MARKES international

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HiSorb™ probes

Instruction for use

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NOTES Short probes are typically used for headspace sampling and long probes are used for immersive sampling*
*Using 20 mL vials

2. Introduction

HiSorb probes are designed to be used directly on the Centri automated sample extraction and enrichment platform, or in conjunction with industry-standard, stainless steel 1/4” sorbent tubes on an alternative thermal desorption system, such as TD100-xr.

When not in use, the probes should be stored either inside empty tubes and sealed with brass caps, or inside the protective shipping packaging provided, in order to protect them from environmental contaminants.

CAUTION Minimise manual handling of the HiSorb probe. To avoid contamination, transfer should be achieved using the HiSorb probe extraction tool (part no. C-HSPH). On Centri, ensure the HiSorb handles (C-HH-6) are securely attached for handling of the probes.

3. Conditioning

HiSorb probes should be conditioned in a flow of inert carrier gas prior to use. This can be achieved using a dedicated probe conditioning mode on Centri, tube conditioning mode on a thermal desorber, or using an off-line tube conditioning unit such as Markes' TC-20.

NOTES If probes are to be used on Centri and require conditioning using the TC-20, probe handles must be removed prior to placing inside empty TD tubes for the conditioning process.

3.1 Initial reconditioning

Prior to sampling it is advised to perform the following conditioning procedure, and test the probes for cleanliness by acquiring blank chromatograms.

Each of the HiSorb probes contains a poly(dimethylsiloxane) (PDMS) coating which is sensitive to oxygen when heated. Purge the HiSorb probes for 10 minutes at low temperature (30–50°C) in a flow of inert carrier gas before conditioning.

- Time: 1–2 hours
- Flow: 50–100 mL/min, inert carrier gas
- Temperature: 250–300°C

CAUTION Do not exceed a temperature of 300°C for conditioning or analysis.

3.2 Subsequent reconditioning

Depending on the application and sample matrix, HiSorb probes may require a short period of conditioning between uses.

The poly(dimethylsiloxane) (PDMS) coating on the HiSorb probes is sensitive to oxygen when heated. Purge the HiSorb probes for 10 minutes at low temperature (30–50°C) in a flow of inert carrier gas before conditioning.

- Time: 15–30 minutes
- Flow: 50–100 mL/min, inert carrier gas
- Temperature: We recommend that this is 5–10°C above the desired desorption temperature. The maximum recommended desorption temperature is 280°C.

CAUTION Do not exceed a temperature of 300°C for conditioning or analysis.

4. Cleaning

Thermal conditioning, as described above, is the recommended method for cleaning HiSorb probes. However, if matrix is persistent, then the probes can be soaked in a solvent. Recommended solvents and maximum exposure times are listed below.

- 50% Methanol–50% dichloromethane (up to 5 hours)
- 100% Acetonitrile (up to 12 hours)
- 80% Acetonitrile–20% methanol (up to 12 hours)

NOTES Immerse the HiSorb probes in solvent for the minimum time required.

NOTES It is not recommended to use other solvents (particularly non-polar solvents) as this may cause the PDMS to swell or become damaged.

5. Sampling

5.1 Sample limitations

HiSorb can be used for both headspace and immersive sampling. For immersive sampling, samples should be aqueous, with

- Up to 10% ethanol
- Up to 3% fat
- Up to 30% salt
- Up to 10% organic modifier (typically methanol, ethanol, propanol or acetonitrile)

5.2 Automated sampling procedure

[1] HiSorb handles attached to HiSorb probes. HiSorb probes inserted into the storage racks. *(Figure 1)*

[2] Set up HiSorb method in software. This allows all parameters for sampling, washing, drying and desorption of the probes (see Analysis section for further information on probe desorption parameters and considerations). *(Figure 2)*

[3] Place sample vials in the sample racks ready for automated sampling. Agitation of the sample vials is recommended to accelerate equilibrium between the sample and sorptive phase.

Figure 1: Centri top deck: HiSorb storage racks (front left), Centri inlet (front right), Centri agitator agitator (back right), Centri wash station (back left)

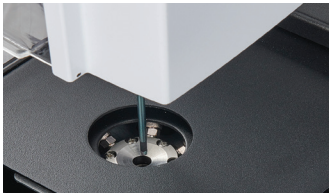


Figure 2: HiSorb probe in wash and dry station.

