High Pressure Heatable Liquid Cells GS05910, GS05915, GS05920, GS05925 Series

User Manual

2I-05910 Issue 9

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1. Introduction

Thank you for purchasing a Specac product.

Specac has a range of High Pressure Liquid Cells designed for transmission spectroscopy measurements of liquids at temperatures covering a range from sub ambient up to 180°C. They are manufactured for high pressure applications and are provided at fixed pathlengths of 0.5, 1.0, 2.0, 5.0 and 10mm versions. Stepped windows are used in the 0.5, 1.0 and 2.0mm pathlength versions of cells, whereas plane windows are used in the 5.0 and 10.0mm pathlength cells.

All of the High Pressure Liquid Cell types are non-demountable. The windows and seals are fixed into position using end cap window assemblies with eight screw bolts that have been tightened to a specific torque. (See **Fig 1**). The screw bolt heads are filled with an adhesive to prevent them from being loosened, thus ensuring the pressure capability to be maintained.

The High Pressure Liquid Cells are rated for 2000psi (136bar) or 5000psi (340bar) pressures and are provided with the specified pressure certification. The standard window materials are Zinc Selenide (2000psi maximum pressures only), Sapphire and Spectrosil B (UV grade quartz), but a few other materials such as Silicon and Calcium Fluoride are offered on special request.

The High Pressure Liquid Cells can be used within Specac's own heating accessories, such that a temperature other than ambient can be applied in operation of the liquid cell. The compatible heating accessories are:

P/N GS21525 Variable Temperature (VT) Cell.
P/N GS20730 Electrical Heating Jacket.
P/N GS20710 Water Heating Jacket.

However, the High Pressure Liquid Cells can also be used for ambient temperature studies when mounted on their standard 3" x 2" slide

mount backplate which is provided with each liquid cell. The 3” x 2” slide mount backplate is fitted to the High Pressure Liquid Cell and is then used with an appropriate 3” x 2“ slide mount accepting baseplate provided with the spectrometer for positioning in the sample compartment.

For heated or sub-ambient applications, the High Pressure Liquid Cell is located inside the VT Cell GS21525 (for temperatures from – 15°C to 180°C), the Electrical Heating Jacket GS20730 or the Water Heating Jacket GS20710 (for temperatures from ambient to 180°C). The 3” x 2” slide mount backplate is removed from the High Pressure Liquid Cell to allow for installation into P/N’s GS21525, GS20730 and GS20710.

Standard 1/16" O.D. stainless steel flow tubes are brazed to the High Pressure Liquid Cell body to give leak-proof inlet and outlet ports. These flow tubes open out into the centre of the High Pressure Liquid Cell where the cell is filled with fluid. External connection to these 1/16” flow tubes can be made via Swagelok couplings, which are supplied.

Important: *Read Section 3 – Safety In Use - before using the High Pressure Liquid Cell.*

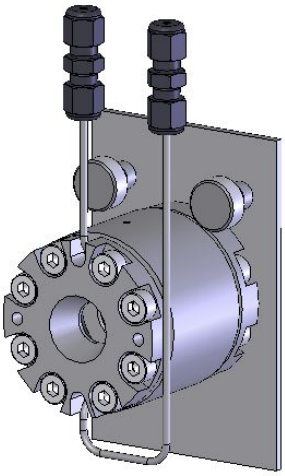


Fig 1. High Pressure Liquid Cell

2. Unpacking and Checklist

The High Pressure Liquid Cell is supplied in a carry case with the following parts:

- 1 High Pressure Liquid Cell assembly including flow tubes, pressure certified window assemblies and set at fixed pathlength of choice.
- 1 High Pressure Liquid Cell 3” x 2” slide mount backplate.
- 1 Front Cover Plate with 2 thumb screws (for use with Electrical Heating Jacket P/N GS20730).
- 2 Open Ended Spanners.
- 1 Certificate of Conformity.
- 1 Instruction manual for High Pressure Liquid Cells

Please check that all the parts are present.

