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IUPAC Recommendations

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Glossary of terms used in extraction (IUPAC Recommendations 2016)

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Abstract: Approaches for analytical-scale extraction are developing rapidly as new strategies are implemented to improve sample throughput, to minimize material use in laboratory methods, and to develop on-site capabilities. In this contribution, definitions and recommendations for symbols for the terms used in analytical extraction are presented. Exhaustive, microextraction, elevated temperature, microwave- and ultrasound-assisted, parallel batch, flow through systems, and membrane extraction approaches are discussed. An associated tutorial titled "Extraction" provides a detailed introduction to the topic.

Keywords: extraction; recommendations; terminology.

CONTENTS

1. INTRODUCTION 517
2. ALPHABETICAL ENTRIES 518
MEMBERSHIP OF SPONSORING BODY 556
REFERENCES 556

1 Introduction

The general procedure for the analysis of complex samples typically consists of several steps identified as sampling, sample preparation, separation, quantification, statistical evaluation, and decision-making. Each step is critical to obtain informative results for optimum decision making. In many cases, the key is the selection of the most appropriate sample preparation step for a particular task. The objective of this step is to isolate target analytes from a *matrix*, because most analytical instruments are designed to handle the analytes but are frequently incapable of accommodating the analytes and matrix together. Sample preparation usually involves *extraction* procedures and can also include "clean-up" procedures for very complex, "dirty" samples. This step must also concentrate the analytes to a level suitable for detection, and therefore, sample preparation methods typically include enrichment. The fundamental basis for extraction processes is often substantially different to those employed in chromatographic separations or other traditional disciplines of analytical chemistry. Analytical-scale extractions generally resemble the corresponding engineering processes, but on a smaller

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scale. There is a tendency to name the extraction techniques according to random criteria. The objective of this contribution is to develop a consistent nomenclature for terms to be used to describe the extraction processes. An associated tutorial, titled "Extraction," provides a detailed overview of the extraction processes.

If a reference is given to a PAC Recommendation without qualification, then the term and definition are unchanged from the earlier version. If the reference reads "Adapted from Reference [xx]," then some changes have been made. Terms from the JCGM VIM [1] are cross referenced without repeating the definition. A qualifying term in parentheses after a term is used to restrict the scope for a term with use in other fields of chemistry or science.

2 Alphabetical entries

2.1 General terms (Fundamental processes)

2.1.1 absorption (extraction)

Process leading to extraction where *analytes* are partitioned from their *matrix* into a liquid
Note: The use of term absorption here should be distinguished from the use of absorption in spectroscopy for the phenomenon in which radiation transfers some or all of its energy to matter.
Adapted from reference: [2].

2.1.2 adsorbent

A condensed phase at the surface of which *adsorption* may occur.
Reference: [2].

2.1.3 adsorption

Increase in the concentration of a dissolved substance at the interface of a condensed phase due to the operation of surface forces. Adsorption can also occur at the interface of a condensed and a gaseous phase.
Reference: [2].

2.1.4 analyte

Part of a system embodying the *measurand* (VIM 2.3 [1]).
Adapted from reference: [3].

2.1.5 boundary layer or diffusion layer (extraction)

Concentration boundary layer
Region in the vicinity of the interface between the sample and *extraction phase* where mass transfer is determined by diffusion.

2.1.6 boundary layer thickness

Distance from the interface beyond which mass transfer is determined by convection.

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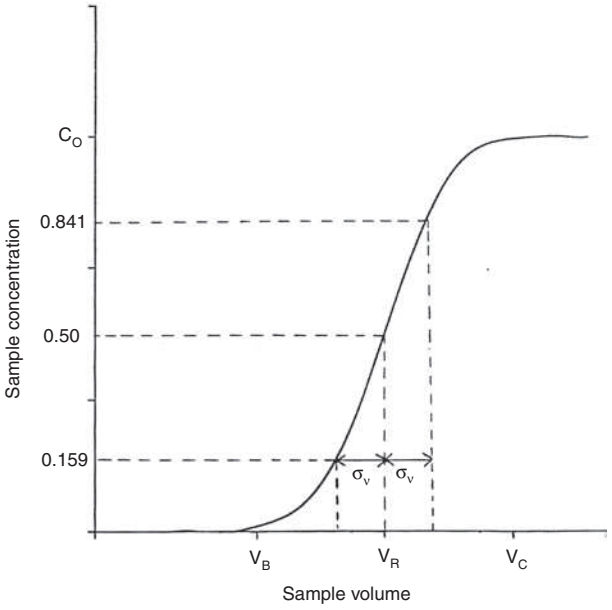

Fig. 1: Typical breakthrough curve indicating the breakthrough volume V_B and the sample volume corresponding to the saturation capacity of the sorbent V_C (V_R corresponds to the chromatographic elution volume and σ_V the standard deviation of the derivative of the curve).

2.1.7 breakthrough volume

The volume at which a defined amount of an *analyte* pumped continuously through an extraction device will begin to be eluted.

Note 1: The breakthrough volume, V_B, for a particular compound and sampling device is determined by its breakthrough curve, Fig. 1 [3].

Note 2: Defined amounts are usually given as a percentage vol/vol of the sample volume (e.g. 1 %, 5 % or 10 %, etc.).

Reference: [3].

2.1.8 distribution

Apportionment of a solute between two phases.

Note 1: The term partition or *extraction* may also be used in this sense where appropriate.

Reference: [4].

2.1.9 distribution ratio, *D*

Ratio of the total concentration of an *analyte* in the *extraction phase*, regardless of its chemical form, to its total concentration in the other phase.

Note: *D* is dependent on experimental conditions (pH, presence of complexing agents, etc.) and does not necessarily imply that equilibrium has been achieved.

Adapted from reference: [4].

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2.1.10 distribution constant, K_D

partition ratio
distribution coefficient, distribution ratio, partition constant, and extraction constant should not be used
Ratio of the concentration of an *analyte* in a single defined form, A, in phase 1 to its concentration in the same form in a second phase (phase 2) at equilibrium.

$$K_D = [A]_1/[A]_2$$

Note 1: If the equation relates to an organic-aqueous biphasic system, the organic phase concentration is by convention the numerator and the aqueous phase concentration the denominator.
Note 2: In the case of non-ideal mixtures, concentrations should be replaced by activities. The distribution constant is invariant only if the activity coefficients are constant, which is not true in concentrated solutions.
Note 3: The use of the inverse ratio (aqueous/organic) may be appropriate in certain cases, e.g. where the organic phase forms the *feed*, but its use in such cases should be clearly specified. The ratio of the concentration in the denser phase to the less dense phase is not recommended, as it can be ambiguous.

2.1.11 ion-pair extraction

Transfer of ions from an aqueous phase to an immiscible liquid (or solid) phase by adding an ion of complementary charge to the aqueous phase.

2.1.12 partition

This term is often used as a synonym for *distribution* and *extraction*. However, an essential difference exists by definition between *distribution constant* (*partition ratio*) and *partition constant*.
Note: This term should be, but is not invariably, applied to the distribution of a single definite chemical species between the two phases.

2.1.13 partition constant, K_D°

partition coefficient should not be used
Ratio of the activity of a species A in the extract $a_{A,1}$ to the activity in a second phase with which it is in equilibrium $a_{A,2}$.

$$K_D^\circ(A) = a_{A,1}/a_{A,2}$$

Note: The value of K_D° depends on the choice of standard states and on the temperature (and since liquids are weakly compressible, on pressure at high pressures).
Reference: [4].

2.1.14 phase ratio

Ratio of the *extraction phase* and sample solution contained in the extraction device.
Note 1: Ratio of the volume of *extraction phase* to the volume of sample solution in liquid–liquid extraction.
Note 2: Ratio of the headspace volume to the condensed phase volume in a closed container for headspace methods.

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Note 3: Ratio of the surface area of the *extraction phase* to the volume of sample solution contained in the extraction device for solid-phase extraction.
Note 4: When the extraction phase is a gas or liquid, the sum of the extraction phase and sample solution volumes equals the volume of the extraction device.

2.1.15 solvent (extraction)

Immiscible fluid added to a process for the purpose of extracting material from the sample.

2.1.16 sorbent

Condensed phase where extraction of *analytes* can occur at the surface and in the bulk.
Note 1: If extraction occurs exclusively at the surface then *adsorbent* is the preferred term and if exclusively in the bulk then *absorbent* should be used.
Note 2: Sorbent is typically used explicitly to describe a condensed phase where extraction occurs simultaneously by *adsorption* and *absorption*, or where the true mechanism has not been established and the use of adsorbent or absorbent would be inappropriate.

2.2 General terms (Process technology)

2.2.1 acceptor phase

Component of a multi-phase system into which the target *analytes* are extracted.
Note 1: When the acceptor phase is a liquid the term acceptor solution may be used.
Note 2: In extractions employing a semipermeable membrane for phase separation the acceptor phase is the phase to which *analytes* are transferred from the *donor phase*.

2.2.2 active sampling

Continuous *extraction* with movement of the sample *matrix* with respect to the *extraction phase*.

2.2.3 batch extraction

Extraction in which the extraction phase and the sample are confined in an extraction vessel and processed according to a defined procedure.
Note 1: Term "batch" is also used in extraction to indicate a number of samples extracted sequentially or simultaneously by the same procedure (e.g. extraction of sample batch(es)).

2.2.4 calibrant in extraction phase

Internal standard that is incorporated in the *extraction phase* for *calibration* [VIM 2.39 [1].

2.2.5 charcoal tube

Tube filled with charcoal typically used to adsorb *analytes* from the gas or liquid phase.
Note: Activated charcoal was used in early applications but a wider selection of carbon materials are currently used, with properties tailored to each application

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2.2.6 continuous extraction

Extraction in which the sample is continuously introduced into the extraction device.

2.2.7 countercurrent extraction

Extraction in which both phases are continuously added (or changed) and flow (or move) in opposite directions as the extraction progresses.
Note 1: The phases are either immiscible or separated by a membrane.

