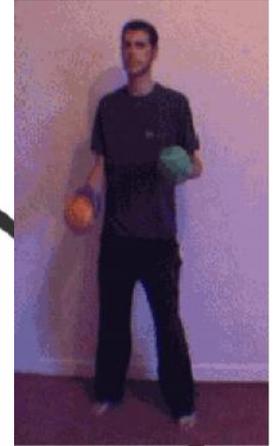




Setup of Accredited Laboratory for Drinking Water Quality Analysis



Dr. Rakesh Kumar

**NABL Lead Assessor and retired from Indian Institute of
Toxicology Research
Lucknow-226001**

Email: rkchitranshi@rediffmail.com

85%

Pass/Fail

A

F

B

C+

93%

Assessment, Scoring, and Evaluation

A-

67%

S

D

Unsatisfactory

GRADES

C



Definitions

- **Assessment** -- *The process of measuring something with the purpose of assigning a numerical value.*
- **Scoring** -- *The procedure of assigning a numerical value to assessment task.*
- **Evaluation** -- *The process of determining the worth of something in relation to established benchmarks using assessment information.*

The **liberalization of economies** and the **globalization of markets** have intensified international competition. This has brought particular challenges to companies in developing countries.

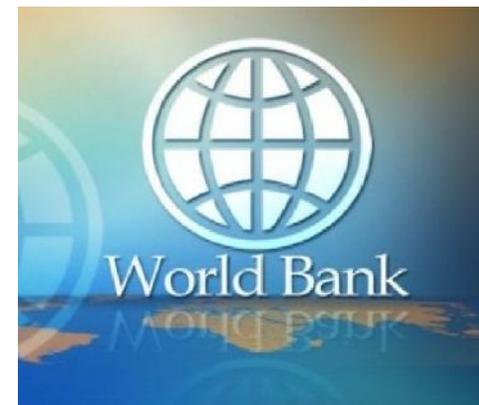
Developing countries need to have a credible conformity assessment infra structure to certify that their products conform to international standards.

Testing laboratories are an essential component of this infra structure.

Testing Labs in developing countries face major problems in



- In measurement**
- Product certificates.**





WTO recognized this as a Technical Barrier to trade and established the **TBT** Agreement.

The test result can only be mutually acceptable if there is a mechanism where by the user has confidence in the technical competence of laboratories and soundness of their measurements.

Global Conformity Assessment System for testing **i.e.**

ISO/IEC-17025 :

Guide Book as a tool in assisting developing countries to achieve international accreditation of their testing laboratories and consequent acceptance of their products in other countries , thus enabling them to over come a major technical barrier to trade and to fully exploit their competitive strength in the global market.



Role of Accredited Test Laboratories in Testing & Calibration Programmes

Accreditation

Definition: Procedure by which an authoritative body gives formal recognition that a body or person is competent to carry out specific tasks - ISO/IEC Guide 2 : 1996.

Key points:

- ☞ Checks conformity to all clauses of ISO/IEC 17025 (which include all ISO 9001 elements).
- ☞ Assures the client that the procedures and test results are technically valid.
- ☞ Recognizes the technical competence of laboratory staff.
- ☞ Endorses the quality management system.

Some Benefits of Laboratory Accreditation

- ⇒ Provides formal recognition to competent laboratories and ensures that they perform their work in accordance with international criteria.
- ⇒ Minimises the risk of unreliable results which, in turn, reduces the risk for manufacturers or suppliers to produce or supply a faulty product.
- ⇒ Minimises the chances of retesting and hence reduces chances of additional financial burden and time delays.

Some Benefits of Laboratory Accreditation

- ⇒ Enhances Customer confidence and Satisfaction.
- ⇒ International acceptability of test results. Based on mutual evaluation and acceptance of other country's laboratory accreditation systems, international agreements called Mutual Recognition Arrangements (MRA) have been established for realizing the ideal of having products "tested once and accepted everywhere". Such agreements are crucial in enabling test data by an accredited laboratory to be accepted in overseas markets and facilitate trade.

Competence

- Raise standard to meet international standards
- Operation
 - ISO/IEC 17025 – testing and cal labs
 - ISO 15189 – medical testing labs
 - ISO/IEC 17043 – proficiency testing providers
 - ISO Guide 34 – reference material producers
 - ISO/IEC 17020 – inspection bodies
 - ISO/IEC 17021 – management system CBs
 - ISO/IEC Guide 65 – Product CBs

About NABL

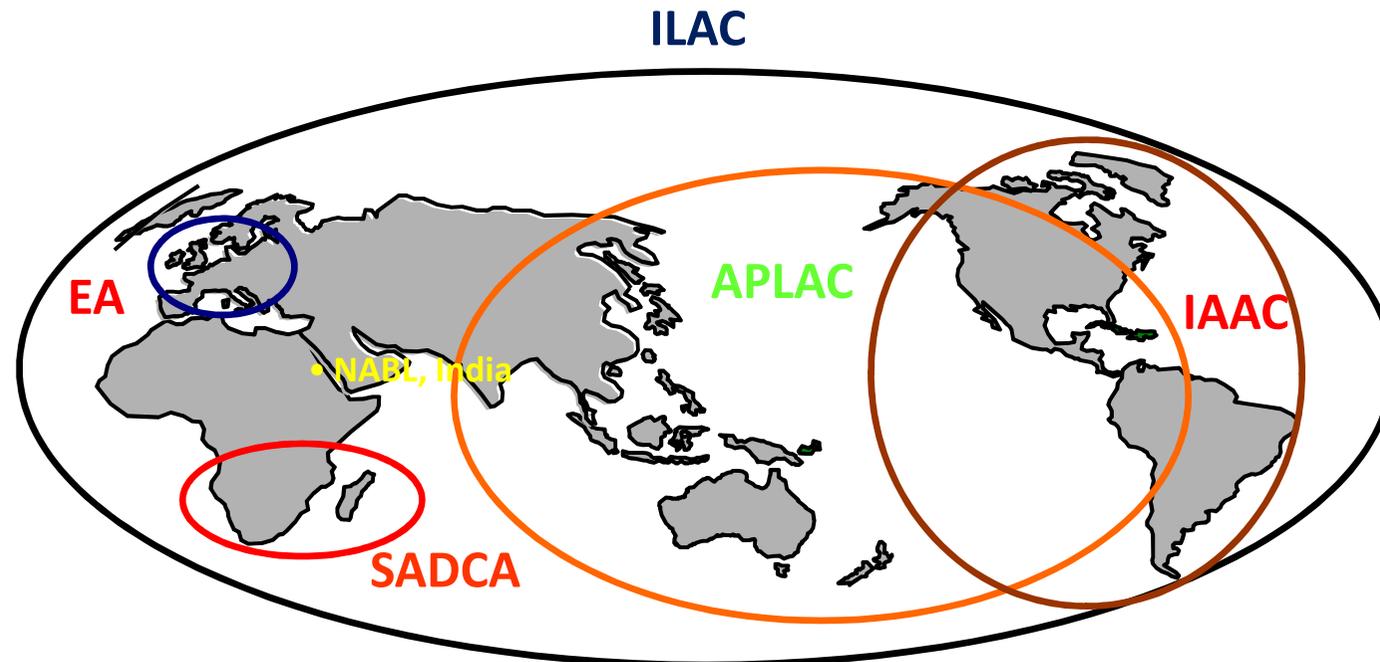
☞ NABL provides laboratory accreditation services to laboratories that are performing tests and calibrations in accordance with ISO/IEC 17025: 1999 (General requirements for the competence of Testing and Calibration Laboratories).

☞ NABL has established its accreditation system in accordance with ISO IEC Guide 58, which is followed internationally.

Calibration and testing laboratory accreditation systems -- General requirements for operation and recognition

☞ NABL is signatory to APLAC Mutual Recognition Arrangement & ILAC Mutual Recognition Arrangement since October 2000 & November 2000 respectively.

The International Picture



- EA** European co-operation for Accreditation
- APLAC** Asia Pacific Laboratory Accreditation Cooperation
- ILAC** International Laboratory Accreditation Cooperation
- SADCA** Southern African Development Cooperation for Accreditation
- IAAC** Inter-American Accreditation Cooperation
- Unaffiliated Bodies** – Peer evaluated ABs who are not geographically located in one of the established regions

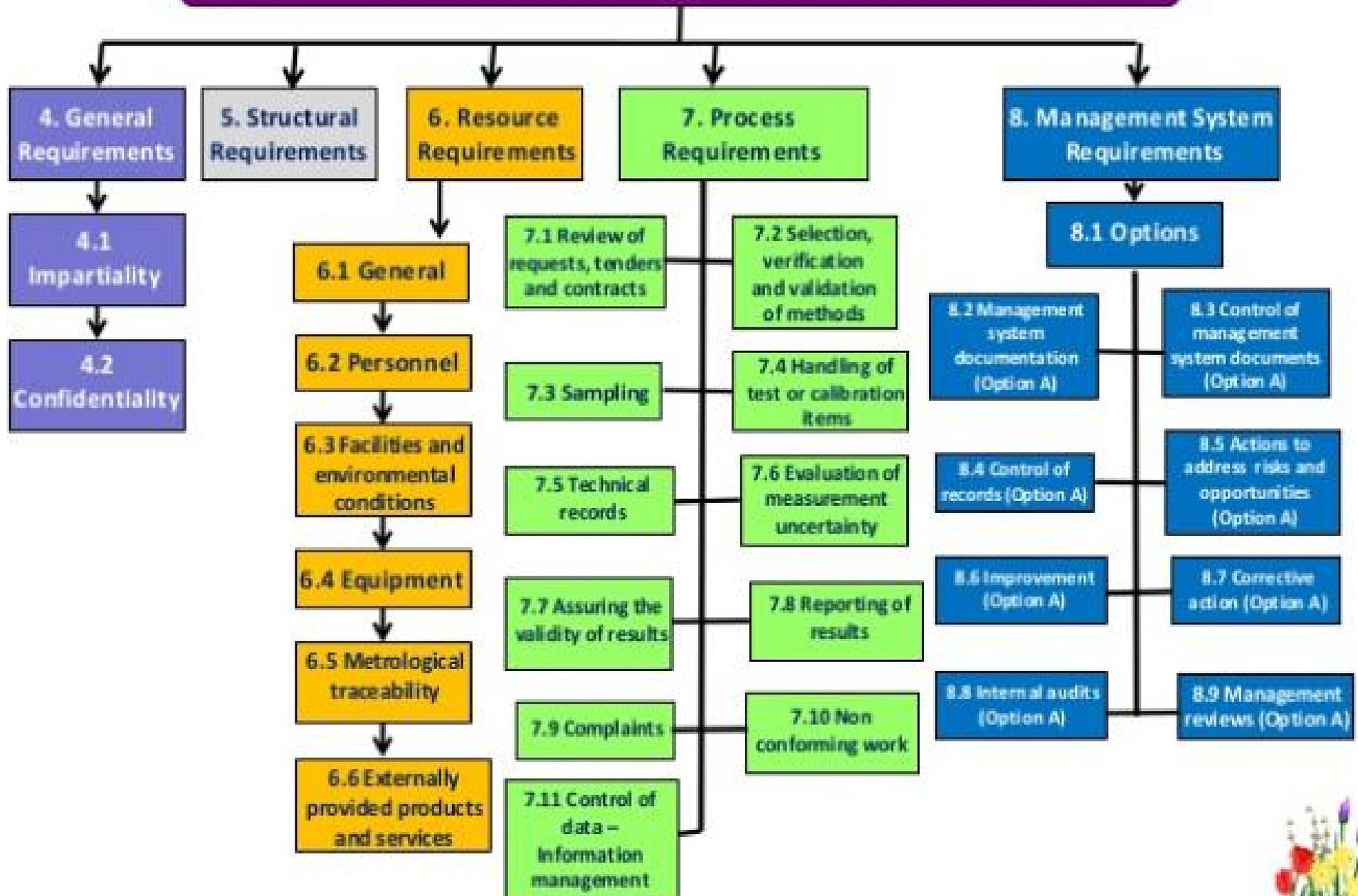
ISO/IEC Guide 25:1990

ISO/IEC 17025:1999

ISO/IEC 17025:2005

ISO/IEC 17025:2017

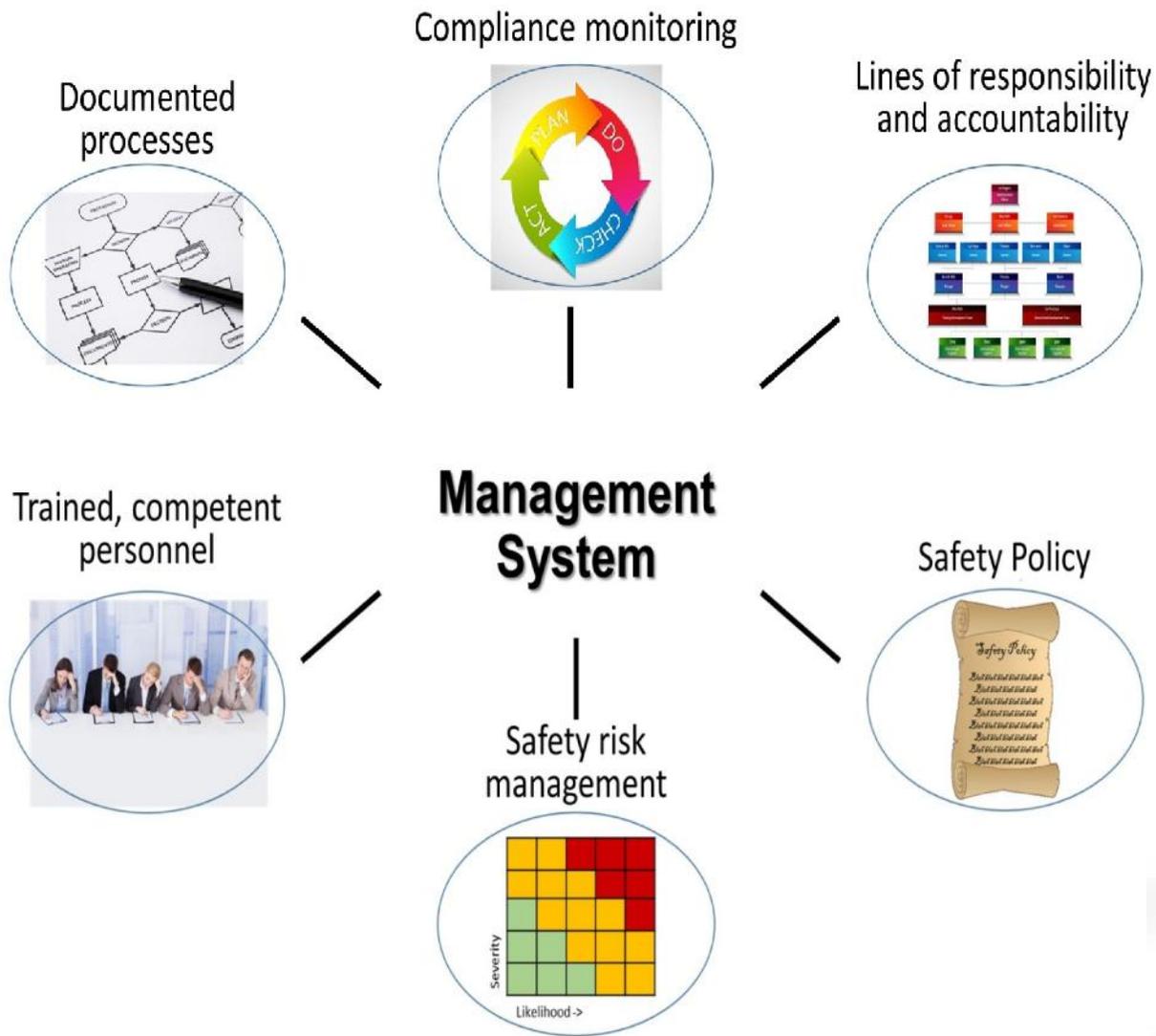
ISO/IEC 17025: 2017 Requirements



Contents of ISO / IEC 17025 : 2017

1. Scope
2. Normative References
3. Terms and Definitions
4. General Requirements
5. Structural Requirements
6. Resources Requirements
7. Process Requirements
8. Management System Requirements

Management System Requirement



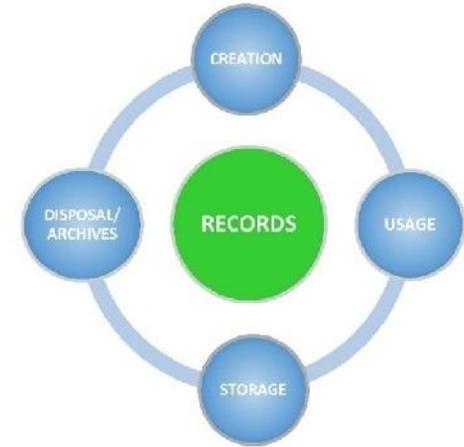
Management System Documentation (Option A)

System acknowledged and implemented all levels

- Level 1 Quality Manual
- Level 2 Quality Assurance Procedures
- Level 3 Standard Operating Procedures for Tests, Equipment's and Calibration
- Level 4 Forms & Formats, Records



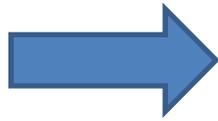
Control of Records



Establish and retain legible records to demonstrate fulfilment of the requirements in this document (NEW)

Technical records .





PERSONNEL

ACOMODATION/FACILITIES

EQUIPMENT



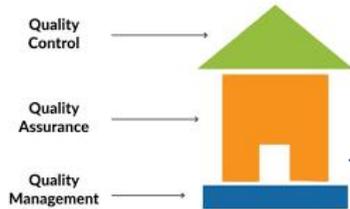
REPORTING



WATER TESTING LABORATORY



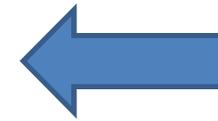
METROLOGICAL TRACEABILITY



QUALITY CONTROL



SAMPLING



SELECTION OF METHOD

Selecting Test Methods

Depends on:

- Objectives
- Range of concentration
- Required accuracy and precision
- Time period between sampling and analysis
- Technical skills and equipment required
- Familiarity with the method
- Availability of resources

1.PERSONNEL

- Sufficient in number
- Qualified
- Trained
- Competent
- Responsible for authorized work only



2. ACCOMMODATION and FACILITIES

- Suitable for different activities
- Well Separated rooms for Equipment, Wet Lab, Hot Zone and Storage room
- Maintenance of Environmental conditions(temperature and Humidity).
- Entry is restricted
- Use PPE in Laboratory
- Good Housekeeping





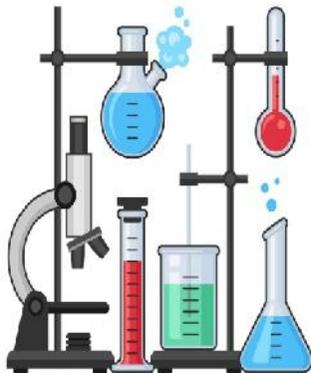
Lab should ensure environmental condition ,lighting, energy sources etc to get correct results





3. EQUIPMENT

- Includes all apparatus, equipment ,software, reagents, glassware, chemicals and Reference standards
- All Equipment must be calibrated and calibration status must be maintained by doing intermediate checks.
- All Equipment must have UID and log books
- All equipment must have maintenance plan.
- Equipment are operated by authorized personnel only.



VectorStock
VectorStock.com/2512068

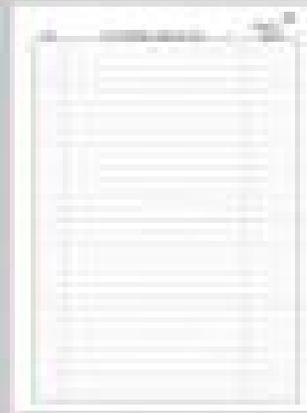




**Unique identification
of each equipment.**



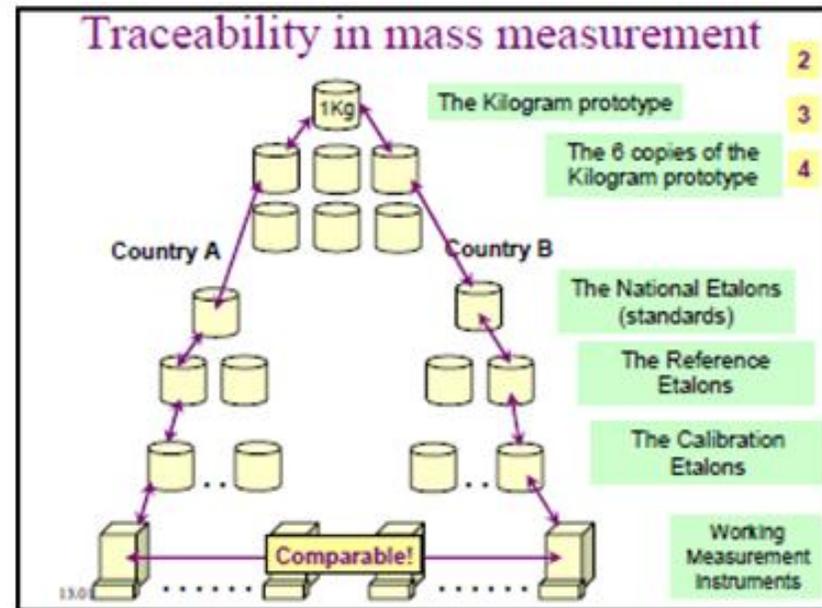
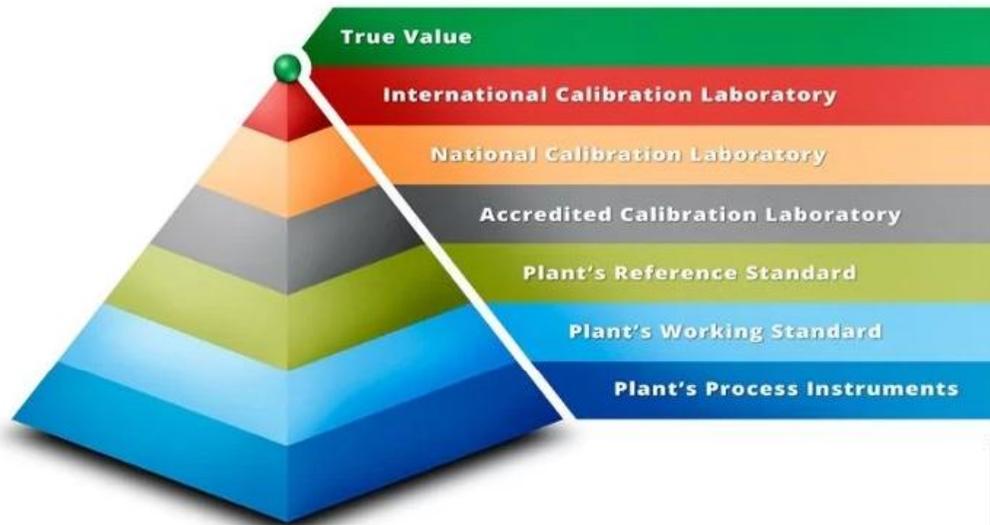
**Equipment
Log Book**



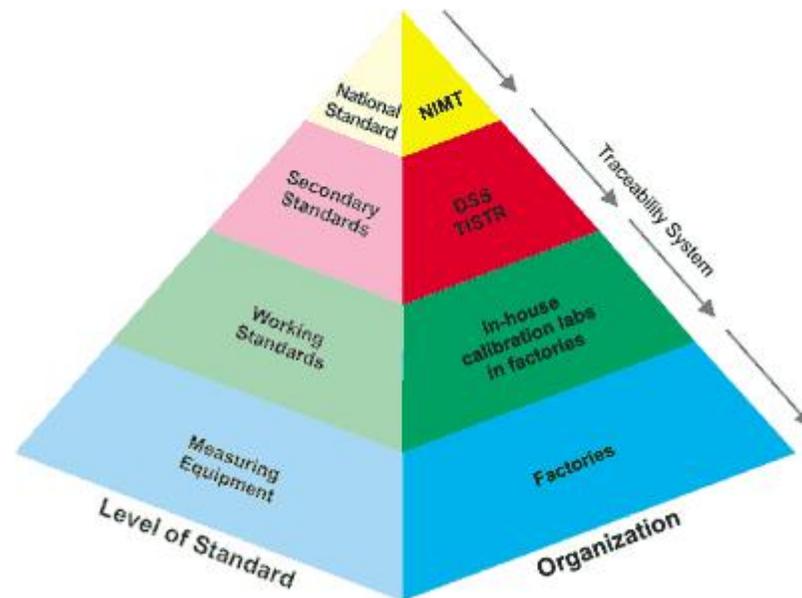
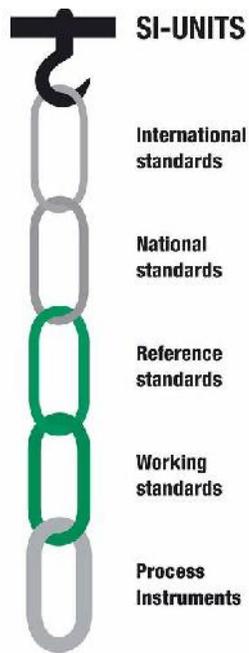
Log Books of Equipment

4. Metrological Traceability

These requirements have been made clearer, with simplified text.



1: The laboratory must maintain metrological traceability of its measurement results by a documented unbroken chain of calibrations, each contributing to the measurement uncertainty, linking them to an appropriate reference.



- 2: Measurement results are to be traceable to SI units through either:
- a) calibration by a competent laboratory;
 - b) certified values of certified reference materials from a competent producer with stated traceability to SI units;
 - c) direct realisation of the SI units.

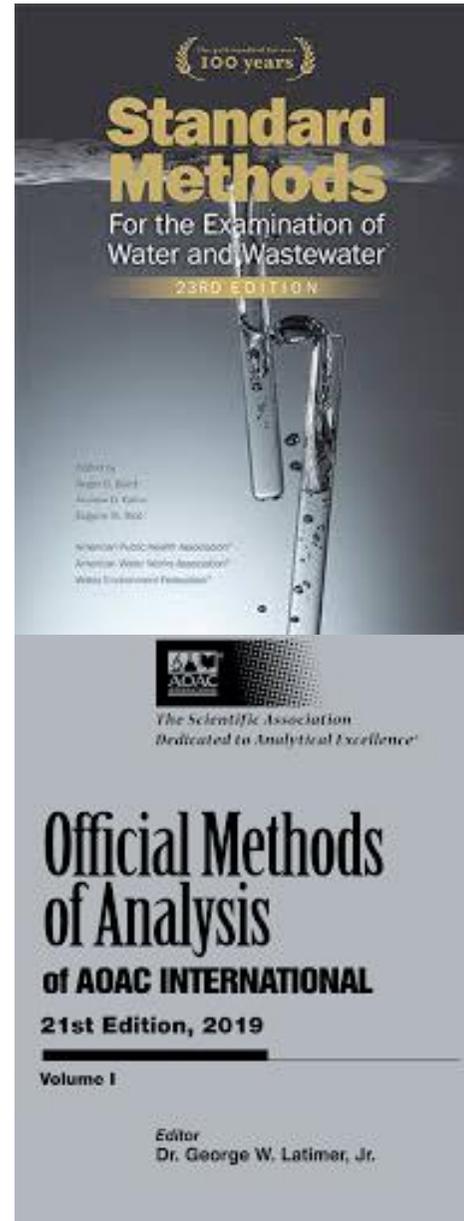
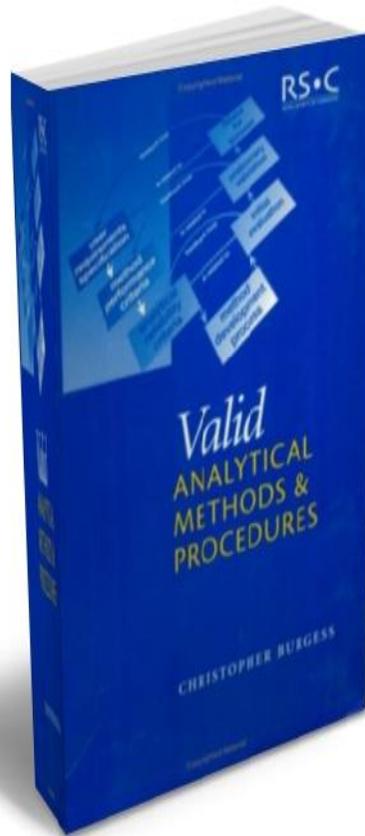
Clarity provided that traceability to SI may be achieved through the use of certified reference materials.



5. SELECTION OF METHOD



Appropriate methods



Method up to date, readily available to personnel

Standard Operating Procedure (SOP)

- A unique identification number of SOP
- Its title.
- Its application and limitations.
- the adaptation and amendment date and amendment number.
- A brief description of its principle.
- Reference to the standard on which the procedure is based, if applicable.
- The name of equipment and chemical and reagents required.
- An unambiguous step-by-step test instruction to be followed by technical staff.
- The precision of the method whenever applicable.
- Detail instructions for handling, transportation and storage and preparation of items (eg Standards), estimation of uncertainty and precaution to be taken.
- Procedure for record of data produced.



