LAB-EXPERIENCE INTRODUCTION

Important knowledges/ requirment

1- Conoscenze di base

2- Attrezzature di laboratorio

3- Come utilizzare le attrezzature di laboratorio

4- Preparazione della soluzione madre

- 1) Preparazione della soluzione da un substrato solido
- 2) Preparazione della soluzione mediante diluizione di una soluzione madre
- 3) Preparazione della soluzione tampone
- 4) Diluizione v/v, diluizione seriale

5- Principio di estrazione degli analiti

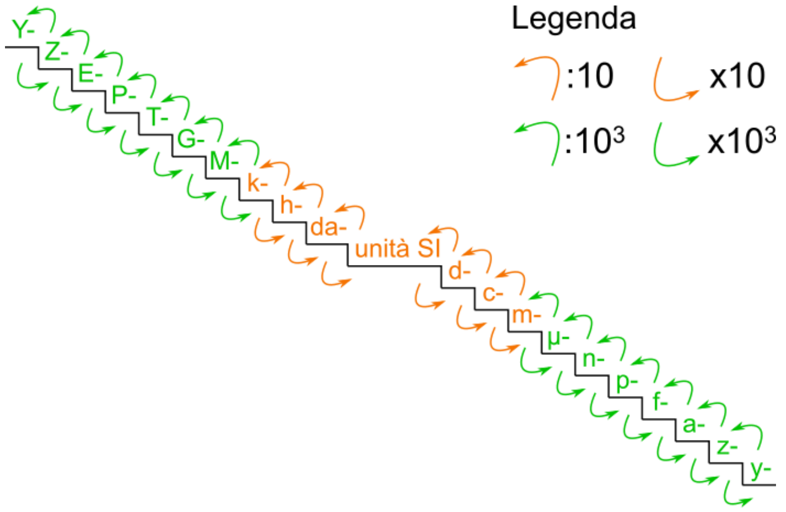
6- Elementi costitutivi di un'analisi

7- Strategie analitiche

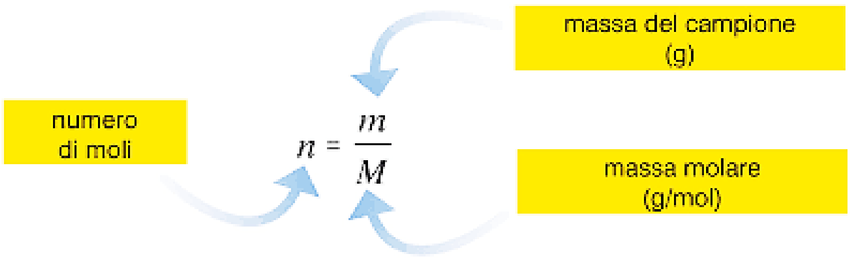
Conoscenze di base

Formulario

SI Base Units		
Physical Quantity	**Name of Unit**	**Abbreviation**
Mass	kilogram	kg
Length	meter	m
Time	second	s
Temperature	kelvin	K
Amount of substance	mole	mol
Electric current	ampere	A
Luminous intensity	candela	cd



Moles and [C]



$$n = \frac{m}{M}$$

$$\text{molarità} = M = \frac{n_{\text{soluto}}\,(\text{mol})}{V_{\text{soluzione}}\,(\text{L})}$$

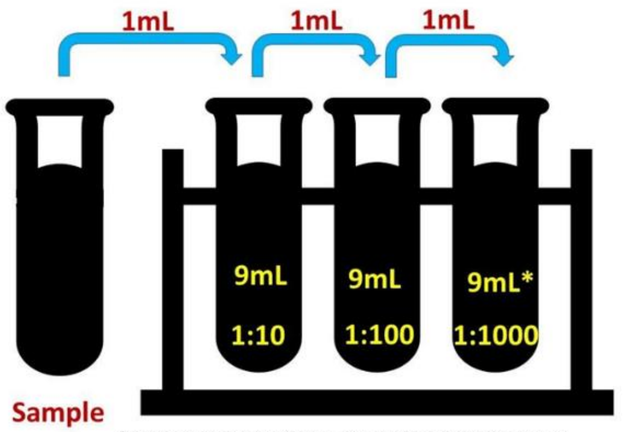
The Dilution Equation

$$M_1V_1 = M_2V_2$$

M_1 = initial molarity ("stock solution")
V_1 = initial volume (Liters)
M_2 = final (desired) molarity
V_2 = final volume (Liters)

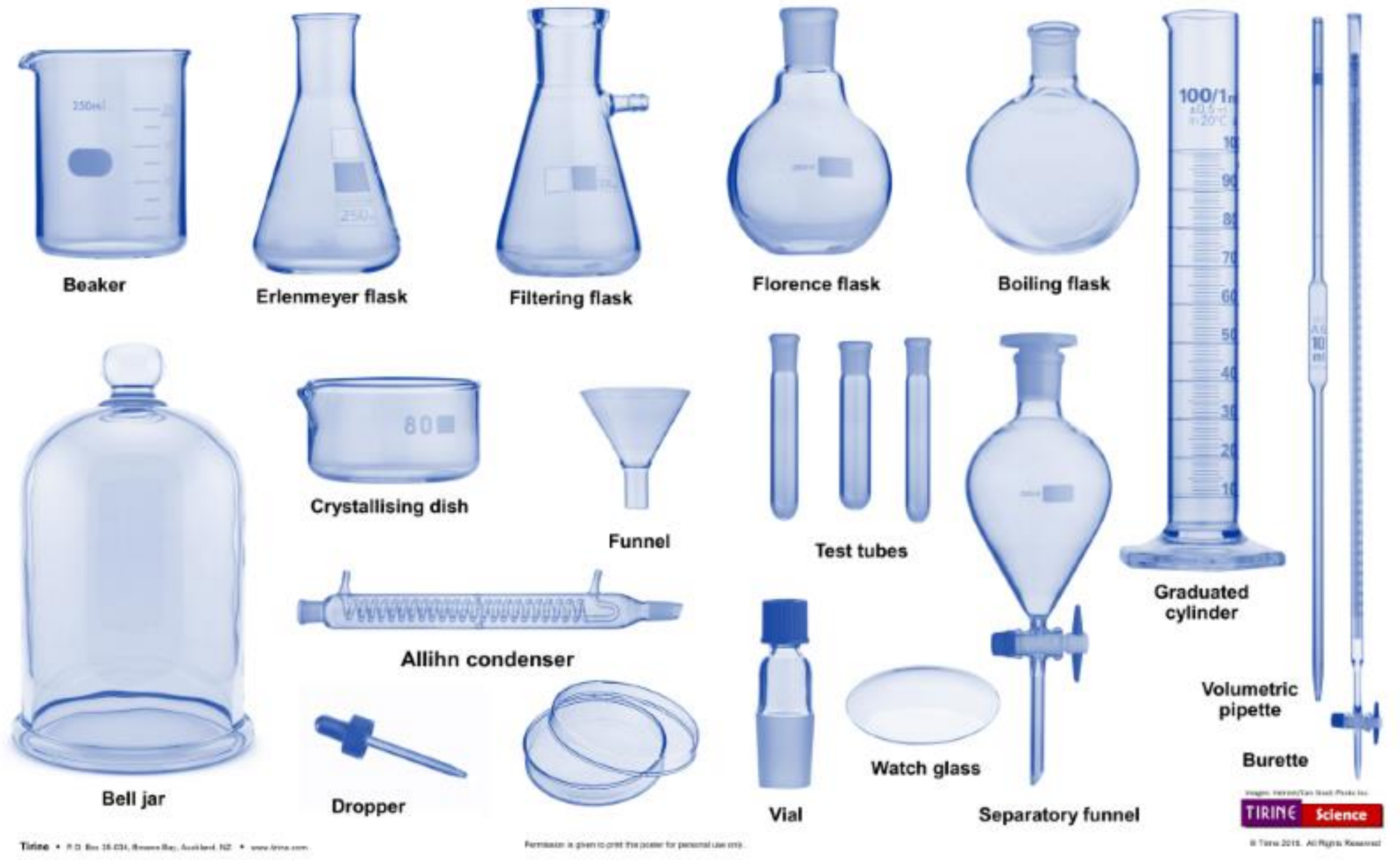
This equation is used when you have a "stock solution" of higher molarity than you need and you need to dilute it to a lower molarity by adding additional solvent.

Diluizione seriale



*Dilution tubes begin with 9mL. 1mL is added, mixed then 1mL is transferred to next tube. The ending volume in last tube would be 10mL

VETRERIA


Beaker
Erlenmeyer flask
Filtering flask
Florence flask
Boiling flask
Graduated cylinder
Bell jar
Crystallising dish
Funnel
Test tubes
Allihn condenser
Volumetric pipette
Burette
Dropper
Vial
Watch glass
Separatory funnel
TIRINE Science

ATTREZZATURA IN PLASTICA

- spruzzetta

- Eppendorf

- Vassoio porta provette

- Falcon

- Essiccatore

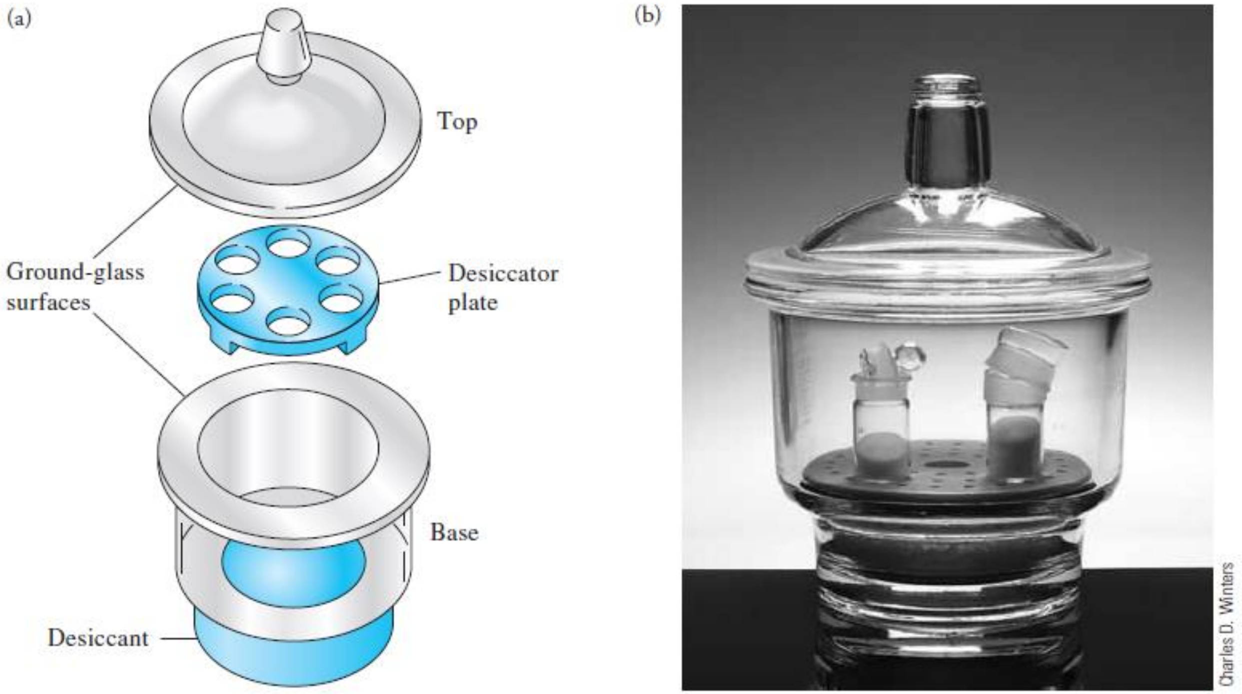
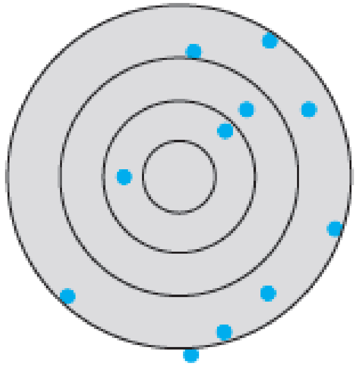
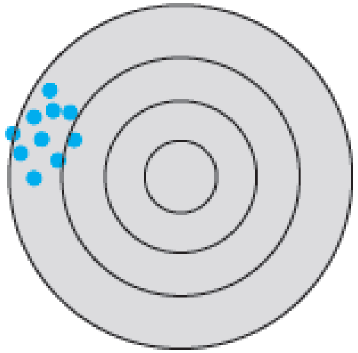


Figure 2-8 (a) Components of a typical desiccator. The base contains a chemical drying agent, which is usually covered with a wire screen and a porcelain plate with holes to accommodate weighing bottles or crucibles. (b) Photo of desiccator containing weighing bottles with dry solids.

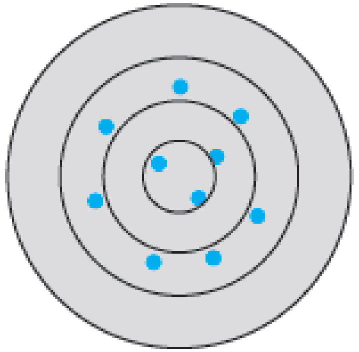
Precise or accurate?



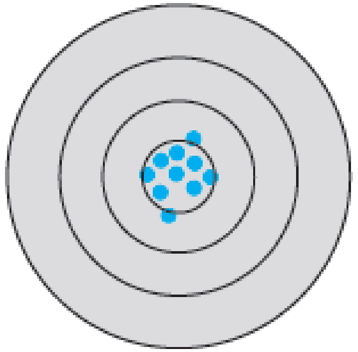
Low accuracy, low precision



Low accuracy, high precision



High accuracy, low precision

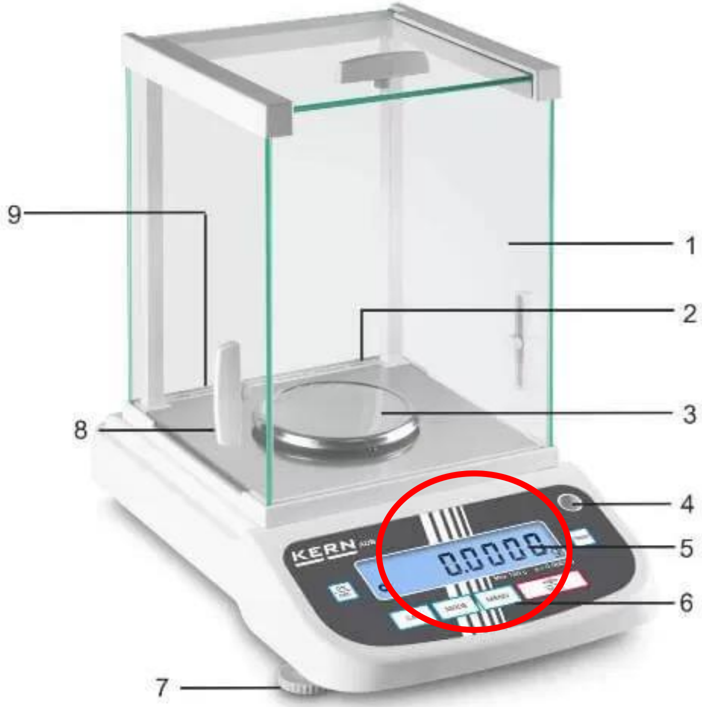


High accuracy, high precision

Cosa significa accuratezza?
Cosa significa precisione?

