Sample Preparation

Introduction

Solid Phase Extraction continues to be the fastest growing technique utilised for sample preparation. The ease of use and flexibility of SPE means that increasingly, this is the chosen pre-step adopted to clean and concentrate samples prior to analysis in HPLC, HPLC/MS, GC or GC/MS.

Advances in the analytical process are placing greater demands and expectations on sample cleaning and therefore increasing the quality required from SPE products. For this reason, polymeric sorbents with high loading capacities and spherical ultrapure silica have become widespread.

Recovery, capacity, selectivity & reproducibility are the principal sample prep. demands of todays analyst. We have developed a state-of-the-art SPE product range incorporating silica and polymer based technology **Upti-Clean™**, **Recovery™** (silica) - **Atoll™, Poly-Clean™** and **BioP™** (polymeric) push the boundaries of expectation from modern day sample preparation challenges.

Sample Preparation - Customers Overview

Clinical Laboratory
Toxicology
Analytical Chemistry
Therapeutic follow-up

Environmental
Pesticides, Dioxines, HAP, ..
Hormones, PCB's …
Water Quality
Industrial wastes

Food
Manufacturers
Distilleries
Beverage Companies
Supervision Agency

Biotechnology
Nucleic acids/proteins
Pharmaceutical Research

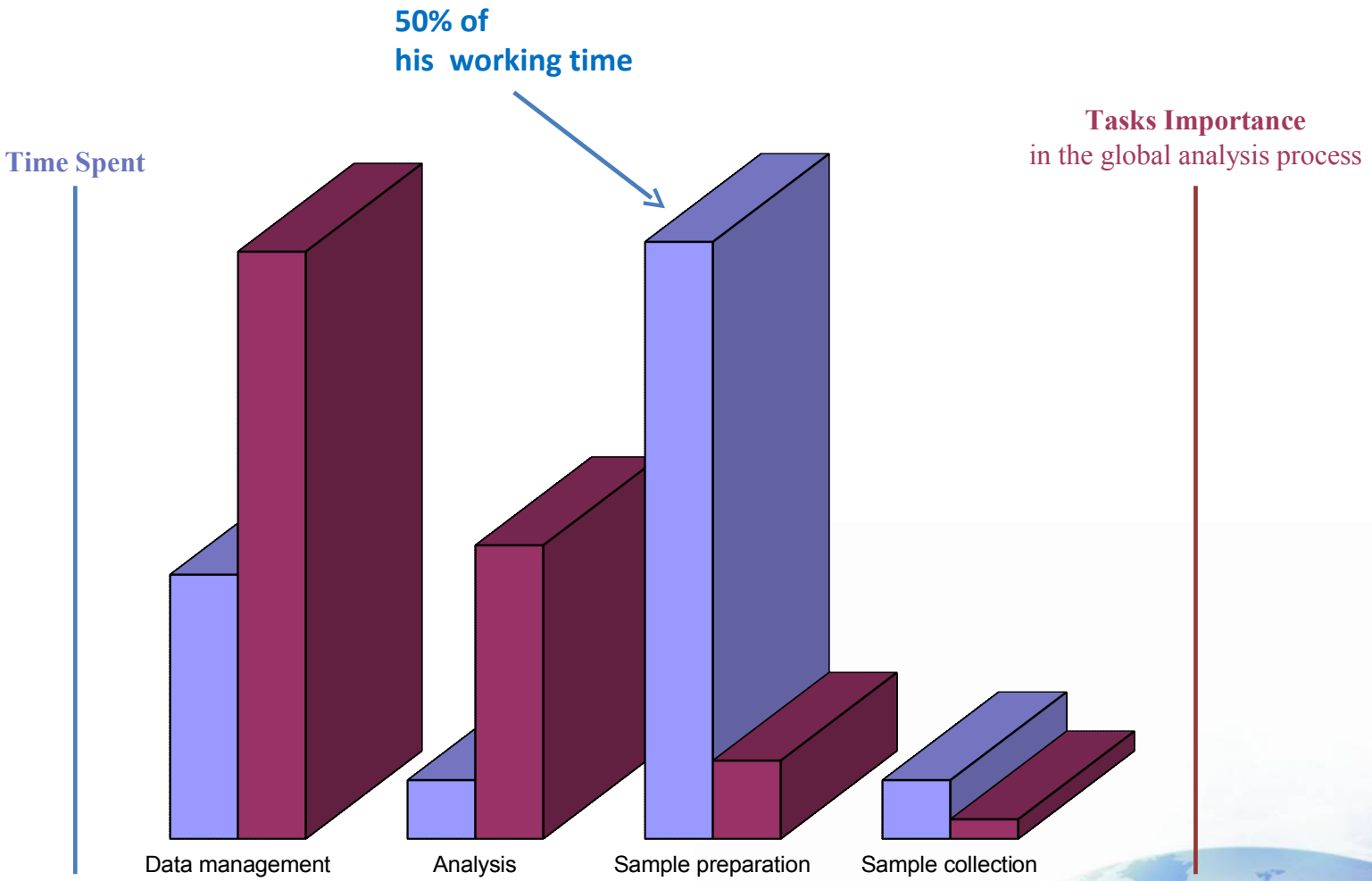
Petroleum
Petrochemical Products
Refineries
Recycling

Chemical
Hydrocarbons
Inorganics
Metals
Organics

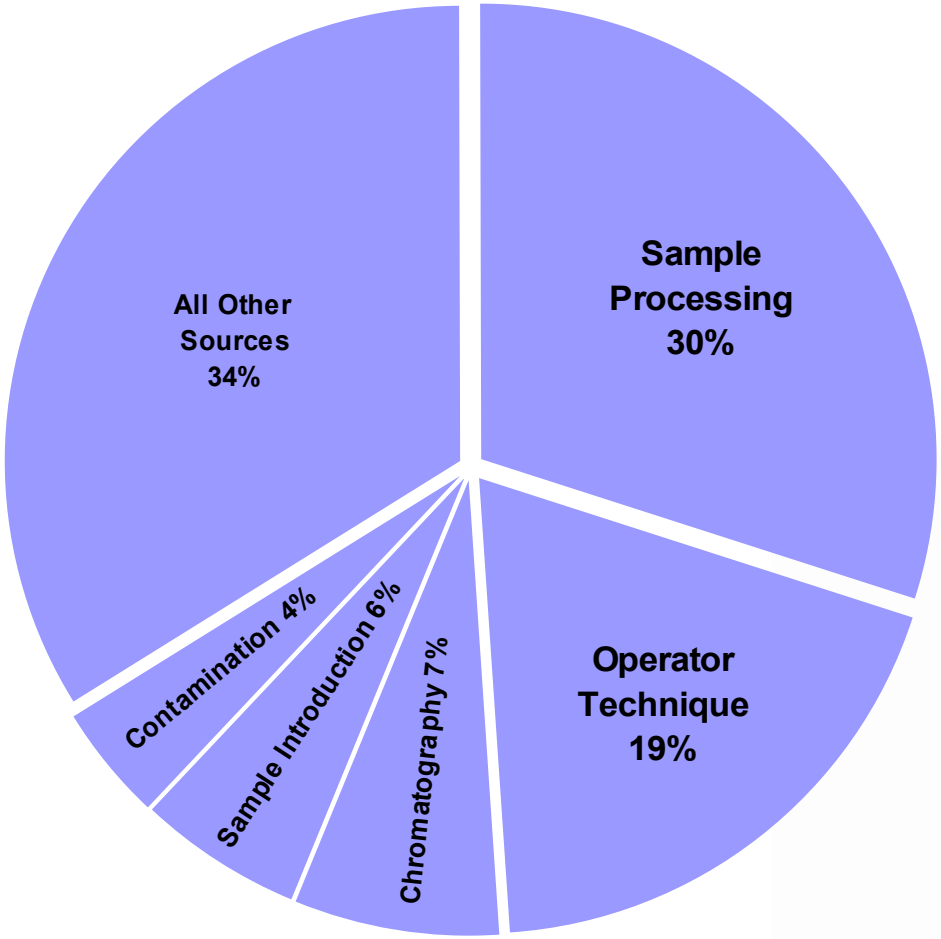
Universities
Chemistry
Environmental
Nutrition Studies

Government labs
Forensic
Food Inspection
Environmental
Drug Enforcement

Sample Preparation - Typical technician's working day



Sample Preparation - Sources of Analytical errors



The importance of sample preparation techniques within the limitation of errors & the increase of reproducibility of the analysis.

Source LC/GC Magazine

Sample Preparation - Overview of Techniques

Chemistry	Analytical Sciences	Biochromatography	Biology

Filtration - non sterile
Syringe Filters, 96 & 384 Well Filter Plates, Membranes, …

Filtration - sterile
Syringe Filters , Centrifugation, ...

Dialysis

Liq-Liq Extraction – Solid-Liq Extraction

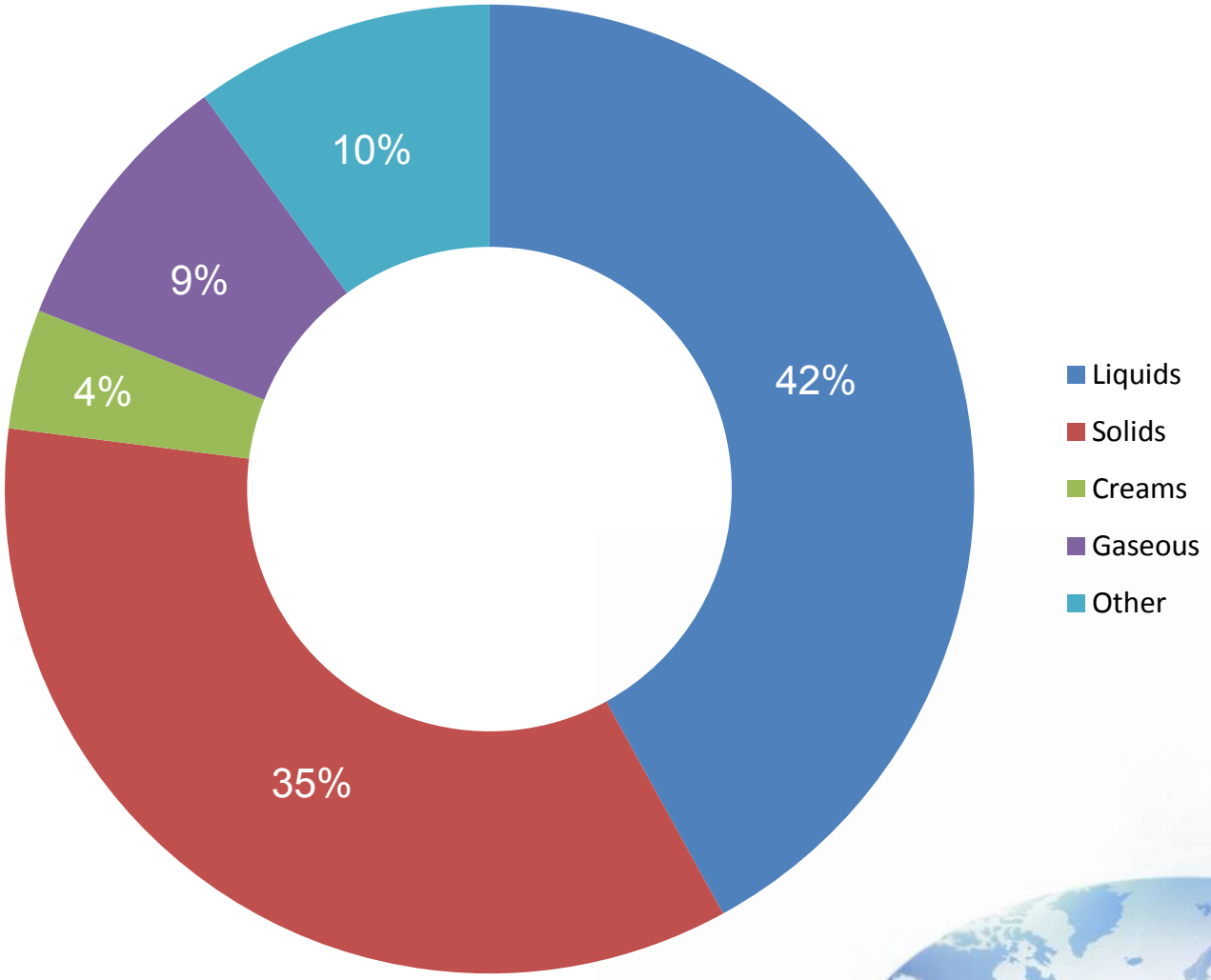
Solid Phase Extraction
Tubes, Cartridges, Disc, 48-96 well Plates, Quechers, Tips, …

Other related techniques
PSE (Pressurized Solvent Extraction), SFC (Supercritical Fluid Chromatography), Soxhlet (solvent extraction), SBSE (Stir Bar Sorptive Extraction), SPME (Solid Phase Micro Extraction), MIP (Molecular Imprinted Polymer), Immonuaffinity, SPD (Solid Phase Desorption), SPE « on-line » …

Purification
TLC, Flash columns, Scavengers, Prep LC columns

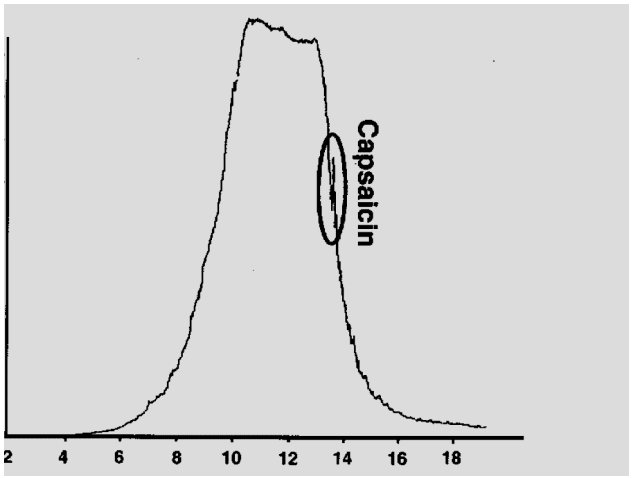
interchim

Sample Preparation - Samples Origins

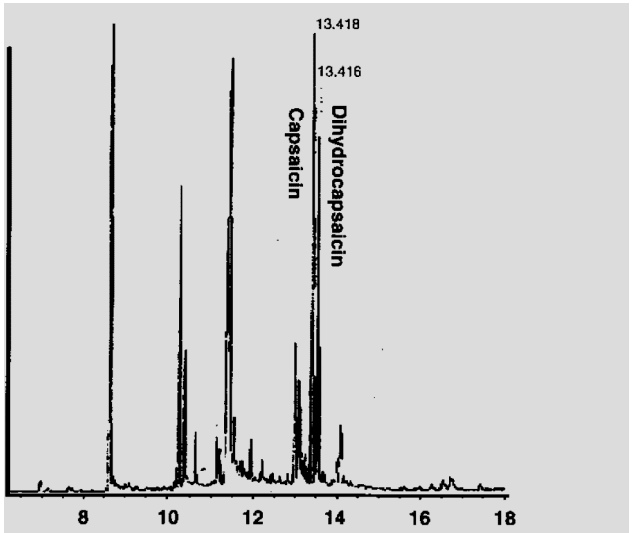


Solid Phase Extraction - Efficient & Green alternative to liq/liq ext.