Verify method before use

Method Verification

- importing a validated method
- show that laboratory can do it at its site
- demonstrate that laboratory can repeat the method performance

Aspect of a Verification

Be extensive enough to show that the method is fit for the intended use and meets the customer's needs



Method Verification

Standard methods shall be verified for:

1. the equipment



2. the required reference materials/standard, reagents



3. the environmental conditions



4. testing staff member competence to perform the test



5. capability to achieve the **method performance**



Validation for car driving



Car driving Training

Verification of Car driving



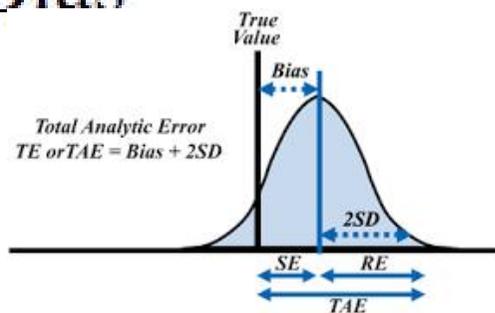
Car driving

Method Verification

To demonstrate you can repeat the method performance, including:

- Detection limits
- Precision
- Bias

NOISE POLLUTION



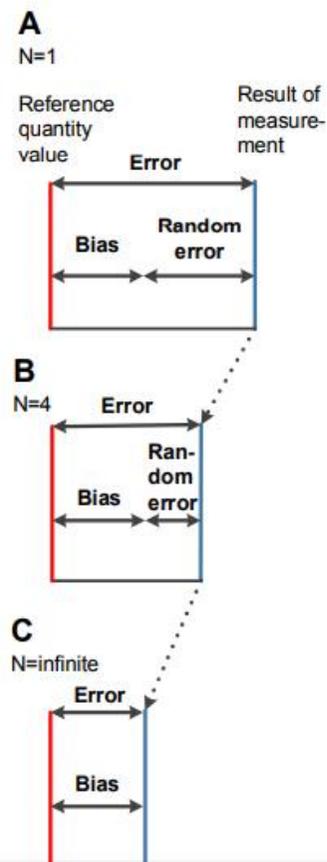
Method Verification – Bias

- To demonstrate the absence of lab bias
- Proficiency tests/interlaboratory comparisons
- Analysis of CRMs



Bias

- **Trueness** is the “closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value”. It is quantitatively expressed as ***bias***.



Water



Who am I?

**I am tasteless, I am Colourless,
I am shapeless, I am Odourless,
But without me, there's no life.**

Can you tell me who m I?

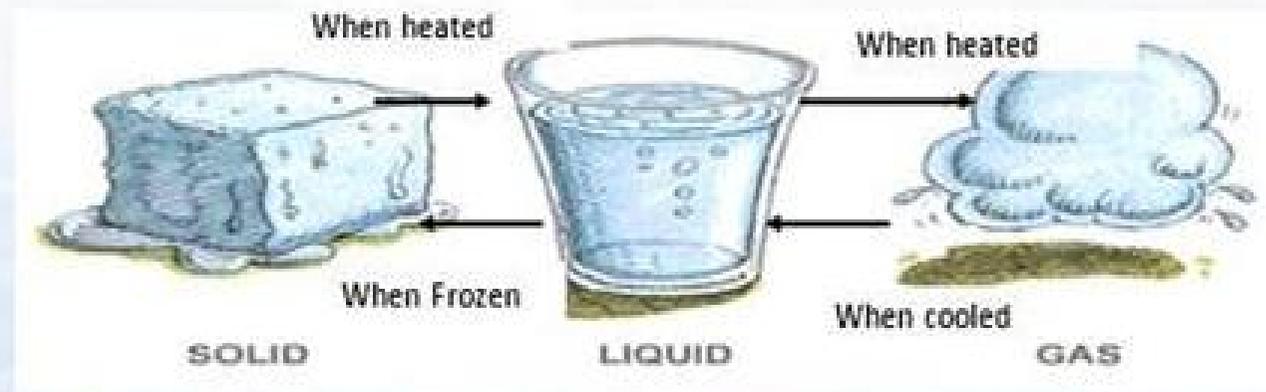
Yes! I 'm Water

Introduction to Water



- Water is the most common substance found on the Earth.
- About $3/4^{\text{th}}$ of the Earth is covered with water.
- Water can be found in ponds, rivers, lakes, oceans and seas.

Three Forms of Water



When water is heated, it into **steam** changes or **water** water vapour. This water vapour is a gas, which can be changed into by cooling. When kept in the 'freezer' of a refrigerator, water will change into solid **ice**. If you keep the ice in a warm place, it will change into water.

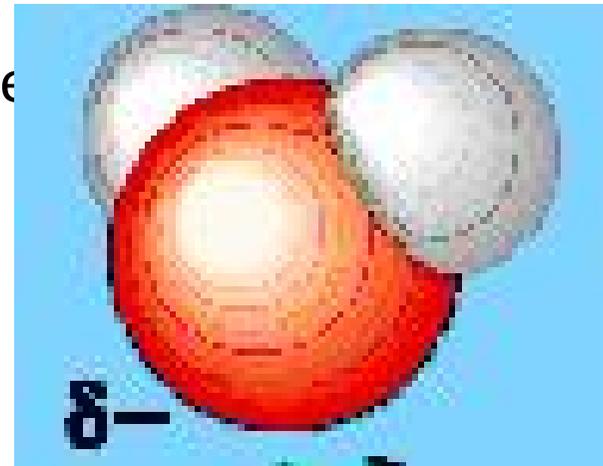
Properties of Water

- Polar molecule
- Cohesion and adhesion
- High specific heat
- Density – greatest at 4°C
- Universal solvent of life



Polarity of Water

- In a water molecule two hydrogen atoms form single polar covalent bonds with an oxygen atom. Gives water more structure than other liquids
 - Because oxygen is more electronegative, the region around oxygen has a partial negative charge.
 - The region near the two hydrogen atoms has a partial positive charge.
- A water molecule is a polar molecule with opposite ends of the molecule with opposite charges.



- Water has a variety of unusual properties because of attractions between these polar molecules.
 - The slightly negative regions of one molecule are attracted to the slightly positive regions of nearby molecules, forming a hydrogen bond.
 - Each water molecule can form hydrogen bonds with up to four neighbors.

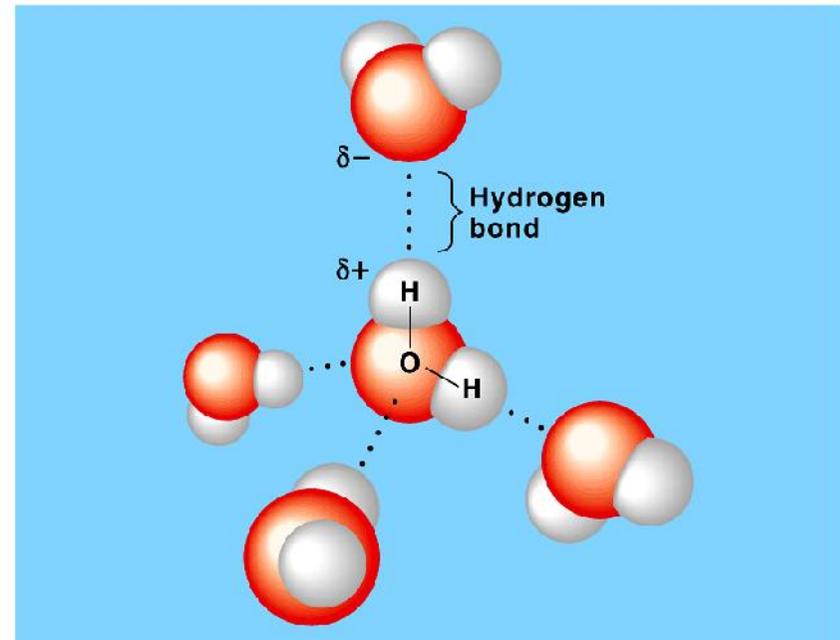
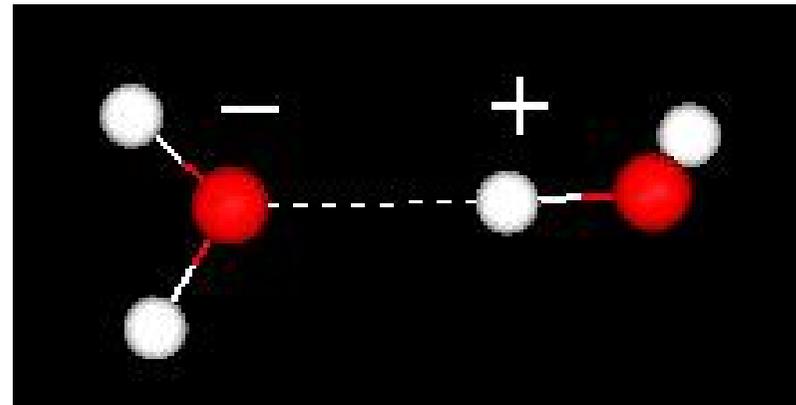


Fig. 3.1

HYDROGEN BONDS

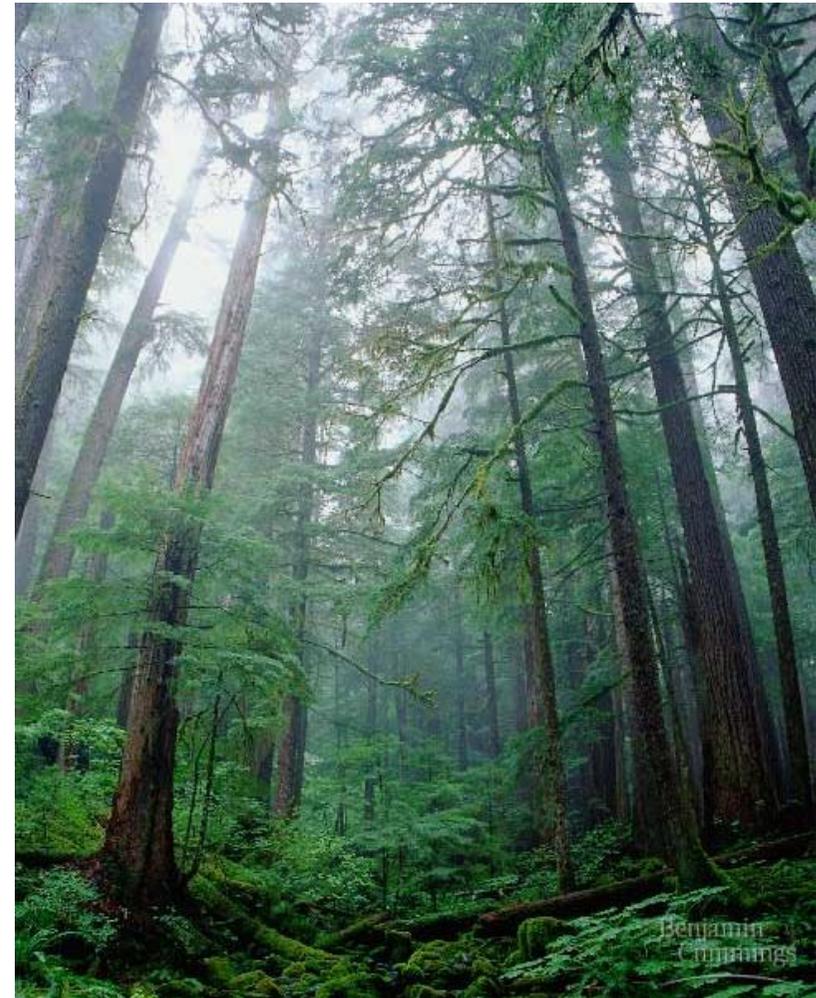
- Hold water molecules together
- Each water molecule can form a maximum of 4 hydrogen bonds
- The hydrogen bonds joining water molecules are weak, about $1/20^{\text{th}}$ as strong as covalent bonds.
- They form, break, and reform with great frequency
- Extraordinary Properties that are a result of hydrogen bonds.
 - Cohesive behavior
 - Resists changes in temperature
 - High heat of vaporization
 - Expands when it freezes
 - Versatile solvent



Organisms Depend on Cohesion

Hydrogen bonds hold the substance together, a phenomenon called cohesion

- Cohesion is responsible for the transport of the water column in plants
- Cohesion among water molecules plays a key role in the transport of water against gravity in plants
- Adhesion, clinging of one substance to another, contributes too, as water adheres to the wall of the vessels.



- **Surface tension**, a measure of the force necessary to stretch or break the surface of a liquid, is related to cohesion.
 - Water has a greater surface tension than most other liquids because hydrogen bonds among surface water molecules resist stretching or breaking the surface.
 - Water behaves as if covered by an invisible film.
 - Some animals can stand, walk, or run on water without breaking the surface.



Fig. 3.3

Moderates Temperatures on Earth

Water stabilizes air temperatures by absorbing heat from warmer air and releasing heat to cooler air.

Water can absorb or release relatively large amounts of heat with only a slight change in its own temperature.

Celsius Scale at Sea Level	
100°C	Water boils
37°C	Human body temperature
23°C	Room temperature
0°C	Water freezes

- What is kinetic energy?
- Heat?
- Temperature?
- Calorie?
- What is the difference in cal and Cal?
- What is specific heat?

Density of Water

- Most dense at 4°C
- Contracts until 4°C
- Expands from 4°C to 0°C



The density of water:

- 1. Prevents water from freezing from the bottom up.**
- 2. Ice forms on the surface first—the freezing of the water releases heat to the water below creating insulation.**
- 3. Makes transition between season less abrupt.**

- When water reaches 0°C , water becomes locked into a crystalline lattice with each molecule bonded to to the maximum of four partners.
- As ice starts to melt, some of the hydrogen bonds break and some water molecules can slip closer together than they can while in the ice state.
- Ice is about 10% less dense than water at 4°C .

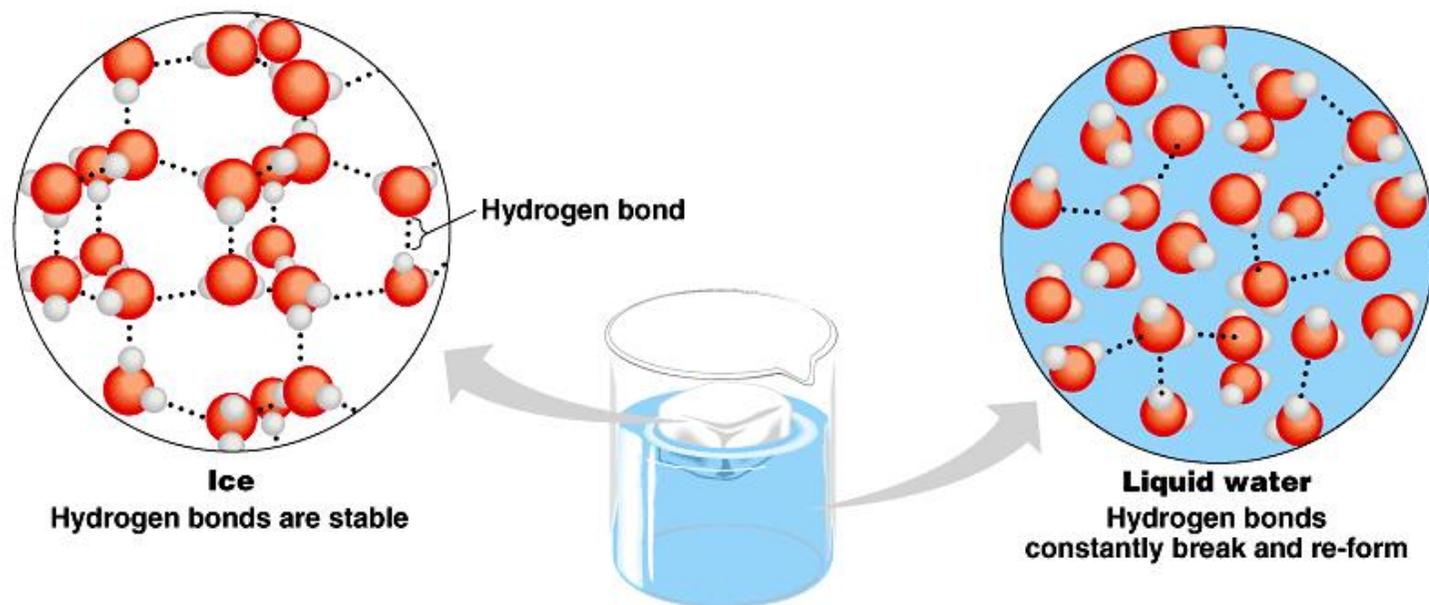
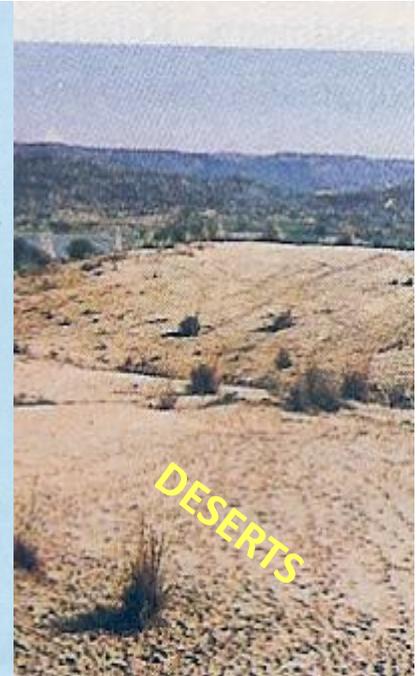
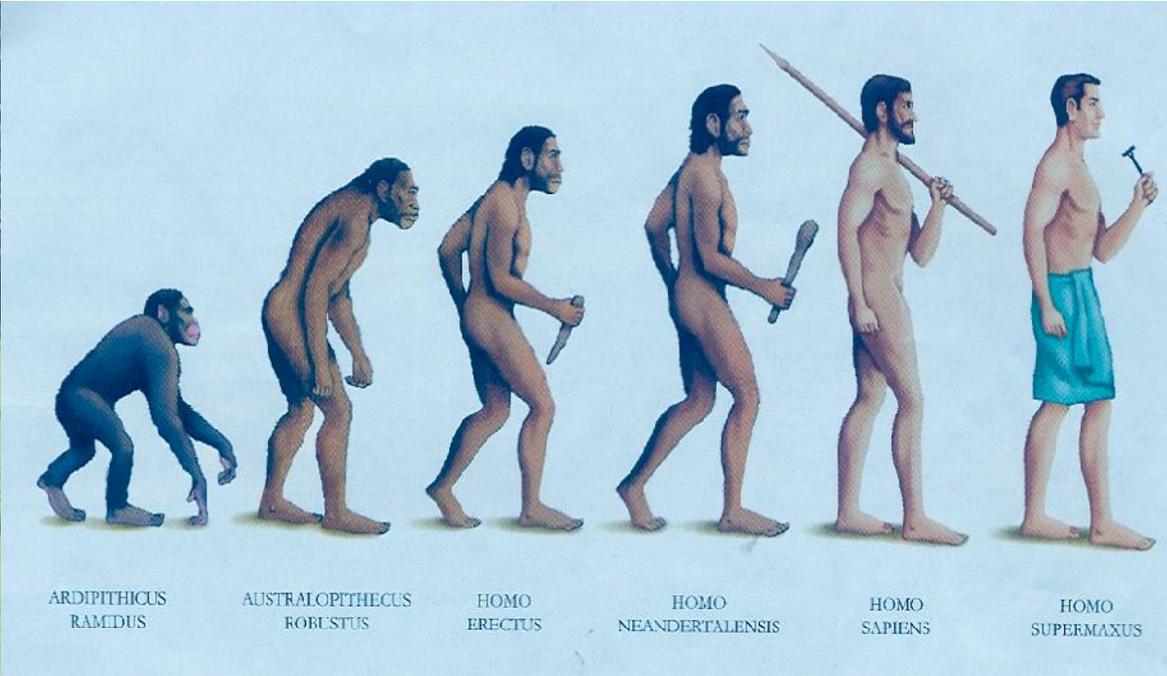
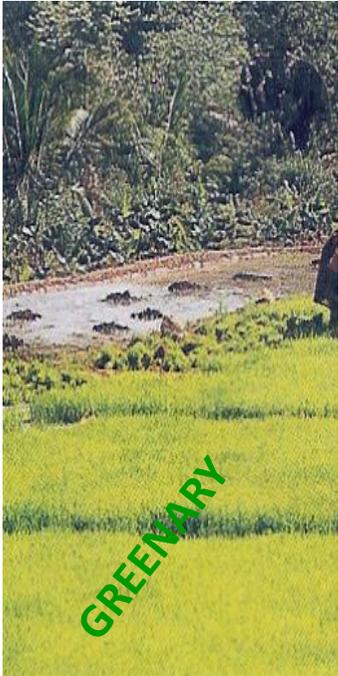
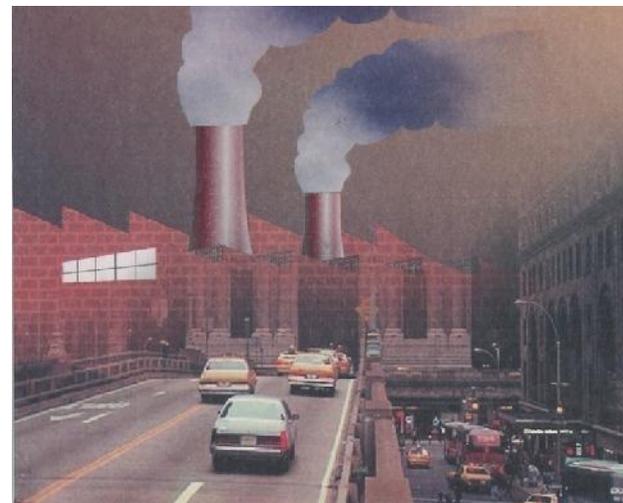


Fig. 3.5



ASCENT DESCENT ?





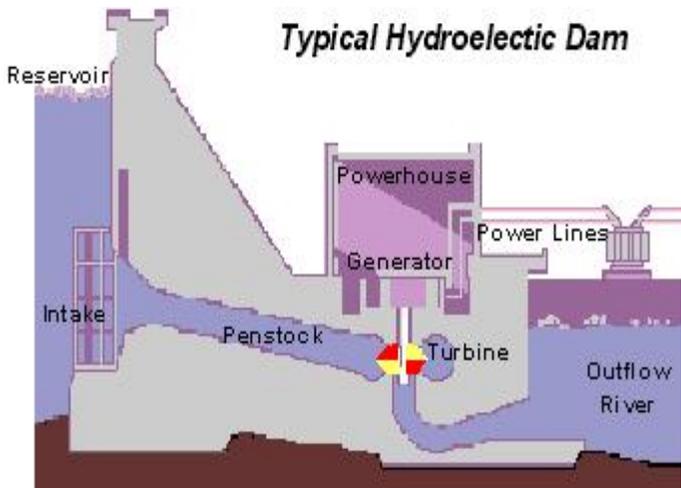
DOMESTIC



AGRICULTURE



PUBLIC WATER SUPPLY



THERMAL POWER PLANT



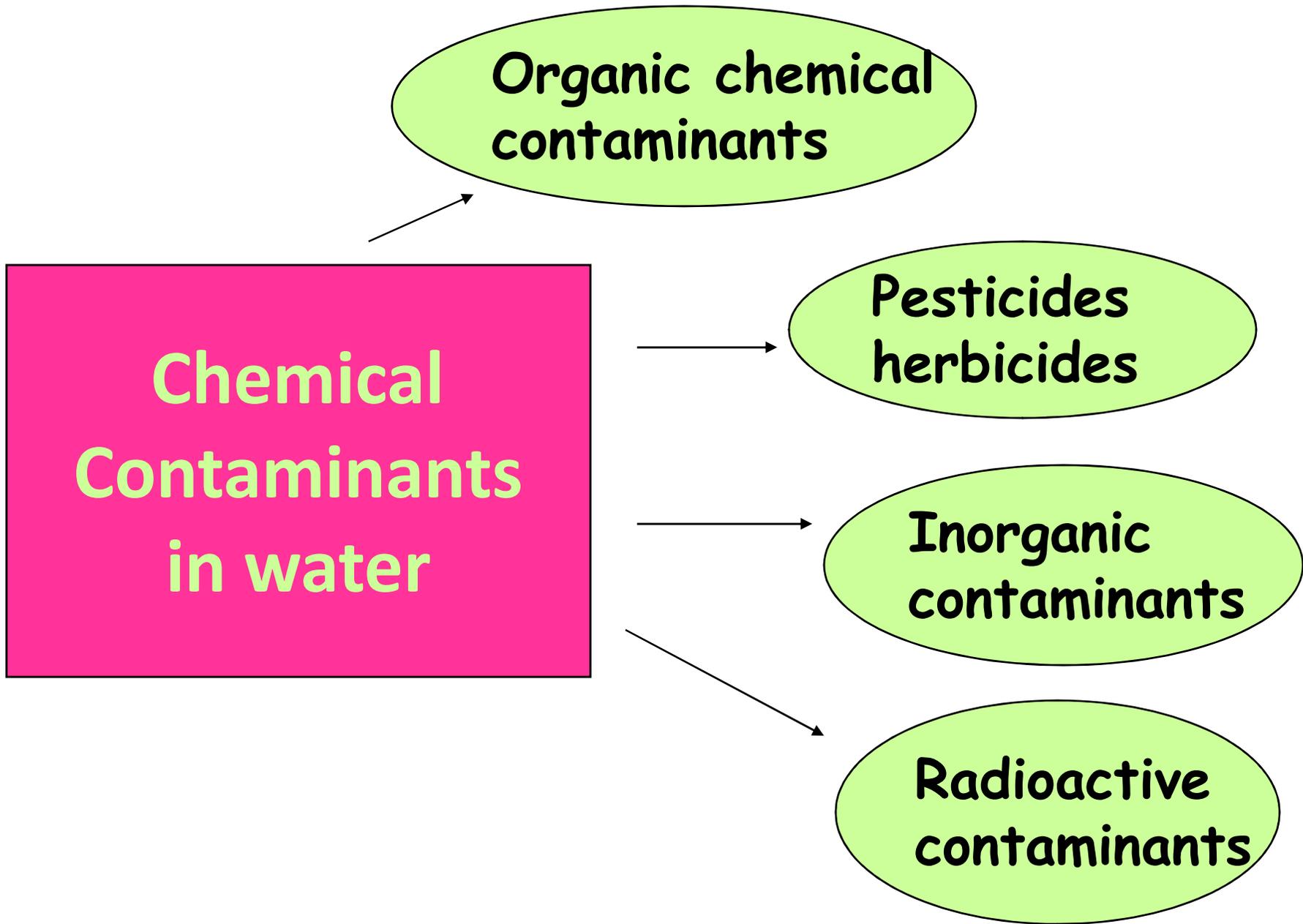
INDUSTRIES

Water Myths and Realities

Myth	Reality
We have less water today than we did 100 years ago	Same amount of water is present on Earth, as was three billion years ago

Water Myths and Realities

Myth	Reality
Water from River Ganga is pure	Untreated water has microbial contaminants. Not fit for drinking
Bottled water is safer than tap water	Unlike tap water, the quality of bottled water is not necessarily monitored by government agencies.



