A cooperation between GERSTEL K.K., Tokyo, Japan and the Research Institute for Chromatography (RIC) in Belgium has produced the multi-Stir Bar Sorptive Extraction (mSBSE) technique. mSBSE has been used successfully for the determination of a wide range of flavor compounds in aqueous matrices such as green tea. A key element of the technique is the GERSTEL Twicester®, which enables the simultaneous use of both a PDMS and an EG-Silicone Twister in one sample vial. These are then desorbed together in the Thermal Desorption Unit (TDU) for efficient combined and comprehensive analysis. The Twicester enables the determination of a wide range of analytes of different polarity in one extraction step and one GC/MS run.

In the search for suitable more polar phases for Stir Bar Sorptive Extraction (SBSE) it was found that generating a rugged and reproducible coating with a more polar phase was more difficult than had initially been expected. A supporting grid was therefore used to produce an ethylene glycol-modified Silicone (EG Silicone) coated stir bar and a novel SBSE procedure termed multi-SBSE (mSBSE) was developed to combine the use of the two Twister types. When performing mSBSE, the PDMS stir bar stirs at the bottom of the vial while the EG Silicone stir bar is attached on the inner side wall of the vial using a magnetic clip mounted on the outside of the vial. After extraction, the two stir bars are placed in a single glass desorption liner in which they are simultaneously thermally desorbed and the combined analytes determined in a single GC/MS run. Compared to conventional SBSE, mSBSE provides more uniform enrichment of a wide range of odor compounds in aqueous samples since the stir bars complement each other. Using the mSBSE technique, more than 30 extraction and desorption cycles could be performed with an EG Silicone stir bar without loss in performance. The recoveries for polar solutes such as 2-acetyl pyrrole (log Kow: 0.55), benzyl alcohol (log Kow: 1.08), guaiacol (log Kow: 1.34), and indole (log Kow: 2.05) were increased by a factor of about 2-7. The mSBSE-TD-GC/MS method showed good linearity ($r^2 > 0.9913$) and high sensitivity (limit of detection: 0.011-0.071 ng/mL) for the test compounds spiked in water. The feasibility and benefits of the method were demonstrated by using the technique for the determination of odor compounds in roasted green tea. The normalized areas obtained from mSBSE showed an improvement in enrichment factor for most of the selected compounds compared to conventional SBSE using the PDMS stir bar or the EG Silicone stir bar respectively. Fifteen compounds were determined in the range 0.15-210 ng/mL (RSD < 14 %, n = 6).

For more information
Sequential dynamic headspace sampling of brewed coffee

Headspace gas chromatography (HS-GC) is frequently used for aroma analysis due to the volatility of aroma compounds. Several established HS techniques are available, for example SHS, DHS, HS-SPME and HSSE. However, in terms of analyte recovery, these techniques tend to discriminate in favor of more volatile and/or hydrophobic compounds. Recently, a full evaporation DHS (FEDHS) method, based on a classical full evaporation technique (FET) was demonstrated for uniform enrichment of aroma compounds from several sample types. FEDHS of 10–100 µL of sample at 80 °C using a valve-less short-path DHS system enables near complete vaporization and uniform recovery of aroma compounds, while largely eliminating non-volatile matrix. However, the FEDHS method often requires a large purge volume in order to remove water from the adsorbent trap. This can lead to loss of volatile compounds due to breakthrough in the adsorbent trap during the purge step. The FEDHS analysis is performed using a DHS module and thermal desorption unit (TDU), both mounted on a GC/MS system using a highly flexible MultiPurpose Sampler (MPS) for automation. Replaceable adsorbent traps are used for analyte concentration, enabling sequential sampling from the same HS vial under different trapping conditions, even using different traps and adsorbents. This flexibility enables the extraction and trapping of more volatile compounds in a traditional DHS step prior to the FEDHS extraction step. A novel multi-volatile method (MVM) with sequential DHS sampling (and desorption) has now been developed based on using a variety of trapping conditions for the determination of a wide range of aroma compounds in aqueous samples. The MVM method consists of three different DHS sampling steps including a final FEDHS step. The DHS parameters were examined with the model aroma compounds spiked in 100 µL of water. Feasibility and benefits of using the MVM method is demonstrated through the determination of key odor compounds in brewed coffee.