Liq-Liq Extraction



Solid Phase Extraction



Better Clean-up with high recovery

No emulsion

Reduce solvent consumption

Less exposure to hazardous solvents

Waste disposal costs reduction

Source M. Telepchak , U.C.T. ; Humana Press ; Forensic & Clinical Application of SPE

interchim

Solid Phase Extraction - Efficient & Green alternative to solid/liq ext.

Better Clean-up with greater recovery

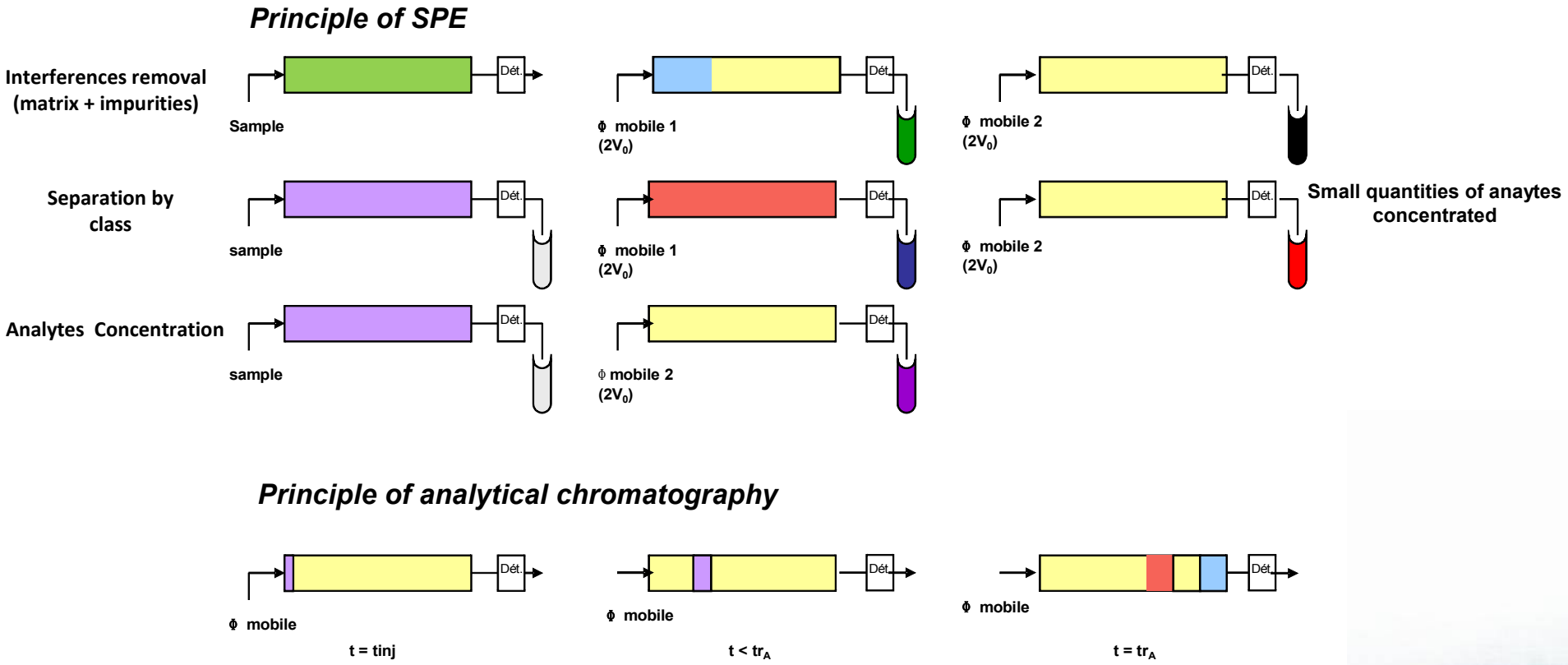
Reduce solvent consumption

Less samples degradation

Waste disposal costs reduction

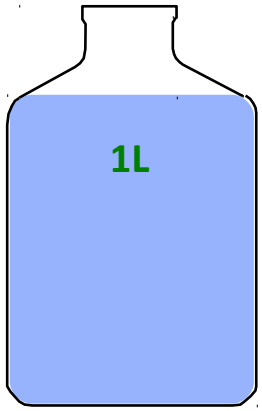
Possible Automation

Solid Phase Extraction - Objectives - Differences SPE vs. HPLC

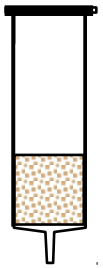


Solid Phase Extraction - Objectives

Large volume sample

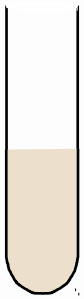


Liquide-liquide or solid phase extraction



compounds of interest fixation

Elution in a small volume



ie: 5 ml

Compounds of interest recovery

Concentration before analysis

ie: 0.1 ml

final concentration factor:

200 to 10000!

after evaporation

interchim

Solid Phase Extraction - Objectives

Recovery of clean analytes and/or re-concentration before future use:

Applications

1- Separation of analyte(s) from impurities

2- Separation by class of analytes (most difficult)

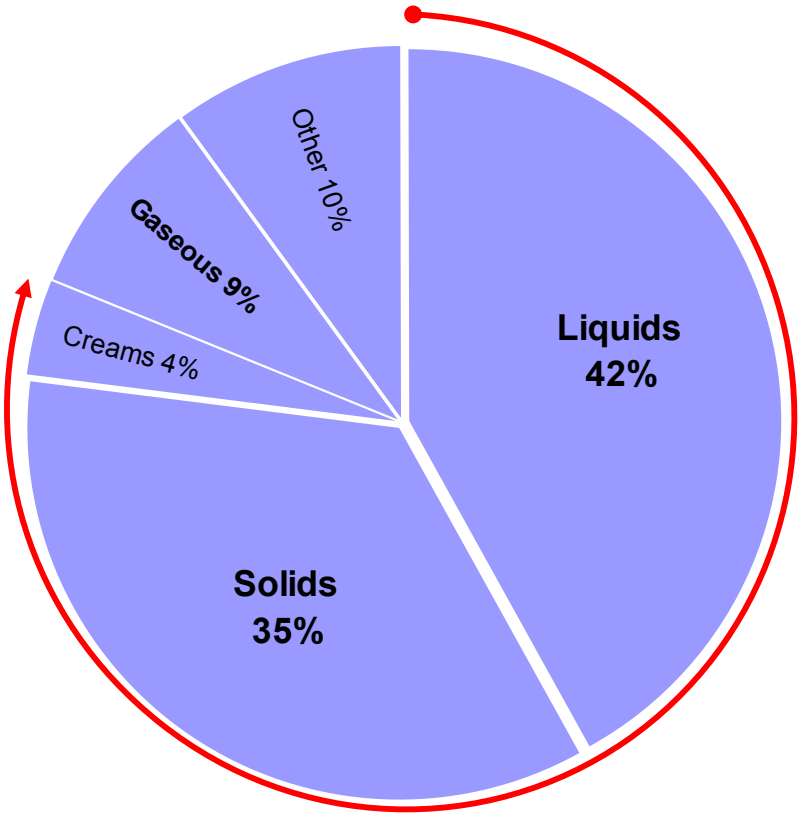
3- Separation of analyte(s) from the solvent

(concentration of analyte traces (low concentration in a large volume))

Solid Phase Extraction - finalization of the objectives

- Final purity require
- Final concentration require
- Final solvent expected
- Define objectives:
  - 1 analyte
  - 1 class of similar analytes
  - Differents classes of analytes

Solid Phase Extraction - Extraction method f^{ion} (samples origin)



The SPE technique needs a liquid sample.

About 80% of the different samples are concerns and can be treated by SPE.

Liquids samples can be pre-treated by a filtration step to remove solid particles

Solid samples need to be extracted first by PSE, SFC, Soxhlet or others or solubilize in an appropriated solvent

Solid Phase Extraction - Advantages

SPE can treat a wide range of samples from 50µl up to > 1L within a wide range of matrix.

Effective Clean-up & Concentration of dirty and dilute samples
Saves time, ensuring reproducible results vs liq/liq & solid-liq extraction

Methods can be automated

The Modern Solid Phase Extraction

The main current technique for sample preparation

Spherical Pure Sorbents perfectly reproducible, no needs to reserve columns batches

Silica & Polymer based high capacity sorbents with wide range of selectivity

Accurate Bed Technology™ : weighted sorbents (+/- 1%) to ensure reproducible and optimal recovery

Fully fits with UHPLC needs

Solid Phase Extraction - evolution vs. HPLC history

70's 80's 90's 00's 10's

Analysis Accuracy increase

HPLC particles

20 - 10 µm

10 - 5 µm

5 - 3 µm

3 - sub 2 µm

HPLC hardwares

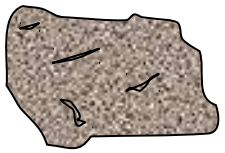
(300 - 250) mm x 4,6 mm

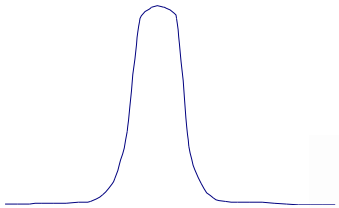
(150 - 50) mm x 4,6 - 2.1 mm

(50 - 25) mm x 4,6 - 2.1 mm

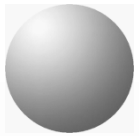
Sample prep Accuracy increase

SPE particles





IR Silica 40/63µm - 60Å, 500m²/g

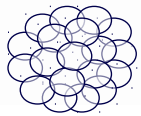


Upti-Clean™
Spherical Pure Silica 50µm - 60Å, 500m²/g

Recovery™
Spherical Ultra Pure Silica 50µm

Polymer Spheroid 200 -100µm - 800m²/g

" Low cost " Solution /competitors



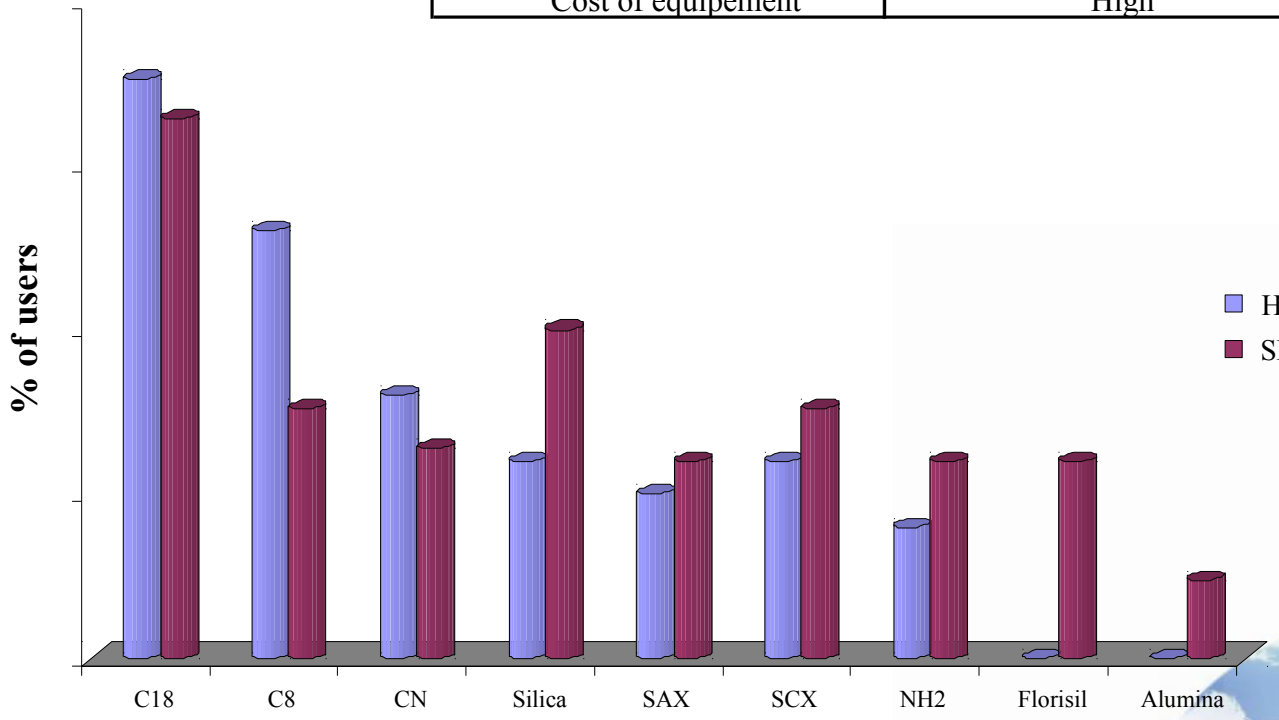
Atoll™
Spherical Pure Polymer 30 , 60& 70µm

