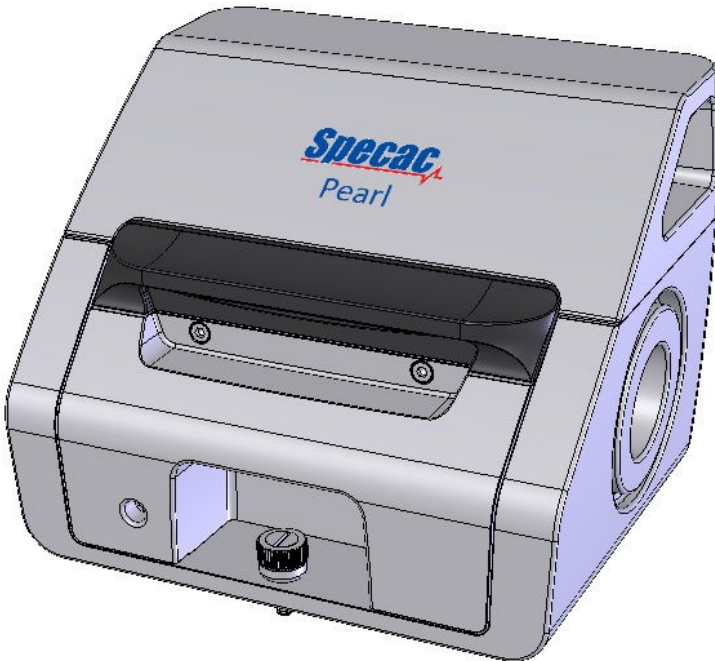




The Pearl

Liquid Transmission Accessory

User Manual



2I-31000-4

The Pearl
Liquid Transmission Accessory

User Manual

The Pearl Liquid Transmission Accessory

P/N GS31000-X

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1. Introduction

Thank you for buying a Specac product. We trust it will provide you with invaluable and excellent service in use.

The Pearl Liquid Transmission Accessory is designed for the qualitative and quantitative analysis of a very wide range of organic and aqueous based liquid sample types using the transmission technique of Infrared light passage through the sample. The liquid sample measurements are taken at ambient (room) temperatures and pressure conditions in a non-flowing environment. The principal features for the design allows for a quick, easy and consistent way of loading a liquid sample into a liquid cell assembly for a transmission spectroscopic measurement with an accurately known and repeatable pathlength for the liquid cell environment.

The Pearl Liquid Transmission Accessory consists of a Pearl optical unit which contains a complete liquid cell assembly called an Oyster. The Pearl Liquid Transmission Accessory can be installed into a very wide range of commercially available Infrared spectrometer systems via use of the Specac Benchmark™ baseplate system.

Pearl Optical Unit

The Pearl optical unit is light grey in colour but is available in a range of seven different colour handle options for a slider tray mechanism assembly where an Oyster liquid cell is installed. The colour handle options are black (B), red (R), orange (O), yellow (Y), green (G), aqua (A) and purple (P). A Pearl optical unit is offered as a GS31000-X part number with the choice of colour code letter as an extension for the particular colour handle as fitted. (e.g. GS31000-A for the aqua coloured handle.)

Oyster Liquid Cells

A complete Oyster liquid cell consists of a separate top and bottom window assembly which are brought together to create a defined, consistent and repeatable pathlength for the liquid cell for the containment of a liquid sample between the inner contacting window

surfaces. The top window assembly of the Oyster cell is placed as a precision fit over the bottom window assembly and the formed Oyster cell sits horizontally within the slider tray of the Pearl optical unit for introduction into the optical beam path of the Pearl accessory for a spectral analysis.

Oyster Cell Types

The Oyster cell can be provided as two different types of a complete liquid cell assembly fitted with either ZnSe or CaF₂ window material options. These types are:-

- 1) Where the top and bottom window surfaces for an Oyster cell are **parallel** to each other.
- 2) Where the top and bottom window surfaces for an Oyster cell have a **wedge angle** set for ***the bottom window assembly only***.

Parallel window Oyster cells will produce a fringing pattern effect (a repeating sinusoidal wave) for the Infrared spectrum collected for an empty cell. Wedge angle window Oyster cells, **will produce a greatly reduced fringing pattern** for peak amplitude by comparison.

From the fringing pattern obtained, the thickness of the Oyster cell (i.e. the pathlength), can be calculated from the following formula.

$$t = \frac{10 n}{2(f_1 - f_2)}$$

Where t = thickness in millimeters (cell pathlength), n = number of fringes (peak to peak measurement), f₁ = frequency at first peak (wavenumber position), f₂ = frequency at second peak (wavenumber position).

Note: *Measuring the period (peak to peak distance) of the residual fringing pattern obtained with a wedge angle window Oyster cell **does not give a reliable value for the average pathlength of the cell.***

For wedge angle window Oyster cells, using either ZnSe or CaF₂ windows, the wedge angle set on the bottom window assembly is circa 6 arc minutes.

When filling a parallel windowed Oyster cell with a liquid sample, for window materials with a **similar refractive index (RI)** to the liquid itself (e.g. CaF₂ windows with an RI of 1.4) the fringing pattern created for an empty cell reference is normally destroyed. The resulting infrared spectrum collected for any indicative absorption peaks measurable for the liquid sample itself may be **quantitatively** assigned. However, when using a window material such as ZnSe which typically has a **higher RI of 2.4**, any introduction of a liquid sample into the parallel windowed Oyster cell will still result in a fringing pattern being observable **along with and overlying** an infrared spectrum for the sample itself. This makes **quantification** of any spectral data unreliable.

Therefore, with use of ZnSe windows, a wedge angle window Oyster cell assembly may be preferred for use in the application where both **qualitative and quantitative** measurements are needed, because of a greatly reduced fringing pattern resulting from an initial empty cell background reference measurement.

Pathlength Options for Oyster Cells

For all Oyster cells, fixed and predetermined pathlength options are available. These pathlengths are:-

- 25microns. (0.025mm).
- 50 microns. (0.05mm).
- 100 microns. (0.10mm).
- 200 microns. (0.20mm).
- 500 microns. (0.50mm).
- 1000 microns. (1.00mm).

There is a specified tolerance of +/- 10% or 5um (whichever is the greatest), on **all** pathlength options of Oyster cell. A complete Oyster cell assembly type for a specified pathlength are offered as a GS312XX Series part number for ZnSe windowed cells and a GS313XX Series part number for CaF₂ windowed cells.

Oyster Top Window Assemblies

The Oyster top window assembly fitted with a particular window material *is common to all* of the complete Oyster cells offered. (GS312XX or GS313XX Series part numbers).

A replacement Oyster top window assembly is offered as P/N GS32200 for a ZnSe window and P/N GS32300 for a CaF₂ window.

Oyster Bottom Window Assemblies

The Oyster bottom window assembly is offered as a choice for the fitting of ZnSe or CaF₂ window material at a parallel or wedge angle window setting. A pathlength option to choose is from the 25, 50, 100, 200, 500 and 1000 micron offerings.

The Oyster bottom window assembly is offered as the following series of part numbers.

P/N GS3321X – ZnSe parallel window.

P/N GS3322X – ZnSe wedged angle window.

P/N GS3331X – CaF₂ parallel window.

P/N GS3332X – CaF₂ wedged angle window.

Hence, if a complete Oyster cell for a window material and at a specific pathlength has been provided, (from a GS312XX or GS313XX Series part number), it is only necessary to swap over and use a different **bottom window assembly** with the top window to create *a different pathlength* Oyster cell.

Note: *In changing the Oyster bottom window assembly to create a different pathlength Oyster cell, the **Oyster cell type** (parallel or wedge angle window cell) can be changed too.*

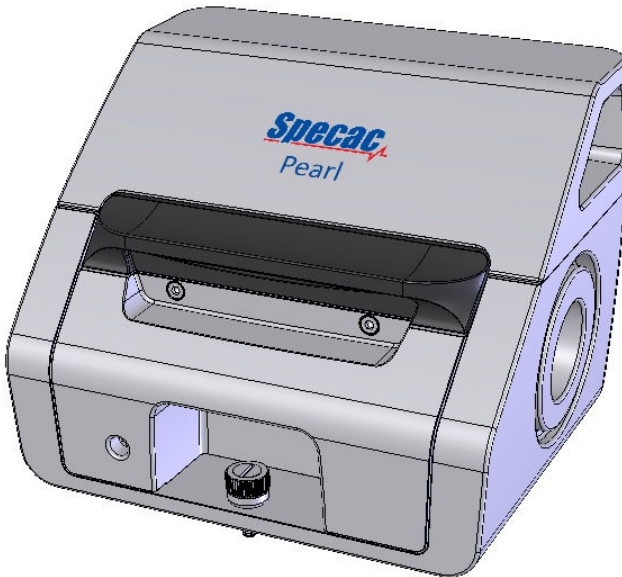
Operation of the Pearl Accessory

For operation, once a complete Oyster cell has been installed into the Pearl optical unit, it is simple and quick to place a liquid sample into the Oyster cell and to introduce (load) the filled Oyster cell into the beam path of the Pearl accessory for a spectral transmission analysis.

The all reflective gold mirrored optics used in the Pearl accessory allows for the full IR transmission range characteristics of the window material type chosen for an Oyster cell to be used. The typical transmission range offered by the window materials are as follows:

ZnSe: 20,000 cm^{-1} to 500 cm^{-1} .

CaF₂: 40,000 cm^{-1} to 900 cm^{-1} .



Pearl Liquid Transmission Accessory

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 is 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 to the Pearl Liquid Transmission Accessory, this uses different window materials for the Oyster cell assemblies where a sample is brought into contact with them for containment and to carry out the analytical spectroscopic study. As standard, Zinc Selenide (ZnSe) and Calcium Fluoride (CaF₂) are the window materials of choice that can be used.



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

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

Provided with each Oyster top and bottom window assembly is a window material safety data sheet for the window material itself that can be consulted for safe handling. A copy of each of these datasheets can also be found in this Pearl Accessory User Instruction Manual following the **Notes On Cleaning** Section found on pages 43 to 45.

3. Checklist of Contents

The Pearl Liquid Transmission Accessory is provided in its own specific packaging. The Pearl optical unit for its coloured handle and any complete Oyster cell(s) type(s), for windows and pathlength options that have been ordered will determine the items to check on delivery.

Please check for the following.

- 1 Pearl Liquid Transmission Accessory optical unit with particular colour handle. (P/N GS31000-X.)
- 1 Oyster cell complete assembly for choice of cell type, window material and pathlength. (P/N's GS312XX or GS313XX Series part number.)
Any additional complete Oyster cells (P/N's GS312XX or GS313XX Series part number) **or** Oyster bottom window assemblies (P/N's GS3321X, GS3322X, GS3331X or GS3332X Series part number) ordered with the Pearl accessory.
- 1 Ball driver 2.5mm (for optical unit cover plate removal and movable mirror alignment adjustments).
- 1 Ball driver 1.5mm (for screw tightening to hold an Oyster top window assembly in the hinge lid of the slider tray and an Oyster bottom window assembly in the recess housing part of the slider tray).
- 5 Cap head screws for Oyster bottom window (permanent) fixing.
- 1 Top window lid housing - (P/N GS33502).
- 1 Pair of purge bellows - (P/N GS10707).
- 1 Air blower (puffer device) - (P/N GS33510).
- 1 Benchmark™ baseplate for your FTIR spectrometer.
- 1 User instruction manual for Benchmark™ baseplate installation.
- 1 User instruction manual for the Pearl Liquid Transmission Accessory.
- 1 Quickstart guide for the Pearl Liquid Transmission Accessory.

Carefully remove your Pearl Liquid Transmission Accessory, Oyster cell parts and Benchmark™ baseplate from the packaging in readiness for use.

4. Installation Using Benchmark™ Baseplate

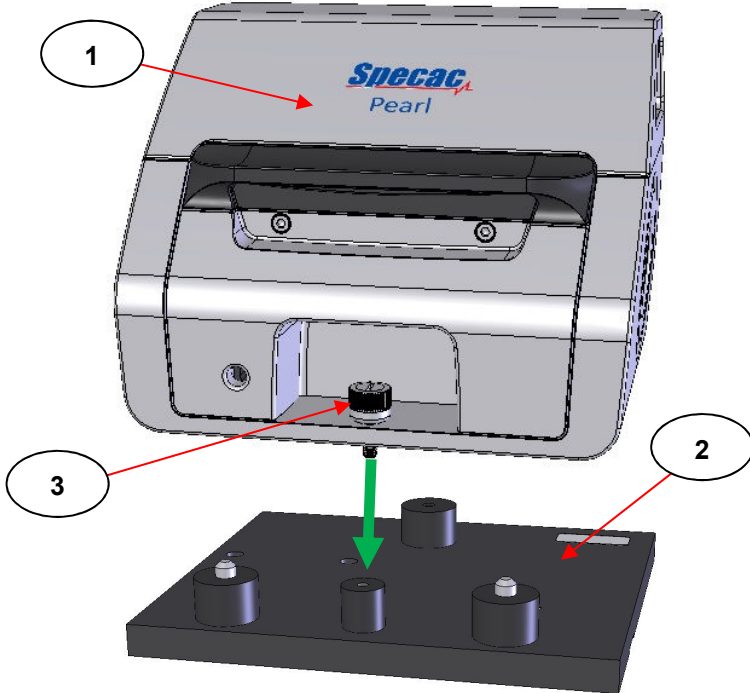


Fig 1. Pearl Liquid Transmission Accessory – Pearl Optical Unit and Oyster Cell (Within) on a Benchmark™ Baseplate

The Pearl Liquid Transmission Accessory (1) is supported on a Benchmark™ baseplate (2) when installed into a spectrometer. The Benchmark™ baseplate (2) typically has three support pillars (one flat support pillar towards the rear and two at the front with location pins) and a fourth front central pillar into which the fixing thumb screw (3) of the Pearl optical unit is tightened.

