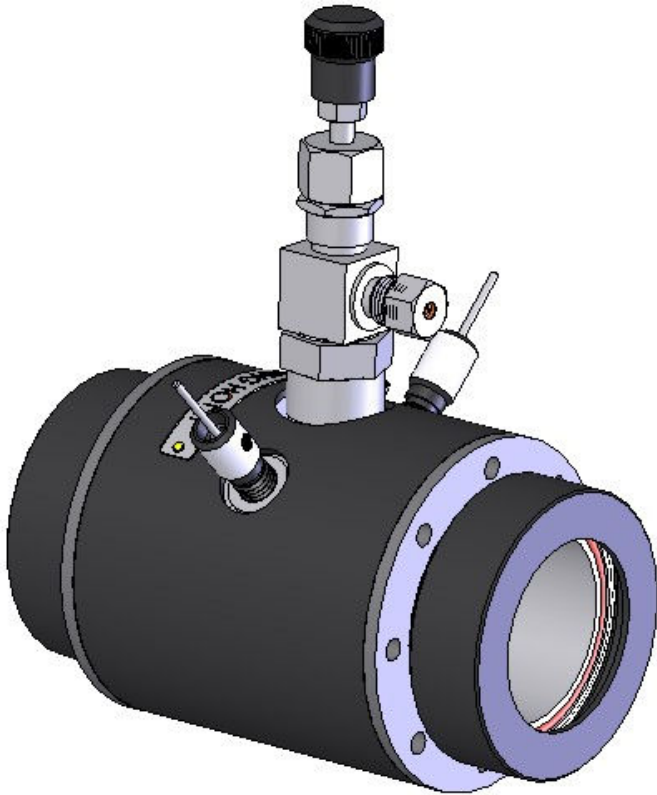




Storm™ 10H Heated Gas Cell

User Manual



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Storm™ 10H Heated Gas Cell P/N GS05670

STORM™ 10H HEATED GAS CELL USER MANUAL

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1. Introduction

Thank you for purchasing a Specac Product.

The Storm™ 10H Heated Gas Cell is a 10cm pathlength gas cell used for the Infrared spectral transmission analysis of vapour and gas phase samples from ambient to 250°C temperatures, and from low vacuum (10^{-2} torr) to 1 Bar pressures (+14.7psi above ambient), irrespective of the window material type used in the Gas Cell. Specific gas containment will depend on the durability of the window material type and seals for their chemical resistance to the vapour conditions. The Gas Cell can also be used to measure vapour and gas spectra obtainable from substances that are normally liquids, solids, or semi-solid compounds at room temperature and atmospheric conditions.

The Storm™ 10H Heated Gas Cell has been designed for safe use from a low 30 Volts for 120 Watt heaters power operation within the Gas Cell body. The power is provided by a dedicated 4000 Series™ Temperature Controller with a WEST 6100+ controlling unit. The Gas Cell incorporates a K-type, NiCr/NiAl thermocouple, permanently inserted into the body of the Gas Cell, to control the heating via the 4000 Series™ Temperature Controller.

The Gas Cell can be evacuated prior to any gas vapour or liquid sample introduction via a stainless steel needle type valve. The evacuation port connection for its operation is independent of any build configuration of the Gas Cell with respect to it being set up as a flow cell for gas vapour measurement, static cell for gas vapour measurement, or for liquid sample injection via a septum injector assembly connection fitted to the body of the Gas Cell.

A variety of window materials can be used within the Storm™ 10H Heated Gas Cell to alter the frequency transmission characteristics for the Cell and to best suit a particular chemical material. There is a choice of standard windows from NaCl, KBr, CaF₂, BaF₂ and ZnSe material options that are supplied separately for fitting into the Storm™ 10H Heated Gas Cell. The variety of window material options allows

for the study of many gas/vapour sample types, although the choice of a particular window material will be dependent upon the particular vapour and gas conditions to be studied with respect to an allowable transmission range and chemical compatibility resistance offered by the window material itself.

The window materials to use are as follows:-

P/N GS05020 Pair of Sodium Chloride (NaCl) windows.
P/N GS05121 Pair of Potassium Bromide (KBr) windows.
P/N GS05022 Pair of Calcium Fluoride (CaF₂) windows.
P/N GS05023 Pair of Barium Fluoride (BaF₂) windows.
P/N GS05096 Pair of Zinc Selenide (ZnSe) windows.

The standard window seals for the Storm™ 10H Heated Gas Cell are in Silicone and PTFE materials. The Silicone O-ring seal is used between the Gas Cell body and the window material surface exposed to the Gas Cell environment and the PTFE gasket is fitted between the window material surface exposed to the external environment and seals with the end cap assembly. Similar to fitting of particular window materials for certain spectroscopic studies, there may be a need to change this internal window seal material if the gaseous sample to be measured will chemically attack Silicone. If in doubt please consult Specac for advice.

The overall dimensions of the Storm™ 10H Heated Gas Cell P/N GS05670 are:-

Overall **length** with both window end cap screw assemblies in place and fitting of 5mm thick windows is 122mm.
Overall **width** at heated cell body (diameter) is 70mm.
Overall **height** to top of the evacuation valve assembly is 148mm.
Overall **volume** of the internal gas cell chamber is 113.5cm³ (mls).

The Storm™ 10 Heated Gas Cell can be installed into the sample compartment of many commercially available IR spectrometer systems via use of its own Gas Cell Mount Holder as supplied.

2. Safety Considerations

With use of any spectroscopic accessory that involves the study of a wide range of chemical samples, the associated risk in handling may mostly be attributed to the specific sample type to be handled itself. As far as it possible you should follow a procedure for safe handling and containment of the type of sample to be used.

With respect to safety of use specifically for the Storm™ 10H Heated Gas Cell, apart from taking necessary precautions when heating the gas cell to elevated temperatures, different window materials can be used for containment of a specific gaseous/vapour type within a stainless steel body for the Gas Cell itself. As standard, NaCl, KBr, CaF₂, BaF₂ and ZnSe windows are the five window materials of choice that can be used.



Caution: *Out of these five different window types, ZnSe is the most potentially hazardous material with respect to toxicity risk in use and handling.*

NaCl, KBr, CaF₂ and BaF₂ window materials can be considered relatively safe to use, although all of them may be harmful to the body if ingested in significant quantity. The general rule when working with **any** window/crystal material (and sample) **is to always wear gloves and safety gear** (e.g. safety spectacles) when handling to obviate the risk of contact with the skin.

Provided with each choice of window material that can be fitted for use in the Storm™ 10H Heated Gas Cell is a window material safety data sheet for the specific material itself that can be consulted for safe handling. A copy of each of these datasheets can also be found in this User Instruction Manual in the **Notes On Cleaning** Section found on pages 34 to 40.

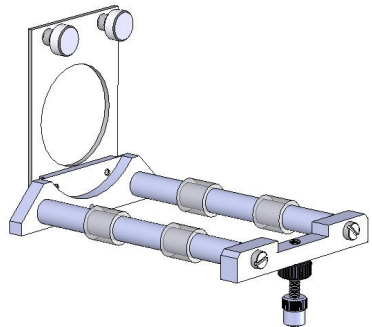
3. Unpacking and Checklist

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

- Storm™ 10H Heated Gas Cell with fixed thermocouple and power cable connection fitted, both adapter ports fitted with stainless steel sealing pin rods, evacuable valve assembly fitted and a set (pair) of window end caps fitted. The silicone O-rings are already fitted into the Gas Cell body and the PTFE gaskets are retained within the window end caps.



- Gas Cell Mount Holder with circular aperture and 3" x 2" backplate.

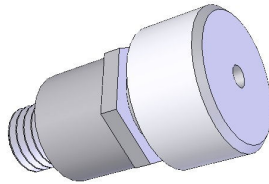


- Spare NiCr/NiAl (K-type) Thermocouple assembly for monitoring gas temperature.

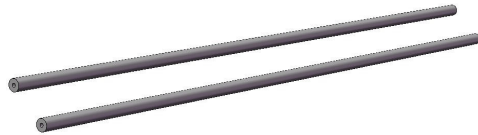


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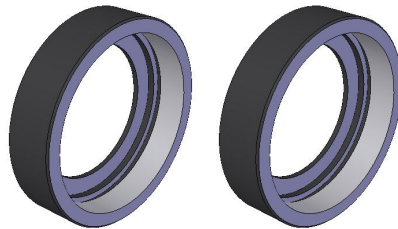
- Liquid/gas septum injector assembly adaptor.



- 2 Stainless Steel Tubes
100 mm long x 1.6 mm
O.D. / 0.51 mm I.D.



- Second pair of window end caps.



- 4000 Series™ Temperature Controller, instruction manual and cables.
- Pair of Storm™ 10H windows (if ordered).

Carefully remove the Gas Cell and associated components from the carry case. Remove the 4000 Series™ Temperature Controller from its packaging.