Poly-Clean™
Spherical Pure mixed Polymer 30 , 60 µm

interchim

Solid Phase Extraction - SPE vs. HPLC

	HPLC	SPE
Hardware	Stainless steel columns	PP columns
Particle size	1.7 to 15 µm	50 µm
Shape	Spherical	Spherical - Irregular
Process	Continuous	Discontinuous
Operation	Reusable	disposable
Cost of consumable	Moderate-high	Low
Cost of equipement	High	Low



Source LC/GC Magazine

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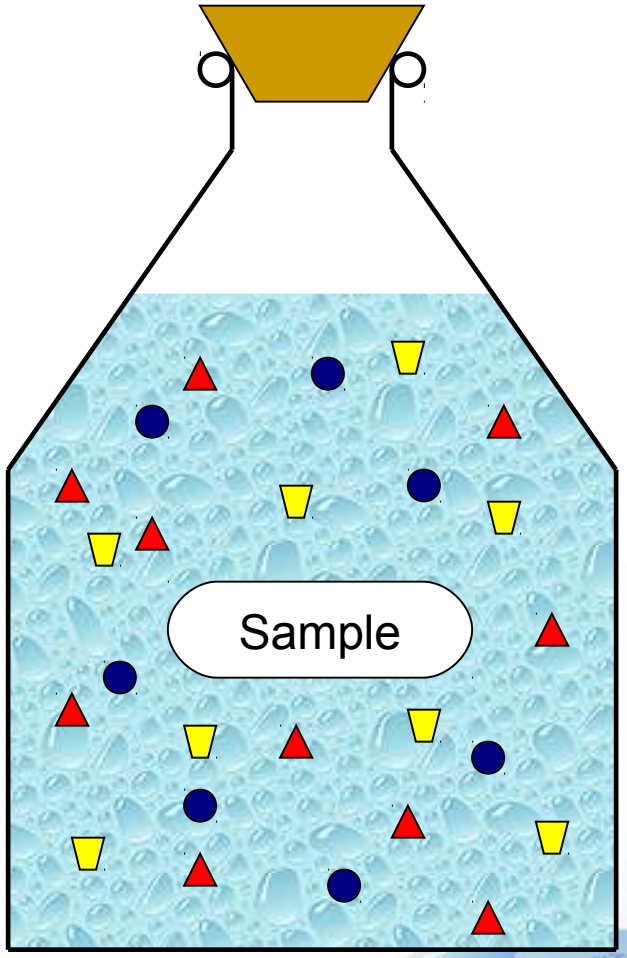
Solid Phase Extraction - Catch & Release protocol

Matrix

Compounds of interest

Impurity

Impurity



interchim

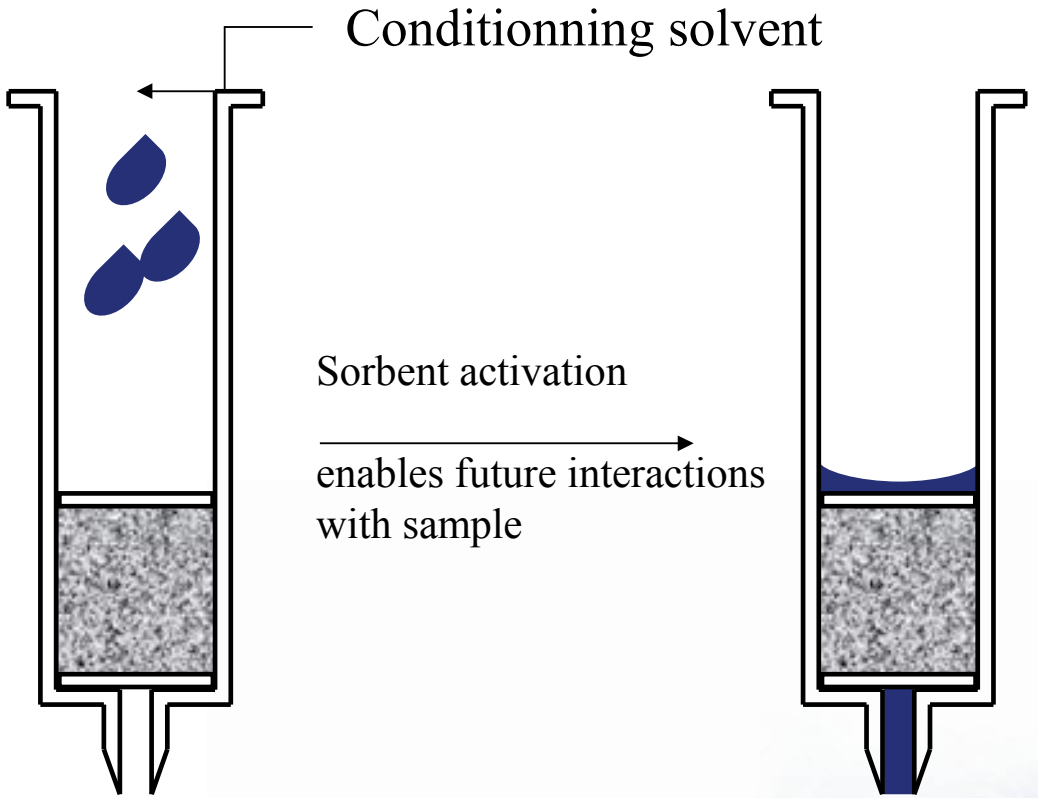
Solid Phase Extraction - Catch & Release protocol

1- Conditionning step

Sorbent activation and functional group activation are achieved by passing a volume of an appropriate solvent or a mixture of solvent, through the column.

Column frits are simultaneously solvated.

Methanol or acetonitrile are commonly used for activating hydrophobic sorbents, whilst hexane or dichloromethane activate hydrophilic sorbents. 2 to 4 bed volumes are typically recommended.



Once the activation is done never let the column dry

Solid Phase Extraction - Catch & Release protocol

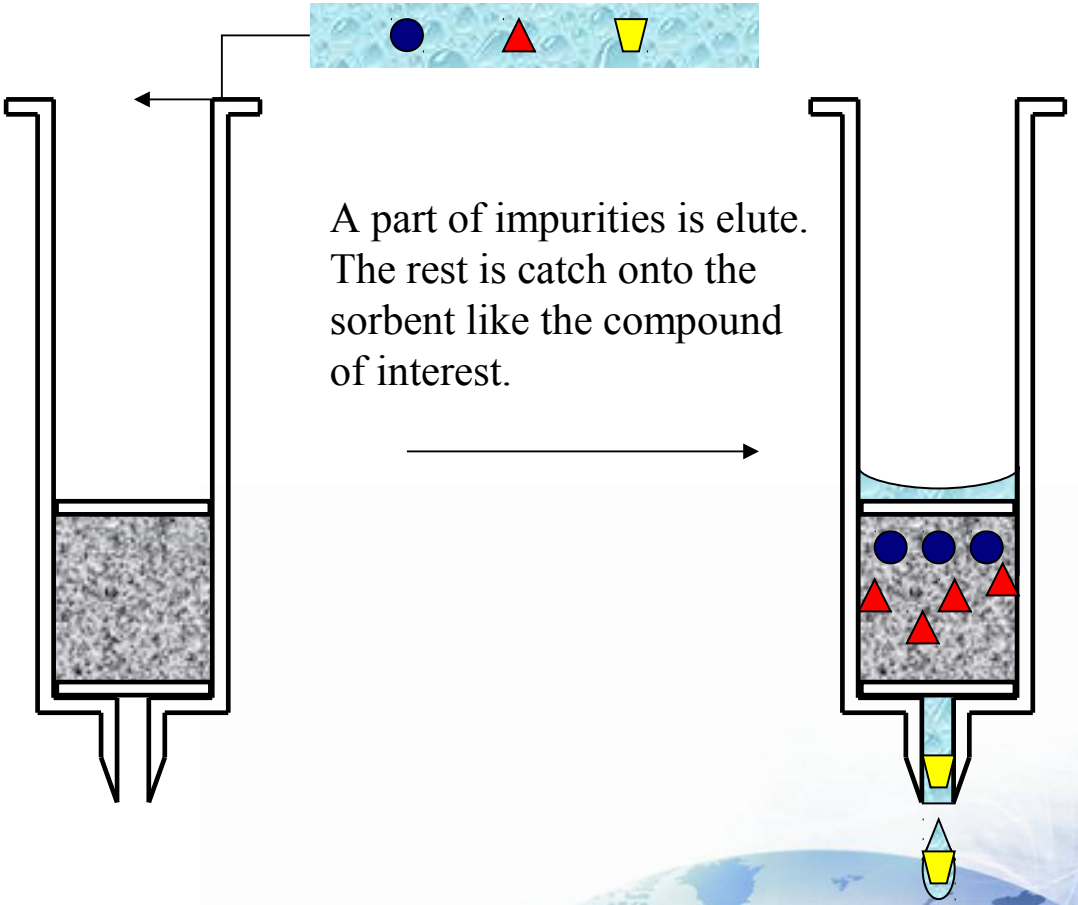
2- Sample Loading step

Apply sample onto the upper part of the sorbent bed. Matrix contaminants may pass through the column unretained, and additionally, other matrix components may be more or less strongly retained on the sorbent surface.

To get a maximum purification efficiency, the sample flow needs to be controlled.

To achieve faster flow of viscous sample through a column, 90 to 140 µm sorbents can be used. The exchange capacity and selectivity are unaffected.

[It is necessary to analyze the unretained fraction to check if all compounds of interest have been retained]



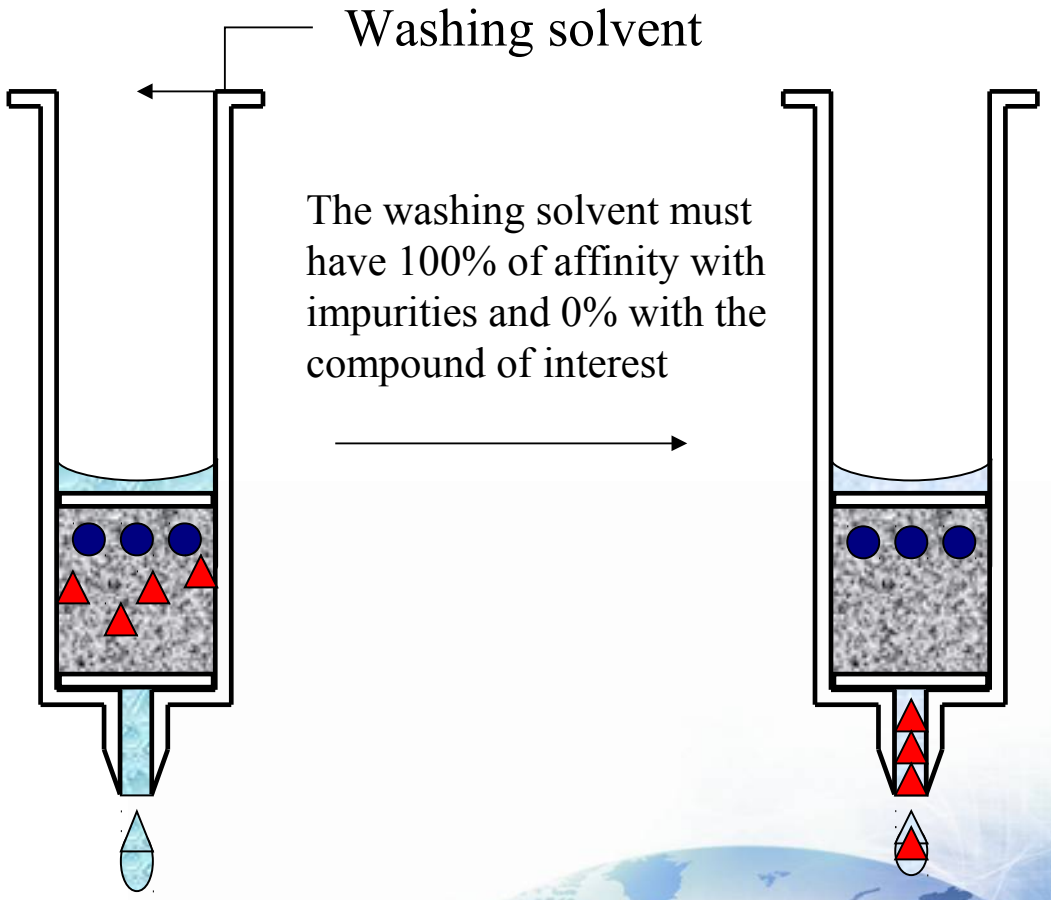
interchim

Solid Phase Extraction - Catch & Release protocol

3- Washing step

Passing solvents through columns washes away interfering compounds whilst leaving the analyte undisturbed on the sorbent bed.

Different solvents or solvent mixtures may be used to improve the rinsing efficiency.



4- Drying step

A drying step may sometimes be necessary.

Solvent traces are evaporated by circulating air through the column over a 2 to 10 minute time period. This improves the extraction yield.