Note: *It is normally best to install the Benchmark™ baseplate in the spectrometer first before locating the Pearl Liquid Transmission Accessory onto the baseplate.*

Fixing holes and studs in the Benchmark™ baseplate (2) will vary depending upon the make and model of the spectrometer into which the Pearl accessory (1) is to be installed. For details on how to install your Pearl accessory correctly into position in the spectrometer, start by referring to the Benchmark™ Baseplate User Installation Manual guide supplied.

When the Benchmark™ baseplate has been installed, carefully align the Pearl optical unit (1) over the Benchmark™ baseplate and secure it in position by a simultaneous pushing down and clockwise rotation of the fixing thumbscrew (3) into front central location pillar of the Benchmark™ baseplate (2). Removal of the Pearl optical unit (1) away from the Benchmark™ baseplate (2) is a reverse action in anticlockwise rotation for loosening of the fixing thumbscrew (3) and separation of the parts.

5. Alignment Procedure for the Pearl Liquid Transmission Accessory

When the Pearl optical unit (1) has been installed into the spectrometer correctly on a Benchmark™ baseplate (2) there will be a need to optimize the throughput of the accessory from an alignment of the mirrors within the lower compartment area of the Pearl optical unit (1).

Note: *Any Pearl optical unit (1) will be supplied from Specac already preliminarily aligned to give an energy throughput reading when initially installed into the spectrometer sample compartment. It is necessary to finely adjust the mirror settings for optimisation of a throughput energy level specifically for an installation into any new spectrometer system.*

An alignment procedure can commence, **with or without** an Oyster cell assembly loaded into the beam path of the Pearl optical unit (1). The introduction of an Oyster cell assembly into the beam path will influence the overall throughput energy achievable depending upon the window material choice of ZnSe or CaF₂ as used in an Oyster cell.

Different spectrometers for their beam characteristics and settings will determine an energy throughput level to aim for from an alignment. A typical range can be tabulated as follows:-

Window Material (Oyster Cell Fitted)	Energy Throughput Level Compared To An Unobstructed Beam at 100% Transmission
None	40 to 70%
ZnSe	20 to 35%
CaF ₂	30 to 60%

Note: *For an initial installation and alignment on receipt of a new Pearl Liquid Transmission Accessory the alignment procedure instructions to follow are **without** any Oyster cell assembly loaded into the beam path of the Pearl optical unit (1). (See Quick Start Guide as supplied.)*

Mirrors to Align in the Pearl Optical Unit

A cutaway front view schematic to help explain the Infrared light beam passage through the Pearl Liquid Transmission accessory is shown as **Fig 2**. There are two movable mirrors (**4**) and (**5**) shown in the lower half of the Pearl optical unit (**1**) which are used for an optimum optical alignment. There are a further four fixed mirror optical components in the top half of the Pearl accessory concealed by a permanently fixed top housing cover. A fitted and loaded for sampling Oyster cell is shown as (**OC**).

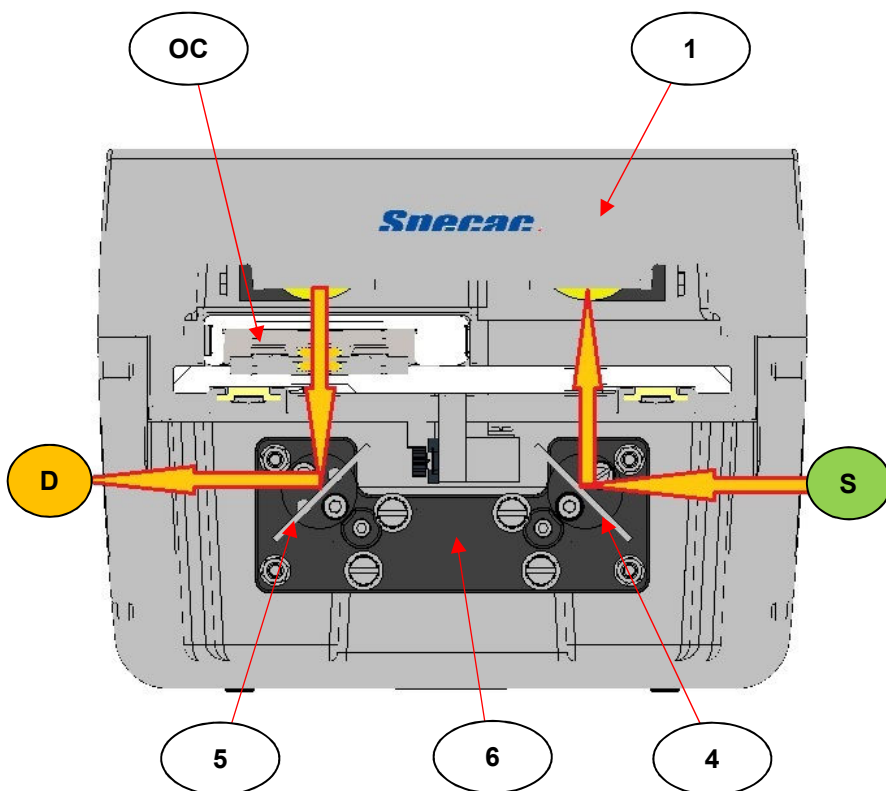


Fig 2. Infrared Light Beam Passage through the Pearl Liquid Transmission Accessory (R to L Direction)

The Pearl Liquid Transmission Accessory can be used in **any** standard spectrometer system irrespective of the beam direction of light through the sample compartment from a source (**S**) to the detector (**D**). As an example, **Fig 2.** shows a right to left (**R** to **L**) beam direction through the sample compartment from source (**S**) to detector (**D**).

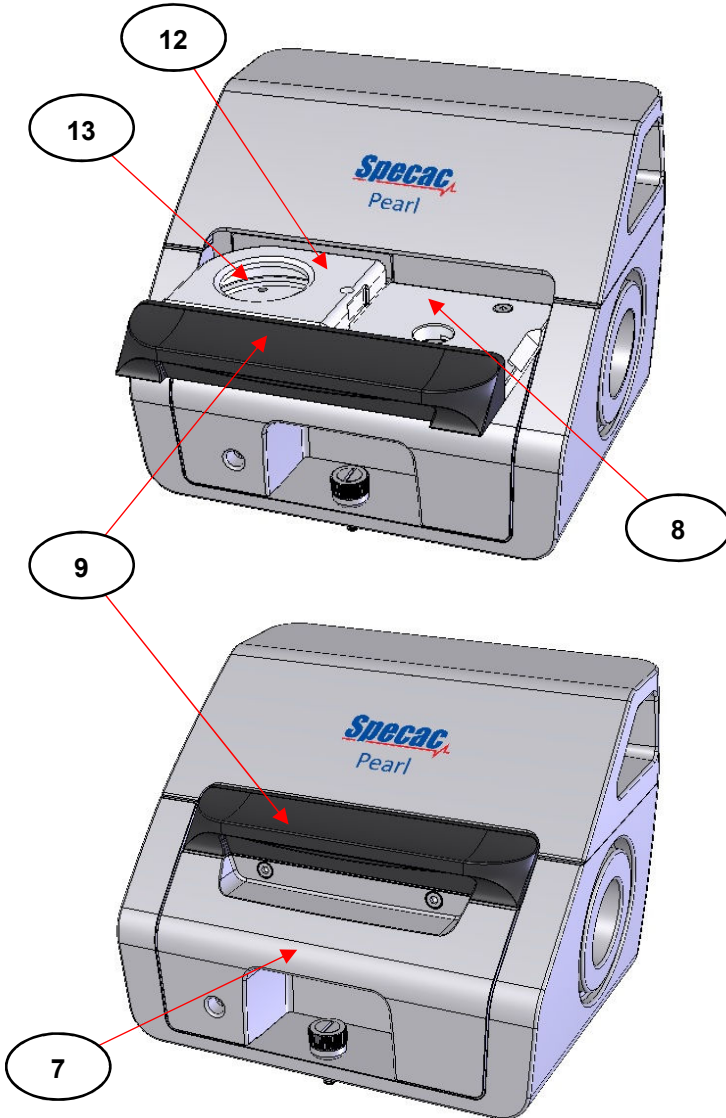
The two mirrors (**4**) and (**5**) to adjust for their alignment in an optimisation of the light beam energy through the accessory, are located within the lower section of the Pearl optical unit (**1**) on a mirror carriage frame assembly (**6**). The mirrors (**4**) and (**5**) are accessible for their adjustment via removal of a front cover (**7**) on the Pearl optical unit (**1**). (See **Figs 4A and 4B.**) The front cover (**7**) is located underneath the slider tray assembly (**8**). (See **Fig 3.**)

The Slider Tray Assembly

To remove the front cover (**7**), the slider tray assembly (**8**) **must be fully closed** and pushed into the Pearl optical unit (**1**). The slider tray (**8**) is opened and closed by pulling or pushing accordingly on the coloured handle (**9**) at the front.

The slider tray (**8**) is pulled fully open to a predefined end stop position to fit an Oyster cell for its constituent Oyster top window (**10**) and Oyster bottom window (**11**) assemblies. The slider tray (**8**) is similarly pushed fully closed to a predefined end stop position to load an Oyster cell into the Pearl optical unit (**1**) for spectral sample analysis.

See **Fig 3.** (Open (upper image) and Closed (lower image) Pearl slider tray assembly (**8**).)



**Fig 3. Pearl Slider Tray Open and Closed
(No Oyster Cell Top or Bottom Window Assemblies Fitted)**

Front Cover Removal

The front cover (7) is removed by an anticlockwise unscrewing of the two captive cap head screws (14) using the 2.5mm ball driver tool supplied. When the screws (14) are loose, pull the cover forward and then up and over to clear the central front thumbscrew fixing (3). (See Figs 4A, 4B and 5. Front cover fitted and front cover removed.)

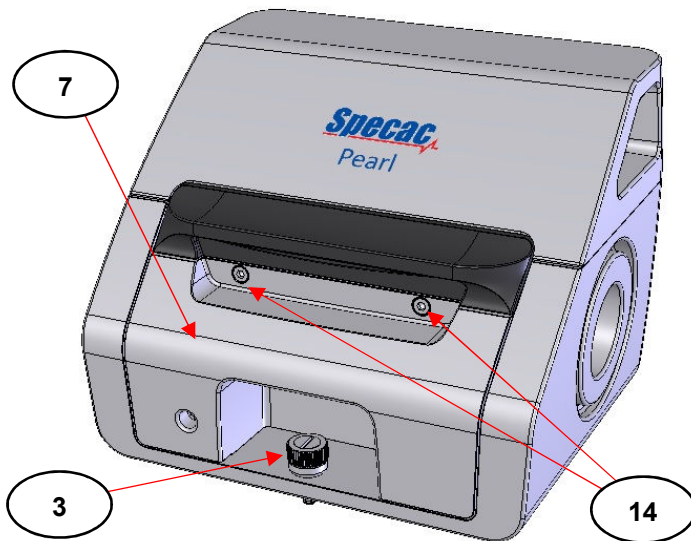


Fig 4A. Pearl Optical Unit with Front Cover Fitted

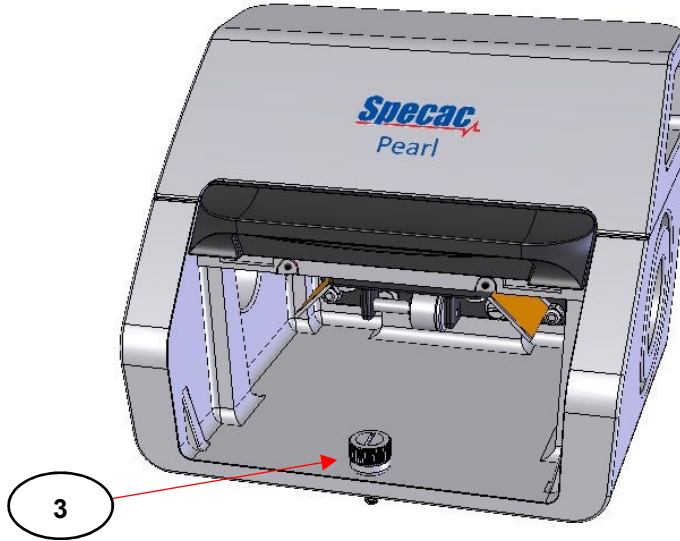


Fig 4B. Pearl Optical Unit with Front Cover Removed

When the front cover (7) has been removed to gain access to the mirror frame assembly (6), the mirrors (4) and (5) can be adjusted for **rotation** and **tilt** in their alignment. (See Fig 5.)