2.2.8 distribution constant calibration

Calibration (VIM 2.39 [1]) for *extraction* when close to equilibrium conditions, using a known *distribution constant* for the *analyte*.

2.2.9 donor phase

In *extraction*, the sample or sample solution.

2.2.10 dynamic extraction

Extraction in which clean (or recycled) *extraction phase* is continuously passed through the sample.
Note: The extraction phase can be a gas or a liquid.

2.2.11 enrichment factor (S)

The factor by which the ratio of two substances in the feed must be multiplied to give their ratio after treatment. $Q_A/Q_B = S_{AB}(Q'_A/Q'_B)$ where Q_A and Q'_A are the final and initial amounts of species A and Q_B and Q'_B are the final and initial amount of species B. Hence, $S_{AB} = E_A/E_B$ where E is the fraction extracted.
Reference: [4].

2.2.12 equilibration

Operation by which a system of two or more phases is brought to a condition where further changes with time do not occur.
Note: This term is not synonymous with pre-equilibrium and should not be used in that sense.
Reference: [4].

2.2.13 exhaustive extraction

Type of *extraction* where the intent is a complete extraction of the *analytes*.

2.2.14 external calibration

Calibration (VIM 2.39 [1]) employing a simplified sample *matrix* spiked with target *analyte* embodying a reference value of the *measurand* (VIM 2.3 [1]).

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2.2.15 extract (noun)

Separated phase that contains material isolated from another phase.
Adapted from reference: [4].

2.2.16 extractant

Synonym: extracting agent
The active component(s) primarily responsible for transfer of a solute from one phase to the other.
Reference: [4].

2.2.17 extraction

Transfer of *analytes* from one phase to a different phase where further processing and analysis occurs.
Adapted from reference: [4].

2.2.18 extraction phase

Liquid, solid, or gas phase in contact with the sample for the purpose of removal of *analytes* from the sample *matrix*.

2.2.19 extraction rate

kinetics of extraction
Rate at which *analytes* are transferred from the sample to the *extraction phase*.

2.2.20 extraction recovery

See fraction extracted.

2.2.21 feed

Solution or gas containing *analyte* introduced into an extraction.
Note: If feed is used without qualification, the term may be taken to designate the initial liquid phase containing the main solute to be transferred or sample solution in a batch process.
Adapted from reference: [4].

2.2.22 fluorous extraction

Extraction in a biphasic system in which the main component of one phase is a highly fluorinated solvent (liquid–liquid extraction) or highly fluorinated *sorbent* (*solid-phase extraction*).
Reference: [5].

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2.2.23 fraction extracted (E)

Extraction recovery
Ratio of the amount of *analyte* extracted under specified conditions to the amount of analyte in the sample.
$E_A = Q_A/Q'_A$ where Q_A is the mass of A extracted and Q'_A is the total mass of A present at the start.
Note 1: Fraction extracted is typically expressed as a percentage with units of mol/mol, or g/g.
Adapted from reference: [4].

2.2.24 fractionation (of analytes)

Extraction of an *analyte*, or a group of analytes, from a sample according to physical (e.g. size, solubility) or chemical (e.g. bonding, reactivity) properties.
Adapted from reference: [6].

2.2.25 leaching

Dissolution of sample components from a solid with a liquid in which the solid phase is not wholly soluble.
Adapted from reference: [4].

2.2.26 mass transfer

Spontaneous (irreversible) process of transfer of mass across non-homogeneous fields. The driving force can be the difference in concentration (in liquids) or partial pressure (in gases) of the component. In fluids, mass transfer may be enhanced by turbulent flow. In biological systems, mass transfer through membranes may result from normal diffusion, facilitated diffusion, or active transport
Reference: [7].

2.2.27 matrix

The components of the sample other than the *analyte*(*s*).
Reference: [8].

2.2.28 matrix-matched calibration

Calibration (VIM 2.39 [1]) employing external calibration in which standard solutions of target *analytes* are prepared in a solution of analyte-free *matrix*.
Note: Matrix-matched calibration is used to minimize the matrix effect on the measurement of target *measurands* (VIM 2.3 [1]) [9, 10].
References: [9, 10].

2.2.29 microextraction (methodological approach)

Extraction method characterized by a low phase ratio, so that at equilibrium the fraction extracted is low (not exhaustive).
Reference: [11].

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2.2.30 microextraction (technique)

Extraction technique in which the extraction phase (liquid or solid) is substantially smaller than the sample volume.
Note: The amount of extraction phase is typically <100 µl or <10 mg and the sample volume > 1 ml.

2.2.31 multiwell sample plate

Plate with multiple wells for individual samples.

2.2.32 optimisation of extraction

Adjustment of extraction parameters to achieve a defined goal.
Note: Many extractions are controlled by several parameters and their interactions. Optimum conditions are more efficiently identified by multivariate methods.
Related terms: *design of experiments*, *optimization*.

2.2.33 passive sampling

Extraction process driven by diffusion of *analytes* towards the *extraction phase*.

2.2.34 preconcentration (extraction)

An operation (process) by which the *analyte*(*s*) and/or sample concentration is increased prior to extraction.
Adapted from reference: [12].

2.2.35 Raffinate

Phase left from the *feed* after being contacted by the extracting phase.
Adapted from reference: [4].

2.2.36 salting in

Addition of an electrolyte to an aqueous phase to decrease the *distribution ratio* of the *analyte*(*s*) or *matrix*.
Related term: *salting out*.

2.2.37 salting out

Addition of an electrolyte to an aqueous phase to increase the *distribution ratio* of the *analyte*(*s*).
Note: The term is also used for the addition of electrolytes to reduce the mutual partial miscibility of two liquids.
Related term: *salting in*.
Adapted from reference: [4].

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2.2.38 saturation capacity

Sample volume at which the concentration of *analyte* at the exit of a sampling device is the same as the sample concentration.
Note 1: Corresponds to V_C in Fig. 1.
Note 2: Corresponds to the sample volume (or sample amount) that results in the isolation of the maximum amount of analyte, but with a lower overall recovery because a fraction of the sample is lost.

2.2.39 sequential extraction

Extraction wherein *extractants* of increasing strength or different chemical character are used in order to extract *analytes* of interest.
Note: The most commonly used approaches in elemental analysis are the BCR [13] (now termed Standards, Measurement and Testing Program) and Tessier [14] procedures.
References: [13, 14].

2.2.40 speciation analysis

Analytical activities identifying and/or measuring the quantities of one or more individual chemical species in a sample.
Reference: [6].

2.2.41 static extraction

Batch extraction in which the *extraction phase* attains a constant composition before phase separation.
Note 1: If the *analytes* are initially present as a solution in an immiscible liquid phase the process is synonymous with *liquid–liquid extraction*.
Note 2: If the sample is a solid that is partially soluble in the extraction phase then *leaching* may be a more appropriate term.
Related term: static headspace analysis.

2.2.42 steady state extraction

The state of a continuous process operating in such a way that the concentration of solutes in exit streams remains constant with respect to time for constant feed concentrations, even though the two phases are not necessarily in thermodynamic equilibrium in any part of the process. The term equilibrium should not be used to describe this situation.
Reference: [4].

2.2.43 time-weighted average sampling

TWA sampling
A passive or active sampling strategy over a relatively long time, during which the amount of *analyte*(*s*) extracted is proportional to its average (mean) concentration over that time.

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3 Extraction techniques

3.1 Gas-phase extraction

3.1.1 chemical hydride generation, (CHG)

Type of *chemical vapor generation* wherein volatile hydrides are generated by reaction with (usually) borane complexes in aqueous solution.
Reference: [15].

3.1.2 chemical vapour generation, (CVG)

Extraction of *analyte* from a sample by a chemical reaction-generating gaseous species.
Note: The analyte is usually an element.

3.1.3 closed-loop extraction

Closed-loop stripping analysis
Extraction by a fixed volume of gas continuously recycled through the sample and a *sorbent trap* by a pump in a closed-circuit arrangement.
Note 1: A typical apparatus is shown in Fig. 2 [16, 17].
Note 2: Closed-loop extraction was developed for trace analysis of volatile compounds (at mass concentrations of ng L^{-1}) in drinking water.
References: [16, 17].

3.1.4 cryogenic trap

Device used to concentrate volatile *analytes* from the gas phase by condensation on a cold surface.
Note: Typically, cryogenic traps have a low thermal mass to respond rapidly to changes in temperature and are prepared from short lengths of capillary tubing. Sub-ambient temperatures are required for the efficient collection of analytes from a flowing gas stream and above ambient temperatures for evaporation into a flowing gas stream for analysis, usually by *gas chromatography* or *atomic spectrometry*.

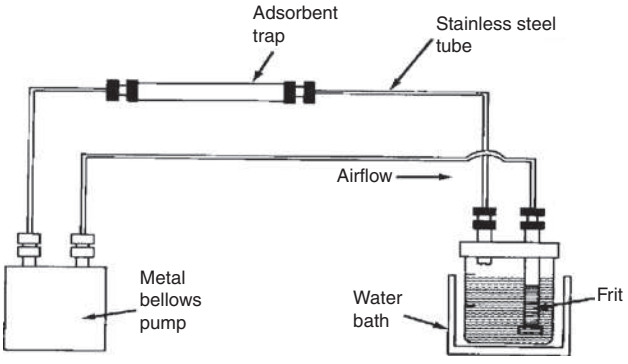

Fig. 2: Typical apparatus for closed-loop stripping analysis.

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3.1.5 denuder (device)

Apparatus for the extraction of volatile *analytes* from a flowing gas stream by diffusion to the wall of a collection vessel, where they are retained by chemisorption or strong physisorption by the wall coating.
Note 1: There are many design variations [18].
Note 2: Both liquid and solid sorbents are used for trapping.
Reference: [18].

