References for "SPME – A Fast and Inexpensive..."

(1) SPME Application Guide – T199925.

Trademarks and Registered Trademarks:

*Carboxen, Discovery* - Sigma-Aldrich

Patents:

*SPME* - Technology licensed exclusively to Supelco. US patent #5,691,206; European patent #523092.

## SPME – A Fast and Inexpensive...

*(continued from page 1)*

can Water Works Association (AWWA) method 6040B, is very time consuming requiring several hours per sample.

### SPME for Detection of Odor-Causing Compounds

SPME provides results comparable to CLS. An SPME fiber extracted the organics from the headspace of a water sample heated at 65°C. The sampling time was 30 minutes. The dual coated divinylbenzene-carboxen-PDMS fiber provided the best extraction and desorption results for MIB and geosmin. Figure 2 shows a chromatogram of the odor compounds desorbed from the SPME fiber. GC/MS ions 95 and 112 confirmed the identity of MIB and geosmin, respectively. Concentrations were determined to be 1ppt by standard addition. Recently, the AWWA approved a new method, 6040**D,** that uses SPME as an alternative to the CLS technique.

### Conclusion

In summary, SPME is a simple and inexpensive sample extraction technique that can achieve very low levels of detection of trace organic compounds present in a variety of sample matrices. In this practical example, SPME provides results comparable to CLS for the extraction and detection of the compounds responsible for musty odors in drinking water at ppt levels in much less time. SPME is a simple, fast and low cost extraction technique that should be your first choice for evaluating a suitable technique for extracting and analyzing trace organic compounds from a variety of matrices.

*For more information on the use of SPME for determination of MIB and geosmin in water, request T398147 – Solid Phase Microextraction of Odors in Drinking Water for Analysis by GC/MS.*

**CASE STUDY 1**

## Resolving Combichem Purification Problems

In recent years, advances in combinatorial chemistry (Combichem) have made a tremendous impact on the pharmaceutical industry by drastically accelerating the drug discovery process. The marriage of robotic liquid handlers, multi-well platforms, and well-established combinatorial techniques has allowed the simultaneous synthesis of large molecularly diverse arrays of potentially biologically active molecules. However, for each synthesis a purification step is required to remove the target molecule from reaction by-products, side-products, and excess reagents. Because many reactions contain ionic impurities and products that could be selectively extracted with ion-exchange resins, ion-exchange solid phase extraction (SPE) has become a routine procedure for purifying solution-phase combinatorial reactions. However, as more compounds are synthesized, there is a larger demand on parallel purification which typically involves the implementation of either smaller bed weights, tube sizes, and/or 96-well SPE. As bed weights scale-down, sufficient loading capacity becomes a major issue for many combinatorial chemists.

This concern was shared by Dr. Bart van Steen, Advanced Drug Discovery Support, Solvay Pharmaceuticals, The Netherlands. Both Supelco scientists and Dr. van Steen agreed that the majority of commercially available pre-packed ion-exchange SPE tubes and well plates contain silica-based sorbents that do not meet the capacity needs required in combinatorial chemistry. For example, most strong cation exchange silica-based sorbents are functionally bonded to either propylsulfonic acid or benzenesulfonic acid, both of which have an average binding capacity of 0.2 and 0.8meq/g, respectively. This translates to a typical loading capacity of just <10mg per well in a 96-well SPE plate. Because most combinatorial synthesis scales are in the range of 25-300mg, silica-based sorbents do not adequately address the capacity needs when scaling up to 24- and 96-well purification platforms. Upon confirming Dr. van Steen's capacity issues, Supelco and Solvay Pharmaceuticals partook in a joint effort in evaluating alternative ion-exchange materials and methods. Using a representative test compound in conjunction with an SPE method suggested by Solvay Pharmaceuticals, we discovered a resin that exceeded Solvay's expectations by providing capacity of 2.5meq/g, or approximately 30mg loading capacity when using a 75mg/well 96-well extraction platform. Unlike most silica-based ion exchange sorbents, this resin is styrene-divinylbenzene co-polymer functionally bonded to sulfonic acid, and stable across the entire pH range. The material comprises of whole spherical beads offering excellent kinetic and packing properties well suited for SPE. Upon determining the appropriate ion-exchange material, Supelco was able to assist Solvay by custom packing the resin into flangeless SPE cartridges and 96-well SPE plates that continue to serve an integral purification role within Solvay's Automated Molecular Assembly Plant, their high throughput Combichem facility.

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