CHEMICAL CONTAMINANTS and DISEASES

MERCURY

NITRATE & NITRITE

LEAD

FLUORIDE

FLUORESIS

BRAIN DISEASE

KIDNEY & BRAIN
DISEASE

CYANOSIS,
BLUE BABY
SYNDROME

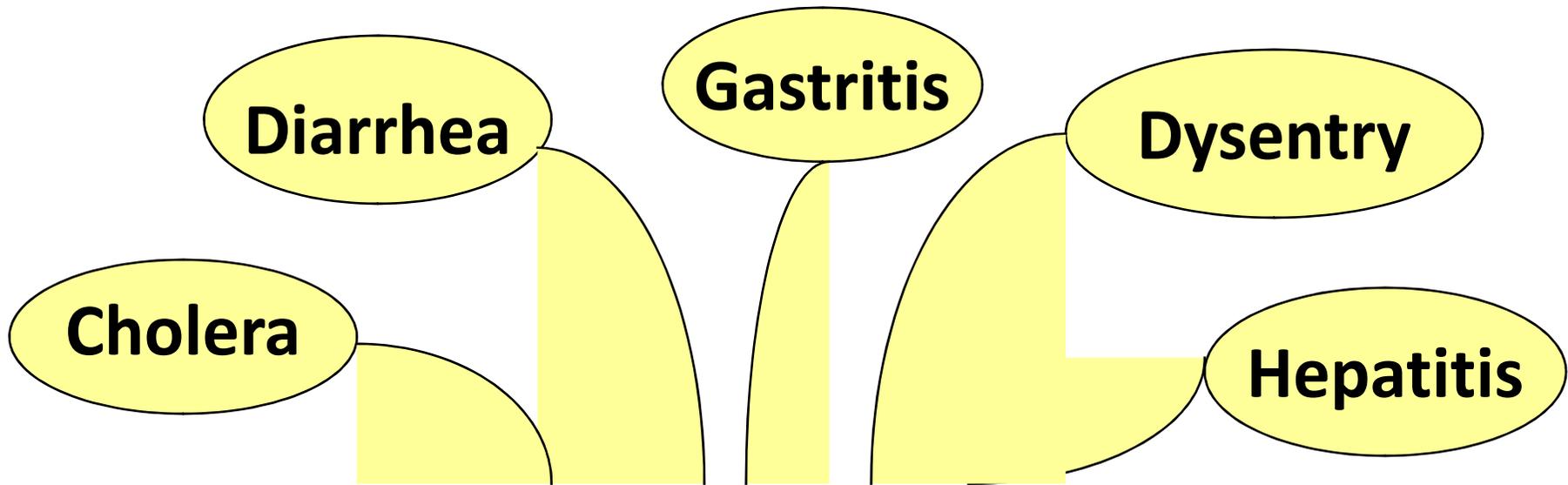
**Certain
Microbes
in
water**

```
graph LR; A["Certain Microbes in water"] --> B["Viruses- Enteric viruses"]; A --> C["Bacteria- Escherchia coli, Vibrio cholerae, Salmonella"]; A --> D["Protozoa- Entamoeba histolytica, Giardia lamblia"];
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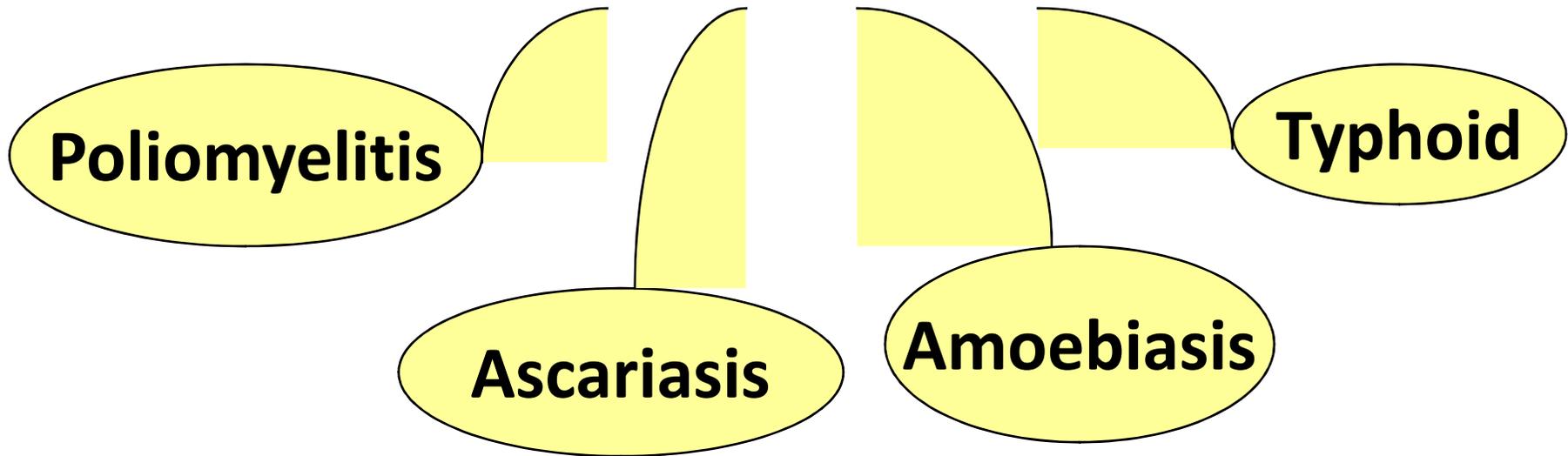
Viruses-
Enteric viruses

Bacteria-
Escherchia coli
Vibrio cholerae
Salmonella

Protozoa-
Entamoeba histolytica
Giardia lamblia



DISEASES caused by WATER BORNE PATHOGENS

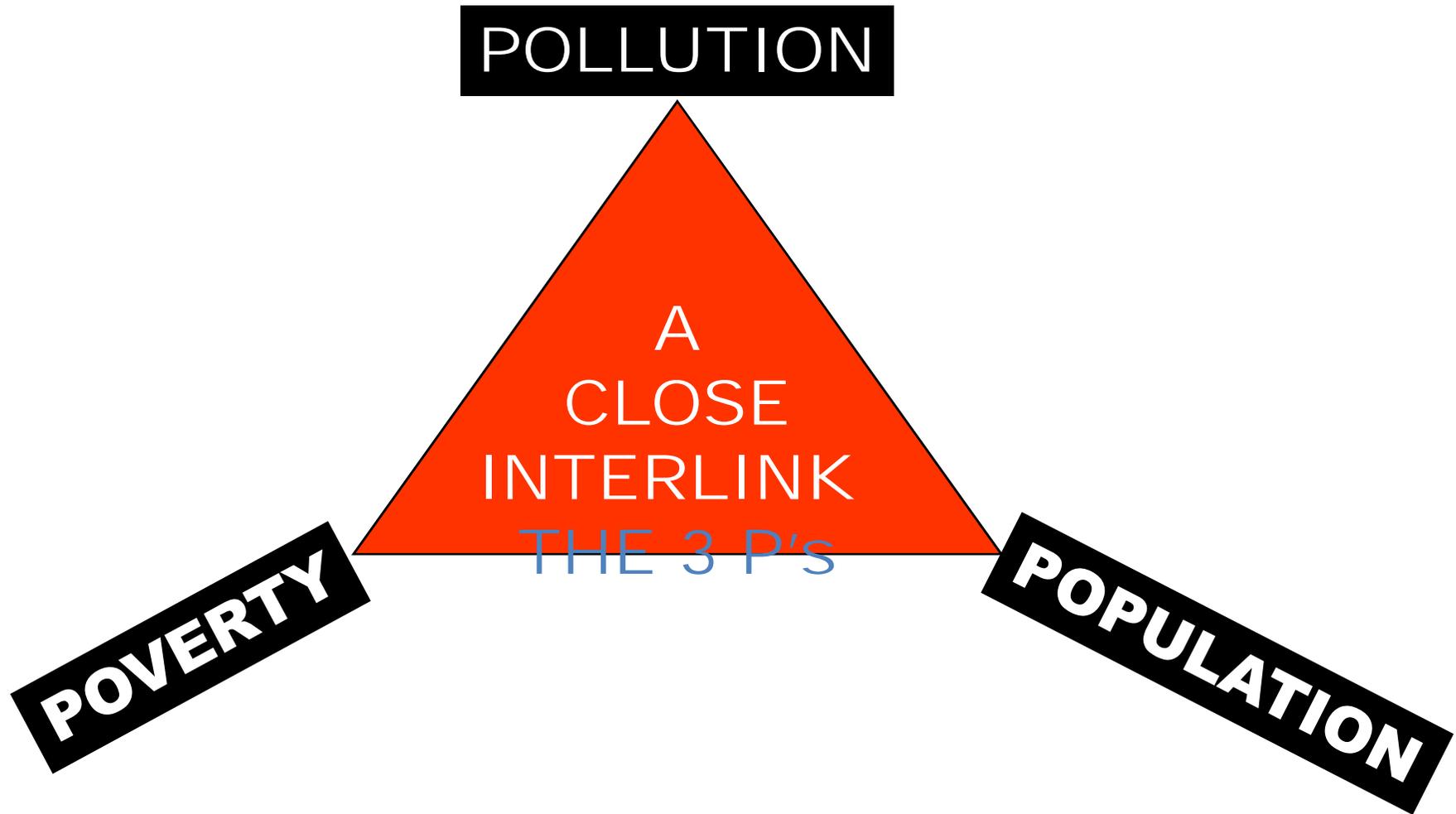


Water use in certain countries

Country	Annual water use per capita (Gallons)	Residential total water use (%)	Total water use in Industry/ Agriculture (%)
USA	525,000	10	90
India	132,000	3	97
China	122,000	6	94

Drinking Water Consumption

- **Adults** - Average 2 - 3 litres per day
- **Children (10 Kg body wt.)** - 1 litre
- **Infants (5 Kg)** - 0.75 litre



Coloured water: Mystery solved

Dirty tap water causes health scare in city

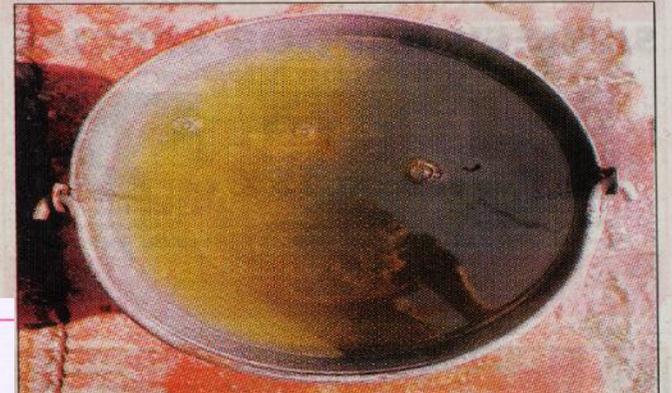
Experts to investigate fish disaster

Times News Network
Lucknow: An expert committee was formed on Monday by housing and urban development minister Lalji Tandon, to get to the bottom of the ecological disaster that occurred in the Gomati, bringing the Jal Sansthan and Uttar Pradesh Pollution Control Board (UPPCB) in direct confrontation.
 The five-member expert committee comprises one nominee from the district administration, two scientists from the Industrial Toxicology Research Centre (ITRC), one engineer from the Lucknow Municipal Corporation (LMC) and Rashid Khan, general manager of Jal Sansthan Rashid Khan. The committee is to submit its report within seven days.
 The committee will collect and



Water-hyacinth chokes the Gomati at Kudi

Times News Network
Lucknow: Guess what do the UP chief minister Mayawati and urban development minister Lalji Tandon share apart from warm personal and political vibes? Stinking and yellow coloured water, supplied by the Jal Sansthan. Provided they ever bother to check, or are informed about it.
 While the duo has not acknowledged the problem for obvious reasons, their



SC slams UP government over Gomati pollution

Times News Network
New Delhi: Perturbed over

Ganga pollution: SC notice to 8 states

New Delhi: Taking serious note of 15 municipalities. Panjwani had and as a matter of fact are totally continued pollution of Ganga river. contended that CPCB was filing the case to the responsibility for the



Saturday, May 18, 2002 5

Epidemic catches officials napping

Times News Network
Allahabad: Two gastro-enteritis deaths in the district and the rising incidence of diarrhoea have not yet woken up authorities

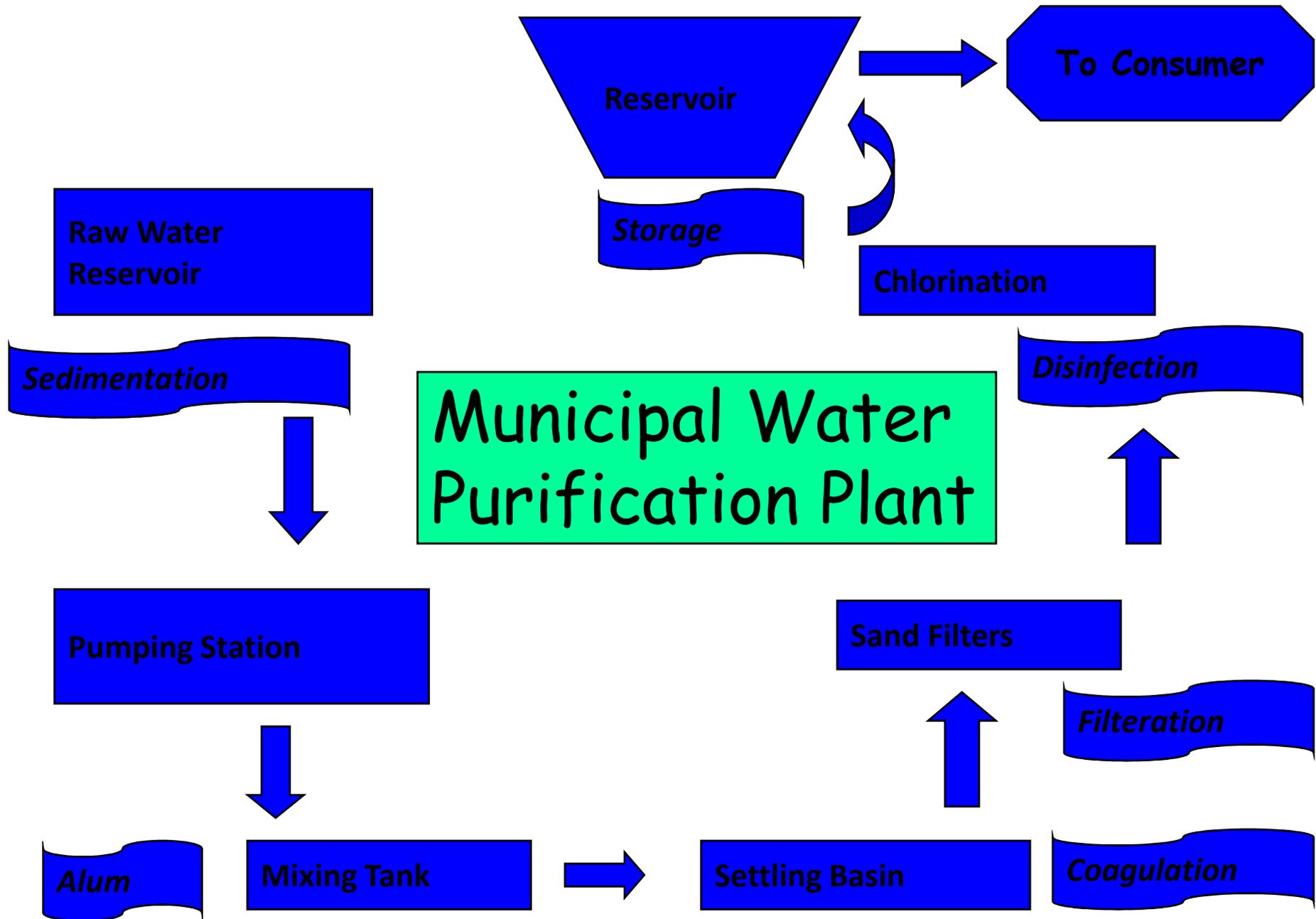
secretaries and director-generals of the department, decided to set up special teams to monitor the sale of open food. However, despite the passage of a week, the urban

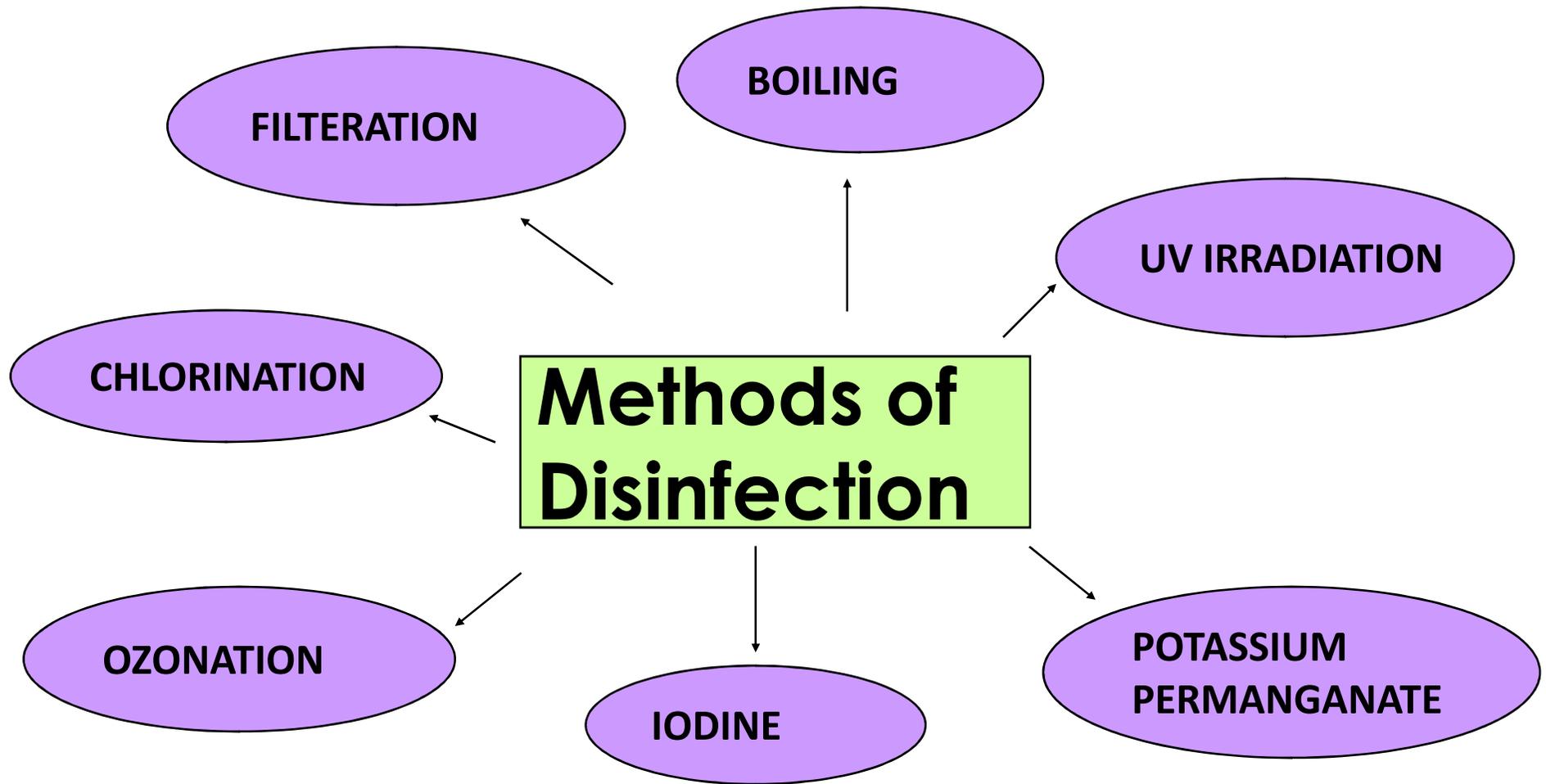
Polio epidemic is on the rise in state

Times News Network
New Delhi: The number of children crippled by the polio epidemic in Uttar Pradesh continues to increase steeply with the latest reports showing 347 cases of paralysis and a spillover into Delhi as well.
 Officials say the numbers are bound to increase as blood samples

- Alarm bells ringing**
- The number of children crippled by the polio epidemic in Uttar Pradesh continues to increase steeply.
 - A reports shows 347 cases of paralysis and a spillover into Delhi as well.

been covered," he says. In Azamgarh, officials say over 40 per cent houses with children were not included in immunisation drive at all.
 Union minister for health and family welfare Shatrughan Sinha met the chief minister in Lucknow on Monday to express concern over this rapid increase in polio in the state. Senior officials from the





WATER DISINFECTION TECHNIQUES

- CARBON BLACK
- HALOGENATION
- OZONIZATION
- UV LIGHT
- REVERSE OSMOSIS
- ELECTROCHEMICAL
- DESALINATION
- EXTREME BOILING

POPULAR DEVICES

CANDLE FILTER
USHA BRITA
AMRIT KUMBH
BACT-O-KILL
AQUA PURA
AQUA PEN
ZERO B
AGUA GUARD

SPECIFICATIONS/GUIDELINES RELATED TO WATER

1	IS 10500:2012	Drinking Water Specification
2	IS 13428: 1998	Packaged Natural Mineral Water – Specification
3	IS 14543:1998	Package Drinking Water (Other than packaged natural mineral water) Specification
4	IS 3025: 1964	Method of Sampling and Test for Water and Wastewater
5	IS 12252: 1987	Polyalkylene terephthalates (PET &PBT) for their safe use in contact with food stuffs, pharmaceuticals, and drinking water
6	IS 10151: 1982	Polyvinyl chloride (PVC) and its copolymers for its safe use in contact with foodstuffs, pharmaceuticals and drinking water.
7	IS 10146: 1982	Polyethylene for its safe use in contact with foodstuffs, pharmaceuticals and drinking water.
8	IS 10148: 1982	Positive list of constituents of polyvinyl chloride and its copolymers in contact with foodstuffs, pharmaceuticals and drinking water.

The next

WORLD WAR

will be over

WATER

? Why...

[Situation]

Oceans 97%

ALL WATER

Freshwater 3%

Ice caps &
Glaciers 79%

FRESHWATER

Accessible surface
Freshwater 1%

Groundwater
20%

ACCESSIBLE SURFACE
FRESHWATER

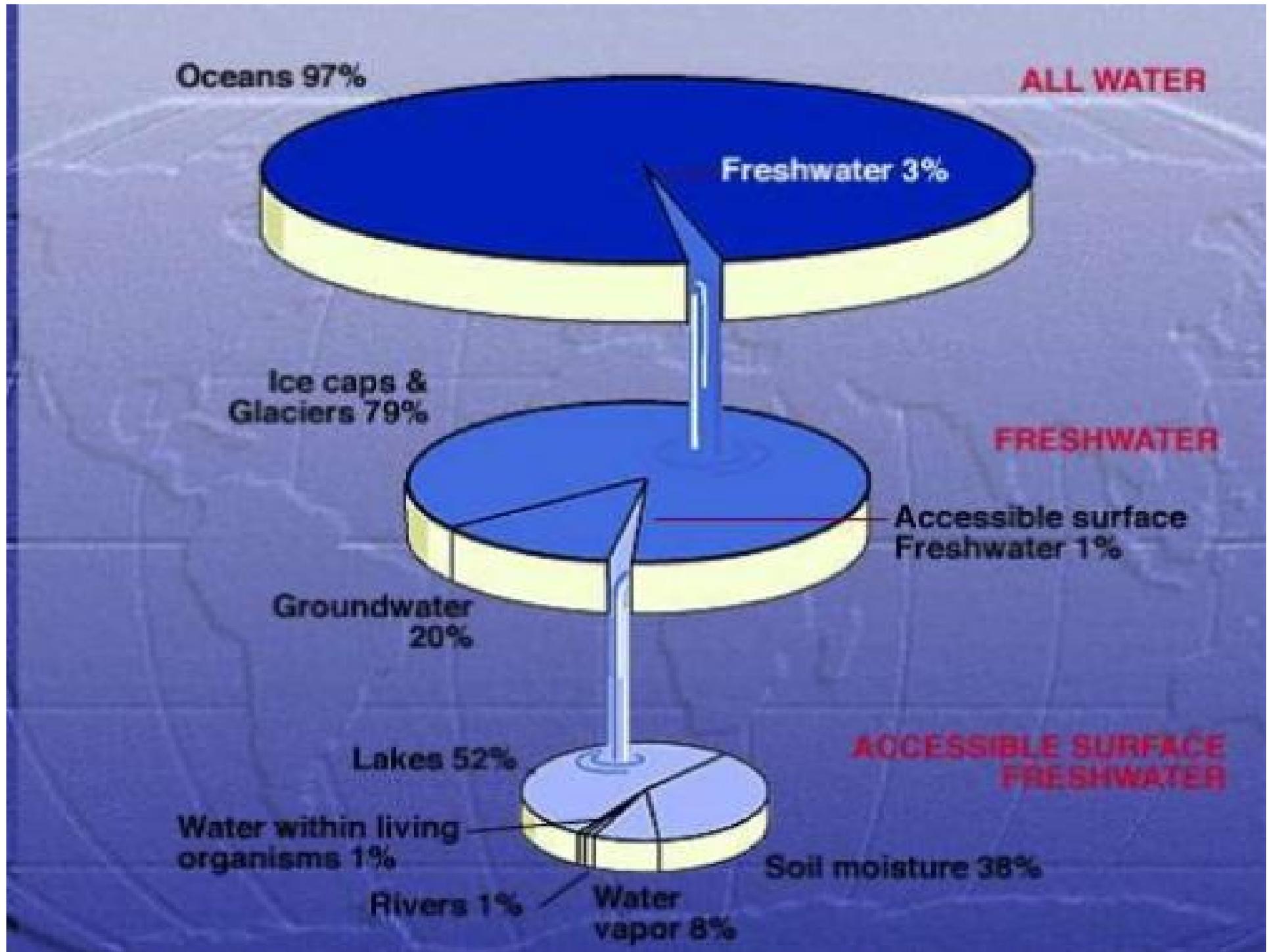
Lakes 52%

Water within living
organisms 1%

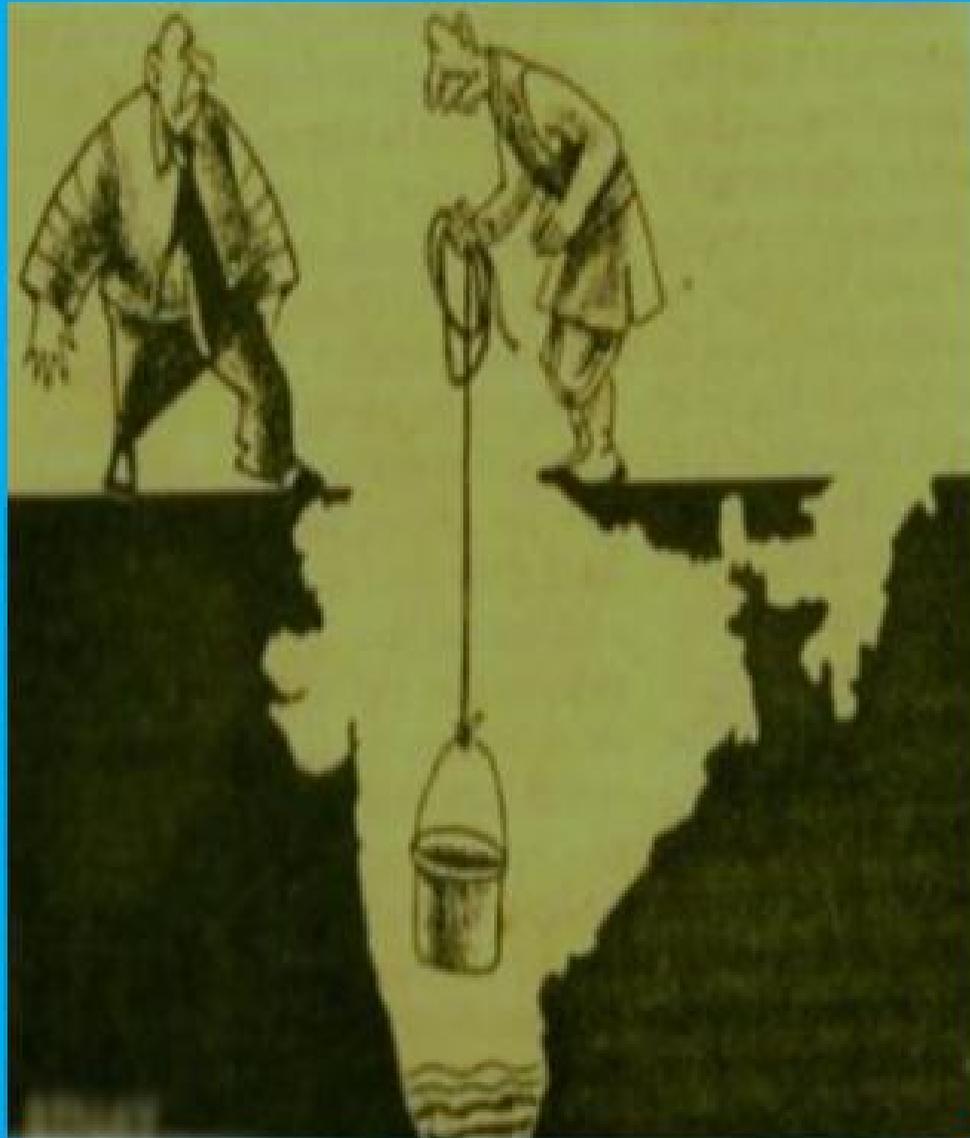
Soil moisture 38%

Rivers 1%

Water
vapor 8%



The **Indian** situation



- Groundwater is the major source of water in our country with 85% of the population dependent.
- Groundwater water table decline - 33 centimeters per year.

17 Countries including India face extremely high water stress

BASELINE WATER STRESS



Extremely high (>80%)

High (40-80%)

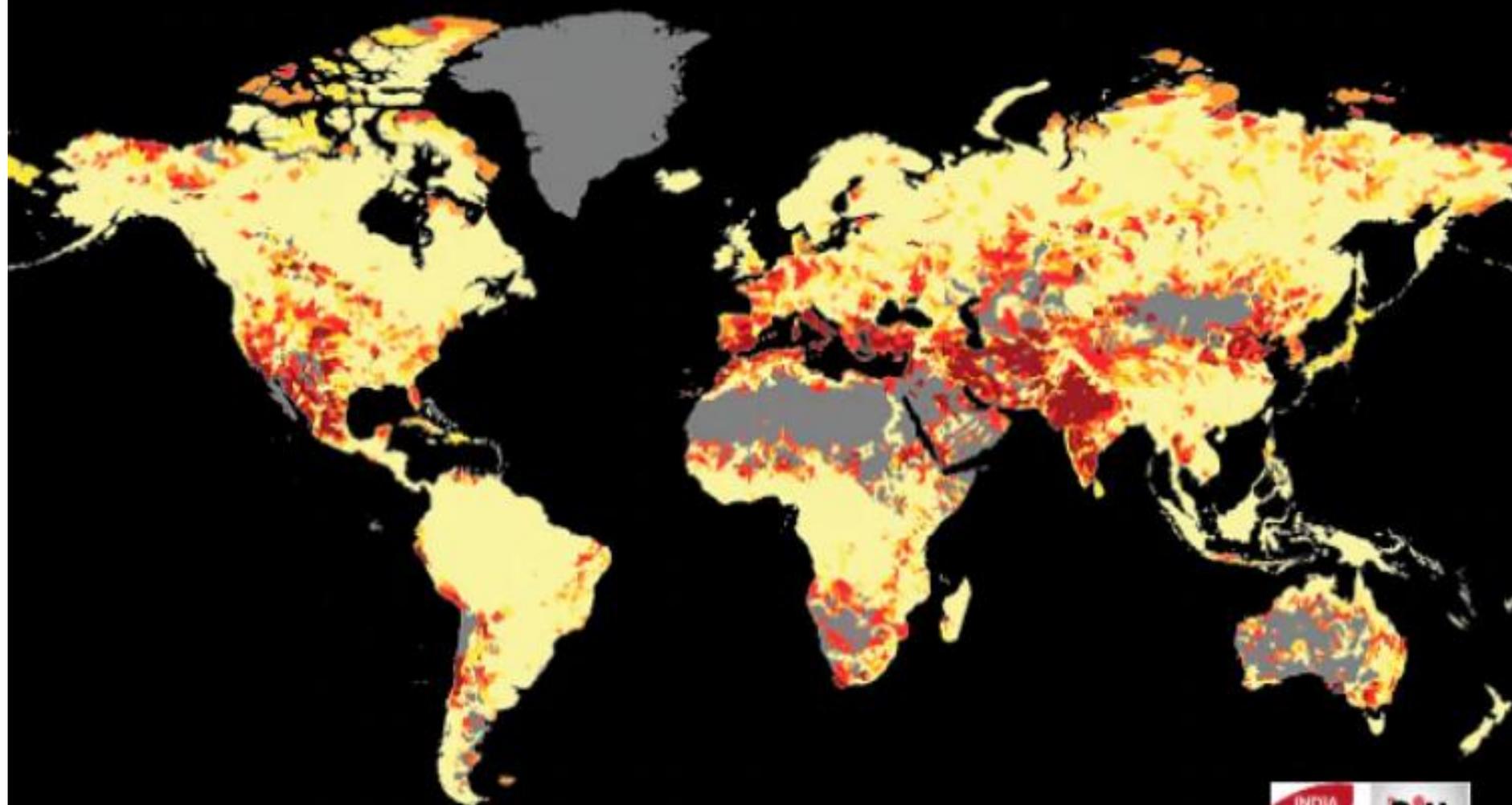
Medium-high (20-40%)

Low-medium (10-20%)

Low (<10%)

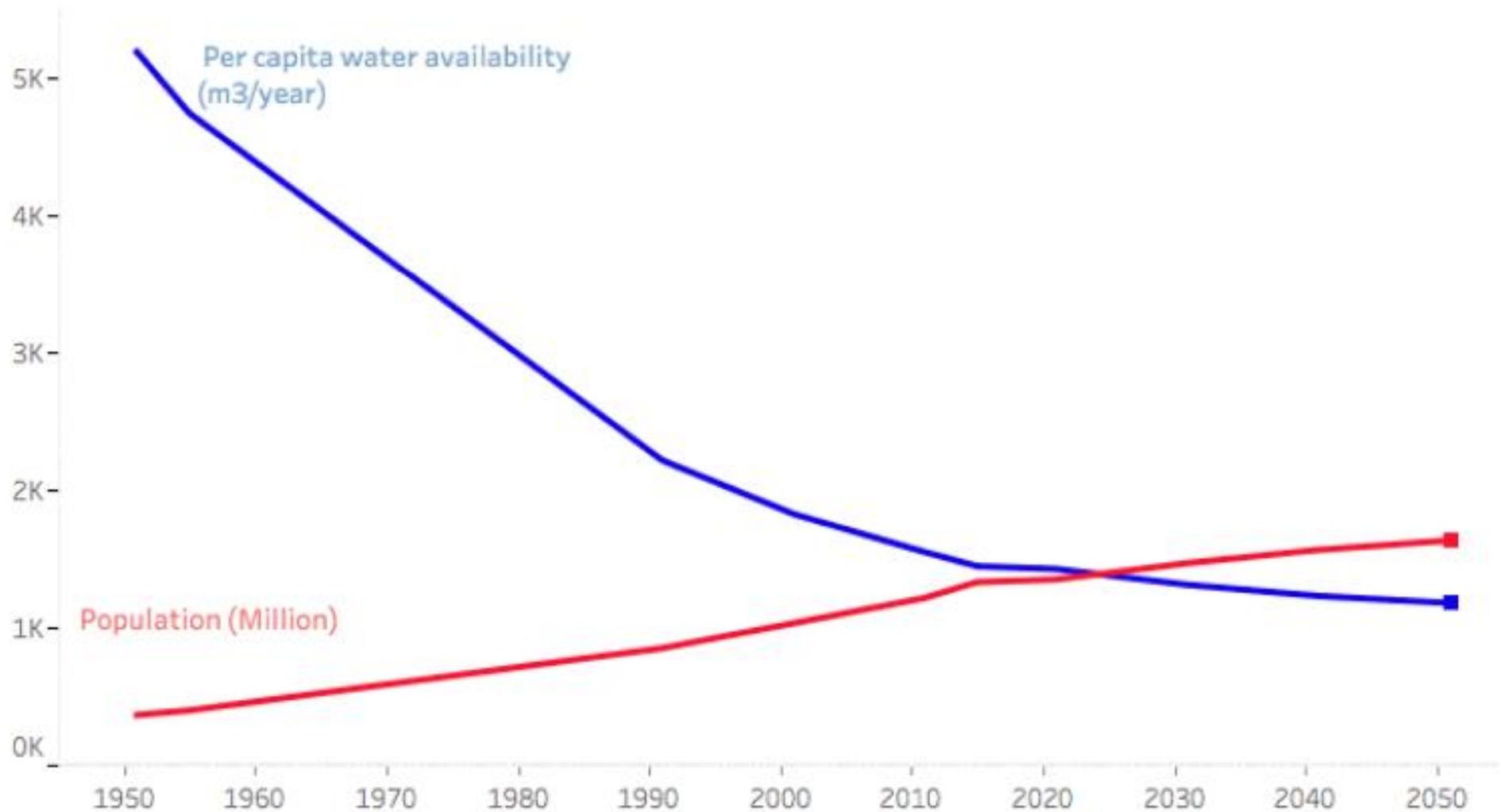
Arid and low water use

No data



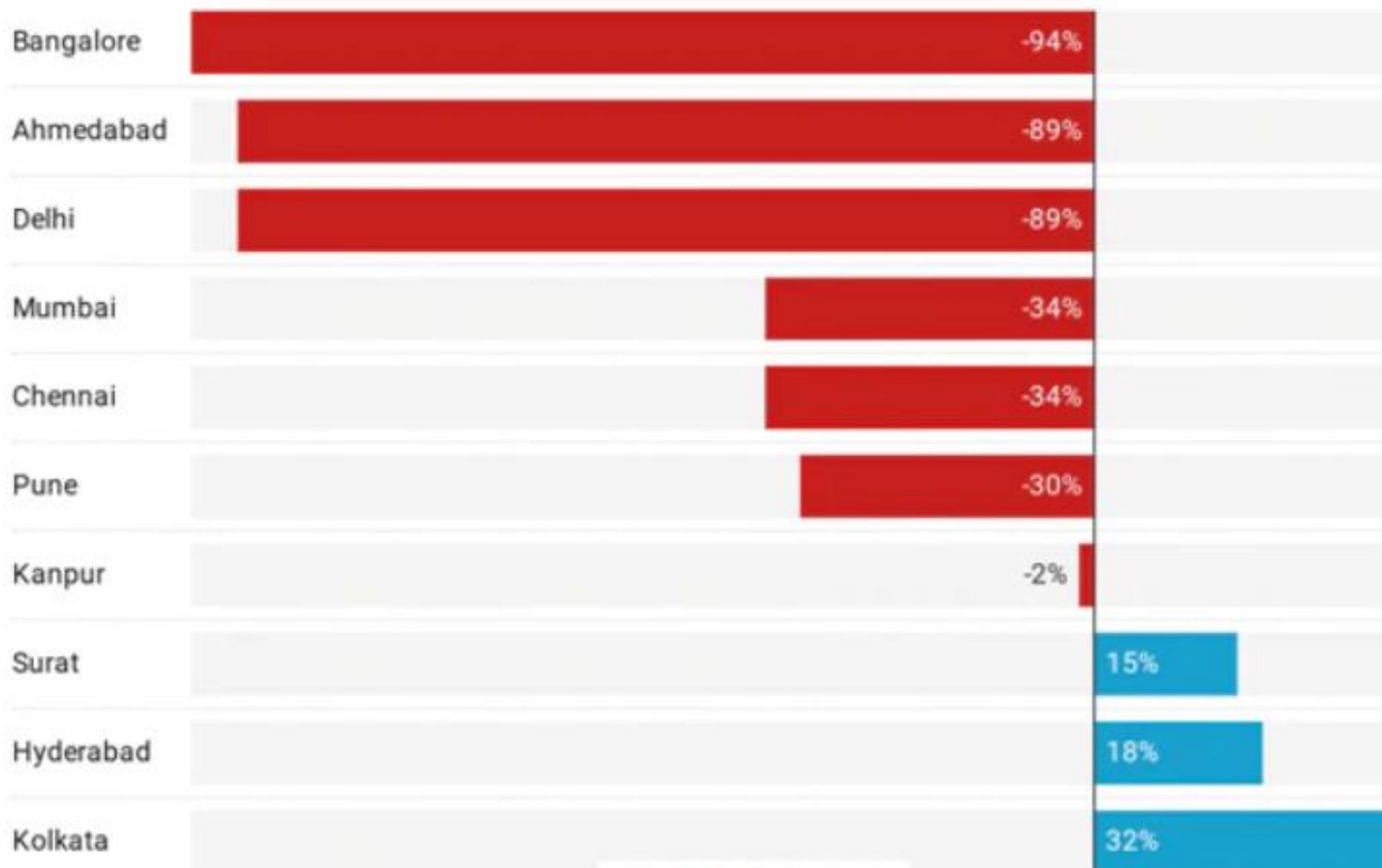
Per Capita Water Availability in India

2051



Cities' ground water levels have fallen

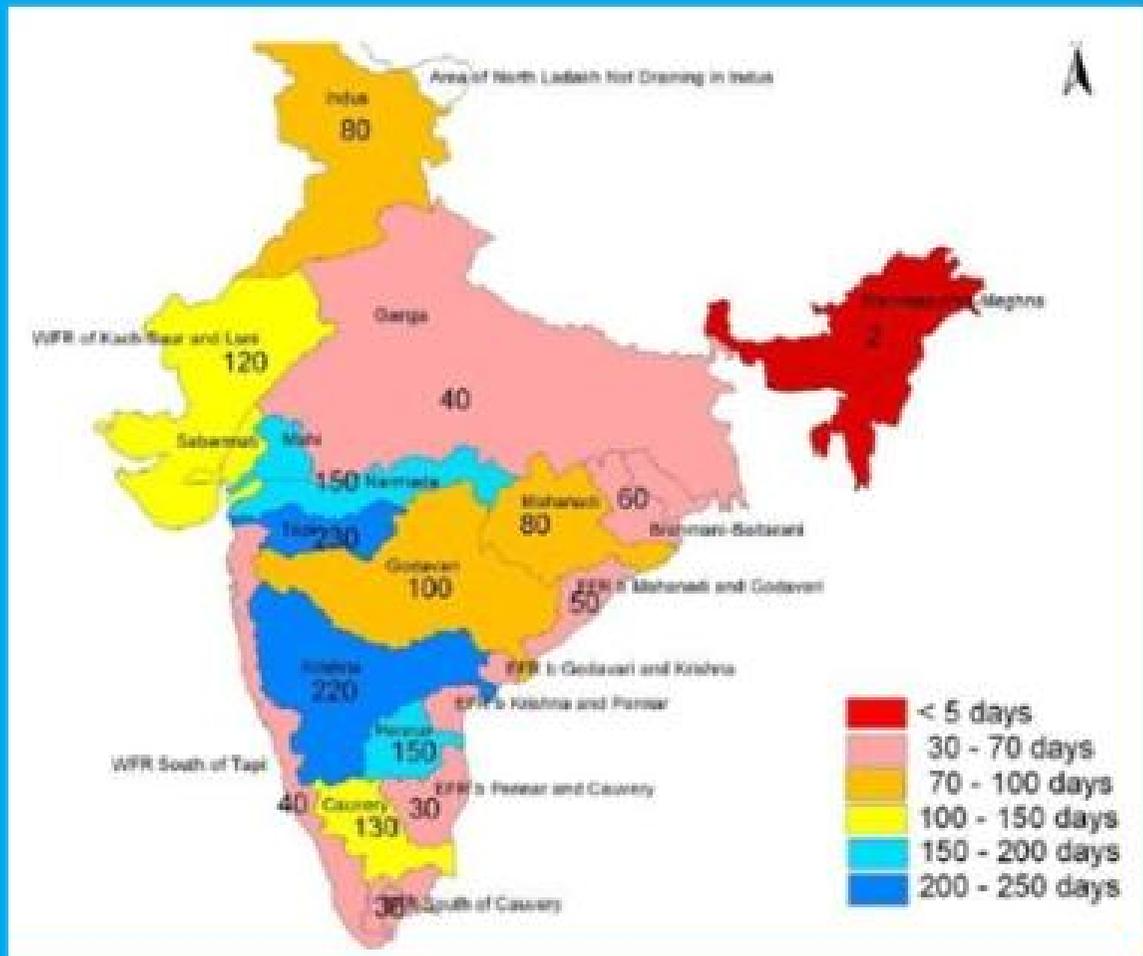
