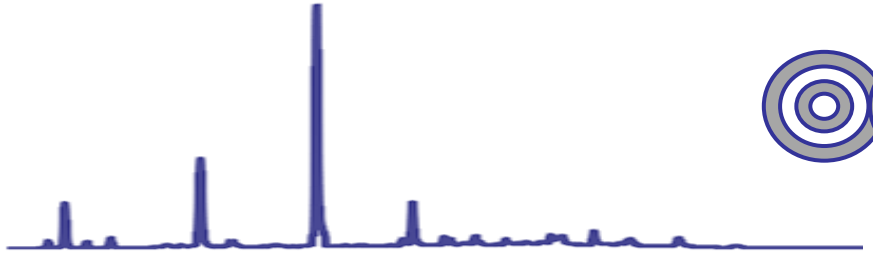
بسم الله الرحمن الرحيم

Basic Tools of Analytical Chemistry



Ahmad Aqel Ifseisi
Professor of Analytical Chemistry
College of Science, Department of Chemistry
King Saud University
P.O. Box 2455 Riyadh 11451 Saudi Arabia
Building: 05, Office: 2A149, Lab: AA53
Tel. 014674198, Fax: 014675992
Web site: https://faculty.ksu.edu.sa/aifseisi
E-mail: aifseisi@ksu.edu.sa
ahmad3qel@gmail.com

كرسي أبحاث المواد المتقدمة
Advanced Materials Research Chair
AMRC

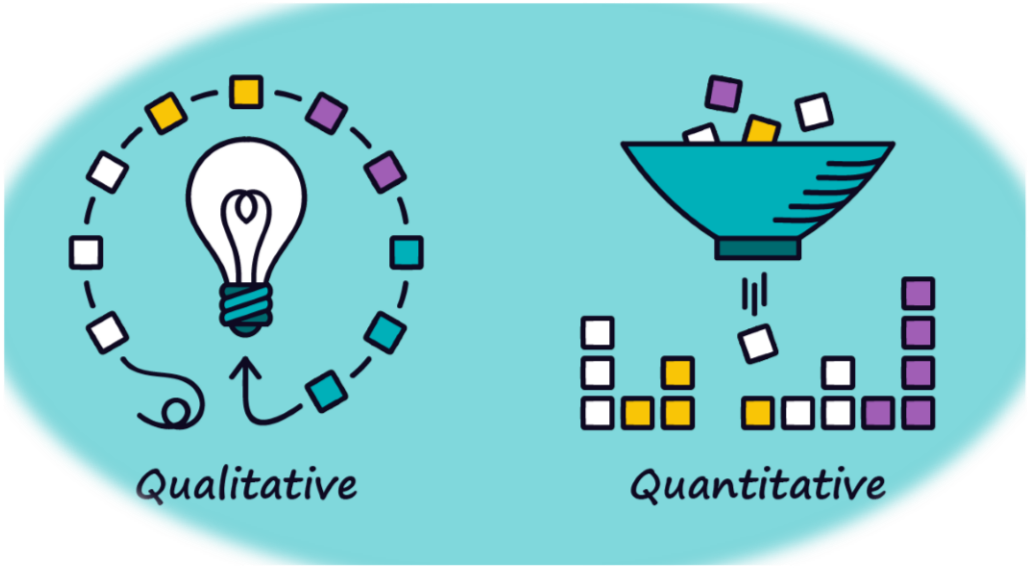
جامعة الملك سعود
King Saud University

The Language of Analytical Chemistry

Analytical Chemistry is the science of,,,

- Analysis,
- Technique,
- Method,
- Development,
- Optimization,
- Validation,
- Methodology,
- Procedure,
- Protocol,
- Determination,
- Measurement,
- Calculation,

Analytical Chemistry - Objectives



Intrinsic aim
attaining analytical information; qualitative and quantitative of the highest quality, accuracy and precision with low uncertainty.

Extrinsic aim
solving analytical problems derived from other fields such as food, biochemistry, health, industry, environment, etc.

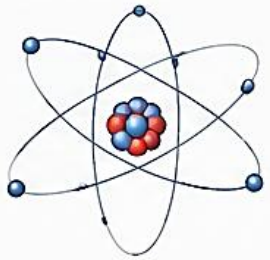
Basic Tools and Operations of Analytical Chemistry

Chemicals, apparatus, equipment, and instruments
Materials, standards, real samples, additives, reagents, solvents, glass and other wares, balances, volumetric wares, laboratory safety, etc.
Numbers, units, and concentration expressions
Base quantities, SI units, derived units, molarity, molality, normality, weight, volume, and weight-to-volume ratios, converting between units, p-functions, etc.
Chemical calculations, stoichiometry, and equilibrium
Chemical reactions, chemical equations, yield, theoretical and practical calculations, calculations based on balanced equations, etc.
Errors in chemical analyses and measurements
Error sources, systematic errors (determinate), random errors (indeterminate), human errors, uncertainty, significant figures, rounding off, etc.
Sampling, standardization, and calibration
Sample collection, sample preparation, standard solution, external standards, standard additions, internal standards, etc.
Methods development and validation
Good laboratory practice, quality control, quality assurance, accuracy, precision, sensitivity, selectivity, robustness, ruggedness, scale of operation, time, cost, laboratory accreditation, etc.
Statistical data treatment and evaluation
Slope, intercept, coefficient of determination, rejection of results, detection limits, statistics software, making the final decision, etc.

What are You Looking for?

ATOMS

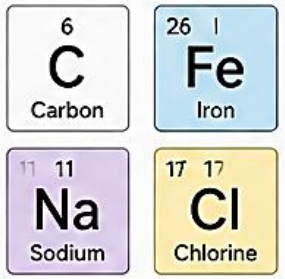
The basic building blocks of matter.



Examples:
H, C, Fe, Na, Cl

ELEMENTS

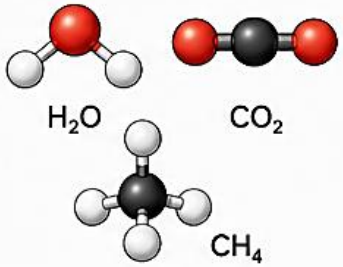
Pure substances made of one type of atom.



Examples:
C, Fe, Na, Cl, O

MOLECULES

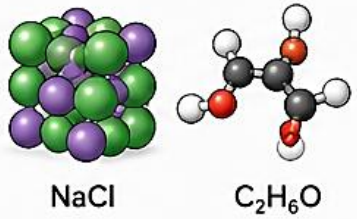
Two or more atoms bonded together.