PESATA

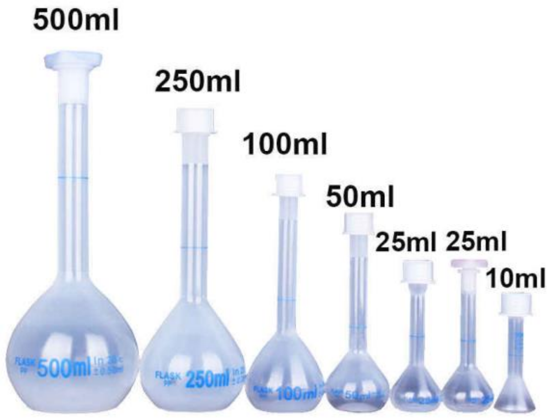
- Bilancia tecnica e analitica



MISURA DEL VOLUME

☐ PALLONE

FARE ATTENZIONE!!!
Parallasse error!



☐ BURETTA

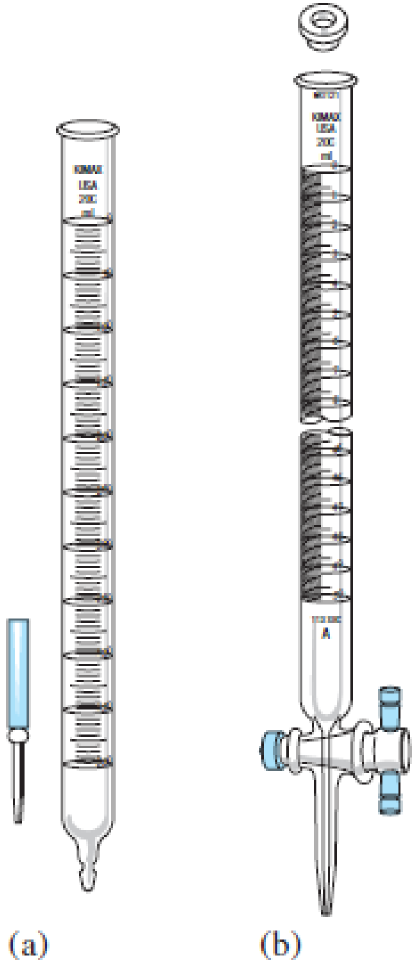
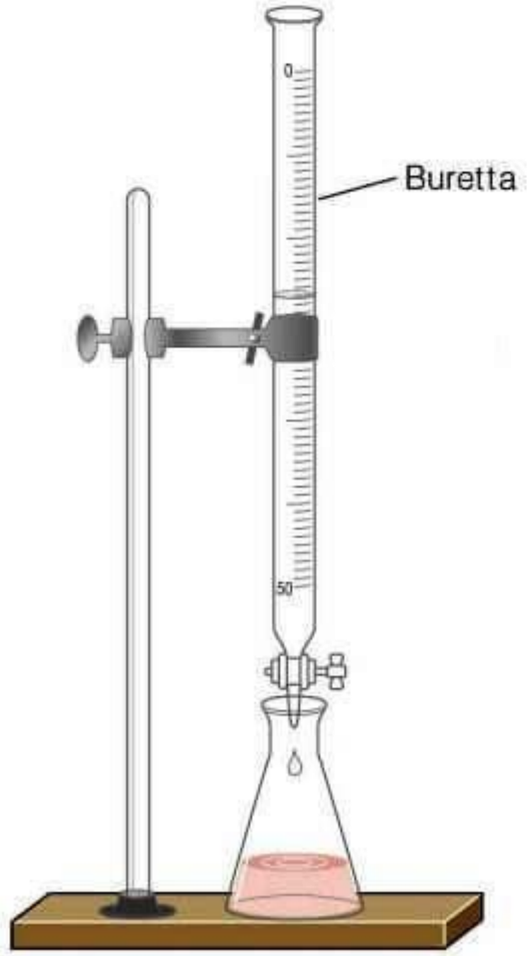
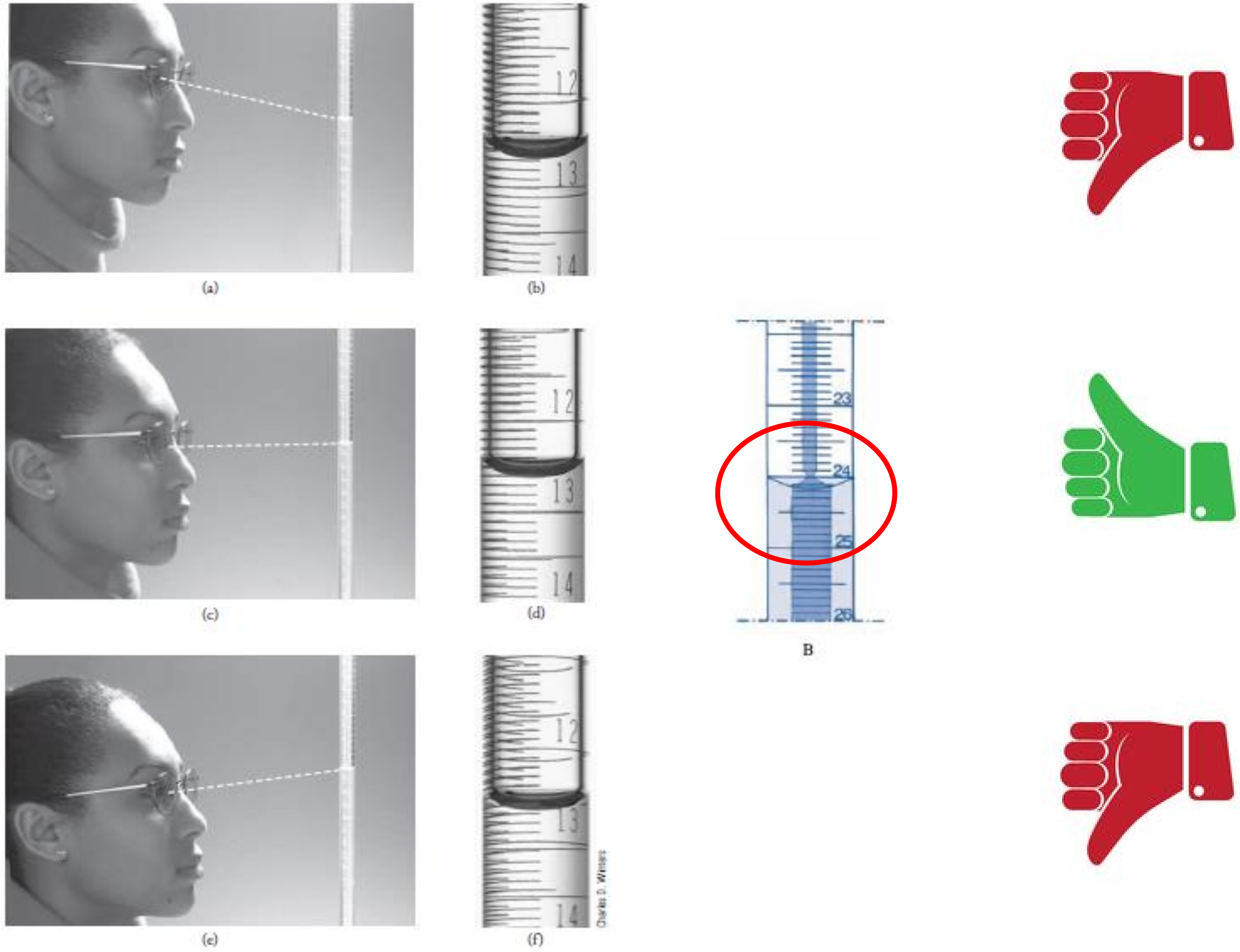


Figure 2-19 Burets:
(a) glass-bead valve,
(b) Teflon valve.

- BURETTA

Fare attenzione all'errore di parallasse





Measuring volume

- Pipette

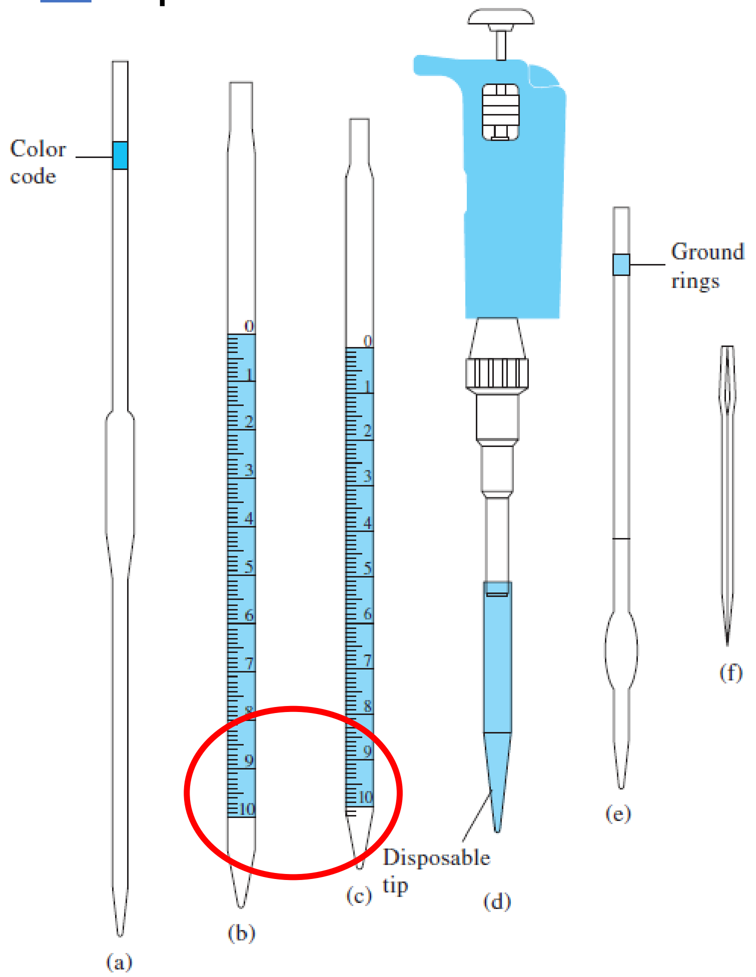


Figure 2-17 Typical pipets: (a) volumetric pipet, (b) Mohr pipet, (c) serological pipet, (d) Eppendorf micropipet, (e) Ostwald–Folin pipet, (f) lambda pipet.

Tolerances, Class A Transfer Pipets

Capacity, mL	Tolerances, mL
0.5	±0.006
1	±0.006
2	±0.006
5	±0.01
10	±0.02
20	±0.03
25	±0.03
50	±0.05
100	±0.08

TABLE 2-2

Characteristics of Pipets

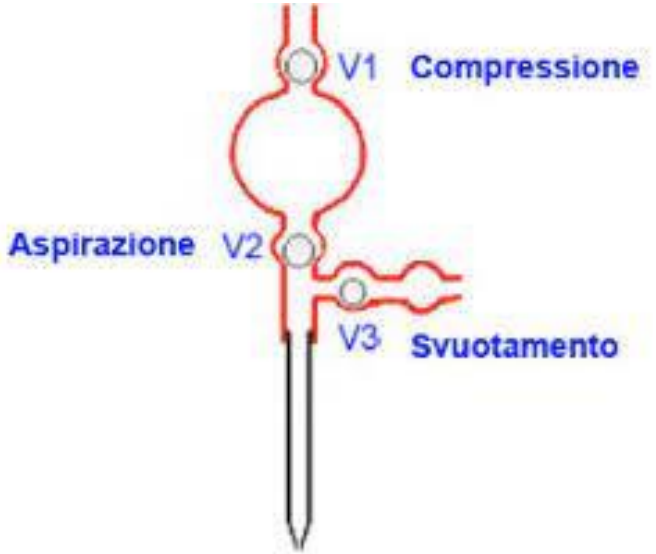
Name	Type of Calibration*	Function	Available Capacity, mL	Type of Drainage
Volumetric	TD	Delivery of fixed volume	1–200	Free
Mohr	TD	Delivery of variable volume	1–25	To lower calibration line
Serological	TD	Delivery of variable volume	0.1–10	Blow out last drop**
Serological	TD	Delivery of variable volume	0.1–10	To lower calibration line
Ostwald-Folin	TD	Delivery of fixed volume	0.5–10	Blow out last drop**
Lambda	TC	Containment of fixed volume	0.001–2	Wash out with suitable solvent
Lambda	TD	Delivery of fixed volume	0.001–2	Blow out last drop**
Eppendorf	TD	Delivery of variable or fixed volume	0.001–1	Tip emptied by air displacement

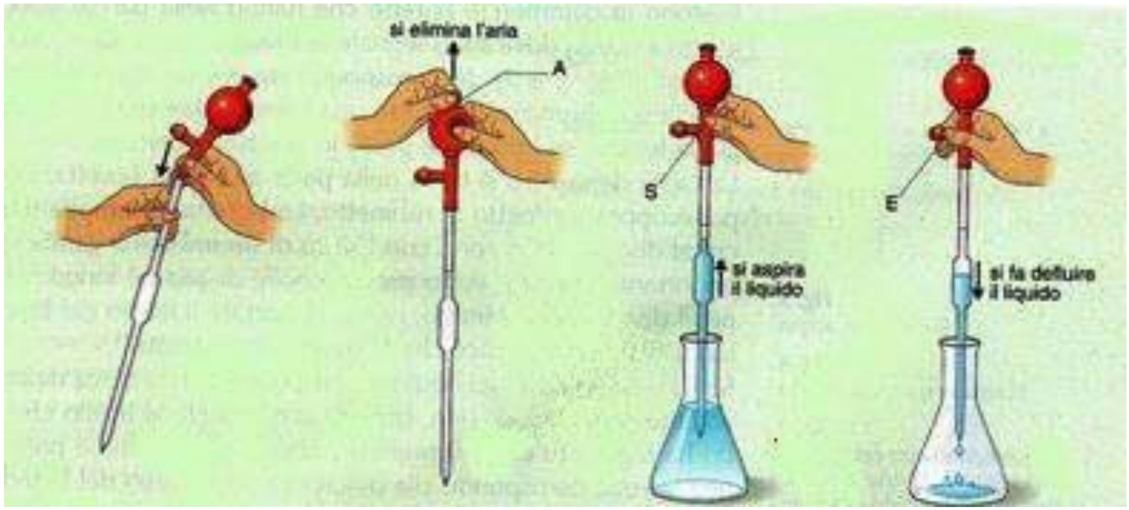
*TD, to deliver; TC, to contain.
**A frosted ring near the top of pipets indicates that the last drop is to be blown out.