Alignment Procedure

For an explanation of the alignment procedure, a right to left (**R to L**), source (**S**) to detector (**D**) beam direction spectrometer system shown as Fig 2 has been used, where mirror (4) is the **initial input** mirror in the light beam path and mirror (5) is the final output mirror.

Note: For a left to right (**L to R**) source (**S**) to detector (**D**) beam direction spectrometer system, the beam sequence is reversed and mirror (5) becomes the initial input mirror and mirror (4) becomes the final output mirror.

For alignment of the Pearl Liquid Transmission Accessory, **if the beam direction of the instrument is not known**, it does not matter if mirror (5) is the initial or final output mirror, as long as it is adjusted first from following the alignment procedure as given.

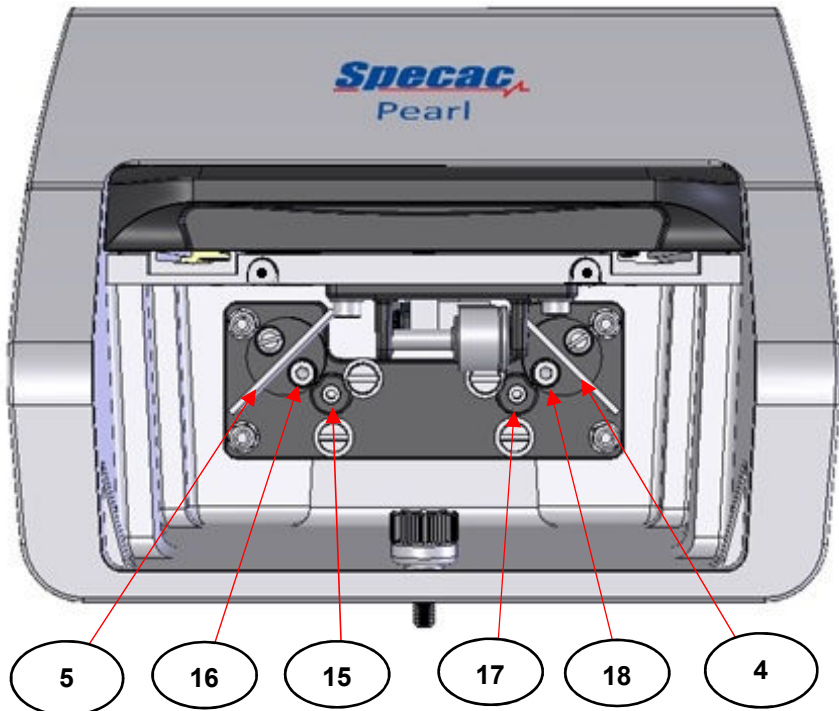


Fig 5. Access to Mirrors M1 and M6 – Front Cover Removed from the Pearl Optical Unit

A 2.5mm ball driver tool has been supplied for adjustment of the rotation and tilt screws for the mirrors (4) and (5). (Please refer to **Fig 5.** for identification of the rotation (15 and 17) and tilt (16 and 18) screws that need to be adjusted.)

User Manual

The grub screw (15) allows for **rotation** of the mirror (5) and the cap head screw (16) allows for **tilt** of the mirror (5), as fitted to the main mirror frame assembly (6).

The grub screw (17) allows for **rotation** of the mirror (4) and the cap head screw (18) allows for **tilt** of the mirror (4), as fitted to the main mirror frame assembly (6).

To begin any alignment, first establish that there is a throughput of IR light radiation energy passing through the spectrometer system from the source (S) to the detector (D) with the Pearl Liquid Transmission Accessory (1) installed correctly on its Benchmark™ baseplate (2) in the spectrometer sample compartment and with the slider tray (8) closed.

Using any appropriate beam energy monitoring signal from the IR spectrometer itself, adjust the output mirror (5) initially, **only**, for its rotation, turning screw (15) clockwise or anticlockwise. An improvement in the overall signal level at the detector may be seen from rotation of the mirror (5) in either direction. If you move away from a peak maximum throughput reading by continuing with either a clockwise or anticlockwise rotation of the screw (15), stop and slowly rotate the screw (15) in the opposite direction. When a peak maximum throughput reading has been reached, leave the mirror (5) at the new rotated setting.

Now, adjust the output mirror (5) for its angle of tilt by turning the screw (16) clockwise or anticlockwise and observe the throughput signal reading. Similarly, if by continuing with a clockwise or anticlockwise rotation of the screw (16) the peak maximum reduces, then rotate the screw (16) in the opposite direction.

When an optimum peak reading has been established for the energy passing through the Pearl optical unit (1) from rotational and tilt adjustments of the output mirror (5) **alone**, then a similar rotation and tilt adjustment can be carried out on the input mirror (4). For rotational adjustment of the input mirror (4) the screw (17) is turned and for a tilt adjustment, screw (18) is turned.

Important: *When making **any** rotation or tilt adjustment to either the output (5) or input (4) mirror, complete the action by leaving the mirror at an optimum peak signal reading before moving to the **other** mirror for any further adjustment.*

If an altering of the rotational and tilt settings of the input mirror (4) from the alignment procedure improves the energy throughput level markedly, it will be necessary to readjust the output mirror (5) again slightly for its rotation and tilt settings. This is to achieve a fine “balance” for mirrors (4) and (5) in their settings for an optimum throughput.

For some spectrometer configurations, if the Benchmark™ baseplate (2) is mounted on a rail or is movable in the spectrometer, then an overall energy throughput may be improved by moving the Pearl Liquid Transmission Accessory on the Benchmark™ baseplate to find the best position where the transmission energy throughput is at a maximum. Firmly secure the Benchmark™ baseplate (2) with the Pearl Accessory (1) attached into this new position if an improvement has been gained.

Note: *In some spectrometers it may be necessary to remove the Pearl optical unit (1) away from the Benchmark™ baseplate before a securing of the baseplate can be made. Where this is the case, ensure that the Benchmark™ baseplate does not move from its new position when removing the Pearl optical unit.*

When rotation and tilt adjustments have been completed for both the output (5) and input (4) mirrors to obtain an optimum energy throughput signal, the Pearl accessory has been aligned for use. The front cover (7) is replaced on the Pearl optical unit (1) to prevent any further adjustments of the mirror settings and sampling from the fitting of an Oyster Cell assembly into the slider tray (8) of the Pearl Optical unit (1) can begin.

Note: *When refitting the front cover (7), **only gently tighten** the captive screws (14) back into position to prevent damage to the Pearl optical unit (1).*

6. Oyster Cell Assemblies

Fitting of an Oyster Cell into the Pearl Optical Unit

A complete Oyster cell assembly that is supplied from new consists of an Oyster top window assembly (10) and an Oyster bottom window assembly (11). (See Figs 6 and 7.)

Note: *The Oyster top window assembly (10) and Oyster bottom window assembly (11) must be fitted with the same window material to create a usable Oyster Cell.*

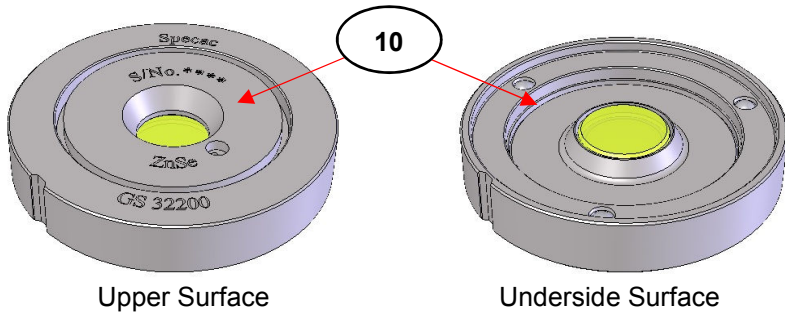


Fig 6. Oyster Top Window Assembly

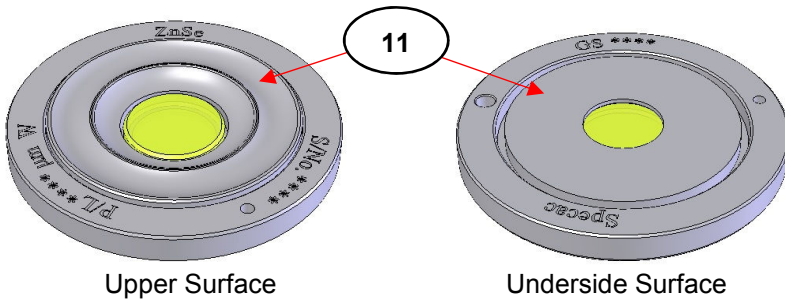


Fig 7. Oyster Bottom Window Assembly

The Oyster top window assembly (**10**) is fitted into an outer retaining top window housing part (**12**) and the Oyster bottom window assembly (**11**) is fitted into the bottom window recess housing area (**13**) of the slider tray (**8**).

When an Oyster bottom window (**11**) assembly has been fitted into position in the slider tray (**8**), the Oyster top window assembly (**10**) in its retaining top window housing part (**12**) is simply placed over the Oyster bottom window (**11**) for a precise and repeatable location of fit to form the Oyster cell.

The Oyster **top window** assembly (**10**) has been shown at **Fig 6** for both its upper surface and underside surface. The Oyster **bottom window** assembly (**11**) has been shown at **Fig 7** for both its upper surface and underside surface.

Note: *An Oyster cell is formed from a bringing together of the **underside surface** of the top (**10**) window assembly to the **upper surface** of the bottom (**11**) window assembly.*

The Oyster top window assembly (**10**) will be marked with its **GS** part number, its serial number (**S/N**) and the window material (**ZnSe** or **CaF₂**) on its upper surface for identification.

The Oyster bottom window assembly (**11**) will be marked with its **GS** part number on the underside surface for identification. On its upper surface, it will be marked with its serial number (**S/N**), its window material (**ZnSe** or **CaF₂**) and its **pathlength** in microns. By the **pathlength** mark, there will be either the letter **W** for a **wedged** bottom window assembly (**11**) or **P** for a **parallel** bottom window assembly (**11**).

With respect to the **pathlength** as marked with either a **W** or **P** setting for the window surface, the tolerance on the **pathlength** that will be set from the bringing together of the Oyster top (**10**) and bottom (**11**) window assemblies to form an Oyster cell will be +/- 10% or 5um (0.005mm), whichever is the greatest.

Fitting of an Oyster Bottom Window



Caution! - When fitting an Oyster bottom window assembly (11) into the Pearl accessory avoid touching the actual ZnSe or CaF₂ window material.

Before any Oyster cell type (parallel or wedged cell), can be used in the Pearl accessory, an Oyster bottom window assembly (11) must be fitted into position in the slider tray (8).

Take the Pearl optical unit (1) and pull out the slider tray (8) assembly of parts towards you by gripping of the handle (9). The slider tray (8) is sprung loaded with a smooth, dampened action and will come to a natural stop when it has been pulled fully out. (See Fig 8.)

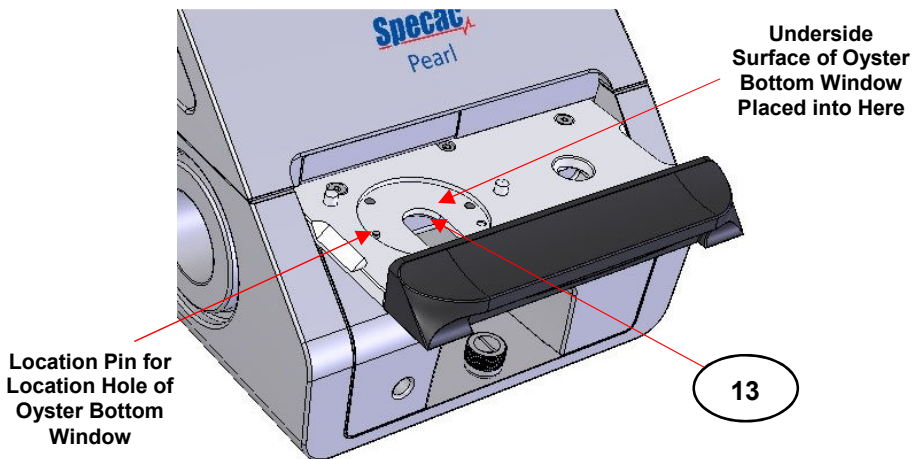


Fig 8. Slider Tray Open to Fit Bottom Oyster Window Assembly

The **underside surface** of the Oyster bottom window assembly (11) is carefully placed into the hollow aperture of the bottom recess housing area (13) of the slider tray (8). There is a location hole on the underside surface of the Oyster bottom window (11) which is aligned

with the location pin in the recess housing area (13) of the slider tray (8). (See Fig 8 for the location pin in the recess housing area (13) and Fig 9 for the location hole on the Oyster bottom window assembly.)

