

LCTech FREESTYLE Automated Solution for Sample Preparation

PRESENTED BY

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General Workflow for Sample Analysis

SAMPLE HANDLING

SAMPLE PREPARATION

INSTRUMENT ANALYSIS

DATA ANALYSIS



- Solvent Extraction
- Solid-phase Extraction (SPE)
- QuEChERS
- Evaporation
- Derivatization
- Centrifugation
- Dilution
- Preconcentration



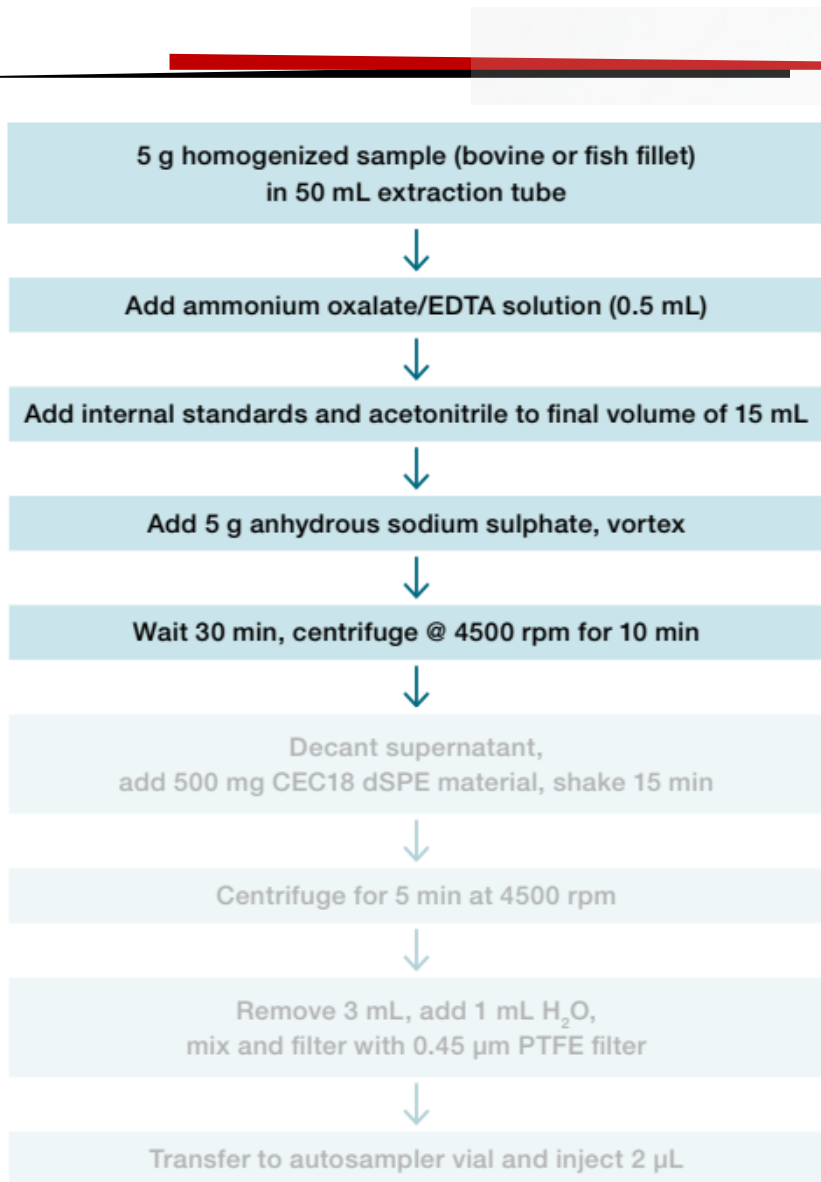
Drawbacks

- Time-consuming
- Labor-intensive
- Human error

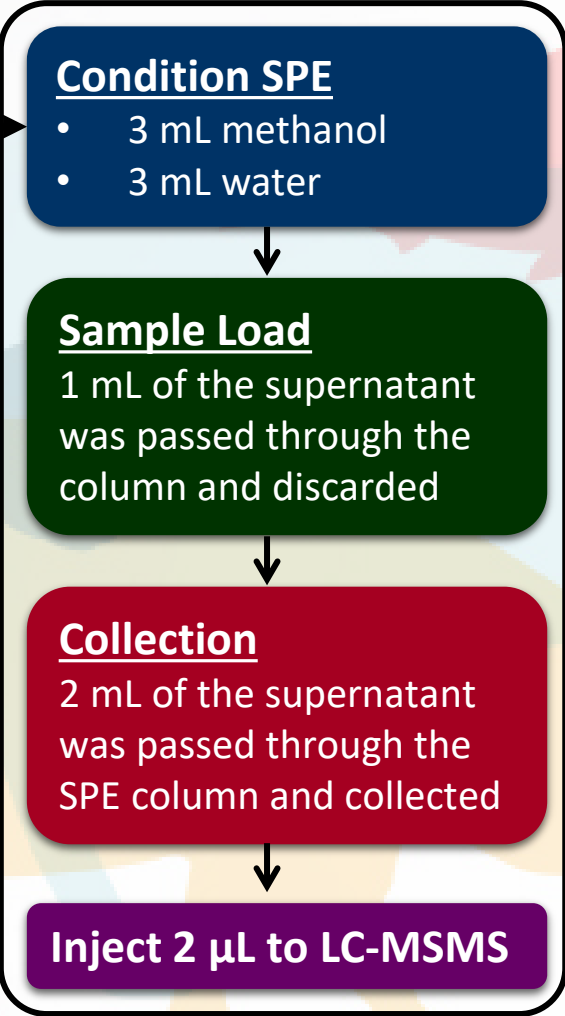


AUTOMATED
SAMPLE PREPARATION

VetDrug Explorer Workflow



SPE Cleanup



Thermo Scientific™
HyperSep™ Retain PEP Cartridges

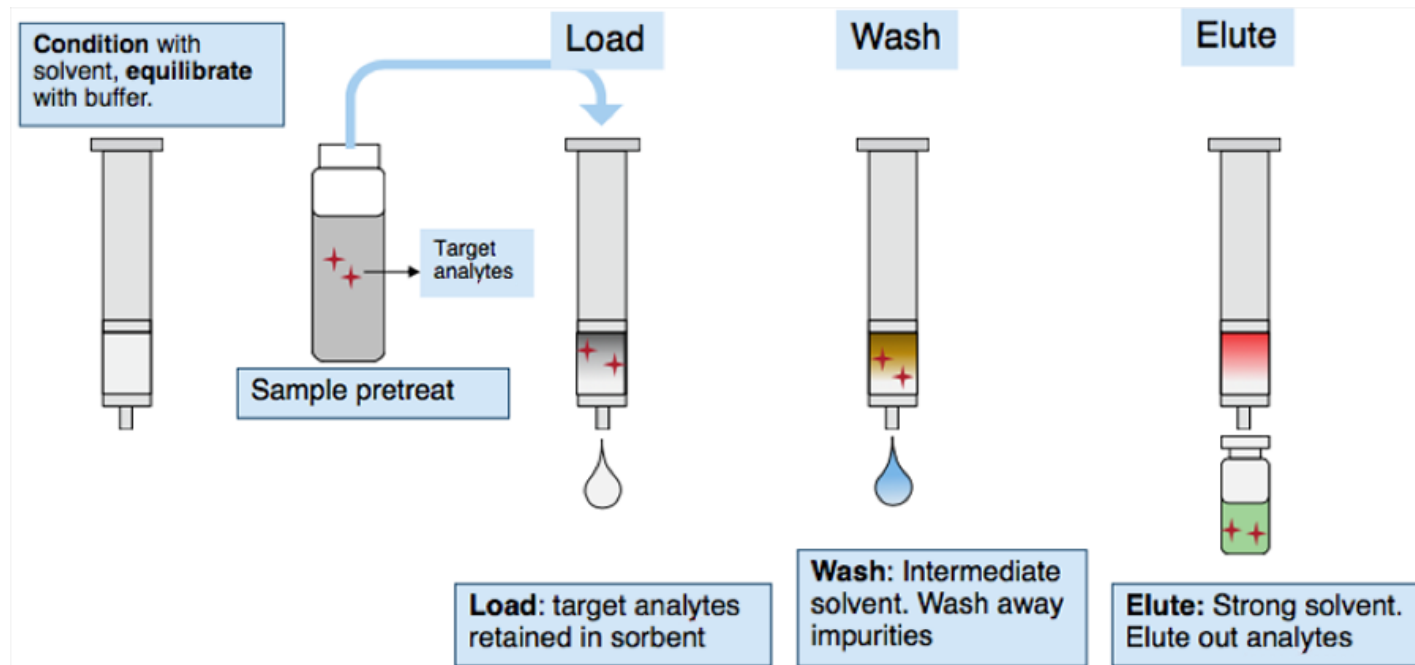
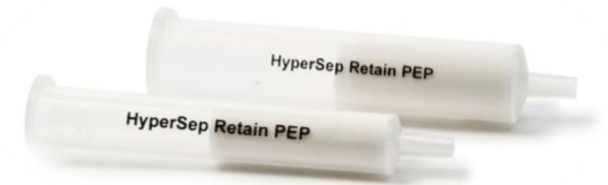


