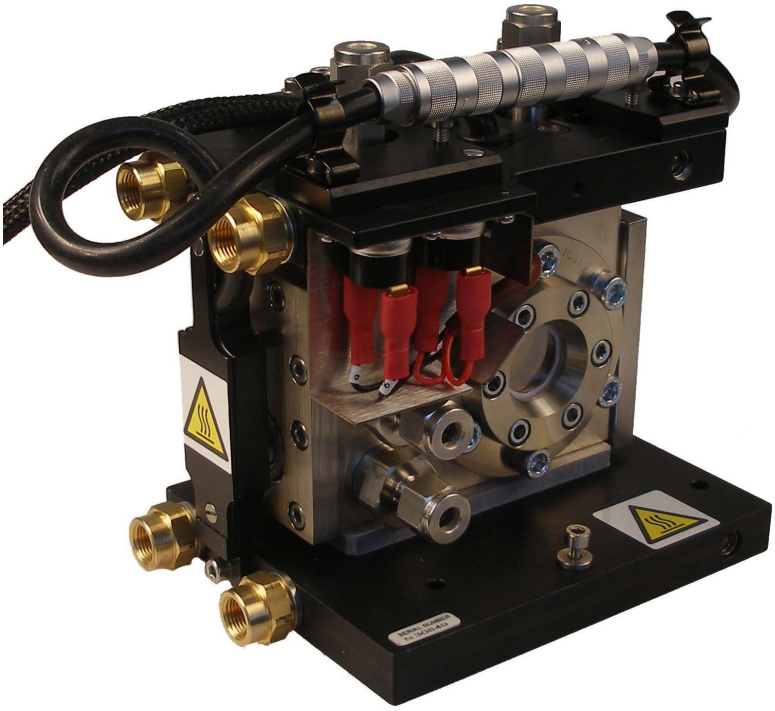
High Temperature
High Pressure Cell

User Manual



2I-05850 Issue 16

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High Temperature High Pressure Cell P/N GS05850

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1. Introduction

The High Temperature High Pressure (HTHP) Cell Accessory is a multipurpose cell for the Fourier Transfer Infrared (FTIR) analysis of sample types in transmission, decomposition or specular reflectance modes of experimental operation. The sample to study is held within a central sample post heater mount assembly which can be heated up to 800°C maximum temperature (under vacuum conditions) in a surrounding cell chamber. The surrounding cell chamber cell can be operated for different environmental gaseous conditions at pressures from vacuum (0.003 torr) to 1000 psi maximum for any in-situ reaction studies against a sample for analysis under such experimental conditions. The HTHP Cell is provided with gas connection ports for gas introduction and vacuum operation capability.

The cell chamber body as standard consists of two pressure certified (to 1000psi) zinc selenide (ZnSe) window assemblies to allow for the transmission of IR radiant light to and from the sample held within the sample post mount assembly. The effective pathlength between the two ZnSe windows for the cell chamber is 30mm. These cell chamber windows and cell body can be heated and controlled up to 240ºC maximum temperature independently of the temperature that can be set (up to 800°C) for the sample temperature itself. In operation this helps to prevent the condensation of any evolved materials in a vapour state from a hot sample forming on the ZnSe windows to reduce the light energy to and from the sample under analysis. Water cooled top and bottom plates on the cell body act as safety devices to prevent any excessive heating of the spectrometer sample compartment in operation of the HTHP Cell. Further heat shields on either side of the cell body are provided to avoid any radiant heat reaching the detector.

The standard P/N GS05850 HTHP Cell is operable in transmission and decomposition mode when attached to a dedicated transmission type of baseplate for the particular spectrometer being used. The transmission mode allows for 13mm diameter solid sample discs mounted in the heater block assembly to be presented to the beam of infrared radiation. The decomposition mode of operation is achieved by repositioning the heater block assembly within the cell body, whereby a

sample cup is placed into the heater block assembly just below the IR optical beam passing through the cell chamber. A solid, semi-solid or liquid type sample is placed into the sample cup and any gases/vapours that may be evolved from the sample at elevated temperatures to fill the cell chamber can be analysed.

To operate the HTHP Cell in specular reflectance mode, an alternative reflectance baseplate with transfer optics (mirrors) is required for the particular spectrometer being used and one of the standard pressurised window assemblies is replaced with an angled (wedged) window housing assembly. In addition, certain mount brackets and cooling water fittings are also required for adaptation of the HTHP Cell, dependent upon the IR radiant beam direction from source to detector through the sample compartment of the spectrometer. The parts required for this conversion of the P/N GS05850 HTHP Cell are available as an upgrade kit P/N GS05860, or all the necessary parts are provided as standard if the HTHP Cell has been ordered as the Advanced Version P/N GS05855. A separate instruction manual is provided with P/N GS05860 for installation and operation of the HTHP Cell when converted into the specular reflectance mode.

The HTHP Cell accessory is manufactured mainly from corrosion resistant and vacuum compatible materials. The main body parts are in stainless steel EN58J but the sample heater body and associated parts are in Incolloy 800HT material. The standard HTHP Cell has ZnSe windows and silicone seals, but other window materials and seals can be used if ordered as special.

Temperature control of the HTHP Cell accessory is provided by its own dedicated power controller. The sample temperature up to 800°C of the sample heating post mount assembly is controlled using a high stability proportional-integral-differential (PID) controller (WEST 6100+) with a continuous digital readout display of both the set and actual sample temperatures. A second WEST 6100+ controller is used to independently set the window and cell body temperature up to 240°C, similarly with a digital display of both the set and actual window/body temperatures.

The WEST 6100+ controller for heating of the sample heating post mount assembly can be operated automatically by computer control from RS232, RS485 or USB cable connectivity options (Specac P/N's GS28000, GS28001 or GS28002 respectively), from a rear connection port on the controller. The connectivity option kits include the fitting of the connection port plug to the controller and an appropriate interface card installed within the WEST 6100+ to operate the HTHP Cell controller via a computer from appropriate controlling software. Specac recommends that you contact your local WEST Instruments representative with regards to specific software packages compatible for control of the WEST 6100+ controller to operate the HTHP Cell heating function accordingly.

Instructions for operation of the dedicated power controller for the HTHP Cell are to be found in the separate instruction manual provided for the controller included with this accessory.

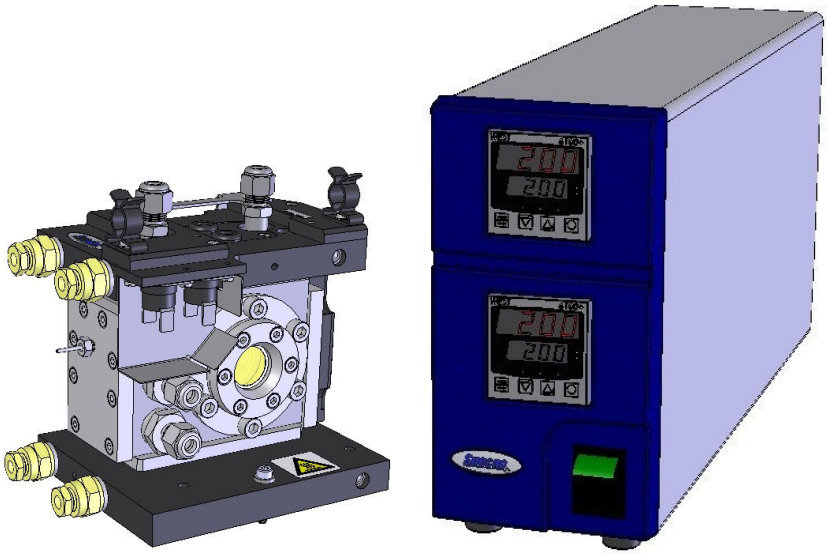


Fig 1. HTHP Cell (Protective Shields and Electrical Cables removed for clarity) and its Dedicated Power Controller

2. Specifications of the HTHP Cell

HTHP Cell Specifications

Pressure: 1000 psi (maximum).

Vacuum: 0.003 torr or better (4.0×10^{-3} mbar).

Cell Pathlength: 30mm (between ZnSe window inner surfaces) in transmission and decomposition modes of operation.

Clear aperture: 11mm dia.(transmission mode for solid sample at sample post) and 17mm dia.(decomposition mode at ZnSe window).

Sample size: 13mm diameter and up to 6mm thick solid sample disc in transmission mode and 0.28ml capacity of sample pan in decomposition mode.

Cell chamber volume: 80ml.

Heated surfaces: Incalloy 800HT for sample heating post assembly and decomposition pans in transmission and decomposition modes respectively. 316 stainless steel for cell body and ZnSe pressurised window assemblies.

Temperature range: Ambient to 800ºC (under vacuum conditions).

Thermocouple type: K-type NiCr/NiAl (heating post and cell body).

Heater power: 250 Watts (sample heating post assembly) and 200 Watts (cell body and ZnSe pressurised window assemblies).

Heater voltage: 30 Volts (sample and cell body heaters).

Water cooling (flow rate): Continuous (typical flow rate = 0.75 to 1 litre/min).

Water cooling (input temperature): Up to 25ºC (water above 25ºC may have to be cooled depending on temperature of the HTHP Cell)

Windows: ZnSe (zinc selenide).

Cell chamber sealing O-rings: Silicone material.

Temperature Controller Specification

Temperature range: Ambient to 800ºC for sample.
Ambient to 240°C for cell body and pressurised window assemblies.

Temperature setting: 1°C step change for sample heating post assembly.
1°C step change for cell body and pressurised window assemblies.

Temperature display: Digital (set and actual temperatures for sample heating post and cell body/windows).

Setting accuracy: 0.2% for sample (+/- 1 digit for display).
1% for cell body/windows. (+/- 1 digit for display).

Control type: Proportional (P), Integral (I), Differential (D) (PID) with temperature rate ramp control for sample.
PID (factory set) for cell body and pressurised window assemblies.

System Specification

Temperature control stability : Better than 1ºC (rms).

Heating rate: Less than 40 mins to 800ºC for sample.
Less than 40 mins to 240ºC for cell body.

Cooling rate: Typically 100 mins - (cooling from 800ºC to 60ºC for sample and from 240ºC to 60ºC for cell body).

Mains Voltage: 240 or 110 volts supplies (+5% - 10%).

Frequency: 50/60 Hz (dependent on voltage).

Power: 750 watts maximum (for sample heating post assembly).

Temperature range allowable for surrounding conditions: 5°C to 40ºC.

Humidity range allowable for surrounding conditions: 20 to 80%.

General Principles of Operation

Temperature Achievable with a Pressurised Gas

The full working temperature of 800ºC for the HTHP Cell can be achieved from vacuum (0.003 torr) to circa standard atmospheric pressure (14.7 psi) conditions.

Pressurised gases have a higher thermal conductivity than unpressurised gases. Therefore, at the full working pressure of 1000 psi for the HTHP Cell accessory, the specified upper temperature limit of 800ºC when under vacuum conditions cannot be achieved. The maximum temperature that can be reached will depend on the nature and thermal conductivity of the pressurised gas being used. A higher thermal conductivity value of a gas to be used, results in a lower attainable maximum temperature. Thermal conductivity also increases as the temperature increases and the rate of increase also has a bearing on the maximum temperature achievable.

As an example, when the HTHP Cell is pressurised to 1000 psi with nitrogen gas, the maximum working temperature is lowered to circa 550ºC.

A guide for thermal conductivity values for some gases are given below:

Gas	Thermal Conductivity $Calsec^{-1}cm^{-1}deg^{-1}$ (x 10^{-6}) @ 26.7ºC	Thermal Conductivity $Calsec^{-1}cm^{-1}deg^{-1}$ (x 10^{-6}) @ 48.9ºC
Nitrogen	62.40	65.71
Helium	360.36	376.07
Hydrogen	446.32	471.11
Oxygen	63.64	68.19
Air	62.20	66.04
Carbon Dioxide	39.07	43.81

Heater Lifetime Versus Operating Temperature

The heater lifetime of the sample heater block assembly for the HTHP Cell is directly related to the amount of power supplied by its dedicated temperature controller and the heating or ramp rate parameter setting on the WEST 6100+ controlling unit.

The figures below give an approximate indication of the heating element lifetime dependent upon the typical temperature level to be maintained in operation of the HTHP Cell. If the parameter values (power level and ramp rate) of the temperature controller are set above the optimum factory set values shown, the lifetime of the heater will be reduced further.

Maximum Set Power Level (%)	Ramp Rate (ºC/min)	Set Temperature (ºC)	Approx. Heater Life (Hours)
86 to 95 *	30	800	500
86 to 95 *	30	720	3000
86 to 95 *	30	700	6000

* See Cleaning of the HTHP Cell, Section 11), pages 72 to 76.

It should be noted from the figures in the table above that higher temperatures of operation for the HTHP Cell, i.e. above 700ºC, have a significant effect on the heater lifetime.

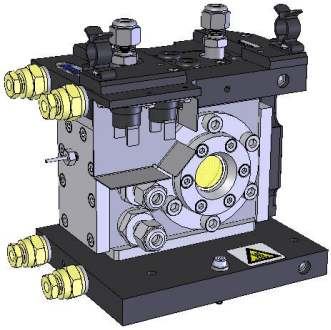
Note: *If the heater for the heater block assembly should fail at any time, the HTHP Cell **must** be returned to Specac to effect a repair. However, a replacement spare complete heater block assembly is available that can be used to make the HTHP cell operational again and any damaged assembly of heater parts need only to be sent back to Specac to effect a repair if necessary. (See Section 5), pages 45 and 46 for more detail of the heater block assembly).*

3. Unpacking and Checklist

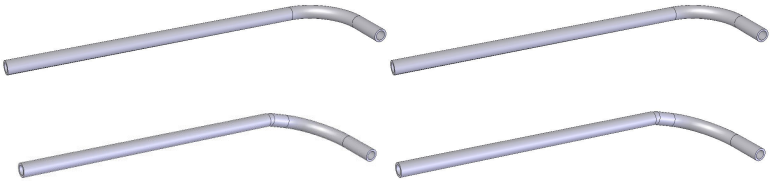
The HTHP Cell and its associated accessories, whether supplied as ordered from P/N GS05850 (Standard) or P/N GS05855 (Advanced; with reflectance mode capability of operation), are packaged in polythene bags and are supplied in a carry case. The dedicated HTHP Cell temperature controller is supplied in its own separate packaging.

On receipt of the items please remove carefully from their packaging and check for the following:

1x HTHP Cell Assembly (has electrical cabling connected and pressure certified ZnSe window assemblies are covered with protective paper).

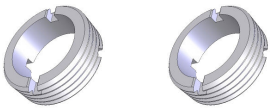


4x Gas feed pipes (2 angled and 2 straight pipes).

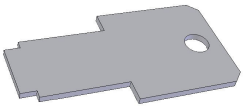


2x Decomposition mode sample cups.

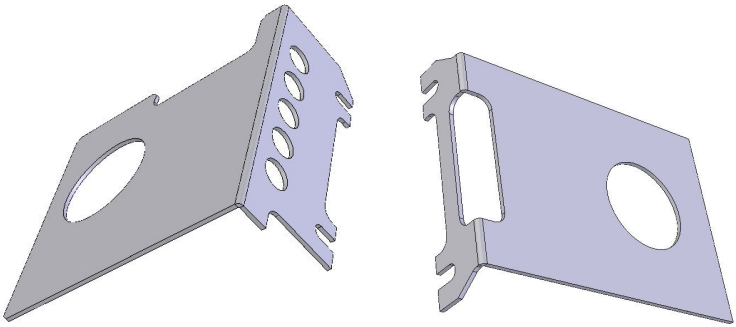
2x Keep rings.



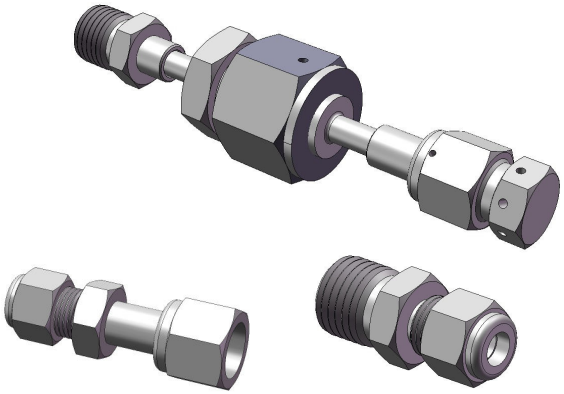
1x Keep ring key.



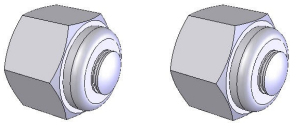
2x Stainless steel heat shields (1 wide, 1 narrow).



1x Burst disc attachment (1/4” tube inlet, 3/8” tube outlet with baffle). Supplied as three separate fitting assemblies of parts (burst disc assembly and two tubing connection adapters).

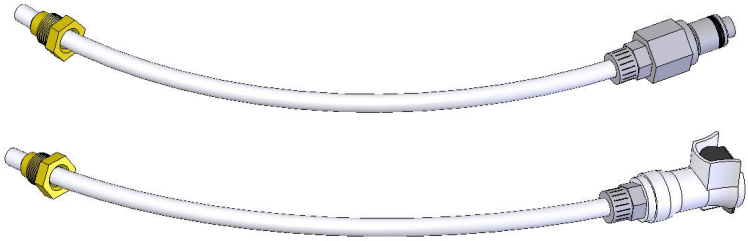


2x Swagelok blanking plug.
(For 1/4” O.D. tubing union fittings).

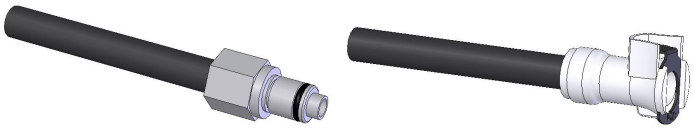


1x Water cooling system parts in a polythene bag consisting of:

2x Quick connects with short lengths of nylon tubing.

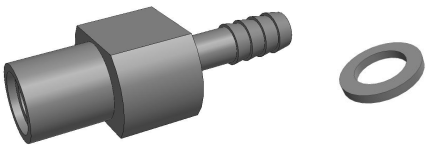


2x Quick connects with longer lengths of rubber tubing.



1x Nylon tubing with ferrules/nuts at both ends.

1x Vacuum pipe adapter and silicone gasket.



1x Forceps.
1x Ball driver 3mm A/F.
1x Torque wrench with 1/4" square drive connector.
1x 3/8” A/F adaptor for torque wrench – 1/4” square drive connection.
1x 3mm A/F adaptor for torque wrench – 1/4” square drive connection.
1x 5mm A/F adaptor for torque wrench – 1/4” square drive connection.
2x Open ended spanners 9/16” and 1/2” A/F.
1x Open ended spanner 12mm and 13mm.
1x Spectrometer specific baseplate. (See installation Section 15) for identification of the baseplate supplied.)
1x Additional monitoring thermocouple and ferrule set
1x Dedicated HTHP Cell temperature controller
1x Mains power cable for temperature controller.
1x Instruction manual for temperature controller
1x Essential Spares Kit of parts (P/N GS05870)
Test certificates: 1 for HTHP Cell body, 1 for HTHP Cell windows and 1 for angled window used in reflectance mode when ordered as Advanced HTHP Cell GS05855).

4. Safety When Using the HTHP Cell

Warning: Before using the HTHP Cell you MUST read the **Safety** information that follows and the **Preparing The HTHP Cell For Use** information (Section 5, pages 21 to 52).

Safety Features

The design of the HTHP Cell incorporates a number of important safety features. In particular these are:-

1) All electrical supplies to the cell are low voltage (30 volts or less) to comply with the 'extra low voltage' condition of Canadian Standards Association (CSA) (C22.2 regulations).

2) The HTHP Cell's dedicated temperature controller is equipped with open circuit detection on the thermocouple inputs.

3) The HTHP Cell itself is fitted with a pressure release device, called a "burst disc", to prevent inadvertent over-pressurisation to the system and potential damage to the pressure certified ZnSe window assemblies. The burst disc device outlet **must** be piped/plumbed to a fume cupboard or other safe repository for any accidental venting of hot gases as necessary.

4) A **thermal fuse** within the HTHP Cell is set to halt **any operation** of the cell should the thermocouple connection fail and overheating occurs. This selective fuse will blow when **the cell body temperature exceeds 300ºC**. If this situation occurs it is recommended that the HTHP Cell is returned to Specac for investigation and subsequent resetting of the thermal fuse.

5) Efficient operation of the cooling system has been safeguarded by fixing **thermal switches** to the top cooling plate. The thermal switches will shut down power to the HTHP Cell if there is an insufficient flow of cooling water passing through the top and

bottom cooling plates and the temperature of the water rises above 40ºC. The thermal switches will reset to enable the HTHP Cell to function normally again with re-establishment of power when the cooling water temperature reaches approximately 25ºC.

General Safety

This apparatus has been designed and tested in accordance with Safety Class 1 requirements of IEC Publication 348, Safety Requirements for Electronic Measuring Apparatus and has been supplied in a safe condition. The current instruction manual contains some information and warnings that must be followed by the user to ensure safe operation and retain the apparatus in a safe condition.

