittings Kit but want to operate the VT Cell without a flow cell, there are two stainless steel blanking rods (38) and some olive and ferrule sets (39) supplied with the GS20080 kit that are used to seal the VT Cell jacket for vacuum operation. The rods (38) are passed through the zero dead volume fittings (35) that have been attached.

Similar to the procedure for fitting of a flow tube as described previously (see **Fig 13.**), take a blanking rod (38) and pass it through a flow fitting screw nut (36) and an olive and ferrule set (39) such that the ferrule set is about halfway along the rod. Then insert this assembly into the top side aperture of the zero dead volume fitting (35) ensuring that the 'V' point of the ferrule is pointing into the fitting. Screw tighten the flow fitting screw nut (36) into the top of the dead volume fitting, thus trapping the ferrule set (39) and the blanking rod (38). The 5/16" spanner supplied is used to tighten the flow fitting screw nut (36) into the zero dead volume fitting (35). As the screw nut (36) is tightened the ferrule set (39) is sealed/clamped permanently to the blanking rod and overall the flow fitting becomes sealed for vacuum operation of the VT Cell Accessory.

The previous operation for fitting of a blanking rod (38) needs to be done for both flow through port positions (20) and (21) on the top pressure plate of the VT Cell Accessory. When a blanking rod (38) is fitted as described to seal the VT Cell Accessory at these flow port positions, it is not necessary to fit the two further flow fitting screw nuts (36) and ferrule sets (39) to the underside of the zero dead volume fittings (35).

6. Installation of the VT Cell into a Spectrometer

First, assemble all the components of the VT Cell Accessory.

- **Place windows into the vacuum jacket assembly (See Section 3, pages 10 to 12).**
- **Install a sample cell into the refrigerant dewar/cell holder assembly chamber (See Section 4).**
- **Place the dewar/cell holder assembly (with a sample cell) into the vacuum jacket assembly.**

This is done as follows:

- 5.1.** Check the silicone O-ring (**42**) that seals between the dewar/cell holder assembly (from P/N GS21525) or dewar/cuvette holder assembly (of P/N GS21530) and the vacuum jacket assembly. (See Fig 15.) Clean it and check the groove (**43**) in the vacuum jacket assembly for foreign matter. Lightly smear silicone/vacuum grease on the O-ring (**42**) and replace in its retaining groove (**43**).

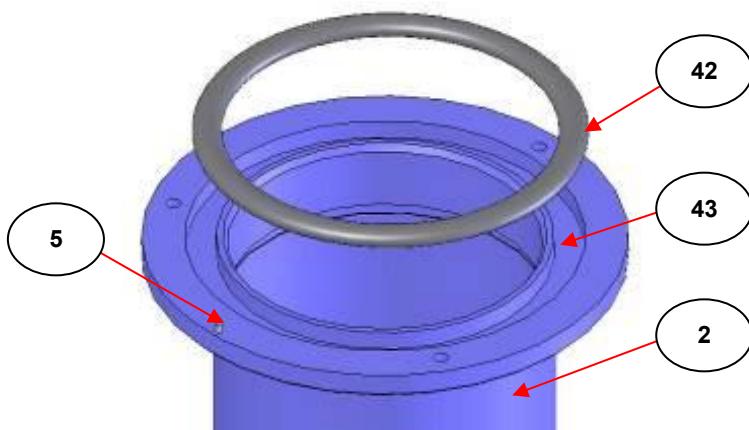


Fig 15. Vacuum Jacket Assembly Silicone O-ring for Sealing

- 5.2.** Insert the dewar/cell holder assembly into the vacuum jacket assembly by aligning the locating slot hole (3) in the pressure top plate with the locating pin (5) on the vacuum jacket assembly jacket (2). (See details on page 8.)