Automated SPE
cleanup & injection



Solid Phase Extraction (SPE)

A sample preparation technique often used by chromatographers prior to analysis. SPE makes use of a **solid phase material** (there are many to choose from) that functions to **retain the interfering substances**, while solvents elute the sample, which is collected and analyzed.



OBJECTIVES:

- In general, SPE can be used for three important purposes:
- Removal of interfering compounds
 - Concentration of the analyte
 - Transfer of analyte into a suitable solvent for analysis

Solid Phase Extraction (SPE)



Sample matrix	Solubility of sample	Polarity of sample	Separation mode	Recommended HyperSep sorbent(s)
Aqueous	Water soluble	Non-polar	Reversed-phase	SOLA HRP, C18, C8, Phenyl, Retain PEP
		Moderately polar	Reversed-phase	SOLA HRP, C18, C8, Retain PEP
		Polar	Reversed-phase	SOLA HRP, Hypercarb Retain PEP
		Cationic	Ion-exchange	SCX
		Anionic	Ion-exchange	SAX Aminopropyl
		Non-polar and cationic	Reversed-phase and Ion-exchange	SOLA SCX/WCX, Verify CX, Retain CX
		Non-polar and anionic	Reversed-phase and Ion-exchange	SOLA SAX/WAX, Verify AX Retain AX
Aqueous	Organic soluble	Non-polar	Reversed-phase	SOLA HRP, C18, C8, Phenyl, Retain PEP
Organic	Organic soluble	Polar	Normal-phase	Silica, Aminopropyl Cyano, Diol
		Moderately polar	Normal-phase	Silica, Florisil, Aminopropyl Cyano, Diol
		Cationic	Ion-exchange	SCX
		Anionic	Ion-exchange	SAX Aminopropyl
		Non-polar and cationic	Reversed-phase and Ion-exchange	Verify CX
		Non-polar and anionic	Reversed-phase and Ion-exchange	Verify AX



AUTOMATED
SOLID PHASE EXTRACTION
INTELLIGENT SYSTEM SOLUTION

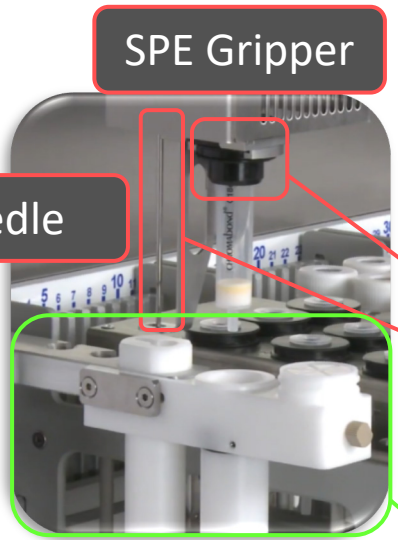


FREESTYLE BASIC



Valves, syringe pump & others on top of y-axis

Flexible robotic area; the appropriate area can be created at any time by choosing the right racks



SPE Gripper

Needle

Rinsing and waste port



NEEDLE – the key for the work



- **Double walled with two independent lines**
 - In the needle
 - Around the needle with spraying holes (360°)
 - Hot sealed – no gap open
- **Thin holes arranged in 360° for**
 - spraying solvent into a vial or into vacuum chamber for rinsing and cleaning
 - removing the air during filling a closed vial with sample
- **Sharp tip for piercing any septum** without taking out a piece of the septum (e.g. needle inlet do not get blocked) and the septum will be restored after taking out the needle



RACKS – The Key for Flexibility

- For vials from 1.0 mL to 1000 mL (e.g. standard flasks, round bottom flasks, TurboVap, Büchi, test-tubes, ...)
- Racks can be cooled or heated.
- With protection for light sensitive samples
- Vials and flasks can be sealed with septa.



- No service or tools needed
- Racks can be individually positioned by hooking them into a toothed plate.

One rack for all kind of columns: reusable adapters allow an easy to change and mixed setup for all kinds of columns.



FLEXIBILITY JUST BY ADAPTERS

- For all **standard SPE-cartridges/columns** from 1 mL up to 15 mL
- For all **standard immunoaffinity columns** (1 mL/3 mL)
- For **glass columns** for max. dimensions 105 mm x 17.5 mm ID (length x diameter)

- Standard **three solvents**, upgrade option up to **six or even fifteen solvents**
- **Positive pressure** up to 4 bar for all steps in which liquids are used.
- **Pressure Monitoring:** If one column is blocked, this one will be taken out of the run, the system gets rinsed and continues the sequence



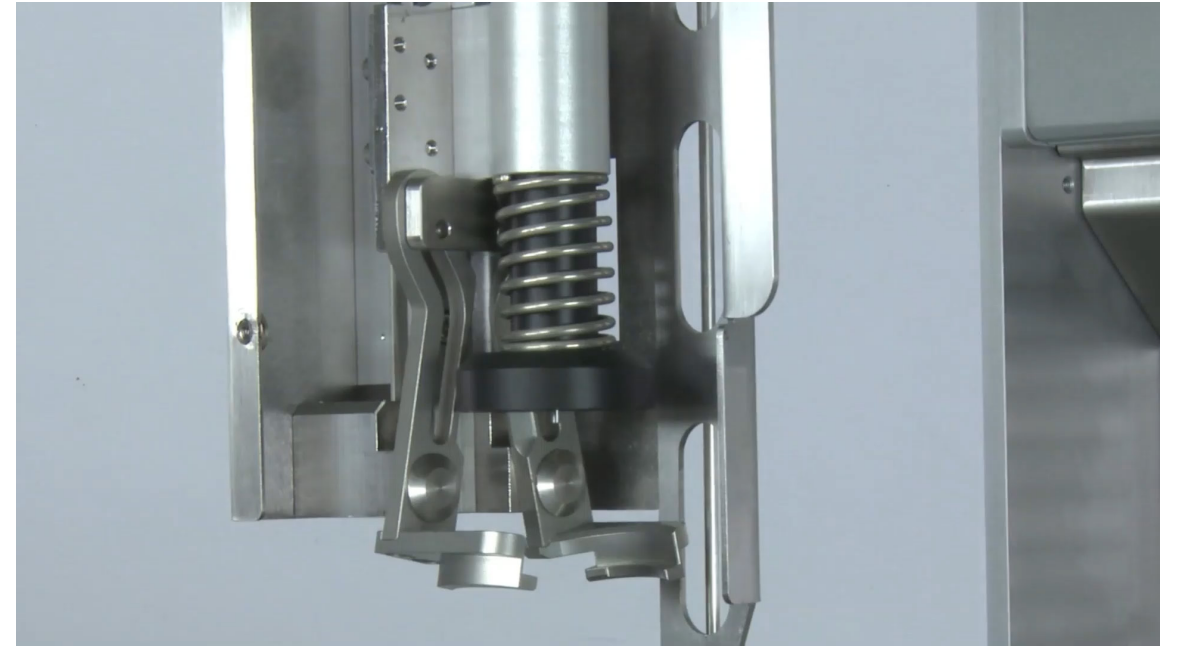
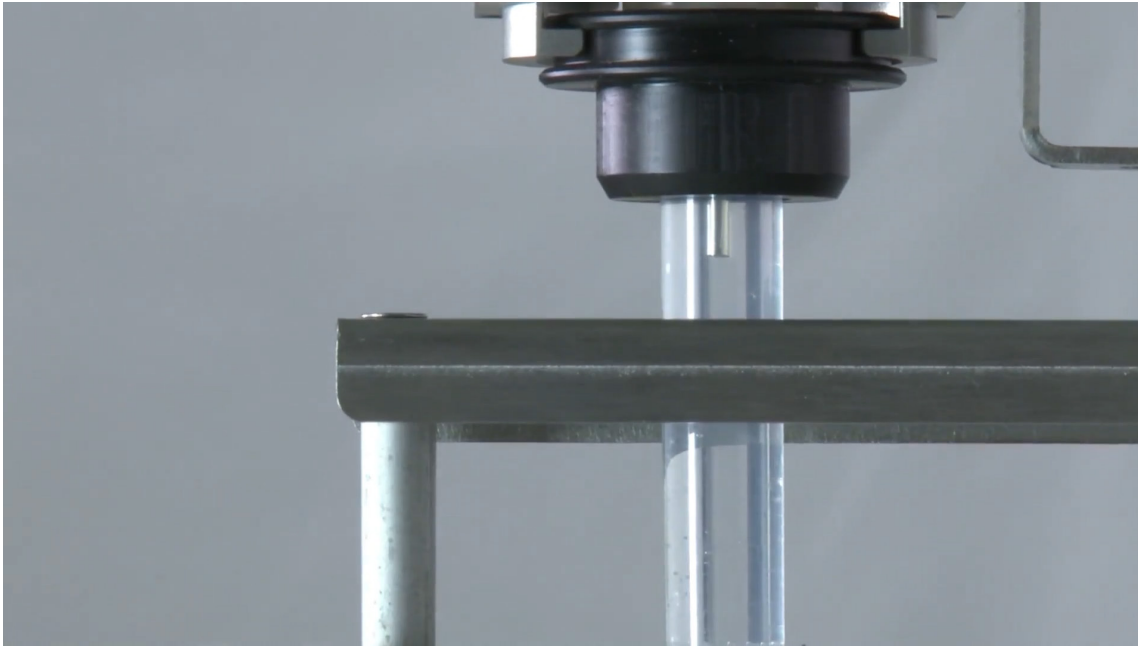
EQUILIBRATION