The apparatus has been designed for indoor use. It will operate correctly under the following conditions:

Ambient temperature : 5ºC to 40ºC
Relative humidity : 80% maximum (non-condensing)

Whenever it is likely that the apparatus is unsafe, it should be made inoperative. The apparatus may be unsafe if it:

- Shows visible damage.
- Fails to perform the intended measurement.
- Has been subjected to prolonged storage in unfavorable conditions.
- Has been subjected to severe transport stresses.

Cylinder Gas

When compressed gases are being used with the HTHP Cell Accessory, the user should provide adequate space for supply of the gas from a cylinder. Any gases should be stored in a safe manner in accordance with good safe practice and when being connected to the HTHP Cell as required. (See Section 5), page 33, on recommended plumbing of gas lines to and from the HTHP Cell for operation).

Do's And Do Not's!

READ THESE SAFETY WARNINGS BEFORE OPERATING THE HTHP CELL ACCESSORY

DO use the HTHP Cell Accessory with care for potentially combustible materials or materials with a low flash point temperature.

DO use the safety heat shields at all times.

DO use the HTHP Cell Accessory with a continuous and adequate flow of cooling water.

DO ensure that any gas or water piping connections to the HTHP Cell are leak free and safe.

DO NOT use the HTHP Cell with any samples that are potentially **explosive** by nature.

DO NOT touch the HTHP Cell body or the window flange surfaces with bare hands. They may be VERY HOT!

DO NOT remove a sample from the HTHP Cell sample cell holder without wearing gloves for handling. (AVOID BURNS!) Samples may take longer to cool than the HTHP Cell body and window parts.

DO NOT touch the ZnSe window material with bare hands. ZnSe is a toxic material and skin contact should always be avoided. Always wear gloves when handling the window housings if they are to be removed or fitted to the HTHP Cell and when cleaning.

5. Preparing the HTHP Cell for Use

Introduction

Operation of the HTHP Cell Accessory relies on correct installation into an appropriate spectrometer sample compartment and connection to service supplies such as cooling water, gas inlet and outlet tubing and electrical power.

The HTHP Cell Accessory is most easily operated in the sample compartment of a spectrometer system that can be left open to the surrounding environment. This allows for relatively straightforward connections of the service supplies (cooling water, gases and electrical power) to the cell. If the spectrometers sample compartment lid is to be kept closed e.g. to purge a system with dry nitrogen gas or to operate in a local vacuum environment outside of the HTHP Cell chamber itself, then it is likely appropriate modifications to the compartment lid will need to be made to allow for the water, gas and electrical connections to the HTHP Cell. If a custom built sample compartment lid is not needed or available, in practice it may be necessary to remove a standard sample compartment lid for the spectrometer and use alternative shrouding (e.g. black plastic material placed over the area and sealed around the edges with masking tape).

Note: *For any operation of the HTHP Cell, if specific customizing is required for installation and connection of the appropriate service supplies, always ensure that the parts are kept safe and away from the possibility of being accidentally heated by being in close proximity to the HTHP Cell. Route any water and gas pipes and electrical cables safely and neatly.*

Fitting of Inlet and Outlet Cooling Water Connections

Certain parts of the HTHP Cell Accessory become very hot during operation (particularly at the maximum temperature of 800ºC for the sample heating post area within the cell chamber) and to prevent any excessive transfer of a heating effect to the spectrometer sample compartment, some form of cooling is required. This is provided by

water being flowed through water cooled plates above (top - **1**) and below (bottom - **2**) the HTHP Cell chamber itself. These water cooled plates (**1**) and (**2**) are physically attached to the HTHP Cell body (**3**) and heat dissipation works via conduction.

On initial/new supply of the HTHP Cell from either the P/N GS05850 or P/N GS05855 part number as ordered, the HTHP Cell is provided with standard water connection brass fittings (**4**) already attached to the upper (**1**) and lower (**2**) cooling plates for connection of the separately supplied Nylon tubing parts (**5**), (**6**) and (**7**) to the HTHP Cell for cooling water circulation. Fitting of the Nylon tubing parts (**5**), (**6**) and (**7**) to the standard fittings (**4**) from the following steps A), B) and C) allows for the HTHP cell to operate in all (**transmission**, **decomposition** and **reflectance**) modes of sample measurement (See **Fig 2.)**

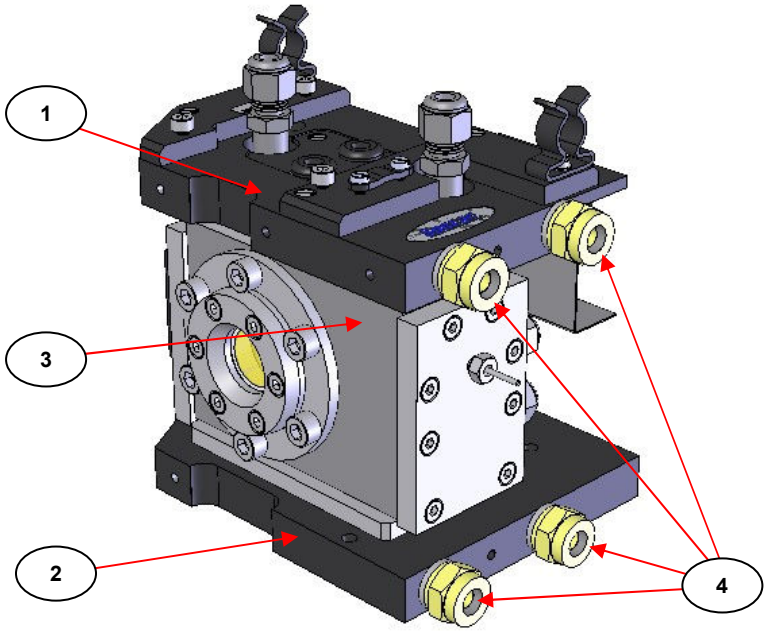
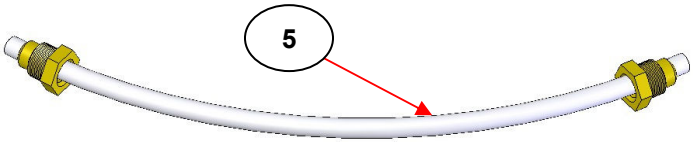


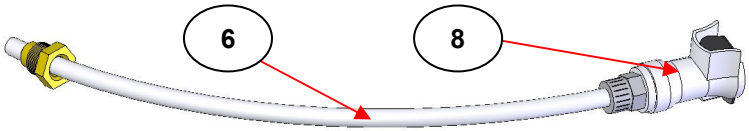
Fig 2. Standard Water Connection Brass Fittings on the HTHP Cell as supplied under P/N GS05850 or P/N GS05855

A) From the pack of water cooling tubing supplied, fit the Nylon tube (**5**) with the ferrules/nuts on both ends of the tube to one of the brass water connection fittings (**4**) on both the upper (**1**) and lower (**2**) water cooled plates. (See **Fig 2.**).

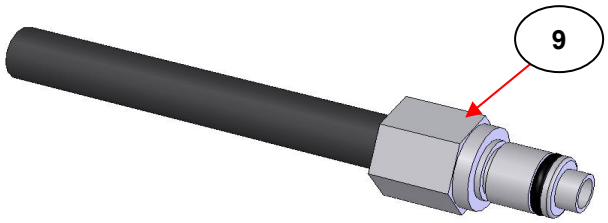


(The tubing (**5**) connects the top (**1**) and bottom (**2**) cooling plates together for water flow). Tighten together the tubing (**5**) ends to the water fittings (**4**) from the tubing nut fittings using the open ended 13mm A/F spanner supplied.

B) Connect the short Nylon tube (**6**) (approx. 130mm long) with a ferrule/nut at one end and the **body** (**8**) part of the Quick Connect fitting at the other end to the remaining brass fitting (**4**) on the **lower** water cooled plate (**2**). (See **Fig 2**.).



Tighten together the nut connection end of the tubing (**6**) to the water fitting (**4**) using the open ended 13mm A/F spanner supplied. Then take the **insert** (**9**) part of the Quick Connect fitting that is attached to the long piece of **black silicone rubber tubing** and push fit the insert (**9**) to click into the body (**8**) part on the Nylon tubing (**6**). (See **Fig 3**.).



This complete length of Nylon and silicone rubber tubing constitutes **the inlet tubing system** for water flow which should **ALWAYS** be introduced to the bottom water cooled plate (**2**).

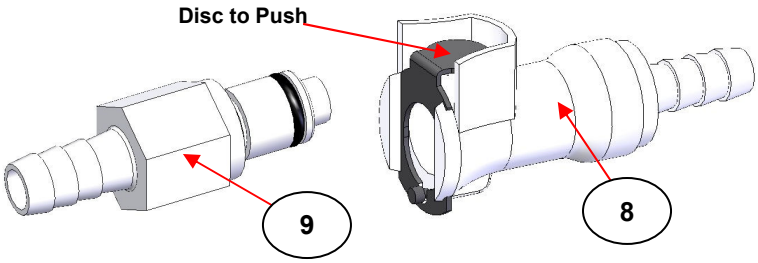
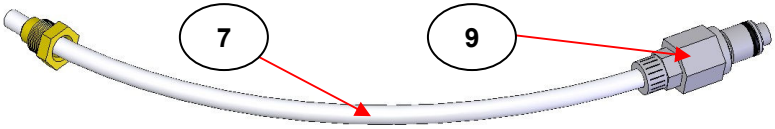


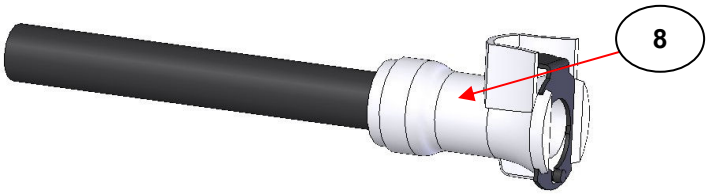
Fig 3. Insert (9) and Body (8) Quick Connectors

Note: *The Quick Connectors (**8**) and (**9**) are a simple push fit (insert into body), which will click when fully engaged. To disconnect, the silver disc on the body (**8**) connector is pushed down whilst the connectors are being pulled apart. When these Quick Connecter fittings are disconnected, water is prevented from leaking out of the tubing, thus avoiding accidental spillage.*

C) Connect the other short nylon tube (**7**) (approx. 130mm long) with A ferrule/nut at one end and the **insert** (**9**) part of the Quick Connect fitting at the other to the remaining brass fitting (**4**) on the **upper** water cooled plate (**1**). (See **Fig 2**.).



Tighten together the nut connection end of the tubing (**7**) to the water fitting (**4**) using the open ended 13mm A/F spanner supplied. Then take the **body** (**8**) part of the Quick Connect fitting that is attached to the long piece of **black silicone rubber tubing** and push fit the body (**8**) to click into the insert (**9**) part on the Nylon tubing (**7**). (See **Fig 3**.).



This complete length of Nylon and silicone rubber tubing constitutes **the outlet tubing system** for water flow which should **ALWAYS** exit away from the top water cooled plate (**1**).

Cooling Water Flow

A continuous cool/cold water supply for the water cooling system circuit is required for operation of the HTHP Cell. To establish a cooling water flow through the HTHP Cell, the silicone rubber tubing for the **inlet** tubing system is connected to a cool/cold water supply (tap). The silicone rubber tubing from the **outlet** tubing system is placed into a waste drain, although ideally the flowing water from the outlet tubing system can be returned to a holding tank to save the water for re-use.

Note: *If a local water supply for use is difficult to access and/or there is a need to conserve water rather than having flowing water running away to a waste, then the water cooling requirement of the HTHP Cell Accessory can be accommodated by use of a "closed circuit" water pumping thermocirculator accessory offered as Specac P/N's GS11127 or GS11128. The water flow from the outlet tubing system is routed back to a holding tank bath to be recirculated via a pumping system to the inlet tubing system.*

Important! The water should be kept running at a flow rate sufficient to keep the cooling plates (**1**) and (**2**) cool throughout the analysis. A typical water flow rate is 0.75 to 1 liter per minute. The cooling water supply should be turned on before any heating is applied to the HTHP Cell and kept running after any analysis until the temperature measurement for both the sample heating block assembly (**10**) and the cell body (**3**) falls below 40ºC.

Efficient operation of the cooling system has been safeguarded by fixing a **thermal switch** to the top cooling plate (**1**). The thermal switch will shut down power to the HTHP Cell if there is an insufficient flow of cooling water passing through the top (**1**) and bottom (**2**) cooling plates and the temperature of the water rises above 40ºC. The thermal switch will reset itself to enable the HTHP Cell to function normally again with re-establishment of power when the cooling water temperature reaches approximately 25ºC.

Water Fittings for Reflectance Mode of Operation

If the **reflectance** mode of operation is required (the HTHP Cell has been ordered and supplied as P/N GS05855), then alternative water connection “banjo” fittings (**58**) in place of the standard fittings (**4**) may need to be attached to the HTHP Cell cooling plates (**1**) and (**2**) before connection of the Nylon tubing parts (**5**), (**6**) and (**7**) to enable for the correct installation. These “banjo” fittings are also supplied along with the GS05860 Reflectance Mode Upgrade Kit for installation of the HTHP Cell specifically into a Left to Right (**L** to **R**) IR beam direction (source to detector) through the spectrometer sample compartment. The instruction manual supplied for P/N GS05860 (and for use with P/N GS05855) is consulted to know how to replace the standard water connection brass fittings (**4**) with the banjo fittings (**58**) for use of the HTHP Cell in **L** to **R** beam direction spectrometer systems for the reflectance mode of operation. (See **Fig 4**.).

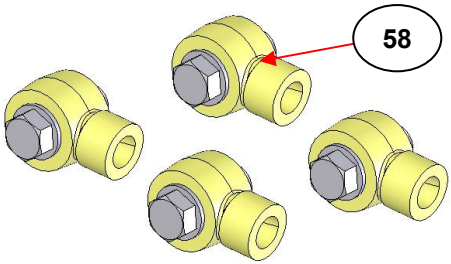


Fig 4. Banjo (Right Angled) Water Flow Fittings for Replacement of Standard Water Fittings in L to R Reflectance Mode Operation

Gas Pipe Connections to the HTHP Cell

The HTHP Cell can achieve a maximum temperature of 800°C at the sample heater block assembly (**10**) when the cell chamber is being operated under vacuum conditions. Alternatively, the cell chamber can be pressurized to a maximum of 1000psi which is the safe pressure limit for use of the standard pressure certified ZnSe window assemblies (**11**).

Note: *Under pressurised gas operating conditions the maximum temperature achievable is dependent upon the gas type being used and a particular thermal conductivity value associated with the gas. (See guide Specification Section 2), page 12.)*

To allow for the introduction of gases under pressurised conditions in, or flow conditions through the cell chamber and for operation of the HTHP Cell chamber under vacuum conditions, the HTHP Cell body (**3**) carries **four** gas connection ports. (See **Fig 5**.)

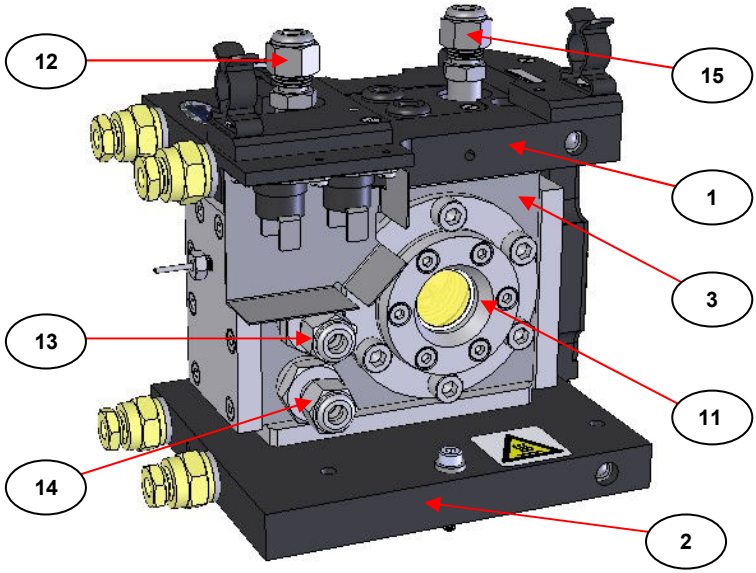


Fig 5. Gas Connection Ports on the HTHP Cell

These four connection ports are:
(**12**) The main **inlet port 1** on top of the HTHP Cell body (**3**).
(**13**) An **inlet port 2** on the side of the HTHP Cell body (**3**).
(**14**) An **inlet port 3** on the side of the HTHP Cell body (**3**).
(**15**) The main **outlet port** on top of the HTHP Cell body (**3**).

The outlet port (**15**) opens into the top of the cell chamber, while the other three inlet ports (**12**, **13** and **14**) connect with each other internally and open, in one place, near the bottom of the cell chamber. A cross sectional view at **Fig 6.** shows how these inlet ports merge.

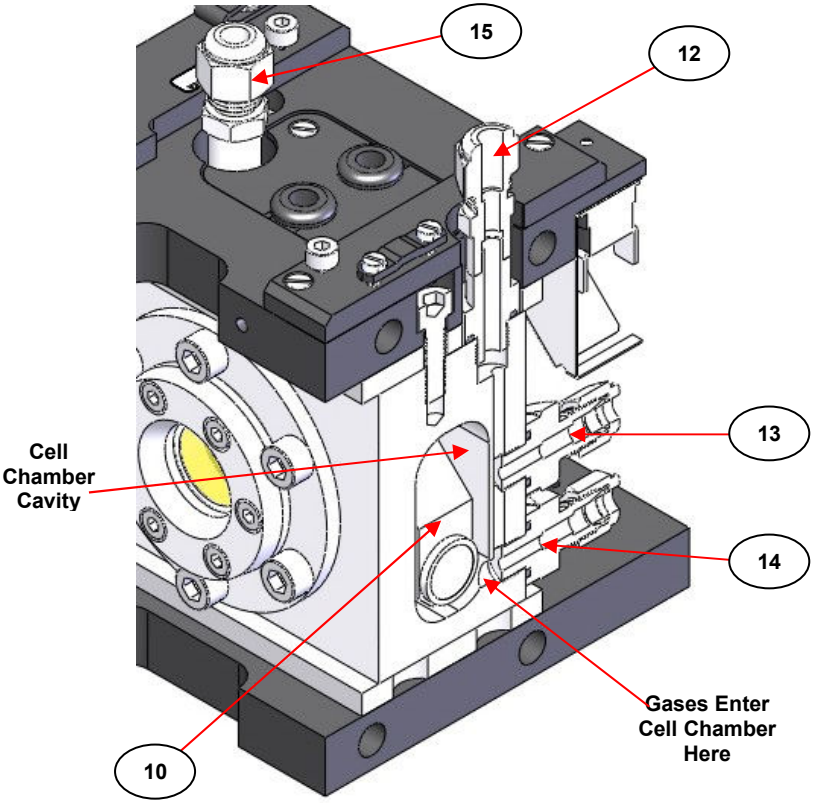


Fig 6. Cross Sectional Cutaway View of Inlet Ports Merging

From the design by introduction of a gas into the bottom of the cell chamber cavity by connection to any of the inlet gas ports (**12**, **13** or **14**) and having the outlet port (**15**) at the top of the cell chamber cavity, then a gas flow may be induced across the sampling area of the sample heater block assembly (**10**) for any mode of operation of the HTHP Cell. (Transmission, decomposition and reflectance).

Gas Introduction into the HTHP Cell

The inlet ports (**12**, **13** and **14**) may be used as desired for connection to appropriate carrier gas line piping via their standard Swagelok cap fittings (**16**) for 1/4" O.D. tubing. At least three different inlet gases can be permanently connected to the HTHP Cell from appropriate in line plumbing, open/shut switches and valve regulators, to be introduced singularly or at the same time as particular gas mixtures depending on the environment required for any experimentation to be carried out.

Note: *The supply gases and any appropriate tubing/plumbing, switches and valve regulators etc are normally to be sourced and provided by the user.* (See **Fig 9.)**

Supplied with the HTHP Cell as standard are lengths of 1/4" O.D. stainless steel tubes (**17**) for initial connection to the inlet ports using the Swagelok cap fittings (**16**) and their respective olive (**18**) and ferrule (**19**) sets of parts. Alternatively, if one or two of the inlet ports are not needed for any operation of the HTHP Cell they can be capped using the Swagelok blanking plugs supplied. The blanking plugs simply screw onto the threaded Swagelok union connection part (**20**) of the port inlet in place of the 1/4” O.D.Swagelok cap fitting (**16**) as supplied.