Where hygroscopic windows, such as KBr, are to be used it is important not to expose them to the atmosphere for longer than necessary. Keep the windows in their container until ready for use.

Note: *The Storm™ 10H Heated Gas Cell is supplied with two pairs of window end caps. One pair are 13.6 mm thick and the other pair are 16.5 mm thick. When using thicker KBr windows (GS05121) with the Storm™ 10H Heated Gas Cell **the thicker window end caps must be used.***

4. Installation

The Storm™ 10H Heated Gas Cell must be positioned correctly within the sample compartment of an IR spectrometer before a spectral analysis of gaseous sample can be made. The Gas Cell can be installed into the sample compartment of many commercially available IR spectrometer systems via use of its own supplied Gas Cell Mount Holder. (See **Fig 1.**)

This part consists of a 3" x 2" size metal plate fitted with a support frame. The metal plate slides into a standard 3" x 2" mount offered from the IR spectrometer system and the Storm™ 10H Heated Gas Cell sits horizontally within the support frame in the correct position for spectral transmission gas/vapour analysis. (See **Fig 1.**)

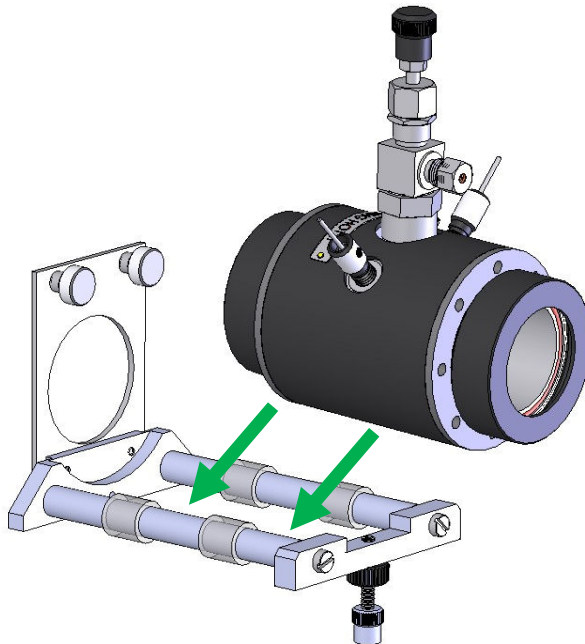


Fig 1. Storm™ 10H Heated Gas Cell with its own Gas Cell Mount Holder

5. Operation of the Storm™ 10H Heated Gas Cell

The Storm™ 10H Heated Gas Cell can be used with a variety of different window materials (1) for the study of high concentrations of gases over a 10cm pathlength from ambient temperature conditions to a maximum of 250°C and pressures up to +14.7psi.

The Storm™ 10 Heated Gas Cell allows for introduction of a gas/vapour or a volatile liquid sample into a low pressure environment to be analysed from different fittings that can be made to the stainless steel Gas Cell body (2). There is a vacuum valve assembly (3) permanently attached to the top of the Gas Cell body (2) which is used to create a low pressure (vacuum pressure 10^{-2} torr) environment within the cell chamber after any windows (1) of choice have been fitted to the Gas Cell.

Suggested Gas Line Connections to the Storm™ 10H Gas Cell for Safe Operation

Whenever raising the temperature for an operation of the Storm 10H Gas Cell, it is advisable to have a pressure safety device plumbed into a gas line connection that is made to the vacuum valve assembly (3). The gas line connection to the valve tap (3) is made using 1/8" O.D. tubing through the 1/8" BSP threaded nut and compression fitting. Additional 1/8" O.D. tubing could be used throughout for the particular gas lines to be plumbed into a system for operation, but alternative "union" connections (e.g. 1/8" to 1/16" reduction) could be used once an initial connection to the valve tap (3) using 1/8" O.D. tubing has been made.

This safety device with associated gas line connectivity is needed to vent away any potential excess build-up of the pressure in the gas cell chamber area (specifically for a **fixed volume** state of the gas cell) when elevated temperatures from ambient conditions are being used, to prevent any accidental damage to the windows that have been fitted or compromising of the sealing from the window end cap assemblies.

A suggested schematic for plumbing of a safety pressure device gas flow line, vacuum line and an additional “vent off” gas flow line to the vacuum valve assembly (3) connection is seen as **Fig 2**.

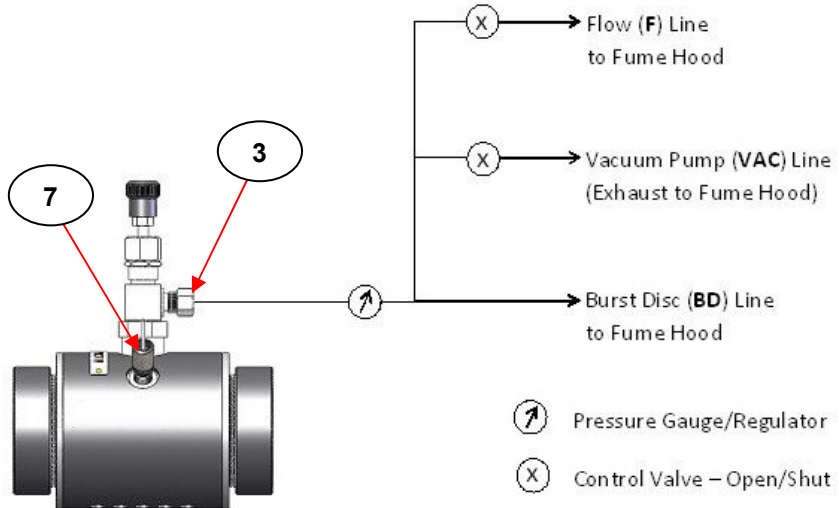


Fig 2. Schematic for Suggested Vacuum/ Gas Line Connectivity for Safe operation of the Storm 10H Heated Gas Cell

As shown in the schematic at **Fig 2**, a single tubing connection is made to the Storm™ 10H Heated Gas Cell vacuum valve assembly (3) via 1/8" O.D. tubing, but this tubing connection branches to three separate line connections. One line is open all the time (no shut off tap/valve) to a pressure safety burst disc device (**BD** line). A second line with an open/shut valve tap runs to a vacuum pump system (**VAC** line) and the third line with its own open/shut valve tap operates as an independent flow line (**F** line), if switched open. Both the safety device (**BD**) line and additional (**F**) line are routed to terminate in a safe containment area such as a fume hood. Any exhaust port(s) from a vacuum pumping system should also ideally be routed to vent off the gas to a safe area too, such as a fume hood environment.

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From connection to the vacuum valve assembly (3) with appropriate gas lines as suggested from the schematic, it does not matter which type of gas fitting (monitoring thermocouple, pin rod, flow tube or septum injector assembly) is made at the two inlet/outlet ports (7) on the gas cell body (2) for a gas analysis measurement to be achieved under a safe mode of operation.

Depending upon the circumstances of operation needed for the Storm™ 10H Heated Gas Cell, the valve taps on the vacuum valve assembly (3), (VAC) line and (F) line can be opened or closed accordingly to control the pressure and allow for safe operation of the equipment. The setting of the valve taps as open or shut can be tabulated as follows for particular experimental conditions with regards as to which line is operable.

Gas Cell Operating Condition	Vacuum Valve Assembly (3)	Valve on (F) Line	Valve on (VAC) Line
Over Pressure Monitoring	Open	Shut	Shut
Gas Flow	Open	Open	Shut
Vacuum	Open	Shut	Open

Note: From the suggested schematic set up, if the vacuum valve assembly (3) tap is **shut**, none of the lines (BD), (VAC) or (F) are operable.

Fitting of Windows into the Storm™ 10H Heated Gas Cell

A pair of windows (1) for the Storm™ 10H Heated Gas Cell are fitted into position as seen from Fig 3.

The stainless steel body (2) of the 10H heated Gas Cell carries two end caps (4) which are screw threaded to the body (2). Any window (1) to be fitted is sealed between a Silicone O-ring (5) that is fitted into a circular grooved recess at the end of the body (2) and a PTFE gasket (6) that seal fits inside the end cap (4). Use the following procedure to fit a window into the Storm™ 10H Heated Gas Cell.