LOADING

WASHING

ELUTING

SPE GRIPPER - Grasp and seal SPE columns tightly



EQUILIBRATION

LOADING

WASHING

ELUTING

VALVES AND SYRINGE PUMP – Pass solvent through SPE column



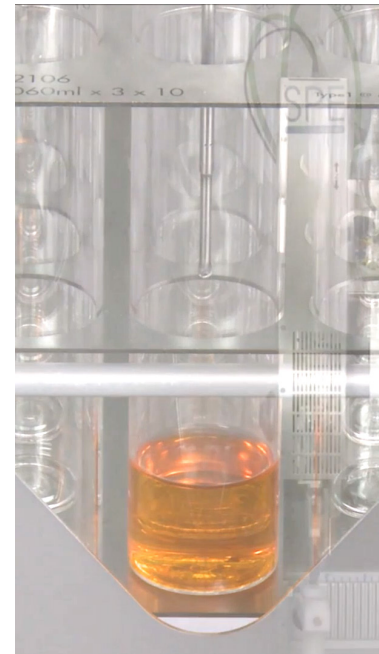
EQUILIBRATION

LOADING

WASHING

ELUTING

NEEDLE, VALVES AND SYRINGE PUMP– Transfer and pass the sample through SPE column



EQUILIBRATION

LOADING

WASHING

ELUTING

VALVES AND SYRINGE PUMP – Pass solvent through SPE column



EQUILIBRATION

LOADING

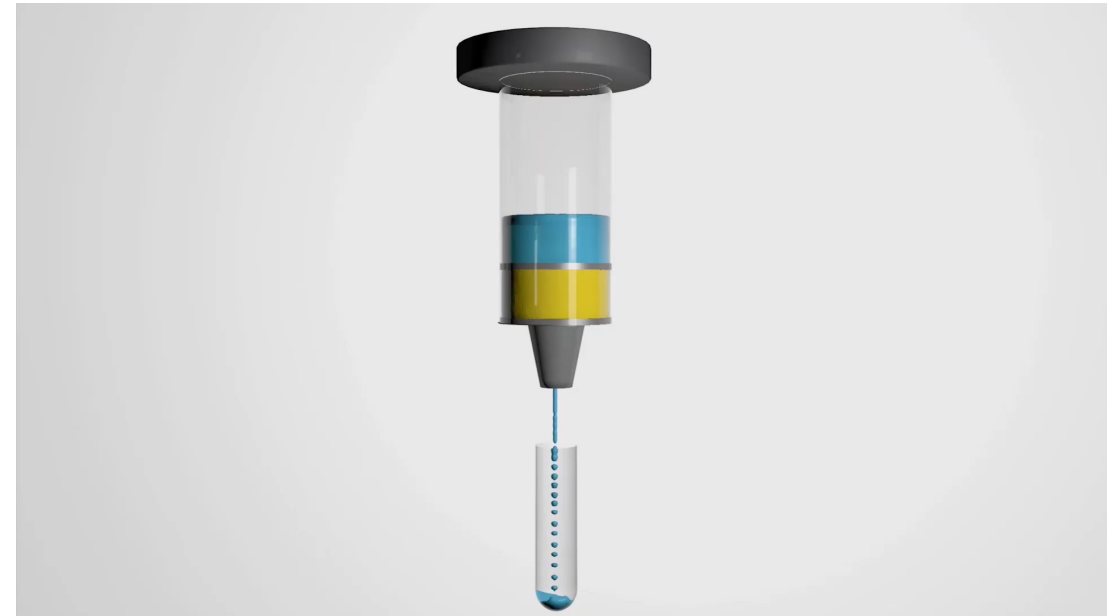
WASHING

ELUTING

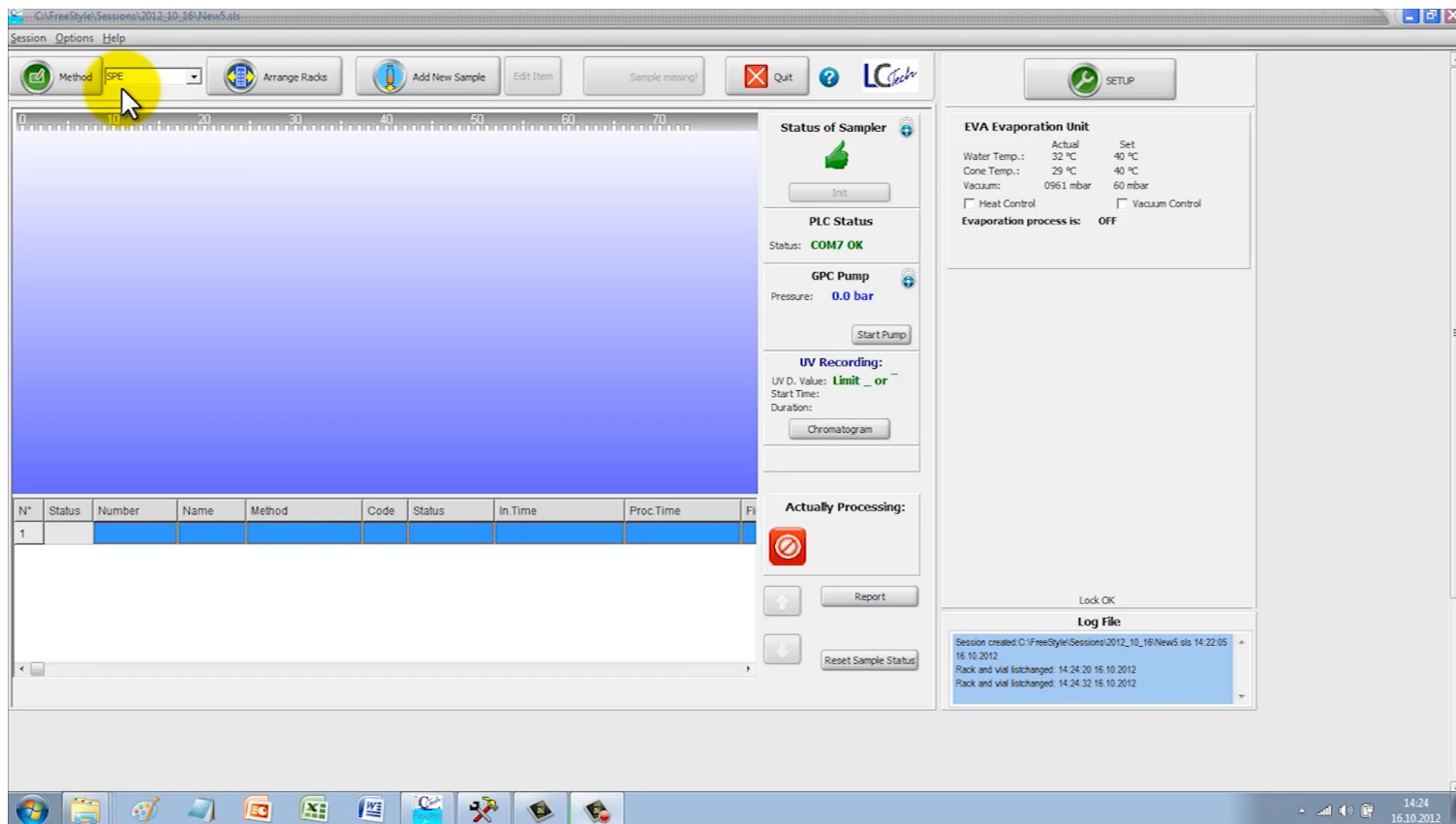
ELUTION, EXACTLY AS YOU REQUIRE



SEVERAL ELUTION PROCEDURES



SOFTWARE – very easy handling

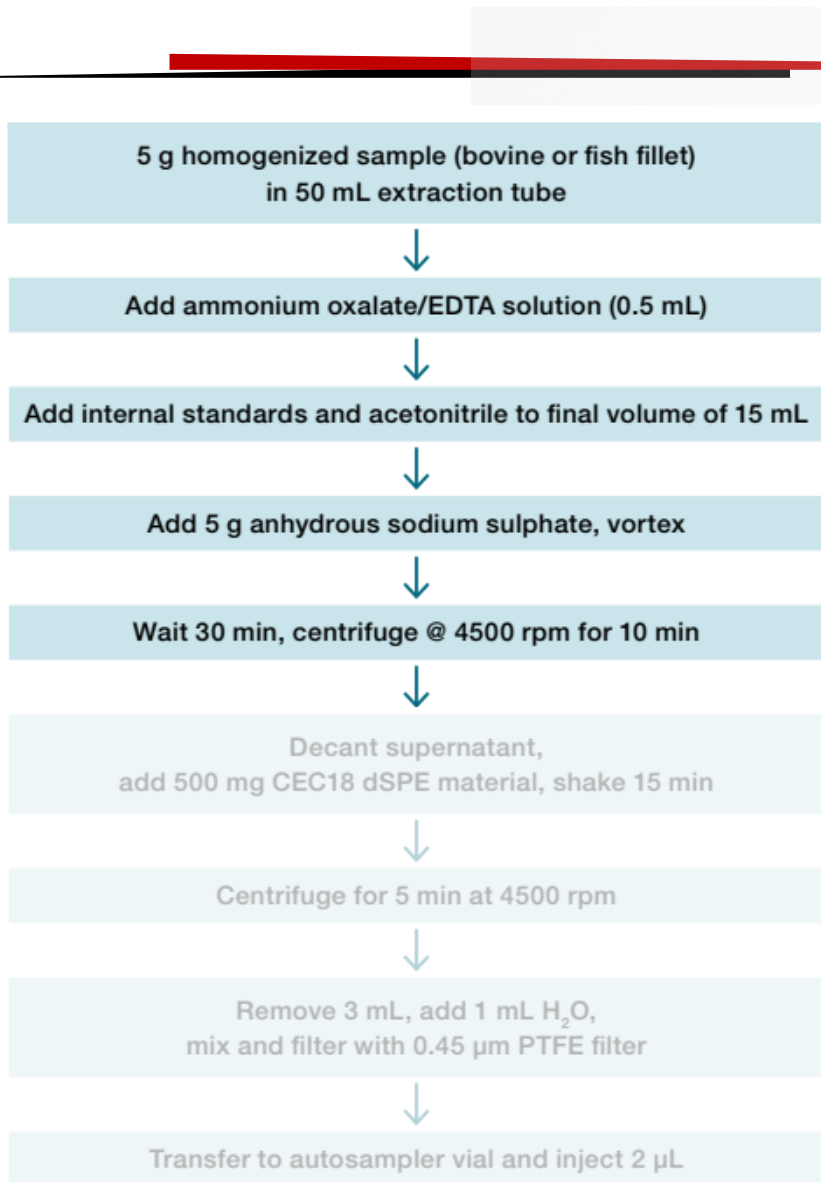


- SPE method: Software allows the transfer of nearly any manual method straight to an automation.

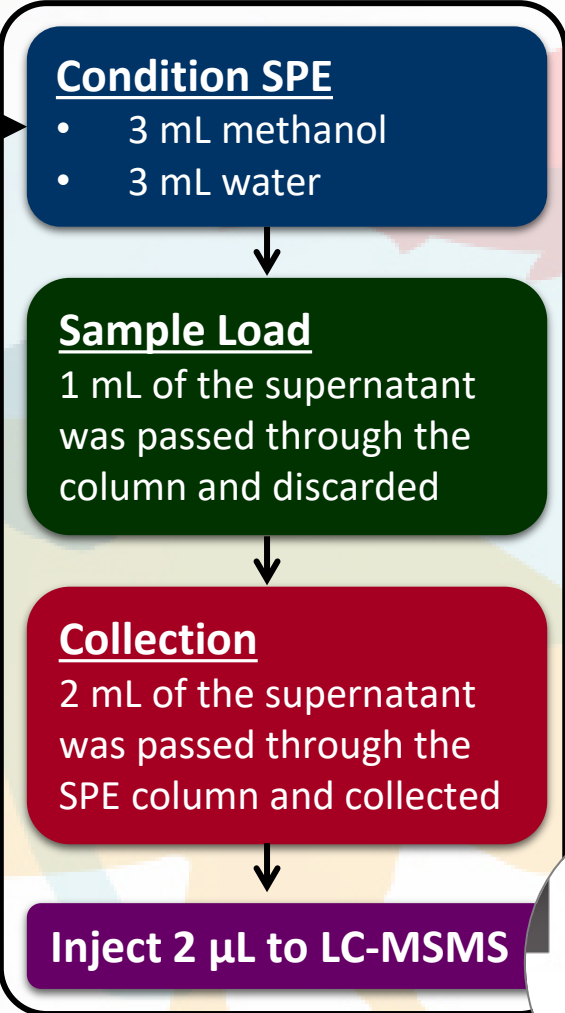
SPE Method Development:

- **Drag & drop** to create the Method.
- Each button there can be individually changed and adopted by using the “bars”.

VetDrug Explorer Workflow



SPE Cleanup



Thermo Scientific™
HyperSep™ Retain PEP Cartridges



Automated SPE
cleanup & injection



Injection Unit

Screening of Veterinary Drug Residues in Animal-derived Foods



thermo scientific

APPLICATION NOTE 21904

Screening of veterinary drug residues in animal-derived foods with a Q Exactive Focus LC/MS system

Authors
Qian Peng, Bing Chen, Teng Guo
