Full Evaluation of the Automated Cold Fiber Device Using Compounds with a Large Range of Volatility

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Introduction

The aim of this project is to evaluate a solid phase microextraction (SPME) method using a cooled fiber extraction phase to achieve higher sensitivity for compounds with a large range of volatility and polarity when compared to the SPME commercial fibers.

In order to effectively exploit the headspace SPME, high temperature usually need to be used to accelerate the mass transfer. Thus releasing more analytes from the sample matrix to the headspace. However, since the sorption is an exothermic process, an increase in temperature decreases the distribution coefficients of analytes between the coating and the sample matrix resulting in lower extracted amount under equilibrium. Therefore, cold fiber SPME was developed to address this limitation by heating up the sample while simultaneously cooling the extraction phase. In this cooling/heating environment, the coating/sample matrix distribution coefficient of analytes increases dramatically according to the following equation [1].

Where, \( K_a \) is the coating/headspace distribution coefficient of analyte when the coating and headspace temperature are both at \( T \).

The cold fiber device was coupled with GERSTEL autosampler to achieve complete automation [2]. A septumless head injector was utilized to accommodate the large external needle. Fourteen compounds within a large range of volatility and polarity were chosen as target compounds for fully evaluate the capabilities of this device.

Experimental set-up

- Instrument: Gas chromatograpgh (GC) and mass spectrometer (MS) was used for separation and analysis. A GERSTEL multipurpose sampler (MPS 2) was coupled to achieve the automation.
- Experimental design:
  - Fabrication of the fully automated cold fiber device.
  - Evaluation of the reproducibility of the cold fiber SPME.
  - Comparison of the extraction efficiency of cold fiber to the PDMS coating.
  - Comparison of the extraction efficiency of fiber to the commercial DVB/CAR/PDMS coating.
  - Comparison the linear range of calibration curve and limit of detection of cold fiber to other fibers

Results and discussion

The cold fiber device was built on a 100 ul syringe barrel. The liquid CO₂ tubing was inserted inside the plunger tubing.

The cold fiber was mounted on the autosampler arm and connected to the liquid CO₂ and the temperature controller.

Discussion:

- The reproducibility of different fibers

Table 1. The comparison of fibers' reproducibility of peak area and the maximum shift of retention time

![Table 1](image)

- The comparison of cold fiber extraction efficiency other PDMS fibers.

![Figure 4](image)

Discussion:

For higher extraction temperature extraction, the extraction efficiency of the cold fiber is much higher than the DVB/CAR/PDMS.

- Comparison the linear range of calibration curve and limit of detection of cold fiber to other fibers

![Figure 3](image)

The linear range and LOD of cold fiber was compared to that obtained by commercial PDMS and DVB/CAR/PDMS fibers.

- The automated cold fiber SPME method can achieve higher sensitivity than other commercial fibers for solid sample sampling due to the cooling effect of the extraction phase.

- The cold fiber SPME is suitable for high temperature sampling exemplified by solid matrix sampling.

Reference and acknowledgment


GERSTEL, Inc.