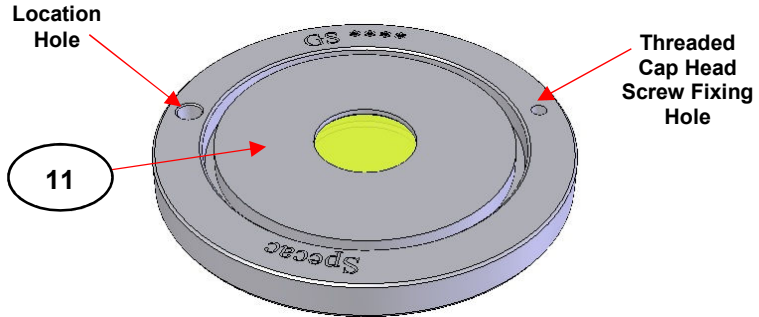


Fig 9. Location Hole of an Oyster Bottom Window Assembly (Underside Surface Shown)

Note: Before fitting the Oyster bottom window assembly (11) into the recess housing area (13), ensure that all contacting surfaces are clean and free from any obstructions to allow for a level, even and flush fit.

When the Oyster bottom window assembly (11) is correctly aligned for its location hole over the location pin, it is pulled down into its correct position in the recess housing area (13) by three small magnets.

Note: To remove and swap over for different Oyster bottom window assemblies (11) to create a new Oyster cell for a different pathlength, the Oyster bottom window assembly (11) is simply pushed out of its recess housing (13) location from below. The aperture slot in the recess housing (13) is elongated to place a finger from below to push up against the surrounding stainless steel housing of the **underside surface** of the Oyster bottom window assembly (11) and to avoid touching the ZnSe or CaF₂ window material as has been fitted.

The fitting of a specific Oyster bottom window assembly (11) into the slider tray (8), determines the window material for a **parallel** or **wedge angle** window setting and the **pathlength** of a complete Oyster cell.

Fig 10. shows when an Oyster bottom window assembly (11) has been fitted into the opened slider tray (8), and is ready to take an Oyster top window assembly (10).

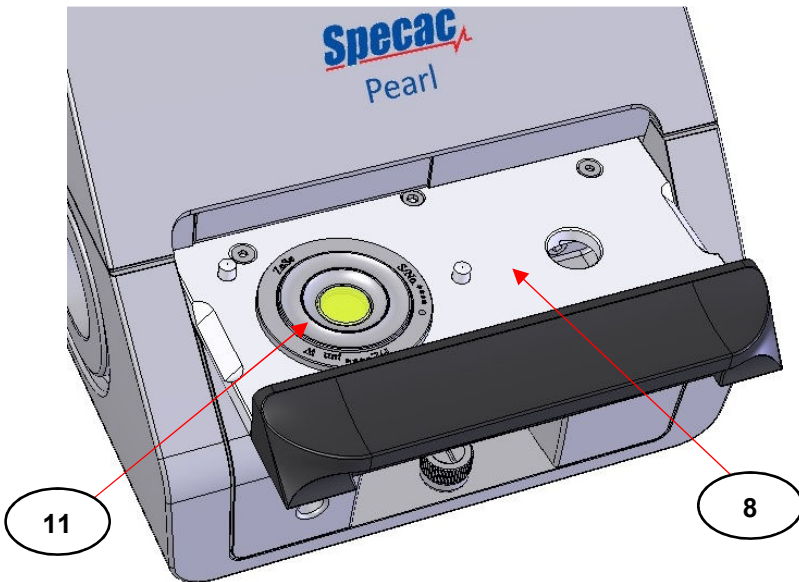
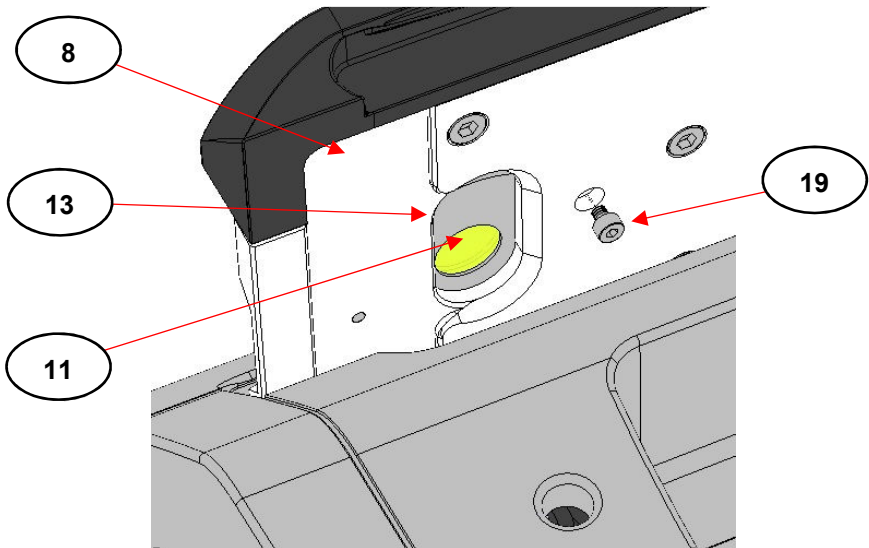


Fig 10. Oyster Bottom Window Assembly Located in the Slider Tray of the Pearl Accessory

Permanent Fixing of the Oyster Bottom Window Assembly

Using just the location hole and magnetic pull down fixings for the Oyster bottom window assembly (11) into the recess housing (13) of the slider tray (8) allows for a quick and easy changeover of Oyster bottom window assemblies if an alternative pathlength and/or wedge angle for an Oyster cell is required. (See **Note**, page 25.)

However, the fixing of the Oyster bottom window assembly (11) **can be made permanent** by the tightening of a cap head screw (19) through the underside of the recess housing into **the threaded cap head screw fixing hole** of the Oyster bottom window assembly (11). (See Figs 9 and 11.)



**Fig 11. Oyster Bottom Window Cap Head Screw Fixing
(Underside View of Slider Tray Assembly)**

To fit the cap head screw (19), place the Oyster bottom window assembly (11) into the recess housing (13) position normally, as described on pages 24 to 26.

With the slider tray assembly (8) open and placing a finger over the top surface edge of the Oyster bottom window assembly (11), invert the Pearl optical unit to gain access to the underside of the recess housing area (13). (See Fig 11.)

Note: *Be careful not to touch the Oyster bottom window (11) optical material when carrying out this action. Wear safety gloves!*

Take the cap head screw (19) and pass it through the hole on the underside of the opened slider tray assembly (8) as seen at Fig 11. Using the 1.5mm ball driver, clockwise turn the cap head screw (19) to engage with the threaded cap head screw fixing hole of the Oyster bottom window assembly (11). While pressing down gently on the Oyster bottom window assembly (11) from the topside of the opened slider tray assembly (8), gently tighten the cap head screw (19) into the threaded screw hole to secure the Oyster bottom window assembly (11) into a permanent fixed position.

To remove the Oyster bottom window assembly (11) for changing of a pathlength and/or wedge angle for an Oyster cell, the cap head screw (19) is simply fully unscrewed anticlockwise - if this screw fixing is being used.

Fitting of an Oyster Top Window



Caution! - When fitting an Oyster top window assembly (10) into the Pearl accessory avoid touching the actual ZnSe or CaF₂ window material.

When an Oyster bottom window assembly (11) has been fitted into the slider tray (8), an Oyster top window assembly (10) can be placed into position within the Pearl accessory to form **a complete Oyster cell**.

Note: *An Oyster top window assembly (10) can only be placed over the installed Oyster bottom window assembly (11), when the Oyster top window assembly (10) has been fitted correctly into its top window retaining housing (12).*

Fitting an Oyster Top Window Assembly into the Top Window Housing Lid Part

The top window housing lid part (12) is shown at Fig 12. for both its upper and underside surfaces.

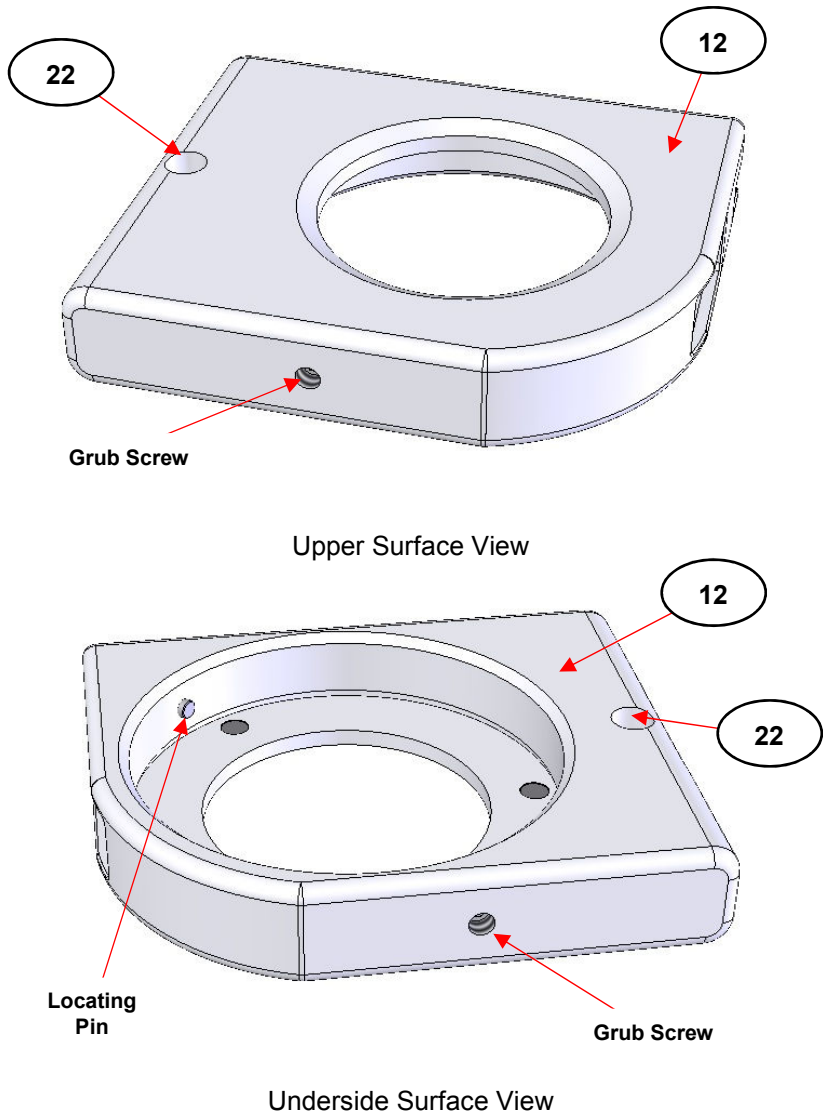


Fig 12. Upper and Underside Views of Top Window Housing Lid

There is a small locating pin inside the circular aperture of the top window housing (12) as shown from the underside view. There is also a small grub screw within a hole in the top window housing (12) as shown from both the upper and underside views.

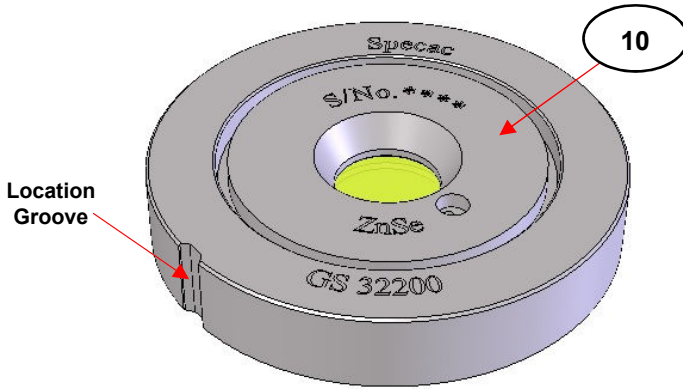


Fig 13. Location Groove of an Oyster Top Window Assembly (Upper Surface Shown)

As shown at **Fig 13**, there is a little notch in the outer stainless steel support ring edge of the Oyster top window assembly (10). Take the Oyster top window assembly (10) and very carefully place the **upper surface** of this assembly into **the underside** of the top window housing (12), aligning the notch of the Oyster top window assembly (10) with the small locating pin in the top window housing (12). Gently push the Oyster top window assembly (10) into the top window housing (12) as far as it will travel until it comes to rest against three small circular magnets in the top window housing (12). The magnets will hold the Oyster top window assembly (10) into position, but it is held securely and correctly from tightening of the grub screw. (See **Fig 14.**)

Take the 1,5mm size Allen key ball driver tool supplied and place it into the hole from outside of the top window housing (12), to gently tighten the grub screw from a clockwise turning. (See **Fig 14.**)

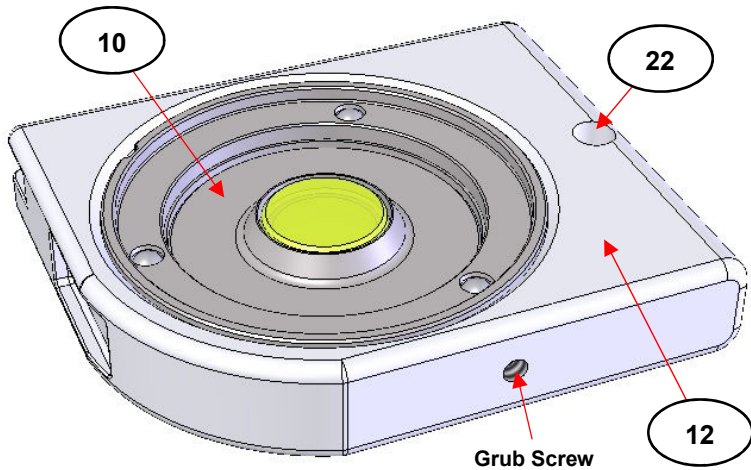


Fig 14 – Oyster Top Window Assembly Fitted into the Top Window Housing Part

Note: The Oyster top window assembly (10) must only be held **gently** by the grub screw when fitted into the top window housing (12). Over tightening of the grub screw risks distortion of the Oyster top window assembly (12) and inconsistent pathlengths being created for an Oyster cell.