Thermo Fisher Scientific, Shanghai, China

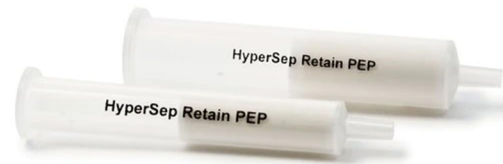
Keywords
HyperSep Retain PEP SPE, Accucore VDX column, Q Exactive Focus, Orbitrap, veterinary drugs, drug residues, meat

Goal
To develop an SPE-LC/MS method for rapid screening and confirmation of 194 multi-class veterinary drug residues in animal-derived foods

Introduction
Veterinary drugs include quinolones, β -receptor agonists, sulfonamides, macrolides, hormones, chloramphenicol, cephalosporins, and penicillins. The simultaneous analysis of multi-class veterinary drugs in animal-derived foods reported in relevant standards and literature is still rare. The main reason for this is that there are hundreds of veterinary drugs and their metabolites, consisting of both water-soluble compounds with strong polarity and non-polar fat-soluble compounds with extremely different chemical properties. In addition, meat samples are a complex matrix, containing protein (15–25%), fat (5–25%), and phospholipids (1–3%). The development of an effective, rapid, and high-throughput sample preparation method for simultaneous screening and confirmation of multi-class veterinary drugs using LC-MS/MS is needed. A complete and reliable analytical method is imperative for residue screening of multi-class veterinary drugs in meat products to improve food safety supervision and to protect the public health and safety.