MISURA DEL VOLUME

- PIPETTE IN VETRO

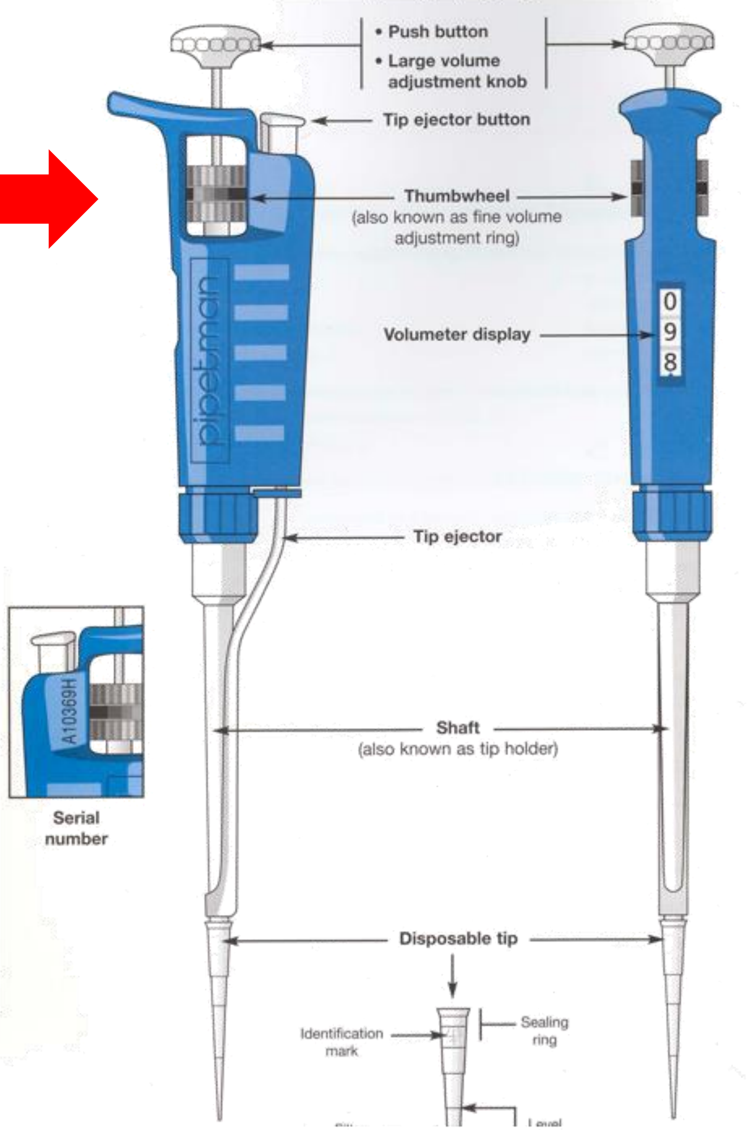
PALLA DI PELEO O PROPIPETTA





- Micropipetta

FARE ATTENZIONE!!!

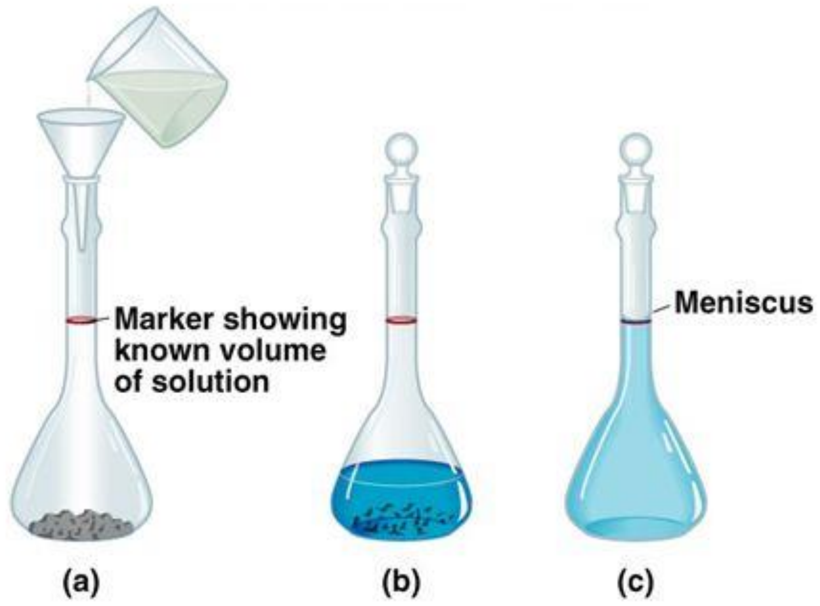


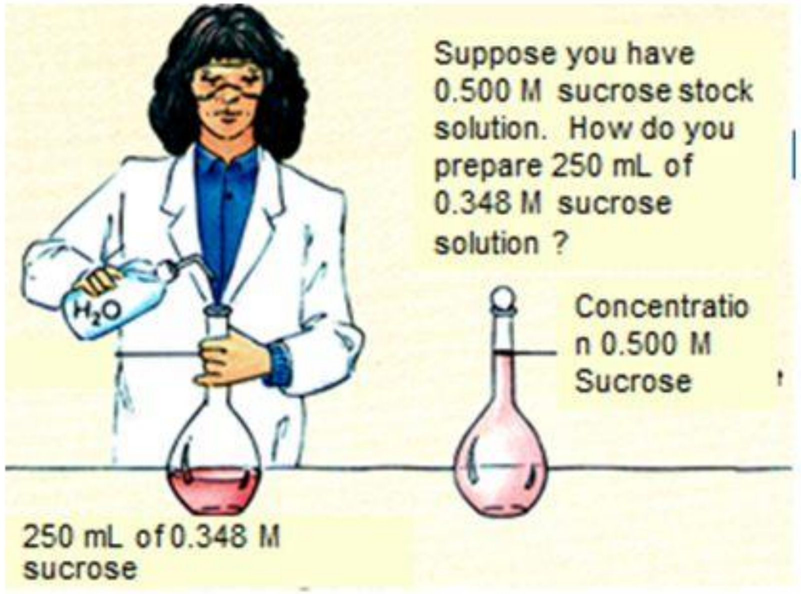
Range and Precision of Typical Eppendorf Micropipets

Volume Range, µL	Standard Deviation, µL
1–20	<0.04 @ 2 µL
	<0.06 @ 20 µL
10–100	<0.10 @ 15 µL
	<0.15 @ 100 µL
20–200	<0.15 @ 25 µL
	<0.30 @ 200 µL
100–1000	<0.6 @ 250 µL
	<1.3 @ 1000 µL
500–5000	<3 @ 1.0 mL
	<8 @ 5.0 mL

Two methods for Preparation of a desired volume of a Molar Solution

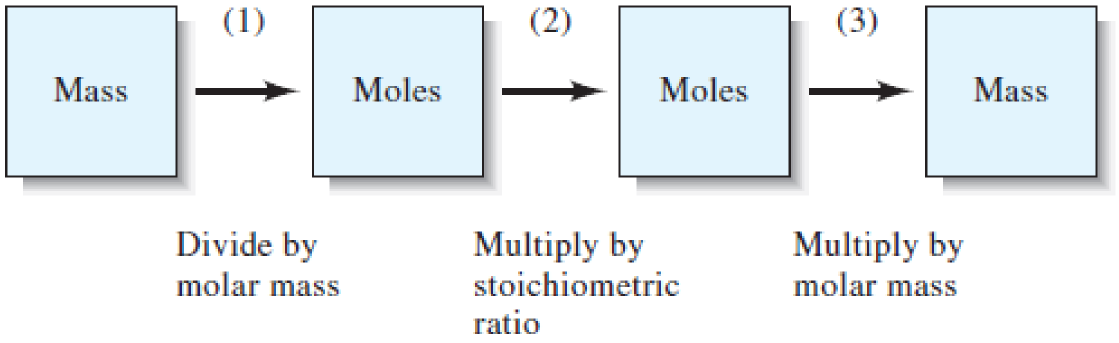
- 1) Preparation from a solid solute.
- 2) Preparation by Dilution of a Concentrated Stock Solution.

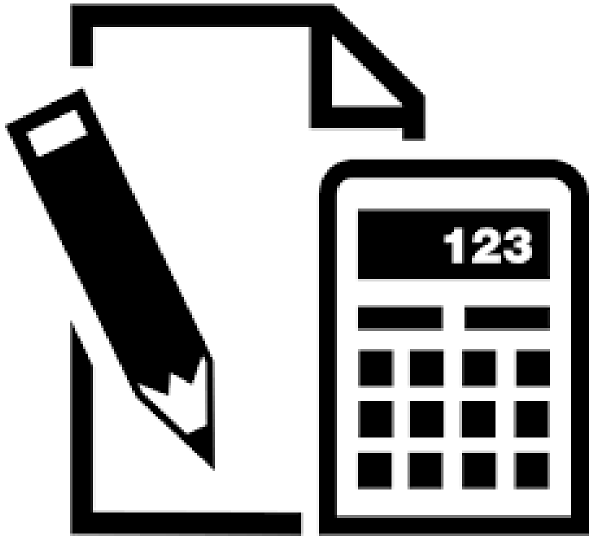




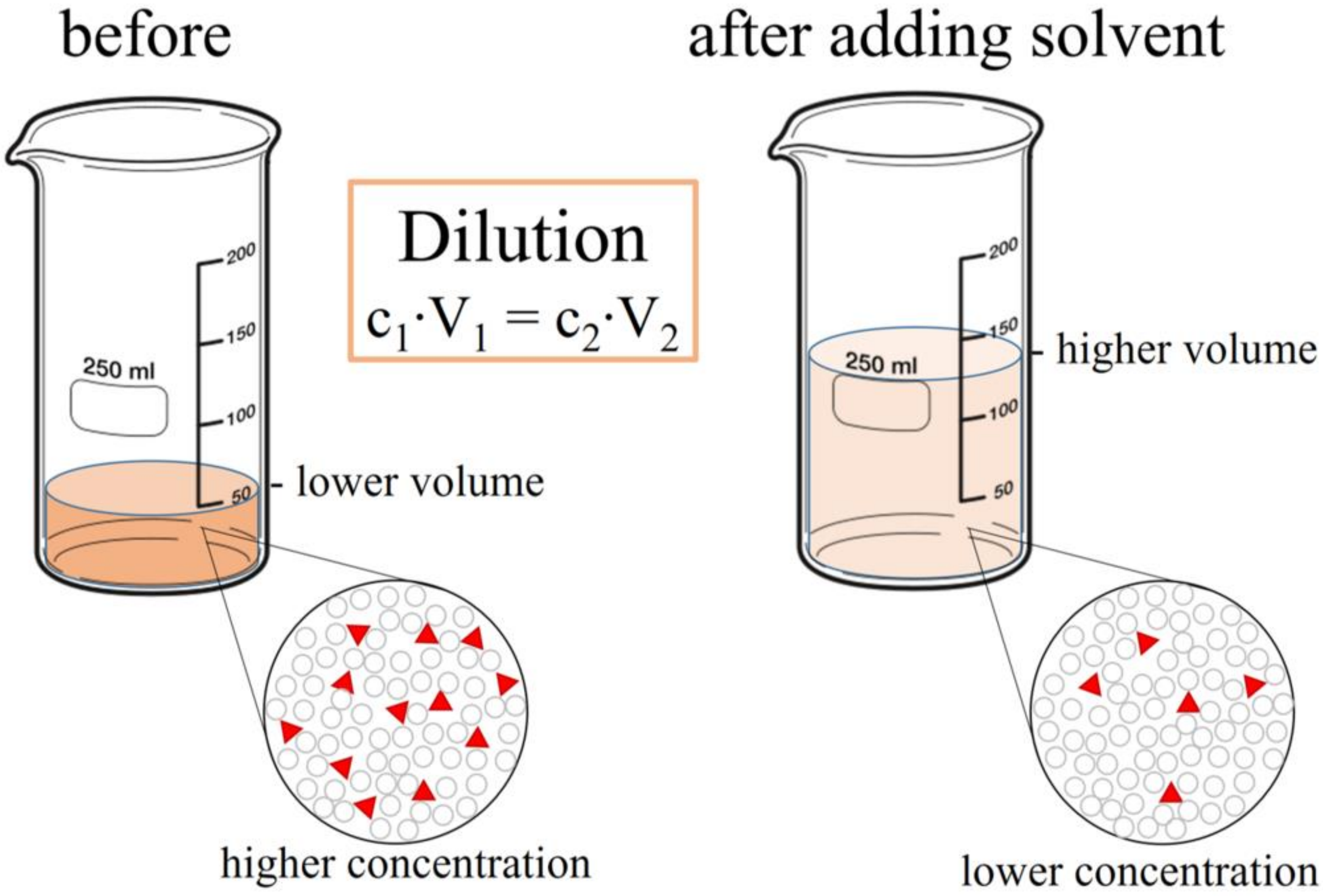
How to solve?