If the 1/4” O.D. stainless tubing (**17**) supplied is to be used, then they are connected accordingly. Carefully undo the Swagelok cap fitting (**16**) away from the threaded body union (**20**) of the port connection to the HTHP Cell body (**3**). Underneath the cap fitting (**16**) are the respective Olive (**18**) and ferrule (**19**) parts to use for compression sealing of the tubing (**17**) to the Swagelok union (**20**). Take the length of tubing (**17**) to connect by passing the tubing through the cap fitting (**16**), the olive (**18**) and ferrule (**19**) over the end of the tubing (**17**) in the correct sequence and orientation as shown. (See **Fig 7**.).

Place this assembly of parts into the open end of the Swagelok union (**20**) and proceed to hand tighten the cap fitting (**16**) to the threaded union fitting (**20**) enclosing the end of the tubing (**17**) with the olive (**18**) and ferrule (**19**) parts. When hand tight, using the open ended spanners supplied, tighten the cap fitting (**16**) further but by no more than one and half complete rotations of the cap fitting (**16**) against the Swagelok threaded union connection (**20**). This provides sufficient tightening together of the components to form a compression leak tight seal for the olive (**18**) and ferrule (**19**) parts against the tubing (**17**) for a tubing (**17**) connection to the inlet gas port (**12**, **13** and **14**).

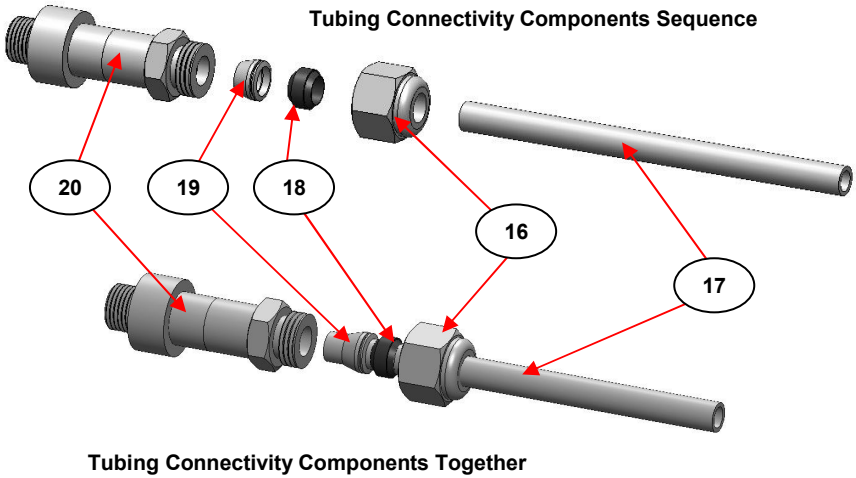


Fig 7. Fitting of 1/4" O.D. Stainless Steel Tubing to the Swagelok Fittings of the Inlet Gas Cell Port Connections.

Note: *If the tubing (**17**) parts supplied are used for initial connection for introduction of a gas into the inlet ports (**12**, **13** and **14**), then any subsequent gas feed supply lines/tubing must be connected accordingly to the open ends of the tubing (**17**) parts. (e.g. an inline Swagelok union part). Any additional parts required are normally to be sourced and provided by the user.*

Gas Outlet Connection, Flow and Vacuum Operation

There is one gas outlet connection port (**15**) on the top of the HTHP Cell body (**3**). Similar to the three gas inlet connection ports (**12**, **13** and **14**), the supplied 1/4" O.D. stainless steel tubing (**17**) can be initially connected to the Swagelok cap fitting (**16**) at this outlet connection port (**15**) as described and shown for **Fig 7**.

However, it is from the **gas outlet connection port (15) alone** that the necessary plumbing of the system has to be made to allow for use of the HTHP Cell as follows:

1) Positive pressure conditions of operation in the cell chamber.
2) A flow of gas(es) in operation through the cell chamber.
3) Vacuum conditions of operation in the cell chamber.

Crucial to any of the above operation conditions is inclusion of the supplied burst disc assembly (**21**) in a system of gas line connectivity to the HTHP Cell gas outlet connection port (**15**).

The Burst Disc Assembly of Components

The burst disc assembly (**21**) parts are vital components to be used in installation from the gas outlet connection (**15**) for the safe operation of the HTHP Cell. As supplied and identified from unpacking, the component parts for the complete burst disc assembly (**21**) consist of the items as shown for **Fig 8**.

The main burst disc assembly (**21**) itself is "split" into the parts (**22**) and (**23**). The body at (**22**) contains the internal "burst disc" diaphragm which is designed to rupture and break if the pressure within the gas line exceeds a level of greater than 1000psi. Should this diaphragm part ever break, it can be replaced easily to make the burst disc assembly (**21**) operational again. (Contact Specac to arrange for return and repair of the burst disc assembly). The gas flow outlet end of the body (**22**) is supplied already fitted with a perforated end plug (**23**) that allows for escape of any gas through the burst disc assembly (**21**) to a safe area when the internal burst disc diaphragm is ruptured.

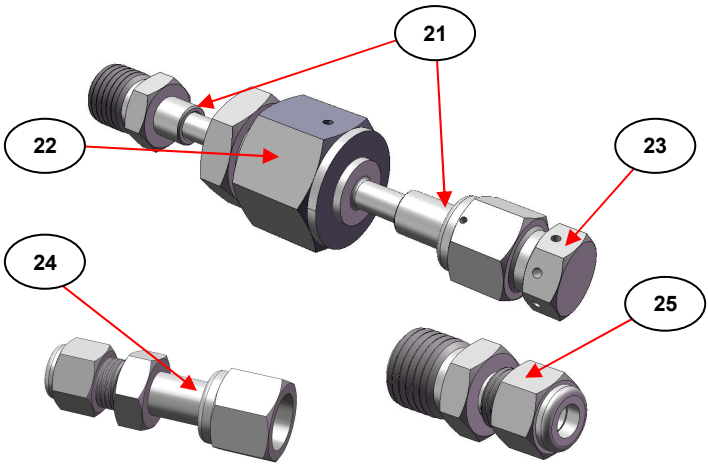


Fig 8. Burst Disc Assembly of Components as Supplied

The tubing connector union (**24**) allows for the burst disc body (**22**) to be connected in line to any 1/4" O.D. tubing (**17**), if this type of tubing is used from initial connection to the HTHP Cell gas outlet port (**15**). The 1/4" O.D. tubing cap fitting end of the connector (**24**) is attached to the tubing (**17)** in exactly the same way for compression sealing with an olive (**18**) and ferrule (**19**) set, as for the cap fitting (**16**) of the Swagelok union (**20**). The other end of the connector (**24**) is screw threaded for attachment to the burst disc body's (**22**) **inlet** end.

The tubing connector union (**25**) is a replacement for the perforated end cap (**23**) which allows for the burst disc body (**22**) to be connected in line to any additional 1/4" O.D. tubing (**17**), to extend a gas line for venting away any gases to a safe area. The 1/4" O.D. tubing cap fitting end of the connector (**25**) is attached to the tubing (**17)** in exactly the same way for compression sealing with an olive (**18**) and ferrule (**19**) set, as for the cap fitting (**16**) of the Swagelok union (**20**). The other end of the connector (**24**) is screw threaded for attachment to the burst disc assembly body (**22**) **outlet** end when the perforated cap (**23**) has been removed.

Inlet and Outlet Gas Line Connectivity

A system of plumbing for supply of gases to the inlet ports (**12**, **13** and **14**) and the necessary connections that need to be made from the outlet connection port (**15**) of the HTHP Cell is shown for **Fig 9**.

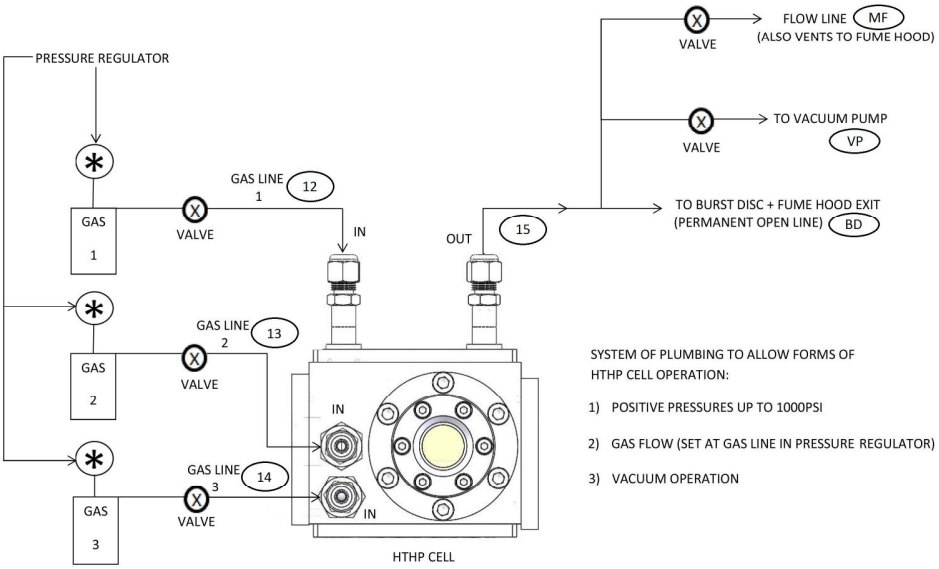


Fig 9. Inlet and Outlet Gas Connection Lines for Operation of the HTHP Cell

Essentially, three separate gas lines are required from the outlet connection port (**15**) to allow for the three different conditions of operation. (Positive pressure holding, flow of gas and vacuum holding.)

Important! *Irrespective of **any operation** of the HTHP Cell under high pressure, gas flow or vacuum conditions, the burst disc assembly (**21**) of parts **MUST** be plumbed in from the outlet connection port (**15**) to be a live and open conduit at all times for the safe evacuation of any gases in an over-pressurisation event. (In excess of 1000psi.)*

From **Fig 9**. the burst disc assembly of parts (**21**) are installed, having been connected as a main open line (**BD**) with no open/shut in line valve restrictions from the main gas outlet port (**15**). This open gas line (**BD**) for inclusion of the burst disc assembly of parts is ultimately routed for its terminal fitting of body (**22**) and perforated cap (**23**) and/or open end of tubing to emerge into a safe containment area such as a fume hood. Under any operating experimentation conditions, this (**BD**) line incorporating the burst disc assembly will be subjected to the same conditions of pressure, (high or vacuum conditions) or a flowing gas environment that can be achieved within the HTHP Cell chamber itself. If there was to be an over-pressurisation event within the cell chamber that would risk damage to the pressure certified window assemblies (**11**) then the excess pressure would activate the burst disc assembly (diaphragm ruptures) via the open (**BD**) line to evacuate any hot and potentially toxic and dangerous gases to the safe containment area away from the HTHP Cell chamber itself.

From the main outlet connection port (**15**), the other two gas lines that emanate from the (**BD**) open line are “branched off” before the burst disc body part (**22**) into a separate vacuum pump (**VP**) line and a main flow (**MF**) line. Both of these gas outlet lines require an open/shut valve connection to be plumbed in line to switch them on or off accordingly for any operating condition of the HTHP Cell chamber itself. See the table following to explain how each valve for both **inlet** and **outlet** connectivity is to be set (open or shut) for the appropriate HTHP Cell operating conditions.

HTHP Cell Operating Condition	Inlet Valve from Gas Line 1, 2 or 3	Outlet Valve (MF) Line	Outlet Valve (VP) Line
High Pressure	**Open (*1)**	**Shut**	**Shut**
Gas Flow	**Open**	**Open**	**Shut**
Vacuum	**Shut**	**Shut**	**Open (*2)**

(*1) Note: *After a gas has been introduced into the HTHP Cell chamber at a specific **regulated** pressure with both the (**MF**) line and (**VP**) line valves being shut, the inlet valve of the specific gas line can then also be shut. Provided all gas line connections are leak tight, the introduced pressure level of gas will be maintained within the HTHP Cell chamber under the same temperature conditions.*

(*2) Note: *The vacuum pump is switched on to draw a vacuum in the HTHP Cell chamber from these inlet and outlet valve settings. When a sufficient vacuum level has been maintained the (**VP**) line valve may then also be shut. Provided all gas line connections are leak tight, the introduced vacuum level will be maintained within the HTHP Cell chamber under the same temperature conditions.*

Similar to the routing of the (**BD**) line, ultimately to a safe containment area (e.g. fume hood), Specac would advise that the (**MF**) line is also routed for the outlet end of any tubing to emerge into a similar safe area for any gas flow containment (disposal). Also, any exhaust venting of a vacuum pump used for the (**VP**) line of operation can similarly be piped to the same safe containment area if desired.

Vacuum Pump Kit Specac P/N GS03640

Specac supplies a vacuum pump kit P/N GS03640, consisting of a suitable vacuum pump and relevant tubing and fittings for connection to any vacuum line (**VP**) created for the system. Supplied with the kit of parts for the HTHP Cell is a specific vacuum pump connector and a corresponding silicone gasket for use of the vacuum pump P/N GS03640. (See **Fig 10.**) The connectivity of these parts is provided from the user instruction manual for the vacuum pump P/N GS03940.

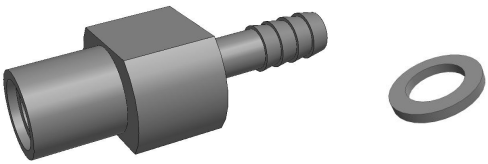


Fig 10. HTHP Cell Vacuum Pump Connector and Silicone Gasket

Pressure Certified Window Assemblies (ZnSe)

The HTHP Cell ordered as P/N GS05850 (Standard) or P/N GS05855 (Advanced; with reflectance mode capability of operation), is provided with two 1000psi pressure certified ZnSe window assemblies (**11**) for transmission and decomposition modes of operation already fitted to the HTHP Cell body (**3**). If the HTHP cell has been ordered as the Advanced version from P/N GS05855, a single **wedged** pressure certified ZnSe window assembly is also provided for use with the HTHP Cell in the reflectance mode of operation. (One of the transmission window assemblies is removed and replaced with the wedged window assembly for reflectance operation).

Note: *This single wedged pressure certified window assembly is also provided along with the HTHP Cell reflectance mode upgrade kit P/N GS05860.*

For use of the ZnSe windows up to their maximum pressure rating capability of 1000psi, the HTHP Cell **MUST** be fitted with an appropriate burst disc assembly of parts (**21**) connected as an open gas (**BD**) line to the gas outlet connection port (**15**).

However, alternative pressure certified window assemblies containing a different window material to ZnSe are available for transmission and decomposition modes of study by use of the HTHP Cell. Similarly, a pressure certified wedged window assembly can also be provided with an alternative window material for reflectance operation.

In operation of the HTHP Cell using an alternative window material to ZnSe, if the window material has a different pressure maximum capability, then an appropriate burst disc assembly of parts (**21**) for the pressure rating **MUST** also be fitted as an open gas (**BD**) line to the gas outlet connection port (**15**). The standard burst disc assembly of parts (**21**) supplied for ZnSe windows allows for operation to 1000psi, but alternative burst disc assemblies of parts (**21**) for a lower maximum pressure operation are available from Specac to be used with a particular window material.

Window Options for the HTHP Cell

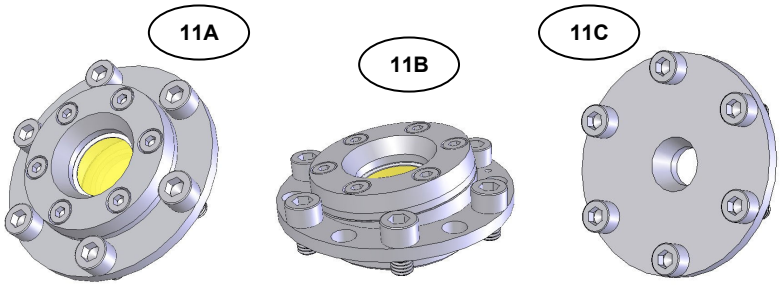


Fig 11. Pressure Certified Window Assembly Types Available for Operation of the HTHP Cell

The example images shown in **Fig 11**. are representative of the different types of pressure certified window assemblies that are available from Specac for specific uses of the HTHP Cell assembly. The use is based upon the chemical resistance and spectral wavenumber transmission range that each particular window material type can offer. The standard ZnSe transmission type (flat) window assemblies (**11**) as fitted are shown as (**11A**). For use of the HTHP Cell in the reflectance mode of operation, the wedged pressure certified window assembly with ZnSe is shown as (**11B**). For a pressure certified flat window assembly with diamond windows, this is shown as (**11C**). The diamond window housing assemblies have a smaller diameter aperture and the window material itself is much thinner than ZnSe used in the transmission and wedged window assemblies. ZnSe windows are permanently sealed into their metal housing assemblies by use of a Silicone O-ring and PTFE gasket either side of the window material surface and bolt clamping the metal housing components together, but diamond windows are fusion bonded into their metal housing part. **All types** of housing assemblies are sealed to the HTHP Cell body (**3**) using Silicone O-rings (**35**).

The following table represents the different window material options that can be supplied as both transmission (flat) and wedged window assemblies.

Window Material	Window Spectral Transmission Range (cm^{-1})	Window Dimensions (dia x thick) (mm)	Window Sealing Type	Pressure Rating (psi)
ZnSe	15,000 to 600	29.8 x 11.0	O-Ring	1000
Diamond	40,000 to 10	18.0 x 1.0	Fusion	200*
CaF_2	70,000 to 900	29.8 x 11.0	O-Ring	700*
BaF_2	60,000 to 800	29.8 x 11.0	O-Ring	500*
Sapphire	40,000 to 1700	29.8 x 3.0	O-Ring	1000
Quartz (UV)	50,000 to 3700	29.8 x 11.0	O-Ring	500*
Silicon	8,000 to 40	29.8 x 11.0	O-Ring	1000

*** Note:** *Window types with a maximum operating pressure rating different to 1000psi MUST be used with an appropriate pressure rated burst disc assembly (**21**) as part of an open (**BD**) gas outlet line. Any alternative window assemblies that can be provided by Specac for use with the HTHP Cell and operate to a pressure rating different to 1000psi will be supplied with an appropriate burst disc assembly (**21**) to use.*

Removal and Fitting of Window Assemblies

The HTHP Cell is supplied with standard ZnSe window material (**26**) within the stainless steel window housing assemblies (**11**) already fitted to the HTHP Cell body (**3**). The complete window assemblies of parts (**11**) have been factory pressure tested and certificated to 1000psi. (The test certificates for the serial numbered window assemblies are provided with the cell).

In operation and use of the HTHP Cell, these complete window assemblies (**11**) may need to be removed for sample introduction to and removal from the sample heater block assembly (**10**) within the cell chamber area of the body (**3**), but certainly they will need to be removed periodically for any inspection and cleaning.

The ZnSe windows themselves (**26**) should **never be removed** from their stainless steel housing assemblies (**11**). To ensure this cannot be achieved the six M4 x 10mm bolts (**27**) that hold the front body window housing (**28**) to the window mounting ring flange plates (**29**) are filled with a blob of black epoxy resin glue. (See **Fig 12**.)

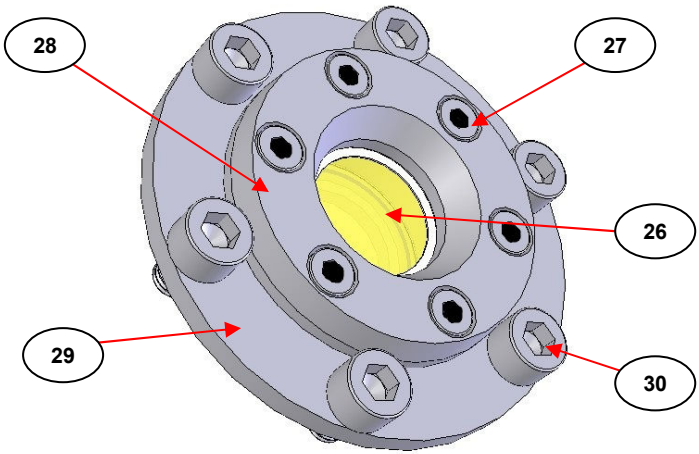


Fig 12. Pressure Certified ZnSe Window Housing Assembly (11)

Important! *For safety reasons, any disturbance to the window material (**26**) itself would require the pressure test to be repeated before use. Each time the HTHP Cell is to be used, the windows (**26**) should be visually inspected for any signs of scratches or defects. If such damage is observed the complete pressure certified window housing assembly (**11**) should not be used. It is recommended to contact Specac or to send the complete window assembly (**11**) to Specac for further examination and report.*

The complete pressure certified window housing assembly (**11**) can be removed from the HTHP Cell body (**3**) via unscrewing of the six M6 x 10mm cap head fixing bolts (**30**) without disturbing the pressure certified window seal itself. The bolts (**30**) are undone using the 5mm A/F hexagon key bit and torque wrench supplied. The complete

window housing assembly (**11**) on either side of the HTHP Cell body (**3**) may be removed for access to the sample if and when necessary.