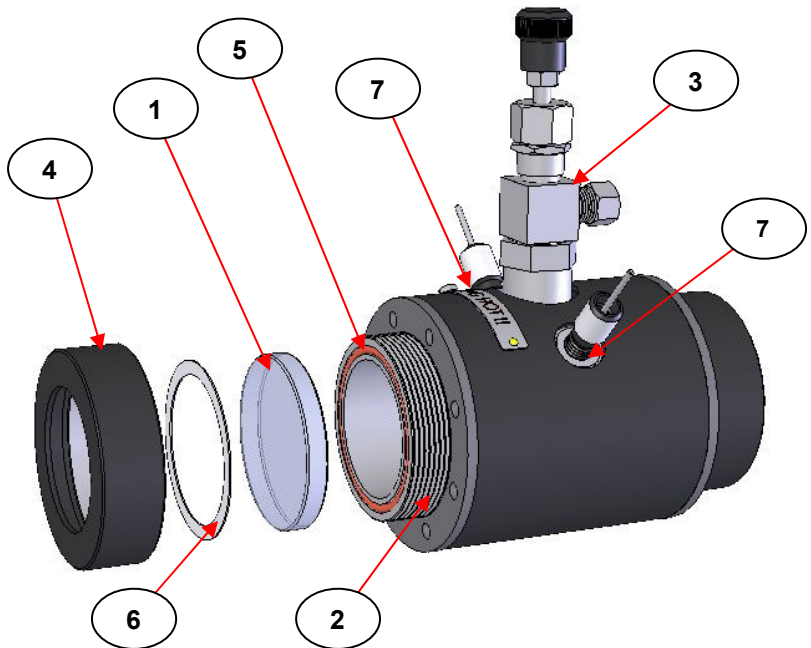


Fig 3. Sequence of Silicone O-Ring, PTFE Gasket and Window Fitting in Storm™ 10H Heated Gas Cell

- 1) Ensure that the face of the body (2), the Silicone O-ring (5) and the screw threads (body (2) and end cap (4)) are clean and free from foreign matter.
- 2) Fit the Silicone O-ring (5) into the groove on the face of the threaded connection end of the Gas Cell body (2).
- 3) Place the PTFE gasket (6) inside and onto the flange of the end cap (4).
- 4) Insert the window (1) into the end cap and PTFE gasket assembly (4 and 6) for it to be held centrally and concentrically in the end cap (4).
- 5) Holding the cell body (2) with Silicone O-ring (5) horizontally, take the window/PTFE gasket/end cap assembly of parts (1, 6 and 4) and screw tighten to the cell body (2). Tighten until firmly clamped, but not too tight to prevent possible damage to the window (1).

Repeat the procedure from steps 1) to 5) for the other end cap (4), PTFE gasket (6) and window (1) assembly.

Build Configuration of the Storm™ 10H Heated Gas Cell for Operation

Before the windows (1) are placed into the gas cell body (2), the Storm™ 10H Heated Gas cell itself will need to be adapted for its build regarding the type of experimental measurement required in its operation. There are a number of ways in adapting the Storm 10H Heated Gas Cell for different operations, but essentially the build configuration and subsequent way the Gas Cell can be used depends upon whether direct monitoring of the gas temperature is a requirement from inclusion of the supplied monitoring thermocouple.

As supplied from new, on the body of the Gas Cell (2) at both of the inlet/outlet connection ports (7) are thermocouple mounting screw fittings (8) that have been filled with a 1/16" O.D. stainless steel pin rod (9). The pin rod (9) is held within the mounting screw fitting (8) by an M3 x 3mm grub screw (10) tightened against the pin rod. (See Fig 4.).

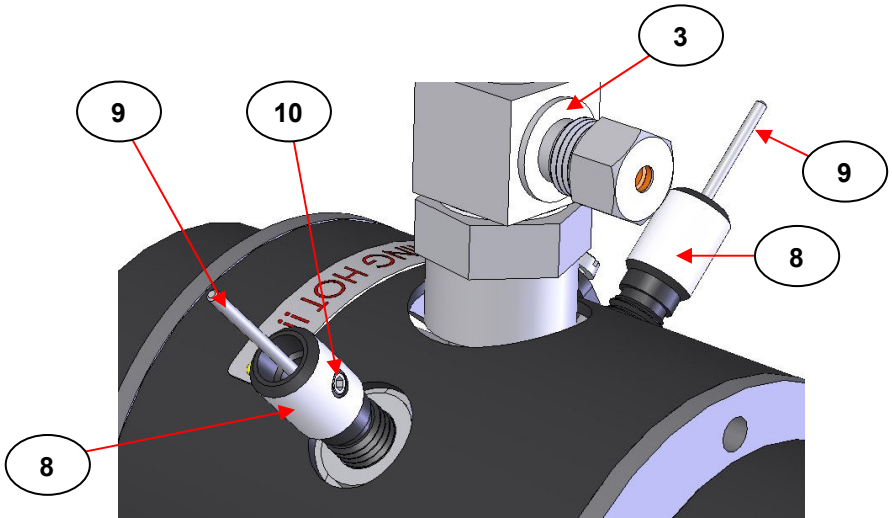


Fig 4. Configuration of the Storm™ 10H Heated Gas Cell for the Inlet/Outlet Ports Supplied from New

The pin rod (9) that passes through the mounting screw fitting (8) also passes through a silicone rubber pad (11) underneath the screw fitting (8). (See Fig 5.)

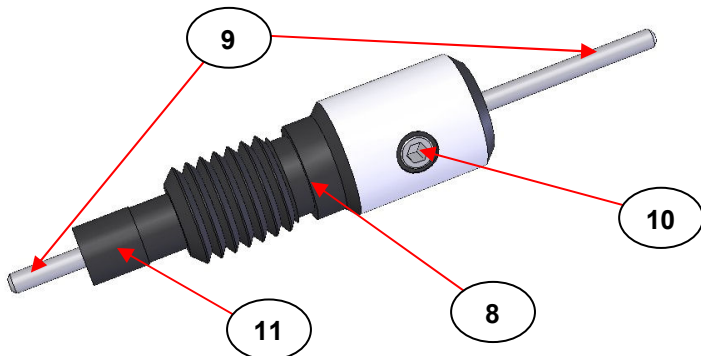


Fig 5. Complete Assembly of Mounting Screw Fitting, Pin Rod, Grub Screw and Silicone Rubber Pad

The complete mounting screw fitting assembly of parts (8, 9, 10 and 11) is screw threaded into the Gas Cell body connection port (7) from turning of the knurled part of the screw fitting (8) to seal the complete fitting assembly of parts via the silicone rubber pad (11). A clockwise turn tightens the fitting assembly of parts (8, 9, 10 and 11) into the gas cell body connection port (7), whereas anticlockwise turning will enable a complete removal of the fitting assembly of parts (8, 9, 10 and 11) from the Gas Cell connection port (7).

To carry out any Gas Cell measurements where **no monitoring** of the temperature of the gas environment is required, the pin rod (9) in one of the mounting screw fittings (8) can be removed and replaced with one of the supplied 1/16" O.D. stainless steel flow tubes (12), OR the **complete** mounting screw fitting assembly of parts (8, 9, 10 and 11) can be removed from the port (7) and replaced with the supplied liquid/gas septum injector assembly (13).

Fitting of the Monitoring Thermocouple

To carry out any Gas Cell measurements where **monitoring** of the temperature of the gas environment is required, one of the pin rods (9) is removed from the screw fitting (8) and replaced with the **monitoring thermocouple assembly** (14). (See Fig 6.)

To remove the pin rod (9) from mounting screw fitting (8), the complete screw fitting assembly of parts (8, 9, 10 and 11) is loosened by a couple of anticlockwise turns of the screw fitting (8) to reduce the sealing of the silicone rubber pad (11) for the whole assembly. The grub screw (10) that is gripping the pin rod (9) can then be loosened by one full anticlockwise turn of the grub screw (10) and the pin rod (9) can then be pulled firmly but smoothly out from its location within the mounting screw fitting (8) and silicone rubber pad (11).

Now take the monitoring thermocouple (14) and very carefully introduce the thermocouple tip (14) into the loosened mounting screw fitting (8) such that the tip passes through the central hole in the silicone rubber pad (11) and to a depth such that the thermocouple tip (14) is just protruding into the internal chamber area of the Gas Cell

body (2). If you wish to have more of the thermocouple tip (14) projecting into the cell chamber, then you need to be careful that it is not going to obscure any of the IR light beam that will pass through the 10cm pathlength of the gas cell when any spectral measurements are being taken.