Thermo Fisher
SCIENTIFIC

SPE Cartridge



Product: Thermo Scientific™ HyperSep™ Retain PEP Cartridges 60 mg 3 mL

Sorbent:

Porous polystyrene divinylbenzene (DVB) material with functionalized group modification, large capacity, and high purity

Property:

Remove most of the protein, fat, and phospholipid interference in meat samples

Meat samples

- Protein (15–25%)
- Fat (5–25%)
- Phospholipids (1–3%)



Screening of Veterinary Drug Residues in Animal-derived Foods

The **194 veterinary drugs** were added to the matrix of pork, pig liver, chicken, duck, etc.

The results show that **the detection rate** of all compounds in the matrix was **>84%** when the concentration was **1.0 µg/kg**.

The detection rate of all compounds in the matrix was **>92%** when the concentration was **5.0 µg/kg**.

The recovery percentage of spiked concentrations of **1.0 µg/kg and 5.0 µg/kg** was **between 50 and 120%**.

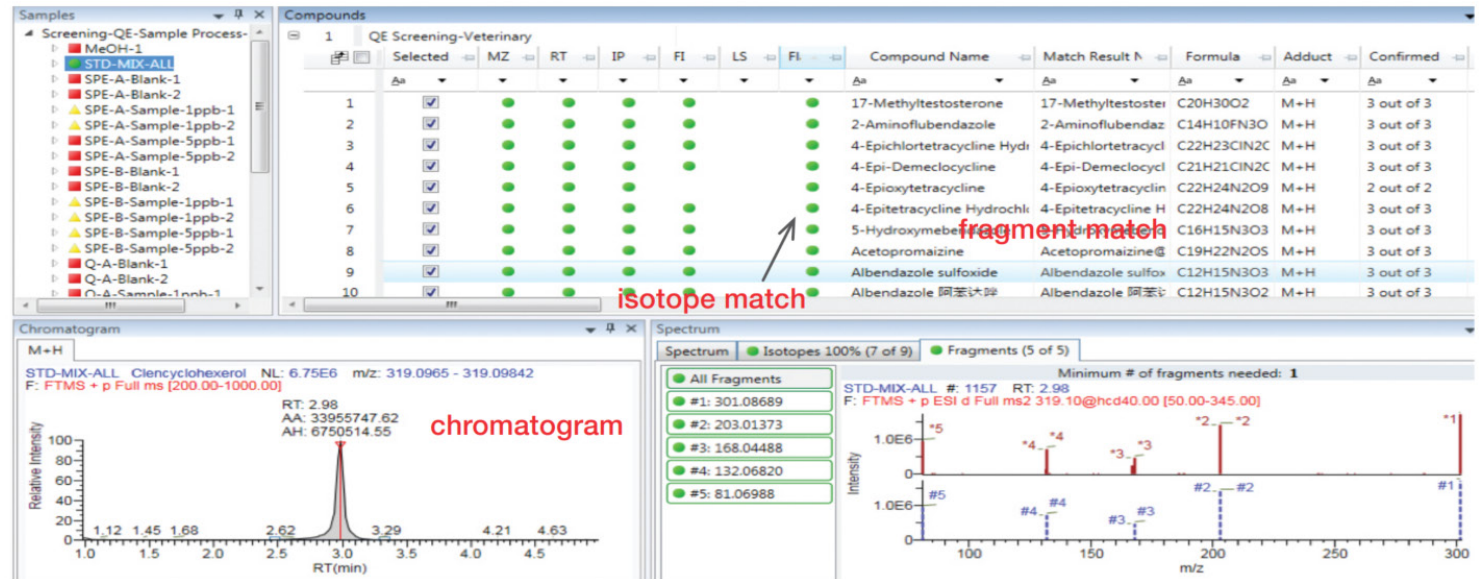


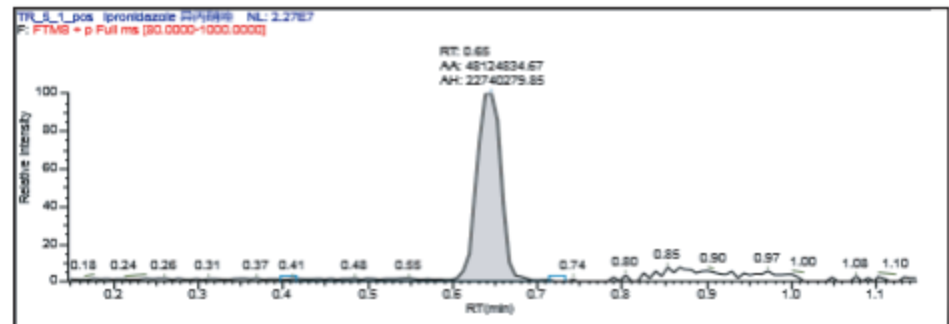
Figure 2. Screening chromatogram in TraceFinder software. A compound is confirmed with RT, isotope match, and fragment match.

Target Database:

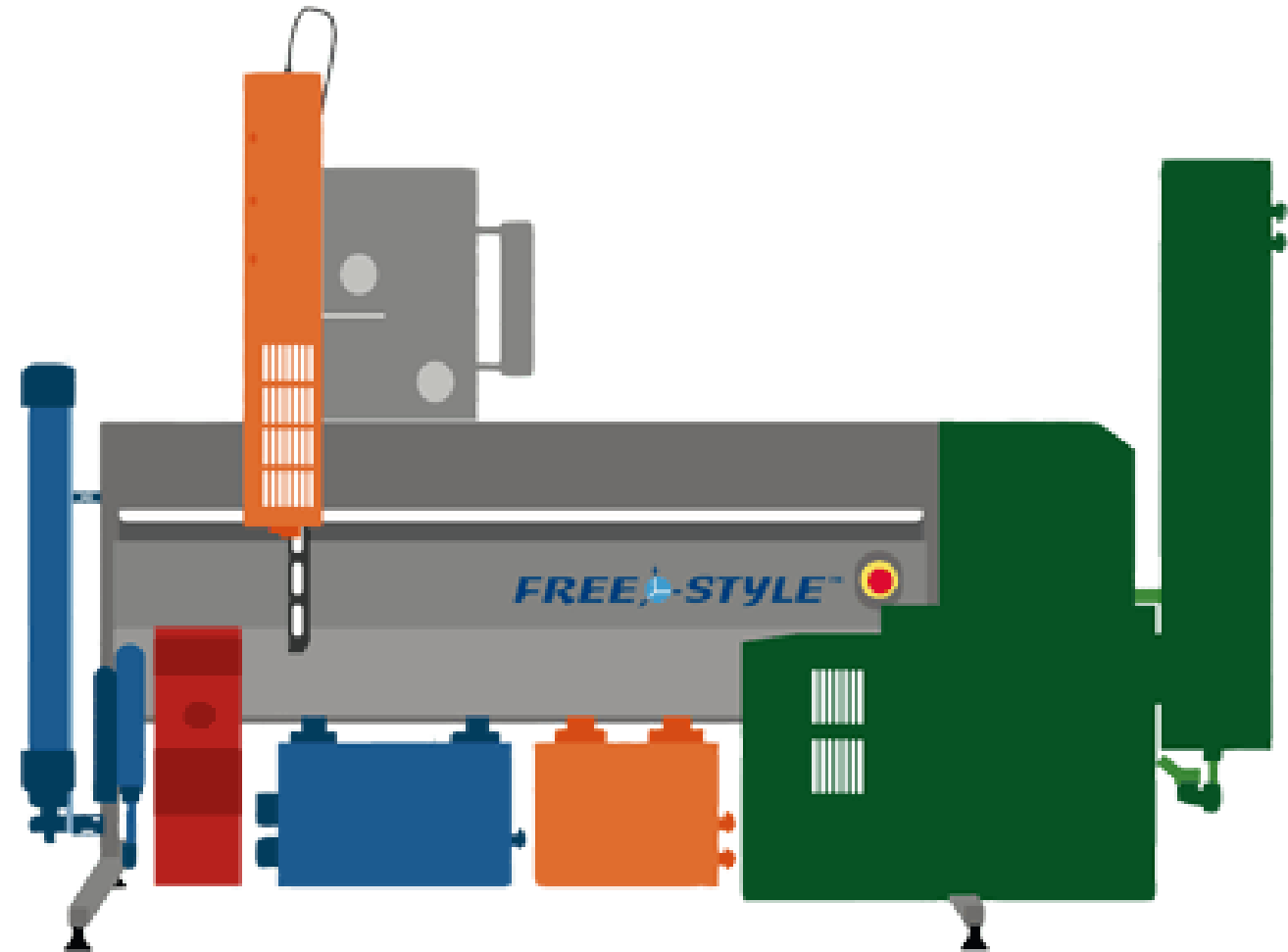
C:\Thermo\TraceFinder\3.3\General\Databases\QE-Plus_screening(200+)-0623.cdb

