Merck

WHAT IF THE UNSEEN WERE SEEN?

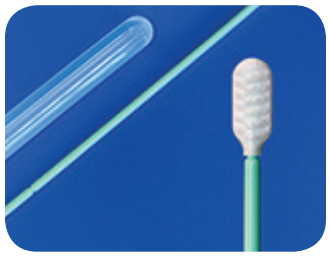
LET'S CREATE ACCURACY IN FORENSIC TESTING. TOGETHER.

Sample Handling

At Merck, we offer tools for sample collection, preparation, analysis and quantification for all your forensic analysis needs - from **small molecules** to **DNA** and **macromolecules.**

Sample Collection

We offer a large variety of sample collection products for use in life science research. From collection and storage, to transportation, we provide containers suitable for proteins, nucleic acids, urine, blood, and sputum.



Vials and Syringes: Protect Your Valuable Sample

Vast selection of vials for MS applications

Clear or amber? Glass or plastic? Choose from a wide variety of vials for any LC-MS application or your everyday usage. We offer the newest and most up-to-date selection of vials for your analytical needs, including our new Low Adsorption (LA) or Center Draining (CD™) vials. Our MRQ30 CD vials allow for maximum sample recovery, down to 2 µL of sample remaining in the vial after sample withdrawal, an improvement over standard CD vials. We know some vials can be instrument specific, so if you have trouble choosing, visit **SigmaAldrich.com/vials**, or call your local technical service representative for assistance.

High-quality syringes for any sample matrix

We carry the best syringe selection and the top brands you use every day. Whether you need a liquid or gastight, manual or autosampler syringe, we have the appropriate quality and size for your application and sample volume. To see our entire syringe selection, visit **SigmaAldrich.com/syringes**

Filtration of Samples

Prepare samples using low-extractable Millex® syringe filters to ensure clean baselines and maximize instrument uptime for sensitive analyses, such as LC-MS, UHPLC, and ion chromatography.

We verify membrane integrity for 100% of Millex® filters shipped each day, you can therefore rely on them to remove all particles from your sample. A wide range of chemical compatibility enables use with virtually any sample composition.

For the best results, choose a filter based on the following criteria:

- Membrane that is chemically compatible with your sample
- Pore size recommended for your analytical method
- Filter diameter appropriate for your sample volume
- Choose an integrated prefilter, such as Millex®-HPF filters, for samples with high particulate load

See our dedicated webpage for more information:
SigmaAldrich.com/onemillex

The vacuum-driven Samplicity® G2 filtration system: the better way to use Millex® filters for HPLC samples

For chromatographers who filter dozens of HPLC samples a day through Millex® syringe filters, especially for hard-to-filter, viscous or particulate-laden samples, traditional filtration can lead to manual fatigue. Millex® filters are specified in numerous HPLC methods and standard operating procedures, but the Samplicity® G2 multi-sample filtration system enables you to enjoy Millex® quality, but skip the pain of manual syringe filtration.

The newest Samplicity® G2 system gives you:

- Vacuum filtration of 1-8 samples in seconds directly into HPLC vials
- An ergonomic alternative to syringe filters
- Broader membrane selection
- Compatibility with any protocol or SOP specifying 33 mm Millex® filters

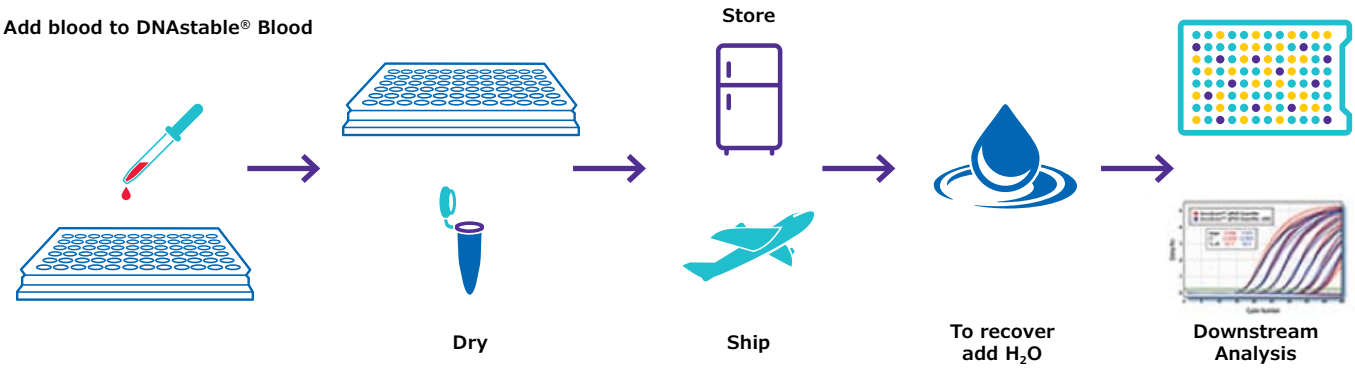
Look at our video on **MerckMillipore.com/samplicity**

Stabilize, Store and Archive DNA and RNA Samples at Room Temperature

Biomatrica® products enable researchers to stabilize ship and store biological samples for long periods of time with complete and rapid sample recovery, all at very affordable cost. Proprietary SampleMatrix® core technology stabilizes and protects biological materials at room temperature without degradation.