3.1.6 dynamic headspace analysis

Continuous removal of *headspace* above a sample by a flow of inert gas over or through the sample until all or a significant portion of the *analytes* have been removed from the sample. Sample *vapors* are typically transferred to a *sorbent trap* or *cryogenic trap* for collection.
Note 1: Purge-and-trap is a version of dynamic headspace analysis in which volatile compounds are purged from solution by a continuous stream of gas bubbles (sparging gas) and transported through the headspace to a sorbent or cryogenic trap for collection.
Note 2: A typical apparatus for purge-and-trap is shown in Fig. 3.

3.1.7 extractive distillation

Separation of close-boiling liquids by fractional distillation in the presence of a low-volatility *solvent* or salt that selectively interacts with one or more components of the mixture, increasing the difference in *vapor* pressure of the sample components.
Note 1: If the separation solvent forms an azeotrope with one or more sample components the process is called azeotropic distillation,

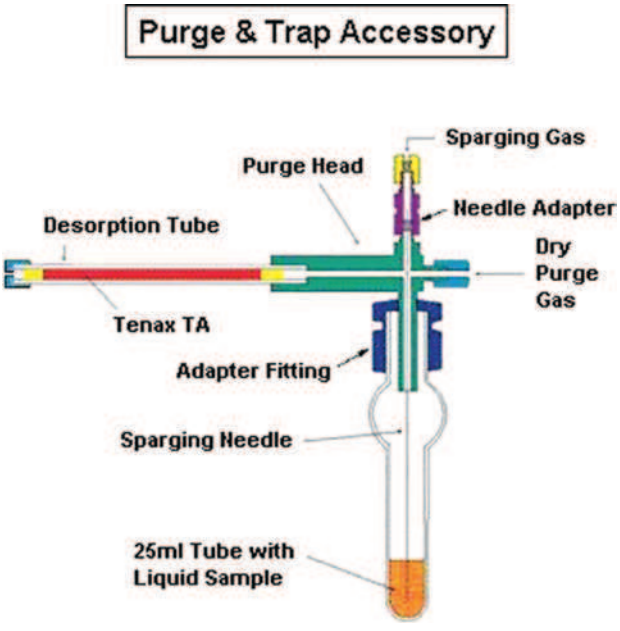

Fig. 3: Typical apparatus for analysis by dynamic headspace (purge and trap). The gas flow (sparging gas) enters at the bottom of the sample container transporting volatile compounds through the headspace and purge head to the desorption tube for collection.

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3.1.8 flash desorption

Thermal desorption where the temperature of a packed tube or cryogenic trap is rapidly increased to transfer condensed or sorbed compounds to the gas phase.

3.1.9 foam fractionation

Extraction method for the isolation of surface active compounds by their preferential *adsorption* at a gas-liquid (bubble) interface formed by passage of an inert gas through (typically) an aqueous solution.

3.1.10 Full evaporation headspace analysis

Small volume of sample in a headspace vessel maintained at a temperature sufficient to cause complete evaporation of *analytes*

Note 1: The full evaporation technique is used with both static and dynamic headspace analysis.

Note 2: Equilibrium between the condensed and vapor phases no longer exists. As a consequence, the possibility of matrix interferences is reduced and the fraction extracted for low-volatility compounds is increased.

3.1.11 headspace

The volume of gas phase above a sample in a closed container.

3.1.12 impinger (extraction)

see also impinger for particle collection

Simple glass bubbling chamber for vacuum-sampling of volatile *analytes* by passage through an extraction solution, the purpose of which is to capture and concentrate target *analytes*.

Note: Isolation by reaction with the extraction solution is common. Selectivity depends on the chemical specificity of the extraction solution.

3.1.13 multiple headspace extraction

Dynamic gas extraction carried out stepwise, with sequential analysis of a number of *headspace* samples taken from the same vial.

Note 1: At equilibrium the headspace is sampled and the remainder (or most of it) is exhausted and replaced by fresh gas phase. The process is repeated and the original sample concentration estimated from the peak areas for two consecutive extractions.

$$A_T = \frac{A_1^2}{A_1 - A_2}$$

where A_T = estimated sum of all partial peak areas corresponding to exhaustive extraction, A_1 the peak area obtained from the first headspace fraction, and A_2 the peak area obtained in the second (consecutive) headspace fraction.

Note 2: *External calibration* is required to convert peak areas into sample concentration.

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3.1.14 simultaneous steam distillation-solvent extraction

A sample dispersed in water and a water immiscible organic solvent (*extraction phase*) in separate flasks are heated simultaneously and their vapours co-condensed and separated into two streams in which water is returned to the sample flask and the extraction phase to the organic solvent flask.

Note: A typical apparatus for simultaneous steam distillation-solvent extraction with a lighter-than-water extraction solvent is shown in Fig. 4 [18].

Reference: [19].

3.1.15 sorbent trap

Tube filled with porous *sorbent* typically used to extract *analytes* from the gas phase.

Note: The performance of a sorbent tube depends on the sorbent type, temperature, gas flow rate, and desorption time.

3.1.16 static headspace analysis

Method of analysis, in which a small aliquot of the gas phase in contact with the sample in a closed container is withdrawn for analysis.

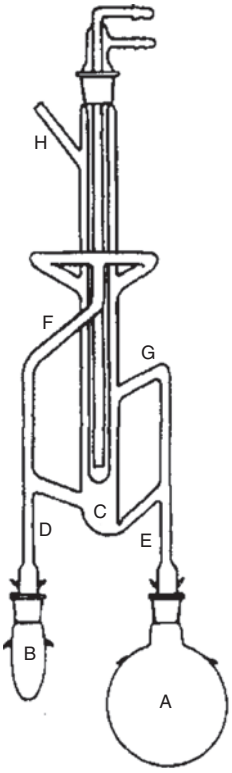

Fig. 4: Apparatus for simultaneous steam distillation-solvent extraction with a lighter-than-water extraction phase. A = aqueous sample solution or slurry; B = immiscible organic solvent and receiving flask for extracted compounds; C = bowl for phase separation; D, E, F, and G are solvent or vapour transfer arms; and H = entry port for additional solvent or alternative sample introduction position for continuous liquid–liquid extraction. Switching the position of flasks A and B allows extraction with a heavier-than-water extraction solvent.

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Note 1: At equilibrium, the *analyte* concentration in the gas phase is related to the sample concentration by a partition constant.
Note 2: The sample vessel is thermostatted.

3.1.17 sublation

Flotation process in which one or more *analytes* are adsorbed on the surface of gas bubbles moving through a solution and collected in an upper layer of immiscible solvent, or in a *sorbent trap*.
Note 1: A typical apparatus for sublation is shown in Fig. 5 [20].
Note 2: As there is no liquid-phase mixing in the bulk of the system, recoveries can approach 100 %.
Adapted from references: [4, 20].

3.1.18 thermal desorption

See flash desorption.

3.1.19 thermal extraction

Release of volatile compounds from a sample by heat.
Note: The volatile compounds are typically transported by a gas stream to a *sorbent tube* or *cryogenic trap* for collection.

3.1.20 thin-layer headspace analysis (TLHS)

Dynamic headspace extraction of *analyte*s from a flowing sample solution in the form of a thin film on the surface of a heated tube.
Note: Volatile compounds in the sample solution pass into the gas phase and are continuously swept out of the tube for analysis or detection.

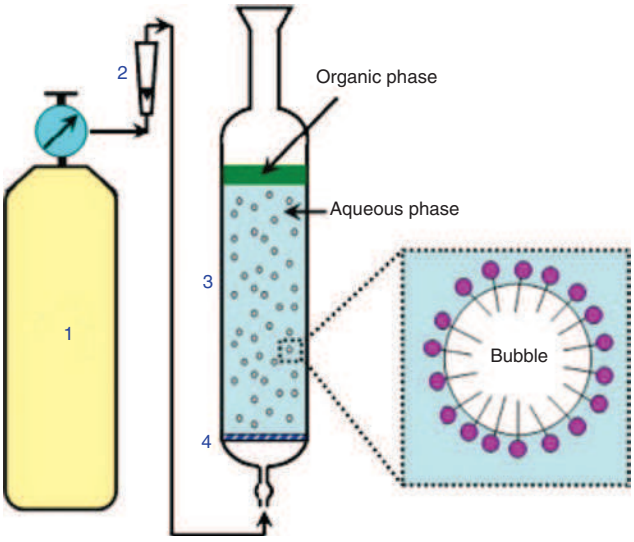

Fig. 5: Typical apparatus for extraction based on sublation. Identification: (1) nitrogen gas supply; (2) rotameter; (3) floatation cell; and (4) sintered glass disc [5].

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3.2 Liquid-liquid extraction

3.2.1 back extraction

stripping
Process of removing *analytes* from an extract by their distribution to a different phase.
Note: As normally used, back extraction refers to the main compounds present or target analytes. It is an operation commonly employed in liquid-liquid extraction to enhance selectivity or to improve the compatibility of the extract with separation and measurement techniques.
Adapted from reference: [4].

3.2.2 biphasic system (liquid-liquid extraction)

Equilibrium system containing two or more solvents forming two phases with a single interface.

3.2.3 chelation solvent extraction

Extraction method wherein charged *analytes* are extracted from an aqueous phase into an organic phase, assisted by a chelating agent.
Note 1: The chelating agent replaces water molecules in the coordination sphere of the metal ions. Consequently, the resultant metal chelates have a hydrophobic character which facilitates their transfer to the organic phase [21].
Note 2: The *analyte*(*s*) are usually metal ions.
Reference: [21].

3.2.4 cloud point extraction

Coacervative extraction at a temperature above the cloud point temperature.

3.2.5 coacervative extraction

Extraction by a surfactant-rich, water-immiscible phase formed by adding a dehydrating agent to a colloidal solution of the sample and a surfactant above its critical micelle concentration.
Note 1: In cloud point extraction a small volume "surfactant-rich" phase enriched in sample components of moderate water solubility and a bulk aqueous phase with dissolved surfactant and highly water soluble compounds are formed [22].
Reference: [22].

