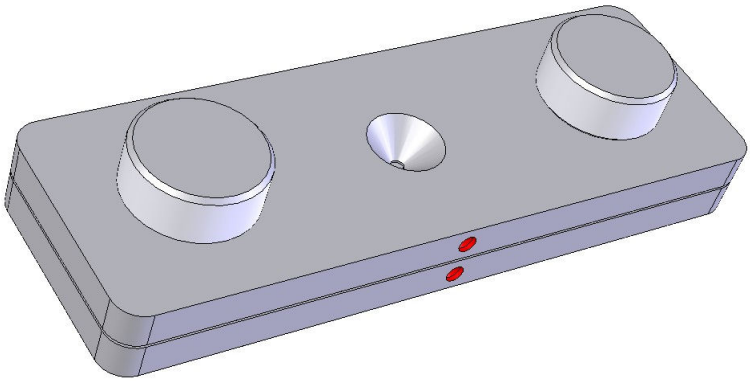
The DC-3 Diamond Compression Cell

User Manual



2I-02555 Issue 3

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USER MANUAL

1. Introduction

Thank you for purchasing a Specac Product.

The DC-3 diamond compression cell P/N GS02555 enables solid and semi-solid type samples to be compressed to an ideal thickness for transmission experiments prior to IR spectroscopic analysis. It uses two type IIIa mono-crystalline diamond windows of circa 3.5mm diameter and 0.5mm thickness, each glue mounted into an upper and lower 440C stainless steel support plate of dimensions 76.2mm long x 25.4mm wide and 4mm thickness.

The clear aperture of 1.5 mm diameter for the diamond window as contained within the 440C stainless steel support plate allows for a good transmission throughput signal when the DC-3 assembly is placed at the sampling focus of an FTIR spectrophotometer using an MCT detector. For optimum performance with a DTGS detector, the Microfocus Beam Condenser P/N GS02560 (with ZnSe lenses) or P/N GS02561 (with KRS-5 lenses)) is recommended to be used with the DC-3 assembly in order to obtain high quality spectra. The entire DC-3 and microfocus beam condenser assembly of parts are mounted within the spectrometer sample compartment via use of an appropriate Specac Benchmark™ type baseplate.

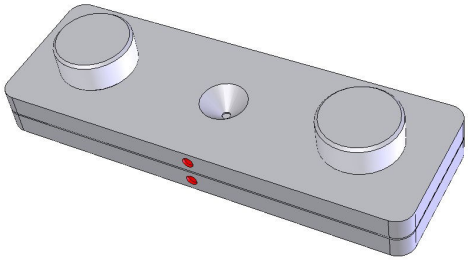
As a kit of parts the DC-3 and **ZnSe** microfocus beam condenser are available as P/N GS02556 and the DC-3 and **KRS-5** microfocus beam condenser are available as P/N GS02557.

The DC-3 is for use with small compressible samples. It is not suitable for crushing large samples such as whole polymer pellets. If these sample types are to be analysed they should be pre-crushed in a die, or a small fragment should be cut off prior to compression between the DC-3 diamond windows. The DC-3 should also 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 or grit etc,) that could cause failure of the thin diamond windows through a “point” loading effect.

The DC-3, because of its small 440C stainless steel support plate size and rectangular shape, can also be used with Infrared (IR) Microscopes. (It is similar in size to many commercially available microscope glass slide mounts). The upper and lower 440C stainless steel plate assemblies have a wide aperture opening of 8.3mm diameter that are cone shaped and taper to the 1.5mm diameter aperture for the diamond window. As such this design allows for the objective lens of an IR microscope with a wide range of incident light angles to be introduced for an optimum focal point and magnification of a sample compressed between the two diamond window surfaces, when the DC-3 has been placed on the microscope stage.

To help achieve a consistent mounting of a sample whilst under compression between the two diamond windows of the DC-3, a compressible O-ring is used between the two fixing screws that pull the 440C stainless steel plate assemblies together. The slight initial separation of the plates when placed together that is provided by the O-rings allows for the plates to be pulled together for an even and level fit of their surfaces with respect to each other when tightening the fixings screws and in turn compresses any sample placed between the diamond windows for a similar even and level contact.

The large aperture of 1.5mm diameter allows for more than one sample to be loaded and compressed at one time if the sample type (and amount of sample available) allows for this to be done. Each individual sample could then be selectively moved into the light beam when under analysis using an IR microscope, saving on the time needed to mount and prepare a new sample between analyses.