% change in groundwater from 1998 to 2018.



The **Indian** situation

India receives abundant rains compared to other water scared countries but...

This map shows how much water the Indian infrastructure fails to store!



The prime minister said on 15th, August, 2020 many states are reeling under the problem of acute water scarcity. PM Modi said his government is committed to provide drinking water to every household. This will be done under the Jal Jeevan Mission.



Challenges



Challenges

Industries



Solution

Solutions to water problems require the consideration of cultural, educational, communication and scientific aspects.

Plant Trees

Avoid Pollution

Conserve water

Technologies and Innovations

Water Purification Systems

Seawater desalination

Water Footprint

Solution

- Rain Water Harvesting
- Irrigation Water Management
- Hydrological projects - Construction of Dams
- Artificial Recharge to Ground Water through Dug well

Solution

sAvE wAtEr
SaVe WoRlD



The concept is simple

- ◆ Collect
- ◆ Store and use
- ◆ Recharge





Tap water



Bottled
water



Distilled
water



Carbonat...
water



**Sparkling
water**

- Tap **water**.
- Mineral **Water**.
- Spring **water**.
- Well **water**. ...
- Purified **water**.
- Distilled **water**: Distilled **water** or demineralised **water** is one where the **water** has been subjected to a treatment that removes all its minerals and salt by the process of reverse osmosis and distillation. ...
- Sparkling **Water**.

Characteristics of Water:

- **Water is a good solvent**
- **Water never occurs in its pure form**
- **All waters contain some dissolved substances**
- **The quality of water is determined by these substances.**
- **It has the ability to dissolve many inorganic and organic substances.**

Water Quality Parameters:

Water has its own

- **Physical properties**
- **Chemical composition and**
- **Biological Properties**

Physical Properties:

- **Temperature**
- **Colour**
- **Odor**
- **Turbidity**
- **Electrical Conductivity**

Chemical properties:

- pH
- **Total Dissolved Solids(TDS)**
- **Major ions**
- **Minor or trace elements**
- **Hardness**
- **Salinity**
- **Alkalinity**

Harmful Chemicals

- Chlorides
- Sulphates
- Iron
- Nitrates
- Heavy Metals
- Pesticides
- Poly Chlorinated Biphenyl (PCBs)
- Polycyclic Aromatic Hydrocarbon (PAHs)
- Tri Halo Methane
- Radioactive materials

Biological Properties:

- **Dissolved Oxygen (DO)**
- **Biochemical Oxygen Demand (BOD)**
- **Chemical oxygen Demand (COD)**
- **Microorganisms-Bacterial counts**

Water Quality Parameters:

- **Limits the suitability of water for different purposes**
- **Drinking**
- **Domestic consumption**
- **Agriculture**
- **Industrial Processes**
- **Cleaning and Recreation.**

Water Quality

Standards

- Permissible limits
- United States Public Health Drinking Water Standards (USPH)
- Indian Standards Institution (ISI)
- World Health Organization (WHO)

WATER AND HEALTH



▶ Quality



▶ Quantity

IS: 10500 - Indian Standard for Drinking Water

S.No.	Parameter	Requirement	Desirable Limit
1.	pH		6.5-8.5
2.	Total Dissolved Solids (TDS) in mg/l		500
3.	Total Hardness (mg/l)		300
4.	Chloride (mg/l)		250
5.	Fluoride (mg/l)		1.0
6.	Nitrate (mg/l)		450
7.	Sulphate (mg/l)		200
8.	Cyanide (mg/l)		0.05
9.	Total Alkalinity (mg/l)		200
10.	Arsenic (mg/l)		0.01
11.	Mercury (mg/l)		0.001
12.	Cadmium (mg/l)		0.01
13.	Lead (mg/l)		0.05
14.	Iron (mg/l)		0.3
15.	Manganese (mg/l)		0.1
16.	Chromium as Cr ⁶⁺ (mg/l)		0.05
17.	Copper (mg/l)		0.05
18.	Zinc (mg/l)		5.0
19.	Pesticide		absent
20.	Total Coliform Bacteria	95% of samples should not contain coliform in 100 ml	10 coliform / 100ml
21.	E. coliform Bacteria		Nil / 100ml

(ix)	Nickel(as Ni)mg/l,Max	0.02	Beyond this,it may cause allergic reaction	No relaxation	3025 (part54)	-
(x)	Poly chlorinated biphenyls mg/l, Max	0.0005	May be carcinogenic	No relaxation	ASTM 5175/APHA 6630	
(xi)	Trihalomethanes					-
(a)	Bromoform mg/l Max	0.1	May be carcinogenic above this limit	No relaxation	ASTM D-3973-85/APHA	
(b)	Dibromochloro methane mg/l, Max	0.1	-do-	-do-	-do-	
(c)	Bromodichloro methane mg/l, Max	0.06	- do-	-do-	-do-	-

Table. 5 Pesticides Residues limits and Test method

SI.No	Pesticide	Limit ~g/l	Test method USEPA AOAC/ISO
(i)	DDT(o,p and p,p-Isomers of DDT,DDE and DDD	1	508 AOAC 990.06
(ii)	Gamma-HCH (Lindane)	2	508 AOAC 990.06
(iii)	2,4D	3	515.1
(iv)	Isoproturon	9	532
(v)	Alachor	20	525.2,507
(vi)	Atrazine	2	525.2,8141A
(vii)	Aldrin/Dieldrin	0.03	508

Table.4 Parameters concerning radioactive substances (Clause 4)

Sl. No	Substance or Characterstic	Requirement (Desirable limit)	Undesirable Effect outside the desirable	Permissible limit in the absence of alternate source	Methods of Test (Ref to IS)	Remarks
(i)	Radioactive Materials:					-
	(a) Alpha emitters Bq/l,Max	0.1	May be carcinogenic above this limit	0.1	IS 14194 (Pt 2)	-
	(b) Beta emitters Bq/l Max	1.0	-	1	IS 14194 (Pt 1)	-

Total Daily Intake (TDI)

Human Health Risk = -----

Acceptable Daily Intake (ADI)

If value is > 1 than health risk

Address uncertainty

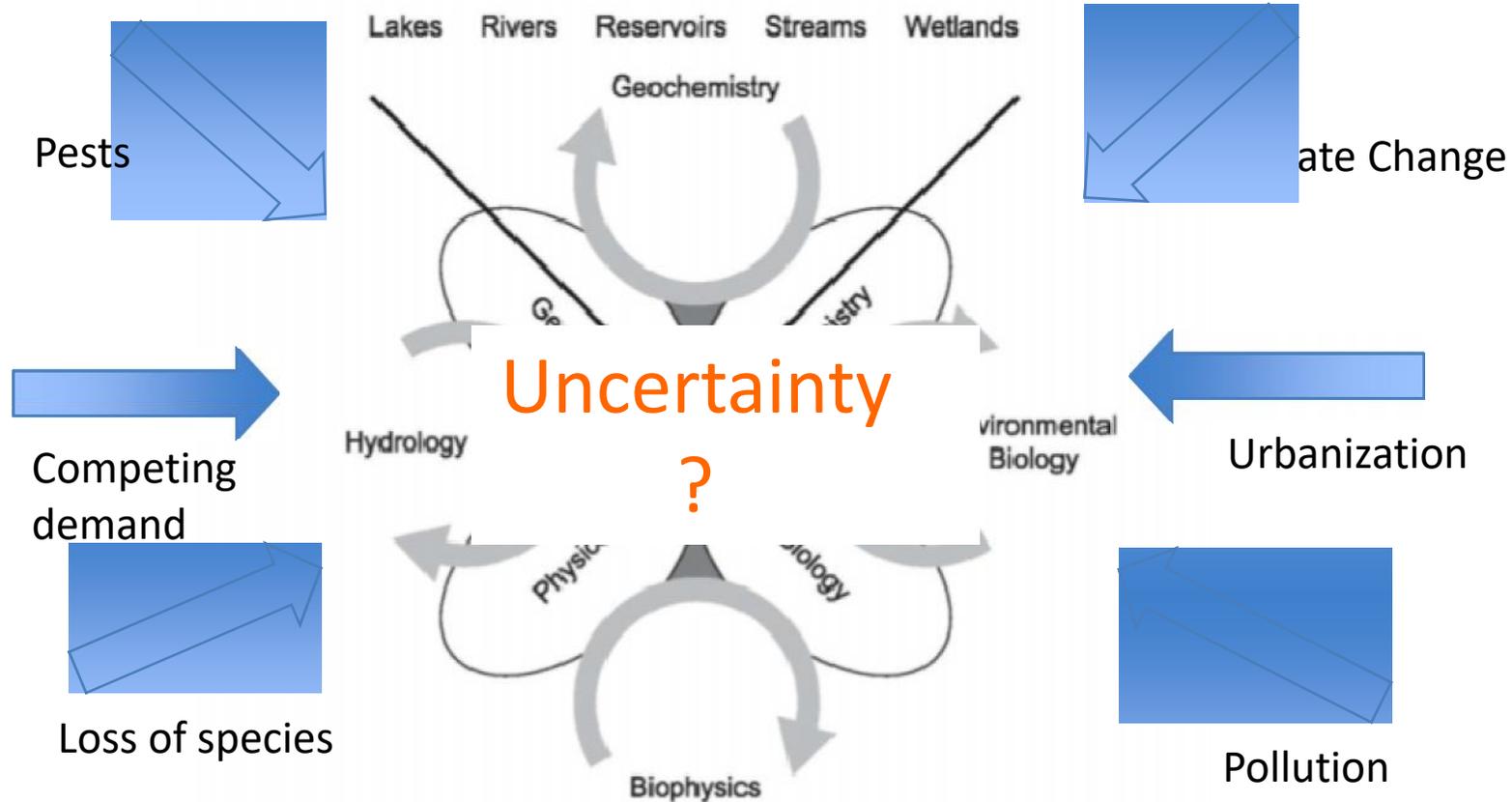


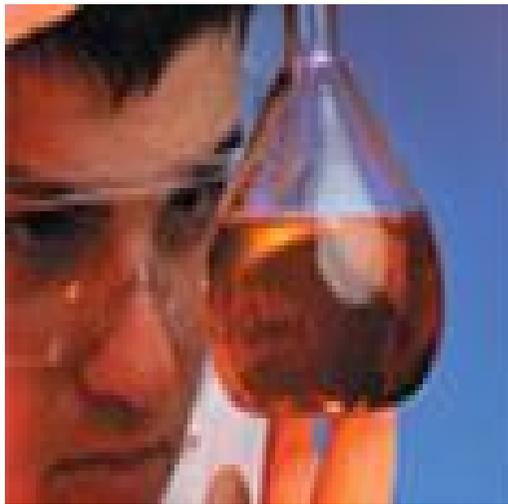
Figure 1. Limnology is the study of inland waters. Like oceanography, it is an integrative science that draws from many disciplines.



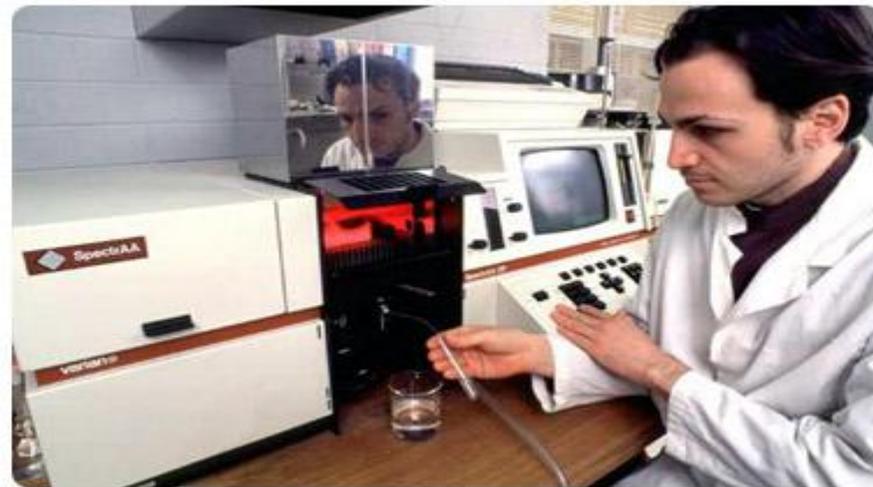
Sampling



Preservation



Processing



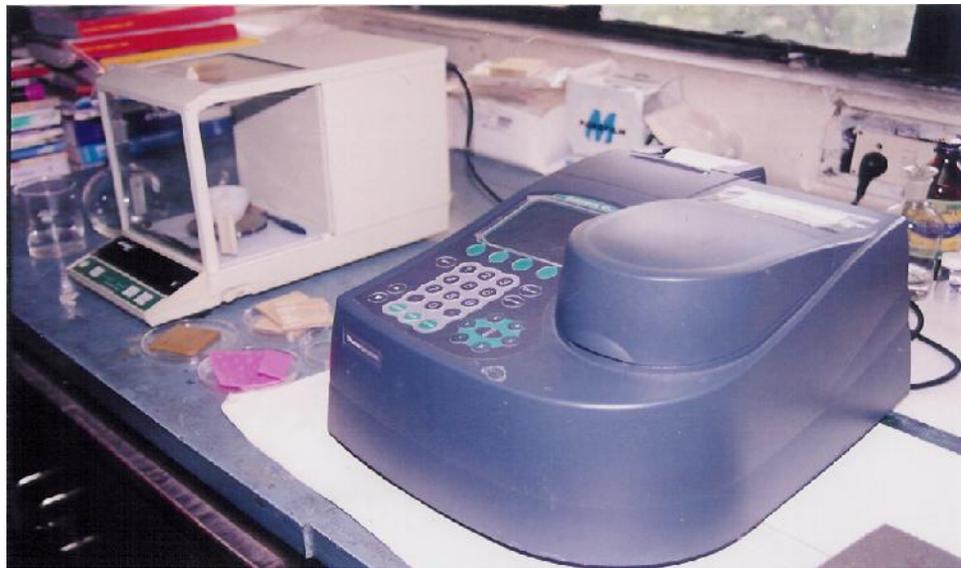
Analysis



Collection of water sample



Analysis



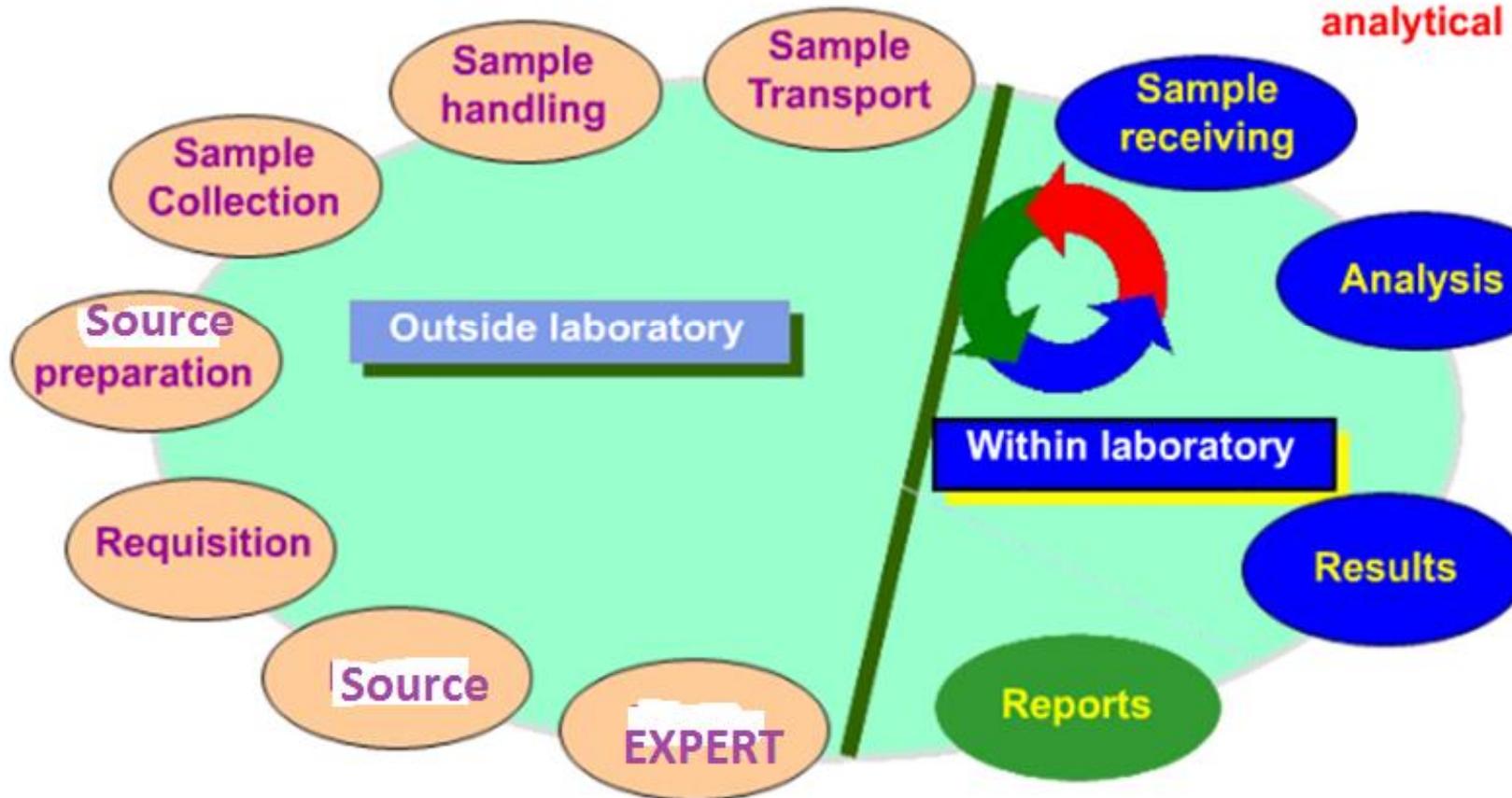
Instrument Application



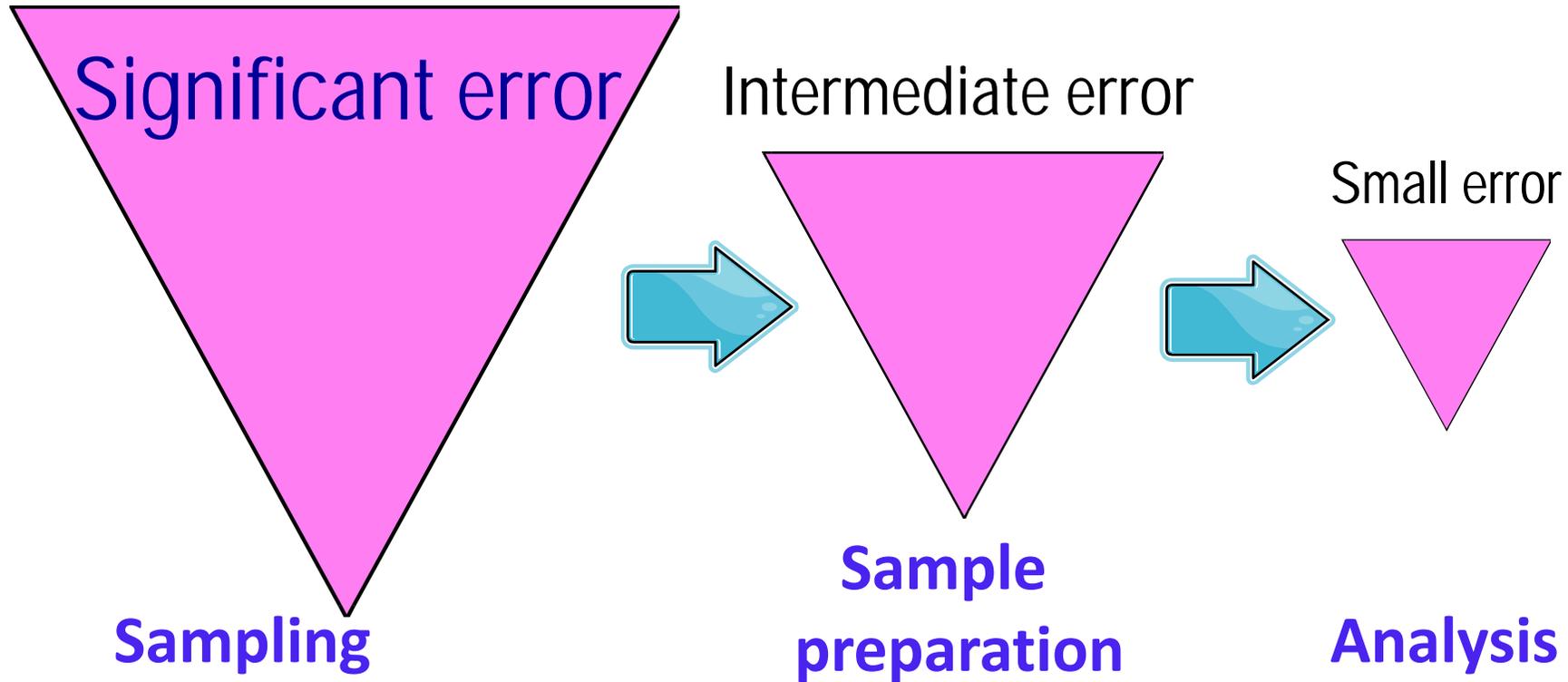
Documentation

Factors influencing quality

Pre-analytical
Analytical
Post-analytical



Relative Contributions to Analytical Error

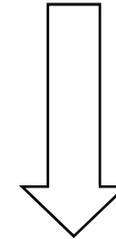


Overall (expanded) error = (individual errors)

Sampling is Important!

- ✓ Clear definition of sampling objectives
- ✓ Sample quality
- ✓ Sample integrity
- ✓ Sample representativeness

Sampling



Chemical Analysis

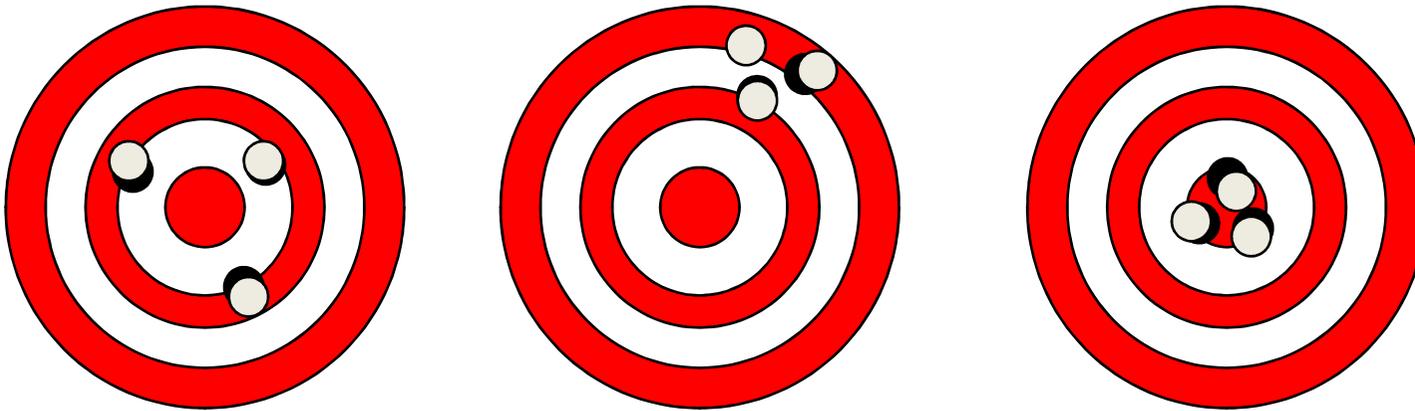


Data Interpretation

Sampling Personnel works under supervision of Qualified and trained personnel.



Accuracy versus Precision



Precision & Accuracy

- Is measured as “repeatability “ or reproducibility

Repeatability