- Next generation liquid preservative for purified DNA—at all temperatures
- Liquid format option compatible with automation and sample handling workflows
- Recover high amounts of DNA and analyze instantly without further purification
- Compatible with most downstream applications, e.g. PCR, RT-PCR, SNP analysis, DNA sequencing, etc.
- Cost-effective, eco-friendly solution to freezer and lab space management
- Environmentally sustainable alternative to all forms of cold storage

See more on **SigmaAldrich.com/biomatrica**



Small Molecules

Sample Preparation

Supel™-Select polymeric SPE

Supel™-Select SPE phases are ideal for the solid phase extraction (SPE) of a broad range of compounds from aqueous samples. While reversed-phase interactions dominate retention on the Supel™-Select HLB (Hydrophilic Lipophilic Balanced), and the retention mechanisms of the Supel™-Select SAX and SCX are predominately based on ion-exchange, the hydrophilic modifications of the styrene-based polymer backbone allow for retention and recovery of more polar compounds. These phases are ideal for, but not limited to, urine samples. See more on **SigmaAldrich.com/supel-select**

For most applications, Supel™-Select SPE is amenable to generic methodology with minimal effort required to optimize recovery, selectivity and reproducibility. This ultimately saves valuable time, money and headaches during method development and routine analysis. The phases are available in SPE tubes and 96-well plates.

See also example applications in the back of this literature.

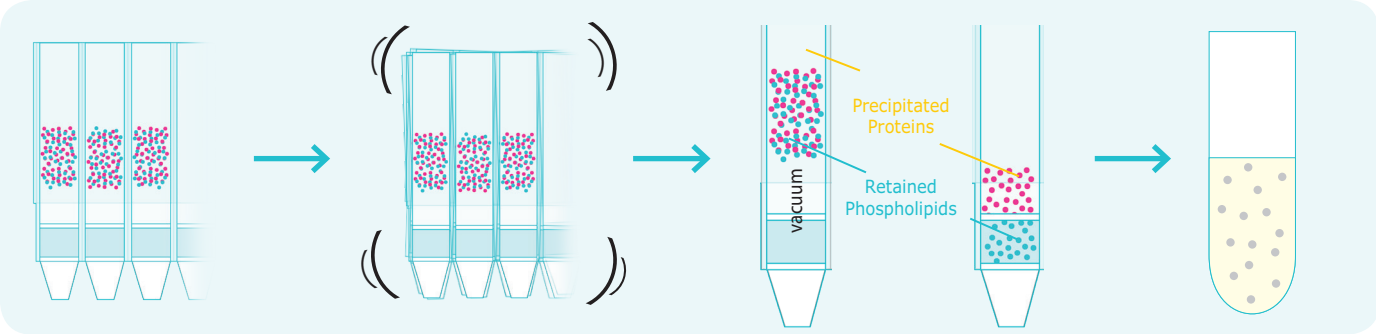
HybridSPE®-Phospholipid products for consistent LC-MS results

When analyzing a compound and its metabolites in biological fluids, such as plasma or serum, analysts frequently deal with interference from the complex sample matrix. Ion-suppression of the mass spec signal due to contaminants in the matrix often limits the ability to properly and reliably identify and quantify the analytes of interest. The presence of phospholipids in biological fluids is one of the major causes of ion-suppression in LC-MS when using positive ion electrospray mode (+ESI). They are not removed by protein precipitation and hence can cause interferences or lengthy HPLC methods. Removing proteins AND phospholipids with HybridSPE®-Phospholipid products is a rapid (same steps as for just protein precipitation required) and reliable means to improve identification and quantification of compounds in biological matrices using LC-MS.

Our HybridSPE®-Phospholipid sample preparation products are available in several configurations.

- Two 96-well plate formats for sample volumes of ~100 µL and 20-40 µL. Both formats allow for in-well precipitation.
- Three SPE tube formats; the Ultra version allows for in-tube protein precipitation.

For more information and to view a video of our HybridSPE®-Phospholipid products in operation, visit **SigmaAldrich.com/hybridspe-pl**



SPME

Solid Phase Micro Extraction (SPME) is an increasingly popular method of extracting analytes directly from the sample matrix and concentrating them for analysis. SPME fibers are available for the extraction of most analytes from most matrices. By tradition SPME is mostly used for GC applications, but can also be coupled with HPLC and LC-MS. To learn more, visit us at **SigmaAldrich.com/spme**

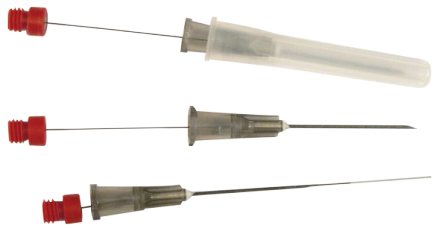
Biocompatible Solid Phase MicroExtraction (SPME) fibers and probes

In recent years, SPME has been developed for use with LC applications by employing biocompatible fibers. We introduced SPME fibers specifically for extraction of polar and nonpolar analytes for LC and LC-MS applications. The fibers comprise C18 silica particles embedded in a proprietary, non-swelling, biocompatible polymer. They are supplied in standard SPME fiber format and set into hypodermic needles.