Note: *Ensure that any flow tubes/connections to the sample cells are not trapped and are clear of the inner sides/wall of the vacuum jacket assembly when inserting the dewar/cell holder assembly.*

- 5.3.** Screw down the three fixing thumb screws (4) on the top plate to tighten to the vacuum jacket assembly (2). Tighten the screws evenly and sufficiently in rotation to give a vacuum tight seal all around the surface interfaces between the Silicone O-ring (42).

Mount the VT Cell Accessory into a Spectrometer

This is done as follows:

- 5.4.** Securely mount the whole VT Cell Accessory by use of an appropriate Benchmark™ baseplate or by use of the affixed 3" x 2" slide mount plate (7). (See pages 8 to 10.)

Note: *The Solids Holder P/N GS20610 has been designed such that the sample position for a solid is as close to the 3" x 2" backplate (7) as possible. In spectrometers whereby the instruments own 3" x 2" mount baseplate is movable, a slight adjustment in the position of the mount baseplate can bring the sample directly into focus with an improved performance.*

Use of the Benchmark™ baseplate as a means of installation provides a more stable support for the whole VT Cell Accessory and as such is recommended in preference to the 3" x 2" slide mount (7) option, if a Benchmark™ baseplate can be used.

Instructions for installation of a specific type of Benchmark™ baseplate into different spectrometers systems is found in the Benchmark™ baseplate instruction manual which has been supplied along with this accessory. Please consult this manual for your spectrometer system.

Make service connections to the VT Cell. (From controller, vacuum lines, liquid flow lines etc.)

5.5. Connect the 6-way plug cable assembly to the back of the 4000 Series™ controller. Connect the larger 4 pin Fischer latching plug to the cell holder heaters electrical connection port (**17**) on the pressure top plate (see **Fig 6**, page 13), and the smaller 4 pin Fischer plug to the vacuum jacket window heater electrical connection (**16**) – (see **Fig 5**, page 11.). (The vacuum jacket window heaters may have already had to be connected and switched on when installing hygroscopic windows into the vacuum jacket).

5.6. Connect a vacuum line (pump and tubing) to the vacuum port (**19**) on the top plate. (See **Fig 6**, page 13.) Switch on the vacuum supply.

Note: *The vacuum line may need independent supporting such that its weight does not move the VT Cell Accessory if the 3" x 2" slide mount plate (7) has been used for installation.*

5.7. Connect the 2 pin Fischer plug of the control thermocouple (**25**), that passes through port (**18**), to the connection in the back of the 4000 Series™ controller. (See **Fig 6**, page 13).

5.8. Connect liquid flow lines to the top of the flow ports (**20** – inlet) and (**21** - outlet) if using a liquid sample flow cell (P/N GS20560 or GS20590 Series). The Flow Fittings Kit P/N GS20080 **must** already be installed to do so.

For temperature operation and control of the VT Cell Accessory, the instructions from the instruction manual for the 4000 Series™ temperature controller should now be followed.

Temperatures to the sample cells are established by use of specific refrigerants where necessary for sub ambient conditions and/or the cell holder heaters (**26**). The next section of this instruction manual explains about the refrigerant dewar chamber part of the VT Cell Accessory.

7. Principles of the Refrigerant Chamber

The dewar/cell holder assembly (1) of the VT Cell Accessory is a single piece assembly. The integrated design allows for precise control of temperature to the sample cells. The dewar (or refrigerant chamber) is used to contain various refrigerants for maintaining sub ambient temperature levels to liquid or solid samples when they are installed within the VT Cell Accessory in their specific sample cell holder.

The refrigerant chamber is equipped with two essential component parts; a bung (44) and a “mushroom” piston assembly (45). The bung is a vented stopper, the vent being a metal “hose type” tube connector. The mushroom piston is a rod with a circular metal base, the underside being covered with a polyurethane foam disc (46). There are two mushrooms with different sizes of foam disc, a large diameter of 33mm and a smaller diameter of 30mm. (See Fig 16.)