Measurement is repeated with a minimum of variations (same person, same laboratory, same equipment, over a short period of time).

Reproducibility

Measurement is repeated with as much variation as possible. (Different analyst, different laboratory, different equipment, over a long period of time).

Limit of Detection (LOD)

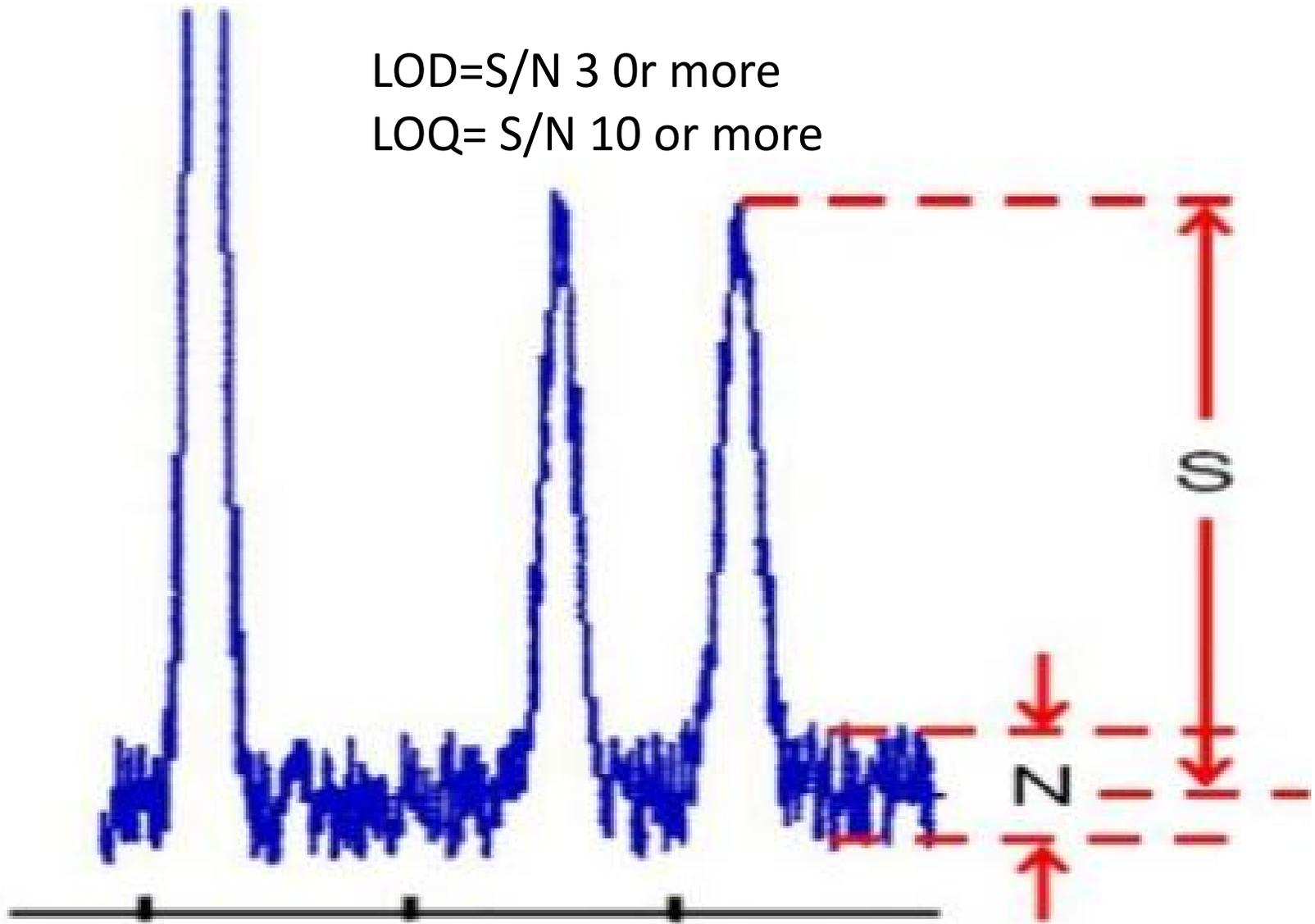
- Point where the variability of the measurement is such that it is impossible to distinguish between random fluctuation in the value measured and actual signal.

$$- \text{LOD} = Y_B + 3XS_B$$

- Y_B = Mean value measured from the blank
- S_B = Standard Deviation of the value measured from the blank

LOD=S/N 3 Or more

LOQ= S/N 10 or more



Where S=Height of Signal
N=Height of Noise

Limit of Detection

TBT in Biota

Replicate (n)	Instrument response	Calculated Concentration (ppb)
1	29550	0.100
2	28653	0.097
3	29401	0.099
4	28532	0.097
5	29712	0.101
6	29983	0.101
7	28167	0.095
Mean		0.099
StdDev		0.002
3 Times StdDev		0.007
Detection Limit		0.106

Limit of Quantification (LOQ)

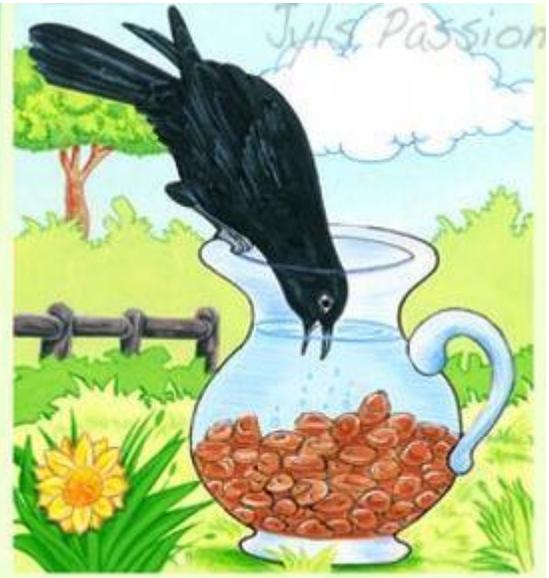
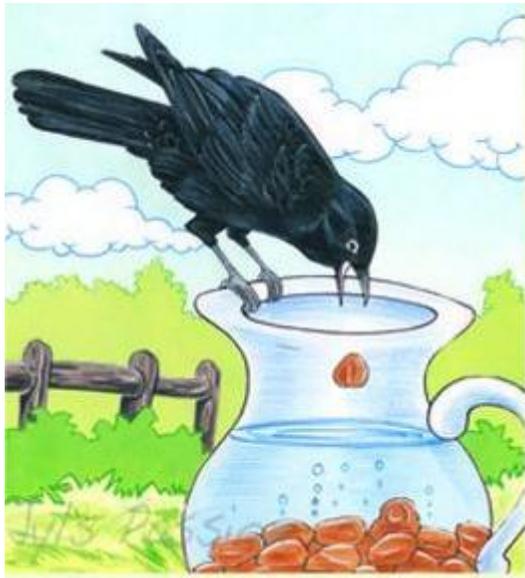
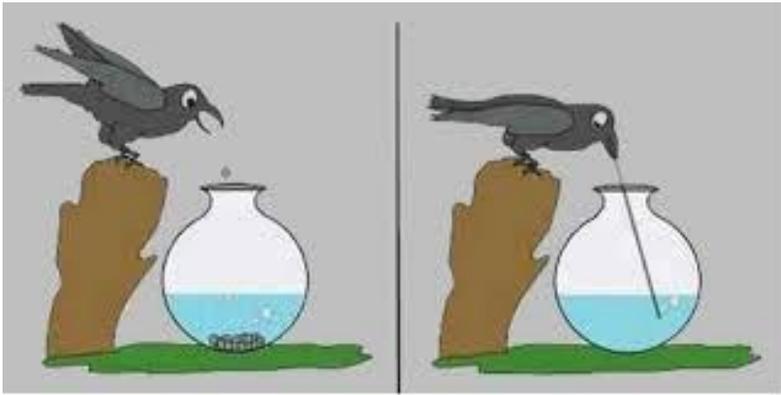
- Point where the measured value could be quantified reliably

$$\text{LOQ} = Y_B + 10XS_B$$

- Y_B = Mean value measured from the blank
- S_B = Standard Deviation of the value measured from the blank

Limit of Quantification (LOQ)

Replicate (n)	Instrument response	Calculated Concentration (ppb)
1	29550	0.100
2	28653	0.097
3	29401	0.099
4	28532	0.097
5	29712	0.101
6	29983	0.101
7	28167	0.095
Mean		0.099
StdDev		0.002
10 Times StdDev		0.020
LOQ		0.119



Requirements

-  **Calibrated instruments**
-  **Certified glasswares**
-  **Analytical grade reagents**
-  **Skilled manpower**



Instrumentation

■ Spectrophotometer/Colorimeter

■ Chromatographs (Gas/Liquid)

■ Analytical Balance

■ Hot Air Oven

■ Refrigerator



Spectrophotometer/Colorimeter

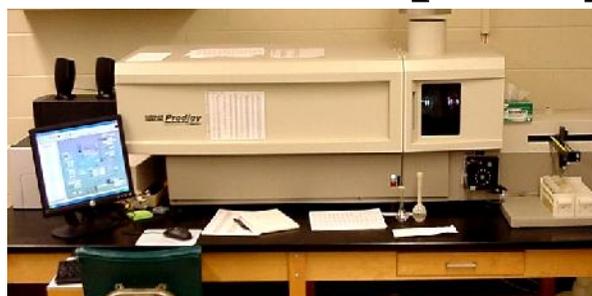
UV Visible Spectrophotometer



Atomic Absorption Spectrophotometer (AAS)



Inductively Coupled Plasma (ICP) Spectrophotometer



Physico-chemical Parameters of Drinking Water

Physico-chemical Tests

- pH
- Temperature
- Conductivity
- **Dissolved Oxygen**
- Turbidity
- Colour
- Odour
- Hardness
- Dissolved Solids
- Nitrate
- **Fluoride**
- **Chloride**
- **Bromide**
- **Iodide**
- Sulphate

Metals

- **As**, Cd, Co, Cr, Cu, Fe, **Hg**, Mg, Mn, Ni, Pb, Zn, **Se**

Organic Contaminants

- Chlorinated Compounds
- Trihalomethanes
- **Pesticides**
- Polycyclic Aromatic Hydrocarbons
- Benzene and lower alkyl benzene
- PCBs
- Phthalates
- **BOD**
- **COD**
- **TOC**



Parameters and Test Methods

No	Parameter	Unit	Methods
1	pH	-	Electrometric method
2	Turbidity	NTU	Turbidity Meter
3	True Color	Pt-Co Unit	Visual Comparison method
4	Electrical Conductivity	$\mu\text{S/cm}$	Electrical Conductivity method
5	Iron	mg / L	FerroVer method
6	Manganese	mg / L	PAN method
7	Sulfate	mg / L	SufalVar Turbidimetric method
8	Fluoride	mg / L	SPADNS method
9	Alkalinity	mg / L	Titration method
10	Total Hardness	mg / L	EDTA Titrimetric method
11	Chloride	mg / L	Argentometric method

Techniques used in water Quality Checks in Chemical

- Direct Instrument use for Physical parameters: pH, Conductivity, temperature, turbidity
- By use of specific equipment after processing : heavy metals, pesticide, PAHs, PCBs, THM
- By Conventional method Like Gravimetric and volumetric analysis.

-

Gravimetric Analysis

TDS
Sulphate

Volumetric Analysis

TH, Ca, Mg
Chloride
Alkalinity

How to Perform a Successful Gravimetric Analysis

- **What steps are needed?**
 1. Sampled dried, triplicate portions weighed
 2. Preparation of the solution
 3. Precipitation
 4. Digestion
 5. Filtration
 6. Washing
 7. Drying or igniting
 8. Weighing
 9. Calculation

Gravimetric Analysis

- Gravimetric Analysis – one of the most accurate and precise methods of macro-quantitative analysis.
- Analyte selectively converted to an insoluble form.
- Measurement of mass of material
- Correlate with chemical composition
- Why?
- Simple
- Often required for high precision

Gravimetric Analysis

- How?
- Quantitative collection of material of known composition
 - Precipitation of analyte with selective agent
 - Volitization and collection of analyte
 - w/o loss of material in handling/processing
 - Free from solvent, impurities
- Determination of mass
 - Direct or
 - By difference

Use a desiccator to cool a dried or ignited sample.

Cool a red hot vessel before placing in the desiccator.

Do not stopper a hot weighing bottle (creates a partial vacuum on cooling).

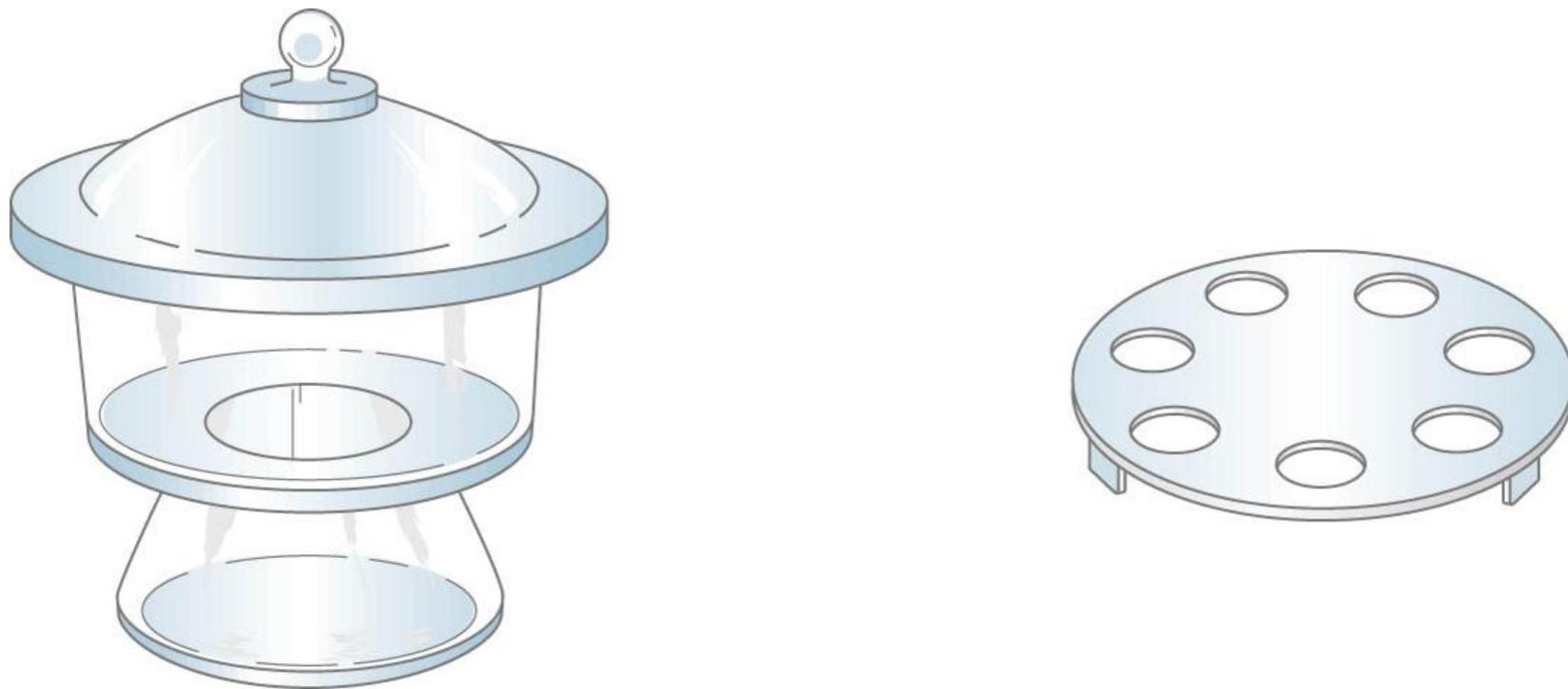


Fig. 2.16. Desiccator and desiccator plate.

CaCl₂ is commonly used.

It needs periodic replacement when wet or caked.

Table 2.5

Some Common Drying Agents

Agent	Capacity	Deliquescent ^a	Trade Name
CaCl ₂ (anhydrous)	High	Yes	
CaSO ₄	Moderate	No	Drierite (W. A. Hammond Drierite Co.)
CaO	Moderate	No	
MgClO ₄ (anhydrous)	High	Yes	Anhydrone (J. T. Baker Chemical Co.); Dehydrite (Arthur H. Thomas Co.)
Silica gel	Low	No	
Al ₂ O ₃	Low	No	
P ₂ O ₅	Low	Yes	

^aBecomes liquid by absorbing moisture.

**This provides a good seal and prevents air bubbles from being drawn in.
Suction from the weight of the water in the stem increases the filtration rate.
Let the precipitate settle in the beaker before beginning filtration.**

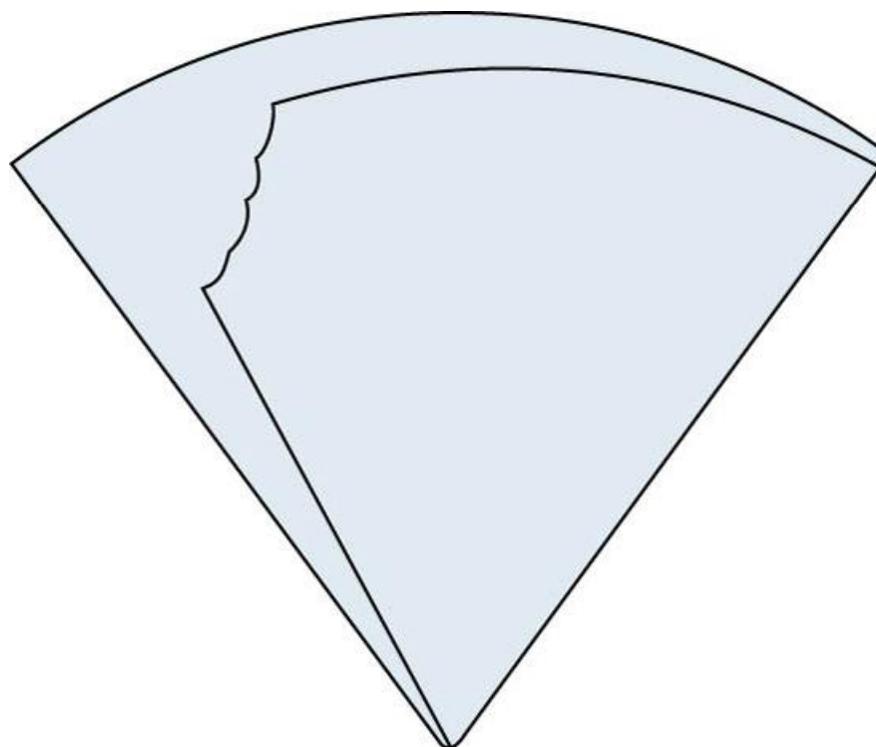


Fig. 2.23. Properly folded filter paper.

These are ashless filter papers.

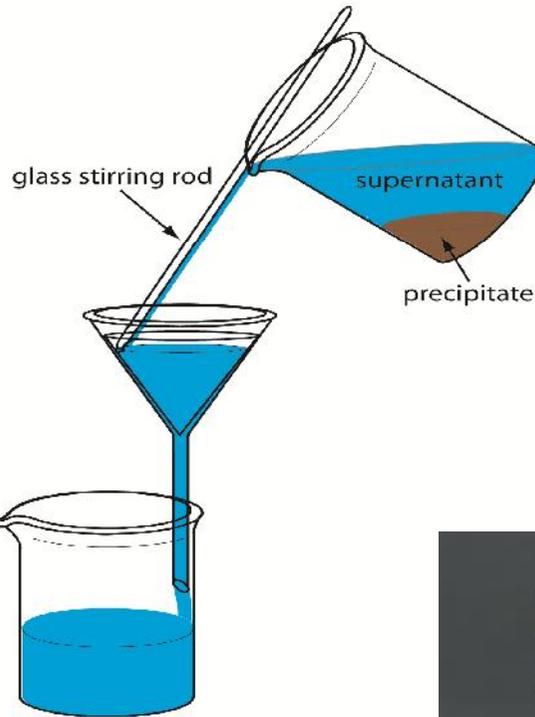
They are ignited away after collection of the precipitate.

Use for gelatinous precipitates.

Table 2.6

Types of Filter Papers

Precipitate	Whatman	Schliecher and Schuell
Very fine (e.g., BaSO_4)	No. 42 (2.5 m m)	No. 589/2 or 5, Blue or Red Band (2–4 m m)
Small or medium (e.g., AgCl)	No. 40 (8 m m)	No. 589/2, White Band (4–12 m m)
Gelatinous or large crystals (e.g., $\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}$)	No. 41 (20–25 m m)	No. 589/1, Black Band (>12–25 m m)



VOLUMETRIC ANALYSIS



Following methods or techniques are being used to measure any chemical parameter

- ❖ Volumetric
- ❖ Gravimetric
- ❖ Potentiometric
- ❖ Coulometric
- ❖ Voltametric
- ❖ Spectrometry