3.2.6 continuous liquid–liquid extraction

Extraction in which a water–immiscible extraction solvent is continuously percolated through an aqueous solution and returned to the extraction solvent reservoir.
Note 1: Typically, a fixed volume of extraction solvent is recycled through the aqueous sample solution by distillation and condensation, and dripped into the sample solution. A siphon mechanism allows continuous recovery of the extraction solvent based on its relative density.
Note 2: Typical apparatuses are shown in Fig. 6.

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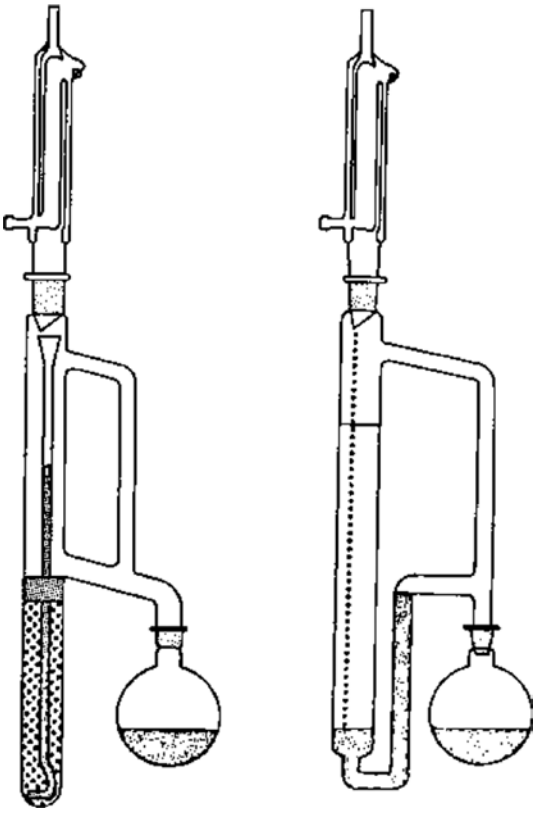

Fig. 6: Apparatus for continuous liquid–liquid extraction with a lighter-than-water extraction solvent (left) and a heavier-than-water extraction solvent (right). In each case the extraction solvent is contained in the round bottom flask and the aqueous sample in the vertical glass column.

Note 3: Continuous liquid-liquid extraction techniques are used when the sample volume is large, the distribution constant is small, or the rate of extraction is slow.

3.2.7 countercurrent chromatography (CCC)

Type of liquid–liquid chromatography using a support-free liquid stationary phase held in place by centrifugal forces while the other phase (the mobile phase) moves through it in a definite direction.

Note 1: CCC is a continuous dynamic process under steady state conditions in which equilibrium may occur but is not essential [23–25].

Note 2: CCC differs from countercurrent distribution (CCD) in that CCD is a discontinuous process based on the attainment of equilibrium prior to phase transfer in a multi-stage apparatus.

Note 3: There are two general approaches used to stabilize the liquid stationary phase based on hydrostatic or hydrodynamic system designs. Hydrostatic systems (known as centrifugal partition chromatography) employ a cascade of geometric volumes with connecting tubes, formed into a pattern and rotated around a single axis, providing a constant centrifugal force field. Hydrodynamic system designs (known as coil-planet centrifuges or high-speed CCC) employ a single flexible tube (column) wound around a former, with a main axis of rotation and a secondary axis with planetary rotation generating a variable centrifugal force field. The variable force field produces mixing and settling zones throughout the column length.

References: [23–25].

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3.2.8 Craig countercurrent distribution apparatus

Apparatus for *extraction* based on a *multistage countercurrent distribution*.

Note: The Craig CCD apparatus consists of a battery of glass vessels designed to allow the mixing, settling, and transfer of extracted phase (sample) and the addition of fresh solvent (extractant) in a repetitive fashion. Movement about the horizontal axis provides gentle mixing of the phases, settling, and decanting of the upper phase. The extraction process is slow but is fully automated.

3.2.9 crosscurrent extraction

Extraction comprising a cascading series of states where the *raffinate* is brought into a subsequent stage and contacted with fresh solvent.

3.2.10 differential contactor

Type of continuous multistage *extraction* equipment in which there is only one interface at which phase separation by settling occurs.

Reference: [4].

3.2.11 dispersing solvent

Solvent used to disperse the extraction solvent as fine droplets throughout the sample solution in *dispersive liquid–liquid microextraction*.

Note: The dispersion solvent is typically a polar organic solvent miscible with water.

3.2.12 dispersive liquid–liquid microextraction (DLLME)

Extraction of *analyte*(*s*) from an aqueous solution by fine droplets of *extraction phase* dispersed throughout the solution.

Note 1: The extraction phase is formed by a mixture of extraction and dispersion solvents injected into the sample solution [26].

Note 2: The extraction phase can be formed by ion exchange when an ionic liquid is used as the extraction solvent.

Note 3: The extraction phase is sedimented by centrifugation and collected for analysis.

Reference: [26].

3.2.13 liquid-ion exchange

A term used to describe the *liquid–liquid extraction* process that involves the transfer of ionic species from the *extractant* to the aqueous phase in exchange for ions from the aqueous phase.

Reference: [4].

3.2.14 liquid–liquid distribution

liquid–liquid extraction

liquid–liquid partition

Process of transferring a dissolved substance from one liquid phase to another (immiscible or partially miscible) liquid phase in contact with it.

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Note: Although extraction, partition, and distribution are not synonymous, extraction may replace distribution where appropriate. Liquid-liquid extraction may be used in place of liquid–liquid distribution when the emphasis is on the *analyte*(*s*) being distributed (or extracted).

Reference: [4].

3.2.15 liquid-phase microextraction

Microextraction technique using a liquid as the *extraction phase*.

3.2.16 multistage countercurrent distribution

Multistage countercurrent separation based on a discontinuous, differential migration process employing stepwise extraction.

3.2.17 single-drop microextraction (SDME)

Extraction of *analytes* from the gas phase or solution by a single drop of extraction solvent.

Note 1: The extraction solvent with a volume typically <50 μL is suspended as a drop from the tip of a microsyringe needle as shown in Fig. 7.

Note 2: Typically, the sample is aqueous and the acceptor phase is an organic solvent, resulting in a two-phase extraction system (aqueous → organic). SDME can also be operated in three-phase mode (aqueous → organic → aqueous), or in the headspace mode (aqueous → gas → organic or aqueous) [26].

Note 3: The sample is stirred or agitated during extraction. After extraction, the drop of acceptor phase is collected by withdrawing it into the syringe used to support the drop for the final analytical measurement.

Reference: [27].

3.2.18 solidified floating organic drop

Droplet of organic solvent recovered from the surface of the sample solution by solidification induced by cooling.

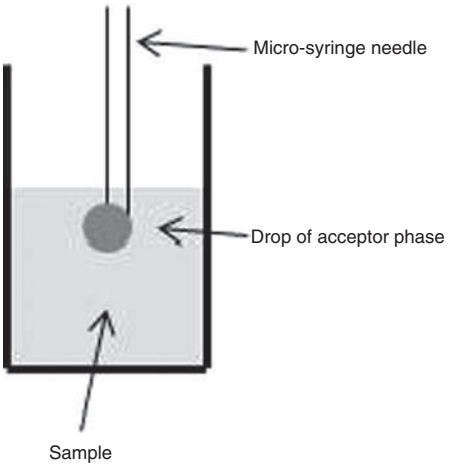

Fig. 7: Typical experimental arrangement for single-drop microextraction.

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3.2.19 solidified floating organic drop microextraction (SFODME)

Extraction employing a solidified floating organic drop.
Note: The extraction solvent has a melting point near room temperature to facilitate collection after solidification induced by cooling. The solidified solvent is then melted and analysed [28].
Reference: [28].

3.2.20 stage (liquid–liquid extraction)

The physically distinct part of an extraction process in which transfer of *analyte*(*s*) occurs, followed by phase separation.
Reference [4].

3.2.21 stagewise contactor

Type of continuous multi-stage *liquid–liquid extraction* equipment in which each stage has a physically distinct cycle of interphase contact and separation.
Note: There are the same number of phase separation interfaces as there are stages.
Adapted from reference: [4].

3.2.22 two-phase aqueous partition

Distribution in a biphasic system formed by two aqueous solutions containing structurally incompatible components (two polymers or a polymer and a salt) above a critical concentration.
Reference: [29].

3.3 Extraction of solids by a fluid

3.3.1 matrix-solid phase dispersion (MSPD)

Extraction in which samples are blended with an abrasive solid *sorbent*, forming a free flowing powder from which target *analytes* are recovered by solvent elution.
Note 1: A typical workflow scheme for matrix–solid-phase dispersion is shown in Fig. 8 [30].
Note 2: For additional sample clean-up the sorbent/sample mixture is deposited at the head of a solid-phase extraction cartridge containing a suitable sorbent to retard the elution of *matrix* components.
Reference: [30].

3.3.2 microwave-assisted extraction (MAE)

Leaching using microwave energy to heat the solvent in contact with the sample or the sample directly.
Note 1: Microwaves are electromagnetic radiation of 300–300,000 MHz. They heat the extraction solution by their interaction with dielectric compounds (solvent or sample). Microwave heating is more efficient and rapid than convection and conduction and enhances the migration rate of compounds into the extraction solvent. Microwave-assisted extraction may be performed in a closed vessel with control of temperature and pressure or in an open vessel at atmospheric pressure [31].
Reference: [31].