5.3 Manual sampling procedure (for use with Centri please refer to Centri operators manual)

[1] Insert the HiSorb probes into the vial through the HiSorb cap/septum (C-HSPCCS).

[2] To accelerate equilibration between the sample and the sorptive phase the sample vial should be agitated or magnetically stirred.

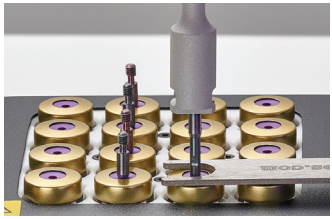
- Agitation can be achieved using Markes International's HiSorb Agitator (U-HSAG-20) or similar instrument.
- Magnetic stirring should be achieved by placing a glass (not PTFE) magnetic follower into the vial, before sealing the vial and inserting the HiSorb probe.

[3] Once sampling is complete, remove the HiSorb probes from the sample vial using the probe extraction tool. This avoids the need to handle the probe directly.

[4] Rinse the probe with HPLC-grade water to remove any residue from the surface.

[5] Dry the probe with a lint-free tissue or a flow of clean air.

HiSorb probes being used for headspace sampling (right) and immersive sampling (left).



Step [3]: Removal of the probe.

Step [4]: Rinsing the probe.

Step [5]: Drying the probe.

5.4 Sampling tips

[1] For immersive sampling, minimise the headspace in the sample vial for the most efficient extraction of volatile compounds.

[2] For headspace sampling, fill the vial to a level that prevents direct contact between the probe and sample

[3] Optimise the sampling time for the compounds of interest to achieve the most efficient extraction. Typical sampling times are 30–120 minutes, but can be much longer for complex matrices.

[4] Sorptive extraction works on the principle of absorption, which is an equilibrium process. The maximum achievable extraction efficiency for a particular analyte therefore depends upon its particular characteristics, and it may not be possible to achieve 100% recovery in all cases.

6. Analysis

6.1 Centri sample extraction and enrichment platform

[1] Ensure that the 'pre-purge' step is set for at least 1 minute to remove any residual oxygen or moisture and prevent damage to the sorptive phase. Trap should be 'in-line' during this step to prevent loss of any analytes released from the sorptive phase at ambient temperature.

[2] Probe desorption *(figure 3)*; exact parameters will depend on the sample and should be optimised during method development. Typical parameters to try are:

- Desorption time: 5–15 minutes
- Trap flow rate: 30–50 mL/min
- Desorption temperature: Typically 200–250°C, but varying depending on the application. The maximum recommended desorption temperature is 280°C.
- For the PDMS phase, some siloxane bleed is to be expected from the HiSorb probes.
- Some bleed is to be expected from the DVB (divinylbenzene) phase. These are aromatic isomers with varying degrees of saturation across the vinyl groups.

[3] After desorption the probe is automatically returned to its original position in the HiSorb storage station. *(figure 4)*

Figure 3: HiSorb probe leak checked, purged and desorbed within the Centri inlet.

Figure 4: HiSorb probe returned to the HiSorb storage station after desorption.

6.2 Thermal desorption of HiSorb using a tube based thermal desorber such as, a TD100-xr

[1] Ensure that the HiSorb probe is clean and dry.

[2] Insert the probe into the TD tube. The sorptive phase section of the probe should be positioned at the sampling/desorption end of the tube.

Step [2]: Insertion of the probe into TD tube.

[3] Ensure that the 'pre-purge' step in the TD method is set for at least 1 minute to remove any residual oxygen or moisture and prevent damage to the sorptive phase. Trap should be 'in-line' during this step to prevent loss of any analytes released from the sorptive phase at ambient temperature.

[4] Thermally desorb the probe. As above on Centri, exact parameters will depend on the sample and should be optimised during method development – see further information above for typical parameters.

CAUTION Do not exceed a temperature of 300°C for conditioning or 280°C for analysis.

NOTES During method development, it is best practice to assess the desorption efficiency. This can be done by desorbing the probes a second time. If >1% of compounds of interest are still present, try increasing the desorption time, flow rate or temperature.

7. Contact details

For technical support, please contact your supplier in the first instance. If they are unable to resolve your query, please contact Markes International's service department:

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T: +44 (0)1443 230935
W: www.markes.com

For an instructional product video, please visit:
chem.markes.com/HiSorbExtraction

Scan the code to watch the video