Liquid Pack

User Manual

2I-01140-4

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Liquid Pack P/N GS01140

CONTENTS

1. Introduction

Thank you for buying a product from Specac.

The Liquid Pack P/N GS01140 consists of the Specac Omni Cell system (P/N GS01800) with some additional windows, spacers and mulling solvents that have been combined together for the analysis of a wide range of liquid sample types.

The Liquid Pack can be offered alone as a cost effective way to procure an Omni Cell system, but when combined with particular solid and gas pack offerings, forms a part offering for the analysis of liquid samples from the individual Specac **Starter Kit** options that are available.

The Starter Kit options from the liquids, solids and gas pack combinations available are as follows:-

Basic Starter Kit P/N GS01180 (Consists of Liquid Pack P/N GS01140 and Basic Solid Pack P/N GS01150).

Analyst Starter Kit P/N GS01185 (Consists of Liquid Pack P/N GS01140 and Advanced Solid Pack P/N GS01160).

Research Starter Kit P/N GS01190 (Consists of Liquid Pack P/N GS01140, Basic Solid Pack P/N GS01150 and Quest ATR Accessory P/N GS10802).

Advanced Starter Kit P/N GS01195 (Consists of Liquid Pack P/N GS01140, Advanced Solid Pack P/N GS01160 and Quest ATR Accessory P/N GS10802).

A Gas Pack P/N GS01170 can be offered for inclusion to any of the above Starter Kit offerings. Respectively, the Starter Kit part numbers become GS01181, GS01186, GS01191 and GS01196 with inclusion of the Gas Pack.

2. Checklist of Contents

Check that the following items have been supplied in the Liquid Pack carry case.

- P/N GS01800 Omni Cell body and mount components (1 assembly).
- P/N GS01811 Potassium Bromide (KBr) liquid cell rectangular windows - one drilled hole and one plane window (2 pairs).
- P/N GS01812 Calcium Fluoride (CaF2) liquid cell rectangular windows - one drilled hole and one plane window (2 pairs).
- P/N GS01831 Potassium Bromide (KBr) mull cell circular windows, two plane windows (2 pairs).
- P/N GS01864 Packet of ten assorted rectangular PTFE spacers – two each of 0.05mm, 0.1mm, 0.2mm, 0.5mm and 1.00mm thickness.
- P/N GS01871 Packet of five circular spacers for mull cell assemblies – 0.1mm thickness.
- P/N GS01110 Luer syringe 2ml volume.
- P/N GS03620 Bottle of Nujol (liquid paraffin) 25mls.
- P/N GS03621 Bottle of Fluorolube (fluorinated mulling oil) 25ml.

Remove the Liquid Pack parts carefully from the carry case and prepare the items for use.

Note: *The KBr windows P/N's GS01811 and GS01831 are hygroscopic by nature and should be retained in their protective packing to keep them dry and free of moisture for as long as possible before use.*

3. Use of the Liquid Pack Items

Please refer to the supplied Omni Cell Instructions in Section 4 of this instruction manual for the Liquid Pack, to understand how the Omni Cell system is used.

Demountable liquid cells (with the KBr and CaF2 rectangular windows and spacers) and demountable mull cells (with the circular KBr windows and spacers) can be constructed from the Omni Cell body, windows and spacers contained in the pack.

The Luer syringe supplied is used to help for the introduction of liquid samples (solvents or solutions) into a demountable liquid Omni Cell assembly.

If analysing a solid sample as a paste by the mull technique, the circular KBr windows are used, with or without the circular spacers. The mulling fluids Nujol and Fluorolube have been provided with the Liquid Pack for use in the mull technique.

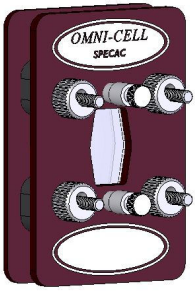
Nujol is a liquid paraffin based solution and when used for mulling, strong carbon to hydrogen bond absorptions are exhibited in the infrared spectrum. The carbon to hydrogen bond absorptions that may be present in the sample itself are masked by those from the Nujol mulling agent.