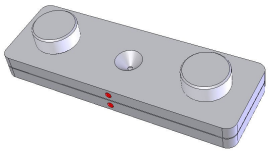


DC-3 Diamond Compression Cell Assembly

2. Unpacking and Checklist

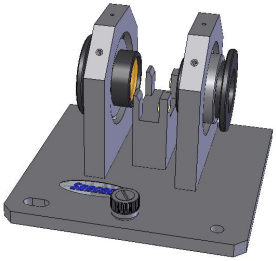
On receipt of your accessory please check that the following have been supplied:

- 1 DC-3 assembly consisting of upper and lower 440C stainless steel support plates, each with a type IIIa diamond window glued into the plate recess, when ordered as GS02555.



If your DC-3 accessory was ordered as part of a kit (GS02556 or GS02557) it will also include:

- 1 Microfocus Beam Condenser (GS02560 – ZnSe lenses or GS02561 – KRS-5 lenses) and its user instruction manual.



- 1 Benchmark™ baseplate for spectrometer to be used.
- 1 Benchmark™ baseplate installation guide instruction manual.
- 1 Tool kit consisting of:
  - Sample forceps.
  - Stainless steel sample needle.
  - Knife, blades (10) and blade remover (2).

Carefully remove the DC-3 diamond compression cell accessory and microfocus beam condenser (if ordered as a kit) from their container(s) and packaging and proceed for use of the items by following the information from the user instruction manual(s).

Please take note of the serial number identification of the DC-3 accessory to include for reference, if ever needing to contact Specac.

3. Warnings for Safe Usage

Please read this section before using the DC-3

Important: *The DC-3 accessory is intended for the preparation of small compressible samples. Incorrect use will break the diamond windows. Please note the following points:*

- The DC-3 diamond compression 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 DC-3 is a **compression cell**; it is not a diamond 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 or grit) that could cause failure and damage to the thin (0.5mm) diamond 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 to full finger tightness.
- Hard samples that do not compress easily to form 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.

Chemical Samples

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 DC-3 diamond compression cell accessory, this uses a diamond (type IIIA) window in each of the upper and lower support plates of the complete assembly where a sample makes contact. However, if the DC-3 is to be used with its compatible microfocus beam condenser accessory, there is a choice of either ZnSe or KRS-5 material for the beam condensing lens assemblies used in this accessory.

Out of these three different crystal material types, KRS-5 and then ZnSe are the most potentially hazardous materials with respect to a toxicity risk in use and handling. Diamond can be considered relatively safe to use, although the general rule when working with **any** crystal material (and sample) is to always wear gloves and safety gear (e.g. safety spectacles) when handling to obviate the risk of contact with the skin. Specifically in operation of the DC-3 diamond compression cell accessory there is a minimal risk associated with the diamond material itself for actual sampling contact. Similarly, there is minimal risk of contact with the KRS-5 or ZnSe materials used for the lens assemblies within the microfocus beam condenser (if this is to be used) as these crystal materials are safely contained within the beam condenser optical unit lens housings and should not require being handled after an initial alignment and optimum throughput has been obtained at installation.

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 **Notes on Cleaning** Section found on pages 14 to 18.

4. Using the DC-3 Accessory

Important! *Be sure that you have read and fully understand the Warnings for Safe Usage information (Section 3) prior to use of the DC-3 diamond compression cell accessory.*

The DC-3 Diamond Compression Cell Assembly

The DC-3 diamond compression cell consists of an upper (**1**) and lower (**2**) 440C stainless steel rectangular support plate that each contain a type IIIa mono-crystalline diamond window (**3**) with an aperture of 1.5mm diameter that has been glue fixed into the centre recess on one surface of the support plate. **Fig 1.** shows the top and underside surface views of the **upper** (**1**) support plate assembly and **Fig 2.** shows the top and underside surface views of the **lower** (**2**) support plate assembly.