Warning: *DO NOT, under any circumstances, attempt to undo the six smaller M4 x 10mm cap head bolts (**27**) that hold the housing assembly (**11**) together and ensure pressure tightness. The blob of black epoxy resin glue in the centre of these bolts (**27**) is to prevent them from being loosened.*

Removal of the Window Assemblies

To gain access for undoing of the six bolts (**30**) for either complete window housing assembly (**11**), it will be necessary to remove the heat shield (**31** - narrow) or (**32** - wide) that covers the window assembly (**11**). This is done by loosening the two small cap head screws (**33**) that hold the shield (these do not need to be removed completely) and sliding the shield away from these screw locations. Refitting of the shield is a reverse procedure. (See **Figs 13A and 13B.)**

Note: *The larger, wider heat shield (**32**) covers the window assembly (**11**) on the side of the HTHP Cell body (**3**) cell with the two inlet gas fitting connections (**13** and **14**) and the thermal switches (**34**) connected to the upper water cooled plate (**1**).*

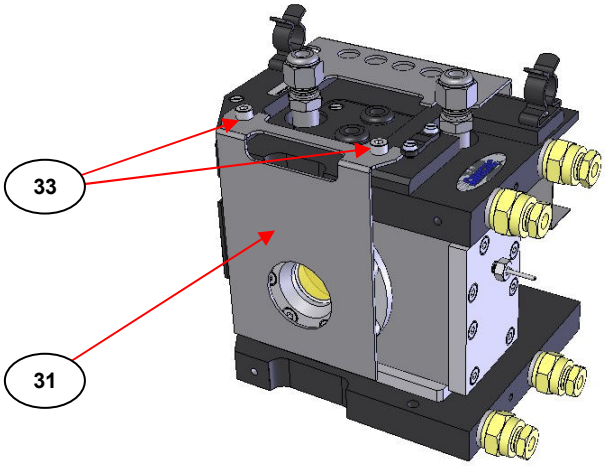


Fig 13A. Narrow Heat Shield Fitted to the HTHP Cell

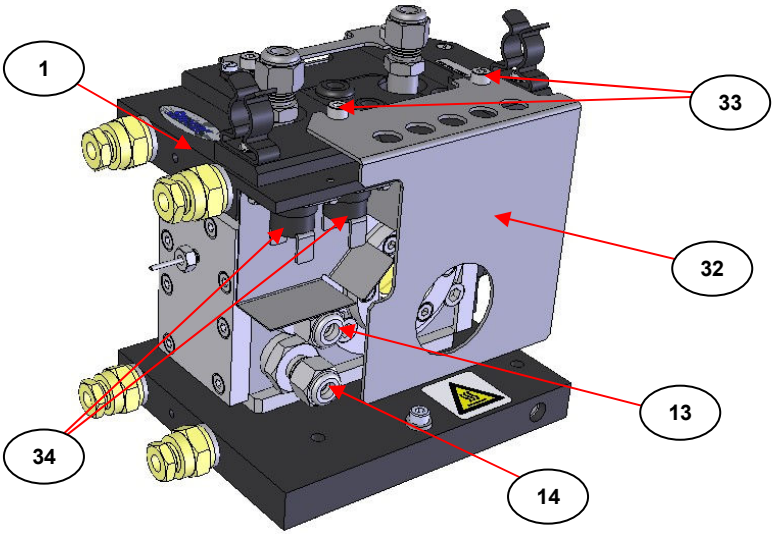


Fig 13B. Wide Heat Shield Fitted to the HTHP Cell

Fig 14. and **Fig 15.** show the stages for removal of the pressure certified window assembly (**11**) covered by the wider heat shield (**32**). With the heat shield (**32**) removed, the six fixing bolts (**30**) are undone and removed from the mounting ring flange plate (**29**).

Note: *There is no specific order needed for undoing the bolts (**30**) but on refitting the window assembly (**11**) they need to be tightened in a specific sequence for evenness of fit of the mounting ring flange plate (**29**) to the HTHP Cell body (**3**).*

With removal of the bolts (**30**) the complete window assembly (**11**) can be removed away from the HTHP Cell body (**3**). There is a silicone O-ring (**35**) that seals the window assembly (**11**) between the flange plate (**29**) and the cell body (**3**). The window assembly (**11**) may require some slight force to separate it from the cell body (**3**) if there is a good seal provided by the O-ring (**35**). Therefore, it may be necessary to prise the window assembly (**11**) away by careful use of a thin screwdriver blade around the circumference of the flange plate (**29**) and between the cell body (**3**).

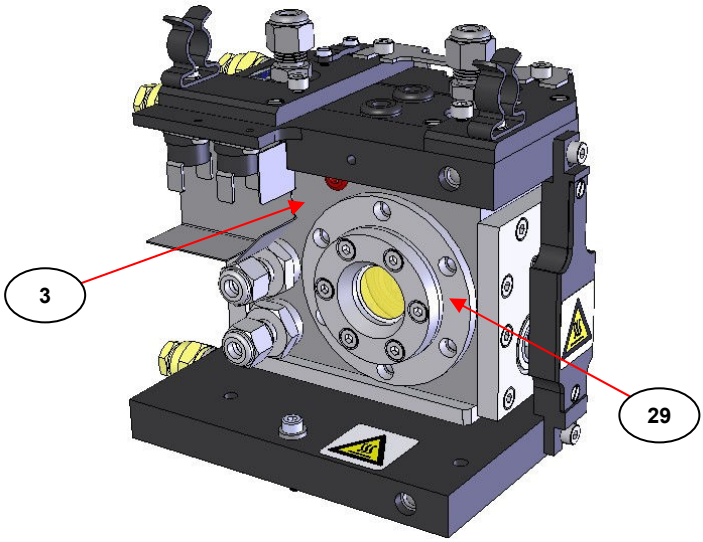


Fig 14. HTHP Cell with Wide Heat Shield (32) and Window Assembly Fixing Bolts (30) Removed

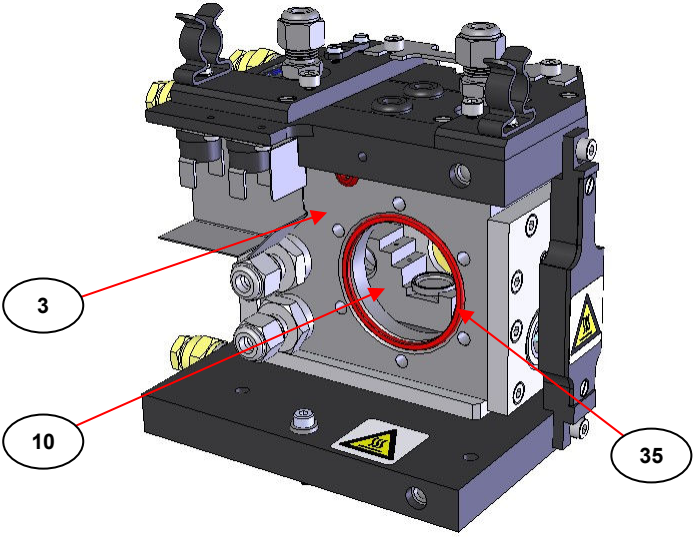


Fig 15. HTHP Cell with Wide Heat Shield (32) and Window Assembly Fixing Bolts (30) and Window Assembly (11) Removed

When the window assembly (**11**) has been removed, the silicone O-ring may come away attached to the flange plate (**29**) or it may be retained in the chamfered groove of the aperture of the HTHP Cell body (**3**) as seen in **Fig 14**. Before the window assembly (**11**) is to be refitted, the silicone O-ring seal (**35**) should be inspected for its condition and cleaned. If there is any damage or imperfections, then it should be replaced with new.

Note: *In the Essential Spares Kit P/N GS05870 of parts supplied with the HTHP Cell, there are six spare fixing bolts (**30**) if these items become lost and four Silicone O-ring seals (**35**) if these need replacing.*

Refitting of the Window Assemblies

In refitting of the window assembly (**11**) for its correct sealing, it is easier to do so with the silicone O-ring (**35**) fitted to the flange plate (**29**) as shown in **Fig 16.** rather than it being fitted into the aperture of the cell body (**3**) as shown at **Fig 15**.

Note: *The condition of the window material itself (**26**) should always be inspected when the window assembly (**11**) has been removed and cleaned accordingly. (See Section 11), Cleaning the HTHP Cell.)*

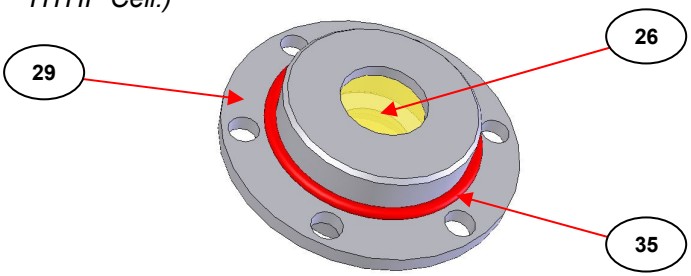


Fig 16. Window Assembly (11) with Silicone O-ring (35) Fitted

For refitting of the window assembly (**11**) the six fixing bolts (**30**) should be tightened in a specific sequence such that the window assembly (**11**) can be tightened flush and evenly to the HTHP Cell body (**3**) for correct sealing by the silicone O-ring (**35**). (See **Fig 17.**)

Carefully place the window assembly (**11**) with its silicone O-ring (**35**) fitted to the rear of the flange plate (**29**) into the aperture on the HTHP Cell body (**3**) ensuring correct alignment of the six bolt holes on the flange plate (**29**) with the threaded holes on the cell body (**3**). Fit the six fixing bolts (**30**) into position and tighten them in a “cross diagonal” sequence of (**30-1**) then (**30-4**), (**30-2**) then (**30-5**) and finally (**30-3**) then (**30-6**) until they are all finger tight. The aim is to bring the flange plate (**29**) surface into contact for an even and level fit all around its circumference when against the surface of the HTHP Cell body (**3**).

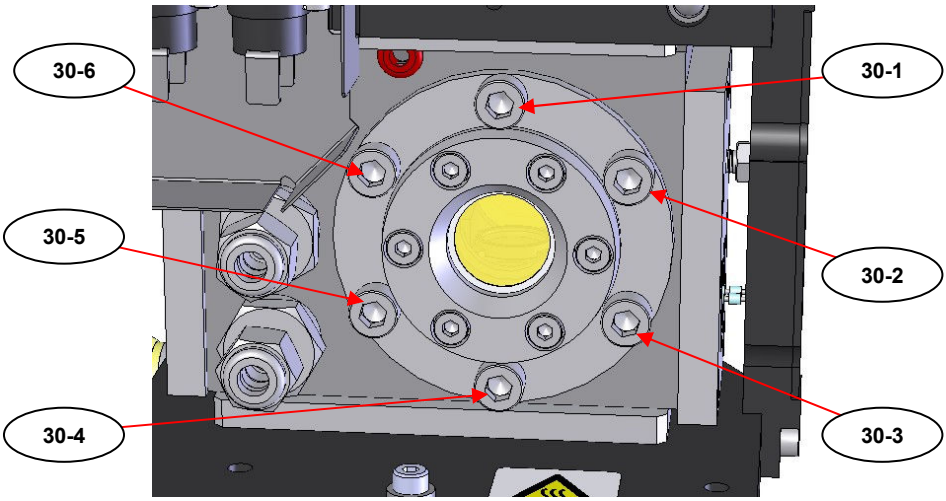


Fig 17. Window Assembly (11) Refitting Bolt Tightening Sequence

When the fixing bolts (**30**) are all finger tight, continue to tighten the bolts in the same “cross diagonal” sequence, but use the 5mm A/F adapter with 1/4" square drive connection supplied in the torque wrench to a torqued tightness of 90cNm to be set on the torque wrench itself. When all the bolts (**30**) have been tightened to this torque setting refitting of the window assembly (**11**) is complete.

Electrical Connections to Power the HTHP Cell

Power for operation of the embedded cartridge heater in the sample heater block assembly (**10**) and the heaters in HTHP Cell body (**3**) is provided by connection of the HTHP Cell to its dedicated temperature controller. (See the separate instruction manual supplied for understanding and operation of the temperature controller.)

There is a single power lead connection to the HTHP Cell which terminates in a 19 way plug for connection to the rear panel of the temperature controller. The 19 way plug simply pushes onto its corresponding connector at the rear of the temperature controller and is locked into position by rotation of the connecting collar on the plug itself. The electrical supply for all the heaters and connection to the thermocouple (**36**) in the sample heater block assembly (**10**) is routed ultimately through this single lead connection via a wiring strap (**37**) prior to passing through the end sealing plate (**38**). (See **Fig 19.**)

The Heater Block Assembly (10)

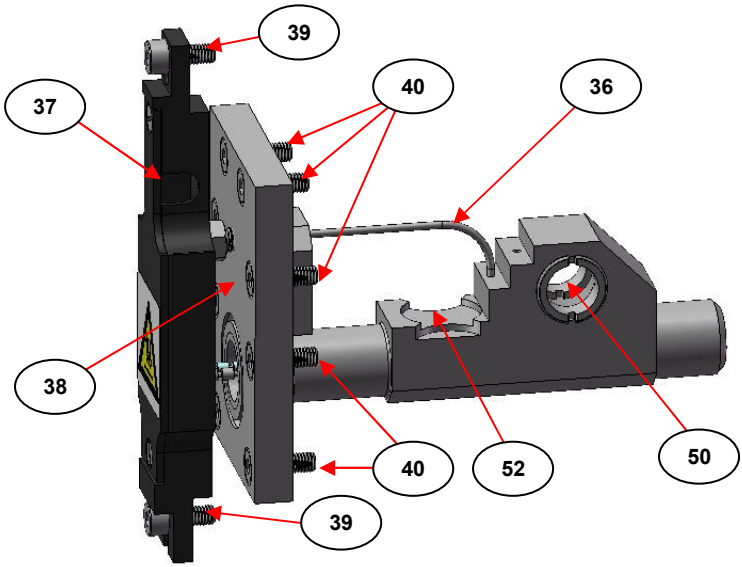


Fig 19. Heater Block Assembly (10) for Sample Introduction

Depending on the mode of operation required for use of the HTHP Cell for transmission, decomposition or reflectance (see Section 7 – Modes of Operation), the complete heater block assembly (**10**) is placed into the HTHP Cell chamber cavity by fitting of the wiring strap (**37**) and end sealing plate (**38**) to a particular side of the HTHP Cell body (**3**).

Note: *For transmission and reflectance modes of operation the heater block assembly (**10**) is fitted on one side of the HTHP Cell chamber body (**3**) and for the decomposition mode is placed on the other.*

The wiring strap (**37**) is affixed into position on either side of the HTHP Cell chamber using two M4 x 10mm stainless steel cap head screws (**39**). The two fixing screws (**39**) are screwed into the upper (**1**) and lower (**2**) water cooled plates. (See **Fig 19.**) The sealing end plate (**38**) is affixed into position using eight M4 x 10mm High Tensile plated cap head screws (**40**) that screw into the end of the cell body (**3**). Similar to the pressure certified window assemblies (**11**), the end plate (**38**) forms a seal by use of a silicone O-ring (**35**) that fits into a grooved recess in the side of the HTHP Cell body (**3**). (See **Fig 20B.**)

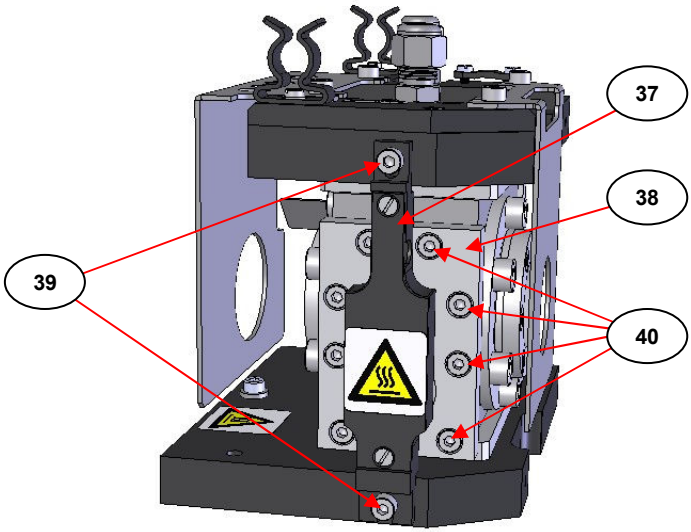


Fig 20A. HTHP Cell Heater Block Assembly (10) in Position on HTHP Cell Body (3) for Decomposition Mode of Operation

Removal of the Heater Block Assembly (10)

To remove the heater block assembly (**10**) from the HTHP Cell chamber cavity, the two wiring strap screws (**39**) are undone first and placed carefully to one side for re-use. (See **Fig 20A**.) (Do not mix these two screws (**39**) with the eight different screws (**40**) that are used for the end plate (**38**).) Now undo and remove the eight fixing screws (**40**) of the end plate (**38**). The wiring strap (**37**) will still be in position covering the end plate (**38**) as it is physically connected by the power cable connection, so it may require slight movement of the wiring strap (**37**) part to gain easier access to the top two screw (**40**) fixings for their removal.

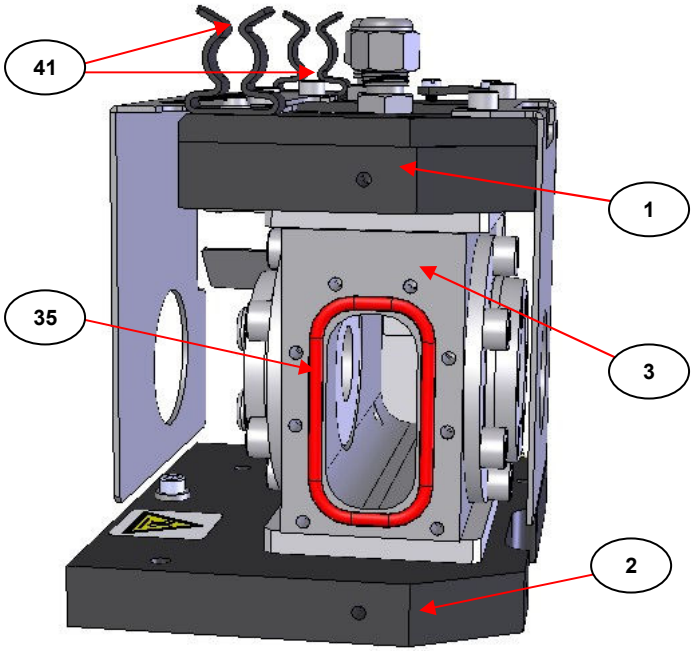


Fig 20B. HTHP Cell Heater Block Assembly (10) Removed from HTHP Cell Body (3) for Decomposition Mode of Operation

When all of the fixing screws (**39**) and (**40**) have been removed, the heater block assembly (**10**) can be pulled away and out from the HTHP Cell chamber by manipulation of the end plate (**38**). (See **Fig 20B.**) The silicone O-ring seal (**35**) may be retained in its groove on the end face of the cell body (**3**) or it may come away attached to the surface of the end plate (**38**). Similar to the same type of silicone O-ring seal (**35**) as used with the pressure certified window assemblies (**11**), when removing the end plate (**38**) and for subsequent re-fitting, the silicone O-ring seal (**35**) should be inspected for its condition and cleaned. If there is any damage or imperfections, then it should be replaced with new.

Note: *In the Essential Spares Kit P/N GS05870 of parts supplied with the HTHP Cell, there are two spare fixing screws (**39**) and eight spare fixing screws (**40**) if these items become lost and four Silicone O-ring seals (**35**) if these need replacing.*

There is an inline straight 6 way plug connection coupling located on top of the upper water cooled plate (**1**) and supported between two spring clips (**41**) for the power cable, that allows for separation (breakage of the connection) of the main power cable from the controller plug connection and before attachment to the wiring strap (**37**) and the end plate (**38)** of the sample heater block assembly (**10**). The inline straight 6 way plug connection consists of a male (plug) and female (socket) coupling that are simply pulled apart to break the power connection. When repositioning the heater block assembly (**10**) from one side of the HTHP Cell body (**3**) to the other, it is necessary to break the power line via this 6 way plug connection to accommodate for the length of cable attached to the heater block assembly (**10**) from a refitting. Separation of the heater block assembly (**10**) from its power cable also enables the heater block assembly (**10**) alone to be moved to a safe place for sample introduction should this be necessary. The power line connection is remade by re-connection of the 6 way plug and socket parts and the complete cable assembly is repositioned back into the two support spring clips (**41**).