- **Reduced matrix interference:** The fiber design minimizes binding of macromolecules, such as proteins and phospholipids, but allows extraction of most smaller analytes of interest. This enables sample collection and preparation in one step.
- **Alternative to DBS cards when dealing with precious samples:** BioSPME permits direct sampling in live organisms, either via a shunt or by directly inserting the fiber into the tissue or fluid. Unlike DBS cards, there is no sample degradation or hematocrit interference, and the fibers are easy to store and ship.

  BioSPME fibers can be used with solvent desorption but also offer direct MS use via DESI or DART interfaces. For more information and to view a video of BioSPME probes in action, visit **SigmaAldrich.com/biospme**

See also example applications in the back of this brochure.



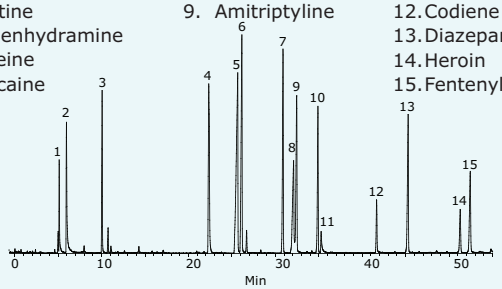
Analysis by GC

Analyses of drugs of abuse can be carried out using both capillary GC and HPLC, but most screening methods use capillary GC. As shown the figure below, our Equity®-1701 columns provide a unique selectivity for this common analysis. In addition, our SLB®ms line of capillary columns provide reliable performance and lowest bleed for GC-MS applications.

GC Analysis of Amphetamine standards on Equity®-1701 (30 m x 0.25 mm I.D., 0.25 µm)

column	Equity®-1701, 30 m x 0.25 mm I.D. x 0.25 µm (28372-U)
oven	45 °C (2 min), 25 °C/min to 110 °C, 15 °C/min to 200 °C, 6°C/min to 280 °C (3min)
Inj.	250°C
MSD interface	280 °C
scan range	40-450 m/z
carrier gas	helium, 0.7 mL/min. constant
injection	0.4 µL, pulsed (40 psi for 0.3 min), splitless (0.5 min)
liner	2 mm I.D. splitless
sample	15-component drug standard, each at 40 ppm in methanol

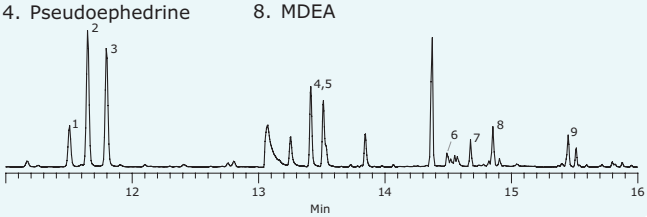
1. Amphetamine
2. Methamphetamine
3. Nicotine
4. Diphenhydramine
5. Caffeine
6. Lidocaine
7. Methadone
8. Phenobarbital
9. Amitriptyline
10. Cocaine
11. Desipramine
12. Codiene
13. Diazepam
14. Heroin
15. Fentenyl



GC Analysis of Amphetamines in Plasma on SLB®-5ms (20 m x 0.18 mm I.D., 0.36 µm) after SPME using 100 µm PDMS Fiber, Fast GC Analysis

extraction	headspace, 300 rpm stirring at 75 °C for 15 min
SPME fiber	100 µm PDMS (57300-U)
column	SLB®-5ms, 20 m x 0.18 mm I.D. x 0.36 µm (28576-U)
oven	75 °C (1 min), 10 °C/min to 190C, 25 °C/min to 300 °C (4 min)
carrier gas	helium, 0.7 mL/min. constant
liner	0.75 mm I.D. SPME
MSD interface	300 °C

1. Amphetamine
2. Phentermine
3. Methamphetamine
4. Pseudoephedrine
5. Ephedrine
6. MDA
7. MDMA
8. MDEA
9. Methylphenidate (Ritalin)



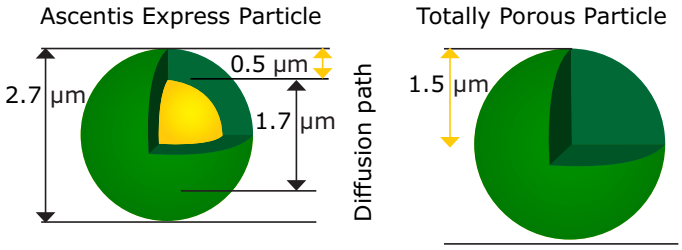
These are complemented by the newest generation of GC phases: Ionic liquids, providing unique selectivities and enabling separations previously not possible.

Read more on our GC offering under **SigmaAldrich.com/gc**

Fused-Core® technology for HPLC and UHPLC: Ascentis® Express columns

Ascentis® Express HPLC columns, based on Fused-Core® particle technology, provide more than twice the speed and efficiency of traditional columns but at half the backpressure of sub-2-µm columns. This performance enhancement is applicable to all HPLC instruments (in addition to UHPLC systems).

Why do columns based on Fused-Core® technology offer superior performance? The Fused-Core® particle has three characteristic features over the traditional porous particle, resulting in a number of performance benefits - especially for fast HPLC and UHPLC applications.