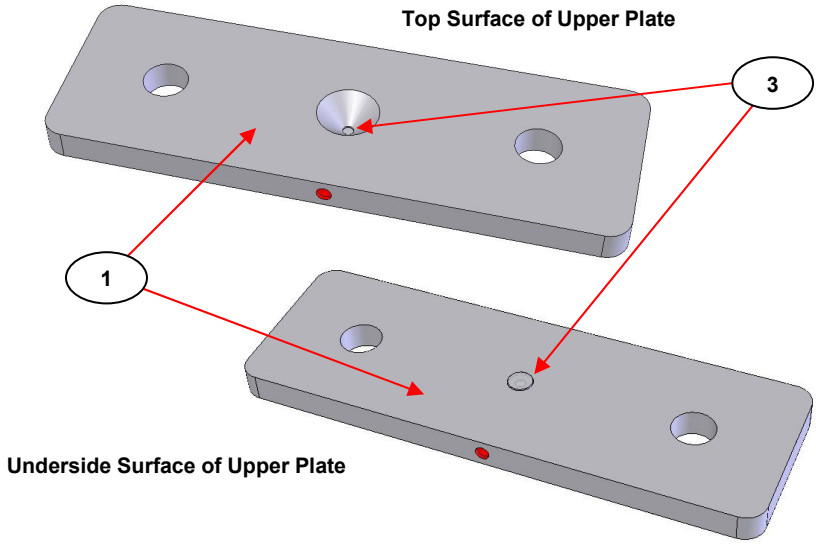


Fig 1. Upper Support Plate Assembly of DC-3

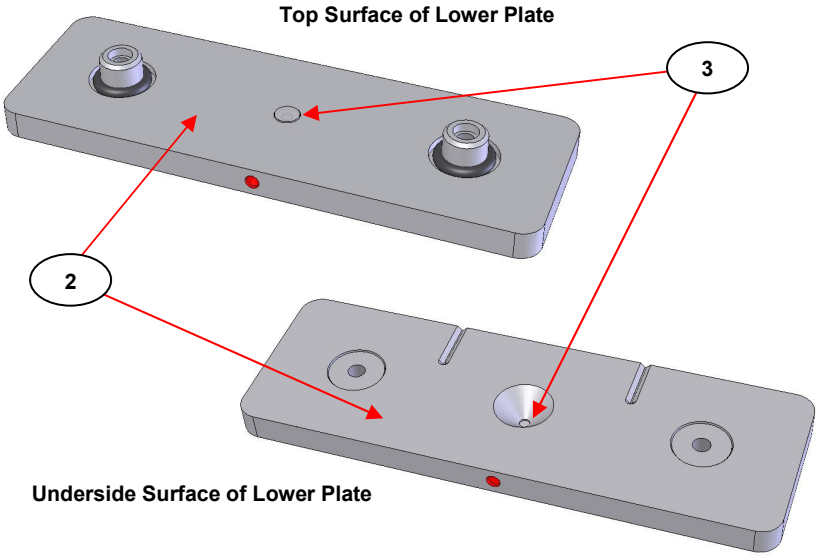


Fig 2. Lower Support Plate Assembly of DC-3

Note: *There are red alignment spots on one edge of each of the upper **(1)** and lower (**2**) support plates of the DC-3 accessory. Whenever the DC-3 is constructed to bring the diamond window (**3**) surfaces together, the plates should always be assembled so that the red spots are together on the same side of the accessory (See **Fig 3.**)*

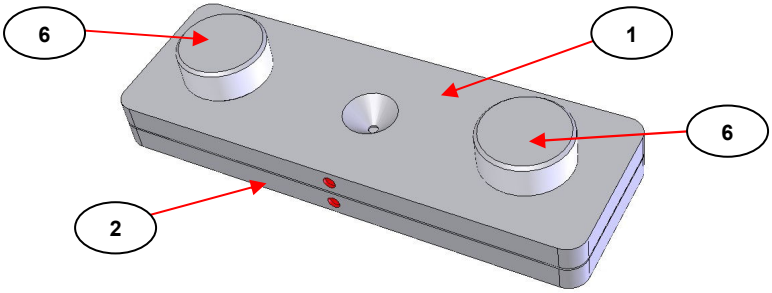


Fig 3. Correct DC-3 Assembly (Upper Plate Top View)

Mounting and Compressing a Sample

In construction of the DC-3 to compress a sample the lower (**2**) support plate is placed onto a level surface with its top surface (see from **Fig 2.**) uppermost. An appropriate amount of sample is taken to cover the 3.5mm diameter diamond window (**3**). Use of the scalpel blades and forceps provided in a DC-3 Kit can help in the manipulation of the sample onto the lower diamond window (**3**) surface. (See **Fig 4.**)