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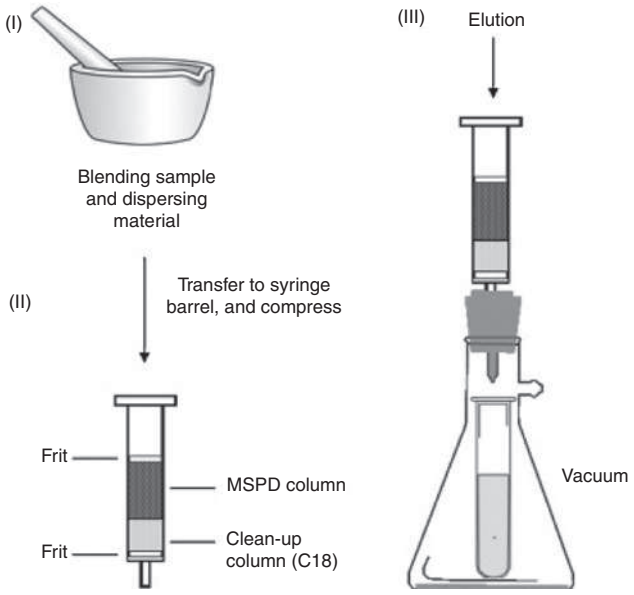

Fig. 8: Typical work flow scheme for solid-phase matrix dispersion. (i) Sample is blended with an abrasive sorbent, (ii) free flowing powder containing sample is packed in a short column, and (iii) target analytes are recovered by elution.

3.3.3 pressurized hot-water extraction

subcritical water extraction
superheated water extraction
Extraction by water below its critical point (374.15 °C and 22.064 MPa).
Note 1: The solvation properties of water become closer to organic solvents at elevated temperatures [32].
Note 2: Instrument requirements are similar to *pressurized-liquid extraction*.
Reference: [32].

3.3.4 pressurized-liquid extraction

Leaching performed at elevated temperature and pressure.
Note 1: A typical apparatus is shown in Fig. 9 [33].

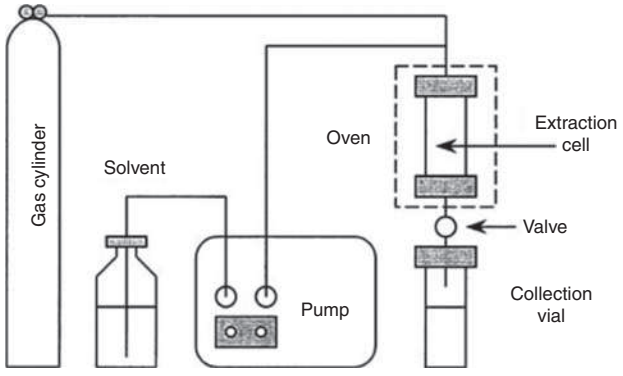

Fig. 9: Schematic diagram of a typical apparatus for pressurized-liquid extraction. Gas flow is used to displace the extraction solvent from the sample chamber.

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Note 2: The sample, in the form of a fine powder or liquid supported on an inert solid, is packed into an extraction vessel and inserted in a flow-through system. The temperature, pressure, solvent type, and static and/or dynamic extraction time are varied to optimize the extraction performance.
Reference: [33].

3.3.5 Soxhlet extraction

Leaching with fresh solvent cycled multiple times through a fine solid sample contained in a porous thimble.
Note 1: A typical apparatus for classical Soxhlet extraction is shown in Fig. 10.
Note 2: The extraction solvent is cycled in a continuous-discrete process by distillation, condensation and siphoning. Extracted material accumulates in the distillation flask.
Reference: [34].

3.3.6 supercritical fluid extraction (SFE)

Use of a supercritical fluid to extract *analytes* from a solid or supported liquid sample.
Note 1: The powdered sample, or supported liquid, is packed into a vessel and extracted using static or dynamic extraction conditions. The extracted material is typically recovered by reducing the pressure of the extraction fluid, allowing it to evaporate [34].
Note 2: The most common supercritical fluid for extraction is carbon dioxide (critical point = 39.9 °C and 73.8 bar). Its extraction properties are varied by changing its density (temperature and pressure) or by addition of polar organic solvents [35].
References: [35, 36].

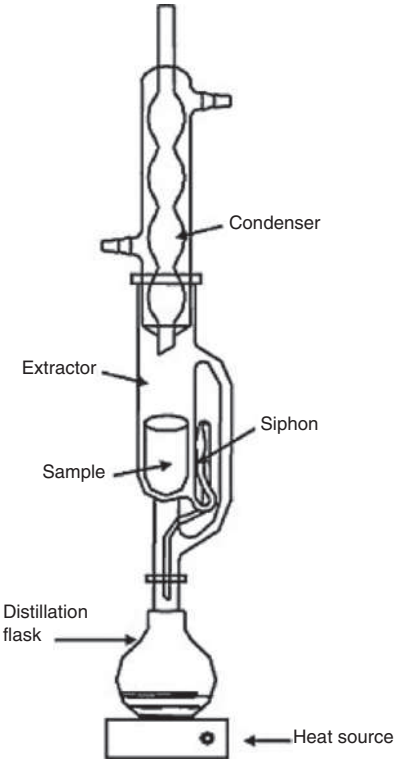

Fig. 10: Typical apparatus for classical Soxhlet extraction.

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3.3.7 QuEChERS (Quick, Easy, Cheap, Effective, Rugged and Safe) extraction

Streamlined multiresidue method for *extraction* and clean-up of *analytes* in complex *matrices*.
Note 1: Often used for samples containing pesticide residues [37].
Note 2: The general procedure includes micro-scale extraction with acetonitrile, salt-assisted phase separation, and sample clean up using dispersive solid-phase extraction.
Note 3: There are several procedural variations of the general method for application to particular compounds or sample matrices.
Reference: [37].

3.3.8 ultrasound-assisted extraction (UAE)

Extraction in which ultrasound energy is used to increase the extraction rate by enhancing the contact area between the sample and solvent and by promoting efficient agitation.
Note 1: Ultrasound energy is applied to samples in a vessel immersed in a water bath or applied more directly using devices, such as horns, probes, or sono-reactors [38].
Note 2: Ultrasound energy causes cavitation in solvents, which generates numerous tiny bubbles in liquids and mechanical erosion of solids, including particle rupture. It promotes digestion and slurry formation with solids and homogenization and emulsification with liquids.
Reference: [38].

3.4 Digestion methods

3.4.1 acid digestion

Process in which the sample (*analyte* and *matrix*) is dissolved by an acid.
Note 1: Acid digestion can also be used to remove a matrix constituent by selective volatilization, e.g. silicon by the use of hydrofluoric acid.
Note 2: Acid digestion can be performed in closed or open vessels.
Reference: [39].

3.4.2 ashing (chemical analysis)

Dry or wet *matrix* decomposition as a method of preconcentration of trace elements.
Related term: *charring*.
Reference: [12].

3.4.3 bomb acid digestion

Type of closed-vessel *acid digestion*.
Note: Materials which are not fully dissolved by acid-digestion at atmospheric pressure may require a more vigorous treatment in a pressure vessel lined with polytetrafluoroethylene (PTFE) glass, silica, or vitreous (glassy) carbon, or in sealed silica tubes; this treatment is called bomb digestion. The sample and acids are heated in a closed vessel so that the digestion is carried out at higher temperature and pressure.
Reference: [39].

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3.4.4 charring

Pyrolysis of samples containing organic matter.
Reference: [40].

3.4.5 closed-vessel acid digestion

Type of *acid digestion* performed in a closed vessel at elevated pressure.
Note: The increased rate of decomposition and retention of volatile trace elements is an advantage over open-vessel acid digestion.
Reference: [39].

3.4.6 digestion (sample preparation)

Sample dissolution with simultaneous *matrix* simplification.

3.4.7 enzymatic decomposition

Decomposition of organic materials by the actions of an enzyme whereby high molecular mass compounds are converted into lower molecular mass species.
Reference: [39].

3.4.8 fusion

Chemical process by which a sample is transformed into a glass.
Note: Fusion is commonly employed in XRF analysis. It typically consists of four steps: (1) mixing the sample with a fusion reagent and occasionally with an oxidant, in a crucible; (2) heating the crucible to a temperature where the fusion reagent melts; (3) agitating the crucible until the sample has dissolved; and (4) pouring the molten glass into a hot mould to form a flat glass disk of a given diameter after cooling, ready for measurement in the spectrometer. Alternatively, the molten glass can be poured into a swirling warm acid solvent near ambient temperature for dissolution.
Reference: [41].

3.4.9 microwave acid digestion

Type of *bomb acid digestion* with microwave radiation used for sample heating.

3.4.10 open-vessel acid digestion

Type of *acid digestion* performed at ambient pressure wherein the digestion temperature is limited by the boiling point of the acid.

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3.4.11 oxygen-flask combustion

Type of *ashing* in which the sample is burned in a closed flask containing oxygen and an absorbing solution, which is subsequently analysed.
Reference: [39].

3.4.12 partial digestion

pseudodigestion
Digestion in which only a part of the sample, or some of the *analytes* present, are brought into solution.
Note: Partial digestion may be preferred over total decomposition if the relative analyte concentrations in the samples provides sufficient information (e.g. materials for geochemical exploration).
Reference: [39].

3.4.13 UV photochemical digestion

Formation of free metal ions in aqueous solution by oxidation under the influence of UV light and possibly an oxidizing agent (e.g. hydrogen peroxide or peroxodisulfate) in order to break down dissolved organic matter and metal complexes.
Note: Typical samples are sea water, waste water, drinking water, or fresh water.
Reference: [39].

3.5 Solid-phase extraction

3.5.1 cartridge

Short disposable column containing a bed of porous particles immobilized between two porous frits.
Note: The particles are sized to allow sample processing by gravity, gentle suction or low pressure.
References: [42, 43].

3.5.2 conditioning solvent

Small volume of solvent used to prepare the sorbent for efficient sample processing.
Note: Principal roles include removal of contaminants from the sorbent bed' to create the conditions for a stable flow of the sample solution during sampling and to facilitate wetting of hydrophobic sorbent surfaces before applying aqueous samples.

3.5.3 elution solvent

Small volume of strong solvent used to displace target *analytes* from the sorbent bed.

3.5.4 foamed polyurethane sorbent

Sorbent consisting of spherical, micron-sized, agglomerated particles bound together in a rigid and highly porous structure used for the extraction of semi-volatile organic compounds from air in high-volume samplers.
Note 1: Their main attribute is their favourable aerodynamic properties, which permit sampling at high flow rates.
Note 2: Foamed polyurethane sorbents have a minor use for extracting compounds from water.