Features of Fused-Core® particles over traditional porous particles

- Narrower particle size distribution
- More consistent packed bed
- Shorter diffusion path within the particle
- Producing more narrow peaks

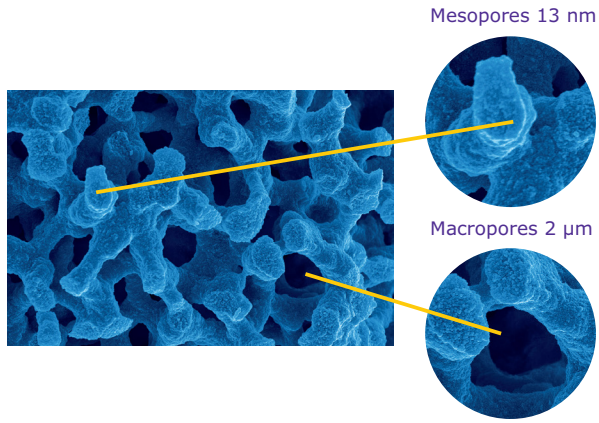
Performance benefits

- More than double the speed of current methods
- Increased resolution over current methods
- Super rugged columns compared to sub-2 µm
- Added sensitivity (from sharper peaks)
- Easily transferable methods, from UHPLC to HPLC

For an in-depth explanation, with practical examples that demonstrate the performance benefits of Fused-Core® technology, watch the presentation linked on this page: **SigmaAldrich.com/express**

Monolithic HPLC columns

Our monolithic silica columns of the Chromolith® family consist of one single monolithic silica rod that exhibits a macro and mesopore structure. There are no frits needed to keep particles in the column, due to this along with the macropore structure, these columns exhibit a very low backpressure and do not tend to clog easily. This allows them also to be used for dirtier samples providing long life time. Connect multiple columns to increase plate count if more resolution is needed, or simply ramp up flow rate to shorten run times. To read more visit us at **SigmaAldrich.com/chromolith**



Derivatization reagents

In some traditional forensic applications based on GC/GC-MS and dealing with non-volatile compounds, derivatization is the only way to make these compounds amenable for GC, turning them into a volatile analytes.

Besides that, modern mass spectrometry techniques such as APCI or ESI are highly successful in providing valuable structural information, and they allow the detection of very low analyte concentrations in various sample matrices. For certain samples e.g. non-polar compounds there are many cases where such methods can be insufficiently sensitive.

Derivatization reactions in mass spectrometry are then used to improve ionization efficiency [1-4]. These reagents have functional groups possessing high proton (cation) affinity that stabilize a positive charge. Of similar importance when derivatizing is the improvement of qualitative analysis by modifying fragmentation behavior to form unique product ions with a shift in retention time. Finally, derivatization can enhance precise quantitative analysis for profiling of relatively small analyte molecules, particularly in metabolomics.

For more information, visit
SigmaAldrich.com/derivatization

References

1. Zaikin V, Halket J, 2009. A handbook of derivatives for mass spectrometry. Chichester: IM Publications LLP
2. Santa T. 2013. Derivatization in liquid chromatography for mass spectrometric detection *Drug Discov. Ther.* 7:9-17
3. Santa T. 2011. Derivatization reagents in liquid chromatography/ electrospray ionization tandem mass spectrometry. *Biomed. Chromatogr.* 25:1-10
4. Santa T, Al-Dirbashi OY, Fukushima T. 2007. Derivatization reagents in liquid chromatography/electrospray ionization tandem mass spectrometry for biomedical analysis. *Drug Discov. Ther.* 1:108-118.

Solvents for LC-MS & GC-MS

LiChrosolv® solvents for liquid chromatography

For reliable and sensitive high-performance liquid chromatography (HPLC) results, high-quality solvents are a must. LiChrosolv® solvents are designed to optimally support analytical HPLC, fast chromatography, and LC-MS applications.

Using LiChrosolv® solvents, you benefit from high reproducibility and sensitivity as well as optimized peak baseline separation. They feature high degrees of UV transmittance, low particle counts, low acidity and alkalinity, and low evaporation residue levels making them ideal for reproducible separations. All our LiChrosolv® solvents are microfiltered through 0.2 µm filters for your convenience.

For analytical HPLC, we provide LiChrosolv® solvents in 'isocratic grade' and 'gradient grade'. Gradient grade LiChrosolv® solvents enable you to minimize the gradient effect of the solvents involved—for example in enantiomeric separations on chiral phases.

For fast chromatography and LC-MS detection, we offer the LiChrosolv® hypergrade family of solvents which are specially optimized and tested for LC-MS suitability. LiChrosolv® hypergrade solvents meet all the requirements of modern LC-MS ionization methods (ESI/APCI – positive and negative mode). Due to their low level of ionic background and low ion suppression, our LiChrosolv® hypergrade solvents ensure high reproducibility and high ionization efficiency—and they are suitable for UHPLC.

To see the list of solvents, visit
SigmaAldrich.com/lc-ms

Solvents for GC applications

When it comes to GC applications, Our SupraSolv® solvents are developed specially for sample preparation in gas chromatography. Ideally suited for all GC applications including sensitive detection tasks in residue analysis, SupraSolv® solvents offer you the security and reliability needed for today's gas chromatography applications.