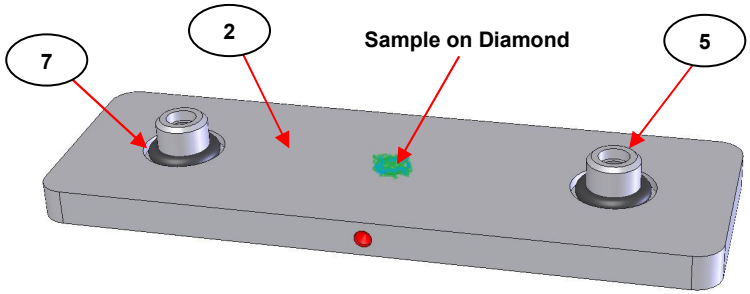


Fig 4. Sample Placed Over Diamond Window on Lower Support Plate Assembly

When the sample has been correctly positioned take the upper (**1**) support plate and carefully place it over the lower (**2**) support plate assembly. The diamond window (**3**) from the underside surface of the upper (**1**) support plate (see from **Fig 1.**) is to be brought into contact with the sample placed over the diamond window (**3**) on the lower (**2**) support plate. The fixing thumb screw holes (**4**) of the upper (**1**) support plate are aligned over the fixing thumb screw support posts (**5**) of the lower (**2**) support plate. When aligning the parts ensure that the red dot indicators on the edge of both support plates (**1**) and (**2**) will be on the same side when the support plates are tightened together.

Take the two fixing thumb screws (**6**) and carefully introduce each one into their support post (**5**) screw threading. Finger tighten them in a clockwise rotation until the flat underside of both thumb screw (**6**) heads begin to make contact with the top surface of the upper (**1**) support plate. The upper (**1**) and lower (**2**) support plates are then

tightened together using the two fixing thumb screws (**6**) by simultaneous rotation of each thumb screw (**6**) in a clockwise direction to bring the support plates (**1**) and (**2**) together for an even and level fit for their contact surfaces all the way around the edges of the support plates. The components are tightened further together to maintain an even and level fit until it is no longer possible to turn the thumb screws (**6**) by hand. (See **Fig 5.**)

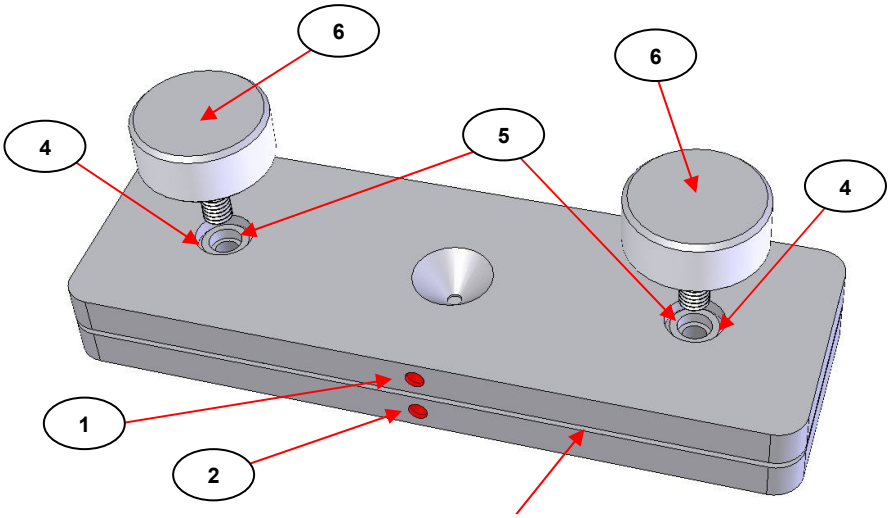


Fig 5. Placement of Upper Support Plate over Lower Support Plate with Sample on Lower Diamond Window and Tightening Together

There is a Nitrile (N70) O-ring (**7**) that is placed in a recess around the base of each of the thumb screw support posts (**5**) on the lower (**2**) support plate. (See **Fig 4.**)

When fitting the upper (**1**) support plate over the lower (**2**) support plate to compress a sample between the two diamond windows (**3**), depending upon the initial amount (and thickness) of sample that has been placed on the diamond window to compress, the upper (**1**) support plate will eventually come to rest on the two O-rings (**7**) for an

even and level fit of the two support plate surfaces with respect to each other. When this stage has been reached for the act of overall compression and tightening together of the DC-3 components, similarly the two diamond window surfaces (**3**) in contact with any sample that is "sandwiched" between them will also be level to each other.