Examples:
H_2O, CO_2, CH_4, NH_3

COMPOUNDS

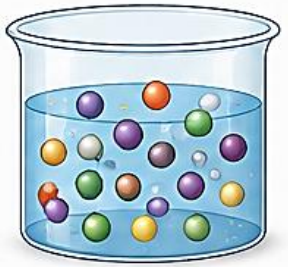
Substances made of two or more different elements chemically bonded.



Examples:
NaCl, C_2H_6O, H_2SO_4

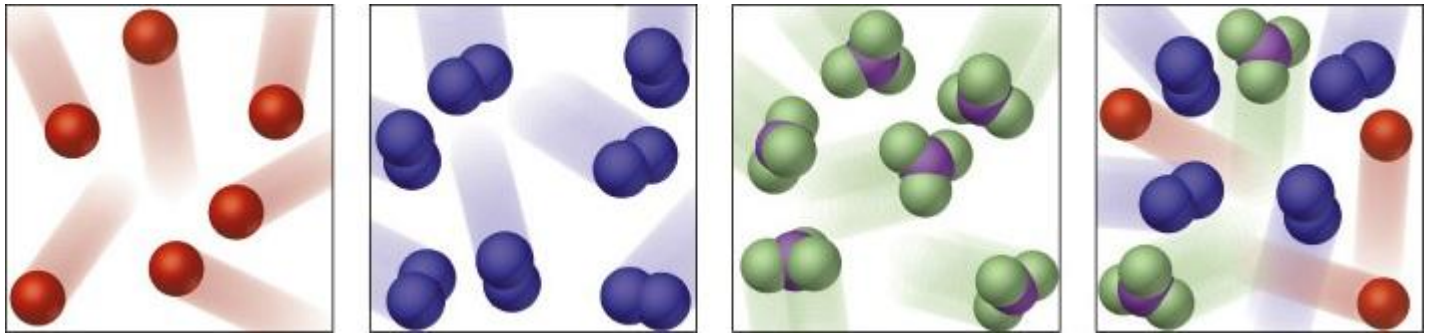
MIXTURES

Two or more substances physically combined.

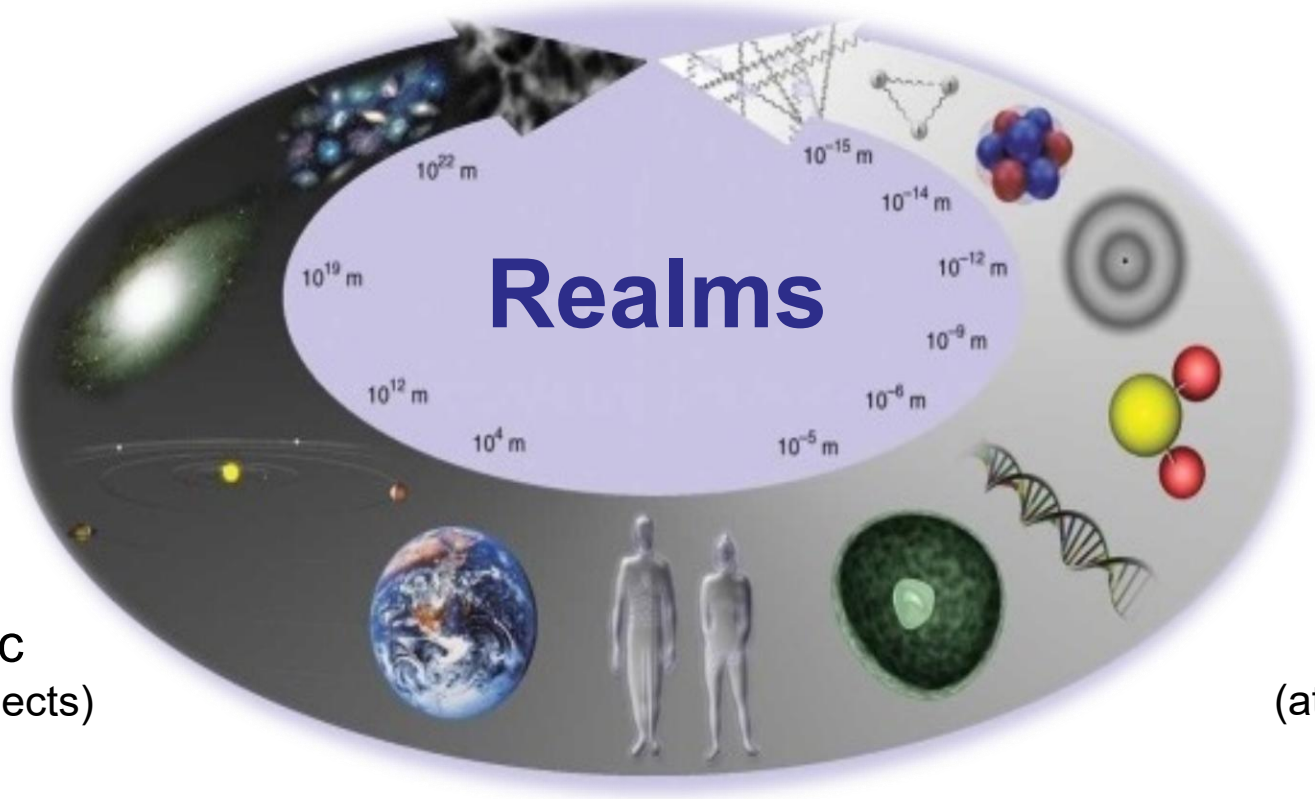


Examples:
Air, Seawater, Soil, Food, Perfume

GOAL: IDENTIFY, SEPARATE, AND QUANTIFY WHAT MATTERS IN QUALITATIVE AND QUANTITATIVE ANALYSIS



From Macroscopic Measurements to Microscopic Insights



Macroscopic
(ordinary sized objects)

Microscopic
(atoms & molecules)

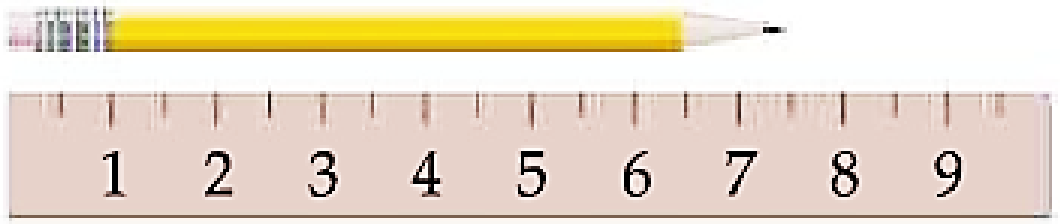
Macroscopic properties, on the ordinary scale, which can be determined directly. e.g., length, mass, volume, temperature.