The High Pressure Liquid Cell is ready for connection to a particular liquid supply and installation into a spectrometer or appropriate Specac heating accessory.

3. Safety In Use

Warnings!

1) The High Pressure Liquid Cell has been pressure tested and certified. Do not dismantle the cell or remove the windows from the cell.
2) Do not operate the High Pressure Liquid Cell below -15°C or above 180°C. Temperatures beyond this range may cause permanent damage to the Viton seals and render the cell dangerous. Ensure that the solvent/sample used is compatible with the Viton seals and window material fitted.
3) Do not operate the High Pressure Liquid Cell above the rated pressure: 2000psi (136bar) or 5000psi (340bar) which is engraved on the body of the cell **(see note on Hazardous Samples**).
4) The tubing for liquid introduction is brazed into the High Pressure Liquid Cell body. Take extra care whenever bending the tubes such that the brazing is not damaged.
5) Ensure when connecting the flow tubes via the Swagelok unions to sample stream pipework, that two spanners are used. One spanner is used to hold securely the Swagelok union nut fixed to the tube connected to the cell body and the other spanner is used to tighten or loosen the Swagelok nut to make or break the connection. This will ensure that the brazed tubes are not damaged unnecessarily at their brazing joints to the cell body.
6) When using the High Pressure Liquid Cell in heating and cooling applications, do not set the heating or cooling rate on the heating accessory too high. High rates may cause thermal shock to some windows and may cause breakage. A cooling or heating rate of 10°C per minute or less is generally recommended.
7) Visually inspect the High Pressure Liquid Cell for damage each time before use.

Note: *If there is any sign of damage to the windows, tubes or body of the High Pressure Liquid Cell,* ***DO NOT USE.*** *Contact Specac or your local representative for the cell to be returned for repair.*

Hazardous Samples

The following variants of High Pressure Liquid Cell must not be used above 2914 psi a (200 bar g) when using a hazardous sample:

P/N GS05915 - all pathlengths.
P/N GS05925 - all pathlengths.

Definition of a Hazardous Sample

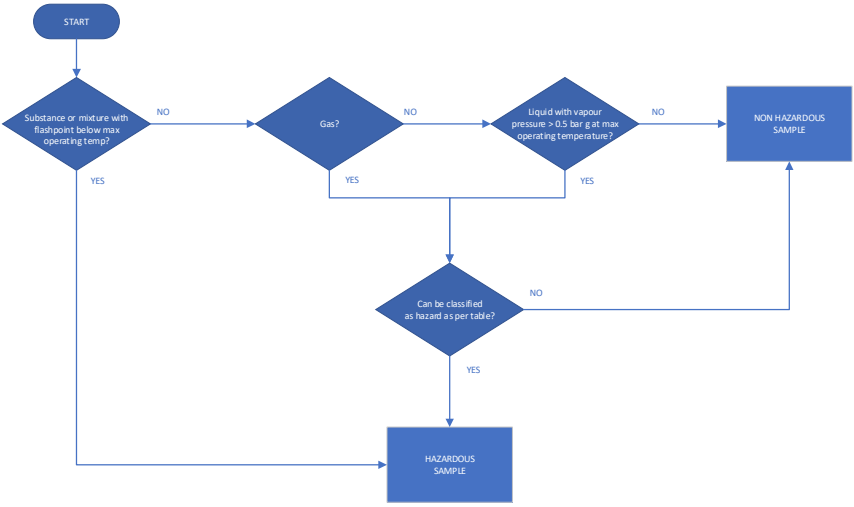
A hazardous sample is defined as a gas (or liquid with a vapour pressure > 0.5 bar g at maximum operating temperature), with a hazard classification shown in the table below or any substance or mixture whose flashpoint is below maximum operating temperature.

The following table defines hazard classification as per Regulation (EC) No 1272/2008. (Applicable Hazard Statement Codes given for reference.)