Fluorolube is a fluorocarbon based solution and exhibits strong carbon to fluorine bond absorptions from 1300cm^{-1} onwards to 400cm^{-1} in the Mid IR spectrum. The useful range for observation of a sample in a Mid IR spectrum when using Fluorolube as the mulling agent is 4000cm^{-1} to 1300cm^{-1}.

Therefore, if possible, it is preferable to run a sample as both a Nujol mull and a Fluorolube mull. This allows for all of the spectral features of the sample to be seen in two separate infrared spectra, because the regions masked by each specific mulling agent are unaffected in the other spectrum.

4. Omni Cell Instructions for Use

Introduction



The Omni Cell has been designed to study liquid solutions in demountable or sealed liquid cell assemblies or as liquid mulls for both Dispersive and FTIR instruments at ambient temperatures and pressures. The Omni Cell is supplied in separate parts: cell body, spacers and windows for assembly by the user. The cell body accepts a standard gasket/window/spacer construction, which is contained between front and back anodised aluminium plates. The plates are of the standard 3" x 2" dimension allowing the cell to be held in all spectrometer mounting systems via the back plate. Rectangular windows are used for liquid cell applications, whereas circular windows are used for mull cell applications.

Operation

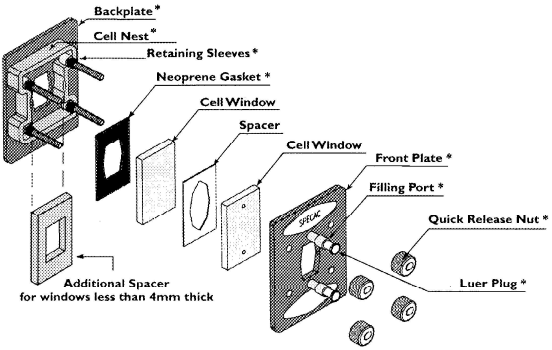
A typical Omni Cell, liquid or mull type, is constructed as shown (Construction Section page 7). Demountable liquid cells have separate windows and a PTFE spacer and sealed liquid cells have windows amalgamated to a lead spacer. The mull cell has circular windows and a PTFE spacer. The front plate has a PTFE gasket permanently bonded into position to allow sealing between the front plate and front window.

The 4 quick release nuts are tightened to provide a sufficient seal between all of the components but not over-tightened such that the windows could break. Window materials have their own physical characteristics; some are soft and can deform, others are hard and brittle. It is a matter of practice and familiarity with the cells and specific window material that determines the ideal sealing conditions.

A recommended method for assembly is to place the back plate onto a flat surface. Build up the cell components as in the diagrams, but before fixing the quick release nuts, hold the front plate centrally and apply an even pressure over the whole assembly. Slip on the quick release nuts and tighten until just holding. The nuts can then be tightened further, but do so in a diagonal sequence.

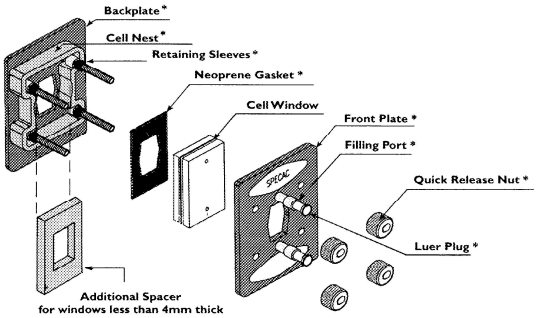
The rectangular windows used are nominally 4mm thick. ZnSe, Fused Silica, AgBr and Si windows are 2mm thick. With the thinner liquid cell windows it is necessary to use an additional spacer (P/N GS01893) placed between the back plate and neoprene gasket shown in all diagrams.