Microscopic properties, on the atomic or molecular scale, must be determined by an indirect method.

Units

A measured quantity is usually written as a number with an appropriate unit

7.5 meaningless
7.5 cm specifies length



Units are essential to stating measurements correctly.

The units used for scientific measurements are those of the **metric units**.

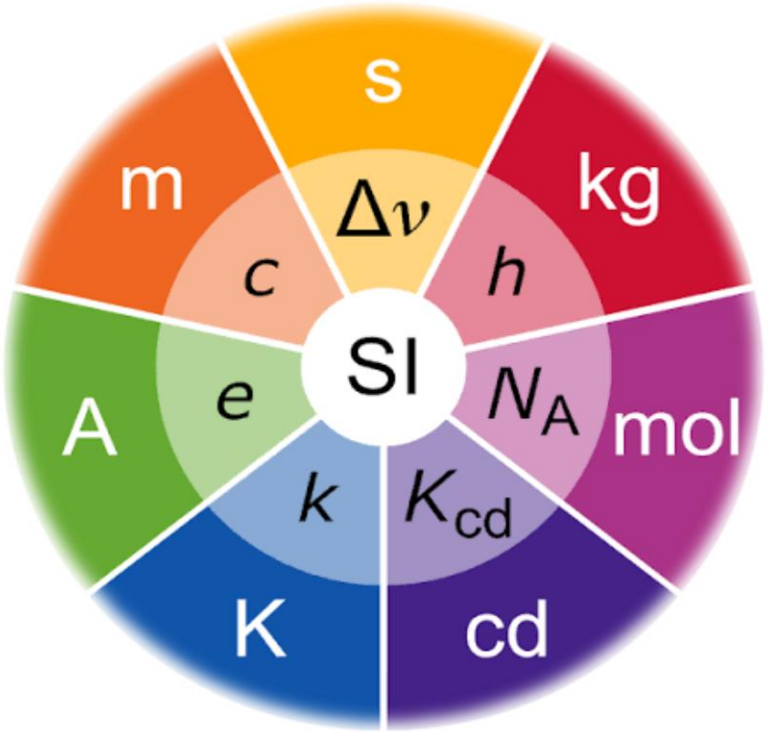
The metric system is an internationally agreed decimal system of measurement that was originally based on the mètre des Archives and the kilogramme des Archives introduced by France in 1799.

SI Units (Base Units)

The General Conference of Weights and Measures; the international authority on units, proposed a revised metric system called the **International System of Units** (abbreviated **SI,** from the French Système Internationale d'Unites, 1960).

SI Base Units (seven base units)

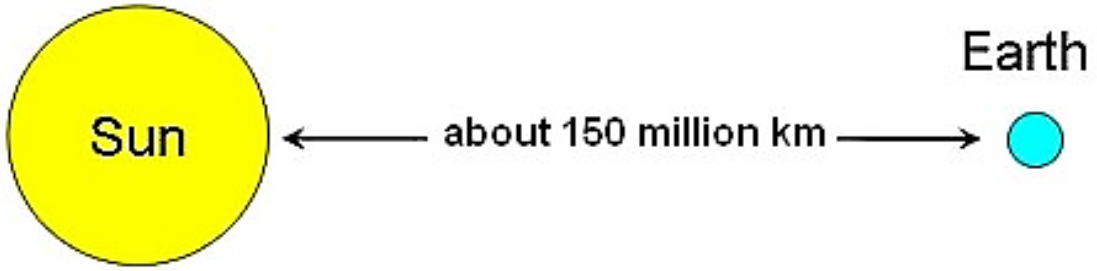
Physical Quantity	Name of Unit	Abbreviation
Mass	kilogram	kg
Length	meter	m
Time	second	s
Temperature	kelvin	K
Amount of substance	mole	mol
Electric current	ampere	A
Luminous intensity	candela	cd



All other units can be derived from these base units

Prefixes

Used to express small or large measured quantities in terms of a few simple digits



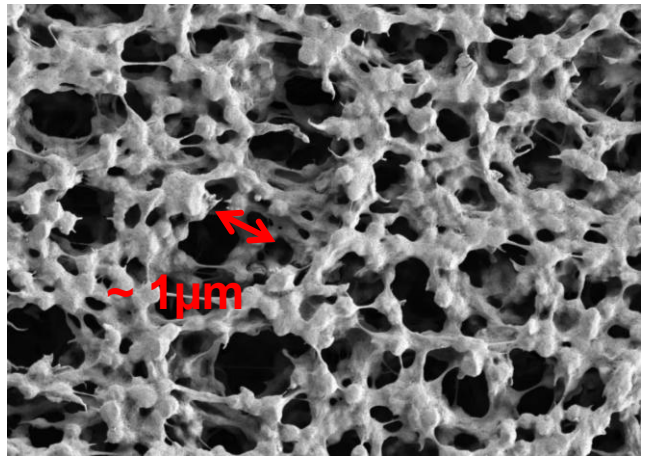
150 000 000 000 m

1.5×10^{11} m

1 µm

0.000001 m

1.0×10^{-6} m



Prefixes are used to indicate decimal **multiples** or **fractions** of various units. Prefixes convert the base units into units that are appropriate for the item being measured.

Prefixes for Units

quecto	q	0.000 000 000 000 000 000 000 000 000 001	10^{-30}	nonillionth
ronto	r	0.000 000 000 000 000 000 000 000 001	10^{-27}	octillionth
yocto	y	0.000 000 000 000 000 000 000 001	10^{-24}	septillionth
zepto	z	0.000 000 000 000 000 000 001	10^{-21}	sextillionth
atto	a	0.000 000 000 000 000 001	10^{-18}	quintillionth
femto	f	0.000 000 000 000 001	10^{-15}	quadrillionth
pico	p	0.000 000 000 001	10^{-12}	trillionth
nano	n	0.000 000 001	10^{-9}	billionth
micro	μ	0.000 001	10^{-6}	millionth
milli	m	0.001	10^{-3}	thousandth
centi	c	0.01	10^{-2}	hundredth
deci	d	0.1	10^{-1}	tenth
-	-	1	10^{0}	one
deka	da	10	10^{1}	ten
hecto	h	100	10^{2}	hundred
kilo	k	1 000	10^{3}	thousand
mega	M	1 000 000	10^{6}	million
giga	G	1 000 000 000	10^{9}	billion
tera	T	1 000 000 000 000	10^{12}	trillion
peta	P	1 000 000 000 000 000	10^{15}	quadrillion
exa	E	1 000 000 000 000 000 000	10^{18}	quintillion
zetta	Z	1 000 000 000 000 000 000 000	10^{21}	sextillion
yotta	Y	1 000 000 000 000 000 000 000 000	10^{24}	septillion
ronna	R	1 000 000 000 000 000 000 000 000 000	10^{27}	octillion
quetta	Q	1 000 000 000 000 000 000 000 000 000 000	10^{30}	nonillion

Note that a metric prefix simply represents a number:

$1\ mm = 1 \times 10^{-3}\ m$

$1\ mg = 1 \times 10^{-3}\ g$

Derived SI Units

The SI are used to derive the units of other quantities.