Solid Phase Extraction - Catch & Release protocol

What forms is the ions in the solution with the pH ?

Ionic form		pH value of the solution				
		pKa-2	pKa-1	pKa	1+pKa	2+pKa
Acid	Anion (-)	1%	9%	50%	91%	99%
Base	Cation (+)	99%	91%	50%	9%	1%

3- Washing - Ion exchange case

Anions Exchange ⟶ pKa of the compound of interest < pH < pKa of the sorbent

Cations Exchange ⟶ pKa of the sorbent < pH < pKa of the compound of interest

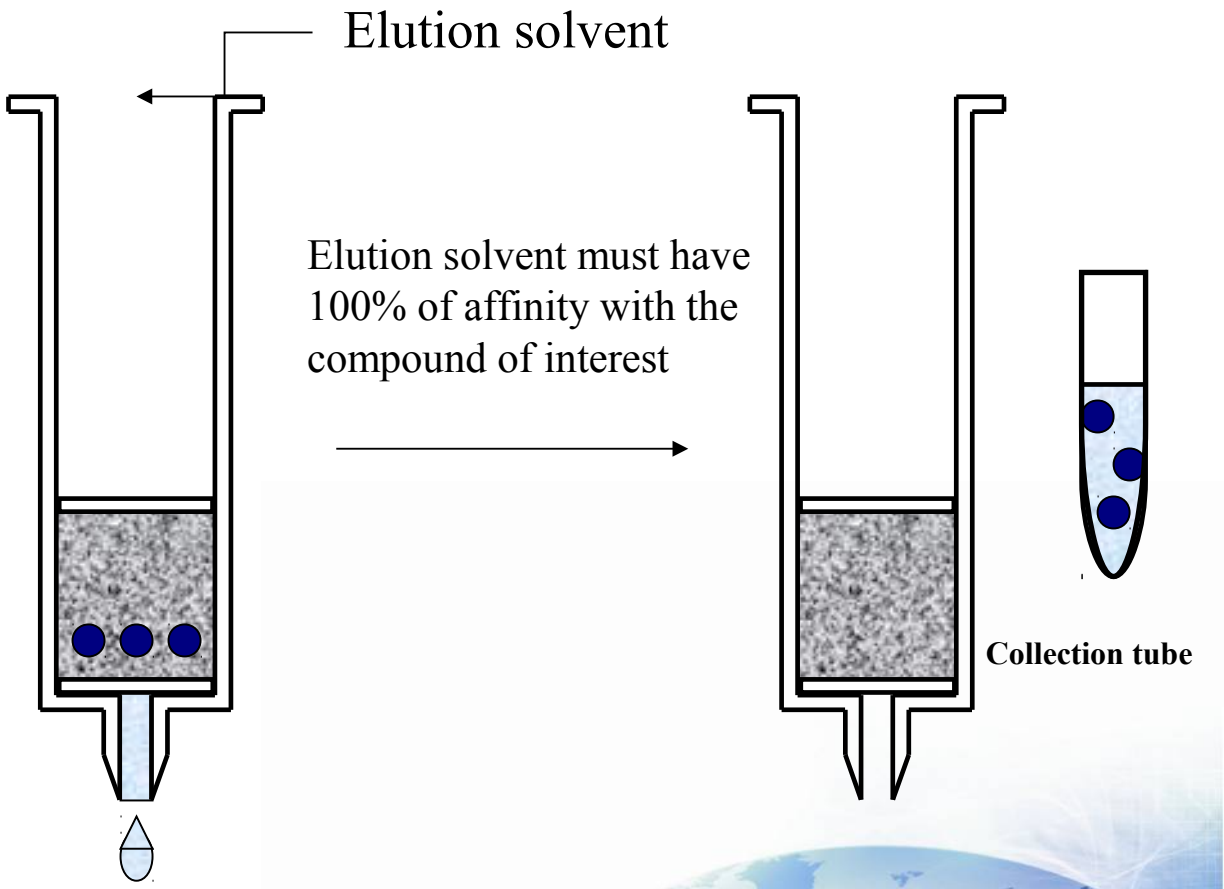
Solid Phase Extraction - Catch & Release protocol

5- Elution step

An appropriate solvent is passed through the column to disrupt the analyte-sorbent interaction and to elute 100% of compounds of interest.

The appropriate solvent must have maximum interaction with the compound of interest and a minimal interaction with the remaining impurities, leaving them undisturbed on the sorbent bed.
In addition the volume of the elution solvent needs to be as small as possible to maximise the concentration factor.

[Sorbent with low particle size (e.g 30,50 µm) gives a lower elution volume than larger sorbent particle size (e.g 90, 140 µm)].



Solid Phase Extraction - Catch & Release protocol

What forms is the ions in the solution with the pH ?

Ionic form		pH value of the solution				
		pKa-2	pKa-1	pKa	1+pKa	2+pKa
Acid	Anion (-)	1%	9%	50%	91%	99%
Base	Cation (+)	99%	91%	50%	9%	1%

5- Elution - Ion exchange case

Anions Exchange ⟶ pKa of the sorbent < pH < pKa of the compound of interest

Cations Exchange ⟶ pKa of the compound of interest < pH < pKa of the sorbent

Solid Phase Extraction - Catch & Release protocol

6- Elution step

Compounds of interest are concentrated by evaporating a part of the solvent.
If necessary, dry the eluate with anhydrous sulfate to remove possible water traces.

The concentrated sample is then ready for analysis.

We recommends that all steps should be carefully optimized according to the customer specific extraction. This will improve the quality of the final analysis.

Spherical vs. irregular

Spherical vs. Irregular silica - Customers main concerns

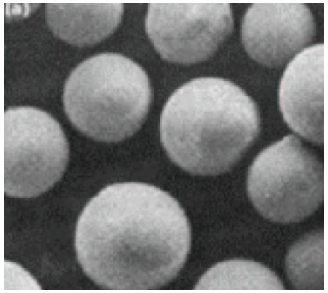
Reproducibility

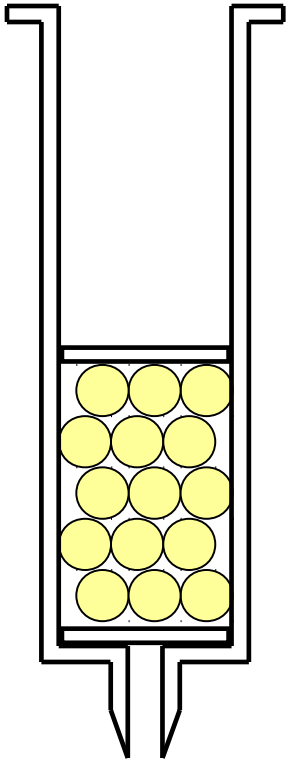
Recovery

Clogging

Find sorbents that can address Selectivity & Capacity

Spherical vs. Irregular silica - Advantages

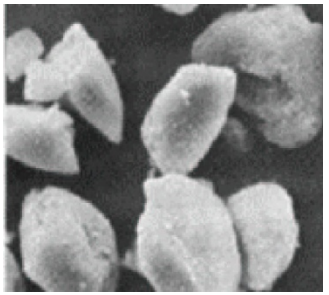


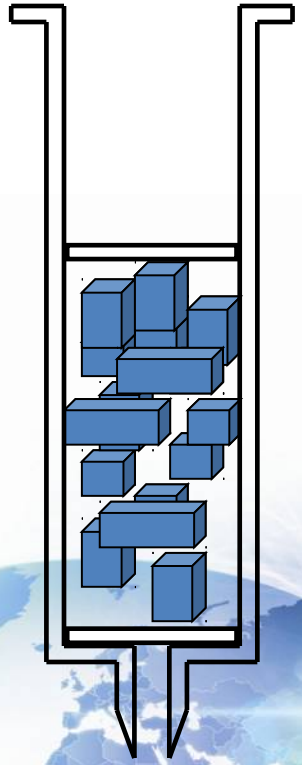


Optimum & homogeneous bed density

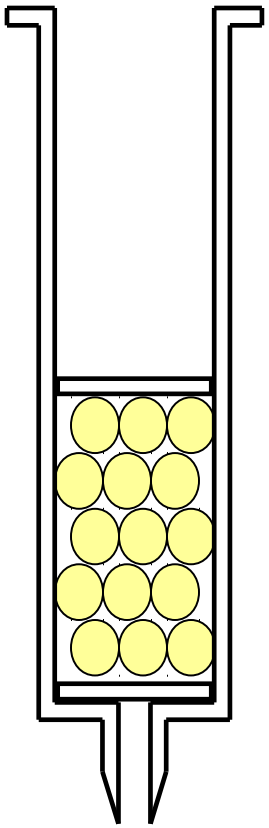
Perfect reproducibility from batch to bacth & column to column

Better sample diffusion





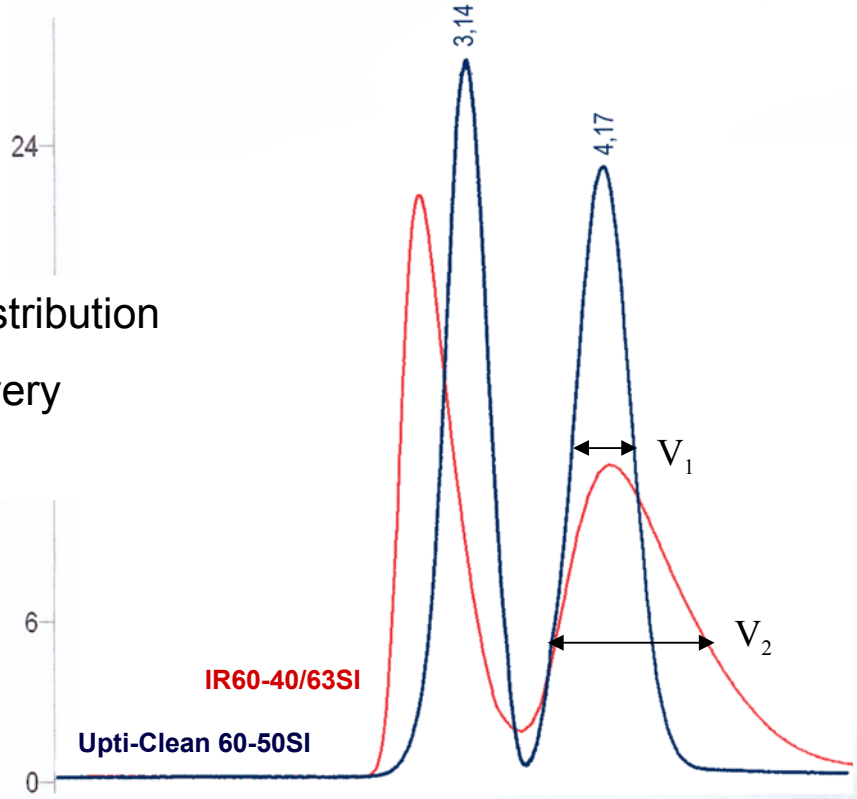
Spherical vs. Irregular silica - Advantages



Narrow particle & pore size distribution

Optimize & reproducible recovery

Lower collection volume



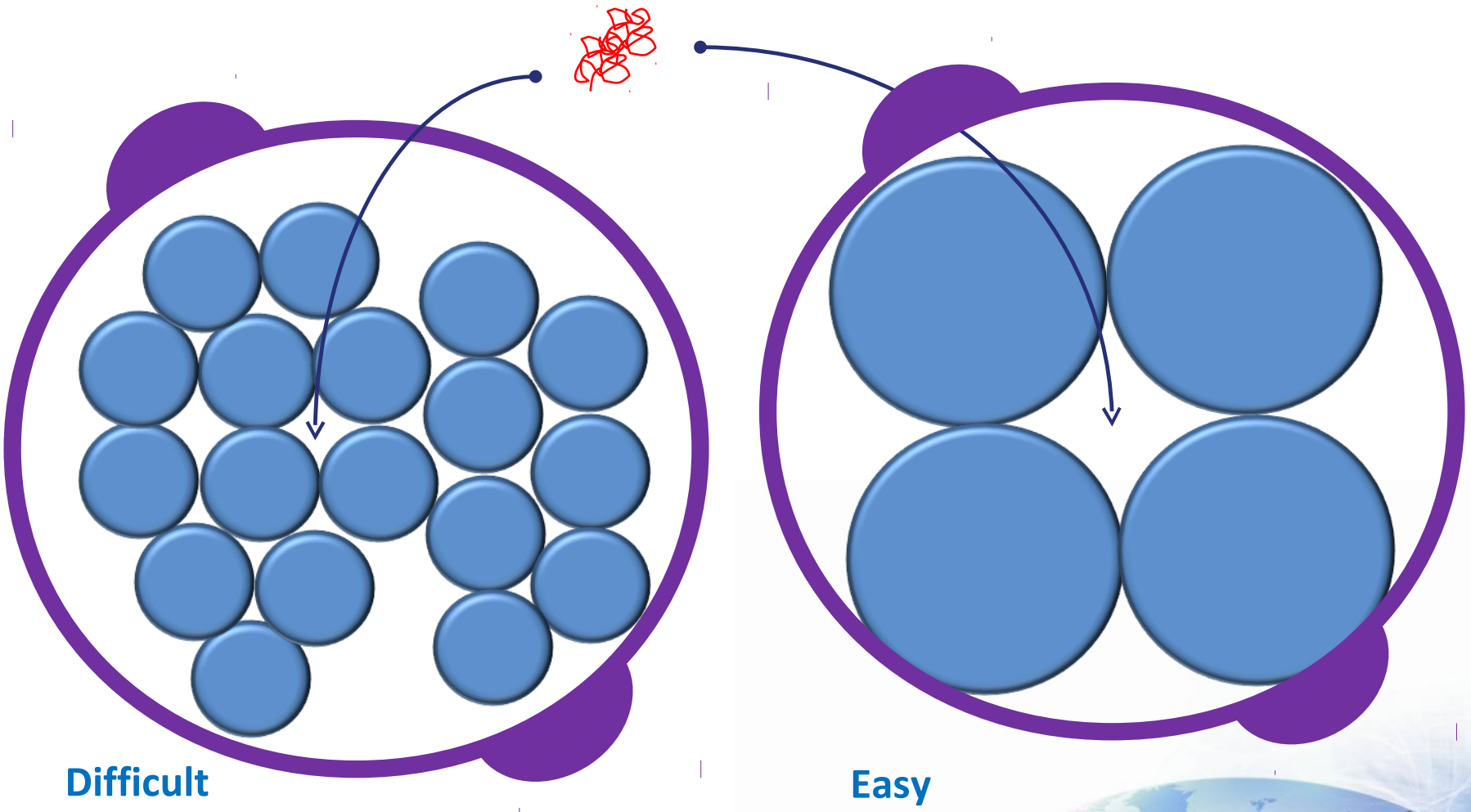
$V_1 < V_2$

250 x 4.6 mm column
Hexane - IsoPropanol (97/3)
Flow rate : 1.0 ml/min
Pressure : 1 bars
UV : 254 nm