- ❖ Mass spectrometry
- ❖ Chromatography
- ❖ Chromatography mass spectrometry

Percentages of different methods of analysis used in the certification of Standards samples of geological objects in 1951-2005. %

Methods of analysis	1951	1971-1973	2004-2005
Gravimetric	28.5	16.4	4.20
Titrimetric	12.8	16.9	3.5
Electrochemical	1.0	1.3	0.4
Colorimetric/photometric	24.1	12.8	7.1
Atomic emission	15.0	21.9	14.2
Flame photometer	6.4	9.5	3.5
Atomic absorption	-	6.7	10.4
X-ray fluorescence	0.7	7.2	22.1
Mass spectrometer	3.7	-	24.1

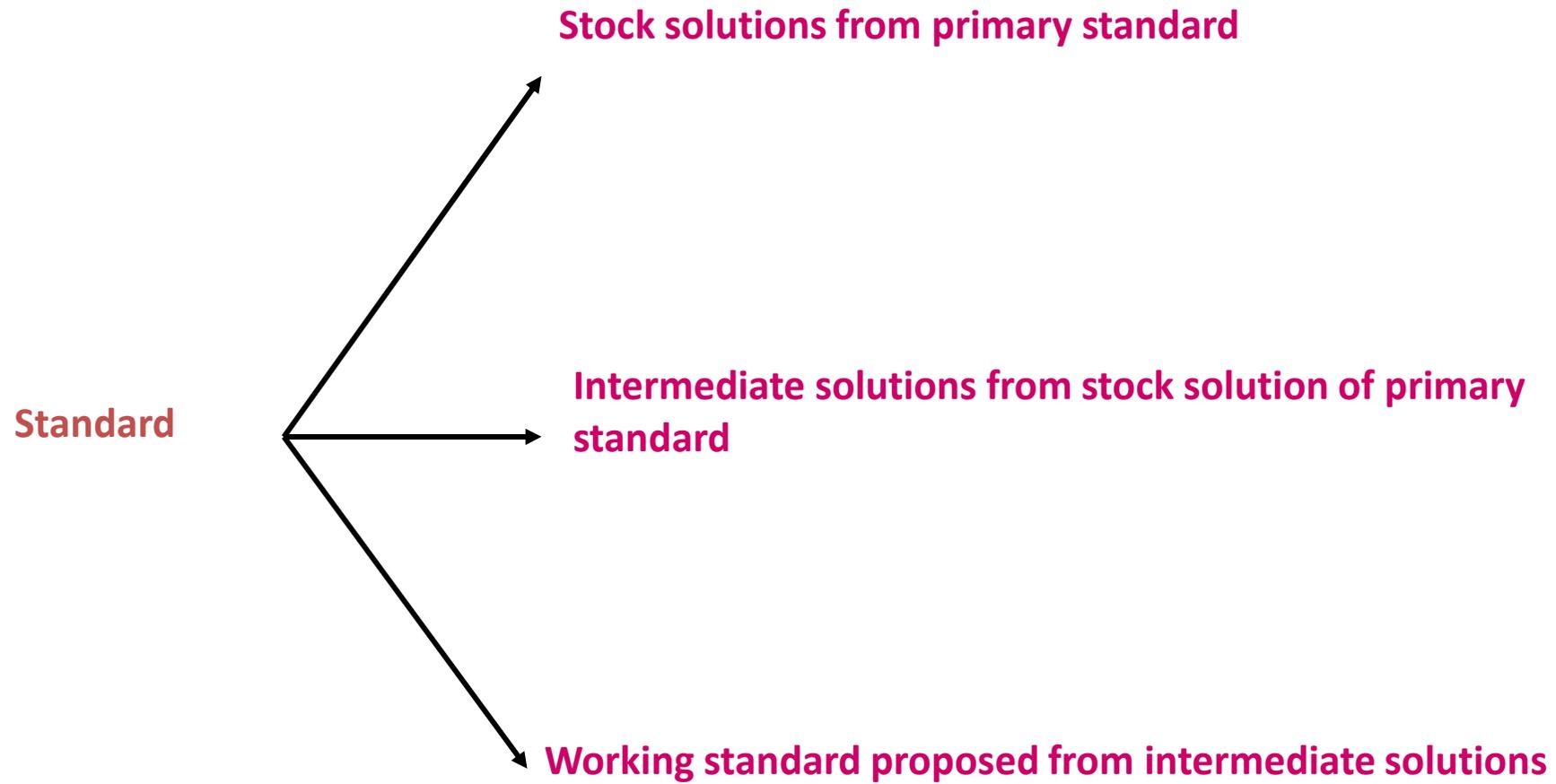
Storage Condition:

Room Temperature= $25 \pm 2^\circ\text{C}$ needs BOD incubator



Fridge or deep fridge:





Traceability to be maintained by having

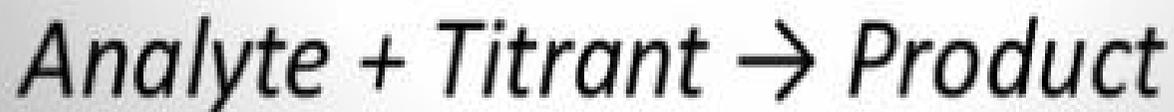
- CRMs or high purity Reference material
- Calibrated analytical balance
- Calibrated weight box
- Calibrated thermometer
- Calibrated Glass wares

Consumption records of CRMs/RM By documentation



Introduction:- A technique for determining the concentration of a solution by measuring the volume of one solution needed to completely react with another solution. Titration process involves addition of solution of known conc. from burette to the measured volume of analyte.

Principle of titration:- It is based on the complete chemical reaction between the analyte and the reagent (titrant) of known concentration.



Terms used in titration

Analyte:- The solution of unknown concentration but known volume.

Titrant:- The solution of known concentration.

Standard solution:- A solution of known concentration is called the standard solution.

Types of standard solution:-

1) Primary standard:- It has certain properties:-

(a) Extremely pure.

For e.g. Na_2CO_3 ,
KHP

(b) Highly stable.

(c) Can be weighed easily.

Secondary standard:- It has certain properties:-

- (a) *Less pure than primary standard.*
- (b) *Less stable than primary standard.*
- (c) *Can not be weighed easily.*

For e.g. NaOH, HCl

Equivalence Point:- Point where the amount of two reactants are just equivalent .

End point:- Point at which the reaction is observed to be complete, this point is usually observe with the help of indicator.

Indicator:- An auxiliary substance which helps in the usual detection of the completion of the titration process at the end point.

For examples:- Methyl orange,
Phenolphthalein, Cresol red, Thymol blue.

Concentration Terms:-

The concentration of standard solutions (titrants) are generally expressed in units of either molarity (C_M , or M) or normality (C_N , or N).

Molarity (M):-It is the number of moles of a solute per liter of solution.

Normality:- It is the gram equivalent weight of solute dissolved per litre of solution.

Molality:- It is the number of moles of solute present in per kilogram of solvent.

Titrimetric calculation :-

It is based on the following law of equivalence:-

$$N_a V_a = N_s V_s$$

or

$$M_a V_a = M_s V_s$$

Where,

N_a is the normality of analyte.

V_a is the volume of the analyte.

N_s is the normality of standard solution.

V_s is the volume of standard solution used.

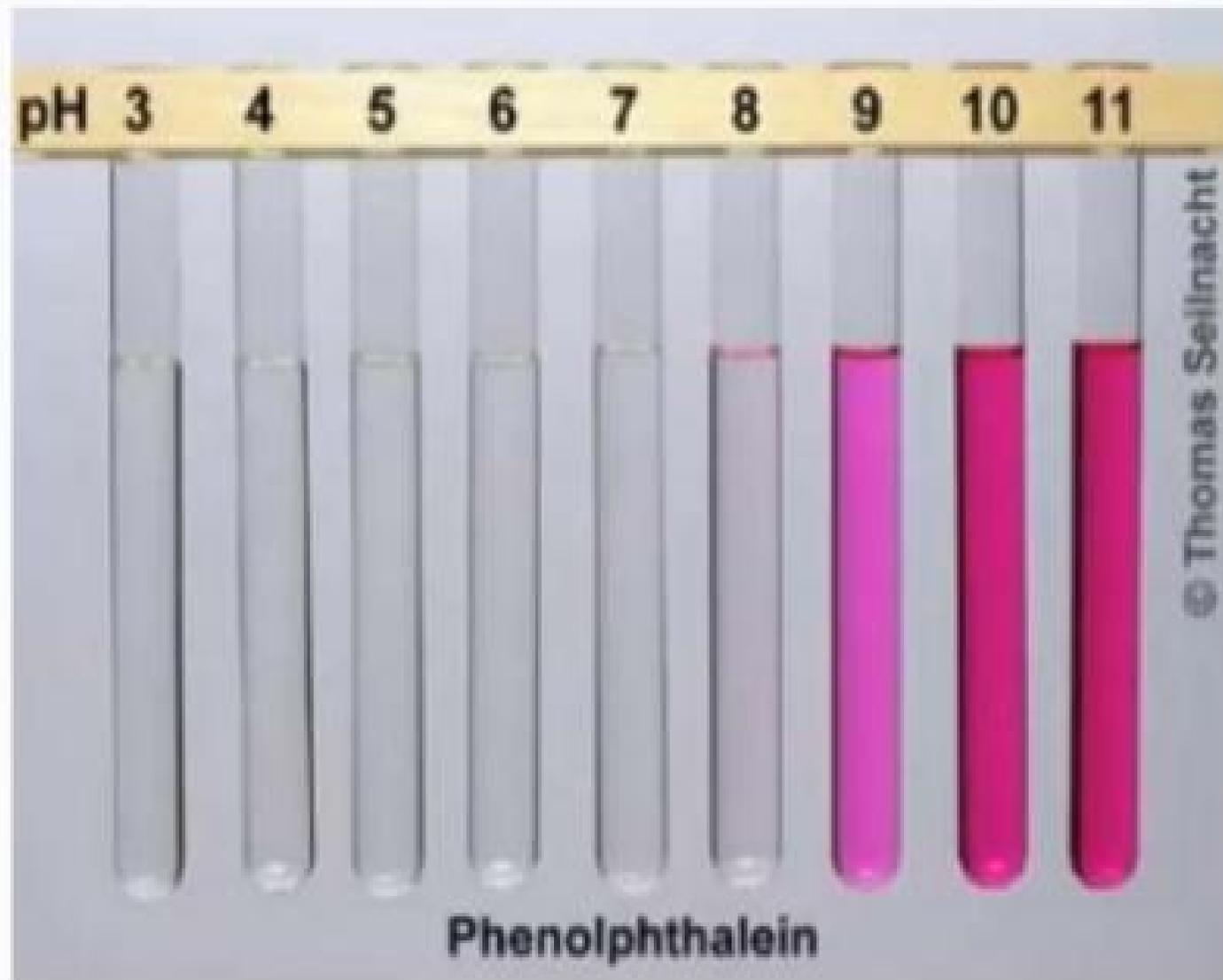
M_a is the molarity of analyte.

M_s is the molarity of standard solution.

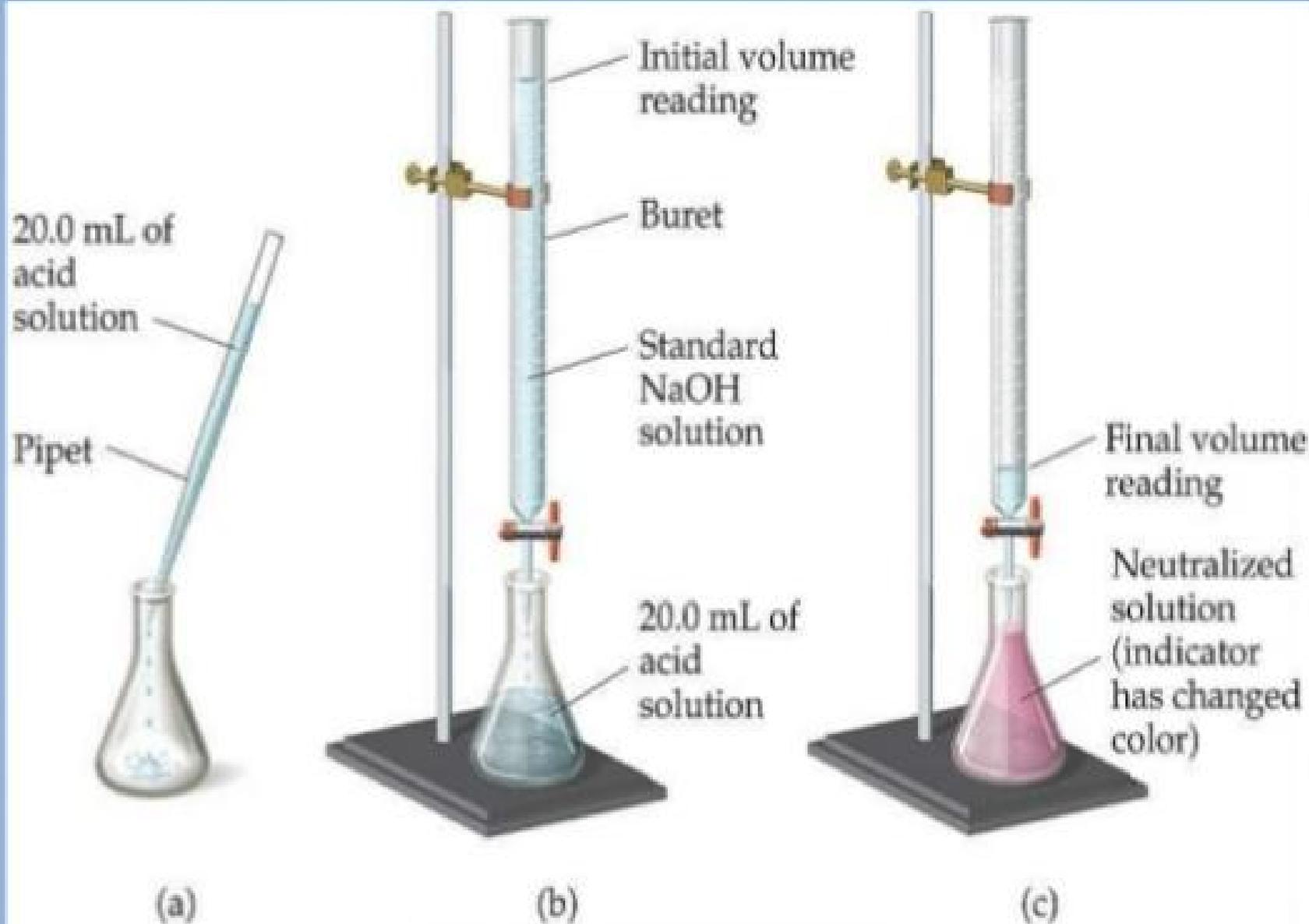
Acid-Base Indicators

Indicator	Color on acidic side	Range of color change	Color on basic side
Methyl violet	Yellow	0.0-1.6	Violet
Bromophenol blue	Yellow	3.0-4.6	Blue
Methyl orange	Red	3.1-4.4	Yellow
Methyl red	Red	4.4-6.3	Yellow
Litmus	Red	5.0-8.0	Blue
Bromothymol blue	Yellow	6.0-7.6	Blue
Phenolphthalein	Colorless	8.3-10.0	Pink
Alizarin yellow	Yellow	10.1-12.0	Red

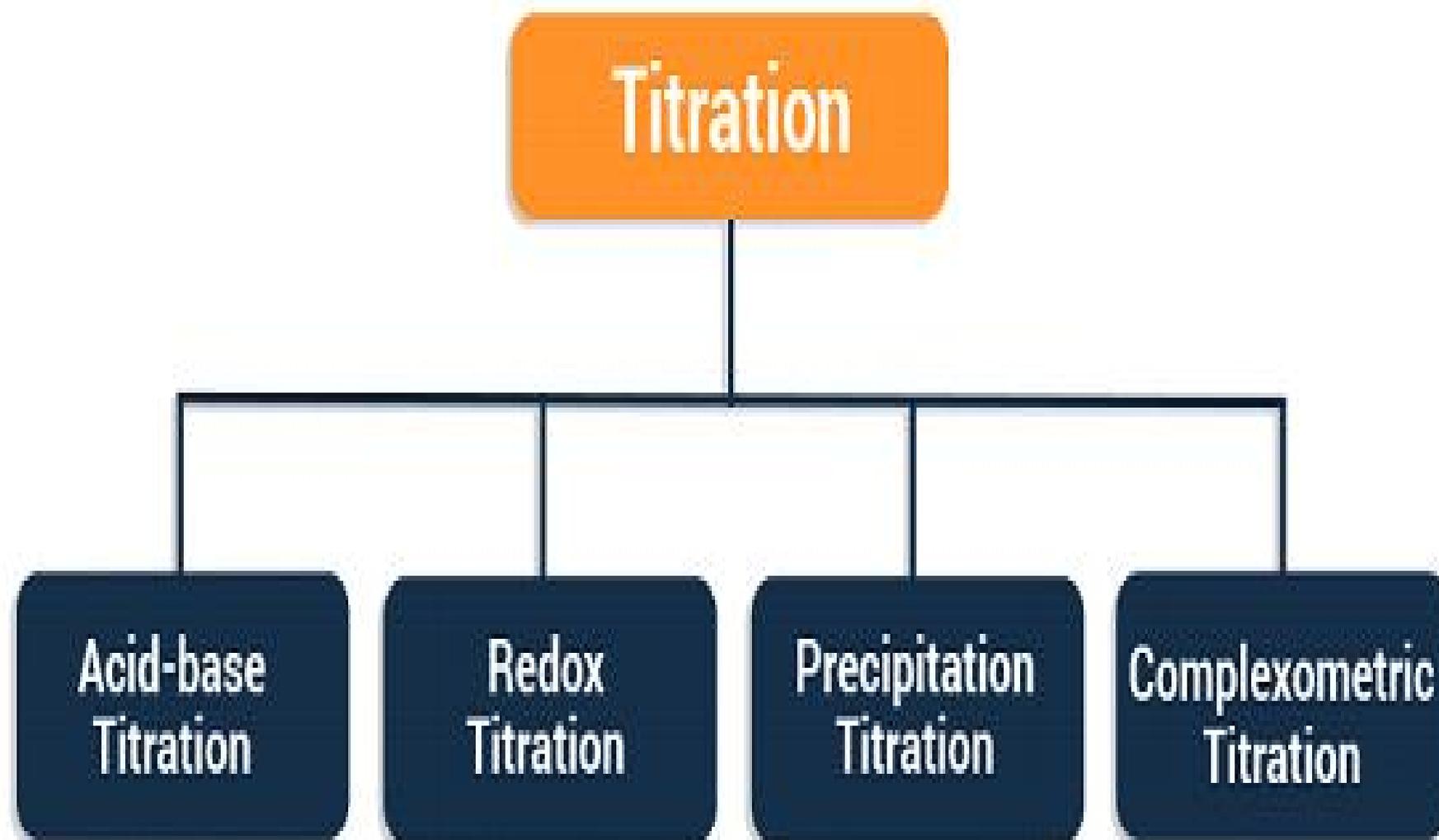
Range of color indicator change



Titrimetric apparatus :-



Types of Titration



Complexometric Titrations:- As the name indicates, the end point is seen by formation of a complex molecule. Here titrant and titrand react to form a complex till end point is reached. Once complex is formed, the complex is stable and no further reaction takes place.

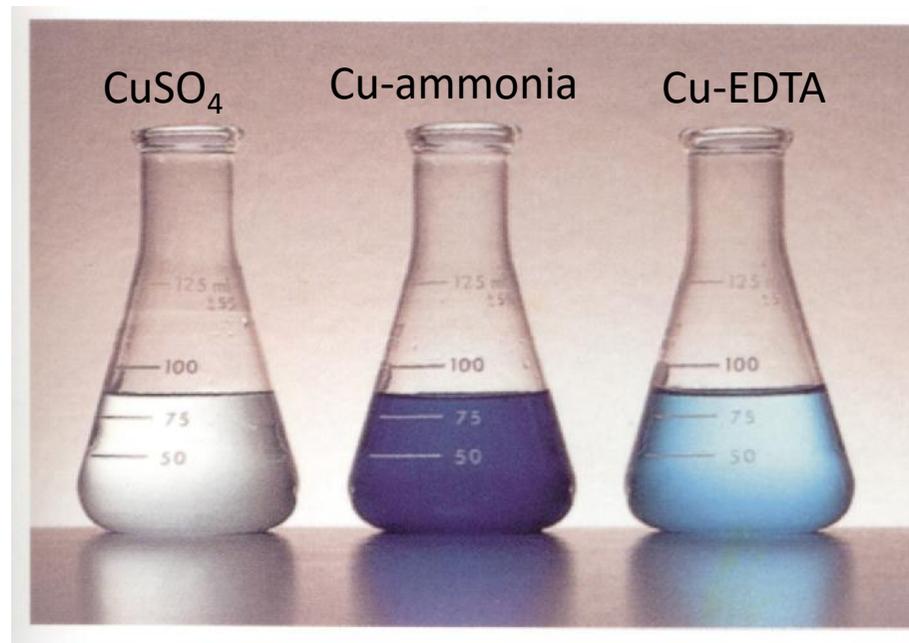


EDTA Titrations

Auxiliary Complexing Agents

2.) **Illustration:**

- Titration of Cu^{+2} (CuSO_4) with EDTA
- Addition of Ammonia Buffer results in a dark blue solution
 - **Cu(II)-ammonia complex is formed**
- Addition of EDTA displaces ammonia with corresponding color change

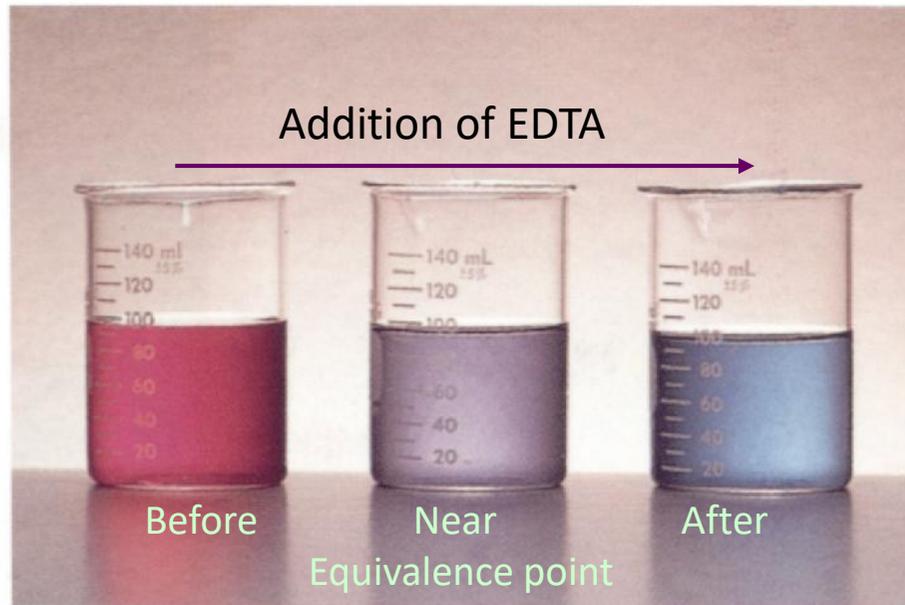


EDTA Titrations

Metal Ion Indicators

2.) **Illustration**

- Titration of Mg^{2+} by EDTA
 - Eriochrome Black T Indicator



EDTA Titrations

Metal Ion Indicators

3.) Common Metal Ion Indicators

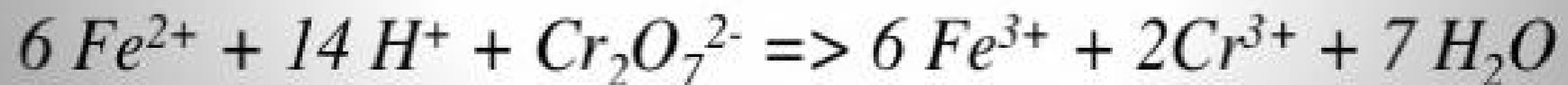
- Most are pH indicators and can only be used over a given pH range

Table 12-3 Common metal ion indicators

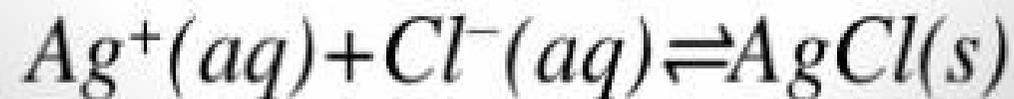
Name	Structure	pK_a	Color of free indicator	Color of metal ion complex
Calmagite		$pK_2 = 8.1$ $pK_3 = 12.4$	H_2In^- red HIn^{2-} blue In^{3-} orange	Wine red
Eriochrome black T		$pK_2 = 6.3$ $pK_3 = 11.6$	H_2In^- red HIn^{2-} blue In^{3-} orange	Wine red
Murexide		$pK_2 = 9.2$ $pK_3 = 10.9$	H_4In^- red-violet H_3In^{2-} violet H_2In^{3-} blue	Yellow (with Co^{2+} , Ni^{2+} , Cu^{2+}); red with Ca^{2+}
Xylenol orange		$pK_2 = 2.32$ $pK_3 = 2.85$ $pK_4 = 6.70$ $pK_5 = 10.47$ $pK_6 = 12.23$	H_5In^- yellow H_4In^{2-} yellow H_3In^{3-} yellow H_2In^{4-} violet HIn^{5-} violet In^{6-} violet	Red
Pyrocatechol violet		$pK_1 = 0.2$ $pK_2 = 7.8$ $pK_3 = 9.8$ $pK_4 = 11.7$	H_4In red H_3In yellow H_2In^{2-} violet HIn^{3-} red-purple	Blue

Redox titration:- Redox titration is based on the redox reaction (oxidation-reduction) between analyte and titrant.

For example:-



Precipitation titrations:- The titrations which are based on the formation of insoluble precipitates, when the solutions of two reacting substances are brought in contact with each other, are called Precipitation titration.



Requirement For Successful Volumetric Titration



- ★ Reaction must be **stoichiometric**, well defined reaction between titrant and analyte.
- ★ Reaction should be **rapid**.
- ★ Reaction should have **no side reaction, no interference** from other foreign substances.
- ★ Must have some **indication of end of reaction**, such as color change, sudden increase in pH, zero conductivity, etc.