For example:

Speed is defined as the ratio of distance traveled to elapsed time.

$$\textbf{Speed} = \frac{length}{time} = \frac{m}{s}$$

Thus, the SI unit for speed is meters per second (m/s).

Other Derived SI Units

Length = L = m (SI unit, Base quantity)
Area = L x L = m x m = m^2
Volume = L x L x L = m x m x m = m^3

Force = m a = kg (m/s^2) = kg m s^{-2} = N

Energy = ½ m v^2 = kg $(m/s)^2$ = kg m^2 s^{-2} = J

Pressure = F / A = kg m s^{-2} / m^2 = kg m^{-1} s^{-2} = Pa

Mass and Weight

Mass: is a measure of the amount of matter in an object (SI unit of mass is the kilogram, kg).

Weight: is the force that gravity exerts on an object (SI unit is Newton, N).

In chemistry, the smaller gram (g) is more convenient:

$$1\ \text{kg} = 1000\ \text{g} = 1 \times 10^3\ \text{g}$$

Volume

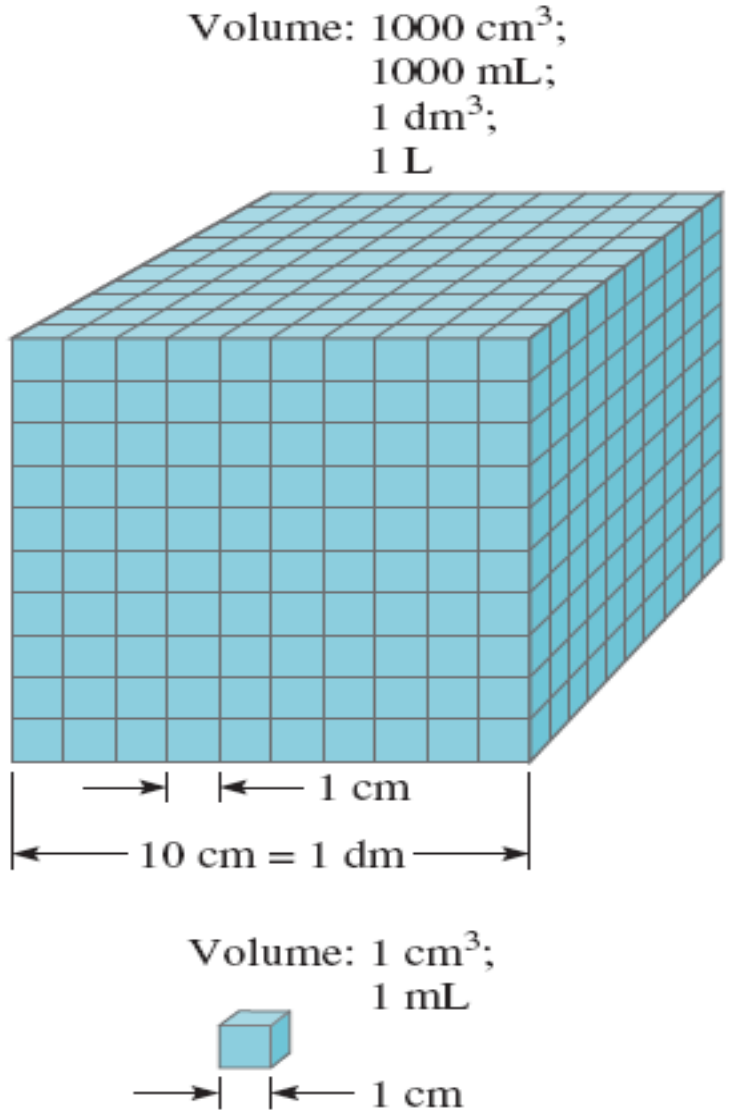
SI-derived unit for volume is the *cubic meter* (m^3).

Generally, however, chemists work with much smaller volumes, such as the cubic centimeter (cm^3) and the cubic decimeter (dm^3):

$$1\ cm^3 = (1 \times 10^{-2}\ m)^3 = 1 \times 10^{-6}\ m^3$$
$$1\ dm^3 = (1 \times 10^{-1}\ m)^3 = 1 \times 10^{-3}\ m^3$$

$1\ m^3 = 1000\ dm^3$
$1\ L = 1\ dm^3$
$1\ dm^3 = 1000\ cm^3$
$1\ cm^3 = 1\ mL$

Even though the liter is not an SI unit, volumes are usually expressed in liters (L) and milliliters (mL).



Laboratory Materials and Reagents

Materials used in the manufacture of common laboratory apparatus

Material	Max. working temp °C	Chemical Inertness	Notes
Borosilicate glass	200	Attacked somewhat by alkali solutions on heating	Trademarks: Pyrex (Corning Glass Works); Kimax (Owens-Illinois)
Soft glass		Attacked by alkali solutions	Boron-free. Trademark: Corning
Alkali-resistant glass			
Fused quartz	1050	Resistant to most acids, halogens	Quartz crucibles used for fusions
High-silica glass	1000	More resistant to alkalis than borosilicate	Similar to fused quartz Trademark: Vycor (Corning)
Porcelain	1100–1400	Excellent	
Platinum	1500	Resistant to most acids, molten salts. Attacks by aqua regia, fused nitrates, cyanides, chlorides at > 1000°C. Alloys with gold, silver, and other metals	Usually alloyed with iridium or rhodium to increase hardness. Platinum crucibles for fusions and treatment with HF
Nickel and iron		Fused samples contaminated with the metal	Ni and Fe crucibles used for peroxide fusions
Stainless steel	400–500	Not attacked by alkalis and acids except conc. HCl, dil. H_2SO_4, and boiling conc. HNO_3	
Polyethylene	115	Not attacked by alkali solutions or HF. Attacked by many organic solvents (acetone, ethanol OK)	Flexible plastic
Polypropylene	120	Translucent. Has replaced polyethylene for many purposes	
Polystyrene	70	Not attacked by HF. Attacked by many organic solvents	Some what brittle
Teflon	250	Inert to most chemicals	Useful for storage of solutions and reagents for trace metal analysis. Is permeable to oxygen

The Analytical Balance

The balance measures mass. The analytical balance is indispensible tool in all laboratory.