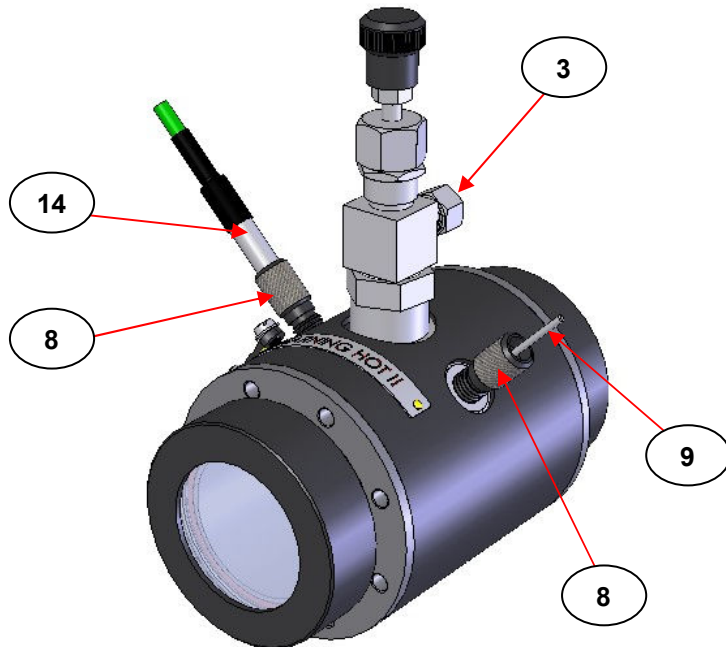


Fig 6. Monitoring Thermocouple Fitted to an Inlet/Outlet Port (Pin Rod Fitted to the other Inlet/Outlet Port)

With the thermocouple tip (14) introduced into the screw mount fitting (8) and through the silicone rubber pad (11) to the required depth, tighten the grub screw (10) to hold the thermocouple tip secure. Then screw turn clockwise the whole assembly of parts (8, thermocouple tip (14), 10 and 11) using the knurling on the mounting screw fitting (8) to seal/tighten the assembly to the Gas Cell port (7) via the silicone rubber pad (11).

Note: *If an inch or longer of the thermocouple tip is protruding into the gas cell chamber, this length may be very carefully bent with a shallow curve profile to lie parallel to the beam path direction as it passes through the gas cell to try and minimize any obscuring of the IR beam energy throughput. Be very careful not to put too much of a sharp bend to the thermocouple tip to avoid damage.*

The monitoring thermocouple (14) can then be connected to an appropriate meter (not supplied) to measure the temperature of the environment within the Gas Cell body (2) chamber.

Configurations of Build for the Storm™ 10H Heated Gas Cell with the Monitoring Thermocouple Fitted

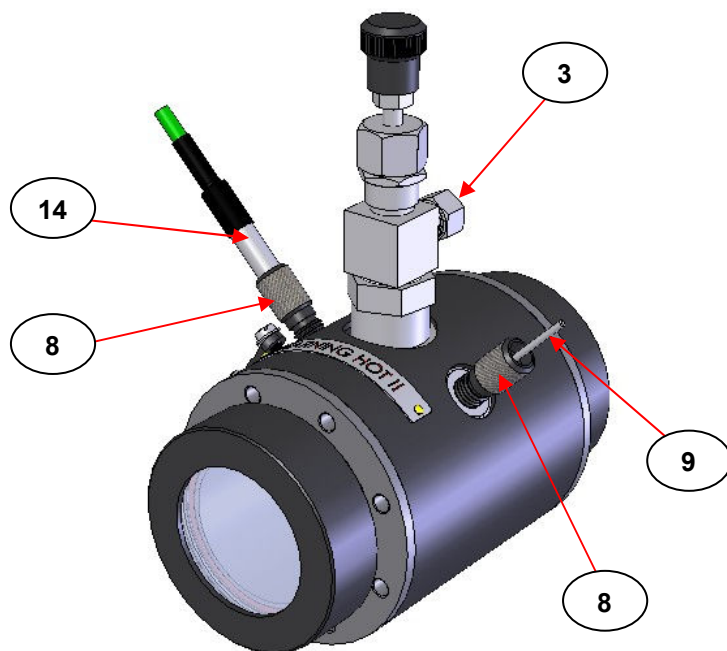
When the monitoring thermocouple (14) has been fitted into one of the inlet/outlet ports (7), the Storm™ 10H Heated Gas Cell can be configured for its build with different fittings into the other connection port (7). There is a choice for fitting of the pin rod (9) or the 1/16" O.D. stainless steel flow tubing (12) into the mounting screw fitting (8), OR the complete mounting screw fitting assembly of parts (8, 9, 10 and 11) can be removed from the port (7) and replaced with the supplied liquid/gas septum injector assembly (13).

Note: *Ensure that the silicone rubber pad (11) is removed from the Inlet/Outlet port (7) if the liquid/gas septum injector assembly (13) is to be fitted. (Use forceps if necessary to remove the pad.)*

Build Configuration (A) – Monitoring Thermocouple and Pin Rod

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at **Figs 6 and 7**.

Applications of use in build configuration (A) allows for the introduction of a solid or semi-solid sample to be placed within the Gas Cell body (2) chamber prior to a careful fitting of an end cap window assembly of parts (1, 6 and 4). A careful fitting of a window (1) helps to keep any solid sample in a position at the base of the inner Gas Cell body (2) chamber such that it does not obscure the IR analysis light beam.



**Fig 7. Monitoring Thermocouple Fitted to an Inlet/Outlet Port
(Pin Rod Fitted to the other Inlet/Outlet Port)**

The Gas Cell body (2) chamber area can then be evacuated by use of a vacuum connection line to the vacuum valve fitting (3) of the gas cell body and when set at a particular vacuum pressure level, the vacuum tap valve (3) can be closed to maintain the vacuum condition. (See gas connection schematic suggestion on page 11.) Under these pressure conditions, if the solid or semi-solid sample itself may evolve a vapour from a decrease in the local pressure environment, then the evolved vapour may be measured spectroscopically over the 10cm pathlength from the fixed volume environment.

In addition, along with a low pressure environment, or even at atmospheric pressure conditions, the Gas Cell body (2) may be heated such that any vapours that may further evolve from an increase in the temperature to the sample itself above ambient conditions can similarly be measured spectroscopically.



Caution! *Whenever raising the temperature for the operation of the Storm 10H Gas Cell, it is advisable to have a pressure safety device plumbed into the vacuum tap valve (3) connection to vent away any excess build-up of the pressure to prevent any accidental damage to the windows or compromising of the sealing from the window end cap assemblies. (See gas connection schematic suggestion on page 11.)*

Under build configuration (A), any gas evolved from the pressure and/or temperature conditions applied can be measured both spectroscopically and specifically for their temperature from the monitoring thermocouple (14).

Build Configuration (B) – Monitoring Thermocouple and 1/16” O.D. Flow Tubing

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at **Fig 8**.

From a Build Configuration (A) construction, the pin rod (9) fitting is replaced with one of the 1/16” O.D. stainless steel flow tubes (12). The fitting of the flow tube (12) into the screw mount fitting (8) is similar to that described for the monitoring thermocouple (14) on page 16. The flow tube (12) must pass through the silicone rubber pad (11) to emerge for a length of approximately 35 to 40mm protruding into the Gas Cell body (2) chamber area. The flow tube (12) will need to be similarly bent with a shallow curve profile when it passes into the Gas Cell chamber to lie parallel to the beam path direction as it passes through the Gas Cell, pointing towards one window (1) to try and minimize any obscuring of the IR beam energy throughput.

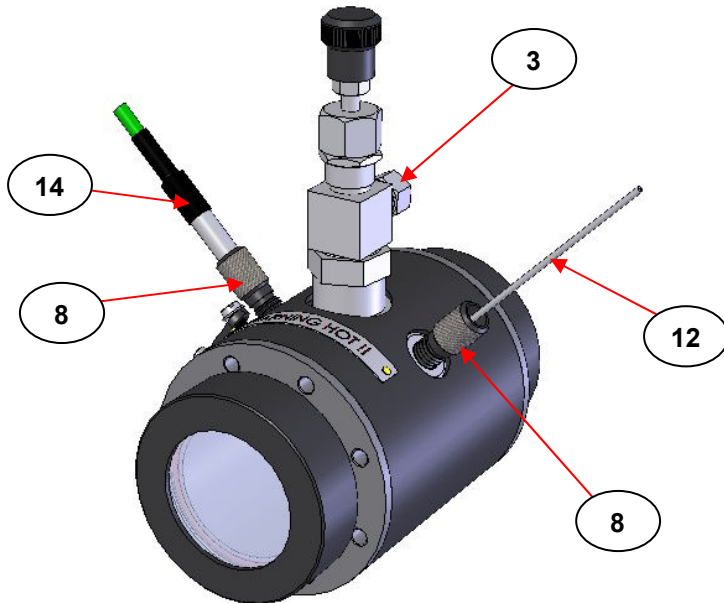


Fig 8. Monitoring Thermocouple Fitted to an Inlet/Outlet Port (1/16" O.D. Flow Tube Fitted to the other Inlet/Outlet Port)

Note: *More of a sharper bend can be applied to the flow tube (12) if necessary for a good fit as the tubing is not as fragile as the thermocouple tip when introducing a bend into the material.*

Applications of use in build configuration (**B**) allows for the introduction of a **gas vapour** into the cell through the flow tube (12) as fitted.

