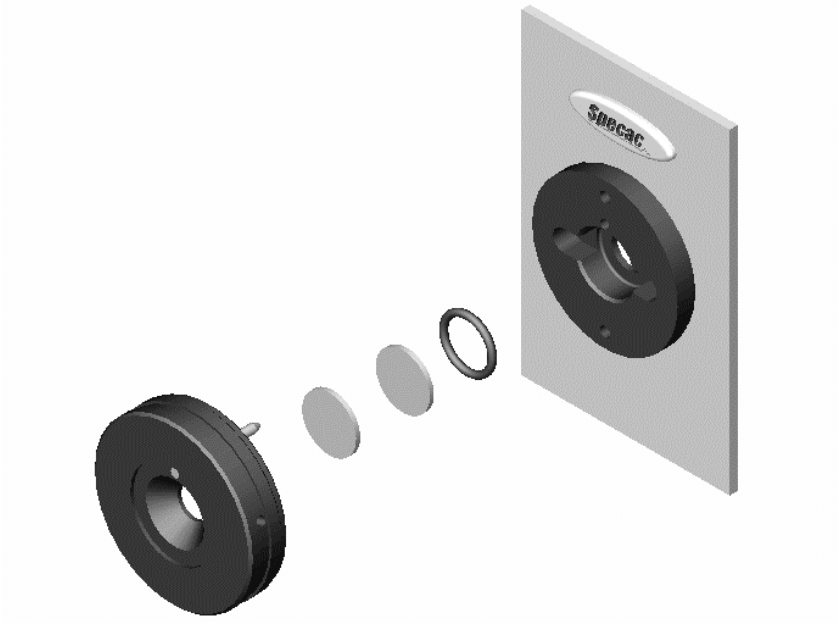
The Micro Compression Cell

User Manual



2I-02520 Issue 5

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THE MICRO COMPRESSION CELL

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1. Introduction

Thank you for purchasing a Specac Product.

The micro compression cell is a simple device to allow for the squeezing of a soft solid or semi liquid sample between two circular windows prior to analysis via transmission spectroscopy.

The windows of choice from NaCl, KBr, CaF_2, BaF_2 or ZnSe options are held within a holder that in turn mounts into the sampling compartment of any spectrometer via a standard 3" x 2" slide mount fixing. After positioning of the windows and the sample within the holder, the windows are compressed together by tightening of a front cap. The design of this front cap allows for its rotation but the windows do not rotate in relation to each other. Hence there is minimal possibility of the sample placed between the windows being disturbed from this rotational movement when tightening the cap.

The clear aperture of 7.0 mm allows for a good signal when placed at the sampling focus of an FTIR spectrophotometer using a standard DTGS detector. The overall performance of the cell is subject to the window materials that can be used within this cell. The entire cell is provided with a choice of either NaCl or KBr windows as standard as supplied against the P/N GS02520 but a list of spare windows that can be used within this cell is found at the end of this manual (see Part Numbers – Section 5).

The micro compression cell can also be used with Infrared Microscopes, where the large aperture allows for more than one sample to be loaded at one time.

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 in operation specifically to the micro compression cell, this uses 13mm diameter x 2mm thick windows of NaCl, KBr, CaF_2, BaF_2 or ZnSe material as standard. Out of these five different window material types, ZnSe can be considered as the most potentially hazardous material with respect to a toxicity risk in use and handling. All of the other window material types can be considered relatively safe to use, although all may be harmful to the body if it is ingested in significant quantity. The general rule when working with **any** window material (and sample) is to always wear gloves and safety gear (e.g. safety spectacles) when handling to obviate the risk of contact with the skin.

Crystal material safety data sheet information for each of the material types can be consulted for safe handling. A copy of each of these datasheets can be found in this instruction manual in the **Cleaning the Micro Compression Cell,** Section 6.

3. Unpacking and Checklist

On receipt of your accessory please check that the following have been supplied:

- 1 Micro compression cell assembly consisting of stainless steel 3" x 2" back plate, O-ring and front cap assembly in its own carry container labeled as P/N GS02520.