Modern **electronic balances** offer convenience in weighing and are subject to fewer errors or mechanical failures than are **mechanical balances**, which have become largely obsolete.

- Zero-point Drift
- Greater precision equals greater cost.

Single-pan mechanical balance

Electronic analytical balance

Weighing bottles

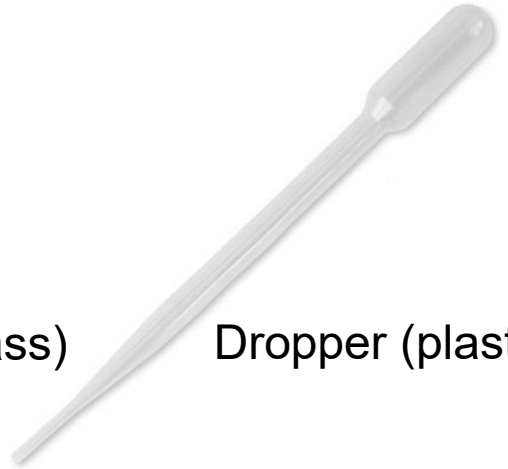
Weighing dishes

Dish metal

Dish plastic

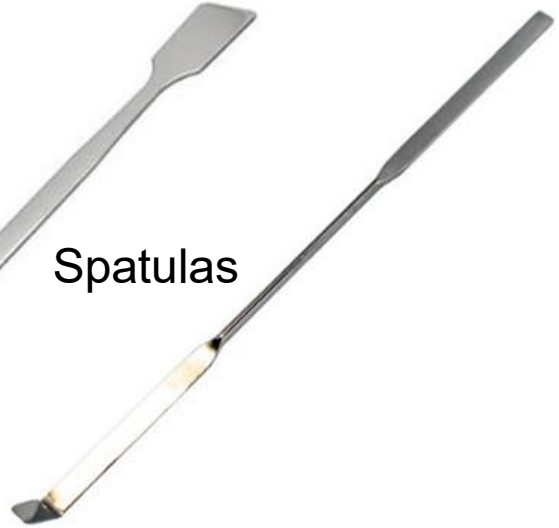
Pan metal

Dropper (glass)

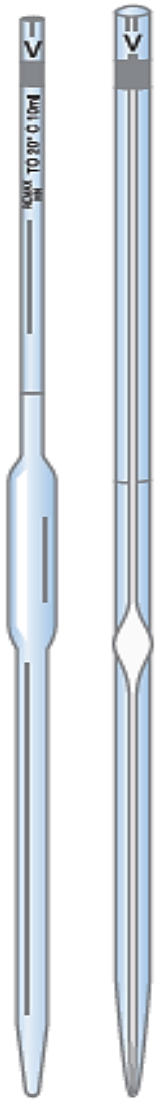


Dropper (plastic)