Note: *The direction of the power cable assembly and 6 way plug connection, as supported between the spring clips (**41**), will be specific to the positioning of the heater block assembly (**10**) as fitted for the mode of operation.*

Non-Heater Block End Plate of the HTHP Cell Body

There is a corresponding end plate assembly (**42**) that is fitted by eight fixing screws (**40**) to the end of the HTHP Cell body (**3**) directly opposite the wiring strap (**37**) and end plate (**38**) parts of the heater block assembly (**10**). (See **Fig 21.**) Similar to the end plate (**38**) of the heater block assembly (**10**), the end plate (**42**) is sealed to the HTHP Cell body (**3**) by use of a silicone O-ring (**35**) when the fixing screws (**40**) have been tightened. (See **Fig 20B.**)

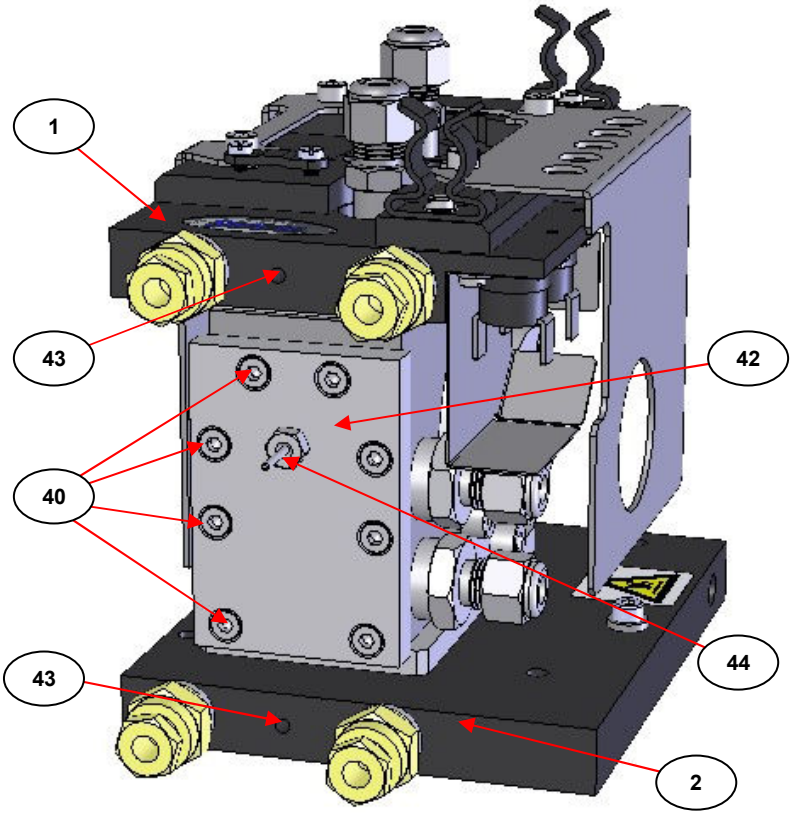


Fig 21. End Plate of the HTHP Cell Opposite to the Wiring Strap and End Plate of the Heater Block Assembly (10)

Whenever the heater block assembly (**10**) is to be removed from the HTHP Cell body (**3**) to change for the mode of operation from transmittance or reflectance to decomposition (and vice versa), the end plate (**42**) must also be removed by undoing of the eight fixing screws (**40**) and swapped over to fit back onto the opposite side of the HTHP Cell body (**3**) from the removed heater block assembly (**10**) of parts. On either end of the upper (**1**) and lower (**2**) water cooling plates there are screw fixing holes (**43**) to take the fixing screws (**39**) of the wiring strap (**37**) when the end plate (**38**) of the heater block assembly (**10**) is to be positioned on the particular side end of the HTHP Cell body (**3**) corresponding to the mode of operation to use.

Refitting of End Plates (38) and (42)

Similar to refitting of a pressure certified window assembly (**11**) for evenness of fit and sealing of the silicone O-ring (**35**) (see pages 43 and 44), the eight fixing screws (**40**) for both end plates (**38**) and (**42**) need to be tightened in a specific sequence when refitting the end plates to the HTHP Cell body (**3**). (See **Fig 22.**)

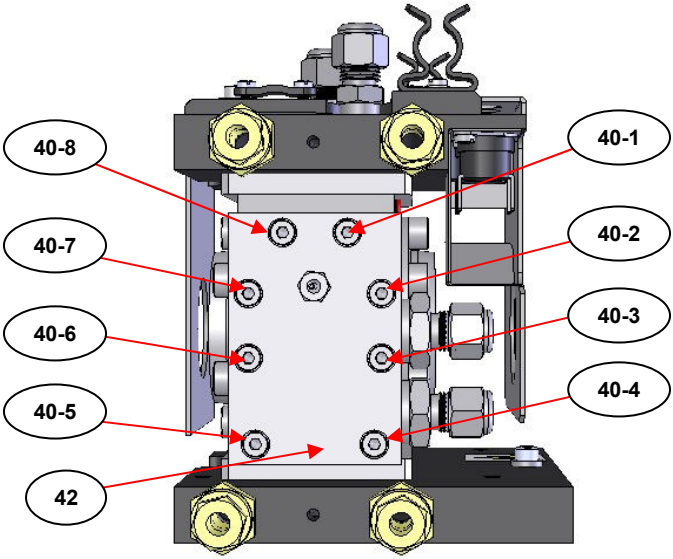


Fig 22. End Plate (42) Fixing Screw Tightening Sequence

Taking the end plate (**42**) as an example of the two end plates to refit, ensure that the silicone O-ring (**35**) is of good quality and is fitted correctly into its groove on the end face of the HTHP Cell Chamber body (see **Fig 20B.**) Offer up the end plate (**42**) ensuring correct alignment of the eight screw holes on the end plate (**42**) with the threaded holes on the cell body (**3**). Fit the eight fixing screws (**40**) into position and tighten them using the 3mm adapter with the torque wrench supplied in a “cross diagonal” sequence of (**40-1**) then (**40-5**), (**40-2**) then (**40-6**), (**40-3**) then (**40-7**) and finally (**40-4**) then (**40-8**) until they are all just hand tight. The aim is to bring the end plate (**29**) surface into contact for an even and level fit all around its circumference when against the surface of the HTHP Cell body (**3**). Then continue in this sequence to tighten the screws (**40**) to a set torque value of 60cNm from the torque wrench.

Additional Monitoring Thermocouple

An additional monitoring thermocouple assembly is provided with the HTHP Cell’s kit of parts if you wish to take an independent temperature measurement of a sample within the HTHP Cell for **any** mode of operation. (This may be required to measure any temperature difference between the heater block assembly’s (**10**) own thermocouple (**36**) reading for particular temperature control.)

In the centre of the end plate (**42**) there is a blocked off aperture hole consisting of a 1/16” Swagelok union and ferrule set fitted with a blanking rod (**44**). (See **Fig 21.**) If the additional monitoring thermocouple is to be used, it is introduced into the HTHP Cell chamber through the aperture hole of the end plate (**42**). To use the monitoring thermocouple the blanking rod is removed from the end plate (**42**) by undoing the Swagelok union (**44**). The blanking rod and its ferrule are removed from the Swagelok union (**44**) and the monitoring thermocouple rod is placed into the Swagelok union instead. (Make sure the metal tip of the thermocouple rod passes through the hexagonal "nut" end of the Swagelok union first). Take one of the spare 1/16” ferrules provided with the HTHP Cell kit of parts to place around the thermocouple rod tip and to seal against the Swagelok union (**44**) when the thermocouple rod tip is to be sealed into the end plate (**42**).

Before sealing the ferrule against the thermocouple by tightening of the Swagelok union (**44**) back into the end plate (**42**), leave enough of the thermocouple tip to pass into the HTHP Cell chamber such that it will make contact with the sample area or sample that you wish to monitor. It might be easier to gauge the amount of thermocouple tip needed to protrude into the cell chamber by removal of a ZnSe sealed window assembly (**11**) to see inside the cell chamber.

When this spare monitoring thermocouple is positioned correctly and the HTHP Cell is made ready for operation, an independent monitoring device is required to connect to the plug of the monitoring thermocouple to obtain any temperature measurement readings (values) from use.

6. HTHP Cell Baseplate Installation

Once you have familiarised yourself with the previous aspects of the HTHP Cell for water cooling connectivity, gas line plumbing, electrical connectivity, removal of the window assemblies (**11**) and the heater block assembly (**10**), then the HTHP Cell can be considered how it is installed into the spectrometer sample compartment via a specific baseplate assembly.

For the **transmission** and **decomposition** modes of operation the HTHP Cell is installed into the sample compartment on a particular **transmission** type baseplate assembly for the spectrometer make and model. This **transmission** type baseplate is supplied with the standard HTHP Cell when ordered against P/N GS05850. Please refer to Section 15) of this user manual for the particular transmission baseplate that is supplied and how it is fitted into the sample compartment of the specific spectrometer.

For the **reflectance** mode of operation the HTHP Cell is installed into the sample compartment using an alternative **reflectance** type baseplate assembly for the spectrometer make and model. This **reflectance** type baseplate is supplied with the HTHP Cell along with the transmssion type of baseplate if ordered as the Advanced version against P/N GS05855 and on its own with the HTHP Cell Reflectance Mode Upgrade Kit against P/N GS05860. Details for installation of the reflectance type baseplate are found within the user instruction manual for the HTHP Cell Reflectance Mode Upgrade Kit P/N GS05860.

Fixing the HTHP Cell to the Baseplate

For **transmission** and **decomposition** modes of operation the HTHP Cell is placed correctly over the transmission baseplate and is connected by an M4 cap head fixing screw (**45**) tightened to the central pillar (**46**) of the line of three pillars or the screw hole location of the baseplate. On the underside of the lower cooling plate (**2**) there are two recessed location holes (**47**) (one slot and one hole) that engage with the two pins (**48**) on the two pillars either side of the central fixing pillar (**46**) of the baseplate. (See **Figs 23.** and **24.**)

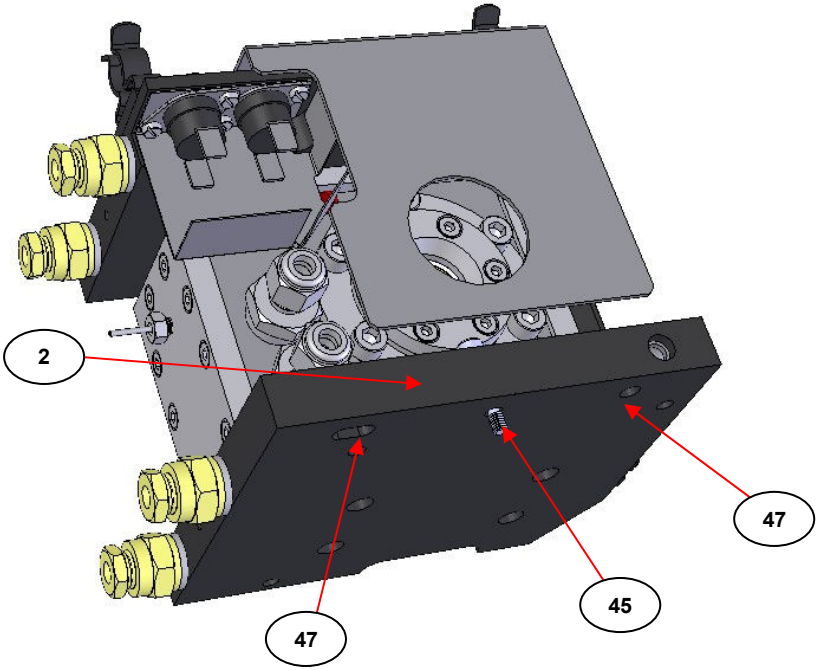


Fig 23. HTHP Cell Baseplate Fixing Screw and Location Slot Holes

The single pillar (**49**) of the baseplate supports the weight of the HTHP cell when fitted into position.

Note: *It is usually necessary to fit the transmission baseplate assembly first into the spectrometer sample compartment and then install the HTHP Cell onto the baseplate. Depending upon the specific sample compartment size and space available, it may also be necessary to have made initial gas line pipe connections at the side inlet ports (**13**) and (**14**) to be routed up and away from the sample compartment prior to installation of the HTHP cell onto its baseplate as access for connectivity may be impaired from the HTHP Cells installation.*

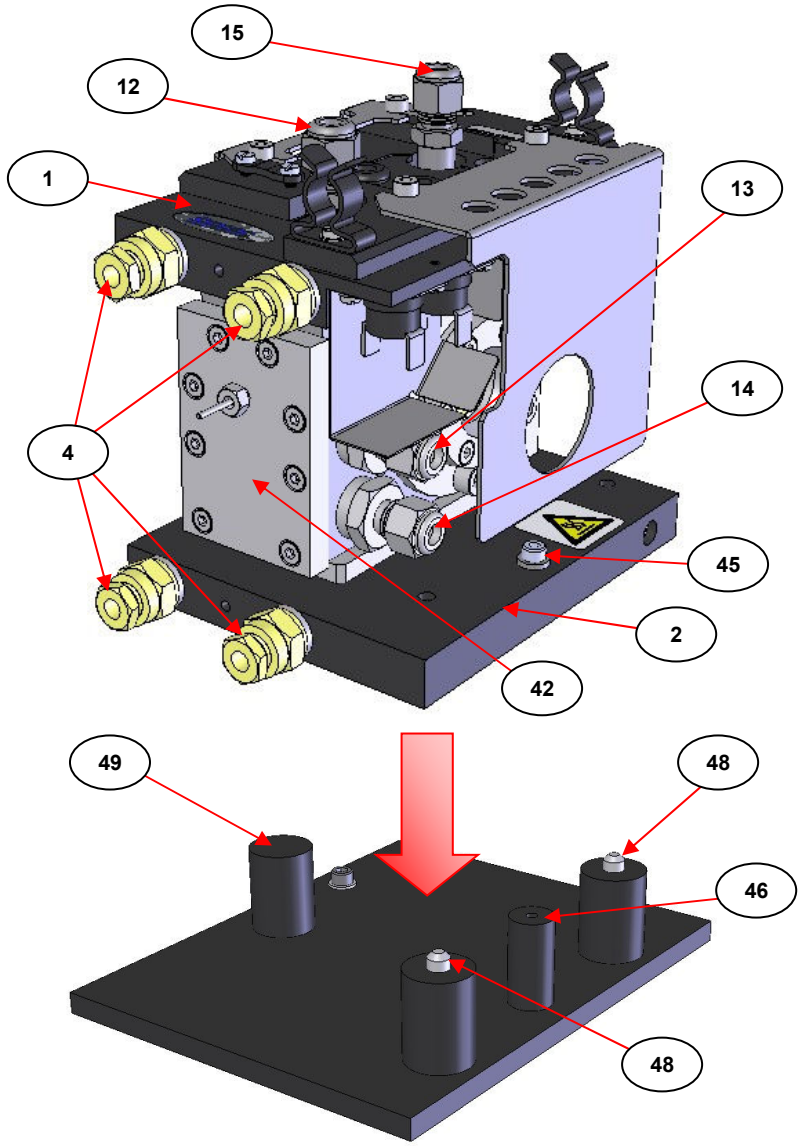


Fig 24. Fixing of the HTHP Cell to the Transmission Baseplate

When the HTHP Cell has been fitted onto the transmission baseplate correctly, the orientation of the HTHP Cell will be upright as shown in **Fig 24.** The water fitting connections (**4**) on both the top (**1**) and lower (**2**) water cooled plates will be facing out towards the front of the spectrometer sample compartment for ease of access to introduce the water cooling tubing.

The top gas inlet port (**12**) will be towards the front of the sample compartment and the outlet gas port (**15**), also at the top of the cell, will be towards the back. The two side gas inlet ports (**13**) and (**14**) will be facing towards the right hand side of the sample compartment, but are towards the front of the HTHP Cell body (**3**) which provides relatively easy access for any gas tubing connectivity (if required) whilst the HTHP Cell is installed within the sample compartment of the spectrometer system.

The transmission baseplate mount provides correct spatial positioning of the HTHP Cell for the beam of IR radiant light from the spectrometer system to pass centrally through the pressure certified window assemblies (**11**) in a 180° (straight through) orientation from the source to detector for either a left to right or right to left beam passage through the sample compartment. It depends on which end of the HTHP Cell body (**3**) the heater block assembly's (**10**) end plate (**38**) and wiring strap (**37**) have been affixed that determines if the HTHP Cell has been installed on its baseplate for transmission or decomposition studies to the HTHP Cell. As shown in **Fig 24.** the end plate (**42**) is seen fitted to the HTHP Cell body (**3**) on the end that will be facing outwards from the spectrometer sample compartment and so from this build configuration the HTHP Cell is used for the **decomposition** mode of operation.

Note: *If the end plate (**42**) is fitted towards the front of the sample compartment, the **decomposition** mode is set and if end plate (**38**) (for the heater block assembly (**10**)) and the wiring strap (**37**) are fitted towards the front of the sample compartment, the **transmission** mode is set.*

7. Loading a Sample in the HTHP Cell for the Transmission Mode of Operation

The HTHP Cell is factory assembled and provided in the **transmission** mode of operation build configuration if ordered as the standard version under P/N GS05850 or Advanced version (with reflectance mode capability) as P/N GS05855.

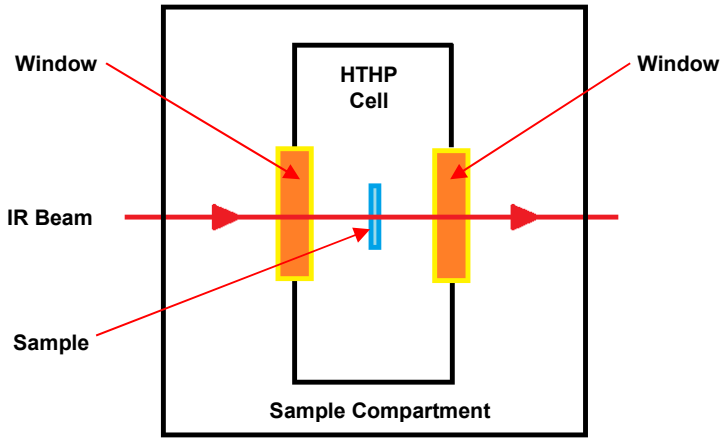


Fig 25. Schematic for HTHP Cell Transmission Mode of Operation

For a **transmission** mode of operation the heater block assembly (**10**) has been fitted into the HTHP Cell body (**3**) such that the wiring strap (**37**) and end plate (**38**) parts will be projected towards the front of the sample compartment and so the heater block assembly (**10**) can be removed from the HTHP Cell body without the requirement to remove the **complete** HTHP cell accessory (including any water tubing, gas tubing and electrical power connections) away from the sample compartment and baseplate installation to introduce a solid sample for measurement. It may be preferable to introduce a sample into the HTHP Cell this way instead of removing a window assembly (**11**).

Sample Introduction to the Heater Block Assembly

For the **transmission** mode of sampling, a solid sample type is placed into the 13mm diameter aperture hole (**50**) of the heater block assembly (**10**). (See **Fig 19.** and **Fig 26.**)

Note: *This way of sample introduction is also needed for the **reflectance** mode of operation. Please see the specific sample introduction details in the user instruction manual for the HTHP Cell Reflectance Mode Upgrade Kit P/N GS05680.*

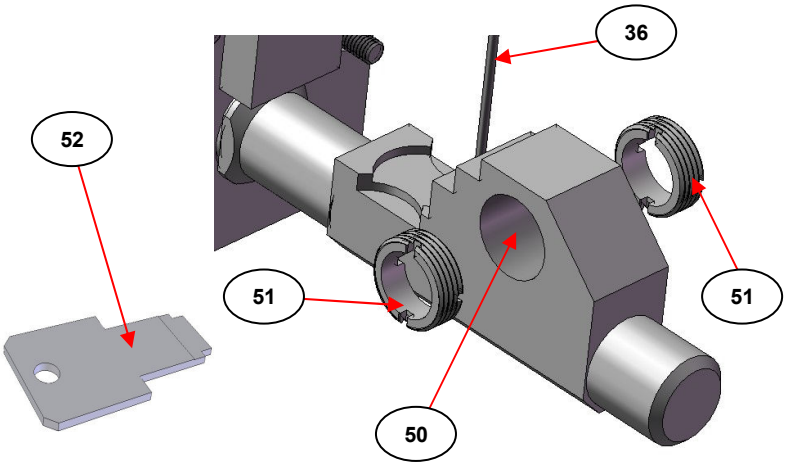


Fig 26. Heater Block Assembly (10) Transmission Sample Position

The length (or width of the heater block part) of the transmission aperture hole (**50**) to accommodate a solid sample of 13mm diameter is 16mm. The aperture hole (**50**) is threaded throughout its length and this allows for the two keep rings (**51**) supplied to be screwed into place from either side of the aperture hole (**50**) to “sandwich” a 13mm diameter solid sample disc between them. Each keep ring (**51**) has an aperture of 11mm diameter and is 5mm thick and so when the two keep rings (**51**) are screwed into the 13mm diameter aperture hole (**50**) and their outside faces are flush with the edge end of the heater block part, the maximum thickness of a 13mm diameter sample disc that can be held between the two keep rings (**51**) is circa 6mm.

To actually place a 13mm diameter sample disc into position within the heater block assembly (**10**), screw one of the keep rings (**51**) into the aperture hole (**50**) from one side using the supplied keep ring key (**52**). (The key (**52**) locates into the notches cut in the keep rings (**51**) to turn them.)