Depending upon any in line gas connections or control valves etc, that may be additionally plumbed to this gas **inlet** flow tube (12) into the Gas Cell, then different static or gas flow experiments can be carried out. If any valve fitted for the gas inlet flow tube (12) is shut for introduction of gas, then the Gas Cell body (2) chamber can be evacuated by setting and switching of the (**VAC**) line capability from

the suggested schematic at **Fig 2**. (Opening of valve tap (3) and (VAC) line valve tap). When the cell chamber has been evacuated, close the valve tap (3) and open the **inlet** flow tube (12) valve to allow for a gas to fill the Gas Cell body (2) chamber for a **static mode of operation**. The Gas Cell can additionally be heated once a gas has been introduced and the gas vapour itself can be measured for its temperature via the fitted monitoring thermocouple (14).

Alternatively, for a **gas flow experiment**, the valve tap (3) can be opened, and with the (F) line valve opened and the (VAC) line valve closed, any gas introduced through the inlet flow tube (12) can be flowed, heated and measured for its temperature via the fitted monitoring thermocouple (14).

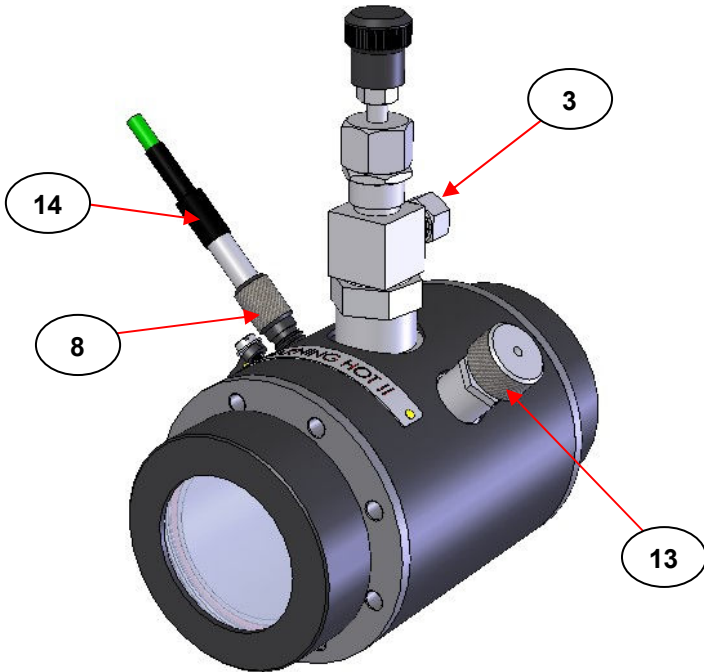
Note: *This build configuration (B) for a gas flow experiment is not the best way to flow gas through the Gas Cell body (2) chamber. Ideally two flow tubes (12) are fitted to the inlet/outlet ports (7) and a flow of gas throughout the cell chamber proceeds this way, with the vacuum valve assembly tap (3) closed, to allow for a complete fill of gas throughout the cell chamber. (See Build Configuration (G) page 29). However, there is no facility to directly monitor the temperature of the gas in the chamber with two flow tubes (12) fitted because the monitoring thermocouple (14) has been removed from one of the ports (7).*

Build Configuration (C) – Monitoring Thermocouple and Liquid/Gas Septum Injector Assembly

For this build configuration of the Storm™ 10H Heated Gas Cell it is constructed as seen at **Fig 9**.

From a Build Configuration (A) construction, the complete mounting screw fitting assembly of parts (8, 9, 10 and 11) is removed and replaced with the liquid/gas septum injector assembly (13).

Note: *Ensure that the silicon rubber pad (11) is removed from the port (7) if the liquid/gas septum injector assembly (13) is to be fitted.*



**Fig 9. Monitoring Thermocouple Fitted to an Inlet/Outlet Port
(Septum Injector Assembly Fitted to the other Inlet/Outlet Port)**

Details of the Liquid/Gas Septum Injector Assembly

The liquid/gas septum injector assembly (13) is shown in more detail for its parts from **Fig 10**. It consists of a body (15) that contains a sealing O-ring (16) in silicone material. The septum injector body (15) has 5/16" B.S.F threading to screw into the gas cell body (2) at the inlet/outlet ports (7), to seal via the O-ring (16). At the other end of the septum injector body (15) there is M12 threading for fitting of the septum injector cap (17). A septum (18) is placed within the body (15) and is held tight for sealing by screwing the cap (17) to the body (15).

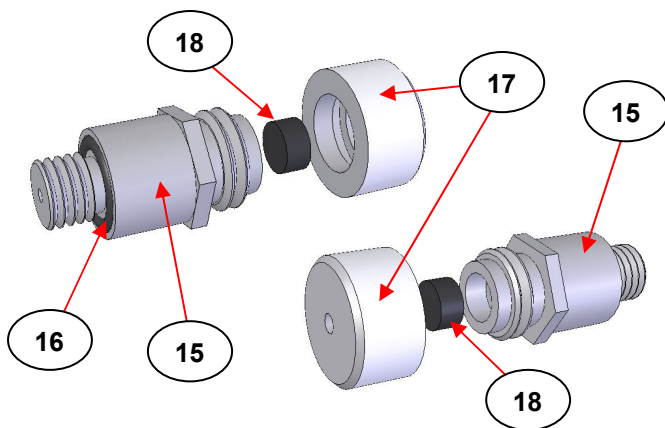


Fig 10. Exploded Views of the Liquid/Gas Septum Injector Assembly

Within the cap (17) is a hole to introduce a needle from a syringe to pierce and pass through the septum (18) to inject volatile liquids or vapours into the gas cell body (2) chamber when the septum injector assembly (13) is fitted to one of the inlet/outlet ports (7). Therefore under build configuration (C), applications of use for the Storm™ 10H Heated Gas Cell allows for the introduction of volatile solvents, solutions or vapours from direct injection into the gas cell chamber that has already been evacuated for a low pressure environment, via the fitted septum injector assembly (13). If sealing of the Gas Cell is compromised, it may mean that the septum (18) has become damaged in some way and a new one is needed for fitting. (A packet of 10 injection septa are available as spares from Specac as P/N GS05665.)

Any subsequent heating of the gas cell body (2), if necessary, will still potentially require a safety pressure monitoring of the system and operation of the appropriate valve taps (open or shut) for the experimental conditions relating to a static holding or a flow of gas for its spectral measurement. Fitting of the monitoring thermocouple (14) also allows for any gas environment temperature to be measured accordingly if required.

Build Configurations of the Gas Cell with No Gas Temperature Monitoring Requirement

If the direct monitoring of the gas temperature for any vapour/gas being measured within the Gas Cell chamber **is not** required, then the Storm™ 10H Heated Gas Cell can be configured for its build **without** a fitting of the monitoring thermocouple (14) into one of the inlet/outlet ports (7). Therefore, any build configurations of the Storm™ 10H Heated Gas Cell starts from the same basic construction of the Gas Cell as supplied from new - i.e. a pin rod (9) is fitted into both inlet/outlet ports (7).

Build Configuration (D) – Pin Rod and Pin Rod

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at **Figs 3, 4 and 11**.

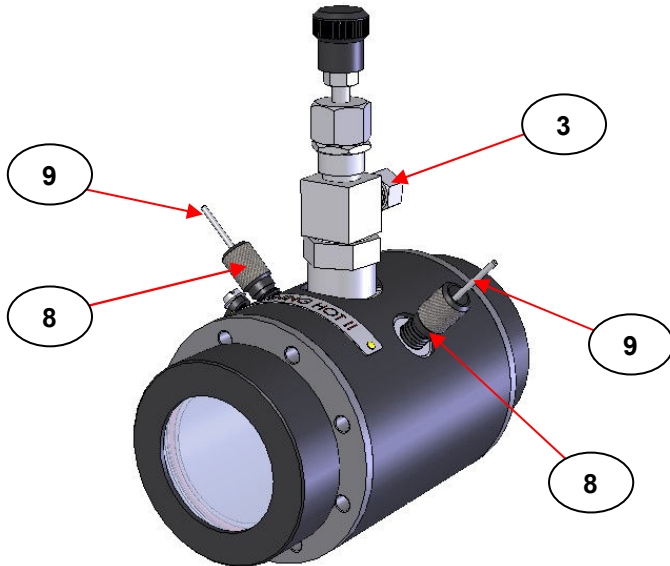


Fig 11. Pin Rods Fitted into Both Inlet/Outlet Ports on the Storm™ 10H Heated Gas Cell Body

Applications of use in build configuration (D) are the same as that for build configuration (A) (see page 18), except that there is no facility to monitor the temperature of the vapour environment because the monitoring thermocouple (14) is not fitted for use.

Build Configuration (E) – Pin Rod and 1/16” O.D. Flow Tubing

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at Fig 12.