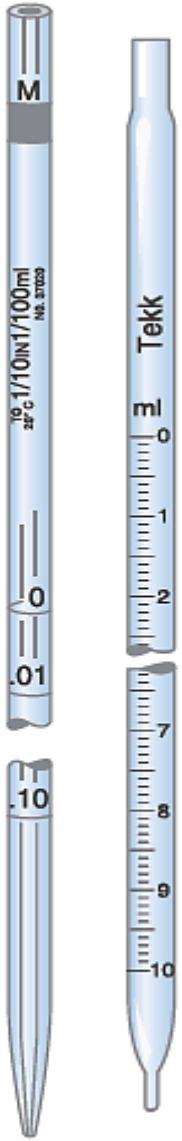
Spatulas



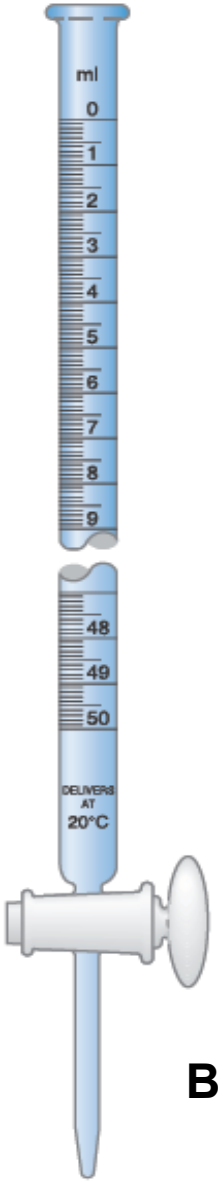
Volumetric Ware



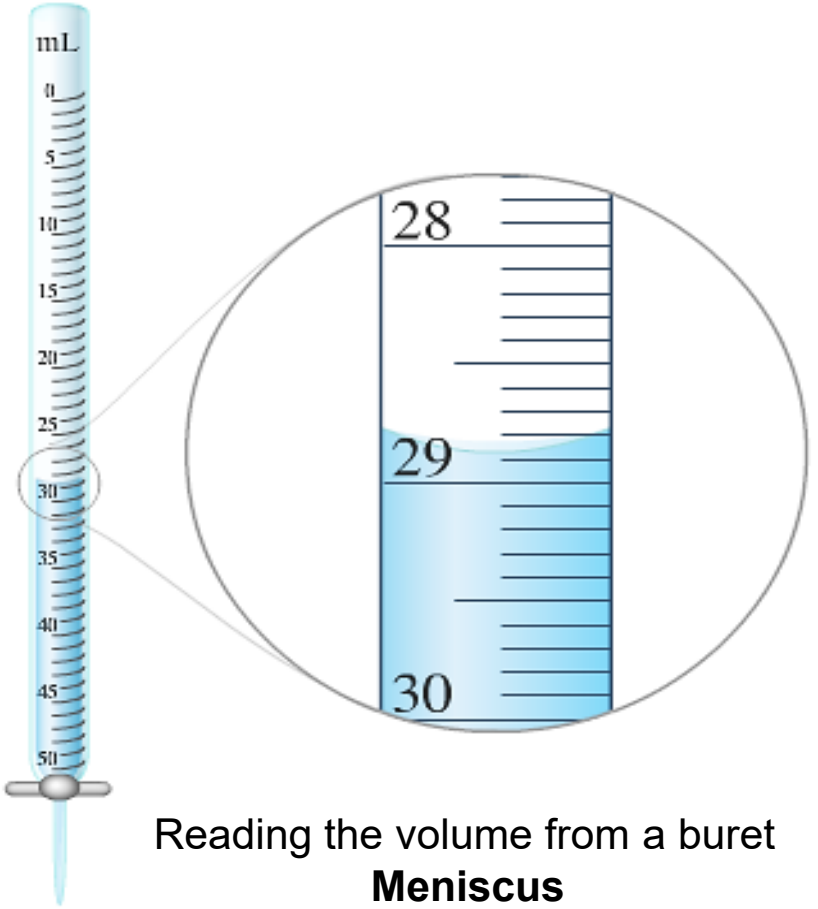
Transfer or volumetric pipets



Volumetric pipets; deliver an accurate volume



Buret



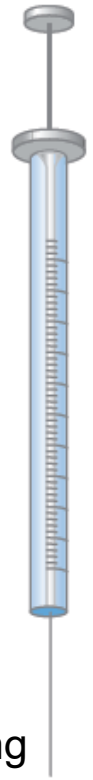
Reading the volume from a buret
Meniscus

Graduated cylinders

Volumetric flask;
contain an
accurate volume

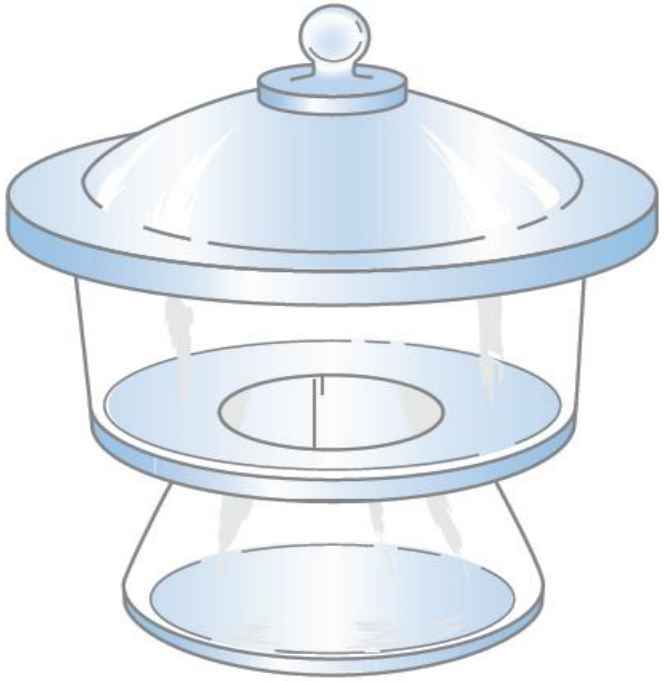
Single-channel & multichannel
micropipettes

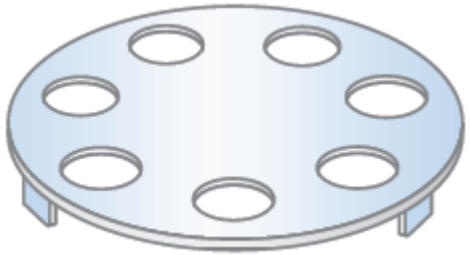
Micropipette tips



Syringe pipets;
useful for delivering
microliter volumes

Other Apparatus





Desiccator plate

Desiccator; used to keep samples dry while they are cooling and before they are weighed and to dry a wet sample. Dried or ignited samples and vessels are cooled in the desiccator.

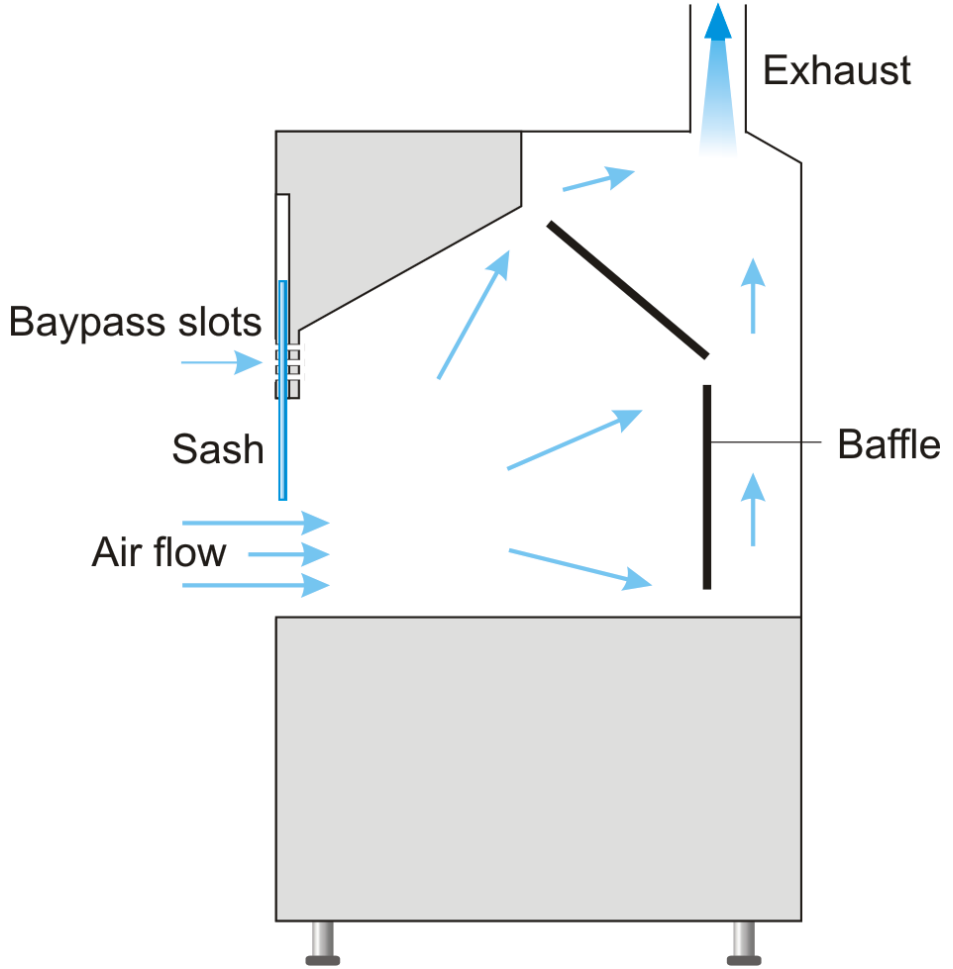
Some common drying agents
- $CaCl_2$ (anhydrous)
- $CaSO_4$
- CaO
- $MgClO_4$ (anhydrous)
- Silica gel
- Al_2O_3

Furnaces and Ovens

Muffle furnace; used to ignite samples to high temperatures, either to convert precipitates to a weighable form or to burn organic materials prior to inorganic analysis
Up to about 1200^{o}C

Drying oven; used to dry samples prior to weighing. These ovens are well ventilated for uniform heating. The usual drying temperature is about 110^{o}C.
Up to temperatures of 200 to 300^{o}C.