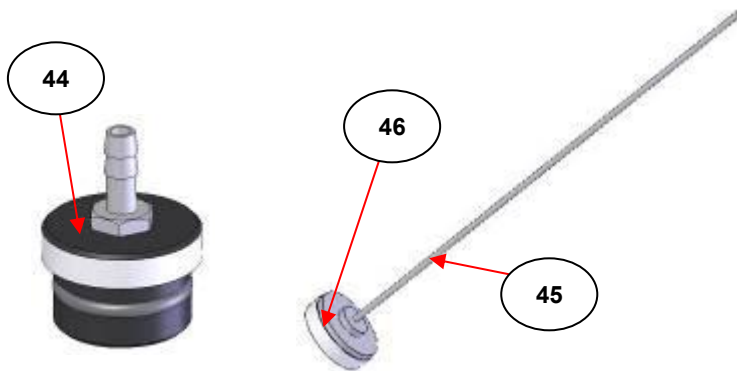


Fig 16. VT Cell Bung and Mushroom Piston Assembly

When any sub ambient temperature is required the bung and “mushroom” piston are used to control and contain the refrigerant within the dewar. A sample temperature between -190°C and sub ambient can be achieved by a combination of the correct refrigerant in the dewar (liquid nitrogen), correct choice of mushroom (45) with bung (44) and power from the controller to the cell holder heaters (26).

For samples to be analyzed at a temperature which is higher than the natural freezing point temperature of the refrigerant, the mushroom (45) is used so that its polyurethane base acts as a partial thermal barrier between the refrigerant in the dewar (24) and the sample cell holder in the sample holder part (28) of the VT Cell Accessory. The sample cell holder may need to reach thermal equilibrium at a temperature considerably higher than that of the refrigerant. The heaters in the cell holder (26) are used to provide higher temperatures to the sample in its holder to compensate (balance) for the lowering of the temperature provided by the refrigerant. However, the mushroom (45) and foam base (46) acts to greatly reduce the rate at which the refrigerant is consumed by heating from the heaters. The heating effect for temperature control is directed preferentially towards the sample itself and allows for a longer lifetime of refrigerant.

The bung (44) aids this overall temperature control by preventing air of ambient temperature from heating a lower temperature refrigerant in the dewar. However, the bung (44) allows some of the refrigerant vapour to boil off through the vent tube to prevent buildup of pressure within the dewar (24). The bung (44) and mushroom (45) are vital for optimum working efficiency of the dewar. When the mushroom (45) is required in operation, the larger diameter (33mm) foam mushroom operates more efficiently between -100°C to ambient. The smaller diameter (30mm) foam is better between -190°C to -80°C.

The base of the dewar (refrigerant chamber 24) is slightly constricted just before the joint with the cell holder part (28) creating a small gap. The constriction prevents the mushroom foam (46) from making intimate contact with the bottom of the dewar during operation. It is therefore recommended that before the mushroom (45) is used this small gap is filled with the stainless steel “thermal block” disc (47) supplied. This can be done by loosely screwing the stainless-steel rod (48) into the disc (47) and gently lowering it down inside the dewar until it reaches the bottom. Unscrew the rod from the disc and remove the rod. (See Fig 17.)

If a sample temperature required is close to that of a refrigerant the bung (44) is used **without** a mushroom (45). Output power from the controller can also be reduced.