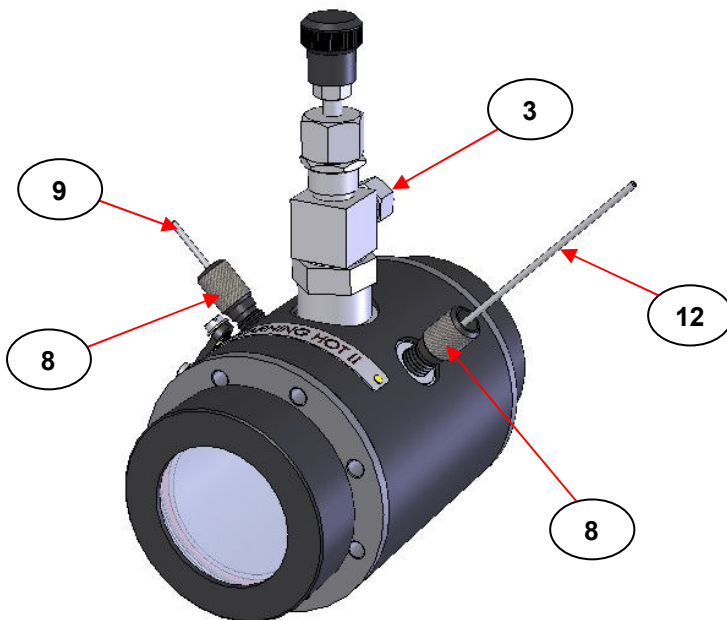


Fig 12. Pin Rod and 1/16” O.D. Flow Tubing Fitted into the Inlet/Outlet Ports on the Storm™ 10H Heated Gas Cell Body

Applications of use in build configuration (E) are the same as that for build configuration (B) (see page 20), except that there is no facility to monitor the temperature of the vapour environment because the monitoring thermocouple (14) is not fitted for use.

Build Configuration (F) – Pin Rod and Septum Injector Assembly

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at **Fig 13**.

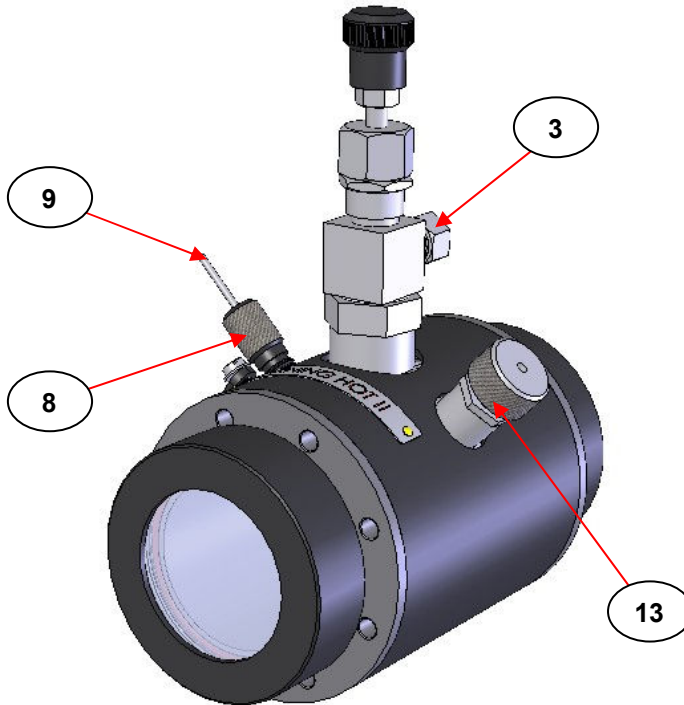


Fig 13. Pin Rod and Septum Injector Assembly Fitted into the Inlet/Outlet Ports on the Storm™ 10H Heated Gas Cell Body

Applications of use in build configuration (F) are the same as that for build configuration (C) (see page 22), except that there is no facility to monitor the temperature of the vapour environment because the monitoring thermocouple (14) is not fitted for use.

Build Configuration (G) – 1/16” O.D. Flow Tubing and 1/16” O.D. Flow Tubing

For this build configuration of the Storm 10H Heated Gas Cell it is constructed as seen at Fig 14.

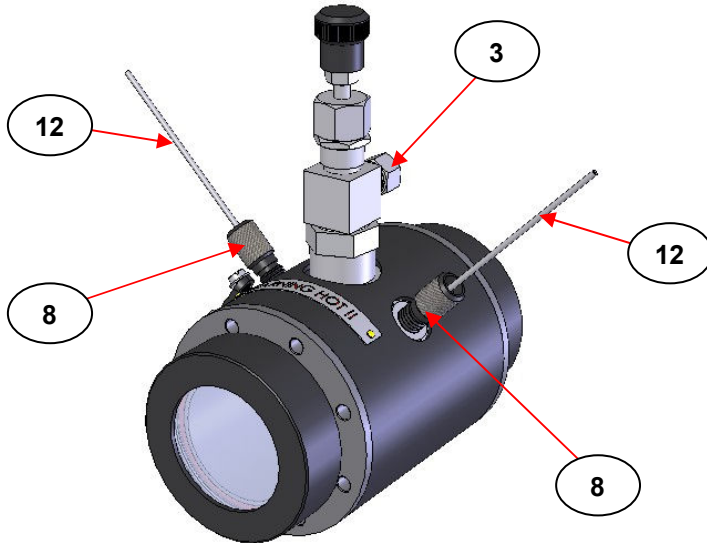


Fig 14. 1/16” O.D Flow Tubing Fitted into Both Inlet/Outlet Ports on the Storm™ 10H Heated Gas Cell Body

Build configuration (G) is where the Storm™ 10H Heated Gas Cell has been configured as a true gas flow cell. One of the fitted flow tubes (12) is used as an inlet for the gas to be introduced and the other flow tube (12) is used as the outlet to allow for a complete and even distribution of gas concentration vapour throughout the gas cell chamber in a flow operation. (Both flow tubes (12) are bent and routed internally for their ends to be close to each window (1) of the gas cell.) Depending upon any inlet and outlet gas flow valve taps that may be added and fitted in line to the flow tubes (12), other applications for build configurations (G) are similar to that as described for build

configuration (B) (see page 20), except that there is no facility to monitor the temperature of the vapour environment because the monitoring thermocouple (14) is not fitted for use.

Build Configuration (H) – 1/16" O.D. Flow Tubing and Septum Injector Assembly

For this build configuration of the Storm™ 10H Heated Gas Cell it is constructed as seen at Fig 15.

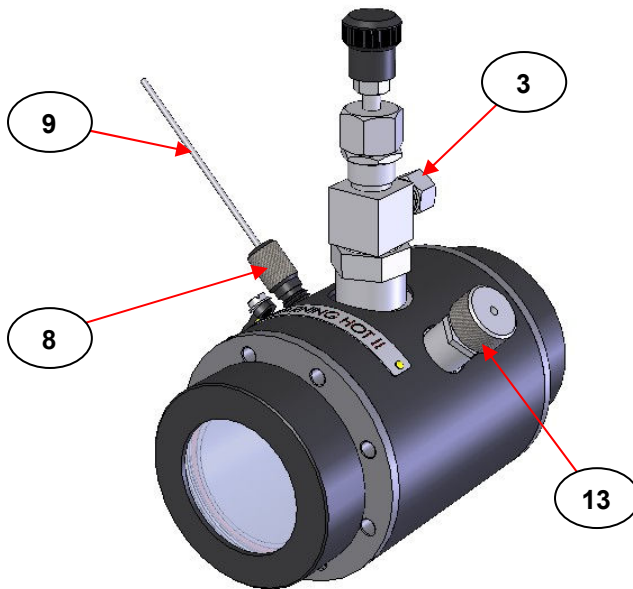


Fig 15. 1/16" O.D Flow Tubing and Septum Injector Assembly into Inlet/Outlet Ports on the Storm™ 10H Heated Gas Cell Body

Applications of use in build configuration (H) can be the same as that offered by either build configurations (B) and (C). Although there is fitting of the septum injector assembly (13) with a flow tube (12), gas flow experiments can be carried out by use of an opening of the vacuum valve tap (3) and the flow tube (12) acts as the inlet for any

gas feed. The fitting of the septum injector assembly (13) in this instance for any gas flow is acting as a blanking plug at this port (7), although there is the capability to use the septum injector assembly (13) for a method of sample introduction into the gas chamber, provided the inlet flow tube (12) is switched off from any inline valve that may be fitted. The analogy of application use similar to build configurations (B) and (C) is limited in that there is no facility to monitor the temperature of the vapour environment because the monitoring thermocouple (14) is not fitted for use.

Summary of Build Configurations and Sample Measurement Applications (At Ambient up to 250°C Temperatures)

Build Config.	Temperature Monitoring	Sample Types Measurable
A	YES	Gases (evolved from solids)
B	YES	Gases - static and flow operation
C	YES	Volatile liquids and gases (injected)
D	NO	Gases (evolved from solids)
E	NO	Gases - static and flow operation
F	NO	Volatile liquids and gases (injected)
G	NO	Gases - static and true flow operation
H	NO	Gases - static and flow operation or volatile liquids and gases (injected)

Changing a Build Configuration of the Storm™ 10H Heated Gas Cell



Special Note For Removing Flow Tubes (12)

If a change to a particular build configuration for the Storm™ 10H Heated Gas Cell is needed that requires removal of one (or both) of the 1/16" O.D. flow tube (12) fittings, any length of tubing that has been bent and routed correctly, internally throughout the length of the gas cell body (2) chamber **MUST NOT** be pulled back through the inlet/outlet ports (7). This action will damage the silicone rubber pad (11) and mounting screw fittings (8) for any further potential sealing.