Iprnidazole 异丙硝唑

RT: 0.65
 Area: 4.8125E+07
 Height: 2.2740E+07
 Flag: S

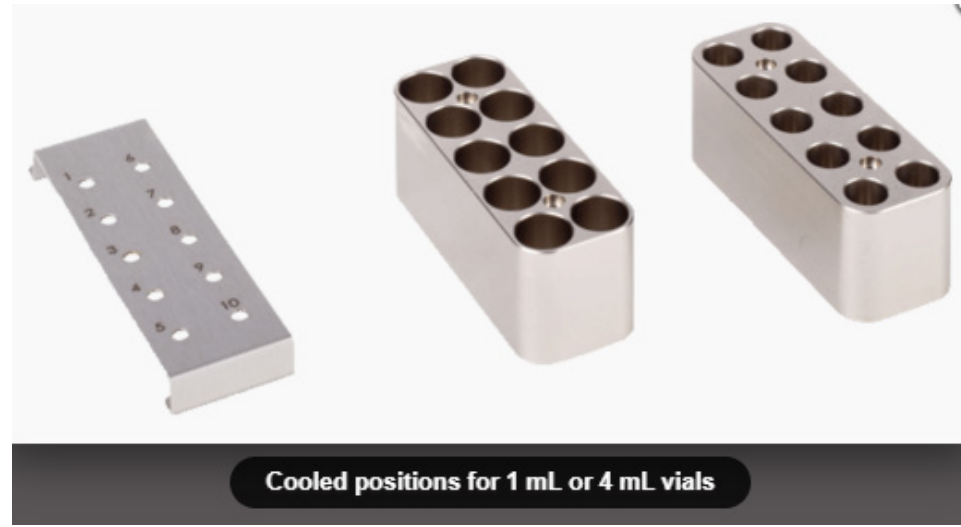


- HPLC Direct Injection Module
- QuEChERS Cleanup
- EVAporation module
- GPC Module
- FREESTYLE XANA



Flexible Configuration and Combination

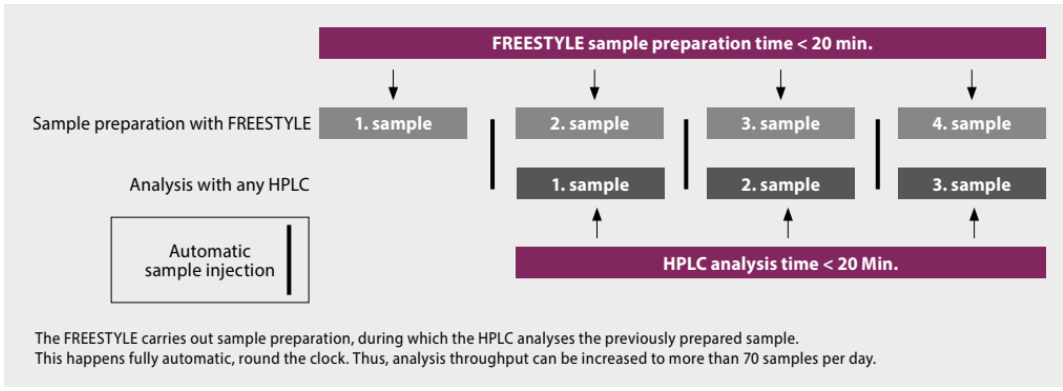
HPLC Direct Injection Module



Cooled positions for 1 mL or 4 mL vials



Direct Injection Module
(Can be mounted on the left or right side)



QuEChERS Cleanup

Unique, Specific Cartridge by LCTech



Contains a LCTech proprietary sorbent with high matrix retention



New QuEChERS

Extraction

10 g hom. sample

Add 10 mL Acetonitrile

Vortex

Add Mix I

Shake

Centrifuge

LCTech QuEChERS Cleanup

Condition SPE

Load 1 mL

Elute with 3 mL Acetonitrile

Aliquotation in vial

Measurement LC-MS/MS with HPLC-Direct Injection

Only around 10 min. run time & adaptable to analysis time in LC-MS

Fully Automated

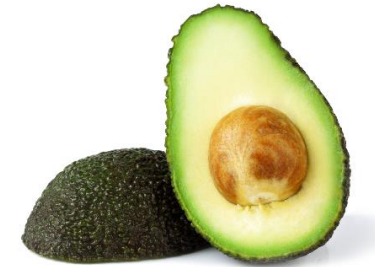
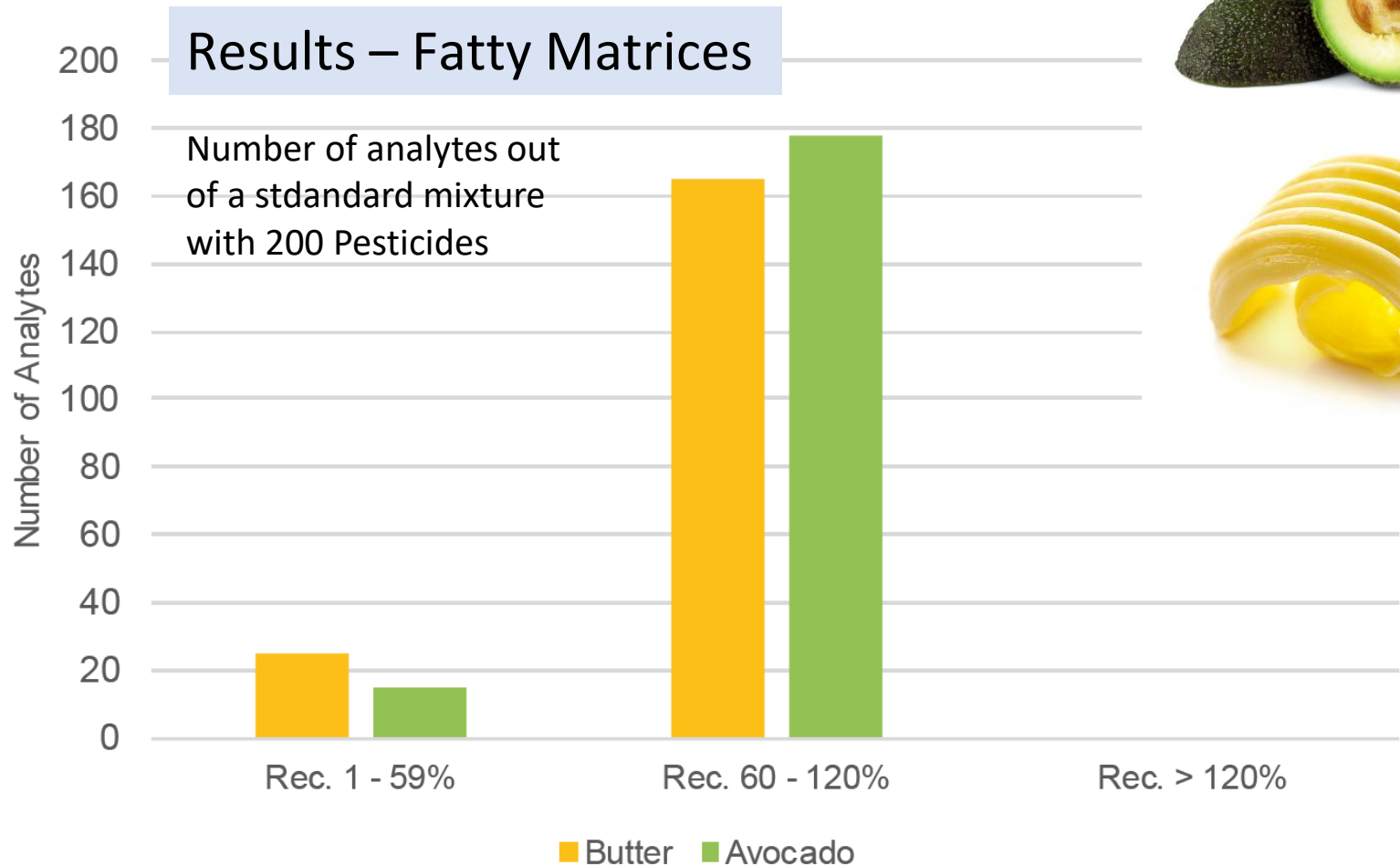