No matter if you use ECD, FID or MS—our comprehensive portfolio of GC solvents offers a dedicated product quality for your specific application and detection method.

To see the list of solvents, visit
SigmaAldrich.com/gc-solvents

Result Interpretation - Calibration & Quantification

Cerilliant® standards for forensic testing - calibrants and internal standards

Forensic science encompasses a very broad range of analytes and analytical techniques. We offer the analytical standards, certified reference materials (CRMs) and analytical tools required for quantitative analyses in this wide field. Our offerings include standards for illicit drug testing, therapeutic drug monitoring, determination of explosives, fire debris analysis, biogenic amine determination, and even snake venom standards.

All of these standards are subject to the stringent manufacturing processes and strict quality control mechanisms you expect from us. Our CRMs are produced under ISO/IEC 17025 and ISO 17034. accreditation. The standards are packaged in convenient, secure vials, bottles, or ampules and are generally available for immediate shipment.

Search **SigmaAldrich.com/standards** for standards by compound name, CAS#, molecular formula, substructure or synonyms. Price, availability, chemical properties, functions, and safety data are all included for each product.

Our organic compound reference materials are complemented by a comprehensive line of inorganic CRMs.

Visit us at **SigmaAldrich.com/ToxicologyStandards** and download our brochures.

DNA Testing

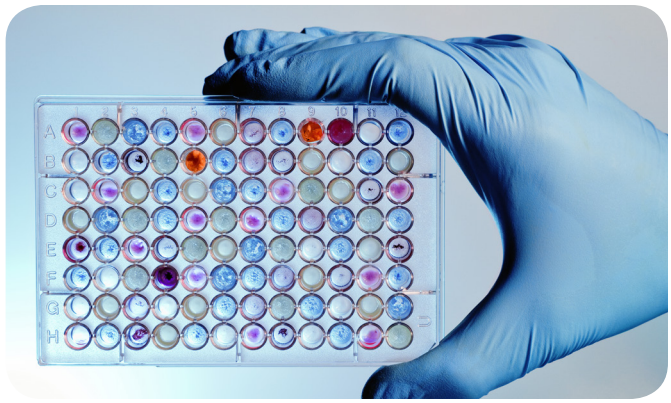
DNA analysis has become the cornerstone of contemporary forensic science. Today, most forensic DNA testing utilizes PCR and capillary electrophoresis (CE)-based analysis methods to detect fragment length variation in short tandem repeat (STR) markers.

Genomic DNA and RNA Purification

We deliver unrivaled quality, expertise and reliability. The GenElute™ portfolio of products includes a variety of kits and reagents for all your nucleic acid purification needs. Whether you are purifying DNA from blood or extracting RNA from fungi, we can help with your experiment.

- GenElute™ plasmid kits offer purification of high quality plasmid DNA that is ready for downstream applications including restriction digest, sequencing, cloning and transfection.
- GenElute™ genomic DNA purification kits provide methods for purifying genomic DNA that eliminate the need for expensive resins and hazardous organic compounds.
- The Extract-N-Amp™ kits provide all the reagents necessary to rapidly extract and amplify genomic DNA for genotyping. The Extract-N-Amp™ method eliminates the need for homogenization and long enzymatic digestions.

Discover more products, including post reaction purification, reagents and high-throughput purifications kits on **SigmaAldrich.com/DNApurification**



Oligonucleotides and Probes

We are recognized as the world's leading supplier of custom and predesigned nucleic acid products, peptides and molecular biology tools servicing the global life science community. Our ability to guarantee quality and performance is directly related to our comprehensive understanding of various synthesis chemistries and manufacturing platforms, our investment in analytical systems, and our experience in methods development. Choose from our comprehensive portfolio of products:

- Custom DNA & RNA oligos
- Custom qPCR probes
- Predesigned primers & probe assays

SigmaAldrich.com/oligos

PCR

We offer a wide variety of PCR reagents to meet any experimental needs. Our range of polymerases is customized to meet your End-Point PCR, qPCR, or RT-PCR needs. Our products vary from routine to enhanced, providing various levels of fidelity, speed, and accuracy.

- The end-point PCR portfolio varies from routine PCR reagents as well as enhanced kits or reagents to ensure accuracy and improve convenience. This category features our unique JumpStart™ taq option.
- Our qPCR portfolio offers a variety of quantitative PCR kits for either probe or SYBR Green applications. The KiCqStart® and LuminoCt® product lines provide convenient, prepared mixes to make your experiment easier. Use our selection guide to find which products are optimized for your instrument.
- The RT-PCR offering includes a variety of kits specific to your needs. Select your instrument to find which products are right for you. Choose the KiCqStart® products for quick and easy, one-step RT-qPCR. Discover more on **SigmaAldrich.com/rt-pcr**

With our PCR Selection Guide, you will be able to select the right product for your application within:

- Routine PCR
  - Standard PCR (REDTaq®, JumpStart™ Taq, KAPA Taq, Taq DNA Polymerase, KOD DNA Polymerase)
  - High Fidelity (Expand™ High Fidelity, Pwo DNA Polymerase, FastStart™ High Fidelity, KOD HOT Start DNA Polymerase)
  - Long and Accurate (REDAccuTaq® LA, AccuTaq™, JumpStart™ AccuTaq™ LA,KAPA LongRange)
  - Long Range (Expand™ and KAPA)
  - Fast PCR & Multiplex (KAPA2G)
- qPCR
  - Detection SYBR® Green (KiCqStart® SYBR® Green)
  - Detection Probe Based (KiCqStart® Probe, LuminoCt®, JumpStart™ Taq, KAPA PROBE FAST…)
- RT-PCR
  - For RT-PCR or RT-qPCR
  - Detection SYBR® Green or Probe Based
  - Choice of instrument

SigmaAldrich.com/pcrselectionguide

New global distribution of high performance Roche reagents for PCR and qPCR

Forensic analysis labs can now turn to us as the exclusive, single-source supplier of the well-respected, high performance reagents developed for PCR and real-time PCR from Kapa Biosystems. This portfolio of reagents uses a high-throughput, directed evolution process that simulates natural selection in the lab, which allows the ability to engineer improvements to the structure and the function of enzymes. These products, paired with our complementary biology offer and industry-leading levels of service, will provide researchers with global access to key materials throughout their workflow.

Features and benefits:

- Used to evolve enzymes that are resistant to inhibitors, enabling direct PCR from samples such as blood, tissue and plant material. Tolerant of the SYBR® Green dye, which improves sensitivity and reaction efficiency for qPCR assays
- High-throughput genotyping with shorter cycling times and higher yields and sensitivity

See more about Roche Reagents for PCR on **SigmaAldrich.com/roche**

Microcon® centrifugal filters – don't amplify contaminants

Microcon® centrifugal filters simply and efficiently concentrate and desalt solutions of DNA, RNA, protein or other macromolecules, using any centrifuge that can accept 1.5 mL tubes. With the low-binding Ultracel® membrane, Microcon® filters offer:

- Typical recoveries of >95%, even for diluted solutions
- Reverse spin to maximize recovery, even in the smallest samples
- Convenient storage of filtrate or concentrated sample in standard microfuge tube

The Microcon® DNA fast flow filter is optimized for the concentration and recovery of genomic DNA. The low non-specific binding characteristics of the membrane and the other device components, coupled with its medical-grade O-ring seal, allows the device to accommodate several wash steps with minimal sample loss. With its inverted recovery spin, the device can concentrate samples up to 100X (for greatest consistency and reproducibility, we recommend concentration factors of < 20X).

SigmaAldrich.com/microcon

Application	10K	30K	DNA Fast Flow
Peptide and growth factor concentration	•		
Protein concentration and desalting of column eluates	•	•	
Protein concentration before electrophoresis or other assays	•	•	
Protein removal prior to HPLC	•	•	
Purification of macromolecular components found in tissue culture extracts and cell lysates	•	•	
Concentration of biological samples (antigens, antibodies, enzymes)		•	
Concentration and desalting of nucleic acids (single-or double-stranded)	•	•	•
Removal of labeled nucleotides	•	•	•
Removal of labeled amino acids	•	•	•
Removal of primers from amplified DNA		•	•
Removal of linkers prior to cloning			

Application Examples

LC/MS Analysis of Illicit “Bath Salts“ in Plasma on Ascentis® Express HILIC after Extraction using SPME LC Tips

The utility of the bioSPME in the pipette tip format for clinical/toxicological applications is demonstrated in the figure below. Here, bath salts, which are illicit “designer drugs” related to phenethylamine and cathinone, were extracted from plasma using mixed-mode C18/SCX SPME fibers, desorbed, and analyzed by TOF-LC/MS. In addition to extracted analyte, overall sample matrix was also monitored and compared with typical dilution/precipitation techniques. The bioSPME extraction had the capability of analyzing sub 10 ng/mL concentration levels of bath salts in plasma samples. It also had 10-fold reduction in detected matrix as compared to standard precipitation techniques, while demonstrating increased analyte response. Ultra-high purity LiChrosolv® LC-MS solvents were used to supply low background interference and low particulate contaminants for robust, trouble-free operation. Cerilliant® CRMs enabled a reliable identification and quantification. This approach demonstrates the ability for quantitative analyte enrichment from limited sample volumes. Typical sample volumes range from 100 µL to 1000 µL.

Synthetic cathinones, marketed as “bath salts” or “plant food” at head shops, convenience stores and on the internet, offer recreational highs that mimic the effects of illegal drugs such as cocaine, methamphetamine and LSD.

These designer drugs are sold in powder or crystalline form under street names including Ivory Wave, Vanilla Sky, Pixie Dust, and White Dove. Packaging labels list the illicit compounds as “not for human consumption,” a tactic used to circumvent regulatory control.