- ★ Known **relationship** between endpoint and equivalence point.

-
- Concentration: is a general term expressing the amount of solute contained in a given material. Expressed by different ways
 - Molarity(M): The number of moles of solute divided by the number of liters of solution containing the solute. (is gram molecular weight dissolved in one liter of solution)
 - Molarity = moles of solute / volume in liters
 - Milli moles of solute / volume in milliliters.
 - Moles = weight (gms) / MW or
 - Millimoles = weight(mg) / MW

Normality (N)

- Defined as no of equivalents of solute divided by the number of liters of solution containing the solute. (gm equivalent weight dissolved in one liter of solution)
- Normality = equivalents of solute / volume in liters
- Milli eq. of solute / volume in milliliters.
- Equivalents = weight (gms) / EW or
- Milliequivalent = weight(mg) / EW

Relation of Normality and Molarity

- Molarity = weight / MW x Volume
- MW = Weight / Molarity x volume similarly
- Normality = weight / EW x Volume
- EW = Weight / normality x volume
- EW = MW / h
 - Where h reacting unit.
 - For acid H⁺ is reacting unit and for base OH⁻
 - For Oxdⁿ redⁿ e⁻ is reacting unit
 - For Ionic species valences

Equivalent weight

- Is defined as part by wt of substance which is chemically equivalent to one part by wt of hydrogen or 8 part by wt of oxygen or 35.5 part by wt of chlorine.
- Thus in finding out equivalent wt we find out how many grams of that substance are directly or indirectly equivalent to one gram of hydrogen
- It depends on reaction in which it takes place

Percent (%)

- Weight percent (w/w) = $\frac{\text{weight of analyte} \times 100}{\text{weight of sample}}$
- Volume percent (v/v) = $\frac{\text{volume of analyte} \times 100}{\text{volume of sample}}$
- Weight percent (w/v) = $\frac{\text{weight of analyte} \times 100}{\text{volume of sample}}$

Requirements of Primary Standards

- It should be 100% pure or with known purity
- Should be stable to drying temp.
- Usually solid to make it easier to weigh
- Easy to obtain, purify and store, and easy to dry
- Inert in the atmosphere
- High formula weight so that it can be weighed with high precision
- It should not absorb moisture, or should not react with oxygen or CO_2
- Reaction with analyte should be single, rapid complete and stoichiometric

Equivalent Weight :It is defined as the number of parts by weight, chemical species combined with or displaced by 1.008 parts of hydrogen, 8 parts of oxygen or 35.5 parts by weight of chlorine and 127 part of iodine..

$$\begin{aligned} 75\text{g Ca(OH)}_2; 8\text{L} \\ 2.027\text{ eq} \div 8\text{L} \\ = \boxed{0.253\text{N}} \end{aligned}$$

Oxidizing Agent :Pot.Permagnate

Reactions of oxalic acid

A. Chemical equations



$$2\text{KMnO}_4 = 50$$

$$2\text{MW} \quad 5 \times 16$$

$$\text{Eq Wt} = \text{MW}/5 = 158/5 = 31.606$$

Oxidizing Agent :Pot.dichromate



Available Oxygen = 3atoms = 3x16

$$\text{K}_2\text{Cr}_2\text{O}_7 = 3\text{O}$$

$$\text{MW} = 3 \times 16$$

$$\text{Eq Wt} = \text{MW}/6 = 294.18/6 = 49.03$$

Reducing Agent Equivalent Weight Ferrous Sulphate



$$2\text{FeSO}_4 = \text{O}$$

$$2\text{MW} = 16$$

$$\text{Eq Wt} = 2\text{MW}/2 = \text{MW} = 152$$

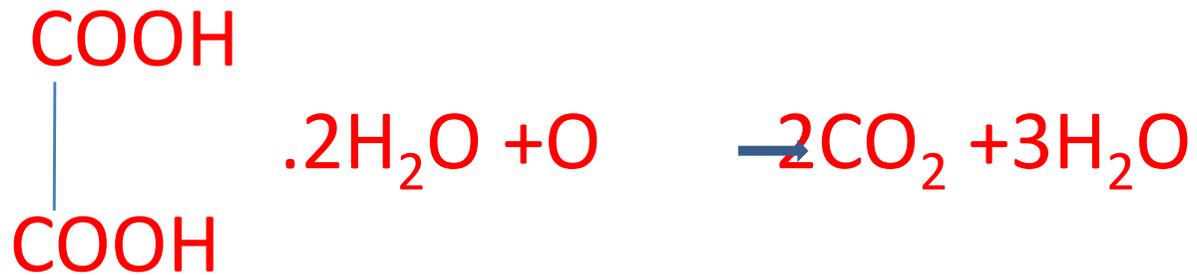
Ferrous Ammonium sulphate



$$\text{O} = 16$$

$$\text{Eq Wt} = 2\text{MW}/2 = 392$$

Oxalic Acid



$$\text{MW} = 126$$

$$\text{Eq Wt} = \text{MW}/2 = 126/2 = 63$$

Sodium thiosulphate(Hypo)



$$2\text{MW} = 214$$

$$2 \text{ MW} = 2 \times 127$$

$$\text{EqWt} = \text{MW}$$

Iodimetric Titrations: Iodimetric titrations are defined as those iodine titration in which a standard iodine solution is used as oxidants and iodine is directly titrated with the reducing agents like thiosulphate, sulphite, arsenite by titrating them against standard solution of iodine run in from a burette .

Iodometric Titrations: Iodometric titrations are defined as those iodine titration in which some oxidizing agent liberates iodine from an iodide and the liberated iodine is titrated against standard solution of reducing agent from the burette.

Some basic question generally asked to chemist :

- Have Chemical Instruments improved in recent years?
- Do Chemists provide meaning full answer to problems?
- Are chemists providing answer with better accuracy and precision compared to 60 years ago?

YES : There has been revolution in Analytical chemistry

- New instruments .
- Computing power and improved quality of information through computing .
- More detailed analysis can be done.
- Detection of ultra trace amounts.
- Faster methods of sample preparation :
 - microwave
 - Ultrasonic

Fluoride

Fluorides are properly defined as binary compounds or salts of fluorine with another element (like Na, K etc.) combined chemically in the form of fluorides.

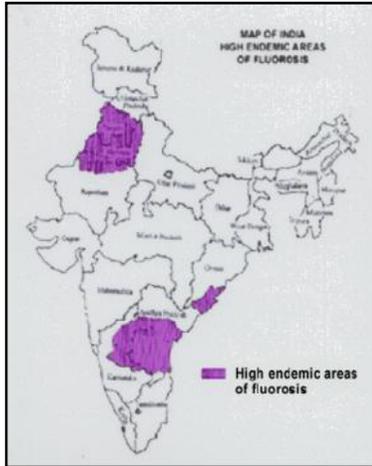
High **pH** of water favours high fluoride content in water .

Naturally, although the controlled enrichment of drinking water with **Fluoride is essential for humans** because traces of fluorine are necessary for normal mineralization, formation of caries – resistant enamel, strong teeth, healthy bones and for normal reproduction.

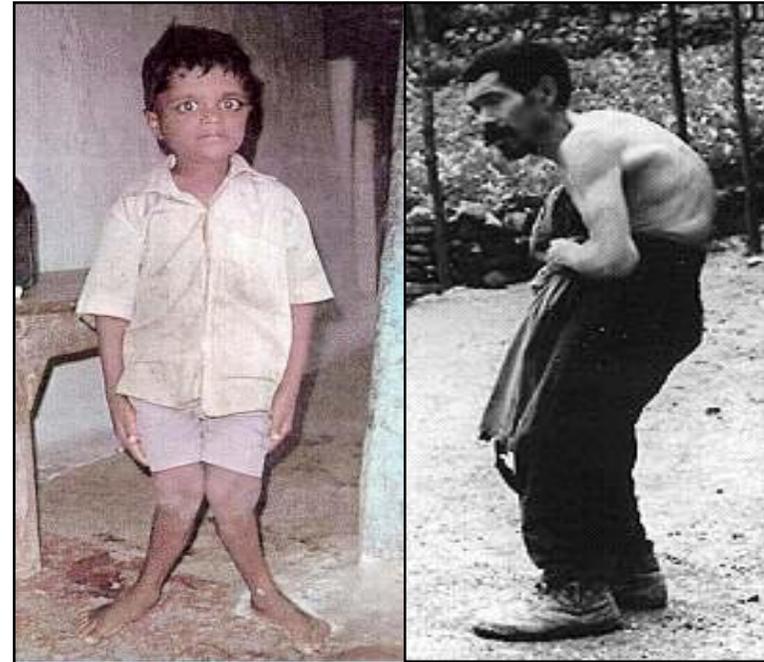
According to WHO 1984 and Bureau of Indian Standard Drinking Water Specification 2002, the **maximum permissible limit of fluoride** in drinking water is **1.5 ppm** and highest desirable limit **1 ppm**.

Fluoride concentration above **1.5 ppm** in drinking water cause **dental fluorosis** and much higher concentration **skeletal fluorosis**, low concentration (**0.5 ppm**) provide protection against dental caries.

FLUOROSIS

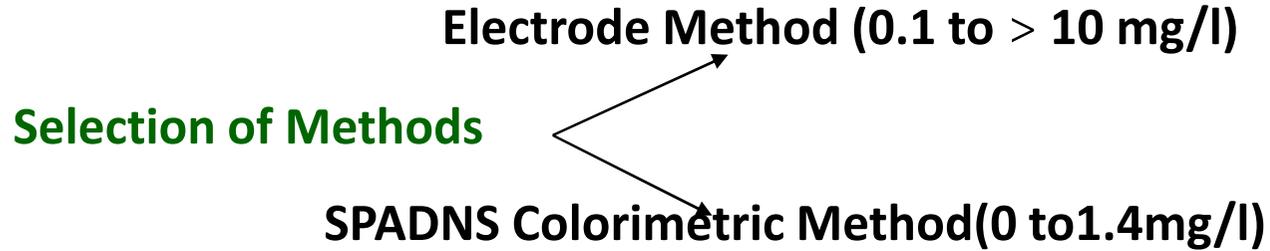


Dental Fluorosis



Skeletal Fluorosis

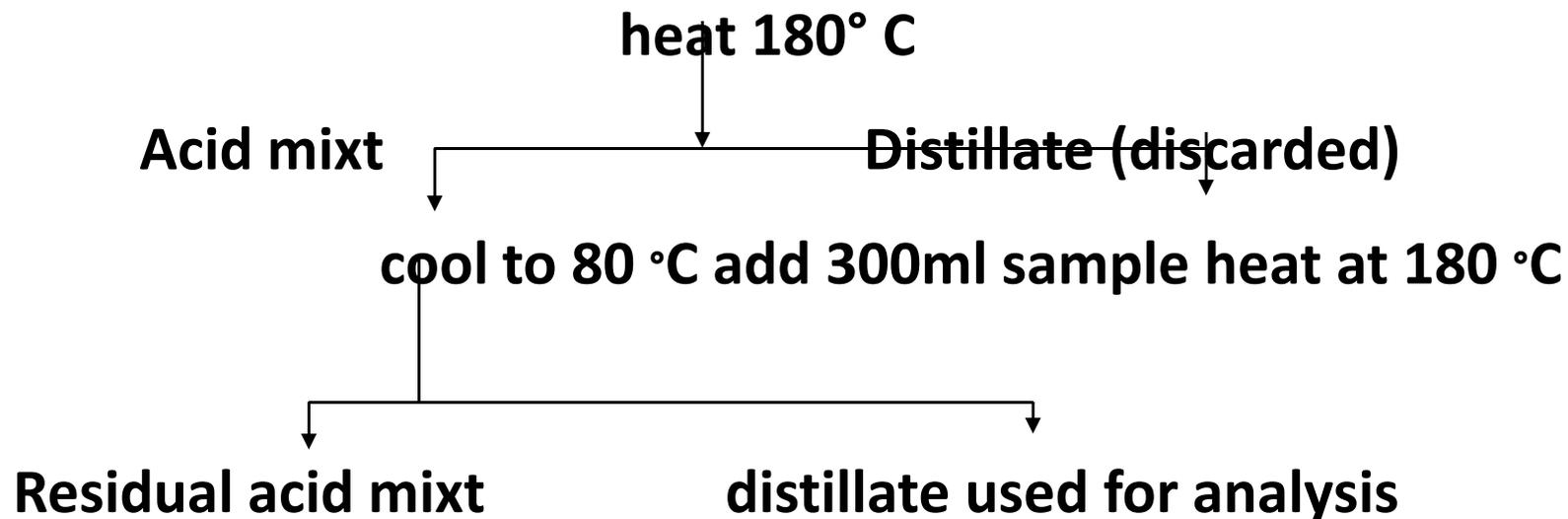
- **Permissible limit of fluoride in drinking water is 1.5 ppm and highest desirable limit 1 ppm.**
- **Fluoride concentration above 1.5 ppm in drinking water cause dental fluorosis much higher concentration skeletal fluorosis.**
- **Low fluoride concentration (0.5 ppm) provide protection against dental caries.**



Sampling & Storage: Polythene/Glass bottle rinsed with portion of sample. Dechlorination using sod. arsenite rather than sod thiosulphate.