Forming an Oyster Cell

When either a ZnSe or CaF₂ Oyster top window assembly (10) has been fitted into the top window housing (12), a complete Oyster cell can be formed to use by placing this complete assembly of parts over an installed Oyster bottom window assembly (11).

Note: The choice in fitting an **Oyster bottom window assembly (11)** determines the type of Oyster cell that will be formed, for a parallel or wedged window cell and for a specific pathlength. The top (10) and bottom (11) **window material** must be the same to create a usable Oyster cell.

With the slider tray (8) open and an Oyster bottom window assembly (11) installed, take the Oyster top window (10) in the top window housing (12) assembly and gently lower the underside surface of the Oyster top window (10) towards and to cover over the upper surface of the Oyster bottom window assembly (11). (See Fig 15.)

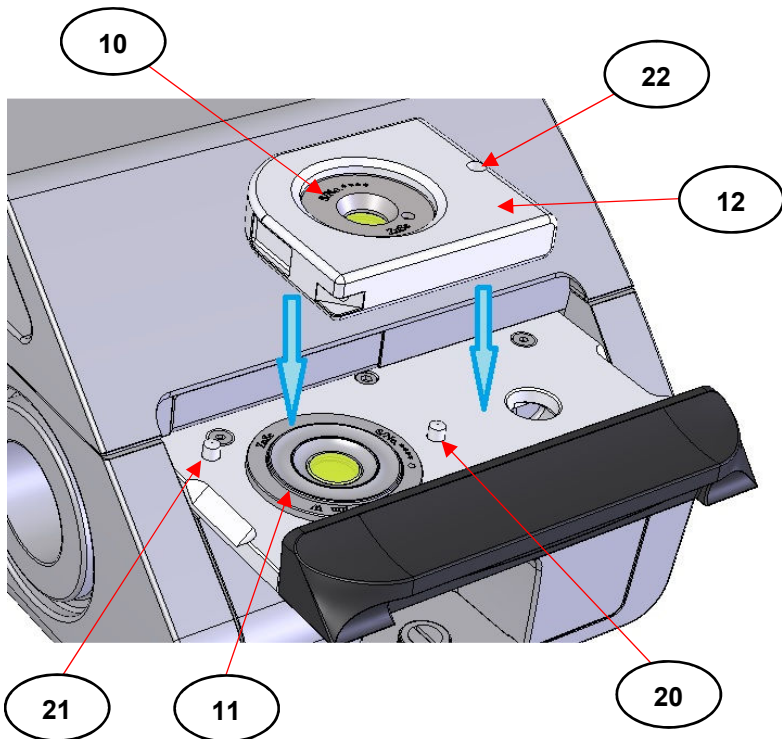


Fig 15. Oyster Top Window Assembly in Top Window Housing Being Positioned over the Oyster Bottom Window Assembly

There are two location pins (20 and 21) on the slider tray (8) that allow for a precise and repeatable positioning of the Oyster top window assembly (10) in the top window housing (12) over the Oyster bottom window assembly (11). The central location pin (20) of the slider tray (8) passes through the location hole (22) on the top window housing

(12) and the curved edge of the top window housing (12) contacts against the side location pin (21) for a correct fitting of the Oyster top window assembly (10) over the installed Oyster bottom window assembly (11) to form a complete Oyster cell. (See Fig 16.)

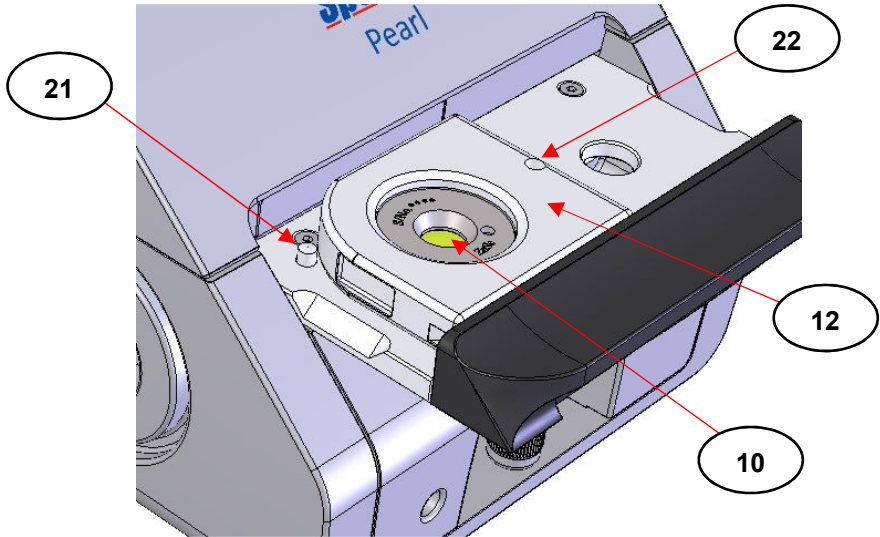


Fig 16. Oyster Top Window Assembly in Top Window Housing Correctly Fitted over the Oyster Bottom Window Assembly

Removal of an Oyster top window assembly (10) away from the Oyster bottom window assembly (11) is a simple matter of holding the edges of the top window housing (12) and pulling this complete assembly of parts up vertically away from the locating pins (20) and (21).

With the Oyster top window (10) and top housing part (12) assembly lifted up and removed, this assembly can be inverted and placed to rest on the right hand side of the opened slider tray (8). Effectively, this allows for ease of access to the Oyster top (10) and bottom (11) window surfaces for their cleaning.

The location hole (22) of the top window housing (12) is placed over the central location pin (20) to hold the inverted top window housing (12) with the Oyster top window assembly (10) securely into position for cleaning of the window surfaces after sampling. (See Fig 17.)

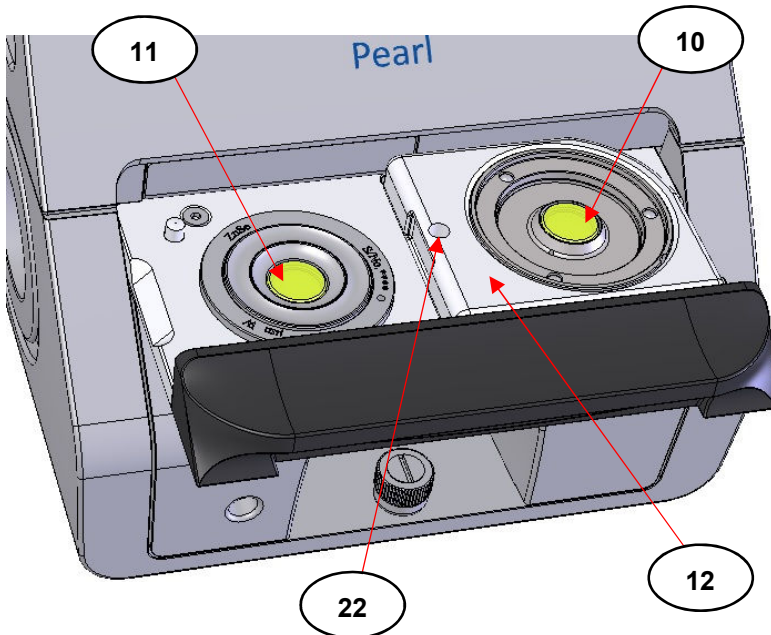


Fig 17. Inverted Top Window Housing with Oyster Top Window Assembly Positioned for Window Surface Cleaning

Loading an Oyster Cell for Spectral Measurements

When a complete Oyster cell has been formed as positioned in the slider tray (8), (see Fig 16), using the handle (9), push the slider tray (8) to its closed position to load the Oyster cell inside the Pearl optical unit (1) for a spectral measurement to be taken. The slider tray (8) will come to a natural stop and be retained when it has been pushed fully in for a closed position. (See Fig 18.)

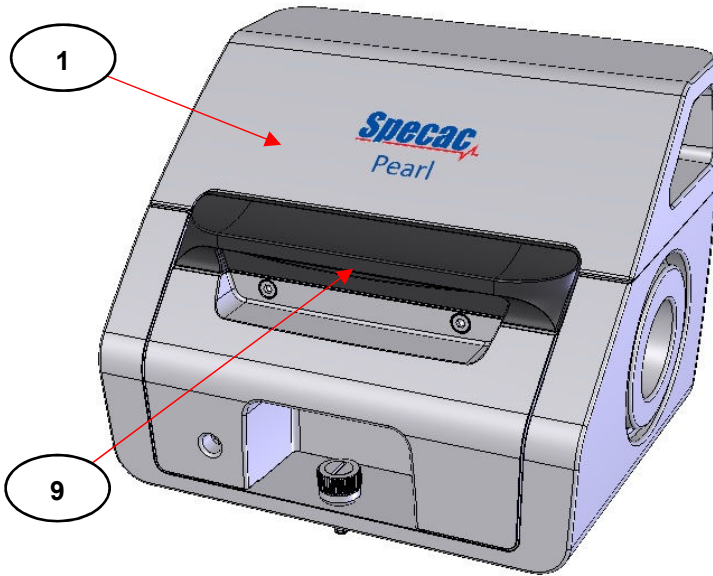


Fig 18. Pearl Accessory with the Slider Tray closed and loaded with an Oyster Cell for Measurement

Measurement of an Empty Oyster Cell

From an initial forming of an Oyster cell by placing an Oyster top window assembly (10) in its top window housing (12) over an installed Oyster bottom window assembly (11), an “empty” Oyster cell is created. (No liquid sample is filling the pathlength created between the two inner window surfaces.)

At this stage an empty Oyster cell can be loaded into the Pearl optical unit (1) for an optical beam passage of light through the Oyster cell assembly. Depending upon whether a **parallel** or **wedge angle** window complete Oyster cell assembly has been formed, will determine what type of reference (background) IR spectrum is to be collected.

For **parallel window** Oyster cells, where the top and bottom window surfaces for a complete Oyster cell are parallel to each other, a fringing pattern will be produced for the spectral throughput range of the window material as fitted. The fringing pattern and periodicity for the peaks and troughs of a parallel Oyster cell can be used in a calculation of the average pathlength of the cell.

For **wedge angle window** Oyster cells, where the top and bottom window surfaces for a complete Oyster cell have a wedge angle determined by the bottom window assembly as fitted, **a reduced absorbance intensity fringing pattern** (as compared to a parallel window Oyster cell), will be produced for the spectral throughput range of the window material as fitted. The fringing pattern and periodicity for the peaks and troughs of a wedge angle Oyster cell **does not give a reliable value** from a calculation of the average pathlength of the cell.

This is tabulated as follows:-

Oyster Cell Type and Window Material	Fringing Pattern Absorption with an Empty Cell (No Liquid Sample)
Parallel - ZnSe	Strong
Wedged - ZnSe	Weaker than parallel - ZnSe
Parallel - CaF ₂	Weaker than parallel - ZnSe (*)
Wedged - CaF ₂	Weaker than parallel - CaF ₂ (*)

(*) Because the refractive index (1.4) of CaF₂ window material is lower than ZnSe (2.4), an Oyster cell with CaF₂ windows will produce a less intense absorption fringing pattern than that observed with ZnSe material. The intensity and periodicity of the fringing pattern peaks are also determined by the actual pathlength of the complete Oyster cell.

Filling an Oyster Cell with a Liquid Sample

A liquid sample can be introduced to create a “filled” Oyster cell assembly in two ways.

- 1) Spotting or placing a liquid sample onto the Oyster bottom window assembly (**11**), prior to positioning an Oyster top window assembly (**10**) in the top window housing (**12**) to form a complete Oyster cell. (**Spotting Method.**)

- 2) Filling the pathlength space created for an empty Oyster cell by injection of a fluid through a filling port (24) on the Oyster top window assembly (10). (**Injection Method.**)

Spotting a Liquid Sample (Spotting Method)

Spotting a liquid sample is a quick and easy way of introducing a sample into an Oyster cell for spectral measurement.

Very mobile to quite viscous fluid types can be spotted onto the window surface of an Oyster bottom window assembly (11) when it has been fitted into the recess housing (13) part of the slider tray (8).

Note: *The Spotting Method may be the preferred method of sample introduction for thicker, less mobile fluids for a transmission spectral analysis, which are difficult to introduce into a conventional liquid sample transmission cell via an Injection Method from a syringe.*

To introduce a liquid sample from the Spotting Method, pull the slider tray (8) fully out of the Pearl optical unit (1) by the handle (9). A complete Oyster cell for choice of Oyster bottom window (11) and Oyster top window assembly (10) in the top window housing (12) should already be installed for use.