Note: *Depending on the actual thickness of the sample disc to be used, try to introduce the keep ring (**51**) being fitted into the aperture hole (**50**) to a depth that will allow the sample disc to be sandwiched as centrally as possible within the aperture hole (**50**) between the two keep rings (**51**).*

When this first keep ring has been screwed into the aperture hole to a sufficient depth, take the 13mm diameter sample disc and introduce it very carefully into the aperture hole (**50**) from the opposite side of the installed keep ring (**51**). The sample disc should be made to rest carefully and flush against the installed keep ring's (**51**) circumference surface in the aperture hole (**50**). Take the second keep ring (**51**) and carefully screw this into the aperture hole (**50**) from the same side as the sample was introduced until it contacts the sample disc to hold it secure within the aperture hole (**50**) of the sample block holder (**10**) between the two keep rings (**51**).

Note: *13mm diameter sample discs (from an appropriate sample type) may be prepared for use in the HTHP Cell from a Specac 13mm evacuable pellet die assembly P/N GS03000 that has been used within an appropriate pressing system (e.g. Specac Atlas™ Manual Hydraulic press P/N GS15011). (For more details contact Specac.)*

Loading a Sample by Removal of the Heater Block Assembly (10) from the HTHP Cell Body (3)

Although it is necessary to mount a particular solid sample type up to 13mm diameter and a thickness of circa 6mm within the aperture hole (**50**) of the heater block assembly, there are two possible ways that the aperture port (**50**) can be accessed for introduction and change of a sample. One way is by removal of the heater block assembly (**10**) from

the HTHP Cell body (**3**). The alternative is to remove one or both window housing assembly's (**11**) from the HTHP Cell Body (**3**).

Note: *Depending upon the various connections for water, gas and electrical supplies for the local system set up for operation of the HTHP Cell, it may be easier and advantageous leaving the HTHP Cell in situ installed into the sample compartment of the spectrometer system and to change over a sample by removal of the heater block assembly (**10**) from the HTHP cell body (**3**) rather than having to remove the HTHP Cell from its baseplate and away from the sample compartment to remove a window housing assembly (**11**).*

For a transmission mode of operation, if you wish to install a sample disc into the aperture hole (**50**) by removal of the heater block assembly (**10**) from the HTHP Cell body (**3**), then it is not necessary to remove the heat shields (**31**) and (**32**). As stated in the above note, it may also be possible to change a sample by this method without having to remove the HTHP Cell from the spectrometer, if the HTHP Cell is already installed into a sample compartment.

In the transmission mode of operation, the wiring strap (**37**) and end plate (**38**) of the heater block assembly (**10**) will be facing outwards from the sample compartment of the spectrometer for easy access to remove. Follow the procedures for its removal and subsequent refitting described in Section 5, pages 45 to 51 of this user instruction manual.

Important: *Any sample change should only be done when the HTHP Cell sample heater block assembly (**10**) and the HTHP Cell body (**3**) parts have cooled down sufficiently (to about 40ºC) for them to be handled safely.*

If the HTHP Cell has been removed from the spectrometer to change the sample by this method of removal of the heater block assembly (**10**), after the sample introduction has been completed, relocate the HTHP Cell into position by securing it onto its baseplate inside the spectrometer and re-establish any necessary gas line connections if these were broken to allow for the HTHP's Cell removal.

The HTHP Cell is now ready to be used for a **transmission** mode of operation with a suitable sample positioned accordingly that can be subjected to a range of different temperatures and environmental gas and pressure conditions and to enable the collection of Infrared spectra.

Loading a Sample by Removing a Window Housing Assembly (11)

Note: *This procedure is possible only if there is sufficient flexibility in the gas connection lines (inlet and outlet ports) to enable the HTHP Cell to be removed from the spectrometer. If the gas line tubing is coiled for its length and allows for flexibility in movement, then the HTHP Cell may be removed from the sample compartment to gain access to a window housing assembly (**11**) without any need for gas line disconnection.*

When the HTHP Cell has been removed from the sample compartment of the spectrometer, depending upon which window housing assembly (**11**) is to be removed to gain access to the aperture hole (**50**) of the sample heater block assembly (**10**), the corresponding heat shield (**31**) or (**32**) on the HTHP Cell must be removed first by loosening the retaining screws (**33**).

With removal of a window housing assembly (**11**) it will be possible to introduce the keep ring key (**52**) into the aperture hole (**50**) to unscrew and remove the keep ring (**51**) on this side (face) of the heater block assembly (**10**) to subsequently gain access for removal of the sample disc for changeover.

Follow the procedures for the window housing assembly('s) (**11**) removal and subsequent refitting described in Section 5, pages 40 to 44 of this user instruction manual.

Note: *If successive samples for analysis do not vary much for their thickness (e.g. by less than 1mm), then only one window housing assembly (**11**) and one keep ring (**51**) will need to be removed for each change of sample such that the sample disc is always kept as central as possible within the aperture hole (**50**) between the two keep rings (**51**).*

Important: *Any sample change should only be done when the HTHP Cell sample heater block assembly (**10**) and the HTHP Cell body (**3**) parts have cooled down sufficiently (to about 40ºC) for them to be handled safely.*

After the sample introduction has been completed and the window housing assembly('s) (**11**) has(have) been refitted, reattach the heat shield(s) (**31**) and/or (**32**).

Then relocate the HTHP Cell back into position by securing it onto its transmission baseplate inside the spectrometer and re-establish any necessary gas line connections if these were broken to allow for the HTHP's Cell removal.

The HTHP Cell is now ready to be used for a **transmission** mode of operation with a suitable sample positioned accordingly that can be subjected to a range of different temperatures and environmental gas and pressure conditions and to enable the collection of Infrared spectra.

8. Loading a Sample in the HTHP Cell for the Decomposition Mode of Operation

When operating the HTHP Cell in **decomposition** mode, the decomposition sample cup recess area (**53**) of the heater block assembly (**10**) is presented just below the radiant IR beam path of the HTHP Cell that passes through the window housing assemblies (**11**). (See Fig 27. for the schematic of decomposition mode of operation).

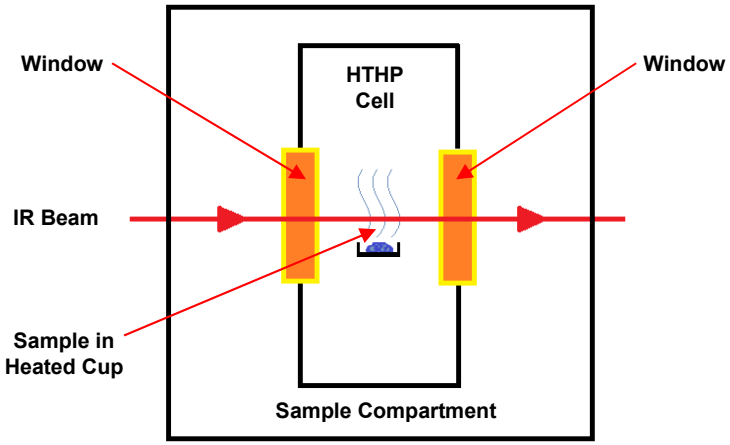


Fig 27. Schematic for HTHP Cell Decomposition Mode of Operation

The heater block assembly (**10**) is placed into the HTHP Cell body (**3**) at the end opposite to the water fittings (**4**) fixed to the upper (**1**) and lower (**2**) water cooling plates. A decomposition sample cup (**54**) is inserted into its location recess (**53**) on the heater block assembly (**10**). (See **Fig 28.**) (Cup (**54**) also shown in recess (**53**) position.)

Similar to operating the HTHP Cell in **transmission** mode, a sample can be loaded into the decomposition cup (**54**) either by removing the heater block assembly (**10**) from the cell body (**3**) or by removing one

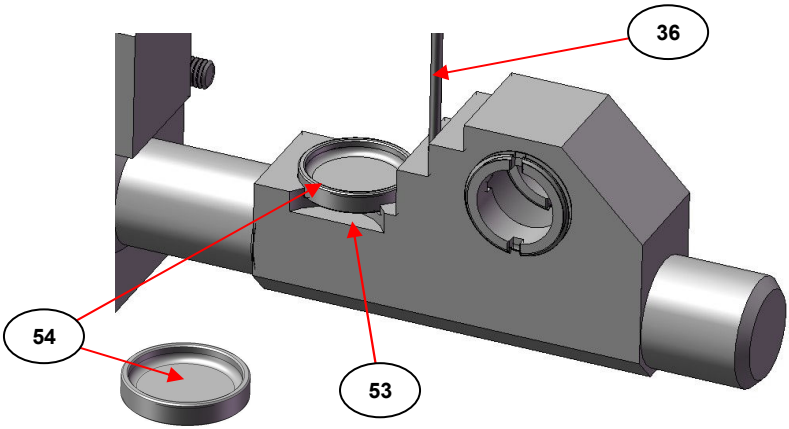


Fig 28. Heater Block Assembly (10) Decomposition Sample Position (53) with Sample Cup (54) in Place

window housing assembly (**11**) from the cell body (**3**). However, because the heater block assembly (**10**) is fitted into the side of the HTHP Cell body (**3**) that is not projecting towards the front of the sample compartment of the spectrometer, if the heater block assembly (**10**) is to be removed for sample introduction, then the complete HTHP Cell accessory **will require removal away from its baseplate installation** within the spectrometer itself to gain access. The HTHP Cell also requires removal from the baseplate installation if a window housing assembly (**11**) is to be removed for sample introduction and access.

To a large extent the favoured procedure for sample introduction and changeover for the **decomposition** mode of operation will depend upon the sample type to be introduced and relative ease of access to the sampling position (**53**). Solid lumps of material may be more easily placed into a decomposition sample cup (**54**) fitted in the sampling position (**53**) of the heater block assembly (**10**) and already within the HTHP Cell body for the decomposition mode of operation, using a spatula or forceps with the removal of a window housing assembly

(**11**). A sticky or viscous material may be more easily applied with the heater block assembly (**10**) being removed from the HTHP Cell body (**3**) if there is risk of contaminating the surrounding area with any excess spilt sample that is tricky to transfer, although a similar way of introducing the sample into the decomposition pan (**54**) already fitted into position (**53**) and from a window housing assembly (**11**) being removed may be suitable.

Loading a Sample by Removal of the Heater Block Assembly (10) from the HTHP Cell Body (3)

For a **decomposition** mode of operation, if you wish to install a decomposition sample cup (**54**) (with sample) into its recess position (**53**) by removal of the heater block assembly (**10**) from the HTHP Cell body (**3**), then it is not necessary to remove the heat shields (**31**) and (**32**).

In the decomposition mode of operation for the HTHP Cell, the wiring strap (**37**) and end plate (**38**) of the heater block assembly (**10**) will be facing towards the rear of the sample compartment of the spectrometer and so to gain access for its removal the complete HTHP cell must first be removed from its baseplate installation.

Note: *This procedure is possible only if there is sufficient flexibility in the gas connection lines (inlet and outlet ports) to enable the HTHP Cell to be removed from the spectrometer. If the gas line tubing is coiled for its length and allows for flexibility in movement, then the HTHP Cell may be removed from the sample compartment to gain access to a window housing assembly (**11**) without any need for gas line disconnection.*

When the HTHP Cell has been removed from its baseplate installation, follow the procedures for the heater block assembly (**10**) removal and subsequent refitting described in Section 5, pages 45 to 51 of this user instruction manual.

Important: *Any sample change should only be done when the HTHP Cell sample heater block assembly (**10**) and the HTHP Cell body (**3**) parts have cooled down sufficiently (to about 40ºC) for them to be handled safely.*

After the sample introduction has been completed from the sample cup (**54**) and sample being placed into the heater block assembly (**10**) and all of these component parts have been carefully replaced back into the HTHP Cell body (**3**), relocate the HTHP Cell back into position in the spectrometer by securing it onto its transmission baseplate. Re-establish any necessary gas line connections if these were broken to allow for the HTHP's Cell removal.

The HTHP Cell is now ready to be used for a **decomposition** mode of operation with a suitable sample positioned accordingly that can be subjected to a range of different temperatures and environmental gas and pressure conditions and to enable the collection of Infrared spectra of any evolved vapours from the sample that will fill the HTHP Cell chamber.

Loading a Sample by Removing a Window Housing Assembly (11)

Note: *This procedure is possible only if there is sufficient flexibility in the gas connection lines (inlet and outlet ports) to enable the HTHP Cell to be removed from the spectrometer. If the gas line tubing is coiled for its length and allows for flexibility in movement, then the HTHP Cell may be removed from the sample compartment to gain access to a window housing assembly (**11**) without any need for gas line disconnection.*

When the HTHP Cell has been removed from the sample compartment of the spectrometer, depending upon which window housing assembly (**11**) is to be removed to gain access to the decomposition sample cup's (**54**) recessed sampling position (**53**) on the heater block assembly (**10**), the corresponding heat shield (**31**) or (**32**) on the HTHP Cell must be removed first by loosening the retaining screws (**33**).

With removal of a window housing assembly (**11**) it will be possible to gain access for removal to introduce or changeover a decomposition sample cup (**54**) and a sample into the sampling position (**53**).

Follow the procedures for the window housing assembly (**11**) removal and subsequent refitting described in Section 5, pages 40 to 44 of this user instruction manual.

Important: *Any sample change should only be done when the HTHP Cell sample heater block assembly (**10**) and the HTHP Cell body (**3**) parts have cooled down sufficiently (to about 40ºC) for them to be handled safely.*

After the sample introduction of decomposition cup (**54**) and sample has been completed and the window housing assembly (**11**) has been refitted, reattach the heat shield (**31**) or (**32**).

Then relocate the HTHP Cell back into position by securing it onto its transmission baseplate inside the spectrometer and re-establish any necessary gas line connections if these were broken to allow for the HTHP's Cell removal.

The HTHP Cell is now ready to be used for a **decomposition** mode of operation with a suitable sample positioned accordingly that can be subjected to a range of different temperatures and environmental gas and pressure conditions and to enable the collection of Infrared spectra of any evolved vapours from the sample that will fill the HTHP Cell chamber.

9. Loading a Sample in the HTHP Cell for the Reflectance (Specular) Mode of Operation

To operate the HTHP Cell in the **reflectance** mode, it must be mounted on a special optical **reflectance** (**OB**) baseplate rather than the standard **transmission** baseplate assembly.

Note: *For the reflectance mode of operation, the HTHP Cell itself must be constructed for mounting of the heater block assembly (**10**) within the HTHP Cell body (**3**) as used for the **transmission** mode of operation. (See Section 7, page 57.)*

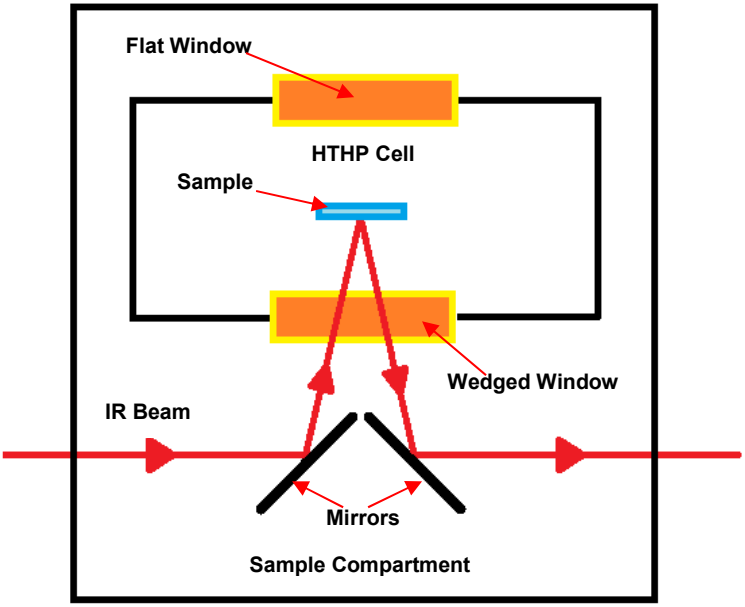


Fig 29. Schematic for HTHP Cell Reflectance Mode of Operation

For the **reflectance** mode of operation a beam of IR light is projected from **below** the HTHP Cell through a **wedged** window housing assembly (**11B**) fitted to the HTHP Cell body in place of one of the plane (flat) window housing assemblies (**11A**). (See page 37 for window housing assembly types). The HTHP Cell is positioned accordingly on a **complete** reflectance baseplate assembly consisting of the optical baseplate (**OB**) on an adapter baseplate (**AB**) (with reflecting mirrors), to direct the IR beam of light into and away from the HTHP Cell. The HTHP Cell is positioned on its side on the optical baseplate (**OB**) to present and orientate the wedged window assembly (**11B**) at the base of the HTHP Cell and this single wedged window assembly (**11B**) allows for both the passage of the inlet (incident) and outlet (specularly reflected) component beams of light to and from the surface of a solid sample 13mm diameter disc held in the heater block assembly (**10**) within the transmission position aperture hole (**50**). The specularly reflected light off of the sample surface passes back through the wedged window assembly (**11B**) to the output mirror of the adapter baseplate (**AB**) and towards the detector of the spectrometer system. The angle of incidence for the specularly reflected light at the sample surface is nominally at 15 degrees to the perpendicular.

Depending upon the particular spectrometer system and whether it is a left to right or right to left beam direction from source to detector (as the light passes through the spectrometer sample compartment when viewed from above and the front of the instrument), along with an appropriate mounting orientation of the wedged window assembly (**11B**), the HTHP Cell also needs to be adapted with particular fixing side plate/bracket parts for attachment to the complete **reflectance** baseplate assembly and alternative water flow connection fittings (**4**) for a left to right beam spectrometer system. (See “banjo fittings Section 5, page 26 of this user instruction manual.)

The appropriate parts are supplied in the Reflectance Mode Upgrade Kit P/N GS05860 along with a user instruction manual to follow for installation and operation of the HTHP Cell in the **reflectance** mode. (This kit of parts (GS05860) and appropriate user instruction manual is included in the Advanced version of the HTHP Cell if ordered as P/N GS05855).

10. Operation of the HTHP Cell

When the HTHP Cell has been correctly configured for fitting of the heater block assembly (**10**) into position within the HTHP Cell body (**3**) and an appropriate sample (or reference material) has been placed into the heater block assembly (**10**) too, the HTHP Cell accessory is installed into the spectrometer onto either its transmission baseplate or reflectance baseplate. After correct installation into the spectrometers sample compartment, it is ready to be operated for spectral data collection.

The specific temperature and pressure conditions regarding the nature of the experiment need to be established prior to any spectral data collection. Consequently, any gas inlet supply and connections at ports (**12**), (**13**) and (**14**) and the outlet connection port (**15**) should be made and checked that they are suitable to allow for appropriate operation after installation of the HTHP Cell into the sample compartment.

Note: *Experimental control of conditions for static (positive pressure or vacuum) or gas flow operation within the HTHP Cell are achieved with an appropriate setting of any inlet gas line valves (open/shut) and outlet gas line valve settings (open/shut). (See Section 5) pages 33 to 35).*

After installation of the HTHP Cell into the spectrometer and the gas line connections have been established and secured for particular operation, the following connections for electrical power and water cooling services should be made.

1) Connect the main power cable of the HTHP Cell to the rear of the temperature controller via the 19 way plug into the corresponding socket. Do not switch on (power up) the temperature controller yet.

2) Having made the appropriate water tubing connections to the upper (**1**) and lower (**2**) cooling plates, connect the inlet tubing to a cold water supply and the outlet tubing to a drain. (See Section 5), pages 25 and 26). Switch on the water supply and establish a **minimum** flow of 0.75 liters per minute through the system.

Important: *The HTHP Cell MUST be operated at all times with a flow of cooling water. The water flow rate should be sufficient to keep the upper (**1**) and lower (**2**) cooling plates cool/cold throughout the entire analysis period. The water supply MUST be turned on before heating and kept flowing after the analysis has been completed and until the temperature indicated for the HTHP cell body (**3**) at the display of the temperature controller falls below 40ºC. Efficient operation of the cooling system has been safeguarded by fixing a thermal switch to the upper (**1**) cooling plate. The switch will shut down the system if insufficient water flows through the cooling plates and the temperature is detected to rise above 40ºC. The thermal switch will reset itself to enable the HTHP Cell to function normally when the water temperature reaches approximately 25ºC.*

3) With the electrical connection established and when cooling water is flowing, the temperature controller can be switched on (powered up) and a specific temperature can be set for the heater block assembly (**10**) and the HTHP cell body (**3**). Please consult the user instruction manual for the temperature controller for operation.

Note: *Although a temperature to reach for the sample can be **set** on the controller, the maximum temperature that can be attained (the **actual** temperature measured) for the heater block assembly (**10**) is dependent upon the pressure conditions and particular gaseous environment with the HTHP Cell chamber. Higher pressures and high thermal conducting gases will reduce the maximum temperature that can be achieved.*

In general operation for collecting of spectral data, the experimentation to carry out for any sample will normally have to be done in the first instance for a **reference** material. Therefore, the specific conditions of temperature, pressures and/or gas flow that will be applied to the HTHP Cell environment for measurement of a **sample** are ideally applied too for the measurement of a **reference background** to try and achieve the best comparison when subtracting the reference from any sample spectrum obtained.