- 1) Preparation from a solid substrate

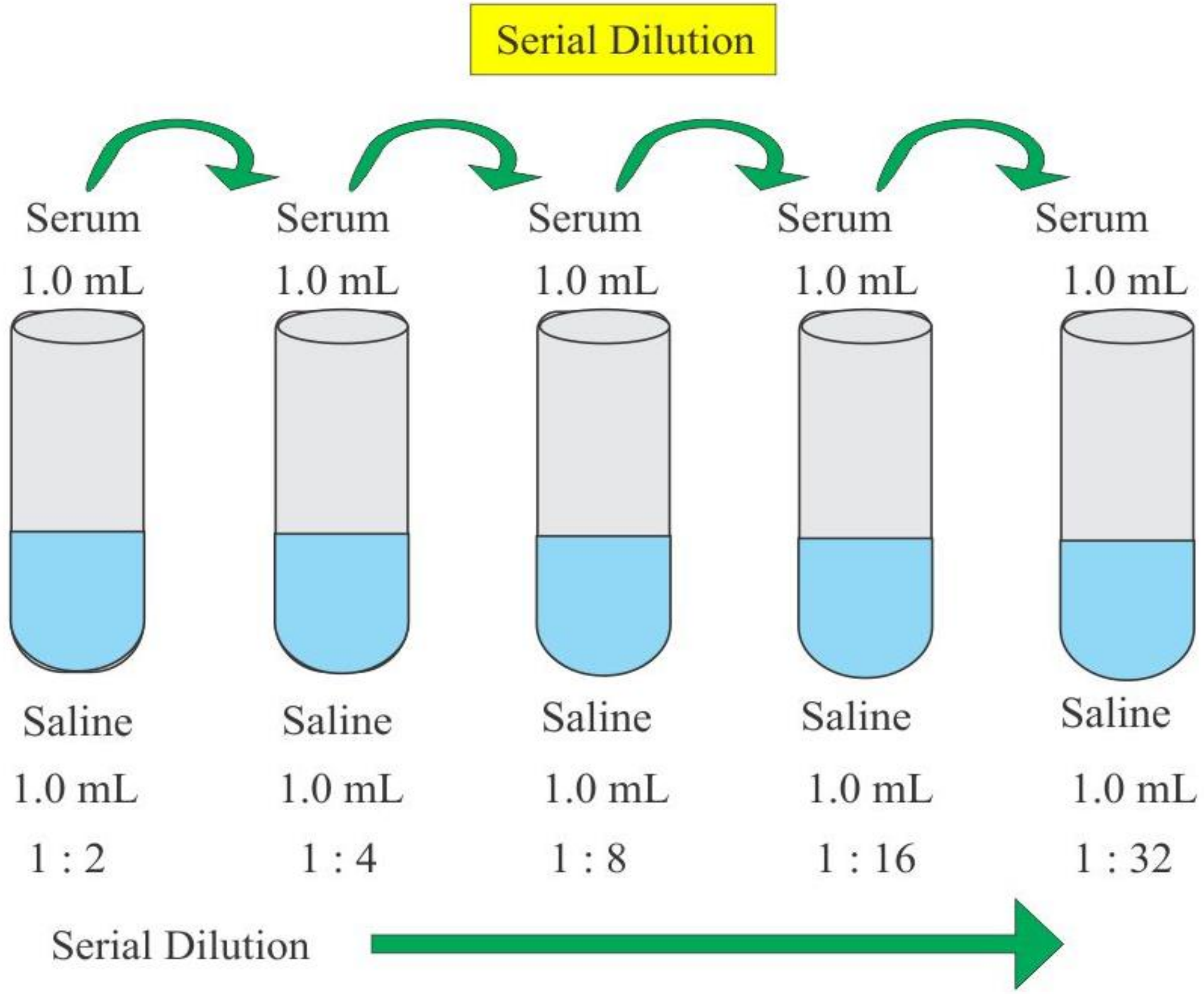




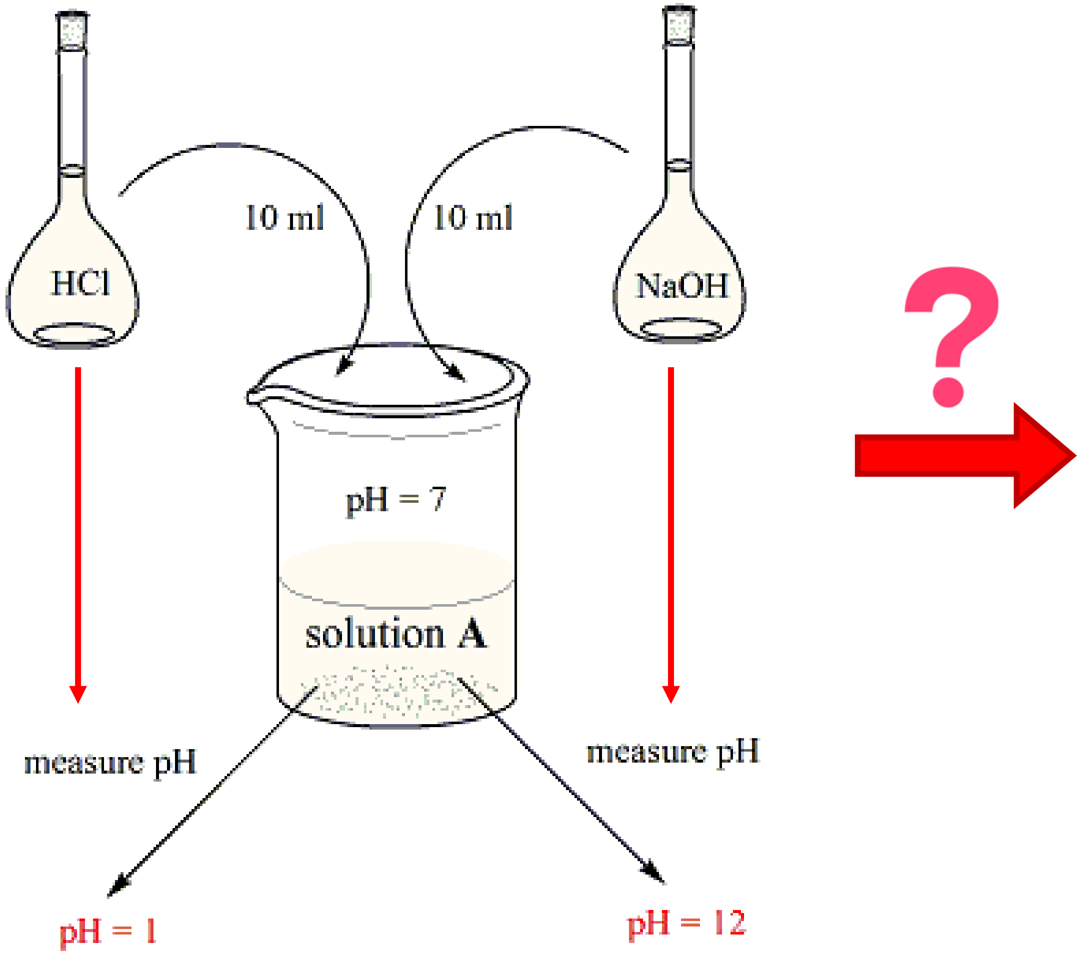
- 2) Preparazione mediante diluizione di una soluzione madre concentrata

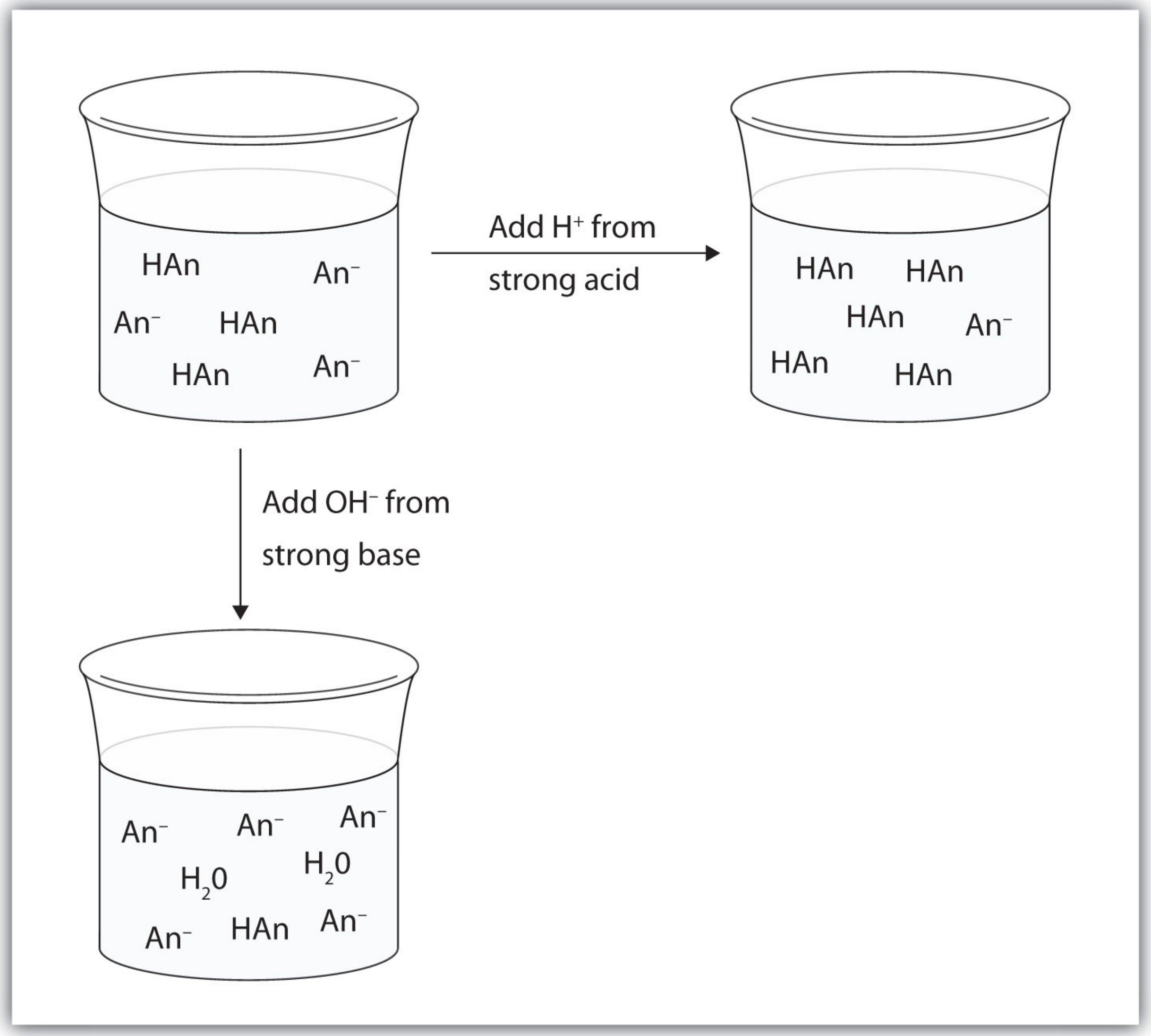


Diluizione seriale



Preparazione della soluzione tampone

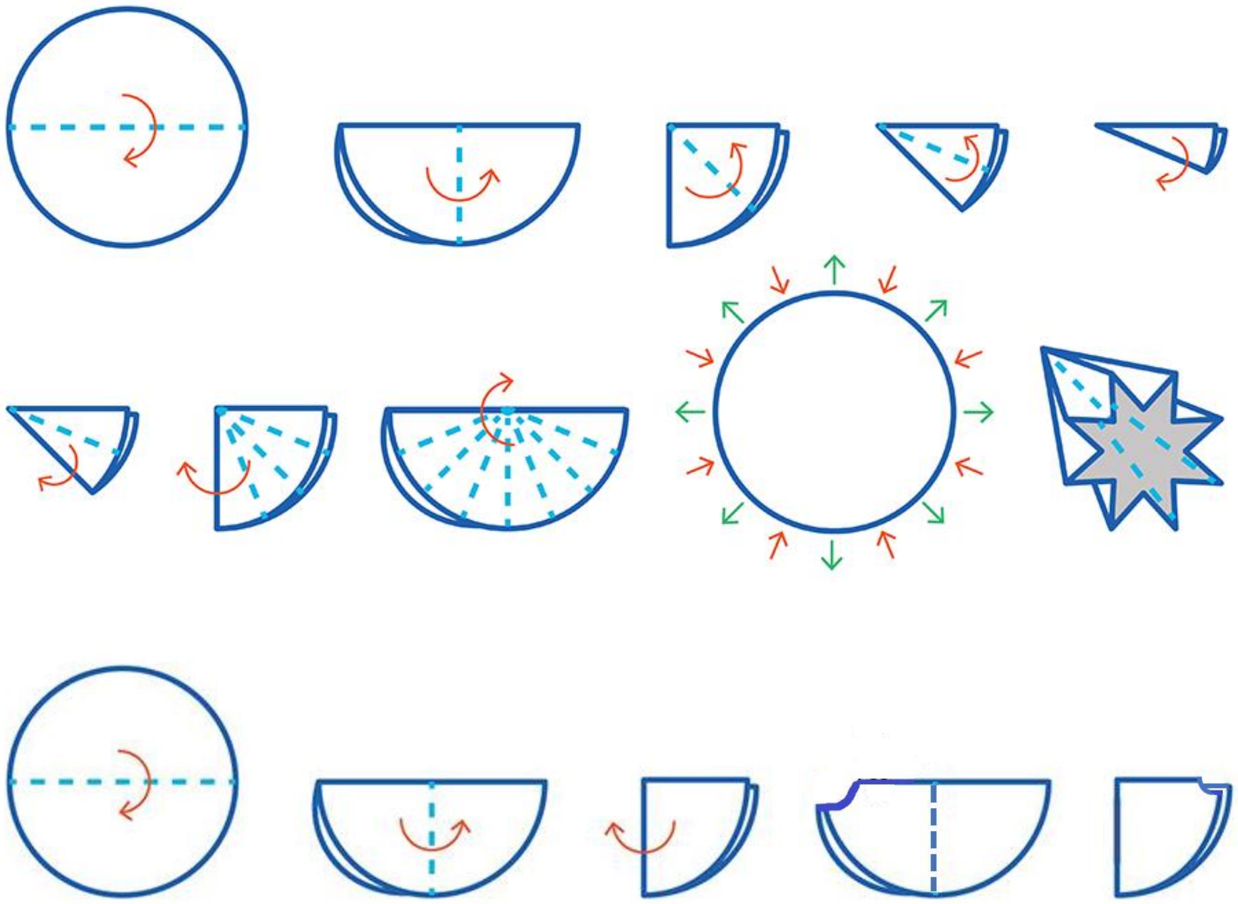


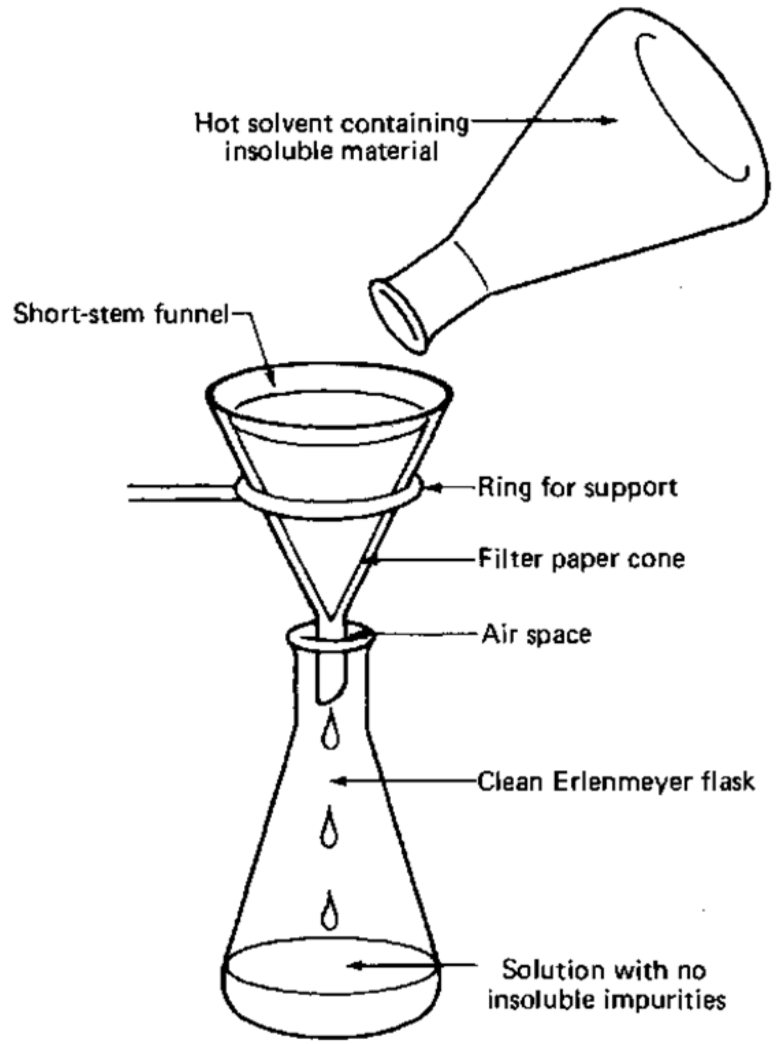


- Come costruire un filtro di carta

A)