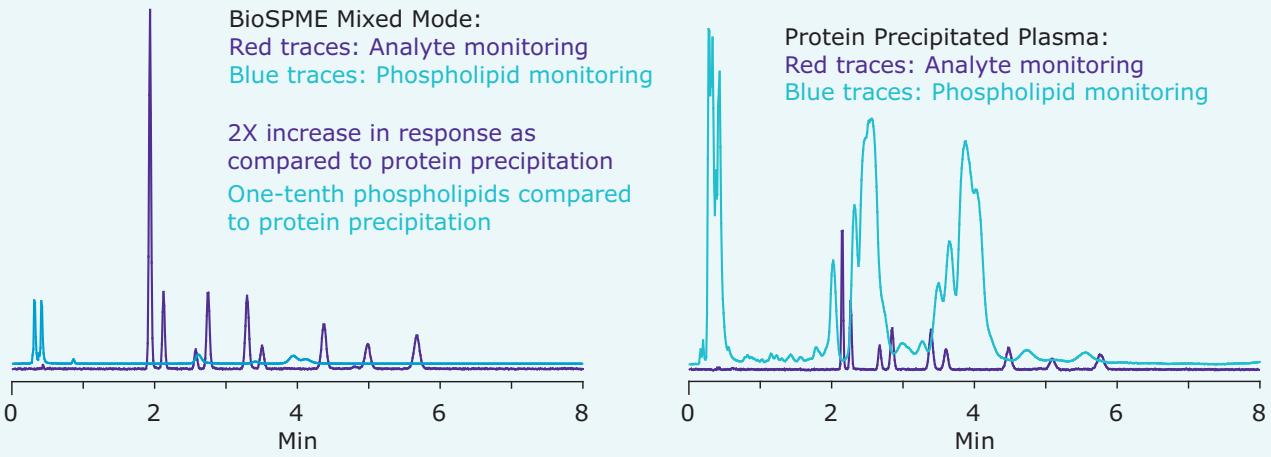
Various states of regulation exist and are evolving in an attempt to control access and use of these potentially dangerous substances.

Our Cerilliant® brand offers the widest selection of Certified Spiking Solutions® for synthetic cathinones and their metabolites and stable-labeled internal standards. Each Certified Spiking Solution® is supported by a comprehensive Certificate of Analysis (COA) that provides all analytical data, as well as uncertainty and traceability information to support regulatory requirements. Our COA includes analytical data on homogeneity and accuracy of concentration by comparison to an independently prepared calibration curve.

TOF-LC/MS of Bath Salts from Plasma Following Extraction with BioSPME vs. Standard Protein Precipitation

system:	Agilent® 1290 Infinity with 6210 TOF
column:	Ascentis® Express HILIC, 10 cm x 2.1 mm, 2.7 µm (53939-U)
mobile phase:	5 mM ammonium formate in acetonitrile: water, 98:2
flow rate:	0.6 mL/min
temperature:	35 °C
pressure:	127 bar
injection:	1 µL
detection:	MS, ESI(+), 100-1000 m/z

Compound	Retention Time (min)
MDPV	2.0
Buphedrone	2.2
3-Fluoromethcathinone	2.7
Butylone	2.9
Ethylone	3.4
3-Fluoromethcathinone	3.7
Mephedrone	4.6
Methylone	5.2
Methedrone	6.0

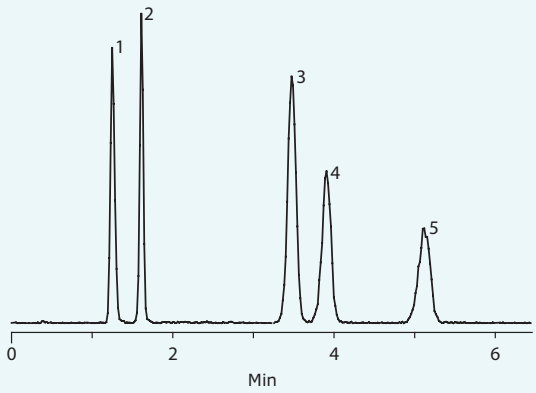


LC-MS/MS Analysis of Synthetic (Spice) Cannabinoids from Plasma using an Ascentis® Express F5 Column after HybridSPE®-Phospholipid Solid Phase Extraction

Synthetic cannabinoids (Spice) are a relatively new type of designer drug used as a pseudo-legal means to get a cannabis-type high. New synthetic cannabinoids are continually being introduced as suppliers tweak the molecular structures. The ability to rapidly and reliably identify the continually changing population of these compounds in the blood or urine of suspected users is a significant analytical challenge facing forensic chemists. A four-pronged approach using column selectivity, high purity solvents, effective sample prep, and reference standards, was used to develop a method to rapidly isolate and identify Spice cannabinoids from plasma. The Ascentis® Express F5 column gave the necessary resolution, and the LC-MS solvents and additives gave adduct-free response for maximum sensitivity. Sample prep employing the HybridSPE®-Phospholipid was rapid and effective and the Cerilliant® reference standards enabled confident identification.

sample/matrix	rabbit plasma, unfiltered K2-EDTA spiked with Spice cannabinoids (5 ng/mL each)
SPE well plate	HybridSPE®-Phospholipid, 96-well plate, 50 mg bed wt., 2 mL well vol (575656-U)
sample addition	to each well add 100 µL plasma, followed by a 300 µL of 1% formic acid in acetonitrile, agitate on orbital shaker for 2 minutes
elution	attach collection plate and apply vacuum at 10" Hg for 4 minutes
column	Ascentis® Express F5, 5 cm x 2.1 mm I.D., 2.7 µm particles (53567-U)
column temp.	35 °C
mobile phase	[A] 10 mM ammonium formate in water, pH 6.8 (unadjusted); [B] acetonitrile; (50:50; A:B)
flow rate	0.3 mL/min
pressure	1296 psi (89 bar)
injection	2 µL
detector	MS, ESI(+), MRM, m/z 344/155 (JWH-073 metabolite), 385/155 (JWH-200), 336/121 (JWH-250), 328/155 (JWH-073), and 342/155 (JWH-018)