Fume hood; used when chemicals or solutions are to be evaporated. e.g., perchloric acid or acid solutions of perchlorates are to be evaporated, the fumes should be collected, or the evaporation should be carried out in fume hoods.



Laboratory Safety

Before beginning any of the experiments, you must familiarize your self with laboratory safety procedures.

Laboratory safety consists guidelines and rules for,,,

- Personal protection,
- Laboratory protocol,
- Operation in the laboratory,
- Recommended laboratory techniques,
- Chemical hazards,
- Chemicals handling,
- Disposal of chemicals,
- Instructions on material safety data sheets (MSDSs),
- Safety equipment,
- Emergency procedures.

- Chemicals safety

Use, handling, storage and disposing of hazardous chemicals.

- Biological safety

Use of biological agents such as human, animal, and plant pathogens; human blood and blood components; recombinant/synthetic nucleic acids; cell and tissue cultures; etc. in addition to the receipt, possession, use, and transfer of biological materials.

- Facilities and construction safety

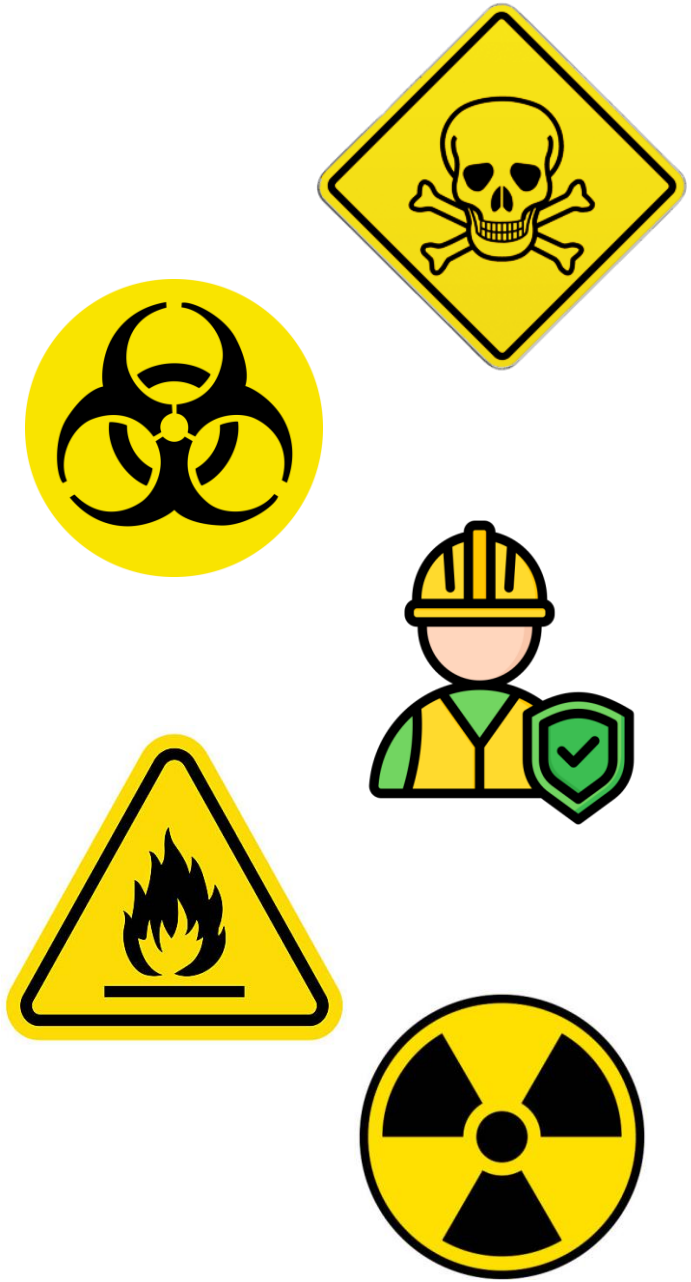
Guidelines for construction and renovation for good health and safety practices, even for small construction or renovation projects.

- Fire safety

The fire safety includes building evacuation, fire prevention methods and the use of portable extinguishers.

- Radiation safety

The use of sources of radiation in various teaching, research, and operating activities.