Note: *At this stage for introduction of a liquid sample into an Oyster cell by the Spotting Method for subsequent spectral analysis, it is likely to be a normal practice that an “empty” Oyster cell assembly has already been measured to collect a background comparison reference spectrum for spectral subtraction purposes.*

Separate (lift), the Oyster top window (10) and top window housing (12) assembly of parts away from the bottom window assembly (11). Invert the lifted away assembly and place it carefully to sit flat onto the right hand side of the slider tray (8) via fitting of the location hole (22) over the central location pin (20). (See **Fig 17.**)

Take a liquid sample and spot this sample onto the window surface of the Oyster bottom window assembly (11). (A dropping pipette can be used to dispense the liquid sample.) The amount of liquid sample to spot into position is determined by the pathlength of an Oyster cell that has been formed. As a general guide, only a couple of drops of aqueous or organic based liquids or solutions are required for a 100 microns pathlength parallel or wedge angle Oyster cell.

With a liquid sample spotted in place on the bottom window assembly (11), carefully lift up the top window housing (12) and the Oyster top window assembly (10) from its right hand side resting position of the slider tray (8), and flip it over to lower over the bottom window assembly (11) for a correct position of fit as shown. (See Fig 16.)

As the top and bottom window surfaces come together to squeeze the liquid sample, any excess liquid that has been spotted in position will be displaced to run off into the overflow catchment well around the bottom window surface area. The liquid sample contained in position between the windows inner surfaces will now be at the pathlength setting as stated and marked on the Oyster bottom window assembly (11) that has been fitted.

Note: *When the Oyster top (10) and bottom (11) window assemblies are being brought together, make sure that there are **no trapped air bubbles** within the liquid sample contained between the window surfaces. For more mobile fluids it can be better to have a slight excess of liquid sample spotted into place to squeeze and displace rather than too little sample, to minimize the possibility of any bubble formation. Slight up and down manipulation of the Oyster top window (10) surface in the top window housing (12) as this assembly fits over the central location pin (20) and before it comes to its final rest position to form a complete Oyster cell can help to dispel any bubbles.*

When the Oyster cell has been filled with a liquid sample using the Spotting Method, the Oyster cell can be loaded into the Pearl optical unit (1) for a spectral collection of the liquid sample by closing the slider tray (8). (See Fig 18.)

Filling an Oyster Cell by Liquid Injection (Injection Method)

As an alternative to spotting a liquid sample into position for analysis within an Oyster cell, a liquid sample can be introduced into an empty Oyster cell construction using an Injection Method, via an injection port hole (23) on the upper surface of the Oyster top window assembly (10). (See Fig 19.)

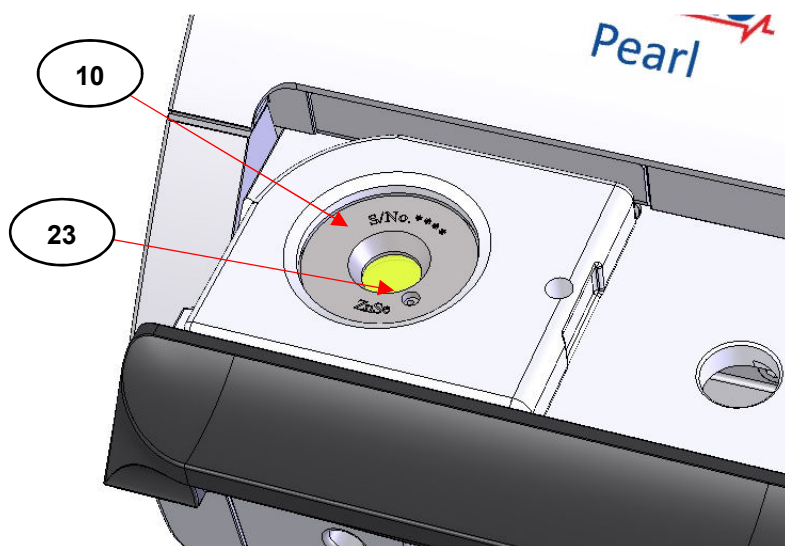


Fig 19. Injection Hole of an Oyster Cell for Injection Method

The Injection Method may be a preferred way of handling for the introduction of very volatile, or highly mobile solvents or solutions for transmission analysis, rather than use of the Spotting Method. There could be a risk that an amount of a very volatile or mobile solvent or solution has significantly evaporated away before the Oyster top window assembly (10) can be placed over a liquid sample on the lower window assembly (11) using the Spotting Method.

To introduce a liquid sample from the Injection Method, pull the slider tray (8) fully out of the Pearl optical unit (1) by the handle (9). A complete Oyster cell for choice of Oyster bottom window (11) and Oyster top window assembly (10) in the top window housing (12) should already be installed for use.

It is not necessary to lift and separate the top window housing (12) with a fitted Oyster top window assembly (10) away from the Oyster bottom window assembly (11) using the Injection Method. A liquid sample is injected straight into the pathlength cavity space of the formed Oyster cell assembly as it is presented on opening of the slider tray (8). This would be a normal sequence of events for sample introduction after an empty Oyster cell has been measured to obtain a reference background spectrum to subtract from the liquid sample spectral conditions.

Take a syringe with a needle tip and inject a liquid sample type into the Oyster cell via the injection hole port (23). As a guide, typically 20 microlitres of a liquid sample is sufficient to fill a 100 micron pathlength cell. It is important not to overfill the Oyster cell with a sample, although there is a surrounding overflow "well" area to accommodate any excess liquid sample. When filling the Oyster cell try not to introduce any air bubbles into the liquid cell environment.

Note: *The design of the Oyster top window assembly (10) for the injection hole port (23) introduces a liquid sample into the surrounding overflow well recess of the Oyster bottom window assembly (11). The introduced liquid flows via a capillary action to cover both window surfaces to fill the liquid cell pathlength cavity. Filling of the cavity is observed as the liquid "front" moves quickly across the window surfaces. The design of the Oyster top (10) and bottom (11) window assemblies when brought together also traps and minimises the escape of any volatile vapours from the cell environment whilst the Oyster cell is loaded into the Pearl optical unit (1) for any spectral analyses.*

When the Oyster cell has been filled with a liquid sample using the Injection Method, the Oyster cell can be loaded into the Pearl optical unit (1) for a spectral collection of the liquid sample by closing the slider tray (8). (See **Fig 18.**)

Cleaning an Oyster Cell

For the cleaning of an Oyster cell after sampling, it doesn't matter if the Spotting or Injection Method has been used for filling.

When an IR spectrum has been collected for a liquid sample, to gain access to the Oyster cell for cleaning, pull the slider tray (8) fully out of the Pearl optical unit (1) by the handle (9). Lift up and away the top window housing (12) fitted with the Oyster top window assembly (10), invert it and place it carefully to sit flat onto the right hand side of the slider tray (8) using the central location pin (20) through the location hole (22). (See as **Fig 17.**)

Cleaning of the Oyster top (10) and bottom (11) window surfaces is simply achieved by removing any excess sample by wiping it away carefully using dry, soft tissues. This can then be followed by using a **moistened** soft tissue with a suitable cleaning solvent and then a final clean using another dry tissue. (See Notes on Cleaning – page 43.)

Tip: *If a long pathlength Oyster cell has been used (e.g. 1000 microns), there may be some excess liquid laying on the bottom window assembly (11) surface and in the surrounding overflow well recess when the top window assembly (10) has been removed. This excess liquid may be sucked away for saving if necessary by careful use of a syringe or dropping pipette prior to cleaning the Oyster bottom window assembly (11) with a soft, dry tissue.*

If using a volatile solvent (e.g. acetone) when cleaning any window materials for a liquid transmission cell measurement, it may require a little time for any residual vapours to disappear after contact. Therefore, after finally cleaning the Oyster top (10) and bottom (11) window assemblies with a dry tissue, it may be preferable to wait for 30 seconds or so, before the Oyster cell is reconstructed and loaded into the Pearl optical unit (1) for a background measurement.

Volatile Liquid Vapour in the Optical Unit

If analyzing volatile liquid samples contained within an Oyster cell, after the spectral measurement and when the slider tray (8) is opened to gain access for cleaning, there may be some residual, lingering vapour from the sample remaining within the optical unit (1) of the Pearl accessory itself. Any traces of vapour will need to be removed from within the optical unit (1) prior to a new sample analysis.

Note: *Any residual vapour effects may be seen from taking a new sample spectrum when compared to the original background spectrum from reintroducing a cleaned and empty Oyster cell back into the optical unit (1) by closing of the slider tray (8).*

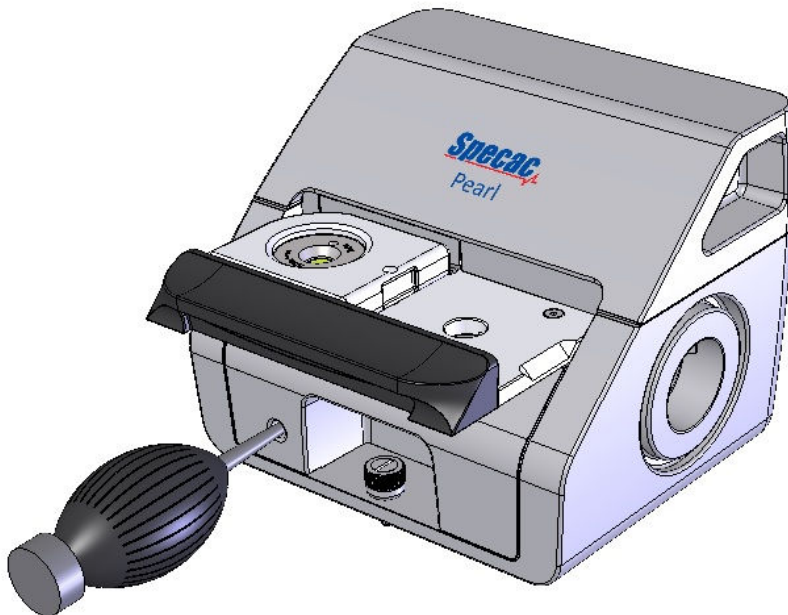


Fig 20. Puffer Device Inserted into Purge Tube Hole of Front Cover to Expel Volatile Vapours from the Optical Unit

As stated in the “Cleaning an Oyster Cell” section (page 41), when using a highly volatile solvent like acetone for cleaning, be careful that the vapour from the solvent does not encroach into the optical unit (1) itself via the opened slider tray (8). It is always good practice to use a minimum of solvent for cleaning purposes rather than an excess. Light moistening of a tissue with a solvent to wipe away at the Oyster cell parts, prior to wiping the window surfaces clean again with a dry tissue is normally all that is required.

If there is any presence of lingering, volatile liquid sample vapour(s) within the optical unit (1) after sampling, a small air blower/puffer device has been supplied to help expel the vapour. The puffer device is inserted into the purge tube hole (24) on the front cover (7). (See Fig 20.)

With the slider tray (8) **remaining open**, two or three pumps on the bulb of the puffer device is sufficient to blow any volatile vapours present out of the Pearl optical unit (1). With removal of the vapour, the Pearl Accessory is now ready to accept a new, clean Oyster cell for both background and sampling measurement regimes.

Note: *If there is a readily available and reliable supply of N₂ gas (laboratory gas supply etc), this could be used to introduce into the optical unit (1) via attachment of a purge tube through the purge hole (24) on the cover (7) to constantly maintain the local environment within the Pearl accessory. A constant, small positive pressure flow of N₂ gas also helps to remove any potential build-up of volatile vapours that could be present in the optical unit (1). (Please see Section 7, Purging the Pearl Optical Unit, on pages 48 to 51 for more information.)*

Notes on Cleaning

When cleaning the ZnSe or CaF₂ window of an Oyster top (10) or bottom (11) window assembly in preparation for a new sample measurement, it is **very important to take care** to avoid damage to the window materials. As also mentioned in the Safety Considerations (Section 2, page 9), of these two window materials, ZnSe is potentially the most hazardous in terms of risk of toxicity.



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

A useful feature of the Pearl Liquid Transmission Accessory is the capability for removal of an Oyster top (10) and bottom (11) window assembly away from the slider tray (8) of the Pearl optical unit (1), for a more thorough cleaning of the window parts if necessary. For example, it may be useful to remove the Oyster cell components away from the Pearl optical unit (1) and carry them to a safe area for solvent cleaning and wiping with a tissue and therefore minimise any risk of contamination being carried over to other components of the Pearl Liquid Transmission Accessory with the Oyster cell parts in situ. However, in most instances the ease and quickness in separation away of the Oyster top (10) from the Oyster bottom (11) window assembly, with placement of the Oyster top window (10) in the top window housing (12) on the opened slider tray (8), is sufficient to gain access to these parts for effective and safe cleaning between samples. (As described on pages 33 to 34 – see **Fig 17.**)

Solvents such as water, methanol and acetone are suitable to use for cleaning purposes. Sample solutions that fall within the pH range of pH4 to pH11 are tolerated by the ZnSe window material. Stronger acids and bases will damage ZnSe irreparably. Strong base ammonium hydroxide solutions will damage CaF₂ window material.

