MARKES international

QUI-1175
Version 1
March 2020

TF-SPME

Instruction for use

Product Reference:

TF-SPME Starter Kit, PDMS	C-TF-P-KIT
TF-SPME Starter Kit, PDMS/CAR	C-TF-PCAR-KIT
TF-SPME Starter Kit, PDMS/DVB	C-TF-PDVB-KIT
TF-SPME Starter Kit, PDMS/HLB	C-TF-PHLB-KIT

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1. Introduction

TF-SPME membranes are designed to be used in conjunction with industry-standard-sized stainless steel 3½″ × ¼″ sorbent tubes for analysis using thermal desorption (TD)–gas chromatography (GC).

CAUTION Do not manually handle the TF-SPME membrane. To avoid contamination, the membranes must be handled with clean handling tools and contact with contaminated surfaces must be avoided.

2. Components

Each TF-SPME starter kit consists of:

- 2 × TF-SPME membranes, 4 cm, supplied in 4 mL vials
- 2 × 20 mL clear screw cap vials with caps and septa
- 2 × cotter pins

- 2 × glass-encapsulated magnetic stir bars
- 2 × industry-standard-sized desorption tubes
- Instruction for use

The following items will also be required, but are not supplied with the TF-SPME starter kit:

- Tweezers
- A method for agitation or magnetic stirring

3. Conditioning

TF-SPME membranes should be conditioned in a flow of inert carrier gas (He or N_2) prior to first use. This can be achieved using a dedicated tube-conditioning mode on a thermal desorber or using an off-line conditioning unit such as Markes' TC-20™, using empty stainless steel sorbent tubes.

NOTES Maximum desorption temperatures differ according to the sorbent phase impregnated on the TF-SPME membrane: PDMS/DVB and PDMS/HLB sorbents have a recommended maximum desorption temperature of 270°C, whilst PDMS/CAR has a recommended maximum desorption temperature of up to 300°C.

3.1 Initial conditioning

Prior to first use, TF-SPME membranes should be conditioned using the following short conditioning procedure:

- Time: 30–60 minutes
- Flow: 50–100 mL/min, inert carrier gas
- Temperature: 250°C

3.2 Subsequent reconditioning

Depending on the application and sample matrix, TF-SPME membranes may require a short period of conditioning between uses. The following procedure is recommended:

- Time: 15–30 minutes
- Flow: 50–100 mL/min, inert carrier gas
- Temperature: The highest desorption temperature the membrane has been exposed to.

CAUTION Do not exceed the maximum desorption temperature for conditioning or analysis.

4. Sampling

TF-SPME membranes, of 4 cm length, can be used for both headspace and immersive sample extractions. Typically, a 20 mL headspace glass vial closed with a septum cap is used.

TF-SPME sampling conditions can vary significantly from one analysis to another, with parameters such as extraction time, temperature and agitation speed all varying depending on analysis.

Figure 1: TF-SPME with PDMS/DVB, PDMS/CAR, PDMS/HLB and sorbent tube (left to right). PDMS not shown.

4.1 Sampling procedure

[1] Remove the TF-SPME membrane from the storage vial using appropriate tweezers.

[2] Unscrew the sample vial cap and pierce the vial cap septum from the outer cap with a cotter pin.

[3] Transfer the membrane to the cotter pin, taking into account positioning for headspace or immersive sampling.

Step [2] Piercing the septum.

Step [3] Transferring membrane to cotter pin.

[4] Seal the membrane in the vial.

Typical TF-SPME vial set up for sample extraction.

[5] Adjust the membrane insertion depth by moving the cotter pin accordingly.

[6] To accelerate equilibration of the analytes between the sample and the sorbent phase, the sample vial should be magnetically stirred or agitated.

- Magnetic stirring can be achieved by placing a magnetic stir bar into the vial before inserting the TF-SPME membrane and sealing the vial.
- Agitation can be achieved using Markes International's HiSorb™ Agitator (U-HSAG-20) or a similar instrument.

NOTES Tests on TF-SPME membranes have shown them to withstand agitation speeds of up to 2000 rpm.

[7] Once sampling is complete, remove the TF-SPME membrane from the sample vial using tweezers. This avoids the need to handle the membrane directly.

[8] In the case of immersive extractions, blot dry the membrane using a dry lint-free tissue before desorption; for difficult matrices, rinse the membrane with deionised water after sample extraction to remove any residue from the surface, followed by blot drying in order to reduce matrix contamination.

Step [8]: Drying the TF-SPME membrane.

4.2 Sampling tips

- For immersive sampling, minimise the headspace in the sample vial for the most efficient extraction of volatile compounds.
- For headspace sampling, fill the vial to a level that prevents direct contact between the membrane and the sample.
- Optimise the sampling time to achieve the most efficient extraction for compounds of interest.
- 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.

5. Analysis

5.1 Thermal desorption

[1] Ensure the TF-SPME membrane is free of residue and dry.

[2] Insert the membrane into the TD tube using tweezers, ensuring the membrane is touching the gauze to guarantee a uniform heating of the membrane during desorption.

[3] Ensure that the 'pre-purge' step in the TD method is set for at least 1 minute to remove all oxygen and prevent damage to the membrane sorbent. The trap should be 'in-line' during this step to prevent loss of any analytes released from the sorbent at ambient temperature.

[4] Thermally desorb the membrane.

Exact parameters will depend on the sample and should be optimised during method development.

Typical parameters are:

- Desorption time: 5–10 minutes
- Flow: 50–80 mL/min
- Temperature: Typically 225–270°C for PDMS/DVB and PDMS/HLB. 250–300°C for PDMS/CAR.

NOTES As for PDMS-based capillary column phases, some siloxane bleed is to be expected from the TF-SPME membrane.

NOTES When used in a clean matrix and properly cared for, TF-SPME membranes have been proven to last for over 100 analytical extractions.

6. Storage

New TF-SPME membranes are best stored within their shipment vials for long-term storage.

After first desorption, membranes can be kept within shipment vials or sorbent tubes sealed with brass caps for long-term storage.

Alternatively, membranes can be stored in sorbent tubes sealed with DiffLok™ caps for up to 24 h.

7. Specifications

Part number	Description	Dimensions
C-TF-P-1	TF-SPME membrane, PDMS (polydimethylsiloxane), pk 1	40 × 4.85 × 0.04 mm
C-TF-PDVB-1	TF-SPME membrane, PDMS/DVB (polydimethylsiloxane/divinylbenzene), pk 1	40 × 4.85 × 0.04 mm
C-TF-PCAR-1	TF-SPME membrane, PDMS/CAR (polydimethylsiloxane/carboxen), pk 1	40 × 4.85 × 0.04 mm
C-TF-PHLB-1	TF-SPME membrane, PDMS/HLB (polydimethylsiloxane/hydrophilic lipophilic balanced), pk 1	40 × 4.85 × 0.04 mm

Compatible tube types:

- Any industry-standard-sized 3½″ × ¼″ sorbent tube

This product is manufactured and sold under the following patents:

- United States, Patents 6,588,255 and 6,941,825
- Canada, Patent No. 2,389,726
- Japan, Patent No. JP2003-521711

8. 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:

E: support@markes.com

T: +44 (0)1443 230935

W: www.markes.com

L-0175 (110320-1)