11. Cleaning the HTHP Cell

When the HTHP Cell is used repeatedly at elevated temperatures, especially in the decomposition mode with evolved gases, tarnishing to the inside of the HTHP Cell body (**3**) and the heater block assembly (**10**) will occur. If this build-up of residual material is allowed to continue, particularly on the heater block assembly (**10**), the performance of the HTHP Cell will be impaired. This can be remedied by cleaning the heater block assembly (**10**) and inside the HTHP Cell body (**3**) on a regular basis.

Cleaning the HTHP Cell Body (3)

The HTHP Cell body (**3**) and the pressure certified window assemblies (**11**), in most instances will not be contaminated to the same extent as the heater block assembly (**10**) of parts. This is because the HTHP Cell body and window temperatures (up to 240ºC) are considerably lower than the temperature that can be attained at the heater block assembly (**10**) (up to 800ºC). Any evolved vapours that become deposited as a residue may be baked and /or charred when in contact with the very hot heater block assembly (**10**).

As stated, the overall performance and efficiency of the HTHP Cell for heating is determined by the cleanliness of the heater block assembly (**10**), but if the HTHP Cell body (**3**) and the window assemblies (**11**) need to be cleaned from any build up of contaminants, this can be done as follows:-

With the HTHP Cell removed from its spectrometer sample compartment installation position, remove the heater block assembly (**10**) from the HTHP Cell body (**3**) by removing its wiring strap (**37**) and endplate end plate (**38**) from undoing of their respective fixing screws (**39**) and (**40**). (See pages 47 and 48.) Carefully remove the heater block assembly's sealing silicone O-ring (**35**) from its recess groove in the HTHP Cell body (**3**) and place all the parts to one side for re-assembly after cleaning.

Remove the other end plate assembly (**42**) from the HTHP Cell body (**3**) by undoing its eight fixing screws (**40**). (See pages 49 and 50.) Carefully remove the end plate (**42**) sealing silicone O-ring (**35**) from its recess groove in the HTHP Cell body (**3**) and place all the parts to one side for re-assembly after cleaning.

Now remove the two pressure certified window assemblies (**11**) from undoing their six fixing bolts (**30**). (See pages 40 to 43.) Ensure that their sealing silicone O-ring (**35**) also comes away from the HTHP Cell body (**3**) and place these parts carefully to one side for re-assembly after cleaning.

The inside of the HTHP Cell body (**3**) is now accessible from four sides (via the end plate positions and the window assembly apertures) to be able to introduce cleaning devices (e.g. fine abrasive papers, solvents, etc). To remove fairly heavy deposits from the cell body walls, 100 grade carborundum paper can be used initially, followed by a finer (600-1000) grade carborundum paper for polishing the inner surface. Any fine dust deposits can then be washed away with detergent and hot water, followed by cleaning with paper towels. Finally, a paper towel soaked in methanol can be used to rub over the inner surfaces to remove any lingering traces of grease etc.

Cleaning the Heater Block Assembly (10)

The heater block assembly (**10**) must be removed from the HTHP Cell body (**3**) to gain access for cleaning. (See pages 47 and 48.)

Any residual build-up of deposits on the heater block assembly (**10**) will first need to be removed using an abrasive. For particular areas of the heater block assembly (**10**) including the internal threads of the transmission position aperture hole (**50**) for the keep rings (**51**) , the thermocouple (**36**) and decomposition cup recess (**53**), use 100 grade carborundum paper or a micro-sand blast. For the flat areas of the holder use 100 grade carborundum paper.

Note: *In all cases rub smooth and bright, with no sign of grey shadow or dark particles beneath the surface.*

When the worst of any deposits have been removed at the initial cleaning stage, the heater block assembly (**10**) can be polished using 600-1000 grade carborundum paper or a sand filled rubber block. Polish until ultra smooth and bright.

After polishing, blow off all the dust. Wash the heater block assembly (**10**) with a suitable degreasent (detergent and hot water) and dry with a paper towel. Finally, clean the surface with a methanol soaked paper towel and then wipe again with a dry paper towel.

The heater block assembly (**10**) is now ready to be inserted back into the HTHP Cell body (**3**) depending upon the mode of operation (transmission, decomposition or reflectance) to be used. When handling the heater block assembly (**10**) after cleaning, use gloves to avoid leaving any fingerprints on the cleaned and polished surfaces.

Cleaning of the Window Assembly (11)

Attention should also be paid to inspection and cleaning of the window assemblies (**11**). It is possible that some contamination can occur (from decomposition mode operation) on the surfaces of the ZnSe windows (**26**) that are in contact with the inside environmental conditions of the HTHP Cell chamber. A build up of contaminants on the window (**26**) will reduce the light throughput performance of the HTHP Cell.

The ZnSe material itself should be cleaned using a soft lens cleaning tissue soaked with a suitable solvent. Water, methanol, ethanol or acetone are all suitable solvents that can be used against ZnSe material in an attempt to remove a particular deposit.

Warning! *Be very careful that you do not scratch or mark the surface of the ZnSe material (**26**) sealed in the window housing assembly (**11**) when cleaning with a lens tissue. Any physical imperfections could render the window unsafe to use in the HTHP Cell accessory at high pressure conditions.*

Use of the HTHP Cell after Cleaning

After cleaning of any specific or all relevant parts of the HTHP Cell chamber and re-assembly of the HTHP Cell, it must be “pre-conditioned” for use by heating at a particular temperature in a vacuum environment. The pre-conditioning heat treatment process produces a very thin layer of protective film on the heater block assembly (**10**) which helps to reduce the build- up of deposits on this assembly of parts during subsequent operation and use.

The pre-conditioning heat treatment process should be carried out after re-installation of the HTHP Cell into the sample compartment of a spectrometer and appropriate connectivity to the cooling water supply and a vacuum pumping facility to the outlet gas connection port (**15**). **No sample** is used in the heater block assembly (**10**), but Specac would recommend to place an empty (cleaned) decomposition sample cup (**54**) into its recess (**53**) and for the keep rings (**51**) to be installed in the transmission aperture hole (**50**) such that these parts are also subject to the pre-conditioning heat treatment process.

Pre-Conditioning Heat Treatment Process

The pre conditioning heat treatment process is as follows. Evacuate the HTHP Cell chamber to circa 0.003Torr by use of the vacuum pumping system. (Please see a typical vacuum set up for operation as shown on pages 33 and 34 – (**VP**) line.)

Next, heat the HTHP Cell window assemblies (**11**) and body (**3**) to 200ºC for the window/body heaters from selection as the **set** temperature value on the HTHP Cell controller of the lower WEST 6100+ controlling unit of the two. Allow 30 minutes to elapse for the HTHP Cell to stabilise at this temperature.

Next, heat the heater block assembly (**10**) to 600ºC for the sample heater in the vacuum environment of the HTHP Cell from selection as the **set** temperature value on the HTHP Cell controller of the upper WEST 6100+ controlling unit of the two. When the **actual** temperature of 600°C has been reached and stabilizes for the heater block assembly (**10**), hold at this temperature for about 5 minutes and then

change the **set** temperature value to 20°C for the sample heater. This will cut power to the sample heater to allow the HTHP Cell to cool down from the prevailing surrounding conditions.

When the **actual** temperature value for the heater block assembly (**10**) (sample heater) has reached circa 200°C, adjust the **set** temperature value for the window/body heaters (lower WEST 6100+ unit), also to 20°C. This will allow the HTHP Cell body (**3**) and window assemblies (**11)** to cool down too, but the rate of cooling of the internal heater block assembly (**10**) will be slower than the rate of cooling for the external HTHP Cell body (**3**) temperature from a co-incident 200°C temperature start point.

Specac advise that you can gain safe access for handling of the internal heater block assembly (**10**) when both the HTHP Cell body (**3**) and heater block assembly (**10**) **actual** temperatures are registering 40°C or less. When a 40°C temperature is reached for the heater block assembly (**10**) from the cooling process, the evacuation process for the HTHP Cell can be ceased and then access can be gained to the heater block assembly (**10**) if required.

When to Clean the Heater Block Assembly (10)?

When the build up of deposits on the heater block assembly (**10**) becomes significant, the HTHP Cell may not attain the **actual** temperature level (input as a **set** temperature), from the **set power level** on the controller system, even under vacuum conditions. (See factory set OPuL parameter on the WEST 6100+ temperature controller for the sample heater. This standard power value is found from the parameter listings in Section 12), page 79 of this instruction manual).

The best option for most efficient operation is to clean the heater block assembly (**10**) to remove the buildup of deposits, but a short term solution to potentially achieve the required **actual** temperature conditions that matches the value **set** on the controller is to increase the power level (OPuL parameter) to between 86 and 95%.

Warning! *DO NOT exceed a value of 95% for the power level parameter OPuL. It is important to make a note of the factory set power level if you raise the power level for operation prior to cleaning.*

Whenever the HTHP Cell fails to achieve its maximum temperature (800ºC) in a vacuum at the 95% power level, the HTHP Cell has to be cleaned. After cleaning the HTHP Cell body (**3**) and the heater block assembly (**10**) thoroughly, reduce the power level parameter (OPuL) back to its factory setting. (For nearly all examples of HTHP Cells this value will be 86%).

If after cleaning, the HTHP Cell fails to achieve its maximum set temperature of 800ºC in a vacuum, specifically at the 95% power level setting for the OPul parameter, then you should contact Specac or your local distributor to seek assistance.

12. The HTHP Cell Temperature Controller

As stated in the introduction of this user instruction manual, the HTHP Cell is operated by its own dedicated temperature controller.

The temperature controller has two independent WEST 6100+ temperature controller units fitted to the front panel. The upper WEST6100+ unit is for control of the temperature to the sample heater in the heater block assembly (**10**) and the lower WEST6100+ unit is for control of the temperature to the HTHP Cell body (**3**) and window assemblies (**11**).

A separate user instruction manual 2I-614-166-10 for operation of the HTHP Cell temperature controller is supplied with the HTHP Cell Accessory if ordered as the standard HTHP Cell P/N GS05850 or the Advanced version as P/N GS05855. The controller user instruction manual for information about operation of the HTHP Cell should be used in conjunction with the HTHP Cell's own user instruction manual.

For general optimum performance and operation of the HTHP Cell, particular values for the control parameters have been factory set and programmed into the WEST 6100+ controllers. (There are some slight differences between the parameter values for the upper and lower WEST 6100+ units.) The control parameters that can be accessed are listed for their factory set values, for both the upper and lower WEST 6100+ units, on the following pages.

If you ever need to change a parameter or autotune the controller for a particular temperature range of operation, these parameter settings will be altered. If you wish to return to the original factory settings, then by gaining access to the control parameters that can be changed, the WEST 6100+ unit can be re-programmed with their factory set values.

Parameters for WEST 6100+ Controller of the Sample Heater in the Heater Block Assembly (10) (Upper Unit)

Parameter Display (In Green)	Parameter Name	Parameter Factory Set Value
FiLt	Input Filter Time Constant	3.0
OFFS	Process Variable Offset	0
PP␣␣	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	2.9
ArSt	Automatic Reset (Integral Time Constant)	1.19
rAtE	Rate (Derivative Time Constant)	0.19
biAS	Manual Reset (Bias)	10
SPuL	Setpoint Upper Limit	800
SPLL	Setpoint Lower Limit	0
OPuL	Primary (Heat) Output Upper Power Limit	86
Ct I	Output 1 Cycle Time	2
PhAl	Process High Alarm	800
AHyl	Alarm 1 Hysteresis	1
PLA2	Process Low Alarm	0
AHy2	Alarm 2 Hysteresis	1
APt	Auto Pre-Tune enable/disable	diSA
PoEn	Manual Control select enable/disable	diSA
SPr	Setpoint Ramping enable/disable	EnAb
rP	Setpoint Ramp Rate Value	1800
SP	SP Value	1
SLoc	Set-up Lock Code	10

Parameters for WEST 6100+ Controller of the HTHP Cell Body (3) and Windows (11) (Lower Unit)

Parameter Display (In Green)	Parameter Name	Parameter Factory Set Value
FiLt	Input Filter Time Constant	3.0
OFFS	Process Variable Offset	0
PP␣␣	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	2.9
ArSt	Automatic Reset (Integral Time Constant)	0.31
rAtE	Rate (Derivative Time Constant)	0.07
biAS	Manual Reset (Bias)	10
SPuL	Setpoint Upper Limit	240
SPLL	Setpoint Lower Limit	0
OPuL	Primary (Heat) Output Upper Power Limit	100
Ct I	Output 1 Cycle Time	2
PhAl	Process High Alarm	240
AHyl	Alarm 1 Hysteresis	3
PLA2	Process Low Alarm	0
AHy2	Alarm 2 Hysteresis	3
APt	Auto Pre-Tune enable/disable	diSA
PoEn	Manual Control select enable/disable	diSA
SPr	Setpoint Ramping enable/disable	EnAb
rP	Setpoint Ramp Rate Value	1800
SP	SP Value	1
SLoc	Set-up Lock Code	10

13. HTHP Cell Diagram Parts Identification List

(**1**) Upper water cooled plate.
(**2**) Lower water cooled plate.
(**3**) HTHP Cell body.
(**4**) Standard brass fitting for water tubing connection.
(**5**) Nylon tubing with fittings for water tubing connection between upper (**1**) and lower (**2**) cooled plates.
(**6**) Nylon tubing with fittings for water tubing inlet connection to the lower (**2**) cooled plate.
(**7**) Nylon tubing with fittings for water tubing outlet connection from the upper (**1**) cooled plate.
(**8**) Body part of Quick Connector water flow shut/off fitting.
(**9**) Insert part of Quick Connector water flow shut/off fitting.
(**10**) Sample Heater Block Assembly.
(**11**) Pressure certified window housing assembly. (ZnSe windows).
(**12**) Top inlet gas connection port (inlet 1).
(**13**) Side inlet gas connection port (inlet 2).
(**14**) Side inlet gas connection port (inlet 3).
(**15**) Top outlet gas connection port.
(**16**) Swagelok cap fitting for 1/4" O.D. tubing connector.
(**17**) Length of stainless steel tubing 1/4" O.D.
(**18**) Olive of Swagelok fitting for 1/4" O.D. tubing connection.
(**19**) Ferrule of Swagelok fitting for 1/4" O.D. tubing connection.
(**20**) Threaded body union of gas connection port.
(**21**) Burst disc assembly.
(**22**) Burst disc body of burst disc assembly (**21**).
(**23**) Perforated end cap of burst disc assembly (**21**).
(**24**) 1/4" O.D. tubing to burst disc assembly inlet Swagelok union.
(**25**) 1/4" O.D. tubing to burst disc assembly outlet Swagelok union.
(**26**) ZnSe window material in window housing assembly (**11**).
(**27**) M4 x 10mm fixing bolt with blob of glue for window housing assembly (**11**).
(**28**) Front body component of window housing assembly (**11**).
(**29**) Mounting ring flange plate of window housing assembly (**11**).
(**30**) M6 x 10mm fixing bolt for mounting ring flange plate (**29**).
(**31**) Heat shield small/narrow.
(**32**) Heat shield large/wide.

(**33**) Cap head screws for fixing of heat shields.
(**34**) Thermal switches.
(**35**) Silicone sealing O-ring of window housing assembly (**11**).
(**36**) Thermocouple in the sample heater block assembly (**10**).
(**37**) Wiring strap of sample heater block assembly (**10**).
(**38**) End plate of sample heater block assembly (**10**).
(**39**) M4 x 10mm stainless steel cap head screw for wiring strap (**37**).
(**40**) M4 x 10mm high tensile plated cap head screw for end plates (**38**) and (**42**).
(**41**) Spring clips to hold main power cable and connection break plug assembly.
(**42**) End plate for HTHP Cell body (**3**).
(**43**) Fixing screw holes for wiring strap screws (**39**).
(**44**) Swagelok union connection in end plate (**42**) for additional monitoring thermocouple.
(**45**) Fixing cap head screw of the HTHP Cell to the transmission or reflectance baseplate assembly.
(**46**) Central fixing pillar/position of HTHP Cell transmission baseplate assembly.
(**47**) Slot and hole recess positions on underside of HTHP Cell for location onto the transmission baseplate assembly baseplate.
(**48**) Location pins on baseplate for slot and hole (**47**) positions.
(**49**) Single support pillar on transmission baseplate assembly.
(**50**) Transmission aperture hole position on the sample heater block assembly (**10**).
(**51**) Keep ring of the sample heater block assembly (**10**).
(**52**) Keep ring key.
(**53**) Location recess on sample heater block assembly (**10**) for decomposition sample cup (**54**).
(**54**) Decomposition sample cup.

14. HTHP Cell Spares and Consumables

P/N GS05860 HTHP Cell Reflectance Mode Upgrade Kit. Consists of a pressure certified to 1000psi wedged window assembly with ZnSe window as standard, a reflectance baseplate, and appropriate bracket fixings. (Please specify the spectrometer make and model when ordering).

P/N GS05867 Replacement ZnSe window housing assembly (tested and certified to 1000psi).

P/N GS05868 Decomposition sample cups (packet of 2).

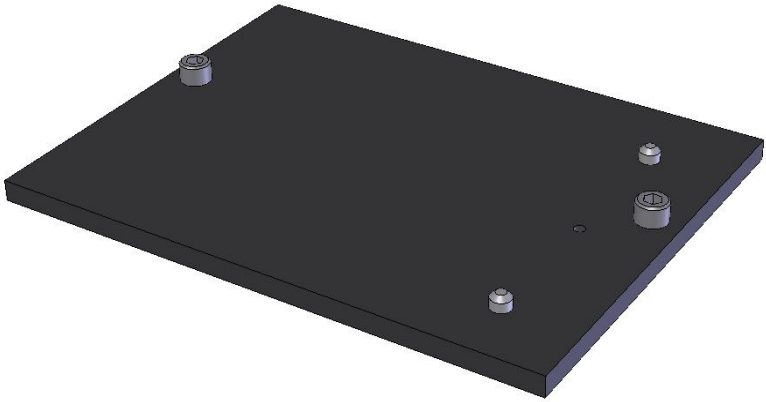
P/N GS05869 Replacement 1000psi burst disc assembly complete. (For window housing assemblies rated to a 1000psi pressure limit.)

P/N GS05870 Essential Spares Kit for the HTHP Cell.

15. Installation of the HTHP Cell Transmission/ Decomposition Baseplate into the Spectrometer

P/N 549-061 - Agilent/Varian/Biorad FTS7, 40, 60, 65, 100, 135, 155, 165, 175, 185, 660, 670, 6000, Excalibur, Scimitar

Beam direction: Right to Left



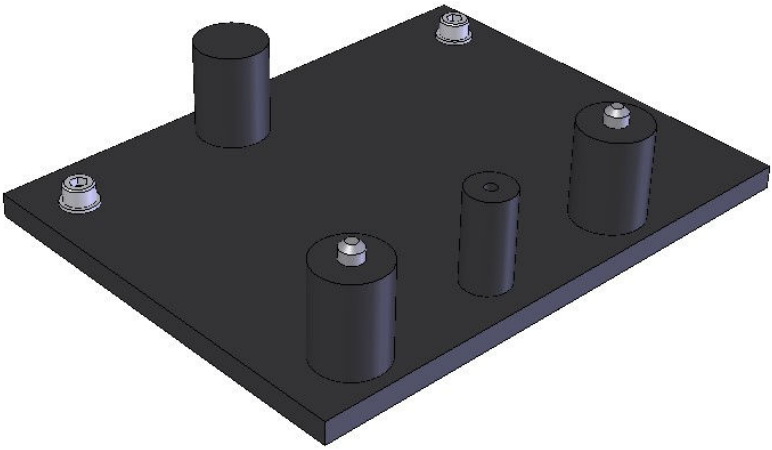
Remove any 3” x 2” mount plate used in the spectrometer.

Place the HTHP Cell accessory baseplate into the sample compartment with the two screw fixing holes of the baseplate over their locating holes in the floor of the instrument. The baseplate is the correct way around with the two support studs and central screw fixing hole on the top surface of the baseplate in a straight line (front to back) to the right hand side of the sample compartment. Secure the baseplate to the floor of the spectrometer through the two screw fixing holes using the two1/4-20UNC x 1/2” cap head screws provided.