To safely remove any flow tube (12) fittings, remove the Storm™ 10H Heated Gas Cell windows (1) by unscrewing them within their end cap window assembly of parts (1, 6 and 4).

Now loosen the grub screw (10) holding any flow tubing (12) within the mounting screw fitting (8) and then loosen the screw mount fitting (8) itself by a couple of anticlockwise rotations within the inlet/outlet port (7). Cut off neatly any external connections to the flow tube (12) fitting and push the remaining part of the flow tube (12) into the cell body (2).

Note: *The requirement for neat cut connections is to remove any burrs on the flow tubes (12) that may damage the silicone rubber pad (11) as the flow tubes pass through it when being pushed into the Gas Cell body (2) chamber.*

If the flow tubes (12) are cut off close to the mounting screw fitting (8), they may be pushed inside easily and drop into the Gas Cell body (2) chamber. When the cut flow tubes (12) have been removed from the inside of the Gas Cell, discard them. (**Do not use the cut, bent flow tubes (12) again.**) (Replacement flow tubes (12) in packs of 10 are available as spares from Specac against P/N GS05662.)

Summary for Operation of the Storm™ 10H Heated Gas Cell

- 1) Choose a configuration of build (**A** to **H**) for the Storm™ 10H Heated Gas Cell specifically for the type of sample measurement to be carried out. The choice of build configuration is based upon whether the monitoring thermocouple (**14**) is fitted to the gas cell body (**2**) and if a specific temperature measurement of the gas environment is required.
- 2) When the build configuration is established, a choice of window (**1**) material can be fitted to the Gas Cell body (**2**).

Note: *If solid samples are to be studied for any vapours that may evolve from them in a low pressure or heated environment, the sample should be placed into the Gas Cell body (2) prior to fitting of a window (1), end cap (4) and PTFE seal (6) assembly.*

- 3) Carefully place the Gas Cell onto its own 3" x 2" slide mounted holder (See **Fig 1.**, page 9) into the spectrometer sample compartment.
- 4) Connect the vacuum valve tap (**3**) to a safety pressure device flow line, vacuum line and flow line system of plumbing as suggested from the schematic – see page 11.
- 5) Connect the 6-way power cable with the fixed system (Gas Cell) NiCr/NiAl K-type thermocouple to the rear of the 4000 Series™ Temperature Controller supplied. It is push fitted from alignment of a "notch and slot" connection and locked by turning clockwise of the outer knurled ring. Follow the instructions from the 4000 Series™ Temperature Controllers own user Instruction Manual supplied to heat the Storm™ 10H Heated Gas Cell.
- 6) If the Storm™ 10H Heated Gas Cell has been configured for its build with the monitoring thermocouple (**14**) fitted, this thermocouple (**14**) should be connected to an independent monitoring device/meter.

Note: *The K-type fixed system thermocouple included in with the power cable assembly connection regulates the heating to the Gas Cell itself via the set and actual temperature values displayed on the 4000 Series™ Temperature Controller.*

- 7) Begin your experiments for any spectral collection of evolved or introduced gas samples by setting any pressure and temperature conditions accordingly on the Storm™ 10H Heated Gas Cell. Usually a reference/background spectrum is required to compare against any spectral sample conditions being monitored and measured and so ideally a reference spectrum is taken under identical pressure and temperature conditions that would be established at the sampling stage.

Cleaning the Storm™ 10H Heated Gas Cell

Depending on the particular type of gas that has been analysed within the Storm™ 10H Heated Gas Cell, before storage, Specac would recommend purging the Gas Cell with a supply of nitrogen gas after use. A purge with nitrogen gas helps to remove any residual vapours that may be trapped within the Gas Cell and to minimise any potential “memory effects” of the previous sample from next use of the Gas Cell. Nitrogen gas can be flowed through if at least one flow tube (**12**) has been fitted to an inlet/outlet port (**7**) and the vacuum valve assembly tap (**3**) is open for gas to be routed along the (**F**) line.

If any vapours are contained in the Gas Cell after use from a build configuration **that does not have a flow tube (12) fitted into one of the ports (7)**, then any gas can be **evacuated** from the cell using the (**VAC**) line route. After evacuation and still with the vacuum valve assembly tap open (**3**), switching to the (**F**) line would allow atmospheric vapour conditions to repressurise the cell body (**2**) chamber environment and then the windows (**1**) and end cap assembly parts (**4** and **6**) can be removed to gain better access for more thorough cleaning of the gas cell and windows if necessary.

The Storm™ 10H Heated Gas Cell can be stored fully constructed with windows in position and containing nitrogen gas if the gas line at the vacuum valve assembly (3) is disconnected and the valve tap (3) is closed. Specac recommends use of a dry cabinet for storage under such conditions.

However, if NaCl or KBr windows (1) have been used, because of their hygroscopic nature and susceptibility to “fogging” if exposed to a moist or damp environment, Specac would recommend that these window (1) material types are removed from the Storm™ 10H Heated Gas Cell body (2) and stored separately in a dry and clean environment. (They may be stored back in the original container they were supplied in.)

Notes On Cleaning

When cleaning any removed window material being used in the Storm™ 10H Heated Gas Cell, it is **very important to take care** to avoid damage to the window materials. As also mentioned in the Safety Considerations (Section 2, page 6), of the five standard window materials supplied that can be fitted in the Gas Cell, ZnSe is potentially the most hazardous in terms of risk of toxicity if it comes into contact with the skin.



Note: Always wear gloves to protect yourself and the window material.

Solvents such as water, methanol, acetone, hexane, chloroform etc are suitable to use for cleaning purposes, but avoid use of any solvents that are “wet” or contain trace amounts of water, as NaCl and KBr window materials will be damaged. CaF₂, BaF₂ and ZnSe window materials are generally chemically tolerant of a wide range of aqueous based solvents or solutions for cleaning purposes, but only sample solutions that fall within the pH range of pH4 to pH11 are tolerated by the ZnSe window material. Stronger acidic or basic solutions if introduced will irreparably damage any ZnSe windows that are fitted.

Caution! *If in doubt that your solvent for cleaning may be damaging to the window material being used with the Storm™ 10H Heated Gas Cell, always try to test a fragment of the window material type, if possible, with the chemical first.*

When wiping away any solid (condensed) residues (if present) on the window surfaces, use a very soft lens tissue moistened with the appropriate solvent to avoid scratches being caused on the surface of the window material. Scratches and blemishes to the window surface will result in poor light throughput for the transmission technique (more risk of light scatter) and an overall degradation in the Storm™ 10H Heated Gas Cell performance.

Water and/or alcohol solvents can be used on all other parts of the Storm™ 10H Heated Gas Cell for cleaning as and when necessary.

Datasheet for Sodium Chloride (NaCl) Material

General

Synonyms: salt, sea salt, table salt, common salt, rock salt.

When fused together as a solid can be polished and used as a transmission window material. Slightly hygroscopic material similar to Potassium Bromide (KBr).

Soluble in water and glycerine. Slightly soluble in lower order alcohols. Fairly good resistance to mechanical and thermal shock and can be easily polished.

Molecular formula: NaCl.

Chemical Abstracts Service (CAS) No: 7647-14-5.

Physical Data

Appearance: Odourless, white or colourless crystalline solid.

Melting point: 804°C.

Boiling point: 1413°C.

Vapour pressure: 1mm Hg at 865°C.

Specific gravity: 2.16 g cm⁻³

Solubility in water: 35.7g/100g at 0°C.

Hardness: 6 Kg/mm².

Refractive Index: 1.52 (at 2000cm⁻¹ - wavenumbers).

Spectroscopic transmission range: 40,000 to 600 cm⁻¹ (wavenumbers).

Stability

Stable. Incompatible with strong oxidising agents.

Toxicology

Not believed to present a significant hazard to health. May cause eye irritation.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material.

Allow for adequate ventilation.

Storage

Keep powder or windows stored in a cool, dry container.

Datasheet for Potassium Bromide (KBr) Material

General

Medium for making Potassium Bromide pellets for IR spectroscopy. When fused together as a solid can be polished and used as a transmission window material. Hygroscopic material similar to Sodium Chloride (NaCl). Soluble in water, glycerine and alcohols. Slightly soluble in ether. Fairly good resistance to mechanical and thermal shock. Molecular formula: KBr. Chemical Abstracts Service (CAS) No: 7758-02-3.