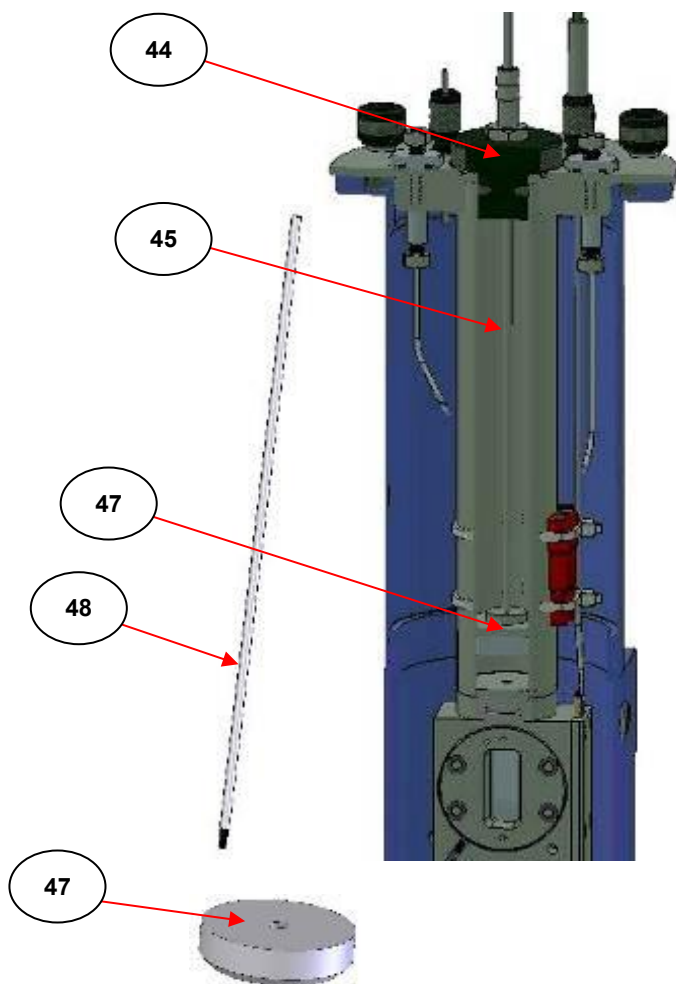


Fig 17. Thermal Block Disc and Rod and Cutaway (Section) View to show arrangement of Thermal Block Disc, Mushroom and Bung positions when in the VT Cell Dewar Cell Holder Assembly

Refrigerants for the Dewar/Cell Holder Assembly

The following table (Table I) is a list of refrigerants that can be used within the dewar of the VT Cell Accessories P/N GS21525 and GS21530.

Specac cannot be held responsible for misuse of the types of refrigerants and refrigerant mixtures recommended for operation, this includes and is not limited to any test sample type being analyzed in the liquid or solids cell fitted within the VT accessory.

Table I

Refrigerant	Mixed Temperature (a)		Hazard
	°C	°K	
Ice Water	0	273	
Sodium Chloride/Ice (33g salt/81g ice)	-21	252	
Calcium Chloride/Ice (100g salt/81g ice)	-40	233	
Toluene/Liquid Nitrogen (slush)	-95	178	
Liquid Air (21% oxygen)	-147	126	
Liquid Nitrogen	-196	77	Asphyxiation

(a) These figures are approximate as actual mixture temperatures are affected by impurities.

It is not advised to use flammable refrigerants, but other refrigerants can be used. It is essential that data sheets are checked, and all safety measures are followed by the operator. Specac cannot be held responsible for the types of refrigerants used.

You should choose the refrigerant mix closest to the temperature you wish to study. Raise the temperature to the target temperature using the bung (44) with large mushroom (45) in the dewar and the temperature controller providing heat to the cell holder.

Note: Although the range of the VT Cell is -190°C to $+250^{\circ}\text{C}$ it is not possible to carry out a controlled rate of temperature rise that passes through the ambient point. This is because the rate of refrigerant loss to allow the temperature to go from sub ambient to above ambient cannot be controlled accurately enough to synchronize with the rate of heating supplied by the cell holder heaters (24). Therefore, temperature rate rise experiments can only be done within a sub ambient or above ambient range.

Operation of the Dewar (Refrigerant Chamber)



CAUTION! Risk of Frostbite, Asphyxiation and Explosion. When using refrigerants with this accessory, wear appropriate safety clothing (PPE) and always follow local laboratory safety practices and procedures.