When wiping away any liquid sample, use very soft tissues to avoid scratches being caused on the surface of the Oyster cell window material. However, if any small scratches and blemishes do appear on the window surfaces, they will not significantly affect the performance of the Pearl accessory.

In common and for general use, it will only be necessary to wipe and clean away at the window surfaces of the Oyster top (10) and bottom (11) window assemblies that contact with a liquid sample. Use a **moistened**, soft tissue only, rather than flooding the window area to be cleaned with a solvent or solution to then wipe away.

If possible try to avoid any solvent or cleaning solution materials from getting to the opposite window surface of the Oyster top (10) and bottom (11) window surfaces that contact a sample, particularly if the Oyster cell components have been removed from the slider tray (8) for remote cleaning. There is a risk that any dried solvent or solution constituents that have been introduced to this window surface of an Oyster top (10) or bottom (11) window assembly could be measured as an “impurity” in any “background” spectrum to be collected, and so this contaminant would need to be removed before any further sampling can continue.

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.

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.)

7. Purging the Pearl Optical Unit

When the Pearl Liquid Transmission Accessory has been installed and aligned in the spectrometer there is an option for purging the Pearl optical unit (1) if this is necessary.

Note: *Purging the Pearl optical unit (1) with nitrogen (N₂) or dry air may be needed to stabilise the local surrounding atmospheric conditions for measurement of a sample within an Oyster cell and/or to remove residual Carbon Dioxide (CO₂) and excess water vapour vibrational features from any resulting IR spectrum. It is also recommended **if using volatile liquid samples** in an Oyster cell, that a purge gas is maintained to prevent potential ingress of any volatile components into the Pearl optical unit (1).*

Fitting a Purge Tube

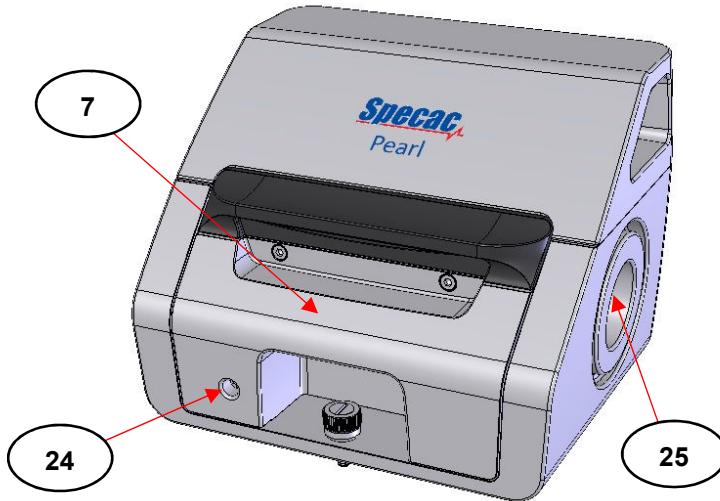


Fig 21. Purge Connection (24) and Aperture Ports (25) on the Pearl Optical Unit

On the front cover (7) there is a small hole (24) into which 1/4" O.D. hard plastic tubing can be inserted to provide a gas supply such as N₂ that creates a purged environment within Pearl optical unit (1). (See Fig 21.) Purge tubing inserted through the hole (24) is held fast by a small curved grip plate (26) behind the front cover (7). (See Fig 22.)

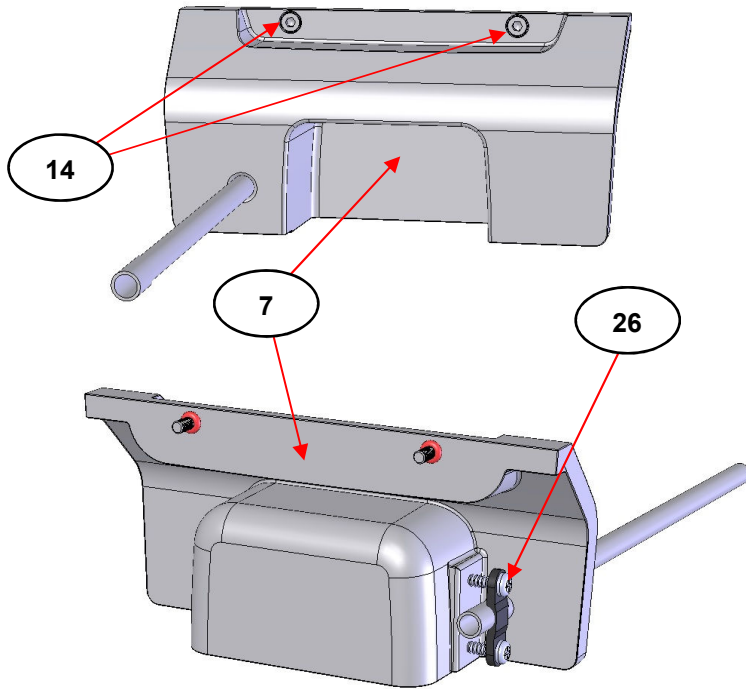


Fig 22. Front Cover with Purge Tube – Front and Back Views

To introduce any purge tubing the front cover (7) is removed from the Pearl optical unit (1) by unscrewing anticlockwise the two captive cap head screws (14) using the 2.5mm ball driver tool supplied.

Note: The slider tray (8) must be fully closed to allow for a removal of the front cover (7).

The screws holding the curved grip plate (26) affixed to the back of the front cover (7) are loosened to allow the open end of the purge tubing which has been introduced into the hole (24) from the front side of the front cover (7) to pass through between the grip plate (26) and its clamp plate. When a small length of tubing has passed through between the grip (26) and clamp plates, the screws are **gently** retightened to hold the purge tubing firmly into position. The front cover (7) with attached purge tubing can now be refitted to the Pearl optical unit (1).

Maintaining a Purged Environment

To help maintain a stable or purged environment within the Pearl optical unit (1), a pair of purge bellows (27) (P/N GS10707) have been supplied with the Pearl Liquid Transmission Accessory for attachment to the optical units two aperture ports (25). (See Fig 21 and Fig 23.)

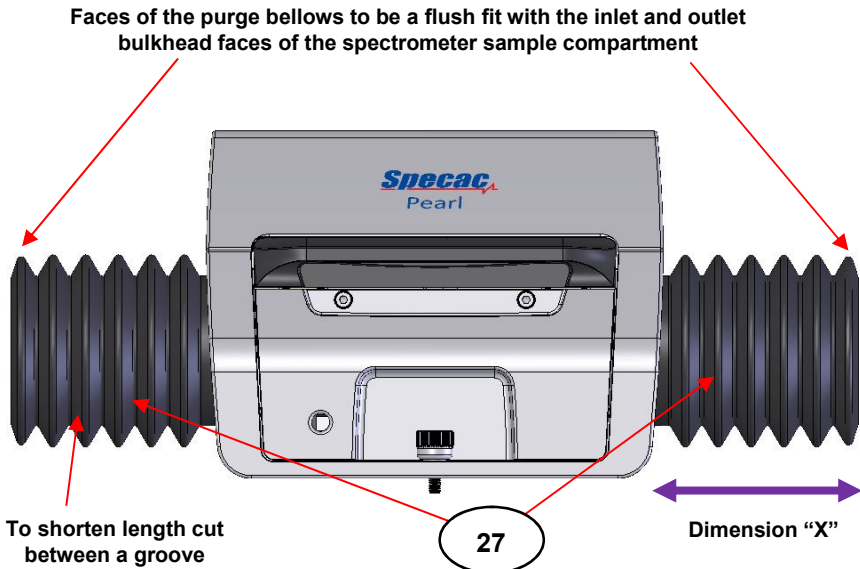


Fig 23. Pearl Liquid Transmission Accessory fitted with Purge Bellows

The purge bellows (27) grip fit between the circular recess and around the circumference protrusion part of the aperture ports (25). They fit the aperture port from one end only of their design as seen in **Fig 23**. The purge bellows (27) bridge any gap between the Pearl optical unit (1) and the spectrometers sample compartment bulkheads from the source (S) and to the detector (D). (Refer to **Fig 2**. for optical beam pathway.) They help to contain any purged environment created within the Pearl optical unit (1) whilst installed. If the gap/distance between the Pearl optical unit (1) and bulkhead is small, the purge bellows (27) can be cut to size for a better compressible fit. It is best to cut all the way around and in between one of the ridged grooves in the purge bellows moulding itself when shortening them for length of fit.

Tip: *It is recommended that purge bellows (27) are fitted during use to help stabilize the spectrometer/instrument background even if purging is not to be carried out.*

Fitting the Purge Bellows P/N GS10707

1. With the Pearl Liquid Transmission Accessory attached to the Benchmark™ baseplate and installed in position in the sample compartment, measure the distance between the spectrometers source (S) and detector (D) bulkhead/side walls and the side end faces of the Pearl optical unit (1). (This is dimension “X”).
2. Using a sharp blade, cut lengths of the flexible purge bellows (27) which are equivalent to the measured length (“X”) allowing for an additional 10mm for each length to cut. (e.g. X + 10mm.)
3. Unscrew the fixing thumbscrew (3) of the Pearl optical unit (1) from the Benchmark™ baseplate and remove the Pearl Liquid Transmission Accessory from the sample compartment.
4. Fit the shortened flexible purge bellows (27) over both end apertures (25) on the Pearl optical unit (1).

Replace the Pearl Liquid Transmission Accessory with the fitted purge bellows (27) back onto the Benchmark™ baseplate. Ensure that the purge bellows (27) are not obstructing the optical beam and re-secure the Pearl optical unit (1) back into position using the thumbscrew (3).

8. Sampling with the Pearl Liquid Transmission Accessory



Important Note for Usage!

The ZnSe and CaF₂ window materials as used in the Oyster top (**10**) and bottom (**11**) window assemblies have their own specific hardness and chemical resistance characteristics and should be treated accordingly.

The window materials have been sealed into position against a 316 grade stainless steel (EN 1.4401) support ring housing for both an Oyster top window (**10**) and Oyster bottom window (**11**) assembly. The outer ring components of both Oyster window assemblies are made from 420 grade stainless steel (EN 1.4021).

For both ZnSe and CaF₂ Oyster window assemblies, these window materials are **glue sealed** to their stainless steel support ring housings using a special chemically resistant adhesive.

The type of liquid sample (organic, aqueous, acid or base) that is to be analysed determines primarily **the choice of window material** to use that will withstand chemical contact to the sample.

Note: *Strong acids (pH1 to pH3) and strong bases (pH12 to pH14) will irreparably damage ZnSe window material.*

As a general rule for sampling, try to keep the contact time of **any sample with any window material** to a minimum, such that the integrity of the window material and constituent components (seals and stainless steel support ring housings) of the top (**10**) and bottom (**11**) window assemblies can be maintained for as long as possible. As soon as a specific liquid sample has been analysed, remove the sample and clean the Oyster cell window assembly components accordingly in readiness for a new sample.

Preparing the Pearl Liquid Transmission Accessory for Analysis

For the ZnSe and CaF₂ Oyster Cell assemblies the active sampling volume of a liquid to be analysed extends across the whole surface area of the 10mm diameter aperture for the window and the pathlength of the complete Oyster Cell type being used. It is therefore necessary to have enough liquid sample to completely fill any volume space created in an Oyster Cell from the Oyster top (**10**) and bottom (**11**) window assembly components being brought together, to interact fully with a transmitting IR beam.

Typically the amount of liquid sample needed to fill between the windows of an Oyster Cell assembly for the particular pathlength of liquid cell created is tabulated as follows:-

Pathlength (Microns)	25	50	100	200	500	1000
Liquid Amount (Microlitres)	5	10	20	40	100	200

Sampling Procedure

The procedure for collecting a liquid sample transmission IR spectrum requires, typically, obtaining a background spectrum first as a reference using the Pearl Liquid Transmission Accessory with **no sample** in position for an installed Oyster cell assembly and then repeating the procedure with a sample filling **the same Oyster cell**.

Collecting a Background Spectrum

1. Choose a ZnSe or CaF₂ windowed Oyster cell assembly to be used in the Pearl optical unit (**1**).

2. Attach the Pearl optical unit (1) to the installed Benchmark™ baseplate and fit the chosen wedged or parallel Oyster bottom window assembly (11) into the opened slider tray (8) of the Pearl optical unit (1). Fit a chosen Oyster top window assembly (10) – same window material as being used for the Oyster bottom window assembly (11) - into the top window housing (12). Form an empty Oyster cell in placing the Oyster top window assembly (10) in the top window housing (12) over the bottom window assembly (11) in the opened slider tray (8).
3. The Pearl Liquid Transmission Accessory should already be aligned to register an acceptable signal throughput when an empty (no sample) Oyster cell is loaded into the sampling position. (Closing of the slider tray assembly (8).) Having ensured that the ZnSe or CaF₂ windows of the Oyster cell are clean, proceed to collect a background spectrum using any preferred acquisition conditions set on the spectrometer.