A- Tri-Terbutylbenzene
B- Diethyl Phthalate

interchim

Spherical vs. Irregular silica - Clogging, influence of the particle size

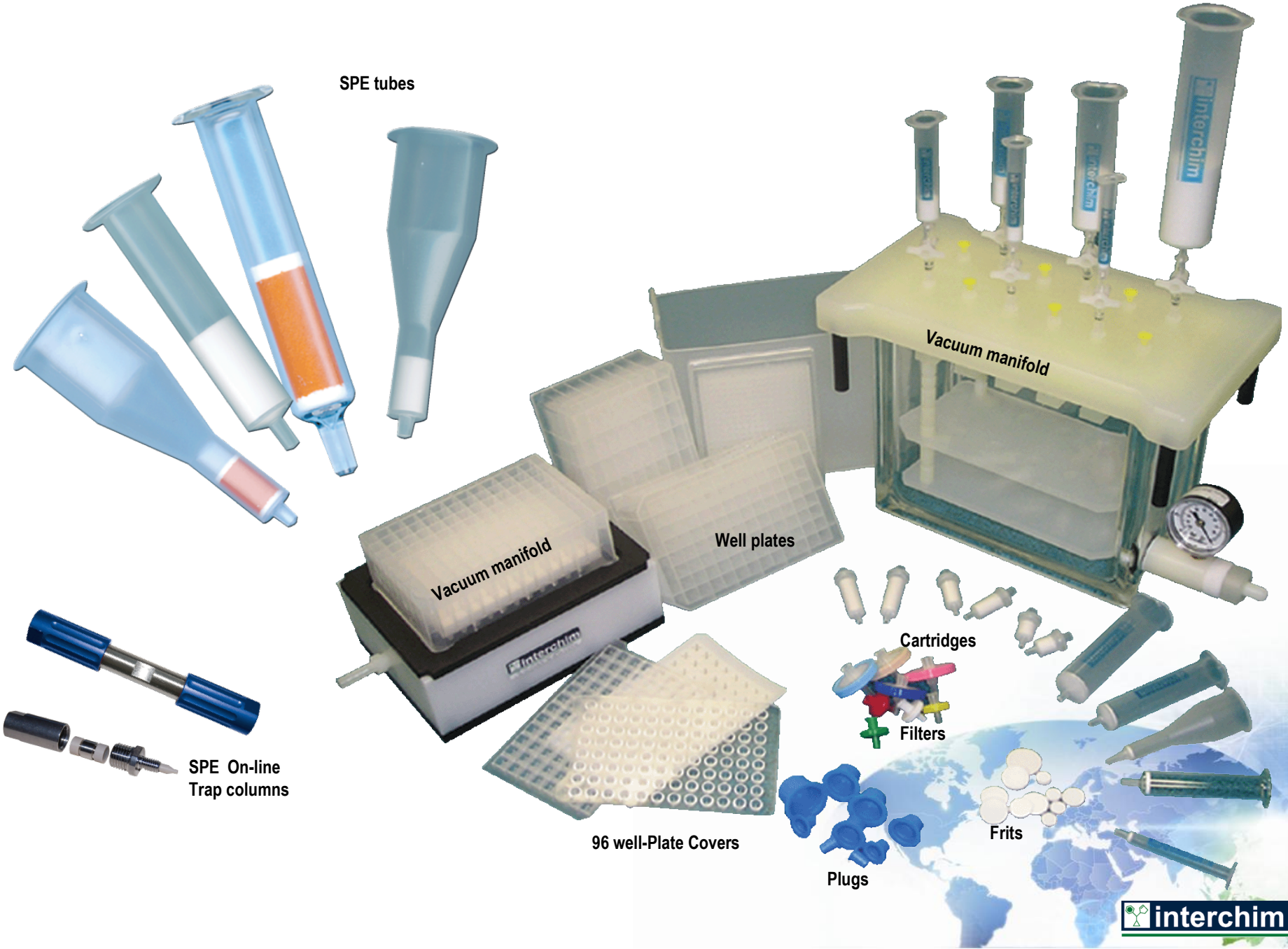


Our specific top frit reduce strongly the clogging of SPE columns.

interchim

SPE tools

SPE tools - overview

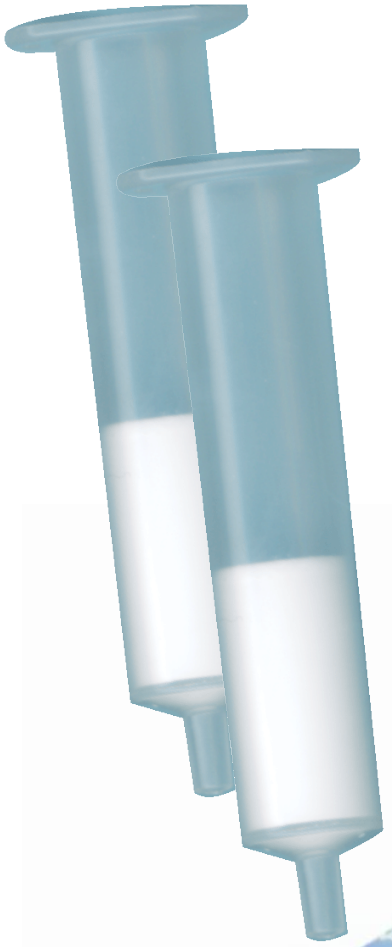


SPE tools - tubes format

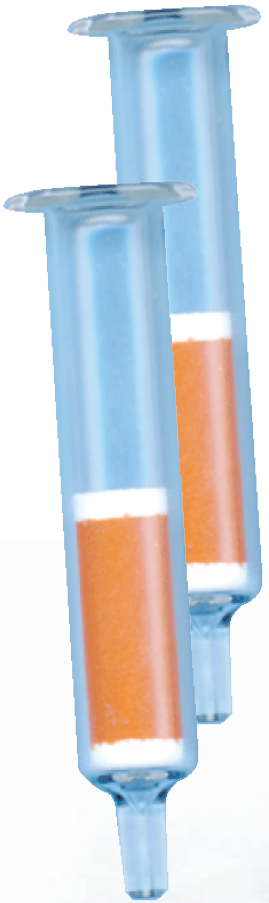
PP - LRC columns

PP cartridges

PP columns

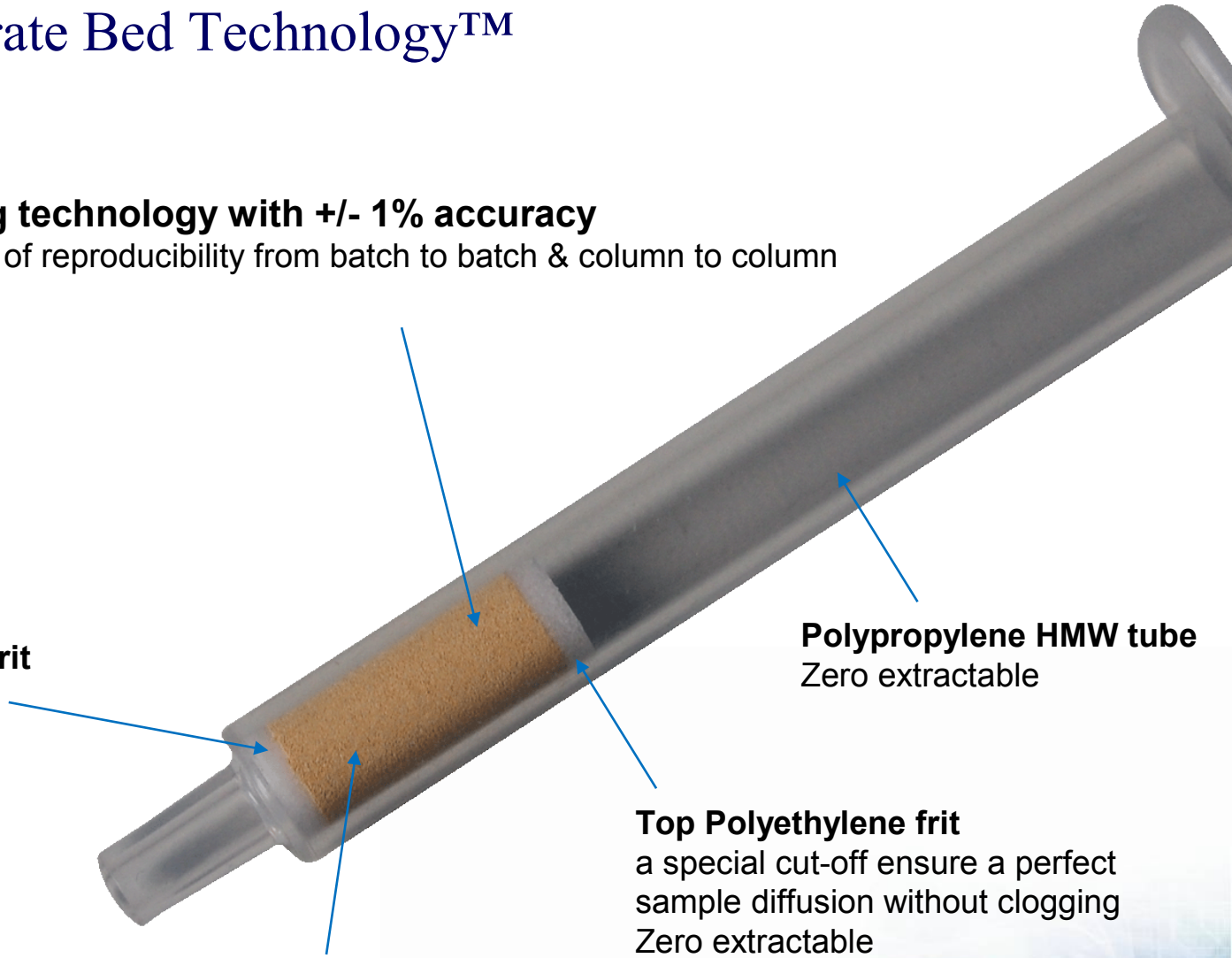


6 ml - Glass columns



SPE tools - Accurate Bed Technology™

Weighing technology with +/- 1% accuracy
Guarantee of reproducibility from batch to batch & column to column



Bottom Polyethylene frit
Zero extractable

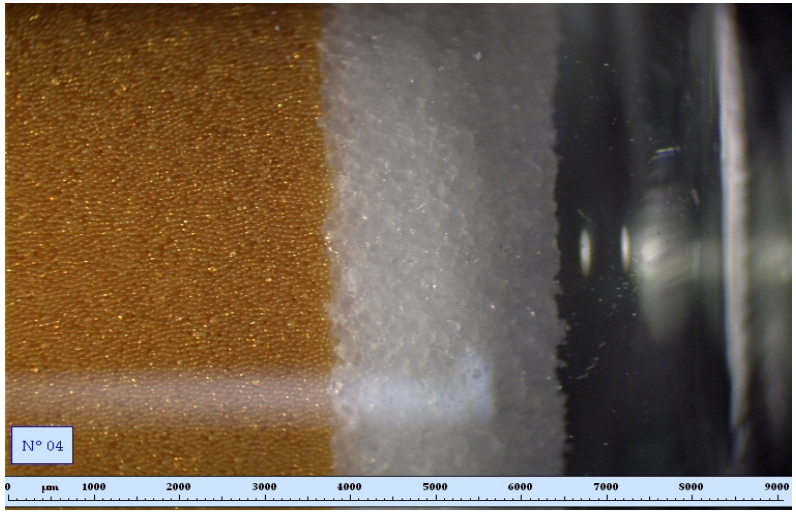
Polypropylene HMW tube
Zero extractable

Top Polyethylene frit
a special cut-off ensure a perfect sample diffusion without clogging
Zero extractable

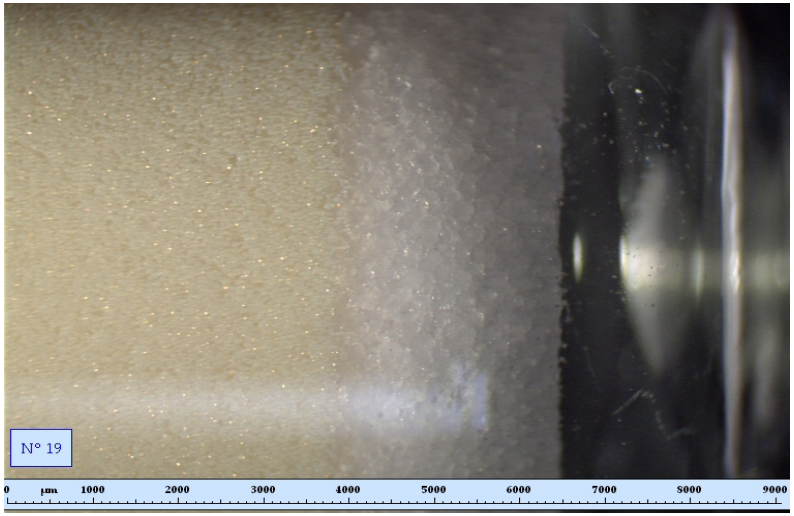
Optimum bed density

All columns are thoroughly quality control tested in-house to guarantee tracability. Products are supplied with an individual certificate detailing the specific production number and sorbent batch.