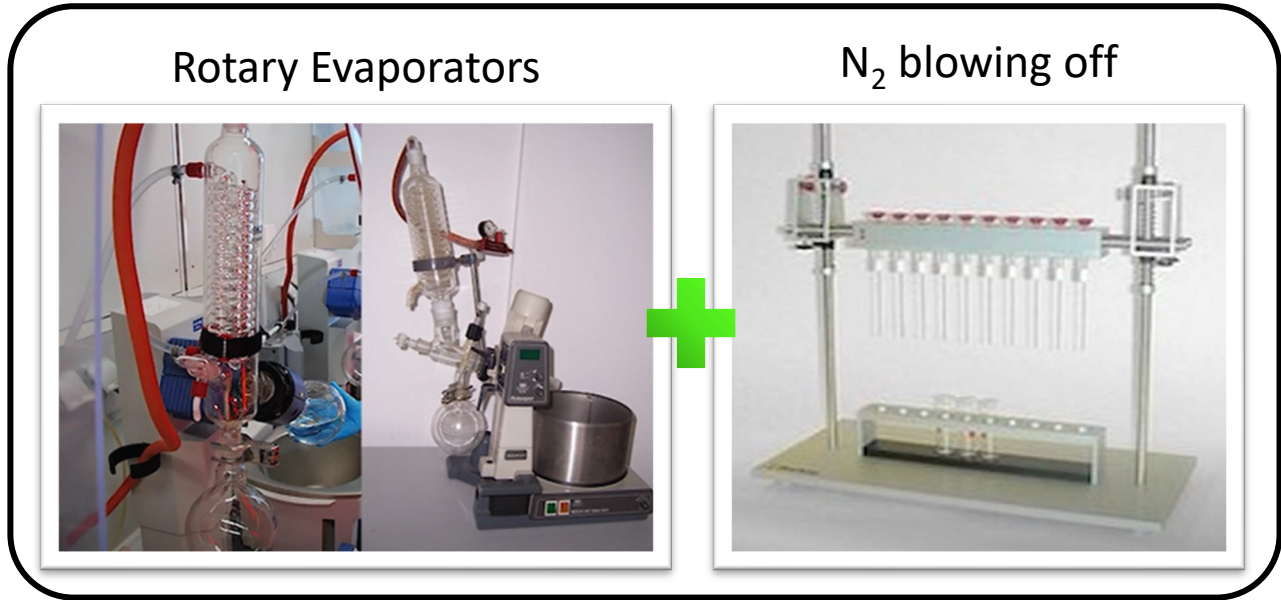
- **Left:** Extract after addition of Mix I
- **Middle:** Extract after dispersive SPE clean-up,
- **Right:** Extract after non-dispersive automated clean-up via FREESTYLE and Pesticide III QuEChERS column



Matrix Reduction with non-dispersive Pesticide III



EVAporation Module

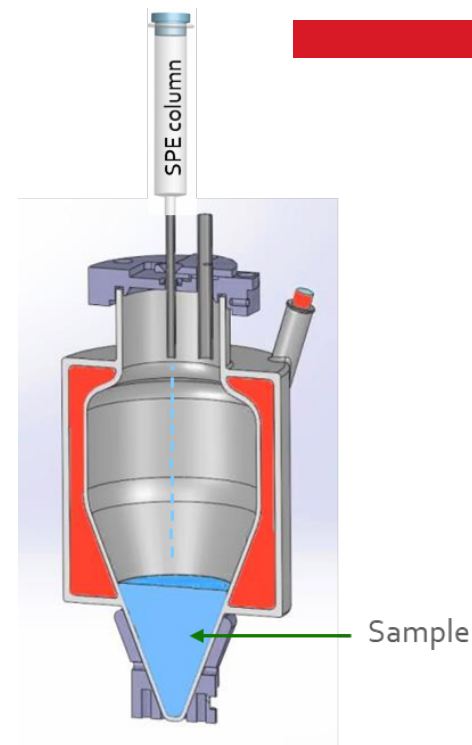
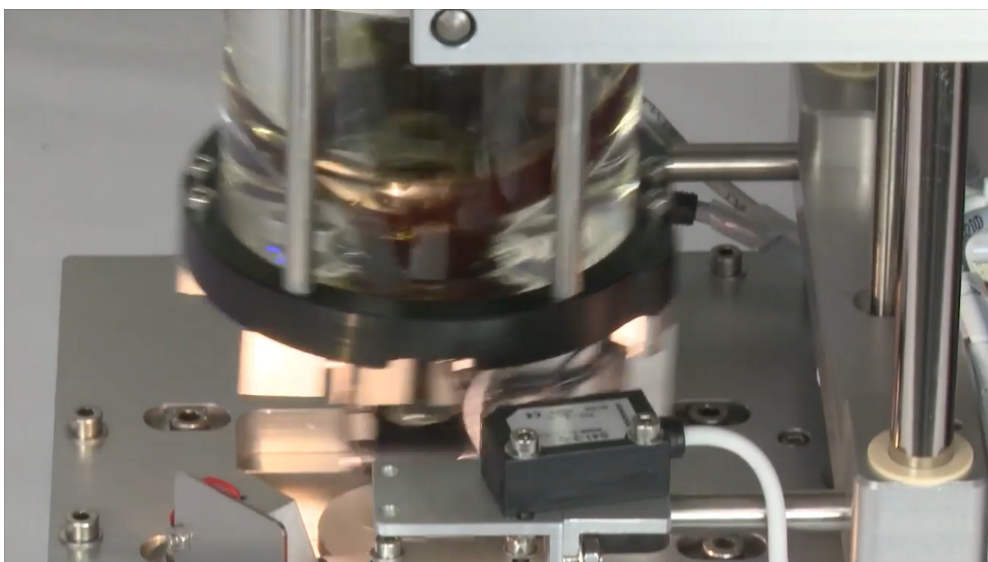


- For precise concentration of the eluate between **0.2 mL and 5.0 mL**
- For monitoring of the EVAporation process: **Addition of an internal standard** possible
- Transfer into autosampler vials, **ready to inject** for subsequent analysis
 - In standard autosampler vials
 - In autosampler vials with inserts