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3.5.5 immunosorbents

immunoaffinity sorbents
Porous material with surface bound monoclonal or polyclonal antibodies capable of selective extraction of target *analytes* by formation of reversible antibody-antigen (compound) binding complexes.

Note 1: A high level of selectivity is possible due to the specificity of antibody-antigen spatial fitting and interactions.

Note 2: The porous support is chosen to minimize nonspecific *matrix* interactions to facilitate processing complex biological, environmental and food samples without additional clean up.

3.5.6 inorganic oxide adsorbent

Adsorbent with site-specific polar and ion exchange functional groups that extract *analytes* from a gas or liquid phase by their complementary interactions with the surface.

Note 1: Typical inorganic oxide adsorbents include silica gel, alumina, magnesium silicate (Florisil), and zirconia.

Note 2: Compounds with hydrogen-bonding or ionic functional groups are extracted efficiently, while neutral dipolar compounds are extracted to a lesser extent and weakly polarizable compounds to a limited extent.

3.5.7 ion-exchange sorbent

Porous material containing fixed ionic sites with a complementary charge to that of the target *analyte*(*s*).

Note 1: Ionic or ionizable compounds are extracted almost exclusively from water by electrostatic interactions with ion-exchange sorbents.

Note 2: Ion-exchange sorbents are usually classified as weak or strong depending on the identity of the ionic group and whether its charge is independent of the sample pH (strong ion-exchanger) or can be varied by changing the sample pH (weak ion-exchanger).

3.5.8 low-specificity sorbents

Sorbent with a non-selective surface used almost exclusively to extract *analytes* from an aqueous solution. The sorbent has a largely passive role in the extraction, with the driving force being the expulsion of the organic contaminants from water to minimize disruption of the water structure.

Note 1: Typical low-specificity sorbents are chemically-bonded porous silica, hydrophobic macroporous polymers, and various forms of carbon.

Note 2: Low-specificity sorbents have surfaces with a low concentration of polar functional groups and retain compounds mainly through non-selective dispersion interactions.

Note 3: From the gas phase compounds are extracted by non-polar and weakly polar interactions with high surface area sorbents.

3.5.9 magnetic nanoparticle sorbent

Sorbent consisting of nanometre-sized, spherical particles with a magnetic or magnetisable core surrounded by a sorbent shell.

Note: Magnetic nanoparticles have a high surface area-to-volume ratio and a low mass. They are easily dispersed in solution. A magnet facilitates recovery of the nanoparticles from the sample solution or suspension [44].

Reference: [44].

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3.5.10 microextraction by packed sorbent (MEPS)

Solid-phase extraction using a syringe with a short *sorbent* bed located between the plunger and the needle.
Note 1: Typically based on a 100- or 250-µl volume syringe with a packed bed containing 1–2 mg of sorbent.
Note 2: The MEPS syringe facilitates low-dead volume sample processing by vertical movement of the plunger.
Reference: [45].

3.5.11 mixed-mode sorbent

Porous material with surface co-bonded ion exchange and alkyl functional groups.
Note: The dual extraction mechanism allows fractionation of mixtures into a neutral and acidic fraction and a basic fraction by a stepwise change in pH of the eluting solvent.

3.5.12 molecularly imprinted polymer sorbent (MIPS)

Porous polymer *sorbent* with synthetic recognition sites with a high specificity for the sorption of a targeted compound or group of structurally-related compounds.
Note: The imprint is obtained by the co-polymerization of functional and crosslinking monomers in the presence of a template molecule (the *analyte*) and a porogen. The resultant imprint possesses a steric (size and shape) and chemical (spatial arrangement of complementary functional groups) memory of the template molecule that enable the sorbent to selectively rebind the template molecule from a complex *matrix*.

3.5.13 multiwell extraction plate

Regular array of wells in a single moulded body with a short *sorbent* bed or disc embedded at the base of each well.
Note 1: The design of a typical multiwall extraction plate is shown in Fig. 11.
Note 2: Multiwell plates typically contain 24, 96, 256, or 384 wells with volumes varying from about 0.1 to 10 ml. They are used for parallel sample processing in high throughput applications.
Reference: [46].

3.5.14 needle trap

Gas-tight syringe with a *sorbent*-filled needle.
Note: Vertical movement of the plunger allows bidirectional flow of sample through the sorbent bed (active sampling). Alternatively, sampling can occur by diffusion of *analytes* into the syringe needle (passive sampling).
Reference: [47].

3.5.15 particle-embedded glass fiber disc

Suspension of micron-sized *sorbent* particles woven into a glass fiber *matrix*.
Note: Small discs are self-supporting while larger discs require external support.

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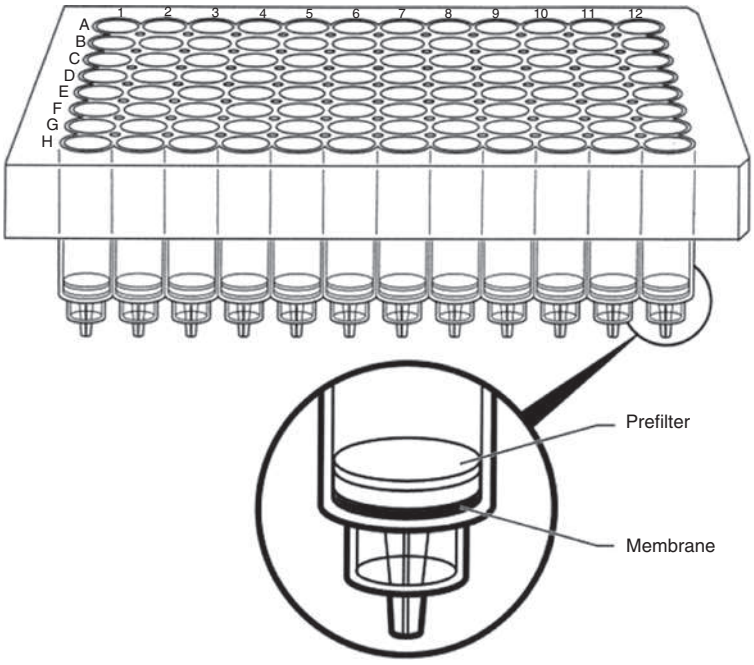

Fig. 11: Multiwell extraction plate.

3.5.16 particle-loaded membrane disc

Suspension of micron-sized *sorbent* particles distributed throughout a web of poly(tetrafluroethylene) microfibrils formed into a thin disc of various diameters.

Note: For general use the flexible discs are supported on a sintered glass disc in a filtration apparatus using suction to generate the desired flow through the membrane.

3.5.17 restricted-access sorbent

Porous material with a biocompatible outer surface and a retentive interior surface.

Note 1: Access to the retentive interior surface is restricted for macromolecules by a physical or chemical diffusion barrier that facilitates elution of macromolecules and extraction of low-mass compounds.

Note 2: The restricted access barrier can be accomplished by size exclusion, differential partitioning, or other phenomenon.

3.5.18 rinse solvent

Weak solvent used to displace *matrix* components from a *sorbent* without displacing the target *analyte*(*s*).

Note: An optional step, intermediate between sample application and elution.

3.5.19 solid-phase extraction

Extraction of *analytes* from a gas, liquid, or fluid by transfer to a solid *sorbent*.

Note: Typical uses of solid-phase extraction include isolation, concentration, solvent exchange, and *matrix* simplification.

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3.5.20 solvent desorption

Use of a solvent, typically combined with agitation, to remove target *analytes* from a *sorbent*.

3.5.21 sorbent-containing pipette tip

A disposable pipette tip with a small packed bed, disc, or monolithic plug of *sorbent* at its narrow end.

Note 1: A typical design of sorbent-containing pipette device for high throughput sample processing is shown in Fig. 12 [48].

Note 2: A micropipettor is used to control sample processing steps. This arrangement facilitates the use of bidirectional flow (the cycling of sample and solvents across the sorbent bed in either direction) for sample processing.

Reference: [48].

3.5.22 sorptive-tape extraction

Extraction from the surface of a solid by *sorbent* or polymer film in the form of a tape in direct contact with, or in the *headspace* above, the sample.

Note: The tape is typically a suitably-sized sheet of poly(dimethylsiloxane) polymer, or a piece of particle-loaded membrane, with a large surface area-to-volume ratio.

3.5.23 stir bar sorptive extraction

Extraction of compounds from solution by a *sorbent* coated magnetic stir bar by rapid stirring (direct immersion) or by exposure to the *headspace* above a sample (*headspace extraction*).

Note 1: Related devices for simultaneous stirring and sampling include rotating disc, stir rod, and stir membrane devices [49].

Note 2: Rapid stirring accelerates mass transfer overcoming some of the limitations of diffusion controlled mass transfer in sessile solutions.

Reference: [49].

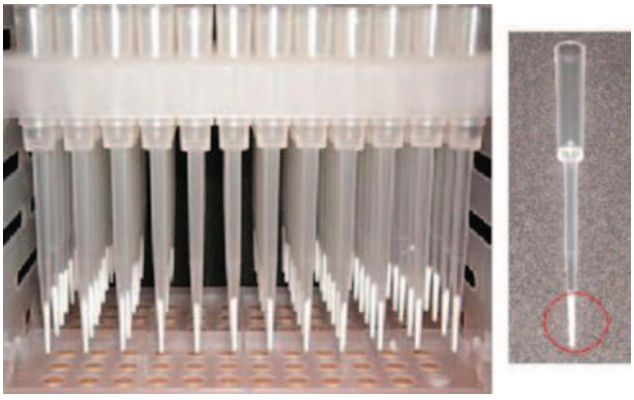

Fig. 12: Sorbent containing pipette tips in an array for use with a multiwall sample plate.