For more information

Aroma Office 2D

MS- and Two-Dimensional Linear Retention Index Database for identification of flavor compounds

The retention time of a compound on a given column phase can be expressed on a scale based on n-alkane retention times. This produces unique retention index values for compounds and serves to standardize gas chromatographic retention data. Both linear retention indices and programmed-temperature retention indices are widely used in the flavor and fragrance field and many published data bases are available. Usually mass spectral information in addition to retention time data is available from a GC run, but either information dimension alone is often insufficient for positive identification – even though modern affordable bench top instrumentation offers highly reproducible retention behavior and information rich mass spectral patterns. Aroma Office 2D, exclusively available from GERSTEL, offers an integrated software approach to automatically process retention index and mass spectral data for improved identification of flavor compounds based on the most comprehensive data base of flavor compounds commercially available. This is a searchable data base with retention index information on >10,000 compounds from greater than 100,000 entries from a wide range of literature references. The program can be integrated into the Agilent ChemStation software and searches are performed using RI values and the CAS No. of a candidate compound after library searching. A manual cross search for a single or limited number of compounds can be performed or an automated cross search can be performed for multiple compounds. Both use a single RI and a mass spectrum for each compound. When the chromatographic analysis is upgraded to two dimensional with heart cutting the software also offers a cross search using two different retention index values obtained from the orthogonal stationary phases used in the first and second dimension analyses. When GC-O organoleptic evaluation is available from both first and heart cut dimensions these signals can provide complementary RI values. This is often sufficient to propose an identification even if the MS signal is weak or absent. Aroma Office 2D is designed to offer significant additional identification strategies to the practicing flavor analyst.

For more information
… please contact gerstel@gerstel.com or your local GERSTEL representative (www.gerstel.com).
**Single Position Preparative Fraction Collector (sPFC)**

The Single-Position Preparative Fraction Collector (sPFC) from GERSTEL can significantly expand the analytical possibilities of the modern flavor analysis laboratory. The sPFC enables collection, trapping and concentration of a compound or fraction for subsequent analysis using other techniques. The fraction in question is transferred to an adsorbent tube through a heated deactivated transfer line for best possible recovery. Analytes from one or more GC/MS runs can be collected for subsequent analysis using other techniques. If needed, and if appropriate, analytes can be detected and classified using olfactory detection by inserting a GERSTEL ODP nose cone instead of the adsorbent tube. To operate an sPFC/ODP, multidimensional column switching using, for example, a Deans’ switch system is required. Software is available to facilitate set-up and method development as well as for addition of spoken olfactory classifiers and intensity rating to an olfactogram, which can be transferred and overlaid with the chromatogram for simple and efficient correlation. More information: Journal of Chromatography A 1218 (2011) 3180-3185.

**Twister on Tap**

It is the classical demonstration effect: Your tap water has had a “medical”, “chlorinated”, “chemical” or “moldy/musty/earthy” off-flavor for a while, but when someone finally shows up to take a sample and presumably fix the problem, everything is fine. As soon as the person is out the door, the off-flavor is of course back – what can a person do? Intermittent off-flavor incidents require Time Weighted Average (TWA) sampling over an extended period of time. A passive sampling device is now available, which can be mounted directly on the tap. ARISTOT (Advanced Relevant Investigation Sampler for Taste & Odor at Tap) enables the extraction and concentration of VOCs and off-flavors from tap water directly on the tap. The extraction devices are six GERSTEL Twister®s, which are mounted inside the ARISTOT module. As tap water flows over the Twisters, analytes are extracted and concentrated in the Twister PDMS coating. Qualitative and quantitative determination then follows using automated thermal desorption coupled to a gas chromatograph with mass selective detector (TD-GC/MSD).


**Literature**

Flavor, Fragrance, and Odor Analysis, Second Edition

There are many advantages to stir bar sorptive extraction (SBSE) for isolating and concentrating flavor active chemicals from foods. These include simplicity, wide application range, efficient analyte concentration, and generally the absence of masking solvent peaks. Written from a practical, problem-solving perspective, the second edition of Flavor, Fragrance, and Odor Analysis highlights this powerful technique and emphasizes the range of applications available.

The final chapter summarizes chemical identities of characterizing aroma chemicals in fruits, vegetables, nuts, herbs and spices, and savory and dairy flavors. It also provides a brief compendium of the characterization of off-flavors and taints that are reported in foods. With contributions from a distinguished panel of international experts, this volume provides chemists and researchers with the latest techniques for analyzing and enhancing food flavor and fragrance.