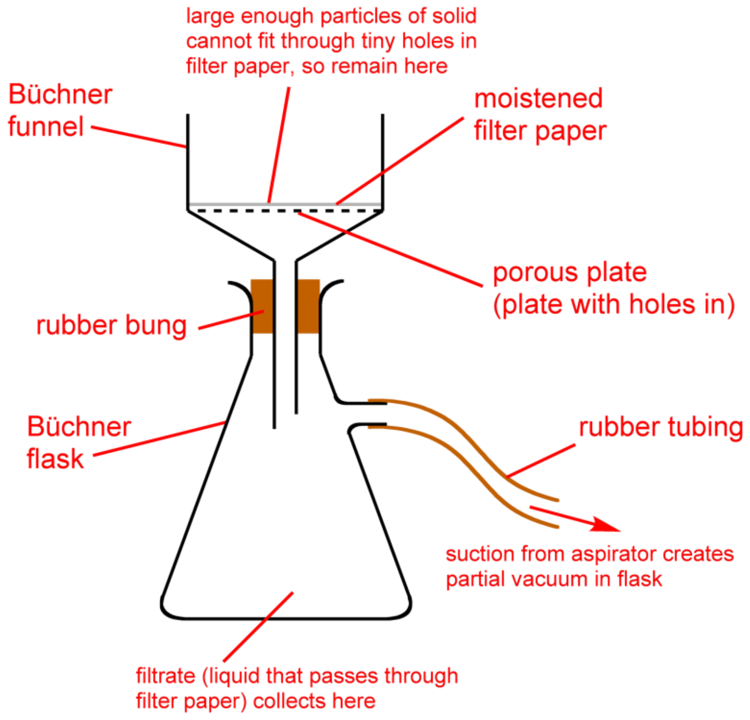
B)

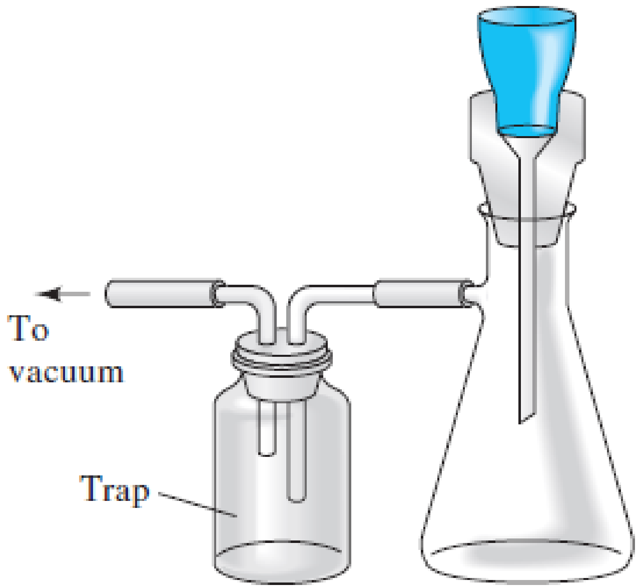




- Filtrazione sottovuoto

Vacuum system





- Centrifuga

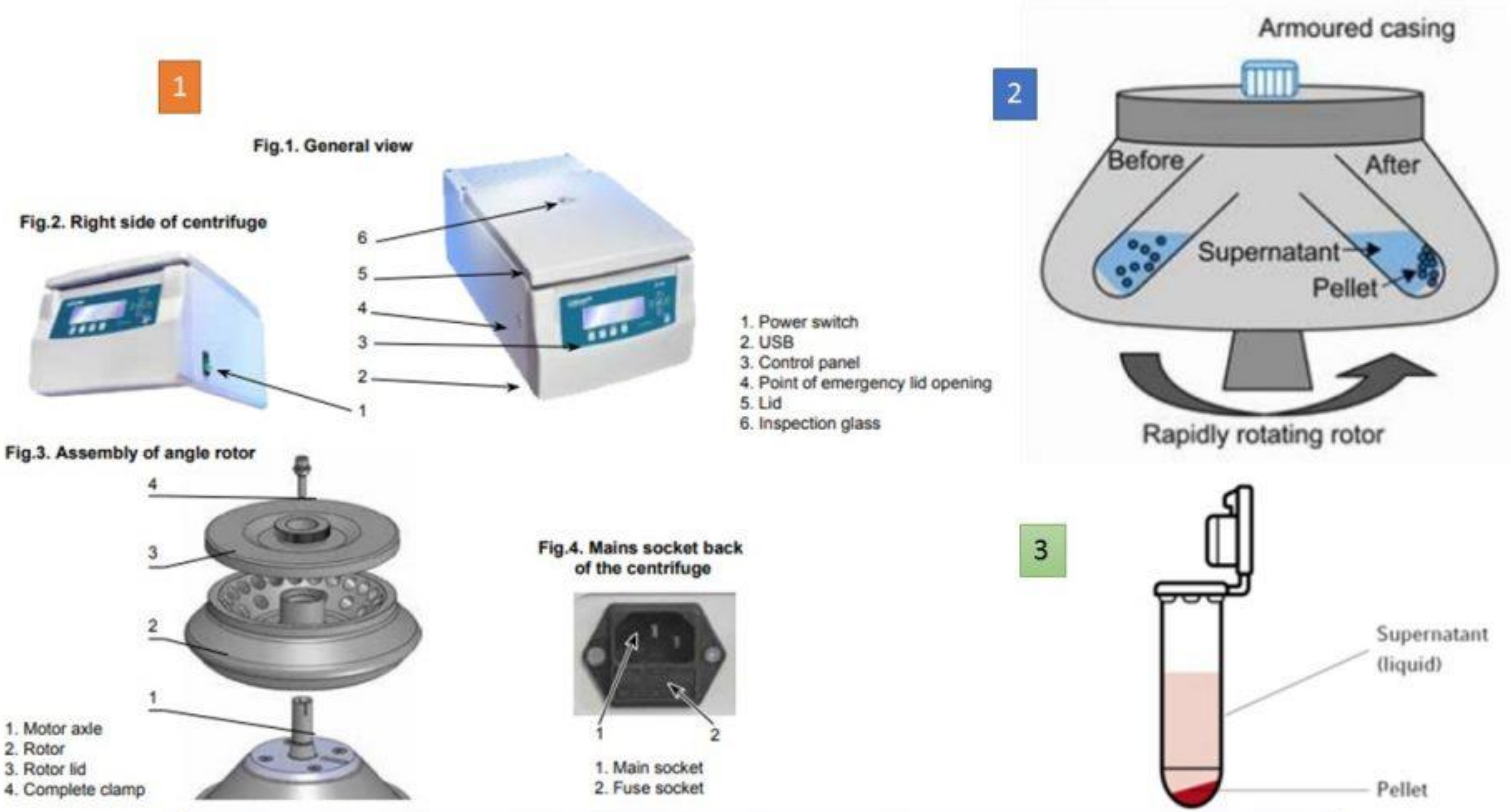


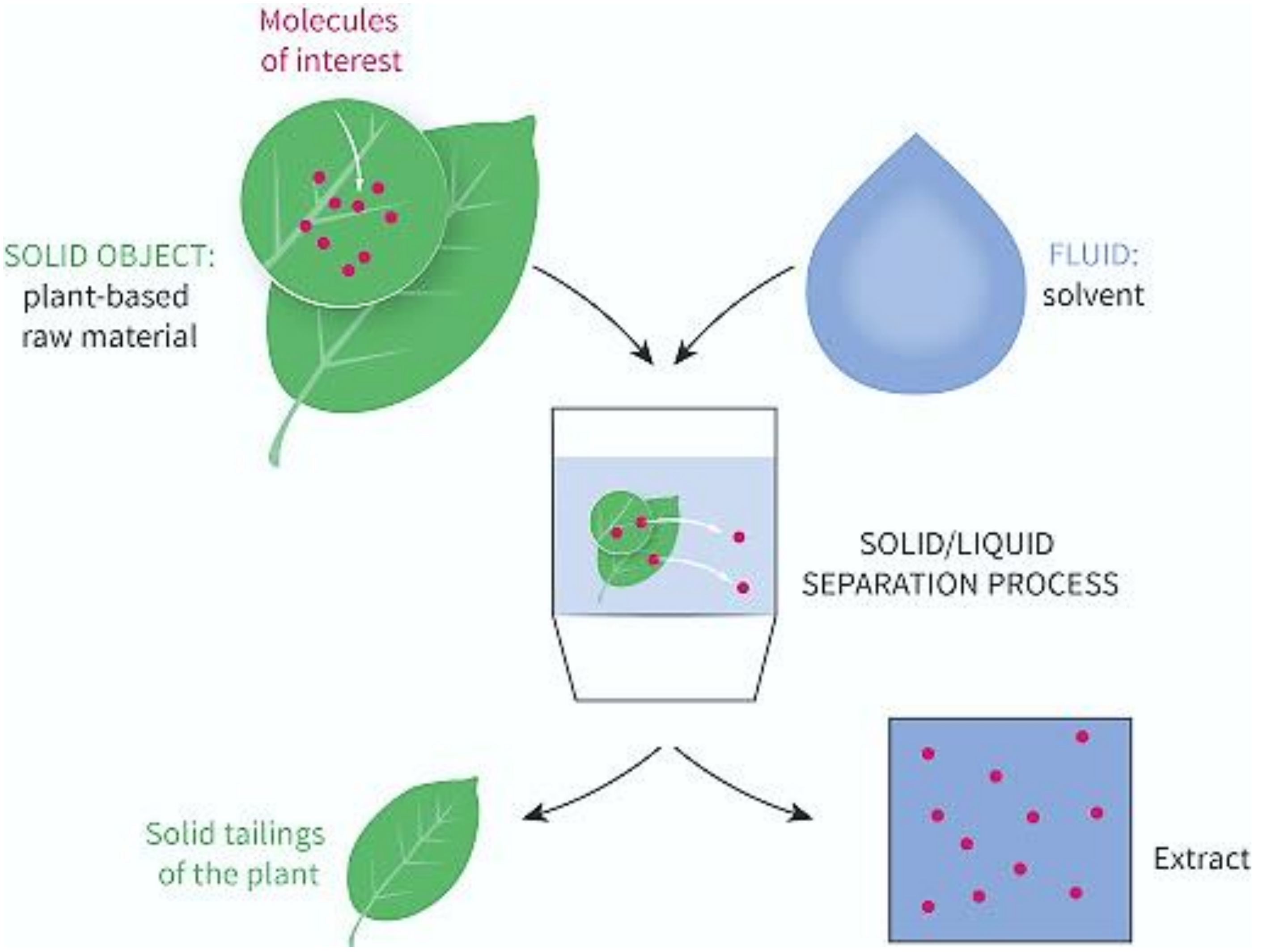
Fig. Centrifuge: General View, Centrifugation procedure and components separation

Image sources: handling-solutions.eppendorf.com & labnetinternational.com

Estrazione solido-liquido. Estrazione forma campione solido.