Construction: Demountable Cell



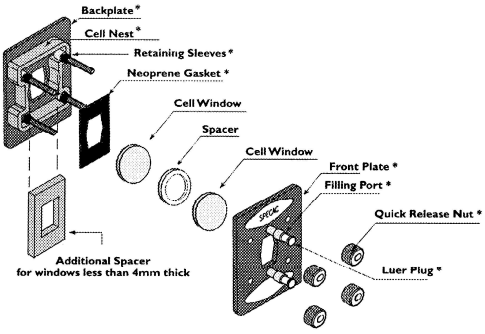
* Asterisked items are included with the Omni-Cell mount P/N GS01800

Construction: Sealed Cell



* Asterisked items are included with the Omni-Cell mount P/N GS01800

Construction: Mull Cell



* Asterisked items are included with the Omni-Cell mount P/N GS01800

Filling the Liquid Cell

A Luer Syringe P/N GS01110 can be used to introduce a solution into the Omni Liquid Cell via the Luer fittings. Lay the Omni Cell on a flat surface, and remove the PTFE plugs and fit the syringe into one of the Luer ports. Allow the cell to fill with sample by gentle pressure onto the syringe. (*Tip: Holding the Omni Cell at an upwards angle whilst filling can help remove any trapped air.*) When the solution is visible in the "open" Luer port remove the syringe and replace the PTFE plugs into the Luer ports. Any seepage around the Luer port when fitting the PTFE plugs can be wiped away with a tissue.

Note: *When using fairly viscous solutions with short pathlength cells (less than 0.012mm) care must taken when filling that there are no trapped pockets of air in the cell. An incomplete filled cell will show a fringing pattern spectrum. Any trapped air pockets can usually be removed by pumping the syringe alternatively sucking and blowing the liquid through the cell cavity. Once the window surfaces have been wetted the liquid will fill the cell.*

Omni Cell Used for Mulls

A mull is generally a mixture of a solid sample ground to a paste with liquid paraffin (Nujol) or Fluorolube. This paste is supported between two windows (with an optional pathlength spacer) and a transmission measurement is made. The Omni Cell mount allows for a mull window assembly to be supported in a spectrometer. The mull cell is built as a "sandwich" construction. The sample paste is placed on to the surface of one circular window and is then squashed between this window and another. Avoid overloading the mull cell as excess sample will be squeezed out and may contaminate the mount. When clamping the windows between the front and back plates a similar method of tightening as for liquid cells should be adopted.

Care of Windows

The Omni Cell's performance is only as good as the quality of the windows. To keep the windows in good and serviceable condition the Specac Polishing Kit P/N GS04000 is recommended. The kit contains all the essential materials required to clean and repolish NaCl and KBr windows to within a few fringes of flatness. Repolishing can be achieved efficiently and economically with a minimum degree of skill.

Catalogue Part Numbers

All Omni Cell windows are 4mm thick except ZnSe, Silica (IR), AgBr and Si, which are 2mm and Polythene which are 3mm thick. These thinner windows require the additional spacer P/N GS01893.

Rectangular Liquid Omni Cell Windows (Pair) (41mm x 23mm)	**Circular Mull Omni Cell Windows (Pair) (25mm diameter)**
P/N GS01810 Sodium Chloride (NaCl)	P/N GS01830 Sodium Chloride (NaCl)
P/N GS01811 Potassium Bromide (KBr)	P/N GS01831 Potassium Bromide (KBr)
P/N GS01812 Calcium Fluoride (CaF2)	P/N GS01832 Calcium Fluoride (CaF2)
P/N GS01813 Barium Fluoride (BaF2)	P/N GS01833 Barium Fluoride (BaF2)
P/N GS01814 Zinc Selenide (ZnSe)	P/N GS01834 Zinc Selenide (ZnSe)
P/N GS01815 KRS-5	P/N GS01835 KRS-5
P/N GS01816 Cesium Iodide (CsI)	P/N GS01836 Cesium Iodide (CsI)
P/N GS01817 Cesium Bromide (CsBr)	P/N GS01837 Cesium Bromide (CsBr)
P/N GS01818 Fused Silica (IR) (SiO2)	P/N GS01838 Fused Silica (IR) (SiO2)
P/N GS01819 Silver Bromide (AgBr)	P/N GS01839 Silver Bromide (AgBr)
P/N GS01820 Silicon (Si)	P/N GS01840 Silicon (Si)
P/N GS01821 Polythene	P/N GS01841 Polythene