CAUTION! DO NOT USE test samples that are potentially explosive under varying temperature conditions in this device.

After installation and connection of the services to the VT Cell Accessory in a spectrometer (see section 5.1 to 5.8.) the dewar (24) chamber can be filled with a suitable refrigerant.

IMPORTANT: It is essential that the VT Cell is evacuated to better than 0.05 Torr (0.067mbar) before filling the refrigerant chamber with refrigerant. If a good vacuum is not achieved before refrigerating, water vapor from the atmosphere would crystallize on the sample cell holder and windows. **N.B.** The vacuum **must** be maintained even when the VT Cell Accessory is used at temperatures above ambient for most efficient temperature control to the sample.

Place the appropriate mushroom (45) into the refrigerant chamber and then fill with the choice of refrigerant. The bung (44) is put in position with the rod of the piston mushroom (45) running up inside the metal vent tube of the bung (44). Depending on the experiment time it may

be necessary to keep the refrigerant level topped up. This is done by removing the bung (44) to gain access to the dewar (24).

An additional length of rubber tubing should be attached to the bung vent tube and vented safely, such that any refrigerant boils off away from the top plate of the refrigerant chamber and the operator. The rubber tube will also prevent icing up of the bung's metal vent tube. Also, when using liquid nitrogen as the refrigerant, a plastic funnel helps to direct the refrigerant into the dewar only. If the top plate area becomes excessively cold and freezes the sealing silicone O-ring (42) will become rigid and vacuum integrity of the VT Cell could be lost.

The following table (Table II) gives the most efficient operating conditions of the VT Cell for various temperature ranges using a mushroom and the refrigerant. In general, when only a small temperature rise above that provided by the refrigerant is required, it is advantageous to use the controller at reduced power. From the instructions found in the 4000 Series™ Controller manual, the parameter OPuL under the SetP parameter list must be set to the appropriate power level as shown in the table for controller power.

Most Efficient Operation (Table II)

Temp. Range (°C)	Mushroom	Refrigerant	Controller Power
Ambient to + 250	None	None	20 - 100%
- 100 to + 30	Large (with bung)*	Liquid N2	20%
- 190 to - 80	Small (with bung)*	Liquid N2	20%
- 196 to - 180	None	Liquid N2	10%

**And Thermal Block Disc (47).*

IMPORTANT: At temperatures above ambient the mushroom (45) **must** be removed. Leaving the mushroom in the dewar (24) will cause damage to the polyurethane foam base (46).

8. VT Cell Operating Parameters on the Dedicated 4000 Series™ Controller

The VT Cell Accessories P/N's GS21525 and GS21530 are provided with their own dedicated 4000 Series™ Temperature Controller. The 4000 Series™ Temperature Controller provides heating of a sample cell holder within the VT Cell Accessory. A separate instruction manual is supplied for explanation in operation of the 4000 Series™ Temperature Controller in conjunction with the VT Cell Accessory.

For operation of the VT Cell Accessory 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.

Parameter Settings on a 4000 Series™ Controller for Operation of the VT Cell Accessories P/N's GS21525 and GS21530

Parameter Display (In Green)	Parameter Name	Parameter Factory Set Value
FiLt	Input Filter Time Constant	2.0
OFFS	Process Variable Offset	0
PP┐┐	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	1.8
ArSt	Automatic Reset (Integral Time Constant)	1.32
rAtE	Rate (Derivative Time Constant)	0.23
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	250
SPLl	Setpoint Lower Limit	-190

OPuL	Primary (Heat) Output Upper Power Limit	100
Ct I	Output 1 Cycle Time	8
PhAl	Process High Alarm	250
AHy1	Alarm 1 Hysteresis	1
PLA2	Process Low Alarm	-190
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	600
SP	SP Value	250
SLoc	Set-up Lock Code	10

9. Direct Temperature Monitoring from the Sample Cell Holder in GS21525

The standard system controlling thermocouple (**25**) supplied and fitted measures the temperature of the cell holder area (**28**) and regulates power to the heaters (**24**) for precise control of the temperature to the system. After time, the temperature of the whole area will reach stability and it is assumed that the sample temperature will be the same as it local surroundings.