Note: *With **no sample** in position between the diamond windows (**3**), when the upper (**1**) support plate is resting on the two O-rings (**7**) of the lower (**2**) support plate, there will be a nominal gap of circa 200 microns between the diamond window (**3**) surfaces prior to **any compression** of the components in tightening of the two support plates together from simultaneous clockwise rotation of the two thumb screws (**6**).*

The O-rings (**7**) alone are compressible to the extent that **without a sample in position** the two diamond windows (**3**) can be brought together for even and parallel contact of their surfaces by hand tightening of the thumbs screws (**6**). This condition is necessary to construct the DC-3 accessory accordingly with no sample, to obtain a reference "background" spectral measurement of the DC-3 accessory, prior to reconstruction using a compressible sample between the diamond windows for spectral measurement.

Warning! ***Always** tighten the support plates (**1**) and (**2**) together from hand tightening alone of the thumb screws (**6**). NEVER use any further gripping devices to try and exert a higher torque force for the tightening of the thumb screws (**6**) as you risk causing permanent damage or breakage to the diamond windows (**3**).*

It is important to try and obtain an even and level fit for contact of the diamond windows (**3**) surfaces to any sample that is being held and compressed between them. This not only helps to reduce the risk of any damage that might occur to the diamond windows (**3**) if they have become canted over, but also maintains reproducibility and reliability of spectral measurement between samples, particularly when comparing a sample measurement against a reference background that ideally has been obtained using the DC-3 accessory with the diamond windows even and parallel when in contact to each other.

Collecting a Reference Background Spectrum

Before any sample can be measured spectroscopically after compression within the DC-3 accessory, a background reference spectrum should be created first.

To obtain a background spectrum, first make sure that the diamond window (**3**) surfaces that will come into contact with a sample are clean. (See Notes On Cleaning – page 14). Assemble the DC-3 accessory as described on pages 9 and 10, **but without** any sample in position on the lower (**2**) support plate diamond (**3**) window. When the components have been tightened together correctly, the DC-3 accessory is ready to be installed into the IR spectrometer or microscope system.

The DC-3 accessory can be used on its own when acting as a sample support mount for transmission spectroscopic studies using an IR (or visible) microscope system, but for use of the DC-3 within an IR spectrometer sample compartment it is usual to mount the DC-3 accessory in the microfocus beam condenser accessory unit, P/N GS02560 (ZnSe lenses) or GS02561 (KRS-5 lenses). For mounting of the DC-3 into the microfocus beam condenser please refer to the user instruction manual 2I-02560-6 supplied with this accessory

Collecting a Sample Spectrum

After collecting a reference background spectrum with the DC-3 alone or used in conjunction with the microfocus beam condenser accessory, a sample spectrum can be obtained.

Dis-assemble the DC-3 accessory and proceed to mount it for reconstruction with a suitable sample following the procedure described on pages 9 and 10.

Note: *Certain regions of the sample spectrum may have a measured transmission of greater than 100%. This is because many samples with refractive index's greater than 1.2 will give lower* ***reflection losses*** *within the DC-3 accessory compared to when no sample (air) is in place for a reference background measurement. This is normal for diamond compression cells.*

Notes on Cleaning

When cleaning the diamond windows (**3**) of the DC-3 diamond compression cell in preparation for a new sample, it is **very important to take care** to avoid damage to the crystal material**.** As mentioned in the Warnings For Safe Usage information (Section 3, page 6), diamond crystal material that is used for sample contact is not particularly hazardous in terms of a risk of toxicity if it comes into contact with the skin. There is more risk associated with the ZnSe and most especially the KRS-5 lens materials in the microfocus beam condenser accessory, should there be a need to clean these items.

Note: ***Always*** *wear gloves to protect yourself and the crystal material from cleaning and when sample handling.*

From the design and size of the DC-3 accessory, a useful feature is the capability for easy installation and removal from a microscope system or the microfocus beam condenser accessory if being used, such that any sample can be prepared remotely and safely within the DC-3 accessory and then can be bought back for fitting into the microscope or microfocus beam condenser whilst installed in the spectrometer. Similarly for cleaning, it is necessary to remove the DC-3 accessory from the working area and carry it to a safe area for solvent cleaning and wiping with a tissue and therefore minimise any risk of contamination being carried over to other equipment or components of the accessory whilst in situ.

Solvents such as water, methanol and acetone are suitable to use for cleaning purposes. Some sample stains or residues if present on or around the diamond window (**3**) area may require removal using a strong acid or base solution, but if these types of cleaning solvents are employed, do so using sparingly. Quickly clean away any excess solvent using a cloth or tissue and follow by rinsing away with further water to dilute any effect of an acid or base that may remain.