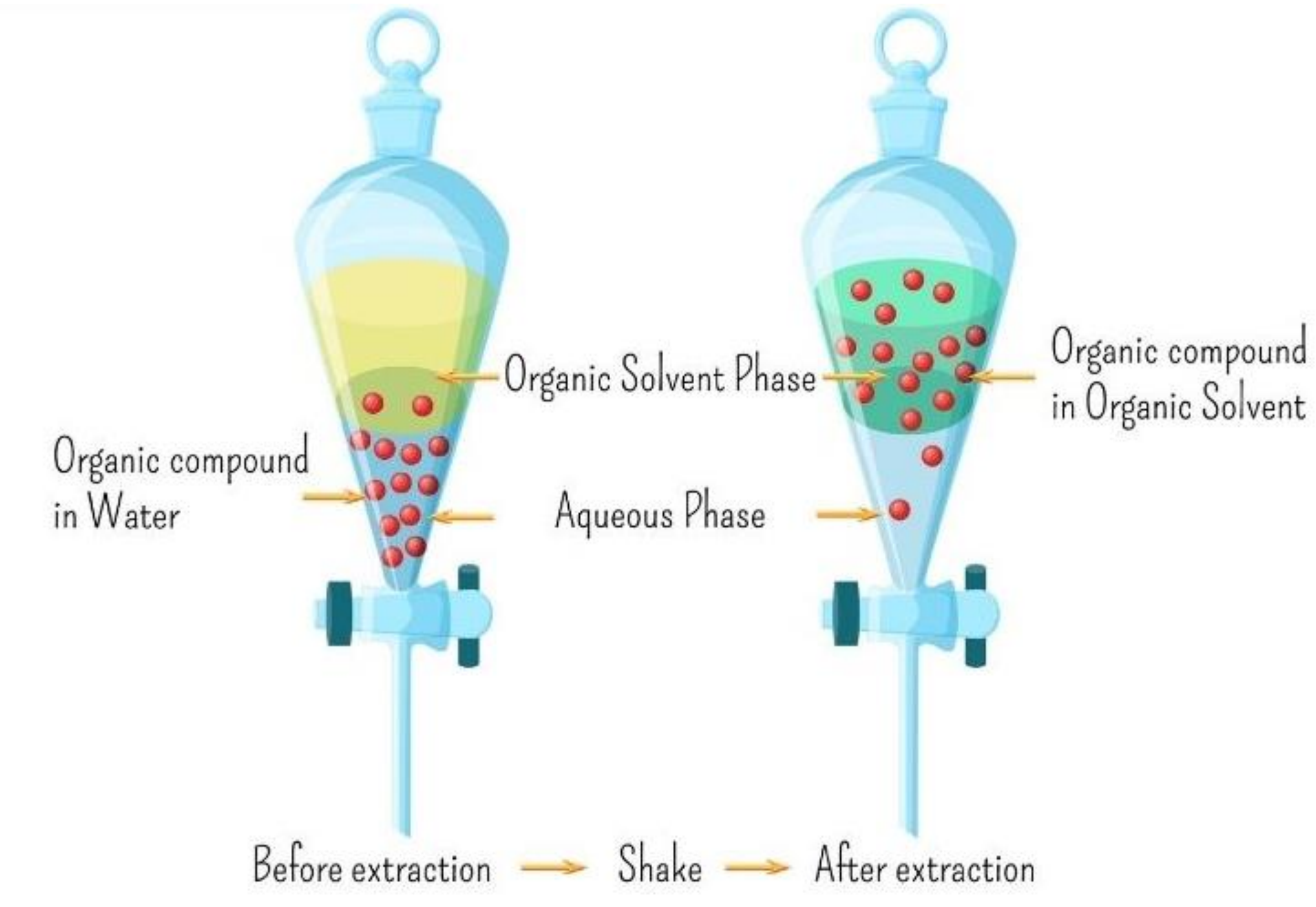
A) **Pesata del campione**

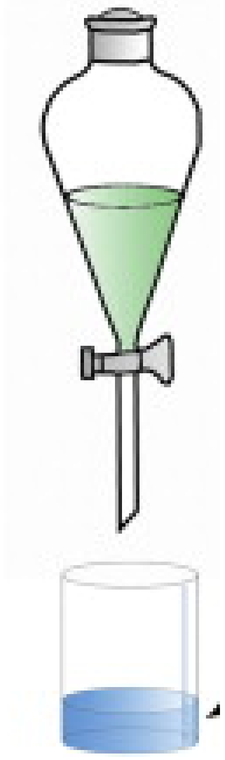
B) **Estrazione del campione**



Principio di estrazione degli analiti

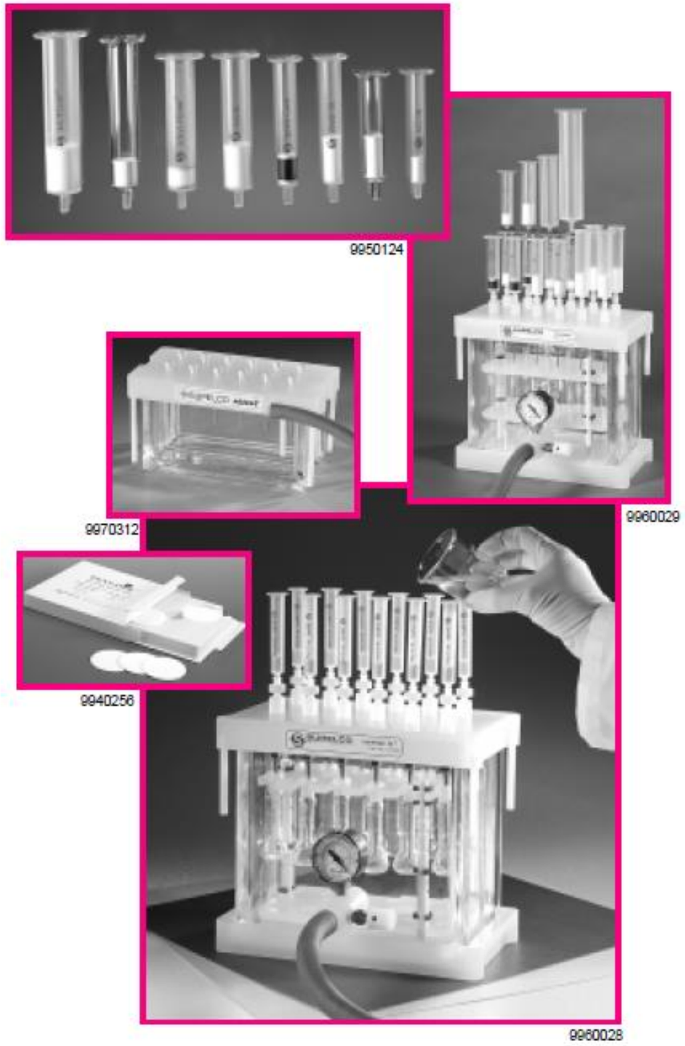
Estrazione liquido-liquido. Estrazione da campione liquido.

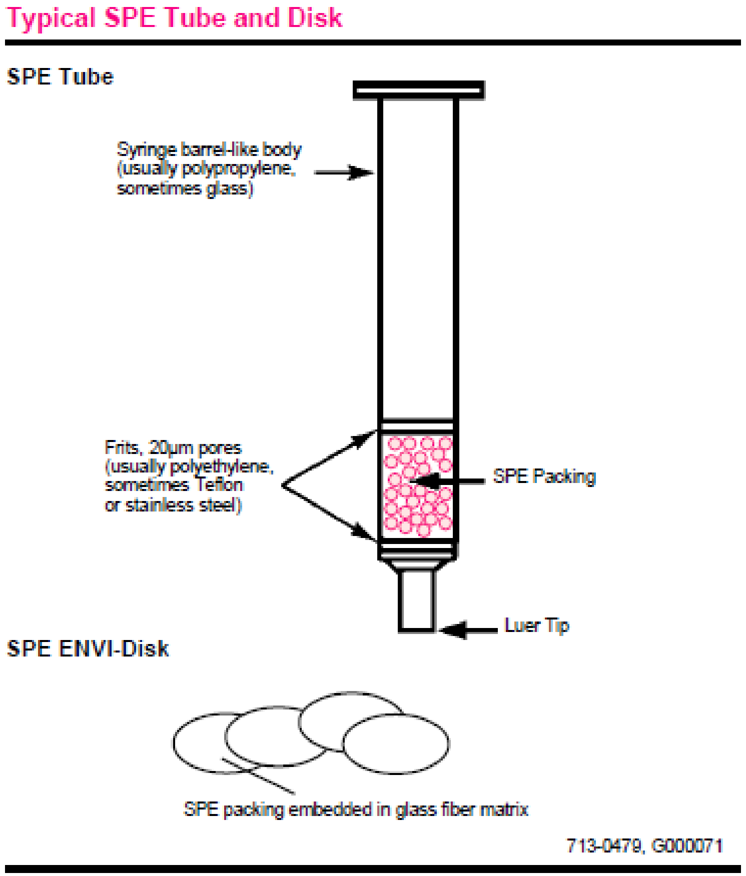




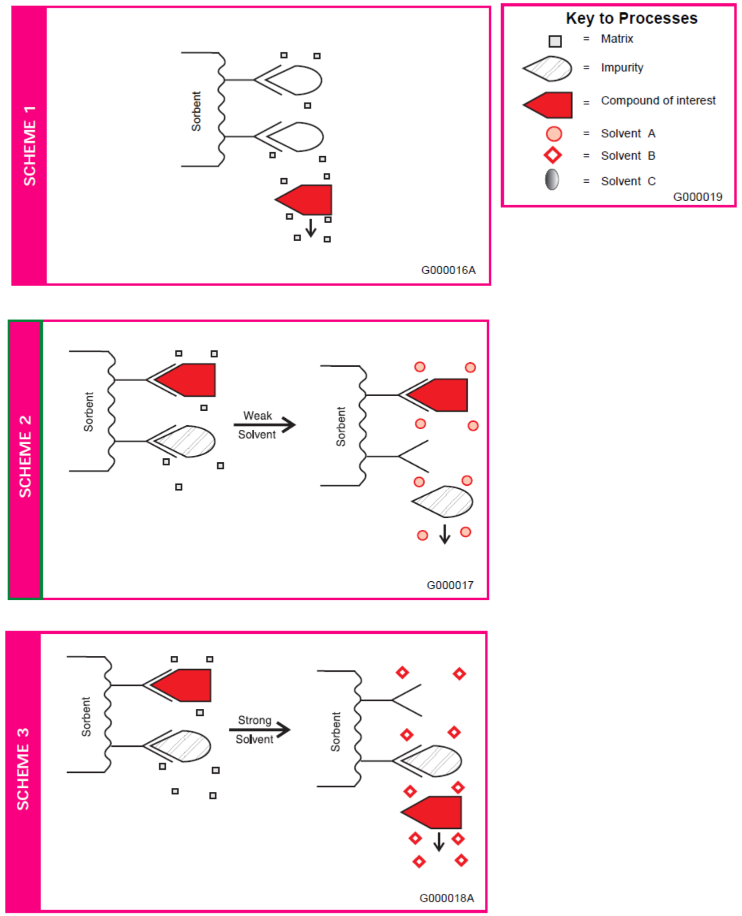
Analytes' extraction principle

Solid phase extraction (SPE). Extraction form liquid sample.





How to use SPE



SOLID PHASE EXTRACTION OF PHOLYPHENOLS FROM OLIVE OIL

Antioxidants could be defined as sacrificial molecules.

Antioxidants are natural or synthetic molecules able to scavenge reactive species, such as reactive oxygen and nitrogen species (ROS and RNS), contributing to oxidative homeostasis.

Phenolic compounds


Beneficial effect on human health


Anti-microbial property


Additives in biomedicine practices


Food supplements (sensory and nutritional properties, shelf-life)


Quality and process indicators


Potential tools for functionalization of materials

Maturitas 132 (2020) 65–69

The Mediterranean diet: A historical perspective on food for health

Juan José Hidalgo-Mora, Alicia García-Vigara, María Luz Sánchez-Sánchez, Miguel-Ángel García-Pérez, Juan Tarín, Antonio Cano

Molecular Aspects of Medicine 67 (2019) 1–55

Benefits of the Mediterranean diet: Epidemiological and molecular aspects

Lluis Serra-Majem, Blanca Román-Viñas, Almudena Sanchez-Villegas, Marta Guasch-Ferré, Dolores Corella, Carlo La Vecchia

Received: 10 April 2019 | Revised: 8 June 2019 | Accepted: 17 June 2019
DOI: 10.1111/bph.14778

Themed Section: The Pharmacology of Nutraceuticals

REVIEW ARTICLE

Mediterranean diet and health status: Active ingredients and ...

Journal of Nutritional Biochemistry 26 (2015) 250–258

Extravirgin olive oil up-regulates CB_1 tumor suppressor gene in human colon cancer cells and in rat colon via epigenetic mechanisms

Andrea Di Francesco, Anastasia Falconi, Clara Di Germanio, Maria Vittoria Micioni Di Bonaventura, Antonio Costa, Stefano Caramuta, Michele Del Carlo, Dario Compagnone, Enrico Dainese, Carlo Cifani, Mauro Maccarrone, Claudio D'Addario

nutrients — Review 2019

The Fluid Aspect of the Mediterranean Diet in the Prevention and Management of Cardiovascular Disease and Diabetes: The Role of Polyphenol Content in Moderate Consumption of Wine and Olive Oil

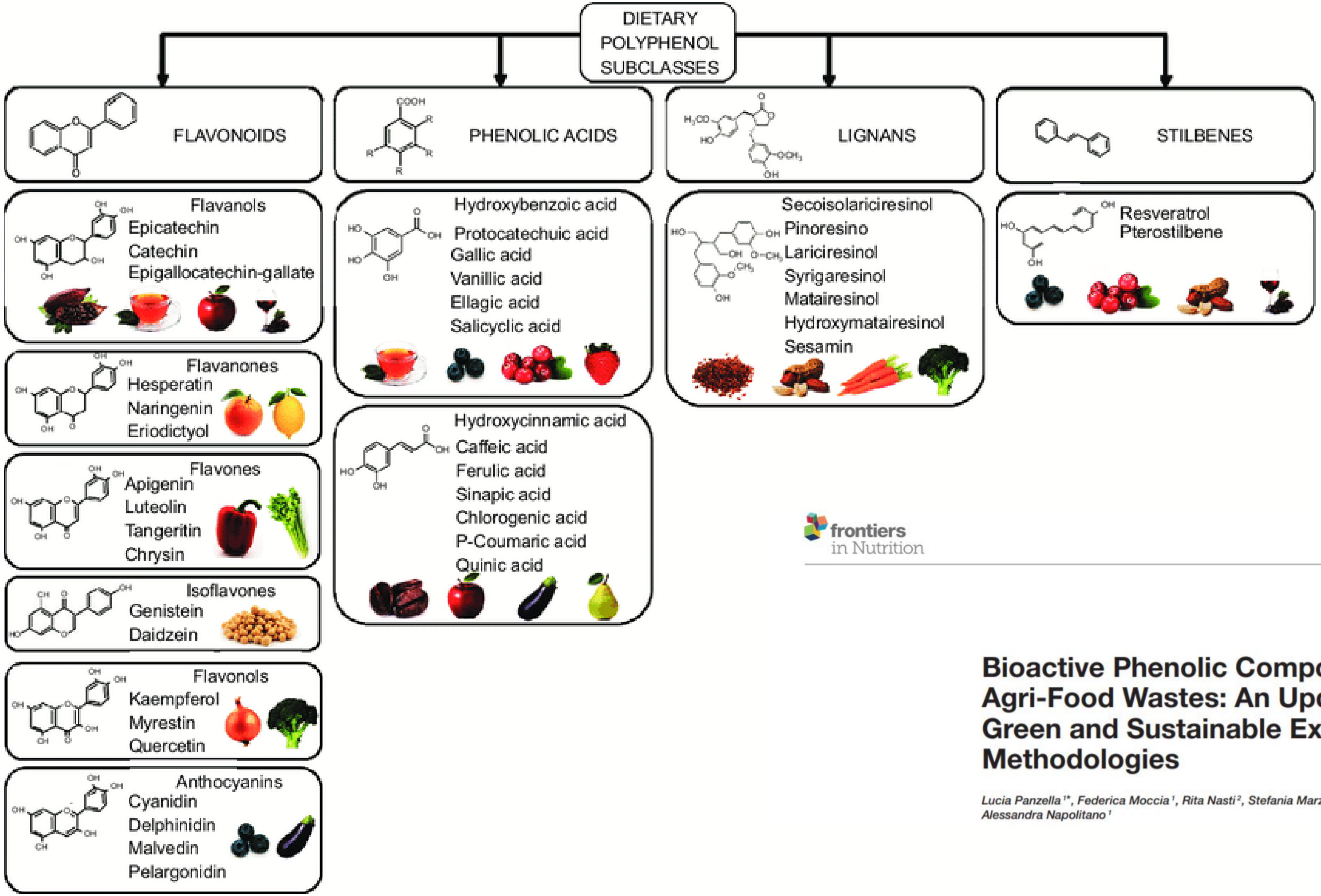
Paola Ditano-Vázquez, José David Torres-Peña, Francisco Galeano-Valle, Ana Isabel Pérez-Caballero, Pablo Demelo-Rodríguez, José Lopez-Miranda, Niki Katsiki, Javier Delgado-Lista and Luis A. Alvarez-Sala-Walther

frontiers in Nutrition

Network Meta-Analysis of Metabolic Effects of Olive-Oil in Humans Shows the Importance of Olive Oil Consumption With Moderate Polyphenol Levels as Part of the Mediterranean Diet