Hazard Class and Category	Hazard Statement Codes
Unstable explosives or explosives of divisions 1.1, 1.2, 1.3, 1.4 and 1.5	H200, H201, H203, H204, H205
Flammable gases, category 1 and 2	H220, H221
Oxidising gases, category 1	H270
Flammable liquids, category 1, 2 and 3*	H224, H225, H226*
Flammable solids category 1 and 2	H228
Self-reactive substances and mixtures type A to F, or organic peroxides type A to F	H240, H241, H242
Pyrophoric liquids or solids, category 1	H250
Substances and mixtures which in contact with water emit flammable gases, category 1, 2 and 3	H260, H261
Oxidising liquids or solids, category 1, 2 and 3	H271, H272
Acute oral toxicity, category 1 and 2	H300
Acute dermal toxicity, category 1 and 2	H310

* Where flashpoint is below max operating temperature.

The following flow chart can help to identify whether a sample is defined as hazardous, in relation to the hazard classification table.



4. Using the High Pressure Liquid Cell

The High Pressure Liquid Cell can be connected to a high pressure liquid pumping system to introduce a liquid sample under pressure into the cells inner chamber, created from the pathlength of the body between the inner surfaces of the windows. The liquid system can be operated in a flow mode or the outlet flow tube can be shut off from an inline valve (not supplied) whilst pumping, to allow for a pressure to build up within the High Pressure Liquid Cell.

Ambient Temperature Use

When using the High Pressure Liquid Cell at ambient temperatures the following procedure can be used.

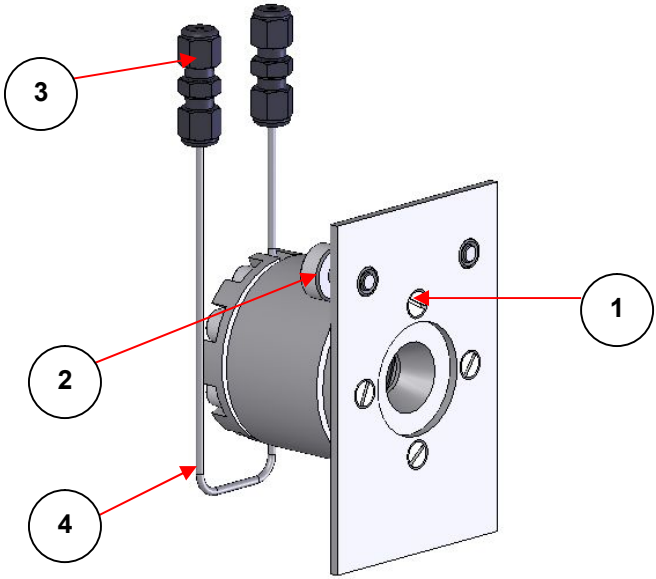


Fig 2. Fitting of the 3” x 2” slide mount backplate to the High Pressure Liquid Cell

1. Secure the 3" x 2" slide mount backplate to the back of the High Pressure Liquid Cell body using the four cross head screws (supplied) in the fixing holes (**1**). (See **Fig 2**). Ensure the slide mount backplate is fixed to the cell body with the two thumbscrew fittings (**2**) being at the top as seen in Fig 2..

2. Slide the 3" x 2" back plate now fixed to the High Pressure Liquid Cell into the 3" x 2" mount plate of the spectrometer and secure using the two thumbscrews (**2**).

3. Connect the High Pressure Liquid Cell to the sample flow stream pipework via the 1/16" O.D Swagelok connectors (**3**).

Tip: *It is better to use the lower of the two tubes brazed to the High Pressure Liquid Cell body as the inlet port (**4**).*

4. Fill the High Pressure Liquid Cell with the solvent and record the background spectrum.

5. Remove the background sample and fill the High Pressure Liquid Cell with sample and record the sample spectrum.

Heated Applications in the Variable Temperature Cell GS21525

The High Pressure Heated Liquid Cell can be inserted into the Variable Temperature (VT) Cell GS21525 in the same way as the standard GS20500 or GS20510 heatable liquid cells used within this accessory. Please see the VT Cell instruction manual for installation and operation for flow conditions of the High Pressure Liquid Cells in this accessory.