1. JWH-073 3-hydroxybutyl metabolite
2. JWH-200 ((1-(2-Morpholin-4-ylethyl)indol-3-yl)-naphthalen-1-ylmethanone)
3. JWH-250 (2-(2-Methoxyphenyl)-1-(1-penylindol-3-yl)ethanone)
4. JWH-073 (Naphthalen-1-yl-(1-butylindol-3-yl)methanone)
5. JWH-018 (Naphthalen-1-yl-(1-pentylindol-3-3yl)methanone)



Arson Investigation: Analysis of Fire Debris using SPME for Headspace Sampling

Furton et al. developed what they described as a simple, inexpensive, rapid, and sensitive method for analyzing gasoline in fire debris, using SPME for headspace sampling[1,2]. According to the investigators, current methods for sampling flammable or combustible liquid residues from fire debris include static headspace sampling (capable of detecting ~10 µL of petroleum product residue) and concentration methods including solvent extraction, dynamic headspace concentration, and passive headspace concentration (capable of detecting ~0.1 µL of petroleum product residue). All of the concentration methods are cumbersome and time-consuming, and require the analyst to use carbon disulfide, a toxic and highly flammable solvent. In a direct comparison of headspace SPME with passive headspace concentration on activated charcoal strips, SPME was faster, simpler, and more economical, being able to extract directly from the headspace of the shipping container, and offering greater sensitivity.

SPME also eliminated the need to expose the technician to carbon disulfide.

Conditions

SPME fiber	polydimethylsiloxane phase fiber, 100 µm headspace sampling (20 min) 10 sec desorption (splitless mode) (57300-U)
Column	100% methyl polysiloxane, 30 m x 0.25 mm I.D., 0.25 µm (Supelco equivalent, Equity®-1, 28046-U)
Oven	35 °C (2 min) to 220 °C at 10 °C/min, hold 2 min, to 300 °C at 30 °C/min, hold 5 min
Carrier gas	helium, 1 mL/min (split 50:1)
Inj. Temp.	splitless (closed 3 min), 220 °C (2 mm I.D. injector liner)
detector	FID, 300 °C

1. Furton K.G., Armirall J.R., Bruna J.C., J. A novel method for the analysis of gasoline from fire debris using head-space solid-phase microextraction. J. Forensic Sci. 1996 41:12-22.

2. Furton K.G., Wang J., Hsu Y., Walton J., Almirall J.-R., The use of solid-phase microextraction–gas chromatography in forensic analysis. J. Chrom. Sci. 2000 July; 38: 297-306

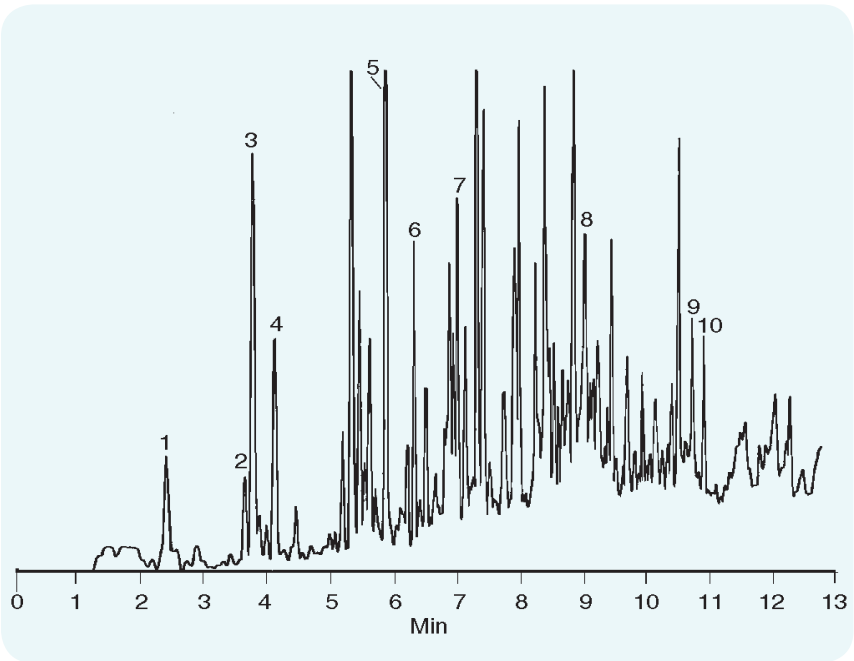


Figure courtesy of José Almirall, Crime Laboratory Bureau, Metro-Dade Police Department, Miami, FL, USA, and Kenneth Furton and Juan Bruna, Department of Chemistry Florida International University, Miami

1. Toluene
2. Ethylbenzene
3. m-Xylene, p-Xylene
4. o-Xylene
5. 1,2,4-Trimethylbenzene
6. 1,2,3-Trimethylbenzene
7. n-Butylbenzene
8. Naphthalene
9. 2-Methylnaphthalene
10. 1-Methylnaphthalene

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