Install the HTHP Cell accessory onto the baseplate dowel pins and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-069 - Bomem M100 Series 110, 120

Beam direction: Left to Right



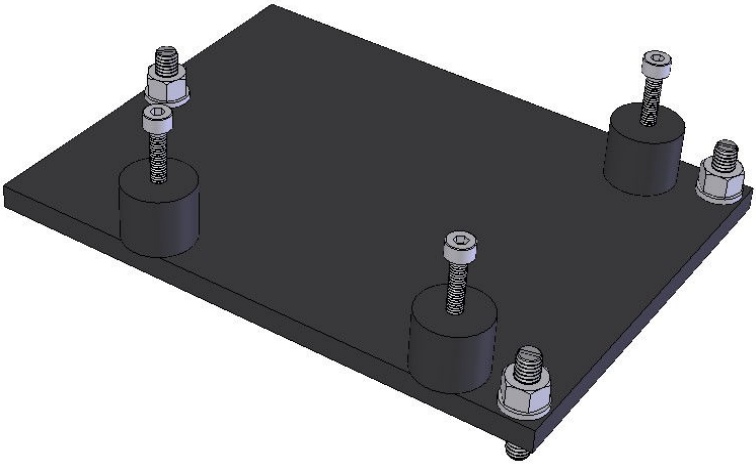
Remove the spectrometer cover and 3” x 2” sample mount to gain access to the sample compartment.

Place the HTHP Cell accessory baseplate into the sample compartment with the two screw fixing holes of the baseplate over their locating holes in the floor of the instrument. The baseplate is the correct way around with the three support pillars in a straight line (front to back) on the top surface to the right hand side of the sample compartment. Fix the baseplate to the spectrometer platform with the two 10-32UNC x 1/2” cap head screws supplied through the screw fixing holes on the left hand side of the baseplate.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-070 - Bomem MB100 Series Kinematic Mount 155, 157 and MB3000

Beam direction: Left to Right



Remove the spectrometer cover and 3" x 2" sample mount to gain access to the sample compartment.

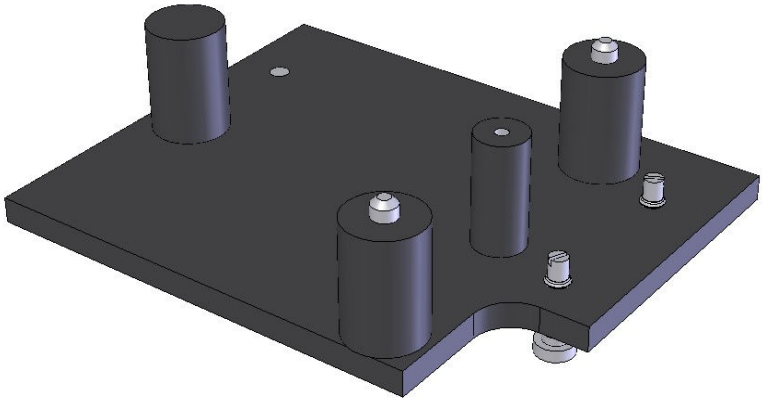
Bolt the HTHP Cell directly to the three baseplate pillars with the three M4 x 20mm cap head screws supplied. The accessory will only affix in one orientation to the three pillars through holes in three of the four corners on the HTHP Cells bottom cooling plate (**2**). The M4 fixing screw (**45**) on the base of the accessory is not used.

Now, place the entire assembly into the spectrometer. This involves placing the screws/feet on the underside of the baseplate onto the three location features (a conical countersink, a 'V' slot and a plain flat) on the floor of the spectrometer. The baseplate screws can be adjusted for height and levelling.

Note: *The baseplate should be secured using two 'L-shape' clamp screws that fix into the spectrometer floor. (These are available from Bomem Inc.)*

P/N 549-064 - Bruker IFS 25, 55, 66, 66V, 88, Vector22, Equinox

Beam direction: Right to Left



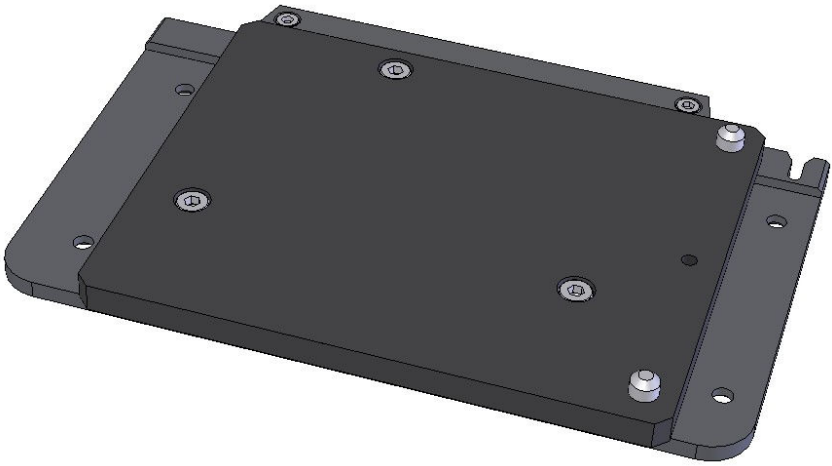
Remove any 3” x 2” mount plate used in the spectrometer from the Bruker baseplate.

Place the HTHP Cell accessory baseplate into the sample compartment with the three support pillars in a straight line (front to back) to the right hand side of the compartment. The two fixing screws and dowel pins of the accessory baseplate (to connect to the Bruker baseplate) are passed through the larger hole ends of the slots on the Bruker baseplate and the accessory baseplate is slid from right to left to centralise it on the Bruker plate. The two fixing screws are then turned anticlockwise to tighten the plates together.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-391 - Bruker Tensor 25, 37, Quicklock 33, Vertex 70, 80

Beam direction: Right to Left



Remove any 3” x 2” mount plate used in the spectrometer from the Bruker baseplate.

Place the HTHP Cell accessory baseplate into the sample compartment to attach to the Bruker baseplate via the Quick Lock mechanism. It will only fit in one orientation for the Quick Lock mechanism to engage.

Install the HTHP Cell accessory onto the flat top section part of the baseplate assembly aligning with the corresponding location pins and secure using the M4 fixing screw (**45**) on the base of the accessory, into the threaded hole in the on the right hand side.

P/N 549-114 - Jasco (All Jasco models)

Beam direction: Left to Right

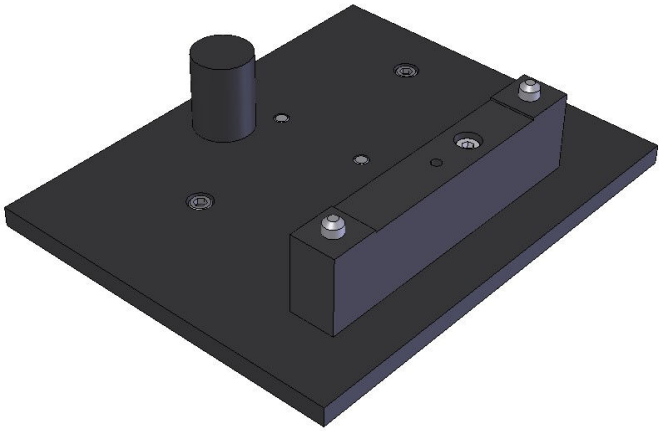
Remove the spectrometer cover and 3” x 2” sample mount to gain access to the sample compartment.

Place the HTHP Cell accessory baseplate into the sample compartment with the two central screw fixing holes of the baseplate over their locating holes in the floor of the instrument. The baseplate is the correct way around with the two support studs and central screw fixing hole on the top surface of the baseplate in a straight line (front to back) to the right hand side of the sample compartment. Secure the accessory baseplate in position using the two M4 x 12mm cap head screws supplied through the central fixing screw holes

Install the HTHP Cell accessory onto the baseplate dowel pins and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-062 - Mattson Galaxy Series 2000, 3000, 4000, 5000, 6000, 7000, 8000 Genesis Series, RS, Infinity

Beam direction: Right to Left (8000 is Left to Right)



A location bar and a support pillar are supplied with the baseplate to allow the accessory to be mounted in both Galaxy Series (bar and pillar required) and Genesis Series (no bar and pillar) spectrometers. Both parts are attached to the baseplate using one cap head screw for each piece.

Remove any 3” x 2” mount plate used in the spectrometer.

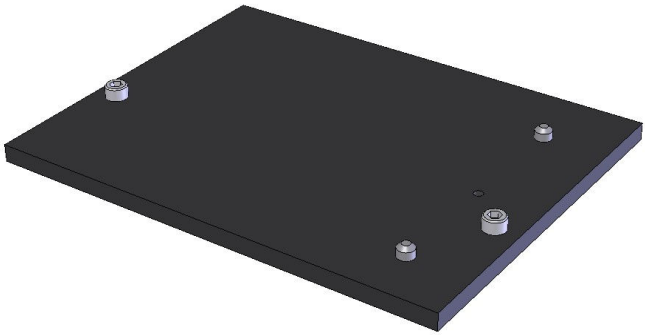
Place the HTHP Cell accessory baseplate into the sample compartment with the location bar fixing to the right hand side of the compartment and such that the dowel pins on the underside of the baseplate locate into the floor of the spectrometer. (The location bar and pillar are not used for Genesis instruments, so must be removed, but the baseplate is still positioned in the sample compartment with the two dowel location pins and central threaded fixing hole being on the right hand side of the sample compartment).

Secure the baseplate in position using the two 8-32UNC x 1/2” cap head screws supplied.

Install the HTHP Cell accessory onto the baseplate location bar and pillar and secure using the M4 fixing screw (**45**) on the base of the accessory, into the central threaded hole of the location bar on the right hand side. (For Genesis instruments the cell installs directly onto the baseplate dowel pins and secures using the M4 fixing screw (**45**) on the base of the accessory, into the small central threaded hole on the right hand side).

P/N 549-123 - Midac (All Midac models)

Beam direction: Left to Right



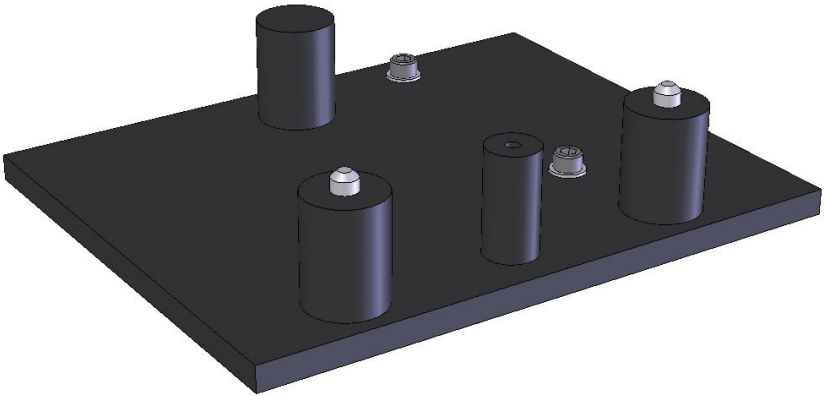
Remove the spectrometer cover and 3” x 2” sample mount to gain access to the sample compartment.

Place the HTHP Cell accessory baseplate into the sample compartment with the two screw fixing holes of the baseplate over their locating holes in the floor of the instrument. The baseplate is the correct way around with the two support studs and central screw fixing hole on the top surface of the baseplate in a straight line (front to back) to the right hand side of the sample compartment. Secure the baseplate to the floor of the spectrometer through the two screw fixing holes using the two 10-32UNC x 1/2” cap head screws provided.

Install the HTHP Cell accessory onto the baseplate dowel pins and secure using the M4 fixing screw (**45**) on the base of the accessory into the threaded hole on the right hand side.

P/N 549-060 - Nicolet 500, 700, 710, 740, 800, 5PC, 5SXC, Magna, Protege, Avatar, Nexus, 6700, 8700, iS10, iS50

Beam direction: Right to Left



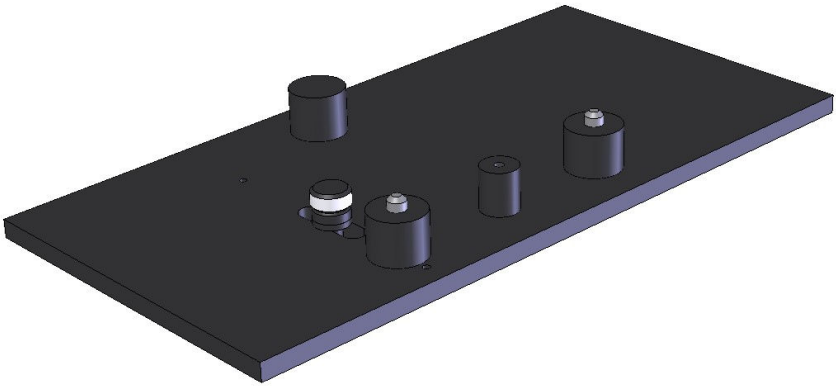
Remove any 3” x 2” mount plate used in the spectrometer.

Place the HTHP Cell accessory baseplate into the sample compartment with the two screw fixing holes of the baseplate over their locating holes in the floor of the instrument. The baseplate is the correct way around with the three support pillars in a straight line (front to back) on the top surface to the right hand side of the sample compartment. Secure the baseplate to the floor of the spectrometer through the two screw fixing holes using the two 8-32UNC x 1/2” cap head screws provided.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-068 - Perkin Elmer PE1700, PE1800

Beam direction: Left to Right



Remove any 3” x 2” mount plate used in the spectrometer.

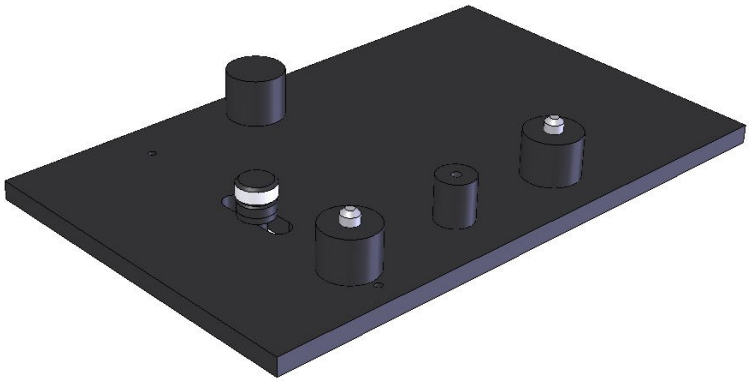
Place the HTHP Cell accessory baseplate into the sample compartment with the three support pillars in a straight line (front to back) on the top surface to the right hand side of the sample compartment. The baseplate is held in position by two location pins into the front support rail of the spectrometer and by tightening of the pull down thumb nut (supplied) to the instruments own fixing screw that passes through the slot at the front of the baseplate.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

Slide the accessory/baseplate assembly along the spectrometer rail to peak the energy where necessary and secure the assembly with the thumb nut onto the instruments fixing screw. The thumb nut is not accessible with the HTHP Cell in place on the baseplate, so you will have to temporarily lift the HTHP Cell off of the baseplate to tighten the thumb screw.

P/N 549-067 - Perkin Elmer PE2000, Spectrum2000, GX

Beam direction: Left to Right or Right to Left



Remove any 3” x 2” mount plate used in the spectrometer.

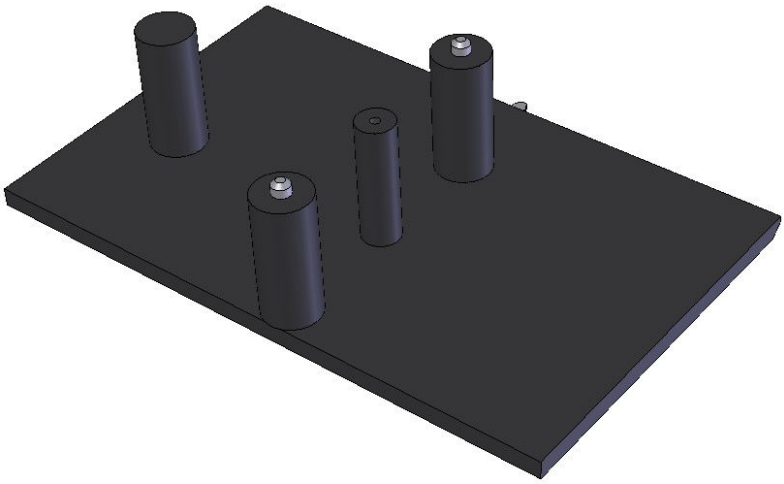
Place the HTHP Cell accessory baseplate into the sample compartment with the three support pillars in a straight line (front to back) on the top surface to the right hand side of the sample compartment. The baseplate is held in position by two location pins into the front support rail of the spectrometer and by tightening of the pull down thumb nut (supplied) to the instruments own fixing screw that passes through the slot at the front of the baseplate.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

Slide the accessory/baseplate assembly along the spectrometer rail to peak the energy where necessary and secure the assembly with the thumb nut onto the instruments fixing screw. The thumb nut is not accessible with the HTHP Cell in place on the baseplate, so you will have to temporarily lift the cell off of the baseplate to tighten the thumb screw.

P/N 549-065 - Perkin Elmer PE1600 Series, Paragon, Spectrum 1000, BX, RX

Beam direction: Left to Right



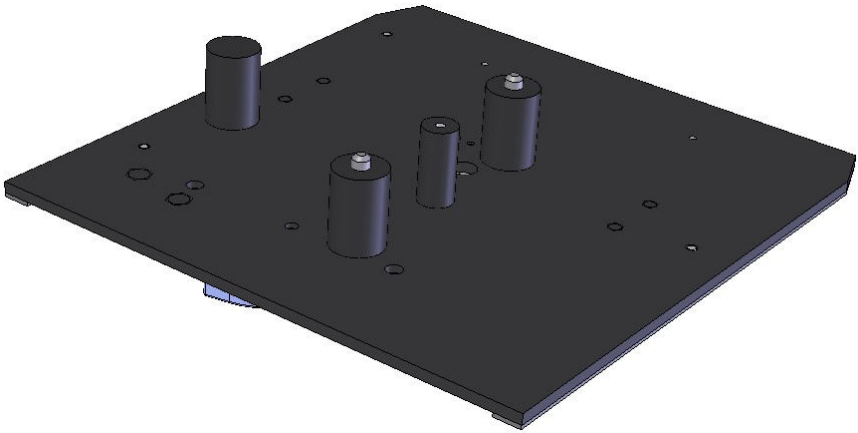
Remove the instruments sliding baseplate floor from the sample compartment by pulling it firmly forward. The catch mechanism at the rear will release automatically.

Slide the HTHP Cell accessory baseplate all the way into the sample compartment with the three support pillars in a straight line (front to back) in the middle of the compartment and the single support pillar to the left hand side. The catch at the back of the baseplate secures the accessory when mounted and the Velcro type strip eliminates any movement.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-293 - Perkin Elmer Spectrum One, Spectrum 100, 400, 4000, 8000 (Frontier)

Beam direction: Left to Right



Remove the instruments sliding baseplate floor from the sample compartment. The lilac colored pivoting handle on the underside of the plate is pulled towards you when you need to remove the plate from the spectrometer.

Slide the accessory baseplate into position along the side edges of the sample compartment with the three support pillars in a straight line (front to back) in the middle of the compartment and the single support pillar to the left hand side. The baseplate locates into the spectrometer by engagement of the 15pin connector at the back of the plate into the spectrometer and by guidance clips on the underside of the plate.

Install the HTHP Cell accessory onto the four baseplate pillars and secure using the M4 fixing screw (**45**) on the base of the accessory, into the small central pillar on the right hand side.

P/N 549-071 - Shimadzu 8000, 8100, 8200, 8300, 8400, 8500, Prestige 21, IR Affinity

Beam direction: Right to Left

Remove any 3” x 2” mount plate used in the spectrometer.

Place the HTHP Cell accessory baseplate into the sample compartment with the two location pins and threaded hole between them on the top surface to the right hand side of the sample compartment. Make sure that the underside bar of the baseplate locates in the slot in the spectrometer sample compartment floor. Move the baseplate from side to side until the location pins in the spectrometer enter the holes in the underside bar.

Secure the baseplate by screwing the two M5 x 10mm cap head screws through the baseplate into the spectrometer sample compartment floor.

Install the HTHP Cell accessory onto the baseplate dowel pins and secure using the M4 fixing screw (**45**) on the base of the accessory into the threaded hole on the right hand side.

Any Other Spectrometers

For advice concerning spectrometers not included in this manual please consult your nearest Specac representative.

Specac

EC Declaration of Conformity

This is to certify that the:

HIGH TEMPERATURE HIGH PRESSURE CELL
5850/5855

Manufactured by:
SPECAC LIMITED

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

by the application of:

1) Testing to the following standard:
   EN-61326:2006/8 EMC (Emissions/Immunity) requirements for Electrical Equipment for measurement, control and laboratory use.
2) Supported by SPECAC Technical File No. **TF5850**

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

by the application of:

1) EN61010-1:2010, Safety Requirements for Electrical Equipment for Measurement, Control and Laboratory use.
2) Supported by SPECAC Technical File No. **TF5850**

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   The Pressure Systems Safety Regulations 2000.
   The Simple Pressure Vessels (Safety) Regulations 1991.

2) Supported by SPECAC Technical File No. **TF5850**
   And adopting (SEP) Sound Engineering Practice as referenced in the above Regulations.

Responsible Person:

Name: Mr.G.Poulter **Signature:** G. Poulter
Position: Technical Director **Of:** Specac Ltd. **Date:** 21st Aug 2013
Serial No: **conforms to the above**
Name: **Signature:**
Position: **Of:** Specac Ltd. **Date:**

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