Warning: *Do not exceed -15°C and 180°C temperatures when using the High Pressure Liquid Cell in the VT Cell. Exercise extreme care if it is a requirement to bend the liquid flow tubes for the High Pressure Liquid Cell to fit in the VT Cell, so that the brazed joints to the cell body are not damaged.*

Heated Applications in the Electrical Heating Jacket GS20730

1. Insert the High Pressure Liquid Cell (after removing its own 3” x 2” slide mount backplate) in the Electrical Heating Jacket. (Please refer to the instruction manual for the Electrical Heating Jacket GS20730 to explain the procedure.)

2. Replace the standard front cover plate of the Electrical Heating Jacket with the front cover plate (**5**) supplied with the High Pressure Heatable Liquid Cell (Fig 3) and secure using the thumb screws (**6**) supplied with the High Pressure Liquid Cell (see **Fig 4**) in place of the standard “quick-release” thumbscrews as supplied with the Electrical Heating Jacket.

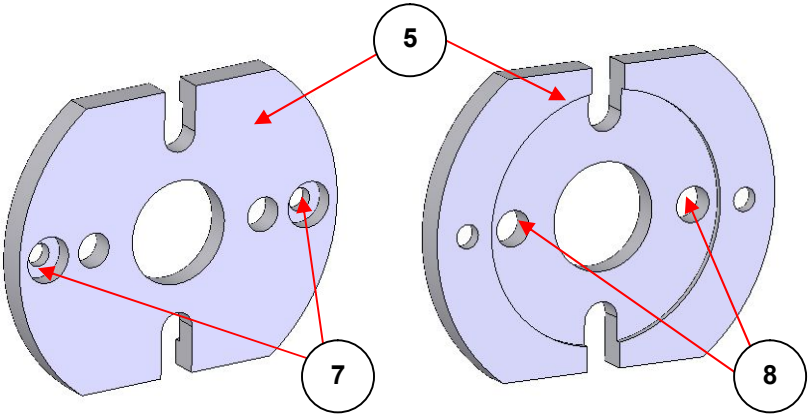


Fig 3. Front cover plate (both sides seen) to hold High Pressure Liquid Cell when placed in the Electrical Heating Jacket GS20730

Note: *For longer pathlength High Pressure Liquid Cells the recessed surface side of the front cover plate (**5**) is fitted to be in contact pressing against the end of the cell where the flow tubes enter.*

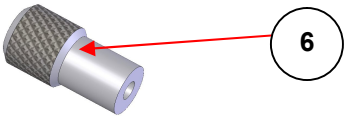


Fig 4. Thumbscrew (2 off) used with the front cover plate (5)

From **Figs 3 and 4** the thumbscrews (**6**) hold the front cover plate (**5**) to the screw threaded studs of the Electrical Heating Jacket through the holes identified at (**7**). For insertion of the controlling thermocouple of the Electrical Heating Jacket into the High Pressure Liquid Cell, one of the holes identified at (**8**) is used on the front cover plate (**5**), depending on which surface of the front cover plate (flat or recessed) is in contact with the High Pressure Liquid Cell.

3. Insert the Electrical Heating Jacket together with the High Pressure Liquid cell in the spectrometer 3" x 2" mount and secure.

4. Connect the High Pressure Liquid Cell to the flow system as described for Ambient Temperature Use from step 3.

Warning: *Exercise extreme care if needing to bend the liquid flow tubes so that the brazed joints to the cell body are not damaged.*

5. Ensure that heating/cooling rate (ramp) of the Electrical Heating Jacket does not exceed 10°C per minute.

6. Connect the Electrical Heating Jacket to its temperature controller.

7. Connect the thermocouple from the temperature controller to the thermocouple hole (**9**) in the body and at the front of the High Pressure Liquid Cell. (See **Fig 5**). The thermocouple will also pass through the front cover plate through one of the hole positions (**8**) as seen at **Fig 3**. Positioning of the thermocouple here in the High Pressure Liquid Cell enables the temperature of the cell to be monitored very close to the liquid sample.