Evangelia Tsartsou, Nikolaos Proutsos, Elias Castanas and Marilena Kampa

- Classification



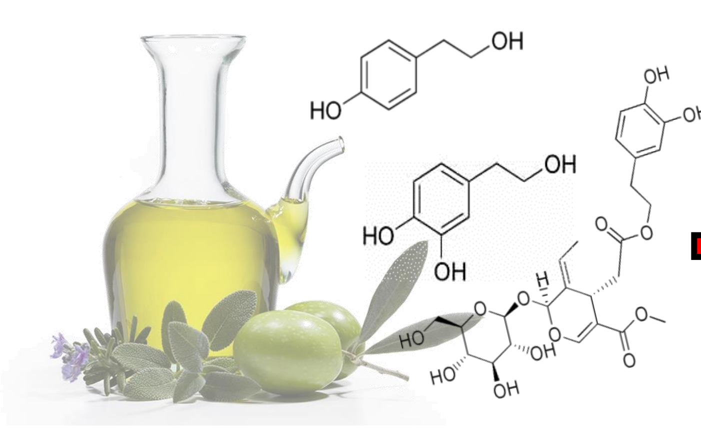
frontiers in Nutrition
REVIEW
published: 07 May 2020
doi: 10.3389/fnut.2020.00060

Bioactive Phenolic Compounds From Agri-Food Wastes: An Update on Green and Sustainable Extraction Methodologies

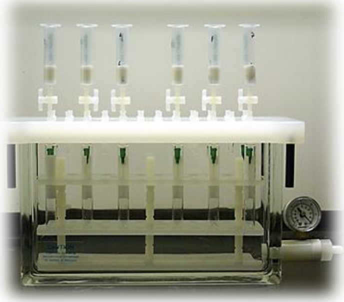
Lucia Panzella[1], Federica Moccia[1], Rita Nasti[2], Stefania Marzorati[2], Luisella Verotta[2] and Alessandra Napolitano[1]*

Phenolic content evaluation in Extra Virgin Olive Oil. Main strategies

UNIVERSITÀ DEGLI STUDI DI TERAMO
UNITE



Extraction (LLE / SPE)



Solvent evaporation and phenols concentration

Separative / Chromatographic strategies

Plant Foods Hum Nutr (2012) 67:326–336
DOI 10.1007/s11130-012-0315-z

ORIGINAL PAPER

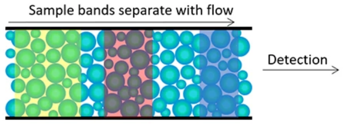
Comprehensive Analysis of Polyphenols in 55 Extra Virgin Olive Oils by HPLC-ECD and Their Correlation with Antioxidant Activities

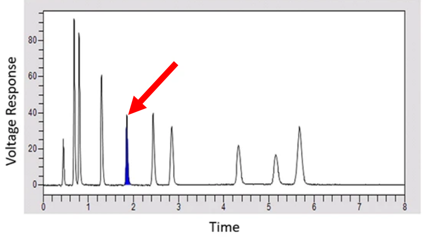
Banu Bayram · Tuba Esatbeyoglu · Nicole Schulze · Beraat Ozcelik · Jan Frank · Gerald Rimbach

Contents lists available at ScienceDirect
Journal of Chromatography A
journal homepage: www.elsevier.com/locate/chroma

Olive oil polyphenols: A quantitative method by high-performance liquid-chromatography-diode-array detection for their determination and the assessment of the related health claim

Massimo Ricciutelli[a], Shara Marconi[b], Maria Chiara Boarelli[b], Giovanni Caprioli[c], Gianni Sagratini[c], Roberto Ballini[b], Dennis Fiorini[b,*]





Electrochemical-based strategies

Microchimica Acta (2019) 186: 363
https://doi.org/10.1007/s00604-019-3418-5

ORIGINAL PAPER

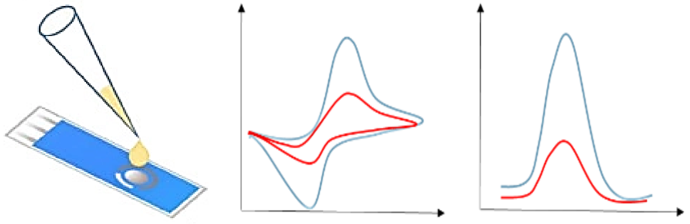
Nanohybrid carbon black-molybdenum disulfide transducers for preconcentration-free voltammetric detection of the olive oil o-diphenols hydroxytyrosol and oleuropein

Daniel Rojas[1,2] · Flavio Della Pelle[1] · Michele Del Carlo[1] · Emiliano Fratini[3] · Alberto Escarpa[2,4] · Dario Compagnone[1]

Food Research International 54 (2013) 2075–2082
Contents lists available at ScienceDirect
Food Research International
journal homepage: www.elsevier.com/locate/foodres

Voltammetric e-tongue for the quantification of total polyphenol content in olive oils

Irina Mirela Apetrei[a], Constantin Apetrei[b,*]



Optical-based strategies

Research Article
(wileyonlinelibrary.com) DOI 10.1002/jsfa.9461

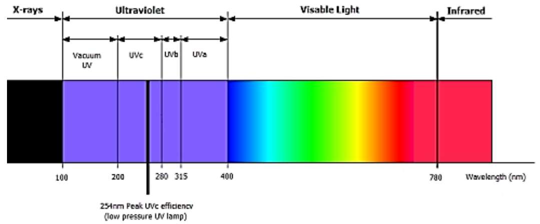
Evaluation of total phenolic content in virgin olive oil using fluorescence excitation–emission spectroscopy coupled with chemometrics

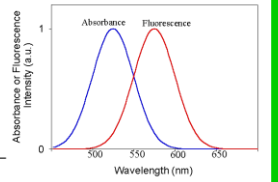
Giacomo Squeo,[a] Francesco Caponio,[a] Vito M Paradiso,[a] Carmine Summo,[a] Antonella Pasqualone,[a] Igor Khmelinskii[b] and Ewa Sikorska[c*]

Computers and Electronics in Agriculture 173 (2020) 105445
Contents lists available at ScienceDirect
Computers and Electronics in Agriculture
journal homepage: www.elsevier.com/locate/compag

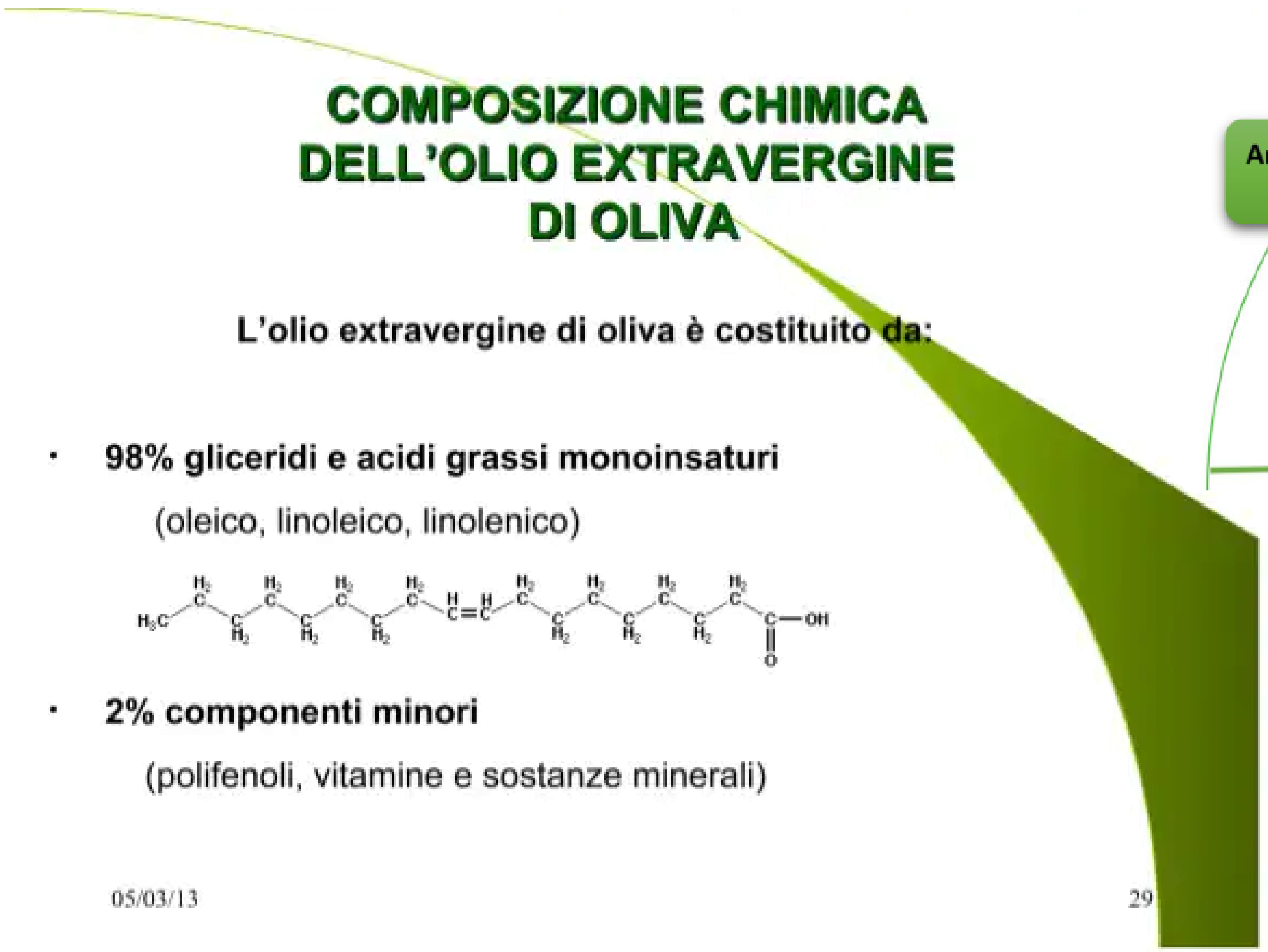
Visible/Near Infrared (VIS/NIR) spectroscopy as an optical sensor for evaluating olive oil quality

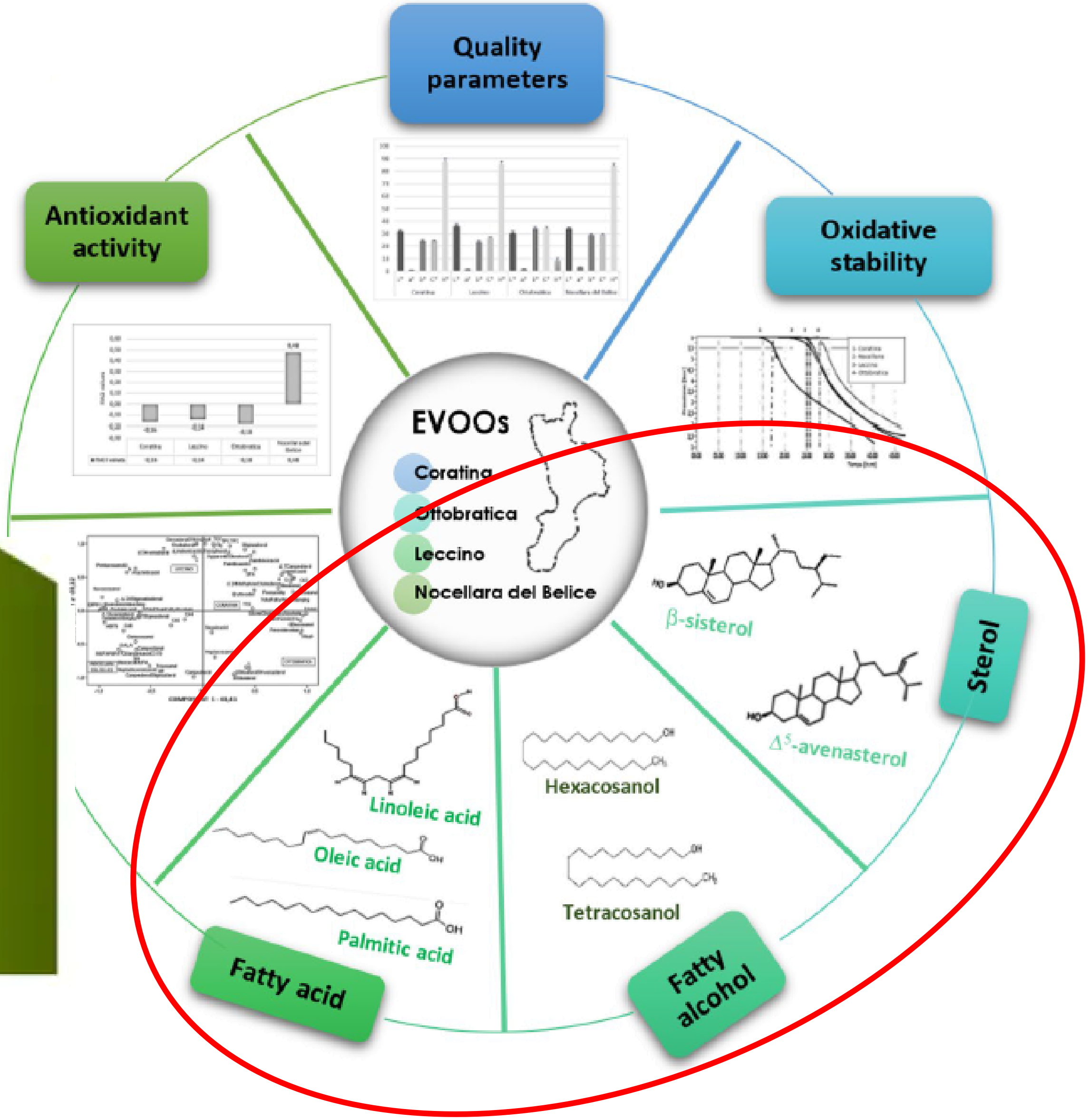
Nawaf Abu-Khalaf[a,*], Mohammed Hmidat[b]





Why the extraction is required?