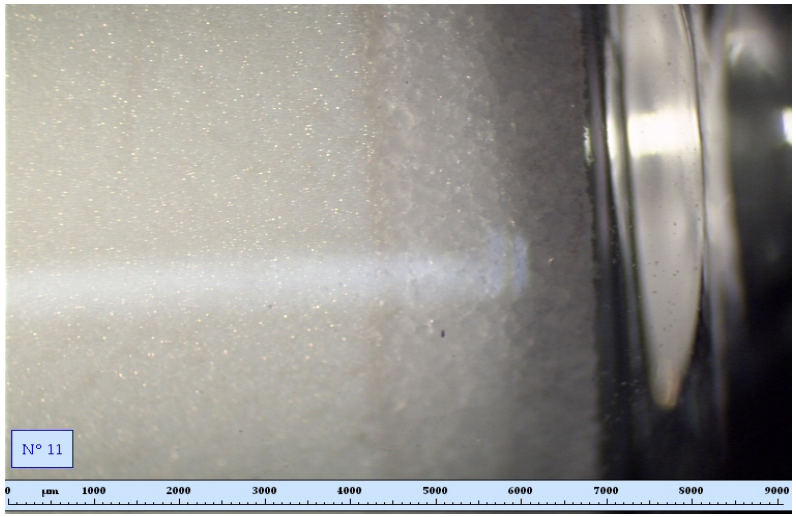
SPE tools - Accurate Bed Technology™ vs. competitors



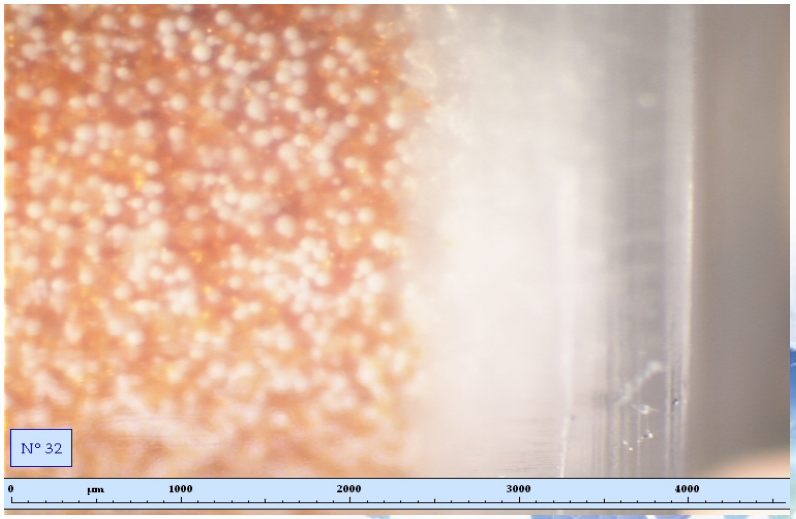
Atoll XC



Competitor 1



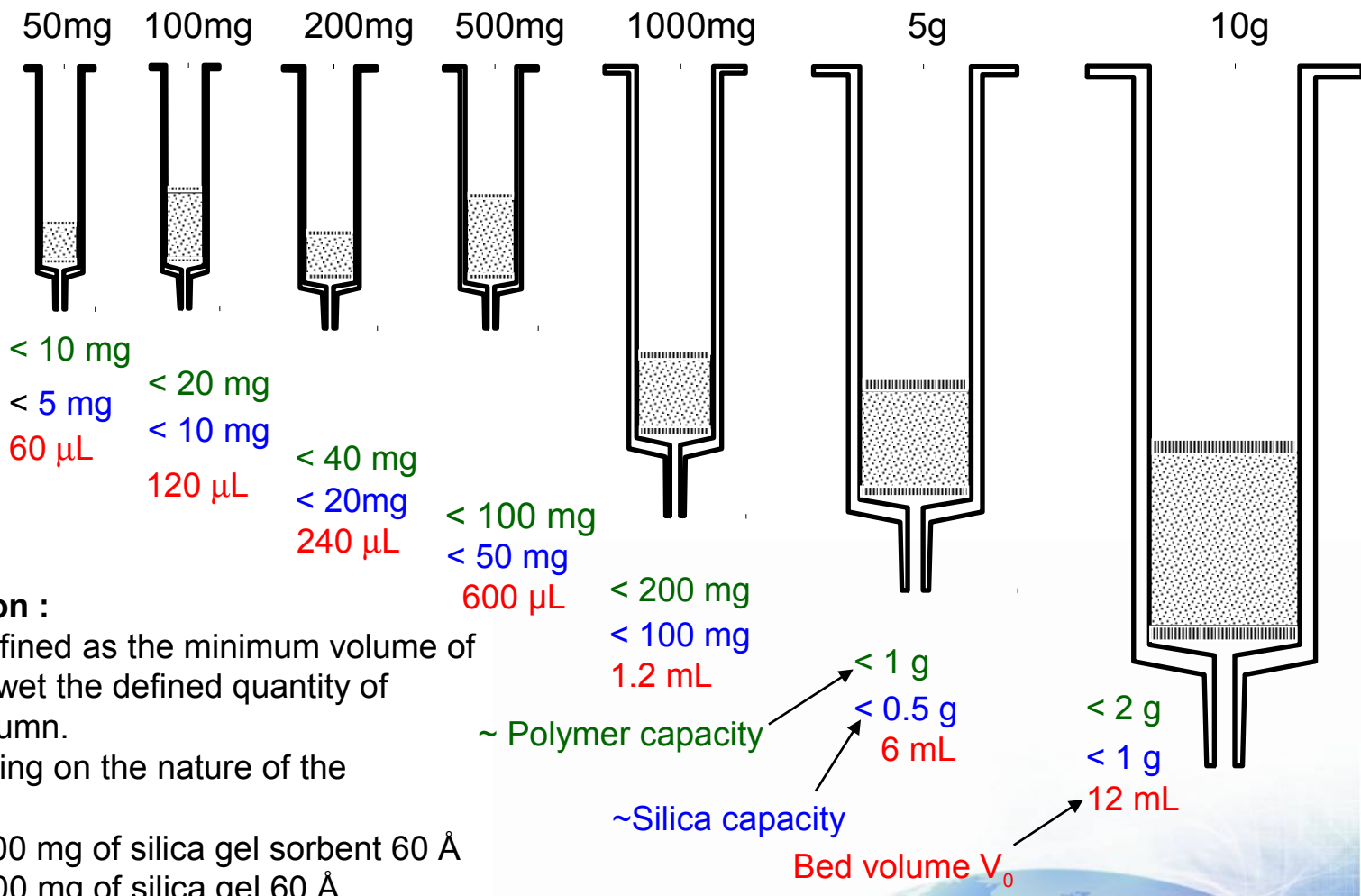
Poly-Clean 2H



Competitor 2

interchim

SPE tools - Bed volume & loading capacity



Bed volume definition :

The bed volume is defined as the minimum volume of solvent necessary to wet the defined quantity of sorbent within the column.

This can vary depending on the nature of the sorbent.

e.g. : ~ 120 µl per 100 mg of silica gel sorbent 60 Å
~ 600 µl per 500 mg of silica gel 60 Å

[Incomplete elution of compound of interest will occur if the sorbent mass is too large for the volume of solvent used.
Incomplete retention of compounds of interest will occur if there is an inadequate sorbent mass leading to compound eluting in the fraction or in the washing solvent. Such cases lead to lower recovery rates].

SPE tools - columns availability

Tubes packed with silica		
	Phase mg/tube	Column volume
Regular Tube /PE frits	50	1ml
Regular Tube /PE frits	100	1ml
Regular Tube /PE frits	100	3ml
Regular Tube /PE frits	200	3ml
Regular Tube /PE frits	500	3ml
Regular Tube /PE frits	500	6ml
Regular Tube /PE frits	1000	6ml
Regular Tube /PE frits	2000	6ml
Regular Tube /PE frits	2000	15ml
Regular Tube /PE frits	2000	25ml
LRC Tube /PE frits	100	LRC
LRC Tube /PE frits	200	LRC
LRC Tube /PE frits	500	LRC
Glass Tube /PTFE frits	200	6ml
Glass Tube /PTFE frits	500	6ml
Glass Tube /PTFE frits	1000	6ml

Tubes packed with polymer		
	Phase mg/tube	Column volume
Regular Tube /PE frits	30	1ml
Regular Tube /PE frits	100	1ml
Regular Tube /PE frits	30	3ml
Regular Tube /PE frits	60	3ml
Regular Tube /PE frits	100	3ml
Regular Tube /PE frits	200	3ml
Regular Tube /PE frits	150	6ml
Regular Tube /PE frits	200	6ml
Regular Tube /PE frits	500	6ml
Regular Tube /PE frits	500	15ml
Regular Tube /PE frits	1000	15ml
Regular Tube /PE frits	1000	25ml
LRC Tube /PE frits	30	LRC
LRC Tube /PE frits	60	LRC
Glass Tube /PTFE frits	200	6ml

Sorbent selection

Sorbent selection requires consideration of sample volume, the nature of the analyte, analyte concentration and the inherent properties of the sorbent itself.

Silicas & Polymers are the most popular sorbents used for Solid Phase Extraction. Polymer loading capacities are higher than silica sorbents, however, silica sorbents exhibit greater selectivity. Sorbent features are highlighted below and provide the analyst with an initial consideration for appropriate sorbent selection.

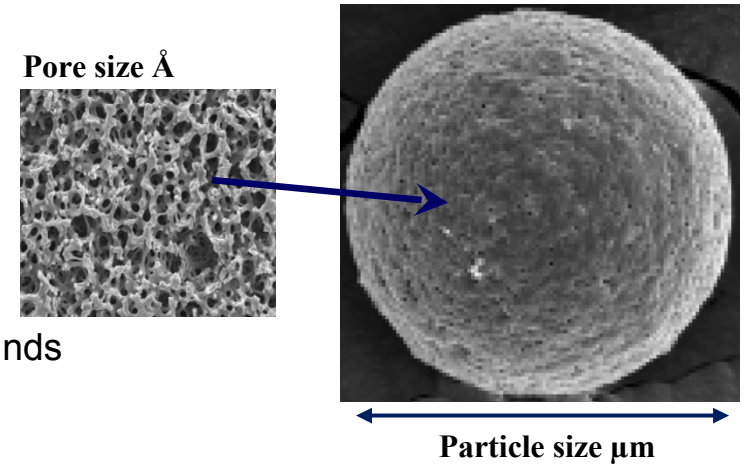
Polymers :
Polymer sorbents are very stable from pH 1 to 14, they exhibit high loading capacities allowing for the cleaning of a broad range of compounds through a variety of matrices (waters, oils, plasma, urines…). Our polymers have a very high specific surface area that maximises pi-pi interactions. The capacity of our polymers are typically 15% greater than competitive polymers and 25% higher than silicas. These polymers are particularly suited for polar compound cleaning. The polymer surface can be easily modified and facilitates a large selectivity range from hydrophobic to hydrophilic interactions.

Silicas :
Silica & bonded silica are rigid supports that do not shrink or swell with solvents. The silica surface can be easily modified, this creates a potential for a large selectivity for SPE from hydrophobic to hydrophilic interactions. The pH stability of bonded silica is limited, typically to within the range of 2 to 7.5, this is chemistry dependant. Interchim offers more than 30 different silica based selectivities. Our sorbents take advantage of our ultra pure spherical silica , and this achieves greater reproducibility, and establishes repeatable extraction and optimized sample recoveries.

Sorbent selection - sorbents main features

Particle size

30µm, 40µm, 50µm, 60µm & 140µm
30µm reduced collection volume
140 µm improves flow rate of viscous sample



Pore size

60Å & 120Å - pharmaceutics, environnemental & food compounds
300Å - biomolécules & bio-medicaments

Surface area & Capacity

Sorbent	Pore size	Surface Area	max. Capacity
Irregular silica gel	60A	450-550 m^2/g	SI: 6% - C18: 3%
Spherical silica gel	60A	400-650 m^2/g	SI: 10% - C18: 5%
Spherical silica gel	120A	280-350 m^2/g	SI: 8% - C18: 4%
PSDVB Polymer	n.d.	800 - 850 m^2/g	10 - 15%
PSDVB Polymer	n.d.	1200 - 1500 m^2/g	15 - 30%

Bonding

Over 20 chemistries are available
hydrophilic, hydrophobic, ion exchange & mixed mode

Sorbent selection - which interaction is drived by the bonding

Hydrophobic interaction

Van der Waals forces

C18, C18U, C8, C4, Phenyl, Cyclohexyl,
Pur PSDVB …

Polar interaction

Dipolar attraction or hydrogen interaction

Virgin Silica gel, Diol, CN, Florisil, NH2
Mixed polymers

Ion exchange

Electrostatic attraction

SCX, SAX, WCX, DEAE, NH2, …
Modified mixed polymers

Mixed Mode

Hydrophobic & Ion exchange

Silica gel bonding (C8/SCX, C8/SAX, C8/WCX, C8/NH2, …)
Modified mixed polymers

Sorbent Selection - methodology

The selected sorbent needs to have an excellent affinity for the compounds of interest and at the same time a weak affinity for irrelevant compounds within the matrix.