When wiping away any solid or liquid sample, it is advisable to use soft lens tissues to avoid scratches being caused on the surface of the diamond window material. Ordinarily, diamond material being extremely resilient will be unaffected from this treatment, but any

scratches and blemishes if introduced to the window surface will result in poor light throughput for the technique and an overall degradation in the accessories performance.

In common and general usage it will only be necessary to wipe and clean away at the diamond window (**3**) surface that comes into contact with a sample for compression. If possible try to avoid any solvent or cleaning solution materials from getting to the conical aperture surface sides of the upper (**1**) and lower (**2**) support plates. There is a risk that any dried solution residues that have been introduced in this way to this surface of the diamond window (**3**) could be seen 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.

Data Sheet For Diamond

General

Hardest substance known for carbon (C) elemental form.
Can be shaped, cut and polished to form spectral transmission window or crystal for ATR spectroscopy.
Has a highish Refractive Index value and can suffer reflection losses but these can be improved with antireflection optical coatings
Extremely chemically resistant to practically all known materials.
Element symbol: C
Chemical Abstracts Service (CAS) No: 7440-44-0 (Synthetic) : 7782-42-5 (Natural)

Physical Data

Appearance: Clear, transparent and generally colourless solid. Has no odour.
Melting point: N/A.
Boiling point: 4827°C.
Vapour pressure: N/A.
Specific gravity: 2.26 g cm^{-3}.
Solubility in water: Insoluble
Hardness: 5700 Kg/mm^2.
Refractive Index: 2.43 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 40,000 to 10 cm-1 (wavenumbers).

Stability

Stable.

Toxicology

Not classified as a dangerous or harmful material according to EC directives.

Personal Protection

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

Storage

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

Data Sheet For Zinc Selenide

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.

Data Sheet For KRS-5

General

Synonyms: Mixture of Thallium Bromide and Thallium Iodide (typically 58% Iodide content). Very toxic red coloured soft 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. Slightly soluble in water, soluble in bases, but not soluble in acids. Not hygroscopic. Organic solvents have no effect.
Soft window material and easily deformed.
Molecular formula: $TlBr_{0.4}I_{0.6}$

Physical Data

Appearance: Red, soft crystals, granular powder or red coloured window material
Melting point: 414°C
Solubility in water: 36g/100g at 0°C.
Hardness: 40 Kg/mm^2.
Refractive Index: 2.38 (at 2000cm-1 - wavenumbers).
Spectroscopic transmission range: 17,000 to 250 cm-1 (wavenumbers).

Stability

Stable.

Toxicology

Very toxic if small amounts are inhaled or swallowed. May be fatal if swallowed. May be absorbed through the skin. Irritant.

Personal Protection

Always wear safety spectacles and gloves when handling the powder or window material.
Allow for good ventilation. If material is machined, polished or ground, precautions must be taken against inhalation of dust.

Storage

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

5. Spare Part Numbers

GS02555 DC-3 Diamond Compression Cell

GS02556 DC-3 Diamond Compression Cell Kit – includes:
GS02555 DC-3 Diamond Compression Cell
GS02560 Microfocus Beam Condenser with ZnSe lenses, Benchmark™ baseplate, forceps, stainless steel sample needle, sample preparation knife, blades (10) and blade remover (2).

GS02557 DC-3 Diamond Compression Cell Kit – includes:
GS02555 DC-3 Diamond Compression Cell
GS02561 Microfocus Beam Condenser with KRS-5 lenses, Benchmark™ baseplate, forceps, stainless steel sample needle, sample preparation knife, blades (10) and blade remover (2).

GS02560 Microfocus beam condenser with ZnSe lenses.
GS02561 Microfocus beam condenser with KRS-5 lenses.
GS02570 Microfocus beam condenser KRS-5 lens kit.
GS02571 Microfocus beam condenser ZnSe lens kit.

6. Legend – Bubble Part Number Identification

(**1**) Upper 440C stainless steel support plate - (76.2mm x 25.4mm x 4.0mm).
(**2**) Lower 440C stainless steel support plate (76.2mm x 25.4mm x 4.0mm).
(**3**) Diamond (type IIIA) window (3.5mm dia. x 0.5mm thick).
(**4**) Thumb screw fixing hole of upper support plate.
(**5**) Thumb screw threaded locating post on lower support plate.
(**6**) Fixing thumb screw.
(**7**) Nitrile (N70) O-ring for threaded locating post.

Notes for Usage

Worldwide Distribution

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Fax 01 42 08 13 65
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Website: www.LOT-Oriel.com/de

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