- 1 pair of windows 13mm diameter x 2mm thick of choice (NaCl;P/N GS09070 **or** KBr;P/N GS09071) as standard in their own container.
- Additional pair(s) of windows as P/N's GS09070 (NaCl), GS09071 (KBr), GS09072 (CaF_2), GS09073 (BaF_2) and GS09074 (ZnSe) in their own containers if ordered.
- User instruction manual

Carefully remove the micro compression cell accessory from its container and packaging and proceed by reading the following instructions.

Note: *NaCl and KBr windows are hygroscopic by nature so it is best to use them as soon as possible after removal from their safe containment environment for use within the micro compression cell assembly.*

4. Warning in Usage

Please read this section before using the micro compression cell

Important: *This accessory is intended for the preparation of small compressible samples. Incorrect use will break the windows. Please note the following points:*

- The cell is for use with small compressible samples. It is not suitable for crushing large samples such as whole polymer pellets. These should be pre-crushed in a die, or a small fragment should be cut off.
- The micro compression cell is a **compression** cell; it is not an **anvil** cell. It should not be used for crushing or compressing extremely hard samples. Care should be taken that the sample does not contain any hard particles (for example, some sand) that could cause failure of the windows.

Important: *The maximum load that can be applied to a sample will vary depending on the area of the sample and its hardness. This makes it difficult to provide guidelines, but the following points may be helpful:*

- Experiment first with softer samples (for example, polystyrene) to get the 'feel' of the product. Learn to judge the minimum size of the sample that will give a just large enough compressed area and optimum sample thickness.
- Samples that flow enough to cover a significant area of the window can usually be tightened sufficiently. Be aware that some window materials are softer than others and apply an appropriate hand tightness.
- Hard samples that do not compress out to a thin film need great care. Use the smallest practical quantity of sample and do not apply high loads unless the sample is seen to compress successfully.

Specac Limited cannot accept responsibility for any window breakage howsoever caused.

5. Using the Micro Compression Cell

Important: *Be sure that you have read and fully understand the Warning (Section 4) regarding use of this accessory.*

Please refer to Fig. 1 in explanation of assembly and use of the micro compression cell.

Measuring Using the Micro Compression Cell

In any sample measurement via IR spectroscopy it is usual that a reference (background) spectrum is taken before a sample spectrum can be collected that has been run under identical conditions. The only change ideally has come from introduction of a sample into the analysis cell or accessory. For the micro compression cell a reference spectrum is collected using transmitted light through **just the windows** of the cell and a sample spectrum is collected from similar transmitted light that is also passes through **a suitably compressed sample** held between the windows. A resultant IR spectrum of the sample alone is subtraction of the reference spectrum from the sample spectrum

To Collect for a Reference Spectrum

Prepare the micro compression cell assembly by unscrewing the front cap (**5**) anticlockwise away from its threaded holder of the 3” x 2” back plate (**4**).

Carefully remove the pair of windows from their container in preparation for mounting within the micro compression cell accessory.

To obtain a reference (background) spectrum, first make sure that both windows for both of their surfaces are clean. Then assemble the accessory (as shown in **Fig. 1** so that the top window (**1**) and bottom window (**2**) faces are in contact with no sample between them. The window pair rest on the O-ring (**3**) which is held inside the recess of the back plate (**4**). The window pair are to be compressed together to be held in the micro compression cell be refitting and tightening of the front cap assembly (**5**). When fitting the front cap (**5**) to tighten against