Collecting IR Transmission Spectra of Liquid Samples

For analyzing a liquid sample, an empty Oyster cell needs to be filled with a liquid sample. This is achieved by using either the Spotting Method or Injection Method for a liquid sample introduction. (Please refer to Section 6), pages 37 to 40.)

1. A **background spectrum** as described in steps 1 to 3 (see pages 53 to 54), must be collected first and saved as a reference.
2. Follow the Spotting Method or Injection Method in filling an Oyster cell with a liquid sample to the point of loading the filled Oyster cell into the Pearl optical unit (1) by closing the slider tray (8).
3. Collect and record a transmission IR spectrum for the loaded liquid sample using **the same acquisition conditions** set on the spectrometer for collection of a reference background spectrum.

After the liquid sample has been analysed, remove the liquid sample as soon as possible from the Oyster cell assembly and clean the Oyster cell components in readiness for a new sample. (See Cleaning an Oyster Cell and Notes on Cleaning – pages 41 and 43 to 45.)

Liquid Sample Types That Can Be Measured

The Pearl Liquid Transmission Accessory has been designed principally for a quick, easy and consistent way of loading a liquid sample into a liquid cell assembly for a transmission spectroscopic measurement with an accurately known and repeatable pathlength for the liquid cell environment.

Volatile and Mobile Liquids

The Injection Method for a liquid sample introduction into an Oyster cell may be a preferred way of handling for the introduction of very volatile, or highly mobile solvents or solutions for transmission analysis. There could be a risk that an amount of a very volatile or mobile solvent or solution has significantly evaporated away before the Oyster top window assembly (10) can be positioned over a liquid sample on the Oyster bottom window assembly (11) using the Spotting Method.

Viscous Liquids

The Spotting Method of a liquid sample introduction into an Oyster Cell is the preferred method for viscous or less mobile types of liquid and fluid samples. Hence these types of liquid samples can be measured via use of the Pearl Liquid Transmission Accessory in place of conventional liquid transmission cells whereby it would be difficult to introduce the sample into the liquid cell environment unless the liquid sample itself has been diluted by a more mobile “carrier” solvent. Therefore, the Pearl Liquid Transmission Accessory allows for a less mobile liquid sample type, like a thick oil or grease sample, to be introduced “as is” into a liquid transmission cell environment for an IR spectral measurement and with no prior sample preparation steps involved from a dilution.

Pastes (As Analysed Using A Mull Cell)

A well-established IR measurement technique is use of a **mull cell** for sample types approximating to pastes and thick immobile fluids for their nature. (At ambient (room) temperature and pressure conditions.) A mull is generally a mixture of a **solid sample** that has been very finely ground to form a paste with liquid paraffin (Nujol) or Fluorolube (a fluorinated lubricant) when the solid sample type cannot be formed into a KBr pellet for transmission spectroscopy purposes of measurement. A finely ground solid sample acts as a “suspension” within the Nujol or Fluorolube fluid solvents as the paste sample.

If the solid sample type cannot be analysed by the classic transmission KBr pellet measurement, an Attenuated Total Reflectance (ATR) technique may be employed for a direct measurement of the sample type instead. However, ATR is a **surface analysis technique** and spectral results may not yield detail for a sample “bulk” make up that might be more readily obtainable using a transmission technique.

For a mull cell measurement, the paste sample is supported between two IR light transmitting windows, sometimes with an optional pathlength spacer between the windows, and a transmission measurement is made to collect an IR spectrum for the paste sample. The mull cell is built as a “sandwich” construction with the paste sample being placed on to the surface of one window, whereby it is then squashed between this window and the other. An optimum strength IR spectrum is obtainable depending on the initial concentration (amount) of the ground solid sample suspended in the Nujol or Fluorolube fluid and how closely the window surfaces need to be brought together to create an acceptable pathlength to obtain the optimum strength IR spectrum. Therefore, in most instances of mull cell use it is practically better to use just the windows with no pathlength spacer between them for a quick **qualitative** IR spectrum to be obtained.

An Oyster cell assembly as used in the Pearl Accessory can be used with the Spotting Method of sample introduction to act as a mull cell for Nujol and Fluorolube **solid/paste** like samples, **but the pathlength of the Oyster Cell is known** because of its construction and design, as

opposed to a conventional mull cell window and optional spacer design. Therefore, when an optimum strength of IR spectrum for absorbance data information is obtained for the paste sample using an Oyster Cell, the exact pathlength knowledge can be applied to make both a **qualitative** as well as a **quantitative** measurement for a mull sample type.

9. Pearl Liquid Transmission Accessory “Bubble Numbers” Part Identification List

- (1) Pearl Liquid Transmission Accessory (optical unit).
- (2) Benchmark™ baseplate.
- (3) Fixing thumbscrew of the Pearl optical unit to the Benchmark™ baseplate.
- (4) Adjustable mirror (for alignment).
- (5) Adjustable mirror (for alignment).
- (6) Mirror carriage frame for adjustable mirrors (4) and (5).
- (7) Front cover of Pearl optical unit (1).
- (8) Slider tray assembly.
- (9) Handle of the slider tray assembly (8).
- (10) Oyster top window assembly.
- (11) Oyster bottom window assembly.
- (12) Top window housing part. (To hold an Oyster top window Assembly (10)).
- (13) Recess housing part. (To hold an Oyster bottom window Assembly (11)).
- (14) Captive screw for front cover (7).
- (15) Rotation screw for adjustable mirror (5).
- (16) Tilt screw for adjustable mirror (5).
- (17) Rotation screw for adjustable mirror (4).
- (18) Tilt screw for adjustable mirror (4).
- (19) Cap head screw for Oyster bottom window (11) permanent fixing.
- (20) Central location pin for top window housing (12).
- (21) Side location pin for top window housing (12).
- (22) Location hole of top window housing (12).
- (23) Injection hole port on the Oyster top window assembly (10).
- (24) Aperture ports on Pearl optical unit (1).
- (25) Purge hole on front cover (7).
- (26) Grip plate for purge tubing.
- (27) Purge bellows.

10. Pearl Liquid Transmission Accessory Spare Parts

Pearl Liquid Transmission Accessory (Optical Unit)

P/N GS31000-A – Pearl Unit – Blue.
P/N GS31000-B – Pearl Unit – Black.
P/N GS31000-G – Pearl Unit – Green.
P/N GS31000-O – Pearl Unit – Orange.
P/N GS31000-P – Pearl Unit – Purple.
P/N GS31000-R – Pearl Unit – Red.
P/N GS31000-Y – Pearl Unit – Yellow.

Oyster Complete Cells

P/N GS31211 – Oyster – Complete ZnSe Parallel 50um.
P/N GS31212 – Oyster – Complete ZnSe Parallel 100um.
P/N GS31213 – Oyster – Complete ZnSe Parallel 200um.
P/N GS31214 – Oyster – Complete ZnSe Parallel 500um.
P/N GS31215 – Oyster – Complete ZnSe Parallel 1000um.
P/N GS31216 – Oyster – Complete ZnSe Parallel 25um.

P/N GS31221 – Oyster – Complete ZnSe Wedged 50um.
P/N GS31222 – Oyster – Complete ZnSe Wedged 100um.
P/N GS31223 – Oyster – Complete ZnSe Wedged 200um.
P/N GS31224 – Oyster – Complete ZnSe Wedged 500um.
P/N GS31225 – Oyster – Complete ZnSe Wedged 1000um.
P/N GS31226 – Oyster – Complete ZnSe Wedged 25um.

P/N GS31311 – Oyster – Complete CaF₂ Parallel 50um.
P/N GS31312 – Oyster – Complete CaF₂ Parallel 100um.
P/N GS31313 – Oyster – Complete CaF₂ Parallel 200um.
P/N GS31314 – Oyster – Complete CaF₂ Parallel 500um.
P/N GS31315 – Oyster – Complete CaF₂ Parallel 1000um.
P/N GS31316 – Oyster – Complete CaF₂ Parallel 25um.

P/N GS31321 – Oyster – Complete CaF₂ Wedged 50um.
P/N GS31322 – Oyster – Complete CaF₂ Wedged 100um.
P/N GS31323 – Oyster – Complete CaF₂ Wedged 200um.
P/N GS31324 – Oyster – Complete CaF₂ Wedged 500um.
P/N GS31325 – Oyster – Complete CaF₂ Wedged 1000um.
P/N GS31326 – Oyster – Complete CaF₂ Wedged 25um.

Oyster Top Window Assemblies

P/N GS32200 – Oyster – Top ZnSe.

P/N GS32300 – Oyster – Top CaF₂.

Oyster Bottom Window Assemblies

P/N GS33211 – Oyster – Bottom ZnSe Parallel 50um.

P/N GS33212 – Oyster – Bottom ZnSe Parallel 100um.

P/N GS33213 – Oyster – Bottom ZnSe Parallel 200um.

P/N GS33214 – Oyster – Bottom ZnSe Parallel 500um.

P/N GS33215 – Oyster – Bottom ZnSe Parallel 1000um.

P/N GS33216 – Oyster – Bottom ZnSe Parallel 25um.

P/N GS33221 – Oyster – Bottom ZnSe Wedged 50um.

P/N GS33222 – Oyster – Bottom ZnSe Wedged 100um.

P/N GS33223 – Oyster – Bottom ZnSe Wedged 200um.

P/N GS33224 – Oyster – Bottom ZnSe Wedged 500um.

P/N GS33225 – Oyster – Bottom ZnSe Wedged 1000um.

P/N GS33226 – Oyster – Bottom ZnSe Wedged 25um.

P/N GS33311 – Oyster – Bottom CaF₂ Parallel 50um.

P/N GS33312 – Oyster – Bottom CaF₂ Parallel 100um.

P/N GS33313 – Oyster – Bottom CaF₂ Parallel 200um.

P/N GS33314 – Oyster – Bottom CaF₂ Parallel 500um.

P/N GS33315 – Oyster – Bottom CaF₂ Parallel 1000um.

P/N GS33316 – Oyster – Bottom CaF₂ Parallel 25um.

P/N GS33321 – Oyster – Bottom CaF₂ Wedged 50um.

P/N GS33322 – Oyster – Bottom CaF₂ Wedged 100um.

P/N GS33323 – Oyster – Bottom CaF₂ Wedged 200um.

P/N GS33324 – Oyster – Bottom CaF₂ Wedged 500um.

P/N GS33325 – Oyster – Bottom CaF₂ Wedged 1000um.

P/N GS33326 – Oyster – Bottom CaF₂ Wedged 25um.

Other Spares

P/N GS33500 - Top ZnSe Oyster window in top window housing assembly lid.

P/N GS33501 - Top CaF₂ Oyster window in top window housing assembly lid.

P/N GS33502 - Top window housing lid for Oyster top window assembly.

P/N GS33510 - Air blower/puffer device for Pearl.

11. Pearl Liquid Transmission Accessory Technical Specifications

	Oyster ZnSe	Oyster CaF₂
Refractive Index of Window at 1000cm⁻¹	2.4	1.4
Aperture of Oyster Cell	10mm dia.	10mm dia.
Accessory Transmission Range	20,000 - 500 cm ⁻¹	40,000 - 900 cm ⁻¹
Oyster Cell Types	Parallel windows. Wedged windows at 6 arc minutes.	Parallel windows. Wedged windows at 6 arc minutes.
Pathlength Sizes of Oyster Cell	25um 50um 100um 200um 500um 1000um	25um 50um 100um 200um 500um 1000um
Pathlength Tolerance	+/- 10% or 5um whichever is the greatest	+/- 10% or 5um whichever is the greatest
Oyster Top Window Assemblies Materials	316 grade stainless steel support housing. Window glue sealed into position.	316 grade stainless steel support housing. Window glue sealed into position.
Oyster Bottom Window Assemblies Materials	316 grade stainless steel support housing. Window glue sealed into position.	316 grade stainless steel support housing. Window glue sealed into position.
Pearl Accessory Dimensions Width (W), Depth (D), Height (H)	W = 160mm D = 129mm H = 121mm	W = 160mm D = 129mm H = 121mm

12. Pearl Liquid Transmission Accessory Serial Numbers

A Pearl Liquid Transmission Accessory and any Oyster Cells (top and bottom window assemblies) will be provided marked with an identifying serial number.

For the Pearl optical unit (**1**), the serial number is a five figure number prefixed by a letter and found on a silver coloured label. For any Oyster top (**10**) and bottom (**11**) window assemblies, the serial number is engraved on the stainless steel ring housing. Please use the table below to make a note of your received Pearl and Oyster Cell parts.

Pearl/Oyster Part Number and Description	Serial Number
P/N's GS31000-X - Pearl Optical Unit (where X is the colour for the handle on the Pearl optical unit.)	
P/N GS32200 (ZnSe) - Oyster Top Window Assembly	
P/N GS32300 (CaF ₂) - Oyster Top Window Assembly	
P/N's GS3321X Series (ZnSe, Parallel) – Oyster Bottom Window Assembly	
P/N's GS3322X Series (ZnSe, Wedged) – Oyster Bottom Window Assembly	
P/N's GS3331X Series (CaF ₂ , Parallel) – Oyster Bottom Window Assembly	
P/N's GS3332X Series (CaF ₂ , Wedged) – Oyster Bottom Window Assembly	

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