Choosing the correct sorbent results in a specific selectivity for the compounds of interest. A sufficient loading capacity also needs to be identified to optimise retention volumes of the desired compound.

Compounds of Interest properties

polar - non polar – ionic

→ Potential extraction mechanisms

Matrix properties

aqueous - organic - ionic stengh - pH

Matrix components

proteins - fats - salts - surfactants

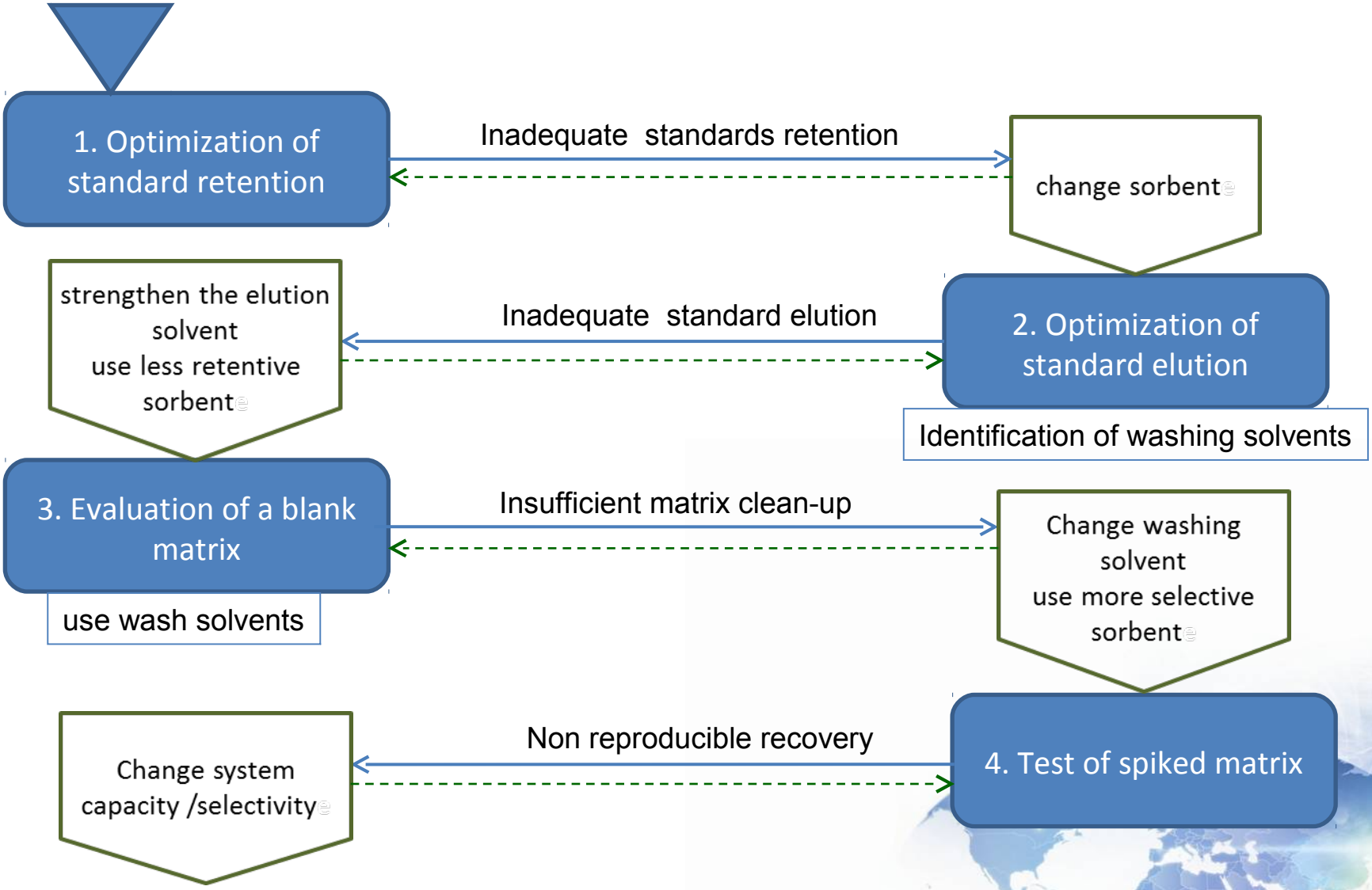
→ Treatment type to use & to avoid

Selection of Potential Sorbents

interchim

Sorbent Selection - methodology

1 + 4 steps to finalize the sorbent selection & develop the method



Sorbent Selection - Guide

Silica — **Polymers** — **Mixed Polymers**

Upti-Clean™
Spherical Pure Silica - 50µm - 60Å, 500m²/g

Recovery™ RESI & REC18
Spherical Ultra Pure Silica - 50µm
100% of the surface is accessible

Accurate Bed Technology™
Excellent batch to batch & column to column reproducibility
Precise sorbent weight +/- 1%

Atoll™ X
Spherical Pure PSDVB 40µm - 800m²/g

Atoll™ XC
Spherical Pure PSDVB - 30 & 70µm - 1500m²/g

Atoll™ XWP
Spherical Pure PSDVB - 90µm - 1200m²/g

Poly-Clean™ 2H
Spherical Pure Mixed Polymer - 30 & 60µm, 850m²/g

Poly-Clean™ HCX, HCW, HAX, HAW
Spherical Pure Mixed & Ion Exchanger Polymer - 30 & 60µm, 850m²/g

BioP™ P, CX, CW, AX
Spherical Pure Mixed BioPolymer - 30 & 60 µm - 400m²/g

Sorbent Selection - Guide

Hydrophobic sorbents

In reversed phase, the non polar functional groups of the sorbent operate according to Van der Waals forces. Alkyl and aromatic chains are function groups that have affinity with non-polar and mid-polar compounds.

Free silanol groups left on the sorbent favour polar interactions.

For aromatic compounds, eg, pharma-based chemistries, we recommend to use phenyl selectivity (polystyrene divinylbenzene polymers).

Hydrophilic sorbents

Normal phase provides an efficient cleaning of molecules with polar function groups.

Cyano (CN) functional groups can be used either in normal phase to extract polar compounds or in reversed phase for mid-polar compounds.

Diol functional groups can enhance polar compound extraction compared to virgin silica.

Amino sorbent (NH_2) can be used either as weak anion exchangers (for strong acids), or as a polar sorbent that can interact with OH, NH, SH ...

Sorbent Selection - Guide

Ion exchange sorbents

Ion-exchange retention is based on ionic interaction.
This sorbent creates a strong attraction with opposite functional groups of the sample compounds.
Ion exchange sorbent interactions depend essentially on counter-ion pH and ionic strength.

Strong anion exchange phases (SAX) possess a strong quaternary amine. They are used to extract weak acids which have one or more negative charges.

Strong cation exchange phases (SCX) contain sulfonic acid that are used to extract weak basic compounds which have one or more positive charges.

Weak anion exchange phases (DEAE, NH2) possess a diethyl amino ethyl and amino group. They are used to extract strong acids which have one or more negative charges.

Weak cation exchange phases (WCX) contain carboxylic acid that are used to extract strong basic compounds which have one or more positive charges.

Sorbent Selection - Guide

Mixed mode sorbents

Mixed mode sorbents exhibit the greatest selectivity. Ion exchange and hydrophobic chains are bonded onto the surface of silica providing unique selectivity.

This technique is used for basic compound extraction especially for drugs and metabolites within biologic fluids. Initially compounds that possess acid or basic functionality are retained by ion exchange functionality. A washing step with an appropriate pH, removes ionizable impurities.

Passing an organic solvent through the column will then remove retained impurities that result from hydrophobic bonding.

Upti-Clean™ & Recovery™

silica based sorbent

Spherical pure silica

Particle size : 50 µm (+/- 5 µm)

Surface Area : 500 m^2/g (+/- 50 m^2/g)

Pore size : +/- 10 Å

Reproducibility batch to batch & column to column

+ 20 chemistries available

C18, C8, NH2, CN, PH, OH, DEAE, SCX, SAX, Mixed Mode, …

Our bonding technology ensures greater batch to batch reproducibility for our bonded silica, there is no longer a requirement for batch reservations. Our products subsequently achieve superior recovery rates relative to traditional irregular silicas, exhibiting excellent reproducibility & consistency

Sorbent Selection - Guide - Recovery™

Recovery™ columns type address recovery and reproducibility problems, highlighted in recent studies, associated to irregular 60 Å silica's for which the surface area isn't fully accessible for clean-up procedures.

These columns utilize an optimized version of Recovery™ sorbent.
They prevent physical phenomenon associated with older generation of silica sorbent and utilize 100% of their specific surface area.

Recovery™ REC18 can be used in all solvent conditions (including 100% water) achieving greater reproducibility and consistency.

REC18 : C18, fully end-capped for the extraction of non-polar, mid-polar compounds in aqueous environment.

RESI : Virgin silica for the extraction of polar and mid-polar compounds from organic matrices.

Sorbent Selection - Guide - Upti-Clean™

Upti-Clean™ SI-S, C18-S

The most common sorbents for Extraction & Purification of Polar to Non-Polar compounds

Matrix: Environmentals, Pharmaceuticals.

Primary mechanism : Hydrophilic or Hydrophobic interactions

Upti-Clean™ FLPR & P6

Florisil PR is a mainly dedicated to cleanup and separation of chlorinated pesticide residue prior to identification and measurement of the pesticide

P6 is based on Nylon 6. Due to its activation process it exhibits a constant selectivity toward flavones, chalkones, anthraquinones,

Upti-Clean™ SCX & WCX

Extraction & Selective Purification of basic compounds

Matrix : Aqueous sample

Primary mechanism : Strong & Weak Cations Exchange

Upti-Clean™ MM1(RP/SCX) & MM2 (RP/WCX)

Extraction & Very Selective Purification of basic compounds

Matrix : Aqueous sample

Primary mechanism : mixed mode interactions (ionic & hydrophobic)

Sorbent Selection - Guide - Atoll™

Atoll™
polymer based sorbent

Spherical pure PSDVB

Particle size : 30 & 70 µm (+/- 5 µm)

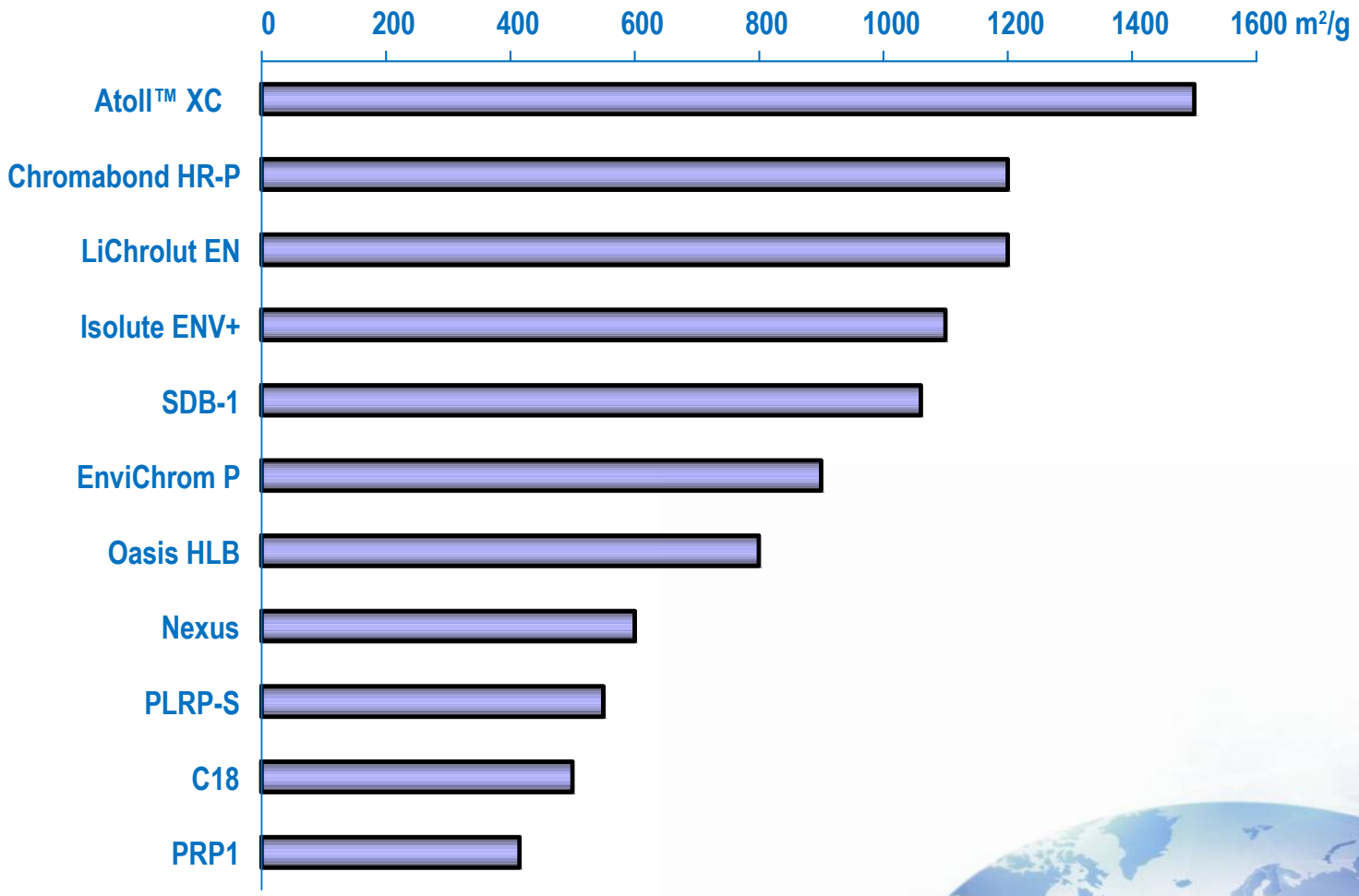
Surface Area : 800 to 1500 m^2/g (+/- 50 m^2/g)