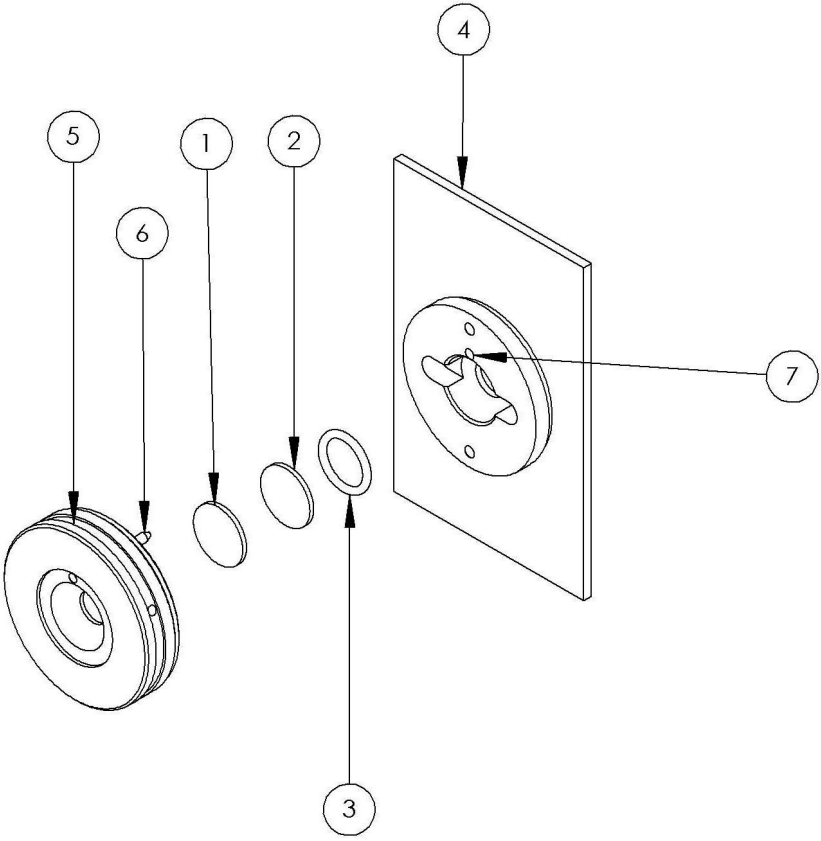

Fig. 1 – Schematic of Micro Compression Cell Components

the 3” x 2” back plate (**4**) by turning front cap (**5**) clockwise, there is a location pin (**6**) on the inside of the front cap that aligns with the hole (**7**) in the holder part of the back plate. Insertion of this pin (**6**) into the hole (**7**) restricts the rotation of the pressing face of the front cap (**5**) itself, but allows for the outer rotation of the cap against the screw threading on the holder part of the back plate. There is a small PTFE gasket permanently sealed to the non-rotational part of the front cap that contacts the top surface of the top wind**o**w (**1**) in the

tightening/compressing process. This PTFE gasket must be in place for correct sealing and tightening of the windows (**1** and **2**).

When taking a reference background do not overtighten the front cap (**5**) onto the windows. The windows need only to be lightly clamped together in the absence of a sample. Insert the micro compression cell into the spectrometer by sliding the back plate (**4**) into a standard 3" x 2" sample mount. Take care when assembling the windows into position that you use gloves to prevent contamination of the window surfaces by your fingers. Record the background spectrum.

To Collect for a Sample Spectrum

To mount a sample in the micro compression cell, disassemble the cell after obtaining the reference background. Lay the back plate (**4**) flat onto a work surface and carefully place the bottom window (**2**) onto the O-ring (**3**) within the back plate (**4**) and then carefully position or spot your sample onto the window (**2**) surface.

Take the top window (**1**) and carefully position it over the sample and bottom window (**2**). Finally, take the front cap (**5**), align the location pin (**6**) into its hole (**7**) on the holder and proceed to tighten the front cap against the back plate squeezing the windows and samples together.

Note: *Be very careful when tightening that the windows being used are not subject to any damage or risk of breakage when a sample is being used for compression.*

Place the assembled cell into the spectrometer and record the sample spectrum.

Certain regions of the sample spectrum may have a measured transmission of greater than 100%. This is because many samples with refractive indices greater than 1.2 will give lower reflection losses within the micro compression cell than when the cell is empty (air between the windows). This is normal for compression cells.

6. Cleaning the Micro Compression Cell

Cleaning of the micro compression cell components between samples is principally going to be confined to care of the windows.