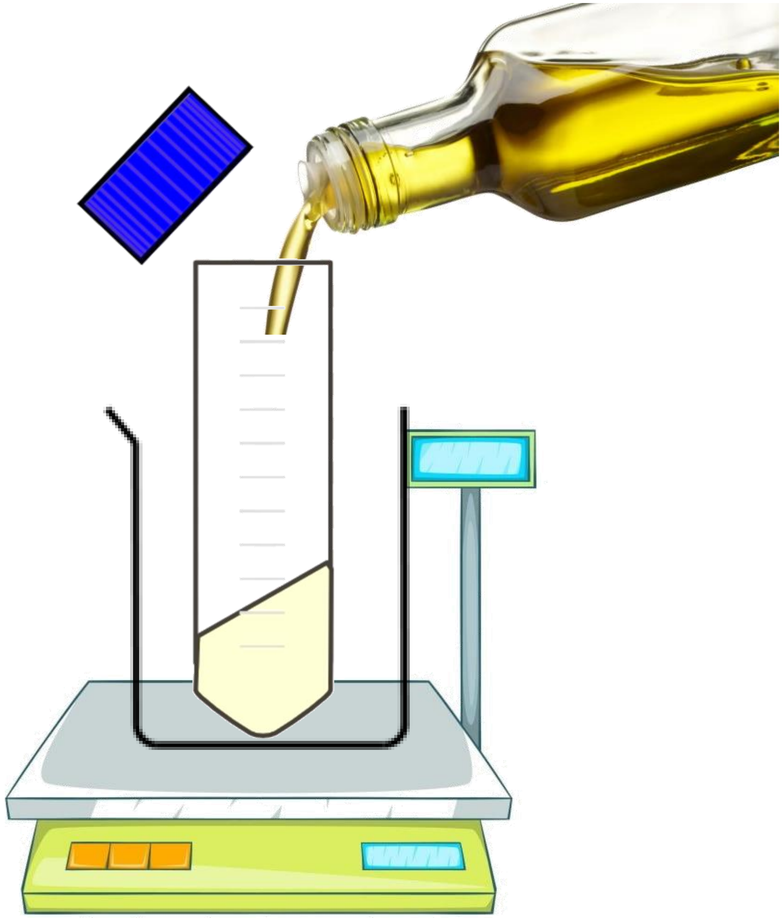




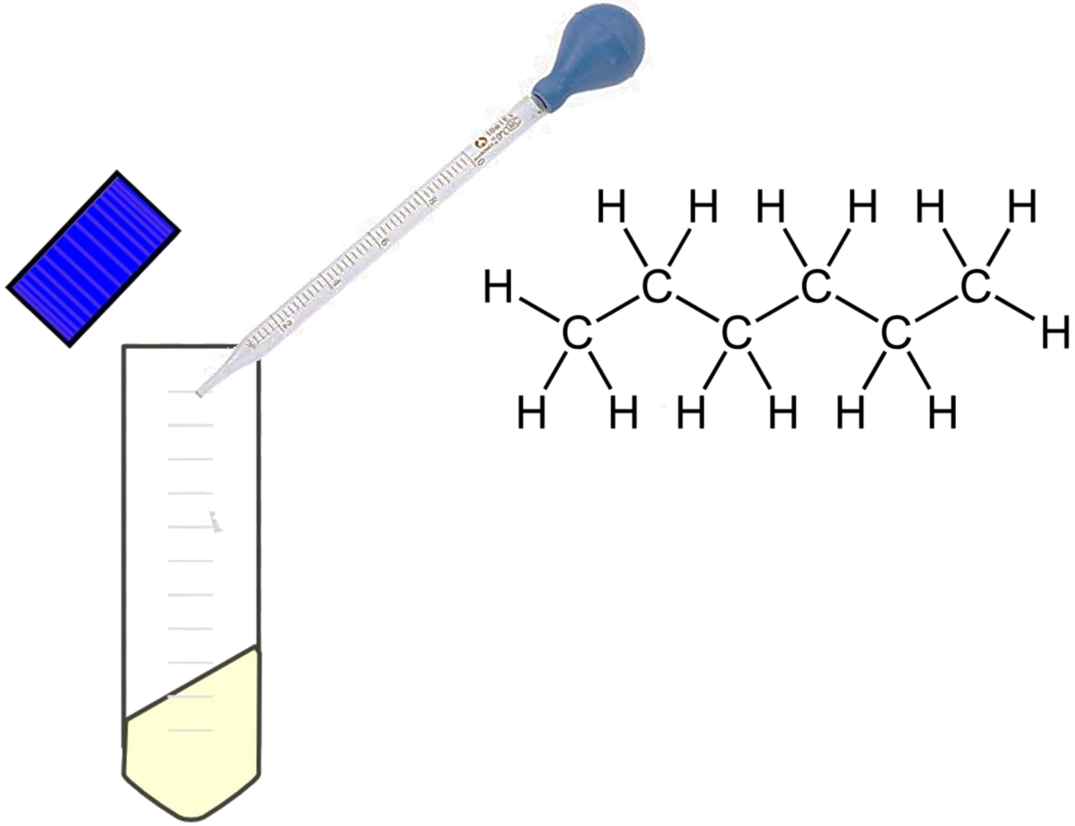
Phenolic content evaluation in Extra Virgin Olive Oil. Main strategies

<u>Preparazione del campione</u>

- Pesare 1.0 g di campione

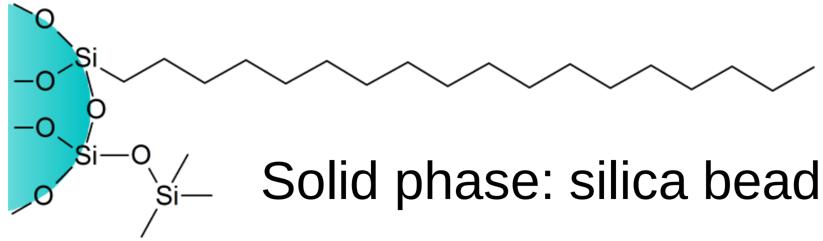


- Scioglierlo in 5 ml di esano.



Polyphenols extraction

Solid phase extraction (SPE). Extraction form liquid sample.



Solid phase: silica beads@C18

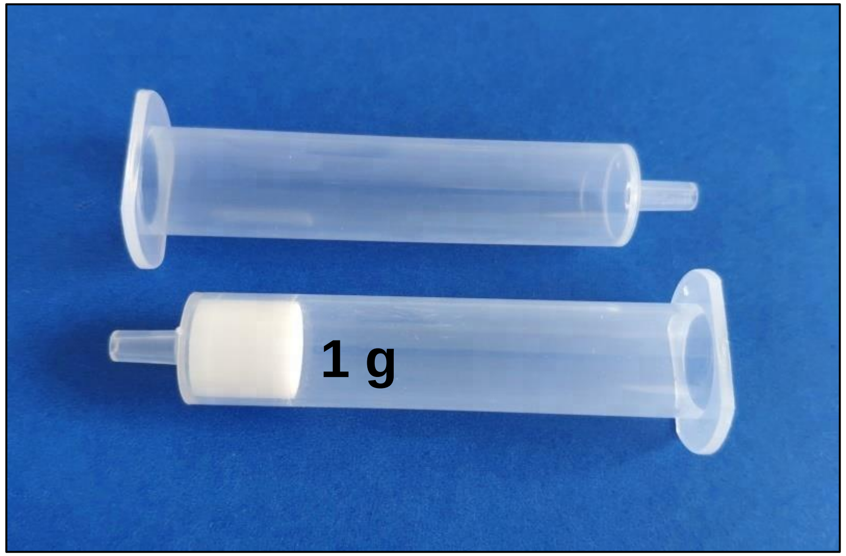
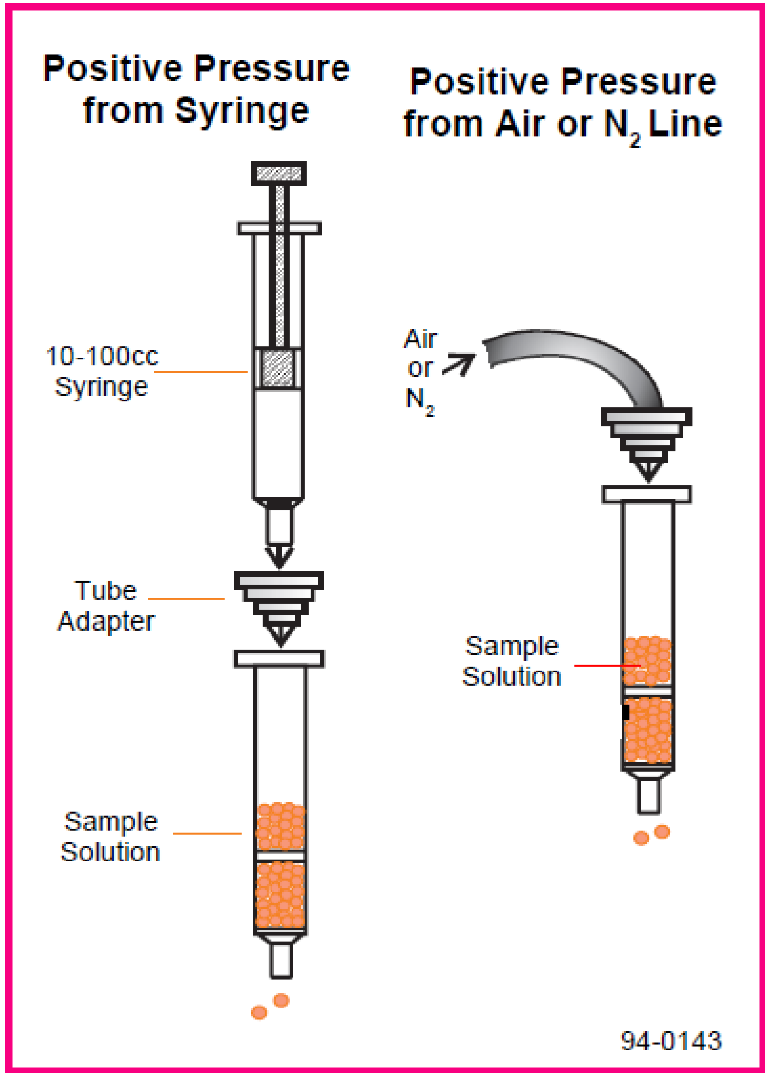
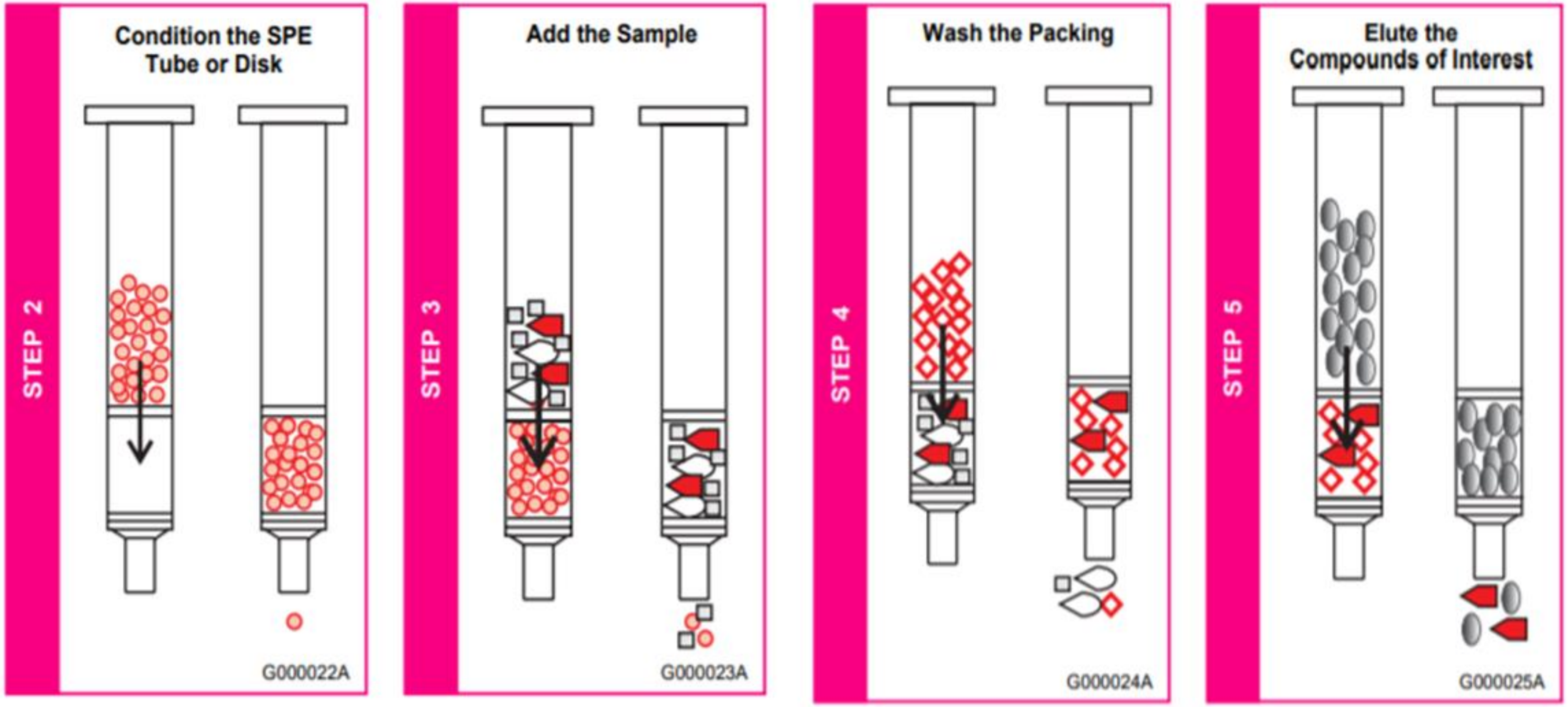


Figure B. Process Using Applied Pressure



Polyphenols extraction

Solid phase extraction (SPE). Extraction form liquid sample.



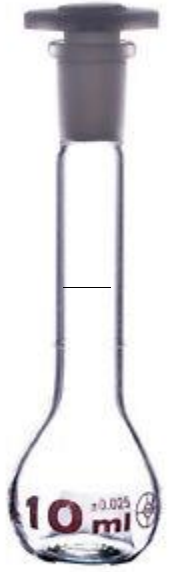
10 mL Methanol
10 mL Hexane

10 mL Hexane

10 mL Methanol

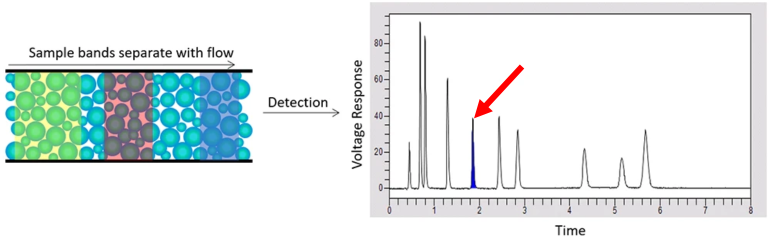
Polyphenols extraction

Solid phase extraction (SPE). Extraction form liquid sample.

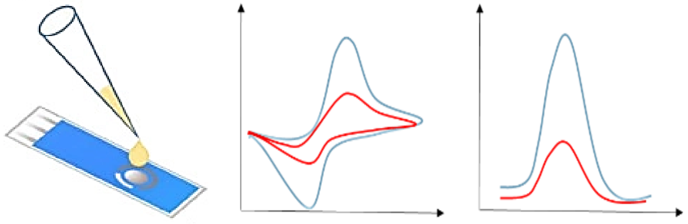


Analysis

Chromatographic strategies



Electrochemical-based strategies



Optical-based strategies