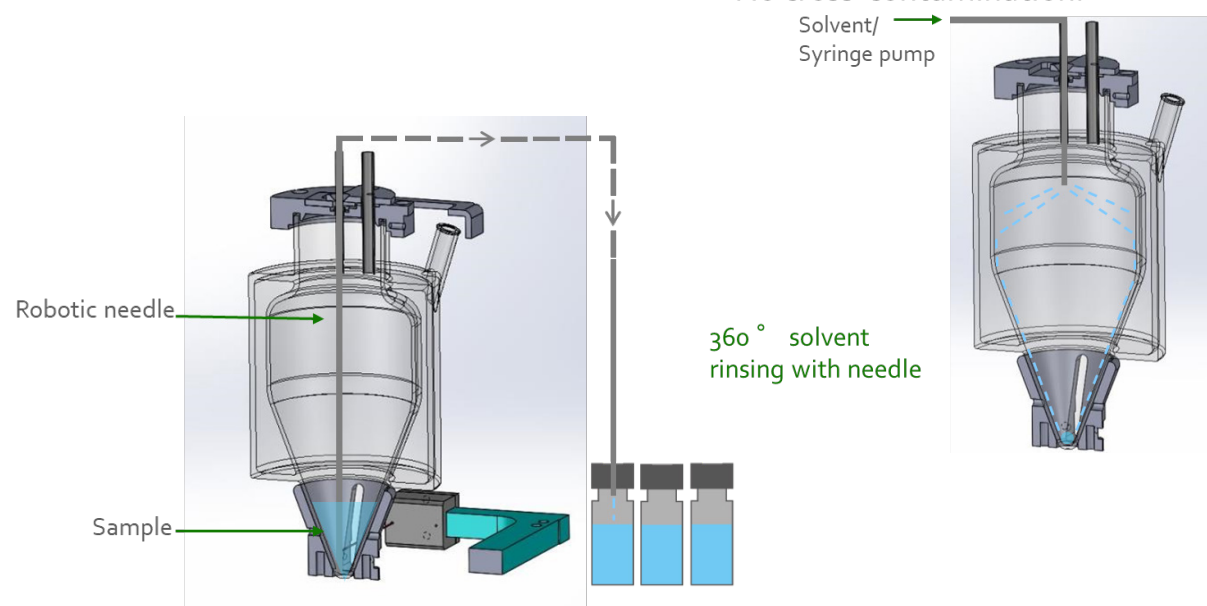
FREESTYLE EVAporation Module

Concentration of all types of organic solvents to a precise end volume between 0.2 mL and 5.0 mL

- **Sample volumes of up to 350 mL** can be concentrated in one operation
- Online connection in **combination with SPE module**
- **Solvent exchange** liquid/liquid or to dryness /uptake in a new solvent



- No cross-contamination!



Clean-up with *Gel Permeation Chromatography*

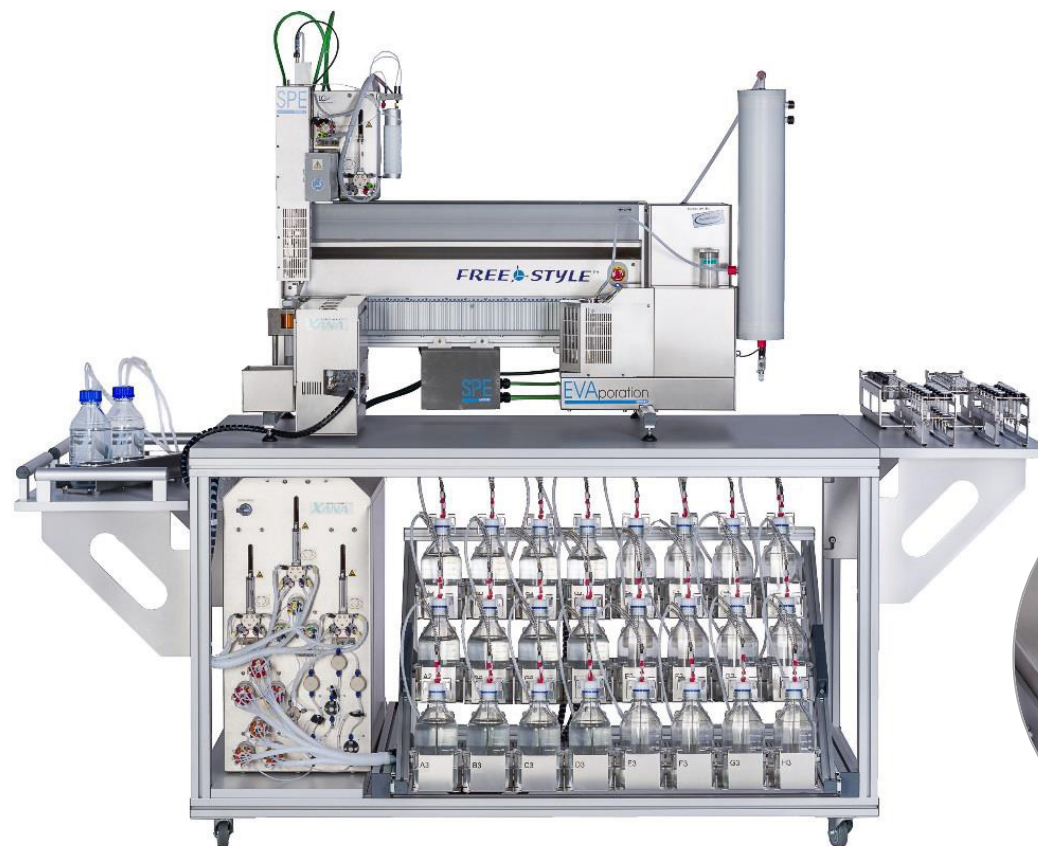
The FREESTYLE GPC is a fully automated chromatographic sample preparation system, that is used for example for clean-up of food, feed and environmental samples. It complies in all areas with the requirements of general methods, e.g. L 00.00-34, EPA 3640A.

Application Note:
Determination of PAH via
GPC/EVAporation and HPLC
Direct Injection



FREESTYLE XANA

- **Sample rack** with 24 positions for 1 L sample containers each.
- **Working station on the FREESTYLE platform:**
3 SPE columns are simultaneously being processed
- **External working station:**
Control centre of the water module including the mechanics for pumping 1-4 L samples, valves and sensors for process monitoring



*Unique Automation for
Sample Preparation in Water Analysis*





FREESTYLE – Unique System with Versatile Application

- FOOD AND FEED
- ENVIRONMENTAL
- WATER
- PHARMA
- DOPING CONTROL
- ETC.



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Automated Solid Phase Extraction (SPE)

The FREESTYLE SPE Module - An Ingenious Principle

In almost every analytical laboratory, samples are routinely cleaned via SPE columns in order to obtain clean solutions for subsequent analysis or analyte concentration. The automation is the perfect solution to simplify these routine working steps, to obtain the reproducibility of the results, and to receive good recoveries.



FREESTYLE

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Brochures

General FREESTYLE brochure (pdf | 3 MB)
FREESTYLE SPE forensics & toxicology brochure (pdf | 924 KB)

Video

FREESTYLE SPE - Automated solid phase extraction (SPE)

Application notes

Determination of PAH in particulate matter with SPE/EVaporation (pdf | 740 KB)

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Application Notes

Mycotoxin Analysis

Multi-Mycotoxin Column CrossTOX®

AN0044	Comparison of Analytical Results of Reference / QC Material Cleaned-up by CrossTOX® Column (EN)
AN0042	Multi-Mycotoxin Analysis CrossTOX® - Sample Clean-up and Matrix Removal

Please contact us

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Further application notes

Application notes for Pinnacle PCX

Applications around IAC columns in mycotoxin analysis

FREESTYLE Robotiksystem

Fully Automated Sample Preparation

FREESTYLE SPE / GPC / EVA / HPLC Direct-Injection

-	Automated Determination of THC Contents in Human Urine - SPE
AN0029	Automated Determination of Aromatic Amines from Azo Colorants According to DIN EN ISO 17234-1 - SPE/HPLC Direct-Injection
AN0027	Automated Determination of Chloramphenicol in Honey - SPE
AN0023	Automated Determination of Drugs in Human Blood - Bidirectional SPE
AN0022	Automated Determination of Drugs in Human Brain - Bidirectional SPE
AN0015	Automated Detection of Acrylamide in Water - SPE
AN0013	Automated Determination of Steroids in Urine of Food-Producing Animals - SPE
AN0008	Automated Determination of PAH in Particulate Matter PM10 - SPE/EVA
AN0007	Automated Determination of the Hydrocarbon Index (H53-Method) -

<https://www.lctech.de/en/products/robotic-system-for-automated-sample-preparation-freestyle/freestyle-spe.html>



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