Topics discussed include:

- Sequential SBSE, a novel extraction procedure
- A simplified method for switching from one-dimensional to two-dimensional GC/MS
- How to improve analytical sensitivity and recovery of phenolic compounds with aqueous acylation prior to SBSE GC-MS
- Analyzing and combating off-flavors caused by metabolites from microorganisms
- A technique for measuring synergy effects between odorants
- The identification of the characterizing aroma-active compounds of tropical fruits with high economic potential
- The parameters utilized during the production of aqueous formulations rich in pyrazines
- How spectral deconvolution can be used to specify the subtle differences in essential oil content and track key ingredients through the manufacturing process

More Information

Olfactory Detection Port (ODP 3)

The GERSTEL Olfactory Detection Port (ODP 3) is used by leading scientists and olfactory analysts in leading companies worldwide. Optimized for excellent recovery, even for high-boiling and polar compounds, the ODP 3 reliably and concisely presents compounds that elute from a GC column to the human nose for accurate determination. Parallel analytical detection by any GC detector, including MSD, FID, and FPD can be set up with flexible split ratio. An olfactogram with reporting of descriptors and intensity is superimposed on the chromatogram and presented as part of a comprehensive report for each GC run. The ODP 3 can be optimized for individual preferences in terms of ergonomic position, flows and humidity. The ODP 3 is an effective tool for obtaining simultaneous sensory and analytical information in determining odors in foods, beverages, fragrances, consumer products and other complex samples - as well as in the associated packaging material. GERSTEL offers regular practical workshops with a highly experienced flavor analyst as instructor.

Company Description
GERSTEL develops and produces automated sample preparation and sample introduction accessories for GC, GC/MS, LC, and LC/MS. GERSTEL technology enhances productivity and significantly improves detection limits. GERSTEL is recognized by Agilent Technologies as one of their Premier Solution Partners. GERSTEL solutions can also be integrated into other leading manufacturer’s systems.

Markets Served
• Food, flavor and fragrances
• Water and beverages
• Personal care and cosmetics
• Forensics
• Microelectronics and semiconductors
• Polymers and packaging — leachables and extractables
• Environmental and industrial hygiene
• Pharmaceuticals

MultiPurpose Sampler MPS: GC, GC/MS, LC, and LC/MS autosampler and sample preparation robot. Performs the sample preparation techniques listed below and more.

Online SPE system: (SPE<sup>200</sup>): Performs online SPE with automated cartridge exchange

Automated SPE: Independent of LC/MS or GC/MS or combined with sample introduction. Based on standard cartridges.

Dynamic Headspace (DHS): Concentrates VOCs from liquids or solids. Ultralow detection limits.

Thermal Desorption System (TDS): Ultra-low detection limits for VOCs and SVOCs up to n-C40. Performs thermal desorption/ extraction and pyrolysis.

Thermal Desorption Unit (TDU): Highly flexible, automated analysis of up to 196 gaseous, liquid, or solid samples.

Twister® Stir Bar Sorptive Extraction (SBSE): Ultra trace-level determination of organic compounds in liquid matrices. Up to 1000 times more sensitive than SPME.


Automated Liner EXchange (ALEX): For samples with a heavy matrix load. Automated Centrifugation, Solvent Evaporation, Weighing, Filtration, Vortexing, and Bar Code Reading

Multidimensional GC with Column Switching: Complex separations using two or more columns.

Olfactory Detection Port (ODP): For GC or GC/MS; complete with voice recognition, intensity indication, and peak annotation.

Preparative Fraction Collector (PFC): For GC or GC MS using up to six traps.

By the way: GERSTEL offers regular practical ODP training sessions with a highly experienced flavor analyst as instructor. For more information, please contact Thomas Albinus, application specialist and trainer at GERSTEL: thomas_albinus@gerstel.com or visit www.gerstel.com.

NEWSLETTER

GERSTEL GmbH & Co. KG
Eberhard-Gerstel-Platz 1
45473 Mülheim an der Ruhr
Germany
+49 (0) 208 - 7 65 03-0
+49 (0) 208 - 7 65 03 33
gerstel@gerstel.com
www.gerstel.com