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3.5.24 surface-bound macrocyclic sorbent

Porous sorbent with surface bound macrocyclic ligands designed for the extraction of metal ions and some anions from aqueous solution.

Note 1: The selectivity of the extraction process depends on the stability of the host-guest complex, which can be optimized for individual ions by the choice of the macrocyclic cavity and the type of donor atoms.

Note 2: Extracted ions are recovered by elution with a solution of complexing agent with a higher binding constant than the surface-bound macrocyclic ligand.

3.5.25 surface-bound phenylboronic acid sorbent

Porous sorbent with surface-bound phenylboronic acid groups for the selective extraction of bifunctional compounds by reversible formation of 5- or 6-membered covalent cyclic boronic acid complexes.

Note: Extraction occurs at a basic pH for compounds with hydrogen-donating functional groups on neighbouring carbon atoms (1,2- and 1,3-). The *analytes* are released by elution with an acidic aqueous-organic solvent solution.

3.6 Solid-phase microextraction techniques

3.6.1 biocompatible solid-phase microextraction

Extraction by a *matrix*-compatible immersion solid-phase *microextraction* device where the extraction phase and support are chosen to eliminate matrix interferences.

3.6.2 blade format

Solid-phase microextraction device where the extraction phase (*sorbent*) is dispersed in the form of a thin coating on the surface of a blade.

Note: This technique is typically used as a 96-blade brush in conjunction with a *multiwell sample plate*.

3.6.3 coated vessel format

Sample vessel with sorbent-coated walls.

3.6.4 cold fiber solid-phase microextraction

Solid-phase microextraction in the *fiber format* with a sorbent temperature maintained below that of the sample matrix during extraction.

3.6.5 fiber format

Extraction phase in the form of a thin layer supported on a fiber made of inert material, such as fused silica, stainless steel, or nickel–titanium alloy.

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3.6.6 headspace solid-phase microextraction

Solid-phase microextraction where the *extraction phase* is placed in the *headspace* above the sample.

3.6.7 immersion solid-phase microextraction

Solid-phase microextraction where the *extraction phase* is immersed directly into the sample.

3.6.8 in-needle capillary adsorption trap (INCAT)

In-tube format device with a *sorbent*-coated capillary placed inside a needle.

3.6.9 in-tube solid-phase microextraction

Extraction utilizing a coated open tubular or packed capillary tube as the extraction phase in a flow system.
Reference: [50].

3.6.10 matrix-compatible solid-phase microextraction

Immersion solid-phase microextraction where the *extraction phase* is designed to minimize sorption of particles or macromolecules present in the sample *matrix*.

Note: Sorption of particles and macromolecules by the extraction phase is a potential source of interference in the extraction process or subsequent determination of analytes when sampling occurs by direct contact of the extraction phase with the sample. In some cases this can be addressed by incorporation of specific properties in the extraction phase that minimize matrix-extraction phase interactions for problem matrix components.

3.6.11 membrane-protected solid-phase microextraction

Immersion solid-phase microextraction in which the extraction phase is surrounded by a permeable membrane that allows access of analytes to the extraction phase while shielding the extraction phase from matrix components with a potential to interfere in the extraction or subsequent analysis.

3.6.12 particle sorbent (microextraction)

Extraction phase in the form of *sorbent* particles.

Note: Particles are typically of a nanometre size and applied in the *dispersion mode of extraction*.

3.6.13 rotating disk sorptive extraction (RDSE)

Sorbent-coated disc consisting of a PTFE base with an integrated magnet and a sorbent film attached to one side.

Note: Facilitates more rapid stirring than conventional sorbent-coated stir bars (see *stir bar sorptive extraction*) without deterioration of the sorbent coating, because it is not in contact with the vessel wall.

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3.6.14 solid-phase dynamic extraction, (SPDE)

Solid-phase microextraction in which the extraction phase is coated on the internal surface of a needle.

3.6.15 solid-phase microextraction (SPME)

Microextraction where the *extraction phase* is a solid.

Note 1: This term is often narrowly used to describe an extraction process employing a syringe-based sampling device with a short length of small gauge solid fiber or wire coated with *sorbent* attached to one end of the plunger of the syringe to facilitate bidirectional movement of the sorbent-coated fiber (wire) through the syringe needle for extraction, storage, and desorption of extractants. For extraction the *sorbent* is exposed to the headspace above the sample or immersed directly into the sample solution. Sample components diffuse into and/or onto the sorbent coating until equilibrium is reached. The extractants are subsequently recovered by thermal desorption into a gas stream (typically in the liner of an injection port of a gas chromatograph) or rinsed from the sorbent coating with solvent for determination by other methods.

Note 2: The extraction phase can also be a liquid physically coated on a fiber or wire support.

3.6.16 thin film format

Solid-phase microextraction where the *sorbent* is in the form of a thin film on a support or self supported *membrane*.

3.7 Membrane extraction

3.7.1 artificial liquid membrane

See *supported liquid membrane*.

3.7.2 dialysis

Process of separation of low molecular weight *analytes* from a *matrix* by a membrane permeable to all components of the system except the high molar mass components, and allowing the exchange of the components of small molar mass to proceed in a continuous fashion.

3.7.3 downstream

Side of a *membrane* from which permeate emerges.
Reference: [51].

3.7.4 electrodialysis

Dialysis in which ions are separated by an electrical potential across the *membrane*.

Note: The membrane is permeable to ions but not neutral compounds.

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3.7.5 electromembrane extraction, (EME)

Hollow-fiber liquid-phase microextraction in which the *extraction* is driven by an electrical potential sustained between the sample solution and the *acceptor phase*, separated by a supported liquid membrane.

Note 1: A typical experimental arrangement is shown in Fig. 13.

Note 2: Electromembrane extraction is typically performed in three-phase mode (aqueous → organic → aqueous) [52].

Note 3: The sample is agitated or stirred during extraction.

Reference: [52].

3.7.6 hollow fiber

Narrow cylindrical tube prepared from a porous polymer.

Note: The internal diameter of the tube is typically 0.5–1.5 mm with walls 100 to 300 μm thick. Fibers are typically prepared from porous poly(propylene), although other materials can be used.

3.7.7 hollow-fiber liquid-phase microextraction (HF-LPME)

Liquid-phase microextraction of *analytes* from a liquid (or gaseous) sample by a supported liquid membrane in the form of a porous *hollow fiber* with solvent-filled pores. The analytes are transferred to an *acceptor phase* contained in the cavity of the hollow fiber.

Note 1: A typical experimental arrangement is shown in Fig. 14.

Note 2: The supported liquid membrane is generally an organic solvent immiscible with water and the sample an aqueous solution.

Note 3: The acceptor phase can be aqueous, which provides a three-phase extraction system (aqueous → organic → aqueous), or an organic solvent, providing a two-phase extraction system (aqueous → organic) [52].

Note 4: The sample is agitated or stirred during extraction.

Reference: [53].

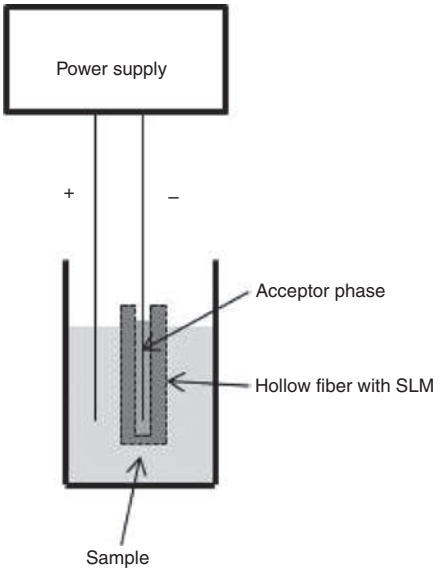

Fig. 13: Typical experimental arrangement for electromembrane extraction of cations (anions extracted by reversing the polarity). SLM, supported liquid membrane in the form of a porous hollow fiber with solvent-filled pores.

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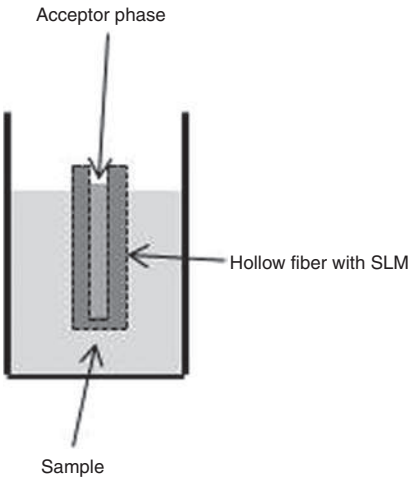

Fig. 14: Typical experimental arrangement for hollow-fiber supported liquid membrane (SLM) for liquid-phase microextraction.

3.7.8 liquid membrane

Liquid phase in either a supported or unsupported form that serves as a *membrane* between two phases.
Adapted from reference: [51].

3.7.9 membrane

Structure having lateral dimensions much greater than its thickness, through which transfer may occur by a variety of driving forces.
Reference: [51].

3.7.10 membrane extraction

Extraction of *analytes* from a sample by permeation through a membrane.

3.7.11 membrane extraction with sorbent interface (MESI)

Membrane extraction with a sorbent trap for concentration of the permeate.
Note: Typically used in a system consisting of three components: (1) membrane extraction module; (2) sorbent interface; and (3) gas chromatography. A flow of gas is used as the stripping phase to transfer permeate to the sorbent trap for concentration and thermal desorption into the gas chromatograph.

3.7.12 microdialysis

Dialysis by a short length of *hollow-fiber membrane* used for sampling.
Note: Microdialysis is performed mostly in a living biological system.

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3.7.13 microporous filtration (microfiltration)

Separation process whereby particles are removed from a sample by application of hydraulic pressure, which forces only particle-free *matrix* to flow through a microporous *membrane*.
Note: Typically the membrane has a pore size down to 0.1 μm.

3.7.14 non-porous membrane

Membrane in which *permeation* occurs by processes other than flow through pores.
Related term: *porous membrane*.