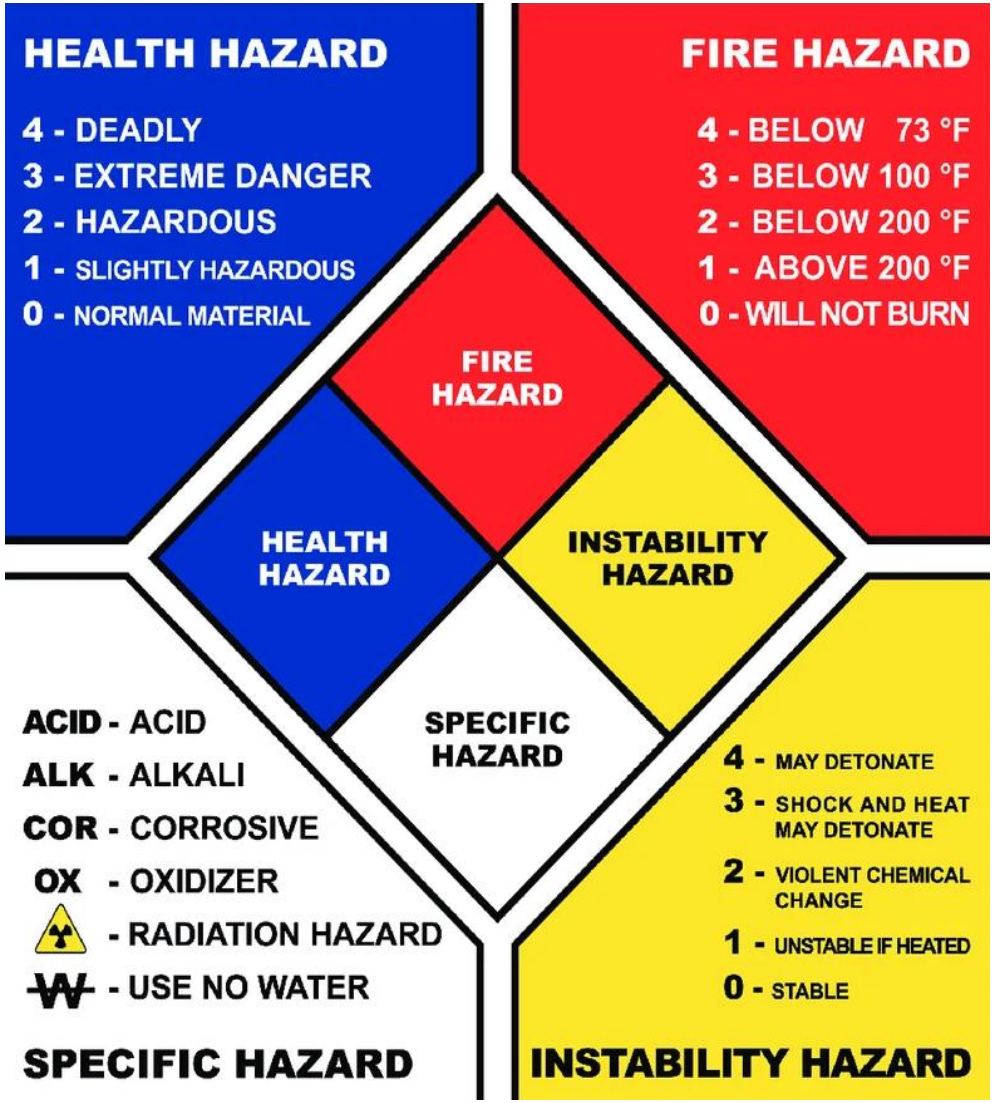


Safety Wears and Protective Equipment

Personal protection

Main personal protective equipment:
- Lab coats
- Safety glasses
- Gloves
- Face masks
- Closed-toe shoes

Hazardous Materials



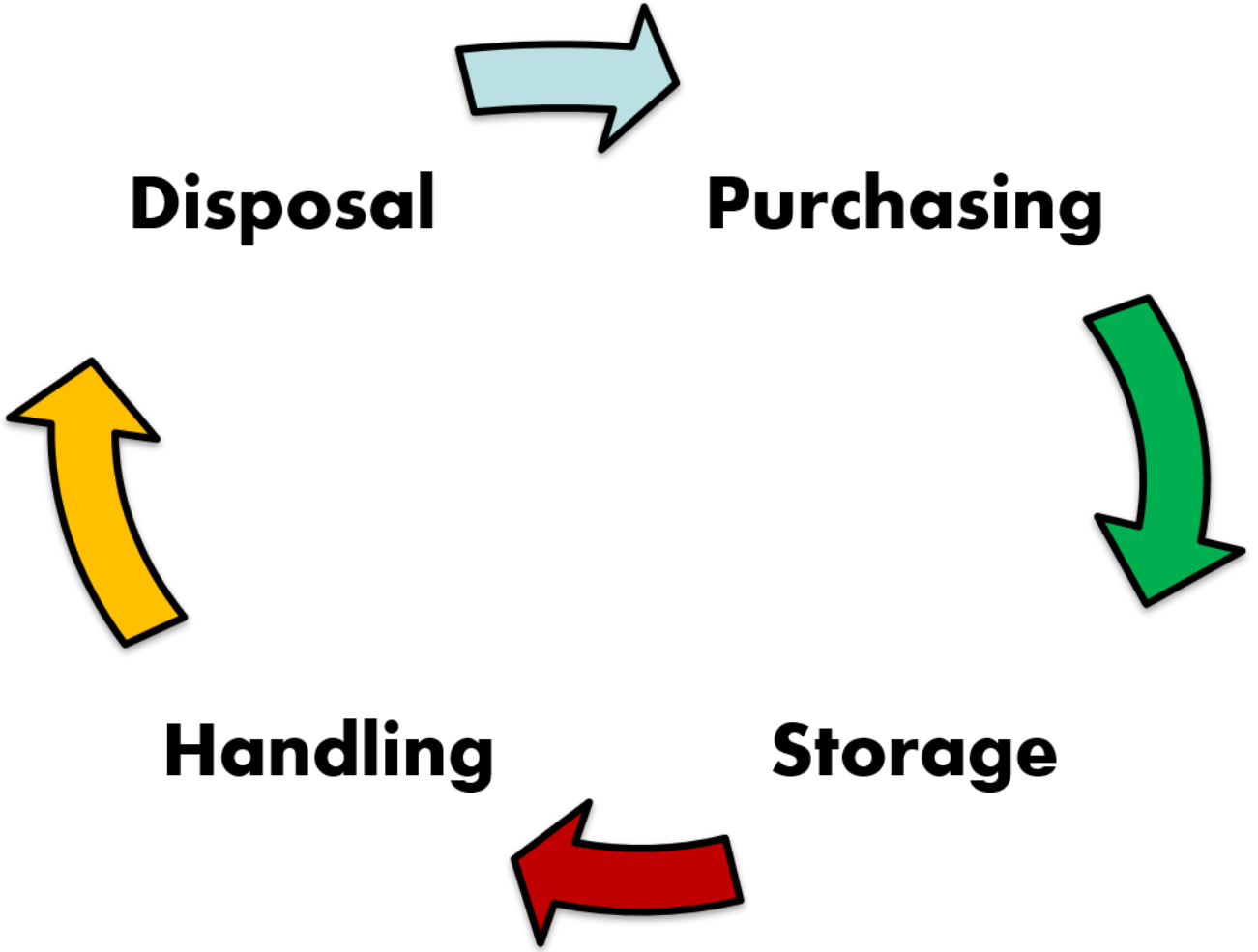
Fire Diamond - Hazardous Materials

Characteristic hazardous wastes are materials that are known or tested to exhibit one or more of the following four hazardous traits:

- Ignitability (i.e., flammable)
- Reactivity (explosions)
- Corrosivity
- Toxicity

Chemicals Life Cycle

The total chemical life cycle defines the stages of a chemical's **purchase**, **use**, and **disposal**.




Thank You!