Physical Data

Appearance: Odourless, white or colourless crystalline solid.
Melting point: 730°C.
Boiling point: 1380°C.
Vapour pressure: 1mm Hg at 795°C.
Specific gravity: 2.75 g cm⁻³.
Solubility in water: 53.48g/100g at 0°C.
Hardness: 6 Kg/mm².
Refractive Index: 1.54 (at 2000cm⁻¹ - wavenumbers).
Spectroscopic transmission range: 43,500 to 400 cm⁻¹ (wavenumbers).

Stability

Stable. Incompatible with strong oxidising agents, strong acids, bromine trifluoride and bromine trichloride.

Toxicology



Harmful if ingested in large amounts, if inhaled, or if in repeated contact with the skin.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material.
Allow for adequate ventilation.

Storage

Keep powder or windows stored in a cool, dry container.

Datasheet for Calcium Fluoride (CaF₂) Material

General

Known as Calcium Fluoride, Calcium Difluoride, Fluorspar or Irtran 3. When powder is fused together, is used as a transmission window material. Insoluble in water, resists most acids and alkalis. Is soluble in ammonium salts. Its high mechanical strength makes it particularly useful for high pressure work. Brittle material sensitive to mechanical and thermal shock. Does not fog. Molecular formula: CaF₂. Chemical Abstracts Service (CAS) No: 7789-75-5.

Physical Data

Appearance: Odourless, white or colourless crystalline solid.
Melting point: 1360°C.
Boiling point: 2500°C.
Solubility in water: 0.0017g/100g at 0°C.
Hardness: 158 Kg/mm².
Refractive Index: 1.40 (at 2000cm⁻¹ - wavenumbers).
Spectroscopic transmission range: 77,000 * to 900 cm⁻¹ (wavenumbers).

Stability

Stable. Incompatible with acids.

Toxicology



Harmful if ingested in large amounts, if inhaled, or if in repeated contact with the skin.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material.
Allow for adequate ventilation.

Storage

Keep powder or windows stored in a cool, dry container.
(* UV Grade material required for this range limit.)

Datasheet for Barium Fluoride (BaF₂) Material

General

Synonyms: Barium Difluoride.

When powder is fused together, is used as a transmission window material.

Very slightly soluble in water, soluble in acids and ammonium chloride. Good resistance to fluorine and fluorides. Does not fog.

Its high mechanical strength makes it particularly useful for high pressure work.

Brittle material - very sensitive to mechanical and thermal shock.

Molecular formula: BaF₂.

Chemical Abstracts Service (CAS) No: 7787-32-8.

Physical Data

Appearance: Odourless, white or colourless crystalline solid.

Melting point: 1280°C.

Boiling point: 2137°C.

Solubility in water: 0.17g/100g at 0°C.

Hardness: 82 Kg/mm².

Refractive Index: 1.45 (at 2000cm⁻¹ - wavenumbers).

Spectroscopic transmission range: 66,666 * to 800 cm⁻¹ (wavenumbers).

Stability

Stable. Incompatible with acids.

Toxicology



Harmful if ingested in large amounts, if inhaled, or if in repeated contact with the skin.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material. Allow for adequate ventilation.

Storage

Keep powder or windows stored in a cool, dry container.

(* UV Grade material required for this range limit.)

Datasheet for Zinc Selenide (ZnSe) Material

General

Toxic and hard yellow coloured crystalline powder when fused together as a solid can be used as a transmission window material or as a crystal material for attenuated total reflectance (ATR) FTIR spectroscopy. Insoluble in water, but attacked by strong acids and bases. (pH range 4 to 11 tolerant).

Organic solvents have no effect.

Fairly brittle as a window material and sensitive to thermal and mechanical shock.

Molecular formula: ZnSe

Chemical Abstracts Service (CAS) No: 1315-09-9.

Physical Data

Appearance: Yellow crystals, granular powder or amber coloured window material

Melting point: 1515°C at 1.8 atmospheres. (26.5psi)

Solubility in water: 0g/100g at 0°C.

Hardness: 120 Kg/mm².

Refractive Index: 2.43 (at 2000cm⁻¹ - wavenumbers).

Spectroscopic transmission range: 20,000 to 500 cm⁻¹ (wavenumbers).

Stability

Stable. Reacts with acids to give highly toxic hydrogen selenide. May be air and moisture sensitive. Incompatible with strong acids, strong bases and strong oxidising agents.

Toxicology



Toxic if small amounts are inhaled or swallowed. In stomach toxic hydrogen selenide (H₂Se) is liberated. Skin and eye irritant. Danger of cumulative effects from frequent handling without protection.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material. Allow for good ventilation.

Storage

Keep powder or windows stored in a cool, dry container, with appropriate safety labelling.

6. Operating Parameters of the Storm™ 10H Heated Gas Cell and Specification

The Storm™ 10H Heated Gas Cell is provided with its own dedicated 4000 Series Temperature Controller. A separate User Instruction Manual is supplied for operation of this Controller.

For operation of the Storm™ 10H Heated Gas Cell the parameters of the 4000 Series™ Temperature Controller have been factory set as shown on the following page. Not all of the displayable parameters can be changed but have been listed for reference purposes. If you ever need to change a parameter or autotune the controller for a particular temperature range, certain parameter settings will be altered. You can get back to original factory settings by reprogramming the controller with these original values.

Specifications

Accessory Type P/N GS05670

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

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

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

Displayable Parameters for Storm™ 10H Heated Gas Cell with WEST 6100+ (4000 Series) Controllers

Parameter Display (In Green)	Parameter Name	Parameter Factory Set Value
FiLt	Input Filter Time Constant	3.0
OFFS	Process Variable Offset	0
PP□□	Primary (Heat) Output Power	0
Pb_P	Primary Output Proportional Band	2.0
ArSt	Automatic Reset (Integral Time Constant)	1.14
rAtE	Rate (Derivative Time Constant)	0.18
biAS	Manual Reset (Bias)	25
SPuL	Setpoint Upper Limit	250
SPLL	Setpoint Lower Limit	0
OPuL	Primary (Heat) Output Upper Power Limit	100
Ct I	Output 1 Cycle Time	2
PhAl	Process High Alarm	250
AHy1	Alarm 1 Hysteresis	5
PLA2	Process Low Alarm	0
AHy2	Alarm 2 Hysteresis	5
APt	Auto Pre-Tune enable/disable	diSA
PoEn	Manual Control select enable/disable	diSA
SPr	Setpoint Ramping enable/disable	EnAb
rP	Setpoint Ramp Rate Value	600
SP	SP Value	0
SLoc	Set-up Lock Code	10

7. Legend (Bubble Number Part Identification)

- (1) Window for Storm™ 10H Heated Gas Cell.
- (2) Body of Storm™ 10H Heated Gas Cell.
- (3) Vacuum Valve Assembly on gas cell body (2).
- (4) End cap that holds window (1).
- (5) Silicone O-ring seal for gas cell body (2).
- (6) PTFE gasket seal for window in end cap (4).
- (7) Inlet/outlet port connection on gas cell body (2).
- (8) Mounting screw fitting for monitoring thermocouple (14), pin rod (9) and 1/16" O.D. flow tubing (12).
- (9) Pin rod for sealing of mounting screw fitting (8).
- (10) M3 x 3mm grub screw in mounting screw fitting (8).
- (11) Silicon rubber sealing pad at base of mounting screw fitting (8).
- (12) 1/16" O.D. flow tubing (100mm long) for mounting screw fitting (8).
- (13) Liquid/Gas Septum Injector Assembly.
- (14) Monitoring thermocouple for mounting screw fitting (8).
- (15) Body of the Septum Injector Assembly (13).
- (16) Silicone O-ring seal for body of Septum Injector Assembly (15).
- (17) Cap for the body of the Septum Injector Assembly (15).
- (18) Septum for the Septum injector Assembly (13).

8. Storm™ 10H Heated Gas Cell Spare Parts

- | | |
|-------------|--|
| P/N GS05020 | Pair of NaCl windows. |
| P/N GS05121 | Pair of KBr windows. |
| P/N GS05022 | Pair of CaF ₂ windows. |
| P/N GS05023 | Pair of BaF ₂ windows. |
| P/N GS05096 | Pair of ZnSe windows. |
| P/N GS05662 | Packet of 10 stainless steel flow tubes. |
| P/N GS05665 | Packet of 10 injection septa. |
| P/N GS05667 | Complete seals and gasket kit. |

This is to certify that the:

**10 cm HEATED GAS CELL & 4000 Series TEMPERATURE CONTROLLER
5670**

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. **TF5670**


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. **TF5670**

Responsible Person:

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

Signature: 
Of: Specac Ltd. **Date:** 21st Feb 2013
conforms to the above
Signature:
Of: Specac Ltd. **Date:**

Original to file/1 Copy to Customer:

FS.No.: 642-112 Rev. No. 02

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