3.7.15 parallel artificial liquid membrane extraction (PALME)

Extraction of *analytes* from aqueous samples in a *multiwell plate* with a porous membrane at the base of each well by migration through the solvent-filled membrane pores into an *acceptor phase* located in each well.
Note 1: The solvent-filled filter pores function as a supported liquid membrane typically based on an organic solvent immiscible with water [54].
Note 2: PALME is usually performed in the three-phase mode (aqueous → organic → aqueous).
Reference: [54].

3.7.16 perfusate

Stream of *extraction phase* entering a *dialysis* module.
Note: Perfusate is also used to describe the stripping phase in *dialysis*.

3.7.17 permeate

Stream containing material that has passed through a *membrane*.
Adapted from reference: [51].

3.7.18 permeation

Process by which mass transfer occurs through a *membrane*.

3.7.19 pervaporation

Membrane-based process in which the *feed* stream and *retentate stream* are both liquid phases with the *analytes* emerging at the downstream face of the membrane as a vapor.
Adapted from reference: [51].

3.7.20 porous membrane

Membrane in which permeation occurs through its porous structure.
Related term: *non-porous membrane*.

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3.7.21 retentate stream

Sample left over in *membrane* separations after initial contact with the membrane.

3.7.22 reverse osmosis

Liquid-phase separation in which pressure applied transmembrane causes selective movement of solvent against its osmotic pressure difference.
Adapted from reference: [51].

3.7.23 semipermeable membrane device (SPMD)

Membrane extraction in which the membrane fully encloses an *acceptor phase*.
Note: Typically, a membrane filled with acceptor phase and formed into a sealed tube is immersed in the sample. As a passive sampling device, it is used to obtain time-weighted average concentrations for trace contaminants or to isolate them for toxicity assessment. With triolein as an acceptor phase, it is used to estimate the bioconcentration of contaminants in fatty tissues of organisms.

3.7.24 stream (membrane separations)

The sample in *membrane* separations prior to contact with the membrane.

3.7.25 stripping phase

Extraction phase in a *membrane* separation.
Adapted from reference: [51].

3.7.26 supported liquid membrane (SLM)

Porous membrane with solvent-filled pores.
Note 1: Porous polymeric *hollow fibers* and filter disks are typically used as support formats for *microextraction* techniques.
Note 2: Water immiscible organic solvents are typically employed.

3.7.27 ultrafiltration

Separation whereby a solution containing a solute of molecular size significantly greater than that of the solvent molecule is removed from the solvent by the application of hydraulic pressure, which forces only the solvent to flow through a suitable *membrane*.
Note: The membrane usually has a pore size in the range 0.001–0.1 μm.
Reference: [7].

3.7.28 upstream (in membrane processes)

The side of a *membrane* into which penetrants enter from the *feed* stream.
Reference: [51].

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3.8 Automated and high-throughput extraction methods-advanced flow technology

3.8.1 at-line extraction

Flow system with extraction device employing a local detector.
Note 1: The *extraction phase* is typically collected in an automated fraction collector and introduced into a detector via a robotic device.

3.8.2 automated extraction

Extraction in a mechanical device regulated by feed-back of information without human intervention.

3.8.3 automatic extraction

Extraction according to manually preselected conditions without further human intervention.

3.8.4 bead injection

Fully renewable (disposable) packed sorbent bed for *solid-phase extraction* in a flow manifold or mesofluidic platform using programmable flow.
Note: A typical workflow for bead injection in a flow manifold is shown in Fig. 15.
Reference: [55].

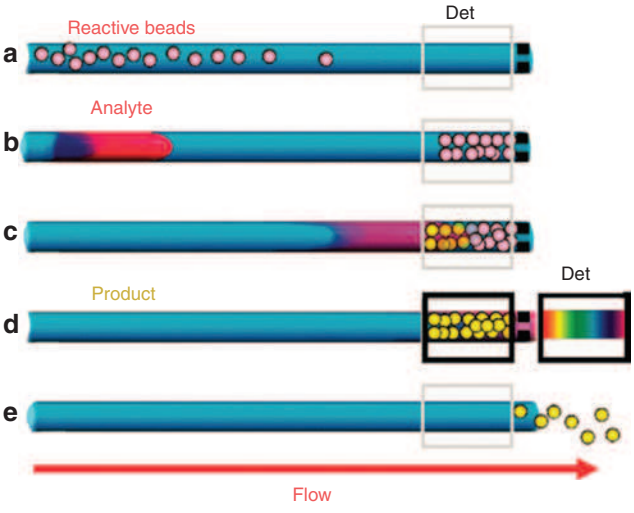

Fig. 15: Bead-injection protocols for automated μSPE with sorbent renewal for each assay [Reproduced from www.flowinjectiontutorial.com with the author's permission (Prof. Ruzicka)].
a) Injection of a metered amount of particles or beads with reactive surfaces in the flow system followed by mechanical trapping or capture with the aid of external energy sources
b) Injection of a metered volume of sample
c) Interaction of analyte(s) with sorptive surfaces
d) Preconcentration of analyte(s) followed by detection of native species or derivatization products
e) Automatic removal of sorptive surfaces.

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3.8.5 bioaccessibility

Fraction of *analytes* desorbed from a sample potentially available to organisms.
Note 1: Bioaccessibility tests are usually performed by operationally defined leaching.
Note 2: Also termed environmental availability.

3.8.6 dynamic bioaccessibility

Leaching of *analytes* from a solid or semi-solid sample in a flow-through device to evaluate their *bioaccessibility*.

3.8.7 flow injection analysis, (FIA)

Analysis of sample by rapid injection into a continuous stream of liquid for extraction, separation, or modification by reaction.
Note: The three cornerstones of FIA are: (i) injection of well-defined sample volumes at the low microliter level; (ii) controllable dispersion, and (iii) precise timing [55].
References: [8, 56].

3.8.8 flow-through extraction

Extraction by flow of sample through or over the *extraction phase*, or vice versa, to enhance the *extraction rate*.
Note: The forced flow of matrix or extraction phase is controlled by an external device.

3.8.9 flow-through solid-phase spectrometry

Continuous optical interrogation of a packed-sorbent bed in a flow system at any stage of the *extraction* process prior to *analyte* elution and *sorbent* regeneration.
Reference: [57].

3.8.10 in-line extraction

Coupling of a flow system with extraction device to a detector without flow interruption.

3.8.11 in-syringe microextraction

Batch-type *extraction* in which the barrel of a syringe pump is used as the device for *liquid-phase microextraction* or dispersive *solid-phase microextraction*.
Reference: [58].

3.8.12 lab-at-valve liquid-phase microextraction

Automatic batch-type liquid-phase *microextraction* by *sequential injection analysis* using an external extraction container (e.g. pipette tip) connected to a multiposition rotary valve.
Reference: [59].

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3.8.13 lab-on-a-chip extraction

Microfluidic platform containing microconduits to facilitate extraction processes with pressure-driven or electroosmotic flow.
Reference: [60].

3.8.14 lab-on-a-valve extraction

Mesofluidic platform containing microconduits for extraction processes mounted on a multi-position rotary valve.
References: [55, 61].

3.8.15 mechanized extraction

Extraction using a device with at least one movable part that replaces or supplements human effort in any (or all) steps of the process.

3.8.16 mesofluidic extraction device

Extraction device with channel widths from 10 μm to 2 mm integrated into a flow platform.
Note: *Lab-on-a-valve* platforms belong to the group of mesofluidic devices.
Reference: [62].

3.8.17 microflow injection analysis (μFIA) extraction device

Miniaturized *flow injection analysis* device for extraction with conduits of a diameter greater than 0.1 mm.

3.8.18 microfluidic extraction device

Extraction device with channel widths from 0.1 to 10 μm integrated into a flow platform.
References: [61, 62].

3.8.19 multicommutation flow extraction

Programmed flow extraction device with microsolenoid valves for processing microliter sample volumes.

3.8.20 on-line extraction

Coupling of a flow system with an extraction device to a detector via a discontinuous interface.
Note 1: A suitable discontinuous interface is a rotary valve with a sample loop.

3.8.21 programmable flow extraction

Multi-bidirectional flow (including stopped flow) of sample and/or *extractant* through an extraction device controlled by a pump.
Reference: [62].

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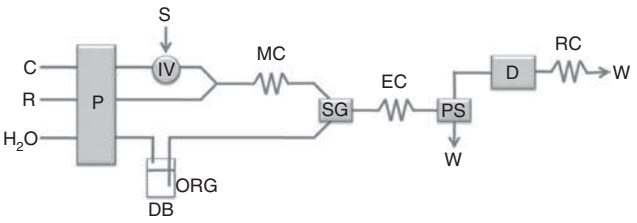

Fig. 16: Typical segmented flow extraction manifold. C, carrier or liquid phase for sample; R, reagent (optional); P, propulsion unit; S, sample; IV, injection valve; MC, mixing coil; DB, displacement bottle (functions as a constant volume pump); ORG, organic solvent; SG, segmenter; EC, extraction coil; PS, phase separator; D, detector (optional); RC, restriction coil; and W, waste.

3.8.22 segmented flow analysis

Continuous-flow system attaining steady-state extraction/reaction conditions facilitated by injection of air bubbles. This might be extended to immiscible liquids injected as droplets in the flow system.
Related term: *segmented flow extraction*.

3.8.23 segmented flow extraction

Extraction by insertion of the sample solution as equal-size droplets (segments) in a flowing stream of immiscible extraction phase.

Note 1: The characteristic components of the system are the segmenter (creates the droplets in the solvent stream); the extraction coil (provides sufficient time for transfer of target *analytes* to the extraction solvent); and the phase separator (reconstitutes the sample solution and extraction solvent into two separate streams [62]).

Note 2: A typical apparatus for segmented flow extraction is shown in Fig. 16.

Reference: [63].

3.8.24 sequential injection analysis extraction device

Automatic programmable flow *extraction* through a multi-position rotary valve housing a *mesofluidic extraction device*.
References: [56, 62].

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