However, it is possible to monitor the temperature of the sample cell directly by insertion of an **additional** monitoring thermocouple P/N GS20200 (**49**) into the well hole (**34**) of any of the sample cell holder types that have been installed in the VT Cell holder area (**28**). This independent monitoring thermocouple (**49**) is used to measure the actual temperature of the sample, if this is needed to be known, as the position of this thermocouple (**49**) can be located closer to the actual sample surface itself. The monitoring thermocouple (**49**) is fitted with a line connector plug (two flat/spade type terminal pins) which can be attached to an independent monitoring readout.



Fig 18. Monitoring Thermocouple GS20200 for VT Cell Accessory

An additional monitoring thermocouple (49) can be fitted is as follows:

- 8.1. Remove the stainless steel blanking rod (27) from the port position (22) on the circular top plate of the dewar/cell holder assembly (1). The rod (27) is held into the screw nut fitting (50) by a grub screw (51). Unscrew the screw nut fitting (50) from the top plate using the knurled surface and the rod (27) with its own ferrule (52) and sealing O-ring (53) will be removed from the port position (22). See Figs 19. and 20.

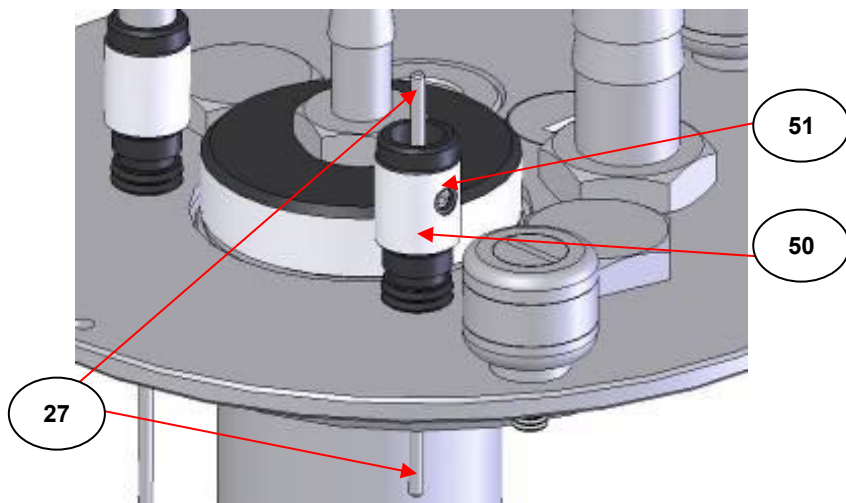


Fig 19. Screw Nut and Blanking Rod at Monitoring Thermocouple Port Position on the VT Cell Accessory



Fig 20. Screw Nut and Blanking Rod Fitting when removed from its Port Position on the VT Cell Accessory

- 8.2** The blanking rod (27) will need to be removed from the assembly seen in Fig 20. and is to be replaced with the monitoring thermocouple (49). Loosen the M3 x 3mm grub screw (51) using an appropriate Allen key, such that the rod (27), ferrule (52) and O-ring (53) can be slid out of the screw nut (50).
- 8.3.** Remove the O-ring (53) and ferrule (52) from the blanking rod (27) as these parts are to be reused. Carefully pass the tip of the monitoring thermocouple (49) through the screw nut (50), ferrule (52) and O-ring (53) in the same sequence that these components were used to secure the blanking rod (27). Ensure that there is at least a 200mm length of the thermocouple (49) projecting beyond the O-ring (53).
- 8.4.** Now carefully pass the tip of the thermocouple (49) from this assembly through the port position (22) until the O-ring (53) and ferrule (52) contact with the base of the port position access hole. Loosely secure/tighten the screw nut (50) into the port position (22), trapping the O-ring (53) and ferrule (52). The assembly needs to be loose to allow the thermocouple (49) to be slid through the screw nut components and adjusted for its length to fit into the thermocouple well hole (34) of the sample cell holder

being used.