Refer to the datasheet information on the next few pages for the window material type being used to understand how they should be handled and cleaned.

Always use gloves when cleaning to minimize the risk of contamination to the window surfaces and to yourself.

Solvents such as water, methanol and acetone with lens tissue material or polishing these window types against a Selvyt (velvet) polishing surface moistened with such solvent types can be used on CaF_2, BaF_2 and ZnSe windows. However, water or any solvent containing water or a considerable amount of moisture **should never be used with NaCl or KBr windows** as they will be destroyed.

After cleaning the windows can be kept within the micro compression cell for storage if required, but it may be better to replace any windows when not being used back into their container and stored in a dry environment.

Data Sheet For Sodium Chloride

Sodium Chloride (NaCl)

General
Synonyms: salt, sea salt, table salt, common salt, rock salt.
When fused together as a solid can be polished and used as a transmission window material. Slightly hygroscopic material similar to Potassium Bromide (KBr).
Soluble in water and glycerine. Slightly soluble in lower order alcohols.
Fairly good resistance to mechanical and thermal shock and can be easily polished.
Molecular formula: NaCl.
Chemical Abstracts Service (CAS) No: 7647-14-5.

Physical Data
Appearance: Odourless, white or colourless crystalline solid.
Melting point: 804°C.
Boiling point: 1413°C.
Vapour pressure: 1mm Hg at 865°C.
Specific gravity: 2.16 g cm^{-3}
Solubility in water: 35.7g/100g at 0°C.
Hardness: 6 Kg/mm^2.
Refractive Index: 1.52 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 40,000 to 600 cm-1 (wavenumbers).

Stability
Stable.
Incompatible with strong oxidising agents.

Toxicology

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

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

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

Data Sheet For Potassium Bromide

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.

Data Sheet For Calcium Fluoride

Calcium Fluoride (CaF_2)

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: CaF_2.
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.)

Data Sheet For Barium Fluoride

Barium Fluoride (BaF_2)

General
Synonyms: Barium Difluoride.
When powder is fused together, is used as a transmission window material.
Very slightly soluble in water, soluble in acids and ammonium chloride. Good resistance to fluorine and fluorides. Does not fog.
Its high mechanical strength makes it particularly useful for high pressure work.
Brittle material - very sensitive to mechanical and thermal shock.
Molecular formula: BaF_2.
Chemical Abstracts Service (CAS) No: 7787-32-8.

Physical Data
Appearance: Odourless, white or colourless crystalline solid.
Melting point: 1280°C.
Boiling point: 2137°C.
Solubility in water: 0.17g/100g at 0°C.
Hardness: 82 Kg/mm^2.
Refractive Index: 1.45 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 66,666 * to 800 cm-1 (wavenumbers).

Stability
Stable.
Incompatible with acids.

Toxicology

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

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

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

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

Data Sheet For Zinc Selenide

Zinc Selenide (ZnSe)

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/mm^2.
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 labeling.

7. Part Numbers

Micro Compression Cell

P/N GS02520 Micro Compression Cell with NaCl GS09070 or KBr GS09071 windows supplied as standard but not fitted.

Windows for the Micro Compression Cell

P/N GS09070 Pair of NaCl windows 13mm dia x 2mm thick
P/N GS09071 Pair of KBr windows 13mm dia x 2mm thick
P/N GS09072 Pair of CaF2 windows 13mm dia x 2mm thick
P/N GS09073 Pair of BaF2 windows 13mm dia x 2mm thick
P/N GS09076 Pair of ZnSe windows 13mm dia x 2mm thick

Legend (Bubble Part Numbers) of Micro Compression Cell

(**1**) Top window 13mm diameter x 2mm thick.
(**2**) Bottom window 13mm diameter x 2mm thick.
(**3**) O-ring for window assembly.
(**4**) 3" x 2" back plate.
(**5**) Front cap assembly.
(**6**) Location pin on front cap assembly.
(**7**) Hole for location pin (**6**) of front cap assembly (**5**).

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