Reproducibility batch to batch & column to column

Sorbent Selection - Guide - Atoll™

Atoll™ X	Atoll™ XC	Atoll™ XWP
800 m²/g	**1500 m²/g** Highest Surface Area available	**1200 m²/g**
High Capacity > 20%	**Xtrem Capacity > 30%**	**Xtrem Capacity > 25%**
Universal Polymer compatible w/all matrix	**Universal Polymer compatible w/all matrix**	**Universal Polymer compatible w/all matrix**
40 µm	30 & 70 µm	90 µm
non Polar compounds MW < 3000D	Polar & non Polar compounds MW < 3000D	Bio-Drugs & viscous sample MW < 400KD

Sorbent Selection - Guide - Atoll™ XC



Sorbent Selection - Guide - Upti-Clean™, Recovery™ & Atoll™ features

Nature	Bonding	Interaction Mode	Comments	Exchange Capacity	End-capping	Pore Size Å	Surface m^2/g	µm
Upti-Clean								
pure spherical silica	C18-S	Reverse Phase	% C : 18	-	yes	60	500	50
pure spherical silica	PH-S	Reverse Phase	% C : 9	-	-	60	500	50
pure spherical silica	SI-S	Normal Phase	-	-	-	60	500	50
pure spherical silica	NH2-S	Normal Phase	% C : 5	-	-	60	500	50
pure spherical silica	SCX	Ions Exchange	Strong Cation Exchanger	0,7 meq/g	-	60	450	60
pure spherical silica	WCX	Ions Exchange	Weak Cation Exchanger	0,22 meq/g	-	60	450	60
pure spherical silica	SAX	Ions Exchange	Strong Anion Exchanger	0,30 meq/g	-	60	450	60
pure spherical silica	MM1	Mixed mode	mixed mode RP/SCX	0,09 meq/g	-	60	450	60
pure spherical silica	MM2	Mixed mode	mixed mode RP/WCX	0,10 meq/g	-	60	450	60
pure spherical silica	MM3	Mixed mode	mixed mode RP/SAX	0,14 meq/g	-	60	450	60
Recovery								
pure spherical silica	REC18	Reverse Phase	% C : 15	-	yes	-	-	50
pure spherical silica	RESI	Normal Phase	-	-	-	-	-	50
Upti-Clean Specialty								
Florisil	FL		Regular grade	-	-	-	-	200
Florisil	FLPR		Pesticide Residue grade	-	-	-	-	200
Polyamide	P6		-	-	-	-	-	100
pure spherical silica	WC4	Wide Pore Reverse Phase	-	-	yes	wide pore	100	50
Atoll								
PSDVB	30XC	Reverse Phase	High capacity	-	-	-	1500	30
PSDVB	XC	Reverse Phase	High capacity	-	-	-	1500	70
PSDVB	XWP	Reverse Phase	High capacity	-	-	wide pore	1200	90

Sorbent Selection - Guide - Atoll™ & Poly-Clean™ new polymer

NEW

	Type	Particle Size	Surface Area	Modification	Ion exchange
Atoll X	hydrophobic	40 µm	800m²/g	no	no
Poly-Clean 2H	Hydrophilic /hydrophobic	30 µm 60 µm	850m²/g	no	no
Poly-Clean HCX	Hydrophilic /hydrophobic	30 µm 60 µm	850m²/g	SCX	1 meq/g
Poly-Clean HCW	Hydrophilic /hydrophobic	30 µm 60 µm	850m²/g	WCX	0,8 meq/g
Poly-Clean HAX	Hydrophilic /hydrophobic	30 µm 60 µm	850m²/g	SAX	0,3 meq/g
Poly-Clean HAW	Hydrophilic /hydrophobic	30 µm 60 µm	850m²/g	WAX	0,7 meq/g

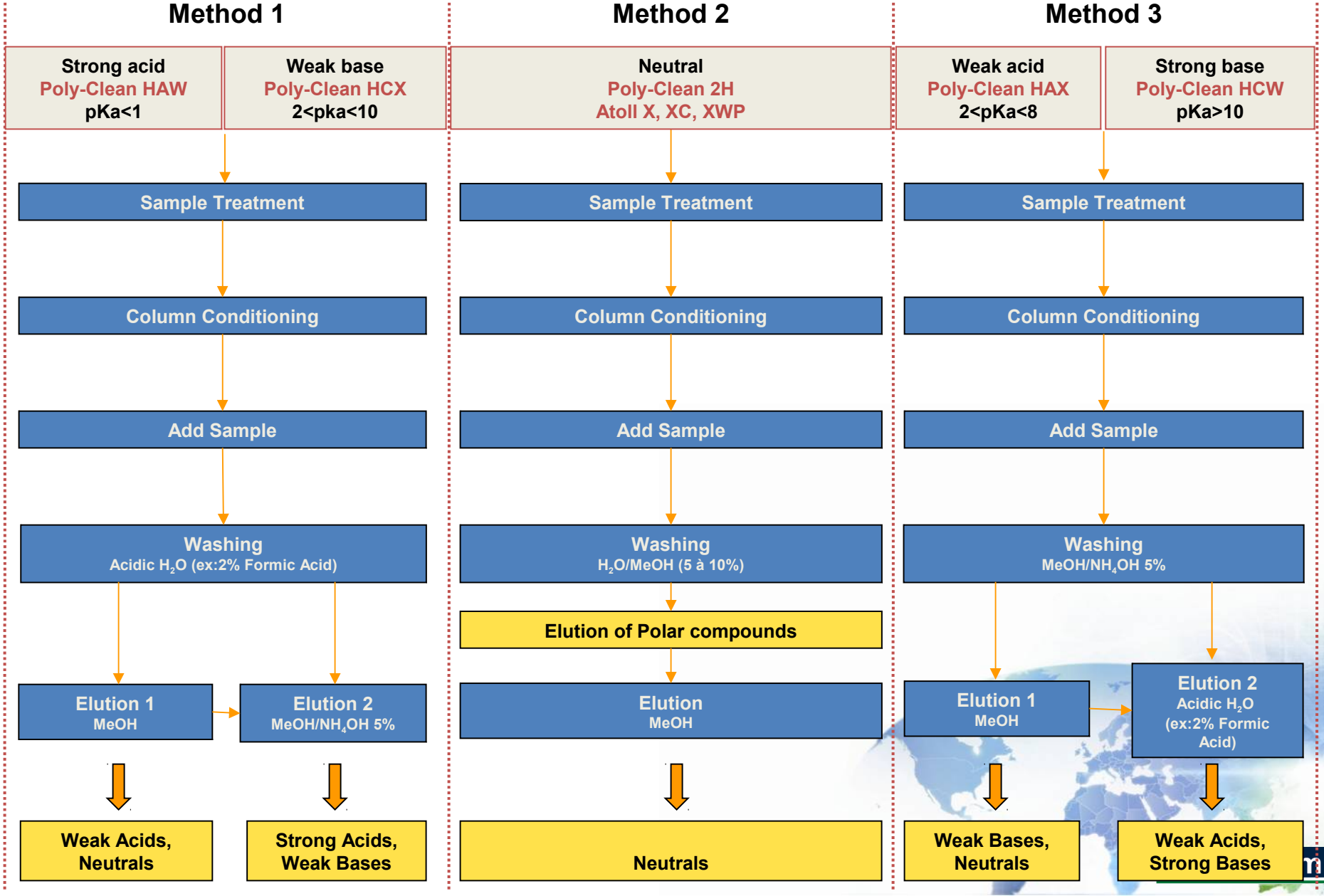
Use 60µm material for viscous samples

Preconcentration factor > 30µm vs 60µm (equal sorbent weight)

interchim

Sorbent Selection - Guide - Atoll™ & Poly-Clean™ new polymer

Indicative protocol for SPE development on Polymer Sorbents



Sorbent Selection - Columns Selection Kits - exemple

1. Pre-concentration of Hydrophobic compounds from aqueous matrix

Description : 6ml SPE columns filled with 200mg of bonded silica or polymer sorbents
P/N : SPE-D137

Including:
Upti-Clean REC18 200mg / 6mL
Upti-Clean C18-S 200mg / 6mL
Atoll XC 200mg / 6mL
Poly-Clean 2H 200mg / 6mL
Atoll X 200mg / 6mL
Qty : 5x10

Description : 3 ml SPE columns filled with 200mg of bonded silica or polymer sorbents
P/N : SPE-D138

Including:
Upti-Clean REC18 200mg / 3mL
Upti-Clean C18-S 200mg / 3mL
Atoll XC 200mg / 3mL
Poly-Clean 2H 200mg / 3mL
Atoll X 200mg / 3mL
Qty : 5x10u

Type of matrix : aqueous
Max. Loading capacity : 40 mg
Principle: concentration of non-polar, mid-polar compounds
Mechanisms : hydrophobic compounds are trapped onto an hydrophobic sorbent. Polar compounds have no typical interactions onto the sorbent. Organic solvent is used for the elution step.

Indicative protocol

Column conditioning: MeOH then DI H_2O or (DI H_2O buffered to improve the adsorption of ionic compounds)
Matrix preparation : set the appropriate pH to extract ionic compounds
Sample volume : 5ml up to 1000ml – (large volume may decrease mid-polar compounds yield).
Column washing: Optional, a polar solvent allows removal of polar impurities
Drying : 5-10min
Elution : MeOH, MeCN, CH2Cl2/MeOH

Sorbent Selection - Columns Selection Kits - list

2. Pre-concentration of Hydrophobic compounds from aqueous matrix
3. Extraction of Hydrophobic compounds from aqueous matrix
4. Pre-concentration of Hydrophilic compounds
5. Removal of polar compounds from aqueous and organic matrix
6. Extraction of Acidic, Basic & Neutral compounds from aqueous or organics matrix
7. Extraction of carboxylic acids and strong bases from aqueous matrix
8. Extraction of weak bases from aqueous matrix
9. SPE method development for extraction of Acidic, Neutral & Basic compounds - I
10. SPE method development for extraction of Acidic, Neutral & Basic compounds – II
11. SPE method development for extraction of Acidic, Neutral & Basic compounds in Biofluids

each kit is shipped with an indicative protocol

Sorbent Selection - Specific Application Kits - exemple

1. Extraction of basic drugs from biological fluids

Description : 3ml SPE columns filled with 200mg of mixed mode sorbent
P/N : SPE-SA1
Qty : 50units

Type of matrix : urine, blood, plasma, serum
Principle : selective extraction of basic compounds
Mechanisms : Mixed mode sorbent traps hydrophobic compounds and basic compounds.
Polar compounds go through the column with the sample fraction. Acidic, neutral and hydrophobic compounds are eliminated by washing. Weak and strong bases are selectively eluted.

Indicative protocol

Column conditioning : 2,5ml MeOH then 2,5ml DI H_2O, 1ml 100mM phosphate pH 6,0
Matrix preparation : adjust pH 6 with appropriate buffer
Sample volume : 1-10ml
Column washing 1 : 2ml MeOH/H2O with 100mM acetic acid (5/95)
Column washing 2 : 2ml MeOH
Drying : 5min
Elution : MeOH/NH4OH 8M (98/2)

Sorbent Selection - Specific Application Kits - List

2. Extraction of Polycyclic Aromatic Hydrocarbons (PAH) from waters or soils
3. Extraction of Polycyclic Aromatic Hydrocarbons from water containing humic acids
4. Extraction of Polycyclic Aromatic Hydrocarbons (PAH) from soils & oils
5. Extraction of Oil & Grease from aqueous matrix (EPA Method 1664)
6. Extraction of Bisphenol A (BPA) from aqueous matrix
7. Extraction of Pesticides & Herbicides from aqueous matrix
8. Extraction of Steroids from biological fluids
9. Extraction of PolyChlorinated Biphenyls (PCB) from oils
10. Extraction of Semi Volatile Organics (SVOCs) from water (EPA 525)

each kit is shipped with a protocol

Sorbent Selection - EPA Methods - List

Related P/N products	EPA methods	Name
Recovery REC18 1G/6ml Upti-Clean C18-S 1G/6ml	608	Organochlorine Pesticides & PCB's from water
Upti-Clean SAX 1G/6ml	552.1	Haloactic Acids & Dalapon
Upti-Clean C8-S 1G/6ml	549.1	Diquat and Paraquat from Drinking Water
Upti-Clean C18-S 1G/6ml Recovery REC18 1G/6ml	525.2	Semi Volatile Organics (SVOCs) in Drinking Water
Upti-Clean C18-S 1G/6ml Recovery REC18 1G/6ml	506 (DW)	Phthalates
Upti-Clean C18-S 1G/6ml Recovery REC18 1G/6ml	8082	Polychlorinated Biphenyls from Aqueous Matrices
Upti-Clean C18-S 1G/6ml Recovery REC18 1G/6ml	8080	Organochlorine Pesticides from waste water
Upti-Clean C18-S 1G/6ml Recovery REC18 1G/6ml	508.1 (DW)	Chlorinated Pesticides in Water

interchim