8.5. With a specific sample cell holder fitted into position in the cell holder area (28), offer up the tip of the thermocouple (49) to the thermocouple well hole (34). If there is an insufficient length of thermocouple (49) to reach the well hole (34), then this length can be adjusted accordingly by sliding the thermocouple up or down through the loose screw nut (50) connection. Very carefully and gently bend the thermocouple (49) tip about 15mm from its end through an angle of 90° with a bend radius of no less than 15mm. (Gentle radius/curving of the bend is important to prevent fracturing the thermocouple sheath).

8.6. Place the tip of the thermocouple (49) into the well hole (34) of the sample cell holder, ensuring that it also enters at least to a 5 to 6mm depth. A small amount of silicon grease on the tip of the thermocouple (49) can give improved thermal contact.

Note: *When using the Solids Sample Cell P/N GS20610 it is not necessary to bend or radius the end tip of the thermocouple (49). The thermocouple tip is inserted into the well hole (34) on the edge of the solids holder. (See Fig 10E.)*

8.7. Straighten out and adjust the remaining length of the thermocouple (49) through the screw nut connection fitting at the port position (22) so there is no excess length below the top pressure plate. Tighten the screw nut (50) into the top plate to firmly compress the O-ring (53) and ferrule (52). Finally retighten the grub screw (51) to fully secure the thermocouple (49) into the screw nut (50).

Note: *Care must be taken not to over-tighten the screw nut (50) thus damaging the O-ring seal (53) by extruding it through the port hole (22) in the top plate. Also, when tightening, care should be taken not to damage the thermocouple (49) lead.*

10. VT Cell Legend

Listed below are the “bubble” number allocations for the various parts that have been described in this manual. This list can act as a guide for quick reference to the part within this instruction manual.

(1) Refrigerant Dewar/Cell Holder assembly	Page 12.
(2) Vacuum Jacket assembly	Page 12.
(3) Locating pin hole on Dewar assembly	Page 13.
(4) Fixing thumb screw on Dewar assembly	Page 13.
(5) Locating pin on Vacuum Jacket assembly	Page 13.
(6) Benchmark™ baseplate adapter plate fixing	Page 13.
(7) 3” x 2” slide mount plate	Page 14.
(8) Fixing screw for Benchmark™ adapter plate fixing	Page 14.
(9) Benchmark™ adapter plate location holes	Page 14.
(10) Benchmark™ adapter plate thumbscrew fixing	Page 14.
(11) Vacuum jacket window retaining ring	Page 16.
(12) Vacuum jacket window PTFE seal	Page 16.
(13) Vacuum jacket window elastomer O-ring seal	Page 16.
(14) Vacuum jacket window key	Page 16.
(15) Vacuum jacket window	Page 16.
(16) Vacuum jacket window heater connection port	Page 17.
(17) Dewar assembly cell heater connection port	Page 18.
(18) Dewar assembly controlling thermocouple port	Page 18.
(19) Dewar assembly vacuum connection port	Page 18.
(20) Dewar assembly liquid flow cell port (inlet)	Page 18.
(21) Dewar assembly liquid flow cell port (outlet)	Page 18.
(22) Dewar assembly additional thermocouple port	Page 18.
(23) Dewar assembly Spectroelectrochemical Cell port	Page 18.
(24) Dewar assembly refrigerant chamber access hole	Page 19.
(25) Controlling thermocouple	Page 19.
(26) Dewar assembly heaters	Page 19.
(27) Blanking rod for port position (22)	Page 21.
(28) VT Cell GS21525 cell holder part	Page 22.
(29) VT Cell GS21530 cuvette holder part	Page 22.
(30) VT Cell GS21525 cell holder part clamping screw	Page 22.
(31) VT Cell GS21525 cell holder part flange ring	Page 22.
(32) VT Cell GS21530 cuvette holder part clamp plate	Page 23.