Rectangular Liquid Omni Cell Spacers (Packet of 5)	**Circular Mull Omni Cell Spacers (Packet of 5)**
P/N GS01850 0.05mm PTFE material	P/N GS01870 0.05mm PTFE material
P/N GS01851 0.10mm PTFE material	P/N GS01871 0.10mm PTFE material
P/N GS01852 0.20mm PTFE material	P/N GS01872 0.20mm PTFE material
P/N GS01853 0.50mm PTFE material	P/N GS01873 0.50mm PTFE material
P/N GS01854 1.00mm PTFE material	P/N GS01874 1.00mm PTFE material
P/N GS01855 0.025mm Lead material	P/N GS01875 0.025mm Lead material
P/N GS01856 0.05mm Lead material	P/N GS01876 0.05mm Lead material
P/N GS01857 0.10mm Lead material	P/N GS01877 0.10mm Lead material
P/N GS01858 0.20mm Lead material	P/N GS01878 0.20mm Lead material
P/N GS01859 0.50mm PTFE material	P/N GS01879 0.50mm Lead material
P/N GS01860 1.00mm PTFE material	P/N GS01880 1.00mm Lead material
P/N GS01861 0.006mm Mylar material	P/N GS01881 0.006mm Mylar material
P/N GS01862 0.012mm Mylar material	P/N GS01882 0.012mm Mylar material
P/N GS01863 0.025mm Mylar material	P/N GS01883 0.025mm Mylar material

Spares

P/N GS01890 Rear neoprene gaskets (Packet of 2).
P/N GS01891 Quick release nuts (Packet of 4).
P/N GS01892 Luer port PTFE filling plugs (Packet of 2).
P/N GS01893 Additional packing spacer for thin windows (1).
P/N GS01110 Luer syringe at 2ml volume (1).

Permanently Sealed Omni-Cell Window Units

	Pathlengths					
Material	0.025mm	0.05mm	0.1mm	0.2mm	0.5mm	1mm
NaCl	GS01910	GS01920	GS01930	GS01940	GS01950	GS01960
KBr	GS01911	GS01921	GS01931	GS01941	GS01951	GS01961
CaF2	GS01912	GS01922	GS01932	GS01942	GS01952	GS01962
BaF2	GS01913	GS01923	GS01933	GS01943	GS01953	GS01963
ZnSe	GS01914	GS01924	GS01934	GS01944	GS01954	GS01964
KRS-5	GS01915	GS01925	GS01935	GS01945	GS01955	GS01965
CsI	GS01916	GS01926	GS01936	GS01946	GS01956	GS01966
CsBr	GS01917	GS01927	GS01937	GS01947	GS01957	GS01967
Silica (IR)	GS01918	GS01928	GS01938	GS01948	GS01958	GS01968

Silicon, AgBr and Polythene are not offered as permanently sealed Omni Cell window units.

5. Spare Parts for Liquid Pack

P/N GS01800 Omni Cell body and mount components (1 assembly).
P/N GS01811 KBr liquid cell rectangular windows - one drilled hole and one plane window as 1 pair.
P/N GS01812 CaF2 liquid cell rectangular windows - one drilled hole and one plane window as 1 pair.
P/N GS01831 KBr mull cell circular windows, two plane windows as 1 pair.
P/N GS01864 Packet of ten assorted rectangular PTFE spacers – two each of 0.05mm, 0.1mm, 0.2mm, 0.5mm and 1.00mm thickness.
P/N GS01871 Packet of five circular spacers for mull cell assemblies – 0.1mm thickness.
P/N GS01110 Luer syringe 2ml volume.
P/N GS03620 Bottle of Nujol (liquid paraffin) 25mls.
P/N GS03621 Bottle of Fluorolube (fluorinated mulling oil) 25ml.

6. Datasheet Information for KBr and CaF2 Windows used in the Liquid Pack

Potassium Bromide (KBr)

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/mm^2.
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.

Calcium Fluoride (CaF2)

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/mm^2.
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.)

Notes for Use of Liquid Pack

Notes for Use of Liquid Pack

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