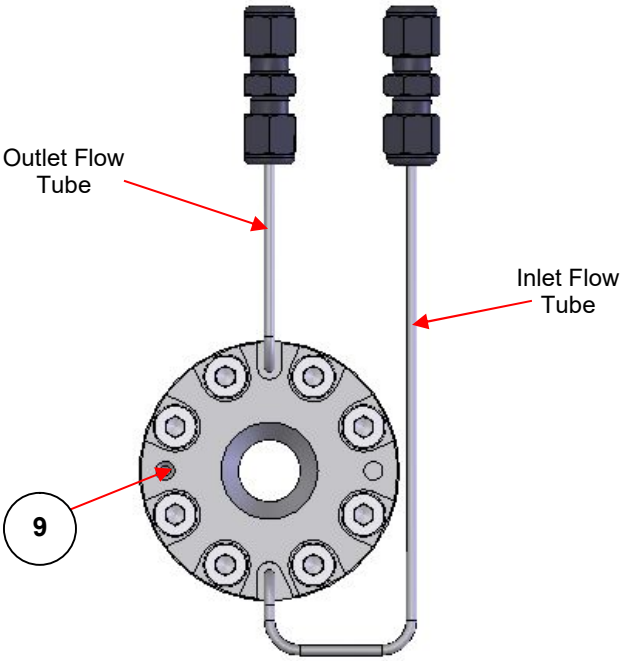


Fig 5. Front view of High Pressure Liquid Cell (without its slide mount backplate) showing thermocouple connection point (9) when cell is installed in the Electrical Heating Jacket

Heated Applications in the Water Heating Jacket GS20710

1. Insert the High Pressure Liquid Cell (after removing its own 3” x 2” slide mount backplate) in the Water Heating Jacket. (Please refer to the instruction manual for the Water Heating Jacket P/N GS20710 to explain the procedure.)

5. Cleaning and Storage

Important: *The High Pressure Liquid Cell must be thoroughly cleaned after use.*

1. Flush the cell through with several milliliters of a suitable solvent in which the sample is soluble.

2. Record the spectrum of the solvent to ensure that all traces of the sample have been flushed out of the cell.

3. Flush the cell through with a volatile solvent (e.g. acetone) and dry by blowing through with clean, dry, compressed air.

4. Store the cell in the carry case or a warm dry cabinet.

6. Part Numbers for High Pressure Liquid Cells

Cells with choice of 0.5, 1.0 or 2.0mm pathlength.

P/N GS05910 ZnSe, Sapphire or Spectrosil B windows at 2000psi pressure rating.
P/N GS05915 Sapphire or Spectrosil B windows at 5000psi pressure rating.

Cells with choice of 5.0 or 10mm pathlength.

P/N GS05920 ZnSe, Sapphire or Spectrosil B windows at 2000psi pressure rating.
P/N GS05925 Sapphire or Spectrosil B windows at 5000psi pressure rating.

7. Specifications for High Pressure Liquid Cells

The High Pressure Liquid Cells have the following specification dependent on their configuration.

Pressure Rating: 2000psi (136bar) or 5000psi (340bar) - see note on Hazardous Samples (page 7.).

Cell Body Material: 316 Stainless Steel (other materials on request).

Flow tube: Stainless Steel 1/16”. O.D. x 0.016" wall thickness.

Window Material: ZnSe, Sapphire or Spectrosil B (other materials on request).

Clear Aperture: 10mm diameter.

Seal Material: Viton (other materials on request).

Temperature Range: - 15C to 180C.

Fixed Pathlengths: 0.5mm +/- 10%, 1.0mm +/-10%, 2.0mm +/- 10%, 5.0mm +/- 2%, 10.0mm +/- 1%.

Liquid volumes (without tubes): 125 microliters for 0.5mm pathlength, 250 microliters for 1.0mm pathlength, 500 microliters for 2.0mm pathlength, 1.25 milliliters for 5.0mm pathlength, 2.50 milliliters for 10.0mm pathlength.

Notes for Usage of High Pressure Liquid Cell

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