(33) VT Cell GS21530 cuvette holder part fixing screw	Page 23.
(34) Sample cell holder thermocouple well hole	Page 24.
(35) Zero dead volume fitting	Page 27.
(36) Flow fitting screw nut	Page 27.
(37) O-ring for zero dead volume fitting	Page 27.
(38) Stainless steel blanking rod 50mm long	Page 27.
(39) Olive and ferrule set	Page 27.
(40) 1/16" O.D. Stainless steel tubing 130mm long	Page 27.
(41) 1/16" O.D. Stainless steel tubing 40mm long	Page 27.
(42) Vacuum Jacket assembly sealing O-ring	Page 31.
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(49) Monitoring thermocouple (P/N GS20200)	Page 42.
(50) Monitoring thermocouple screw nut fitting	Page 42.
(51) Grub screw for screw nut fitting (50)	Page 42.
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11. Specifications of the VT Cell Accessory

The VT Cell types P/N GS21525 and GS21530 have the following specifications.

Maximum temperature capability: +250°C.

Minimum temperature capability: -190°C (with liquid nitrogen LN₂).

Thermocouple: T type (Cu / CuNi).

Heating rate: 2 Port VT Cell typically, ambient to +250°C in 25 minutes. (Specified rise rate or fall of circa 10°C per minute maximum).

4 Port VT Cell typically, ambient to +250°C in 50 minutes. (Specified rise rate or fall of circa 10°C per minute maximum).

Pressure range (normal): Vacuum to 1.5 bar (maximum).

Fixing: 3" x 2" (76mm x 50mm) slide mount backplate or Benchmark™ baseplate installation.

Weight: 4.0Kg

Overall Dimensions when installed in spectrometer.

Length (side to side dimension): 120mm.

Width (back to front dimension): 130mm.

Height (base to top of bung): 300mm.

Vacuum Jacket inner diameter of cylinder: 70mm.

Dewar assembly volume: 125cc maximum.

VT Cell holder electrical specifications:

Rated voltage: 30VDC maximum.

Rated current: 3A peak

Operating ambient: 5-40°C (10-80% humidity, non-condensing).

Storage temperature: -20 to +60°C.

Altitude: Up to 2000m

Pollution Degree: 2

12. Spares and Consumables

Vacuum Jacket Assembly Windows for VT Cells

P/N GS20800	Pair of NaCl Windows
P/N GS20801	Pair of KBr Windows
P/N GS20802	Pair of CaF ₂ Windows
P/N GS20803	Pair of BaF ₂ Windows
P/N GS20896	Pair of ZnSe Windows
P/N GS20898	Pair of UV Quartz (Spectrosil B) Windows

Spares

P/N GS20810	Set of replacement O-Rings for VT Cells GS21525 and GS21530
P/N GS20200	Monitoring thermocouple (Cu/Constantan) for VT Cells GS21525 and GS21530.
P/N GS20080	VT Cell Flow Conversion Kit.
P/N GS21526	VT Cell Essential Spares Kit.

End of Lifetime Equipment Use



Warning: Handle with care! Environmental damage in the case of incorrect disposal.

Correct disposal will ensure environmental protection and prevent any potentially harmful impact on people or the environment.

Follow all relevant local requirements when disposing of the appliance and its accessories. If appropriate check SDS's for disposal information.



Within the European Community, national regulations are specified for the disposal of electronic appliances, based on EU Waste Electrical and Electronic Equipment Directive 2002/96/EC (WEEE). In accordance with this, the appliance cannot be disposed of with local or domestic waste.

The appliance will be accepted at local collection points or recycling centers.

Notes for use of your VT cell accessory

[illegible]

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