Preliminary Distillation Step: To remove nonvolatile materials from water.

400ml DW +200ml Conc. sulphuric acid

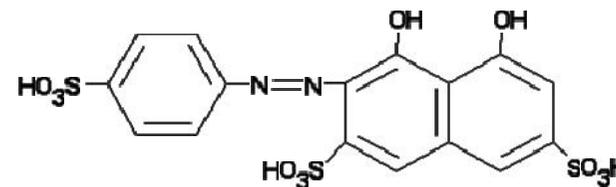


SPADNS Spectrophotometric Method for Analysis of Fluoride

- Fluoride reacts with certain zirconium dyes to form a colourless complex and another dye
- The dye becomes progressively lighter as fluoride concentration increases
- Absorbance is measured at **570 nm**
- Prepare calibration curve using absorbance values for known standards
- Read fluoride values for the samples



SPECTROPHOTOMETER



SPADNS

Sod. 2-(parasulfophenylazo)-1,8
dihydroxy-3,6-naphthalene disulfate

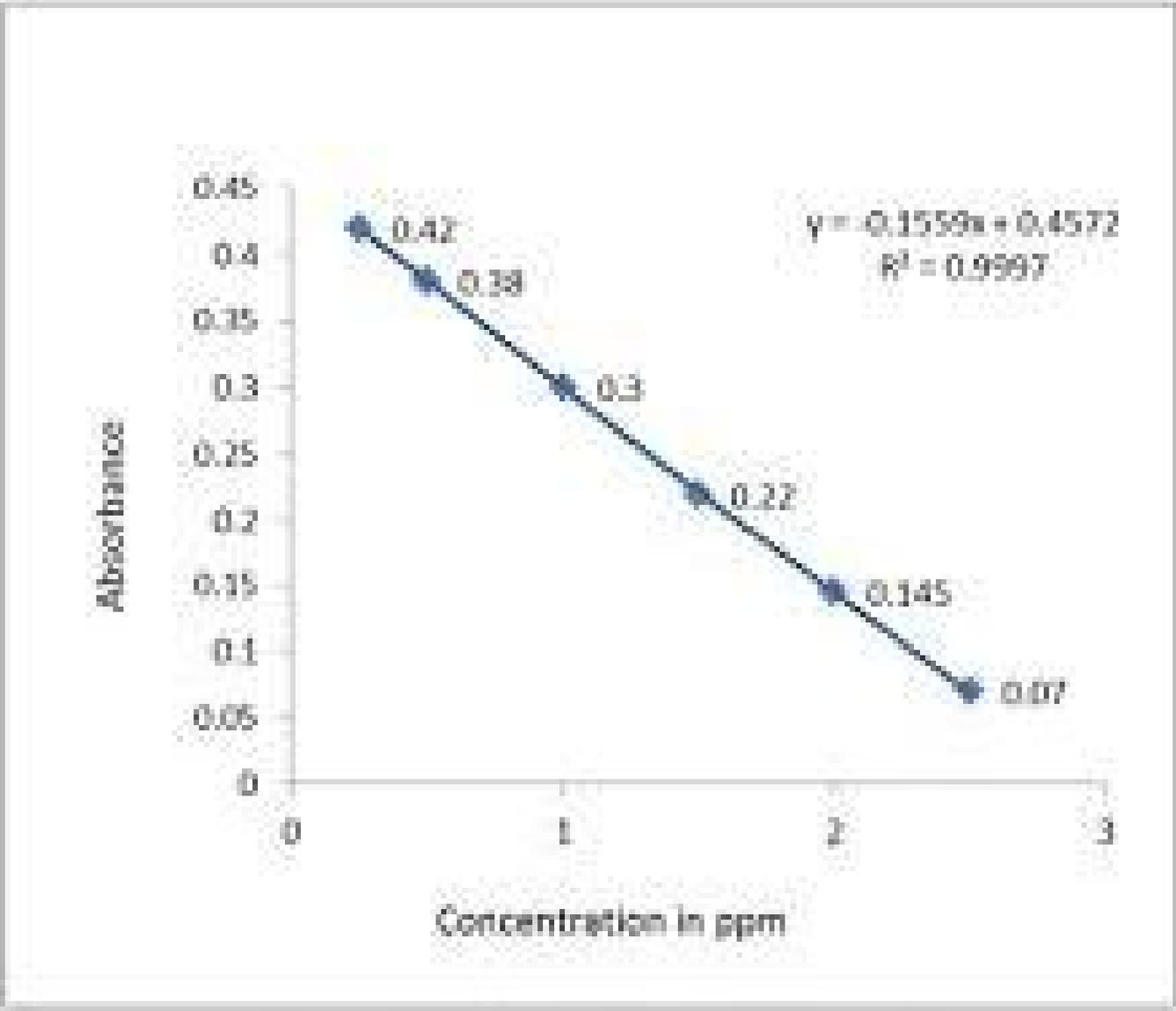
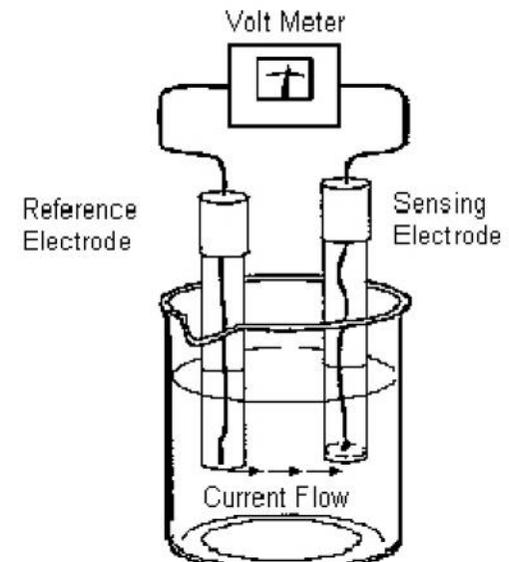


Figure 2: Fluoride standard calibration curve.

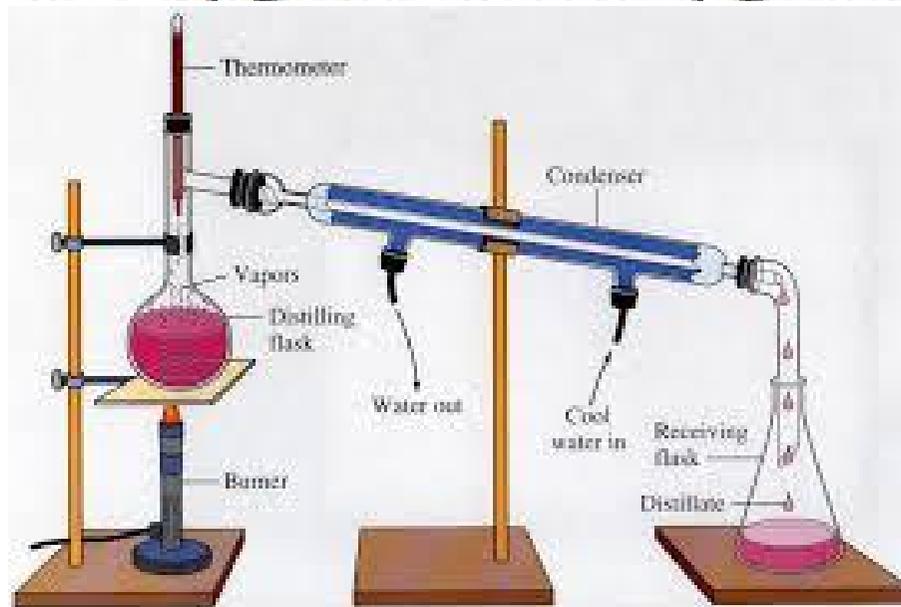
Ion Selective Electrode Method

- It is ion selective sensor, Laser type doped Lanthanum fluoride crystal across within a potential is established by fluoride solution.
- $\text{Ag}|\text{AgCl}, \text{Cl}(0.3\text{M}), \text{F}90.001\text{M}) \text{LaF}_3 \text{ Test}$



Phenolic Compound

a. Principle: Steam-distillable phenols react with 4-aminoantipyrine at $\text{pH } 7.9 \pm 0.1$ in the presence of potassium ferricyanide to form a colored antipyrine dye. This dye is extracted from aqueous solution with CHCl_3 and the absorbance is measured at 460 nm. This method covers the phenol concentration range from $1.0 \mu\text{g/L}$ to over $250 \mu\text{g/L}$ with a sensitivity of $1 \mu\text{g/L}$.



Sample Collection for Heavy Metals

All metals except Hg :

Container : Reagent bottle or polyethylene bottle

Combining with 0.5% HNO₃

Store up to maximum 1 month.

Hg:

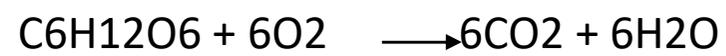
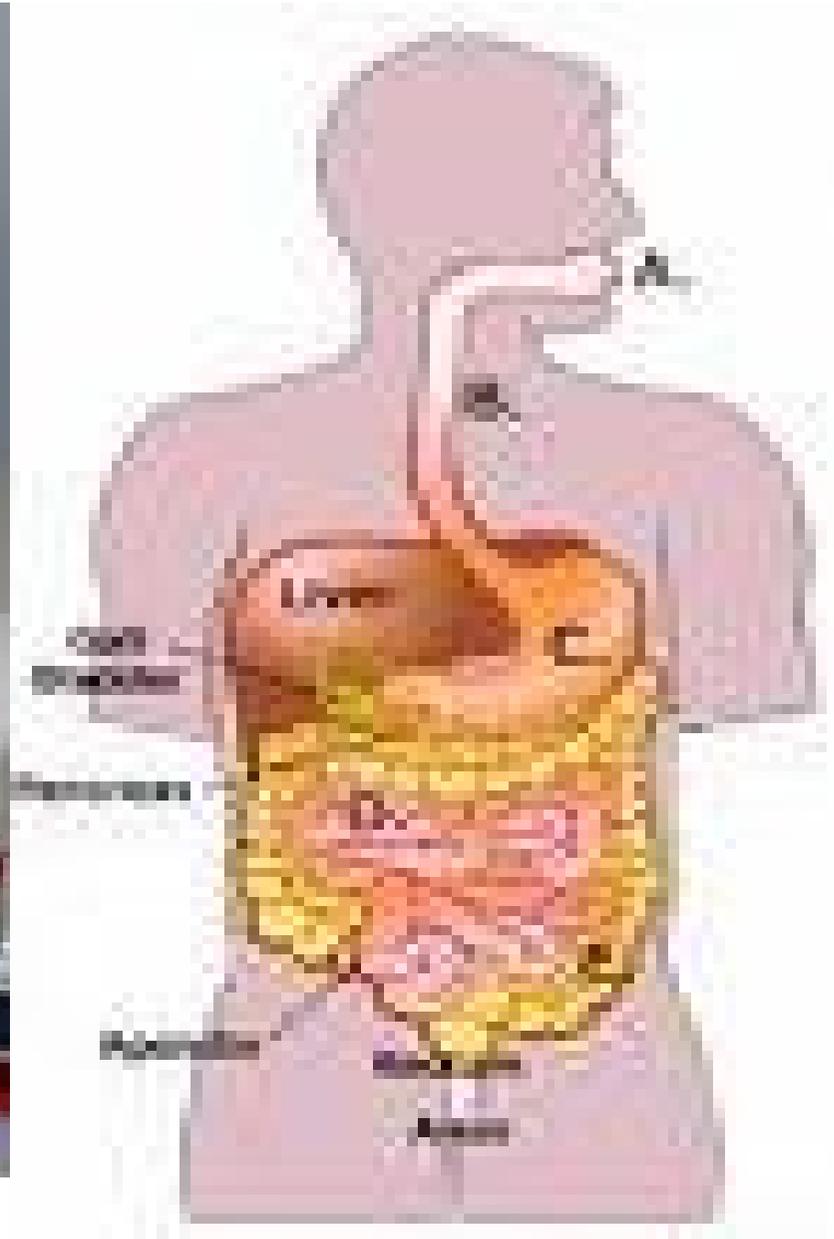
Prewashed reagent bottle. Rinse the bottle twice with sample.

2 ml of 4M nitric acid and 5 ml of 2.5% Pot. Dichromate.

Store up to 5 weeks (maxm) in freeze.



Organic matrix + Oxidizing agent
 $\text{CO}_2 + \text{H}_2\text{O}$



Type of Sample Preparation

- Acid Digestion
 - Hot
 - Cold



- Fusion With alkali carbonates



- Ashing in muffle furnace

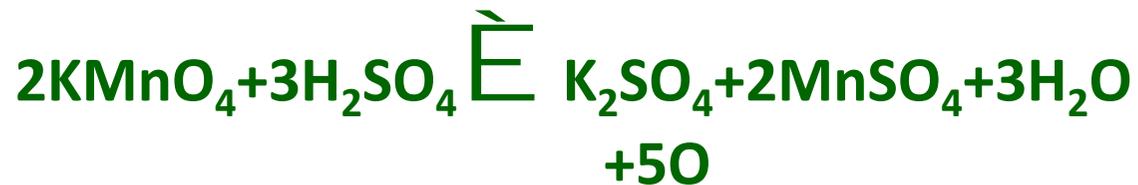


- Micro wave digestion



Cold digestion For Volatile metals (Hg, As) estimation

Digestion of organic using nascent Oxygen produced by reaction of Potassium permagnate with Conc. sulfuric acid.



Example:

0.5 g of tissue or 1ml blood in conical flask containing 5 ml conc. sulfuric acid(cooled in ice bath).

Stopper the flask and heat for 2 hrs at 70 °C.

15 ml of 20% w/v hydroxylamine hydrochloride is added to reduce the excess of permagnate.

The sample is ready for analysis using AAS –VGA.

Figure : Times typically required for digestions

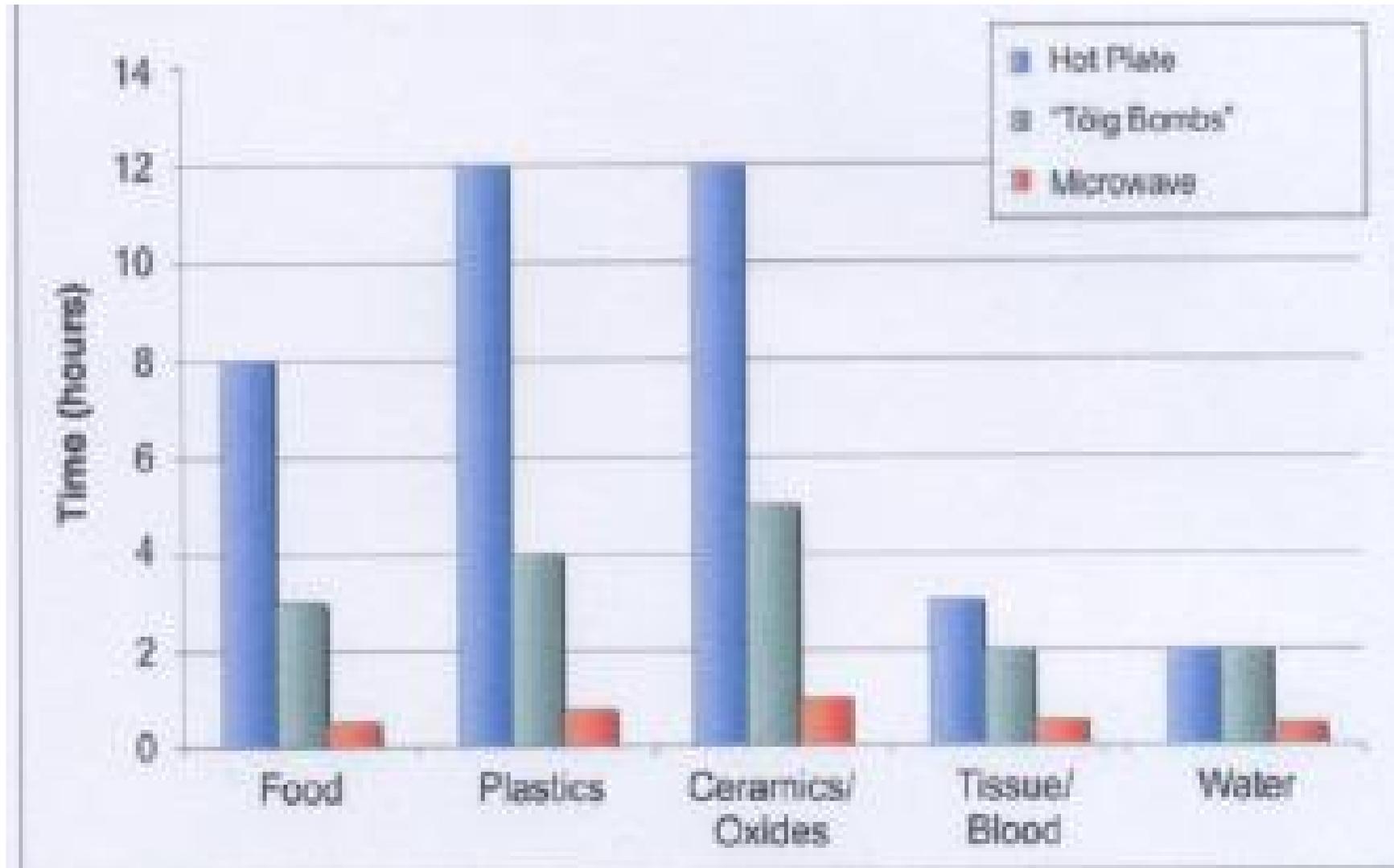


Table: Effect of Laboratory Atmosphere on Pb Blank Levels

	ng Pb found in 1 mL of Acid
Acid Blank	2
Covered for 2 hrs on bench	1, 1.5
Uncovered for 2 hrs on bench	5.5, 6.2
Covered in hood for 3 hrs	4.5
Uncovered in hood for 3 hrs	25

ARSENIC TOXICITY

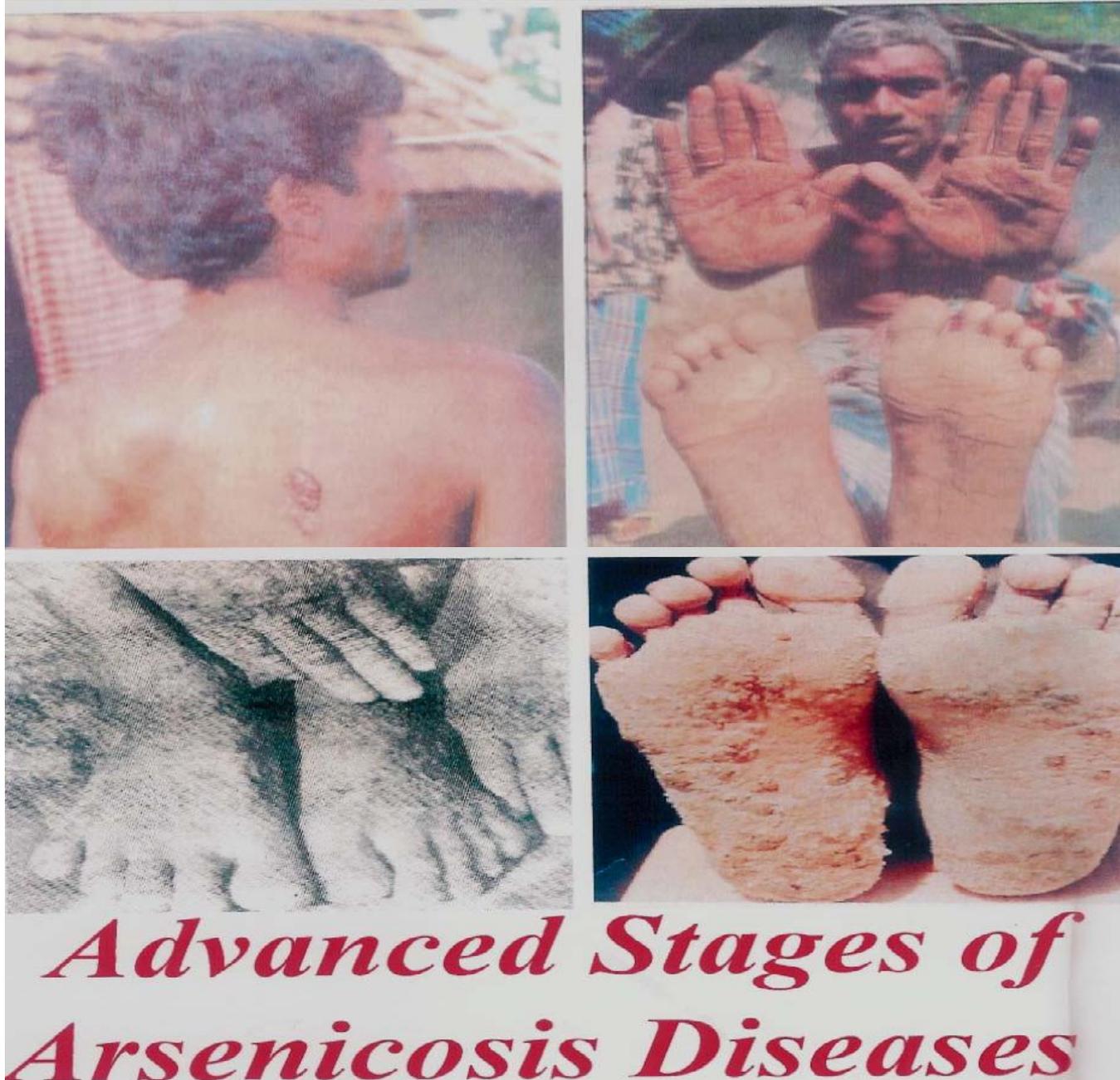


School of Environmental Studies, Jabarpur University

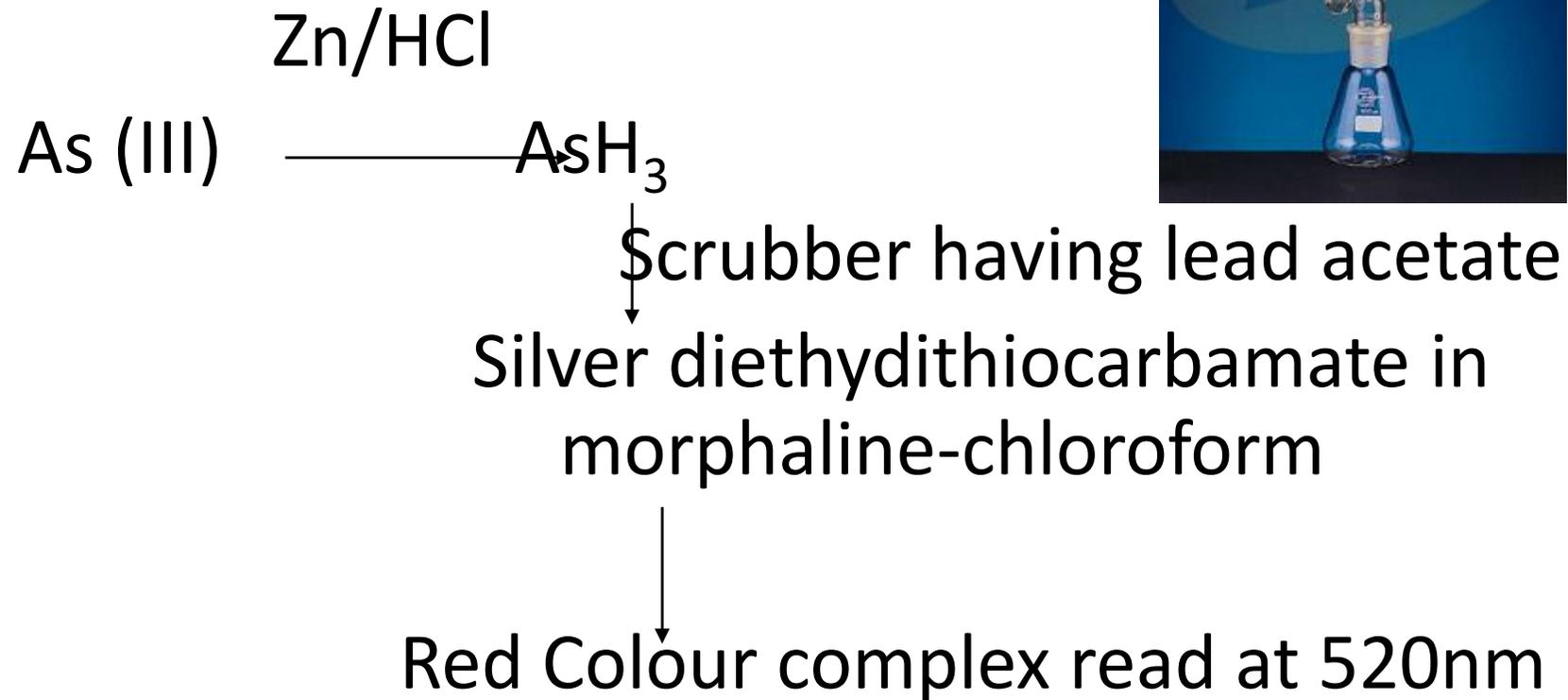


- Arsenite - As (III) is more toxic than Arsenate – As (V).
- Maximum permissible level for arsenic in drinking water is 10 ppb.

Arsenic



Arsenic

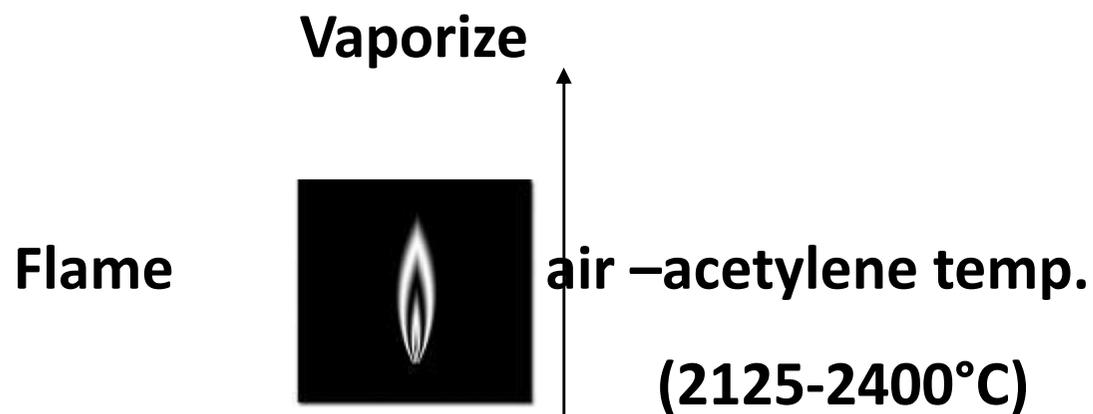


MDL : 1μ As



182
Spectrophotometer

Vapour Generation Accessory (VGA)



Atoms like As,Sb,Se,Te,Bi,Sn,Hg and Zr

Causing less availability of atomize atoms ,resulting less sensitivity

Hence it is required to convert them into their respective hydride

Atomize at low temperature say 1000°C.

Atomic Absorption Spectrophotometer



Atomic Spectrophotometer



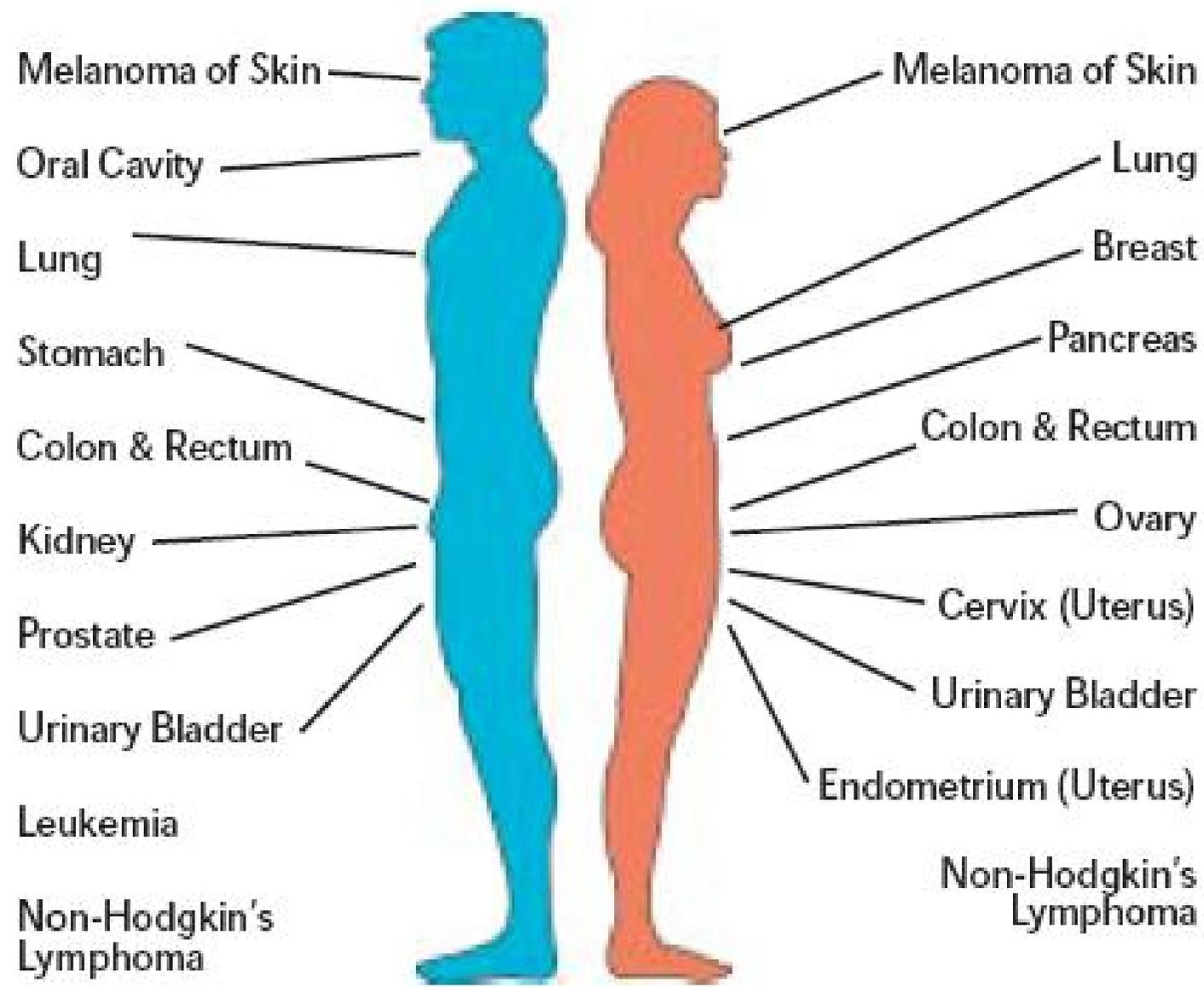
Quantitation \longrightarrow AAS



Pesticides

Pesticides are widely used in agriculture to protect crops against insects (insecticides) fungus (fungicides) and weed (herbicides).

- **Organochlorine**
- **Organophosphorus**
- **Carbamates**
- **Botanical pesticide**



Some of the cancers linked to pesticide use.

Strength of solution is expressed in

- Percentage
- Molality
- Molarity
- Normality
- Parts per million (ppm)
- Parts per billion (ppb)
- Parts per trillion (ppt)

Concept of ppm & ppb

One part per million (ppm): Denotes one part per 1,000,000 parts, one part in 10^6 , and a value of 1×10^{-6} . This is equivalent to one drop of water diluted into 50 liters (roughly the fuel tank capacity of a compact car), or **one second of time in approximately 11½ days. 1mm in 10 Km.**

ppm = mg/kg = µg/g

One part per billion (ppb): Denotes one part per 1,000,000,000 parts, one part in 10^9 , and a value of 1×10^{-9} . This is equivalent to 1 drop of water diluted into 250 chemical drums (50 m^3), or **one second of time in approximately 31.7 years.**

10 ppb = A pinch of salt in 10 tons potato chips .

1mm in 1000 Km (approxm distance between Lucknow and Kolkata)

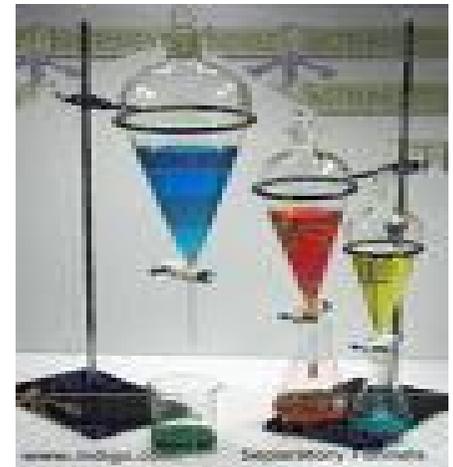
ppb = µg/kg = ng/g

Pesticide Residue Analysis :

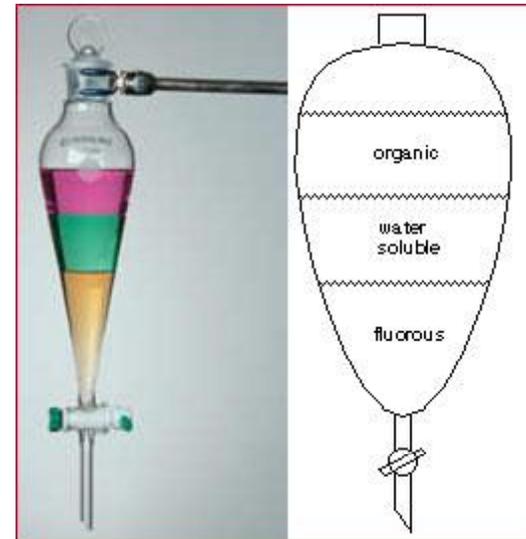
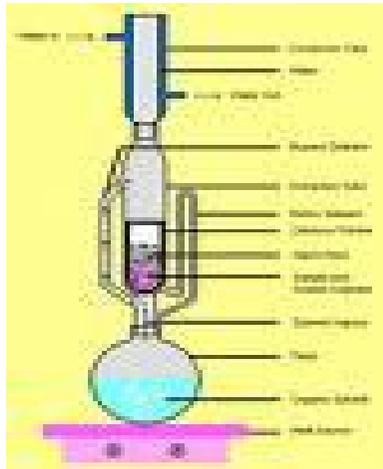
1. Sampling
2. Storage
3. Extraction
4. Demoisturization
5. Clean up
6. Concentration
7. End Analysis
8. Identification and Quantification

Extraction

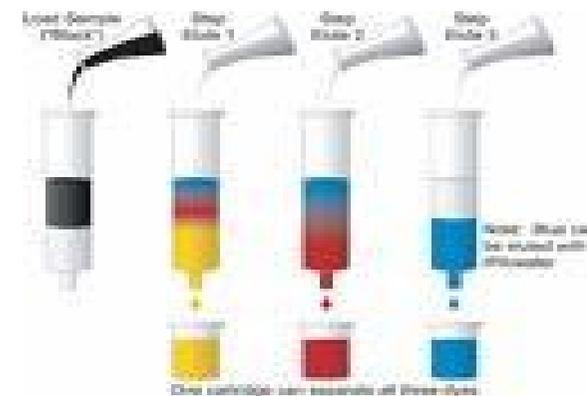
Liquid –Liquid Extraction using separating funnel



Solid Sample using solid-liquid Extraction: Soxlet Apparatus



Solid Phase Extractor (SPE)



Demoisturization : Using anhyd. Sodium sulphate

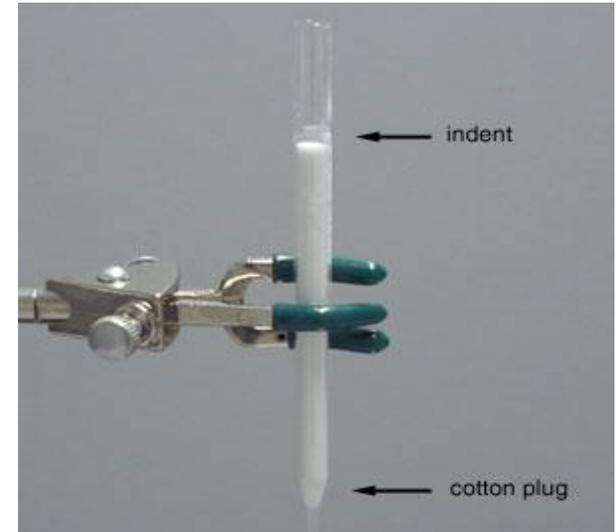
Cleanup: Using column Chromatography:-

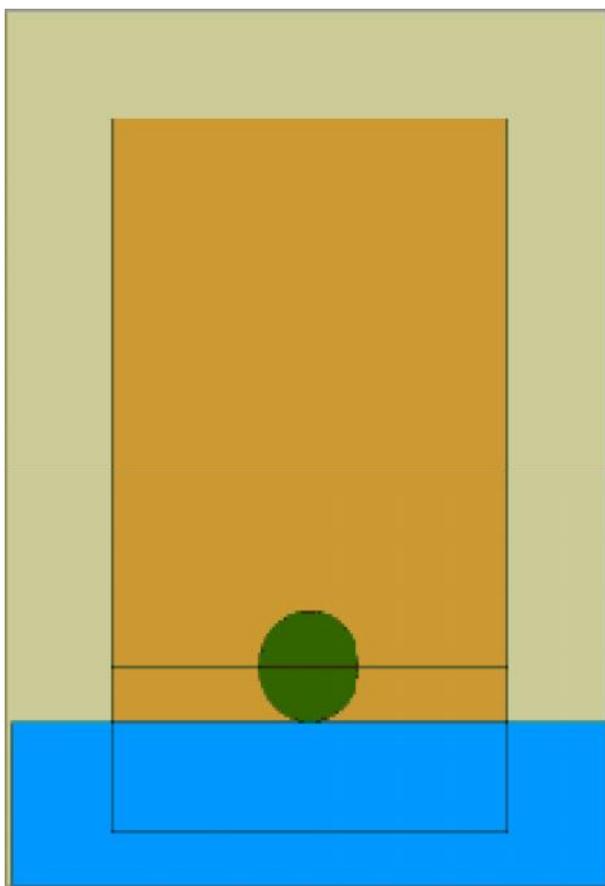
- Alumina
- Silica
- Florosil (Magnesium silicate)

Pre wet column with 20ml n-hexane, load the concentrate extract and elute with mixture of diethyl ether with n-hexane (6%,15%,50%)

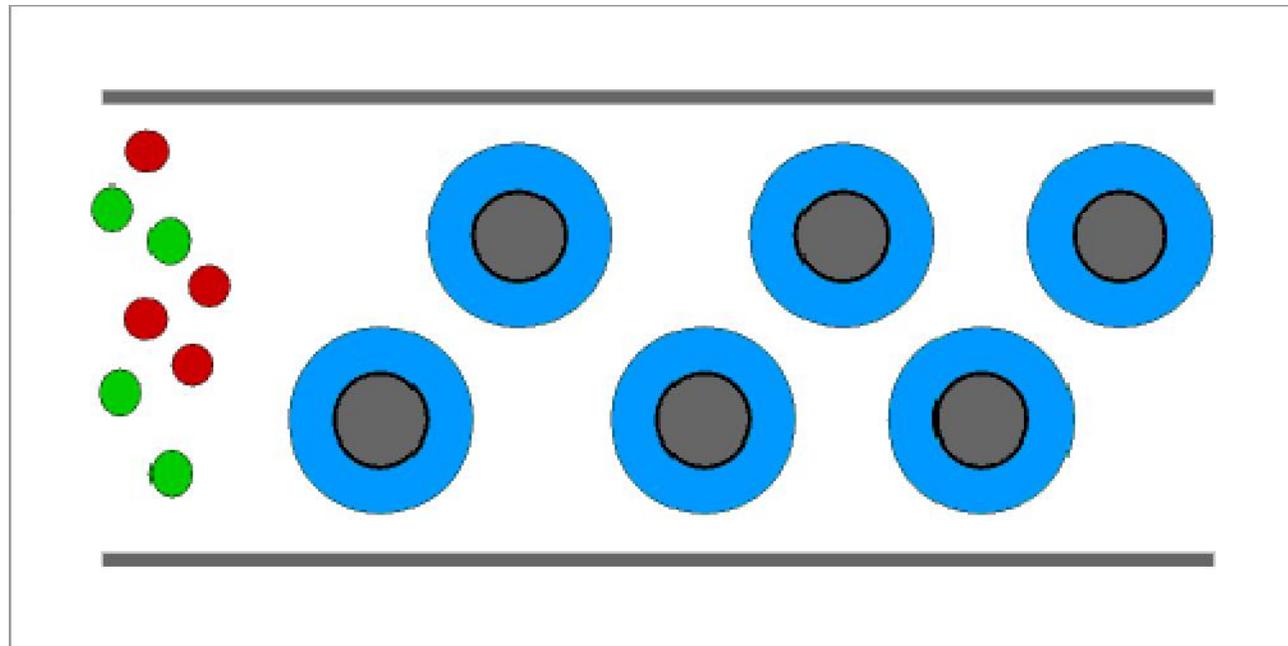
Concentration of Extract (i) By using Rotary Evaporator

(ii) By using gentle stream of nitrogen

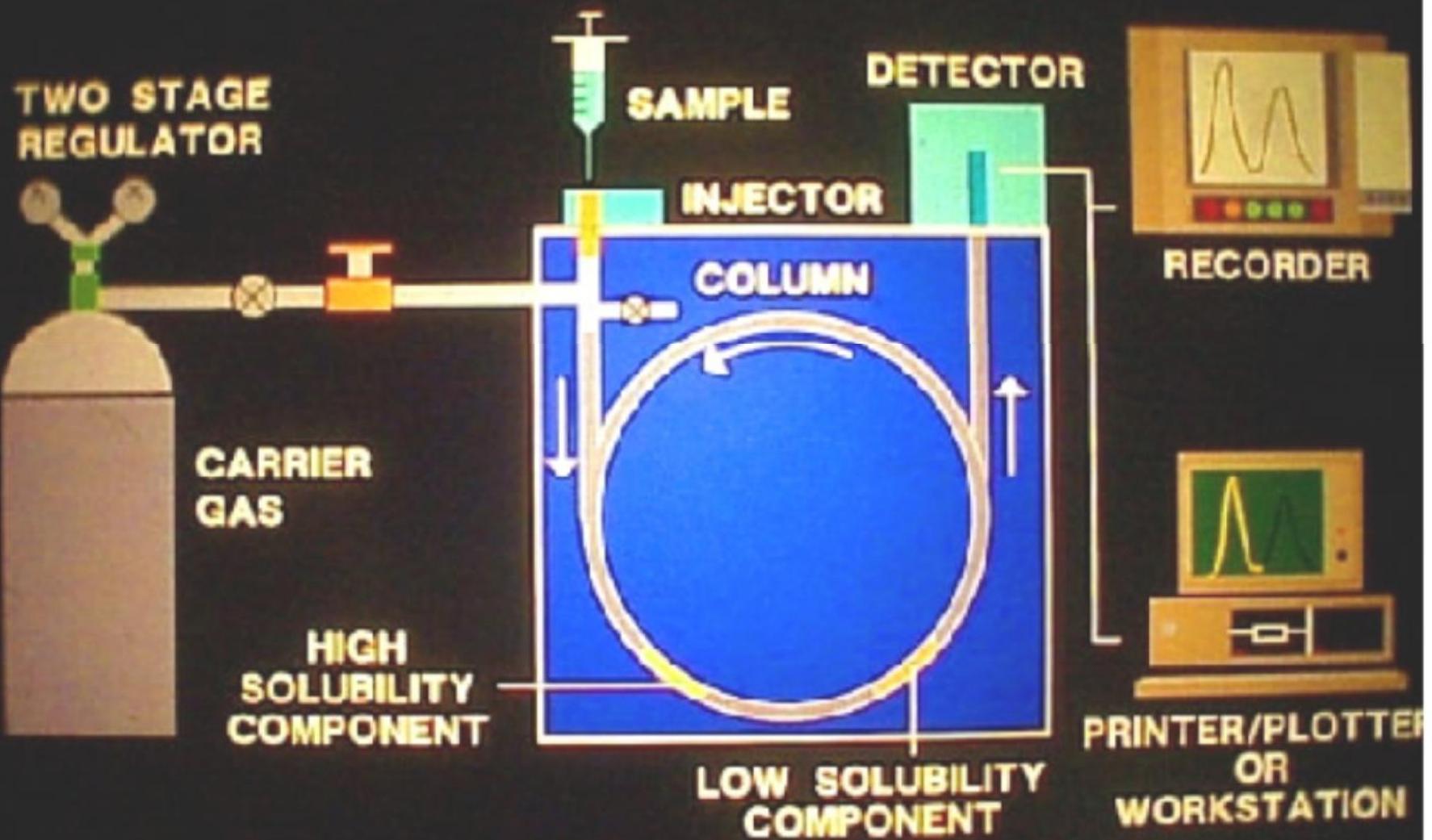




In the animation below the **red** molecules are more soluble in the **liquid** (or less volatile) than are the **green** molecules.

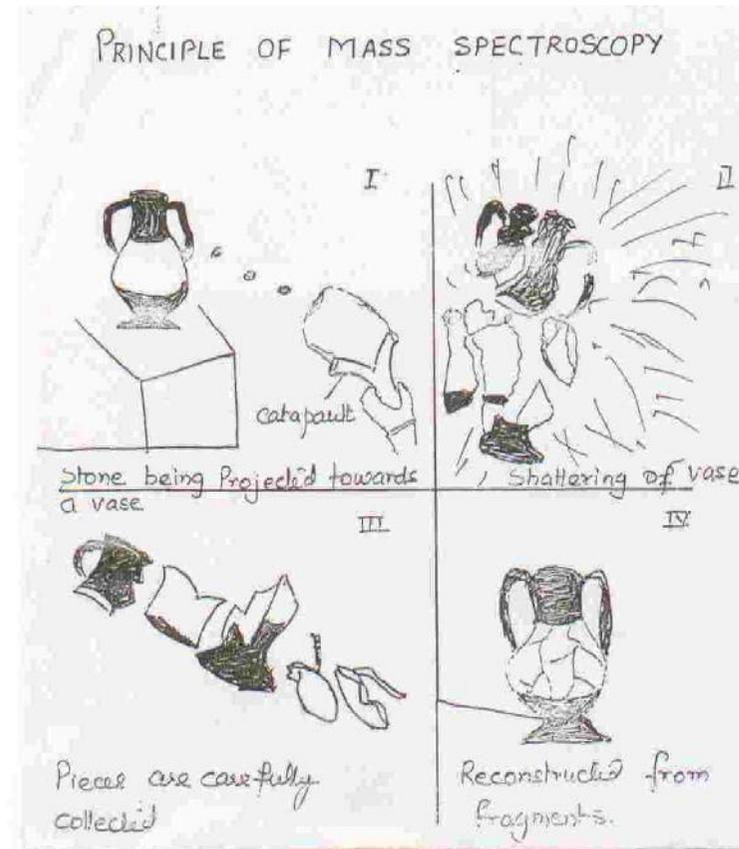
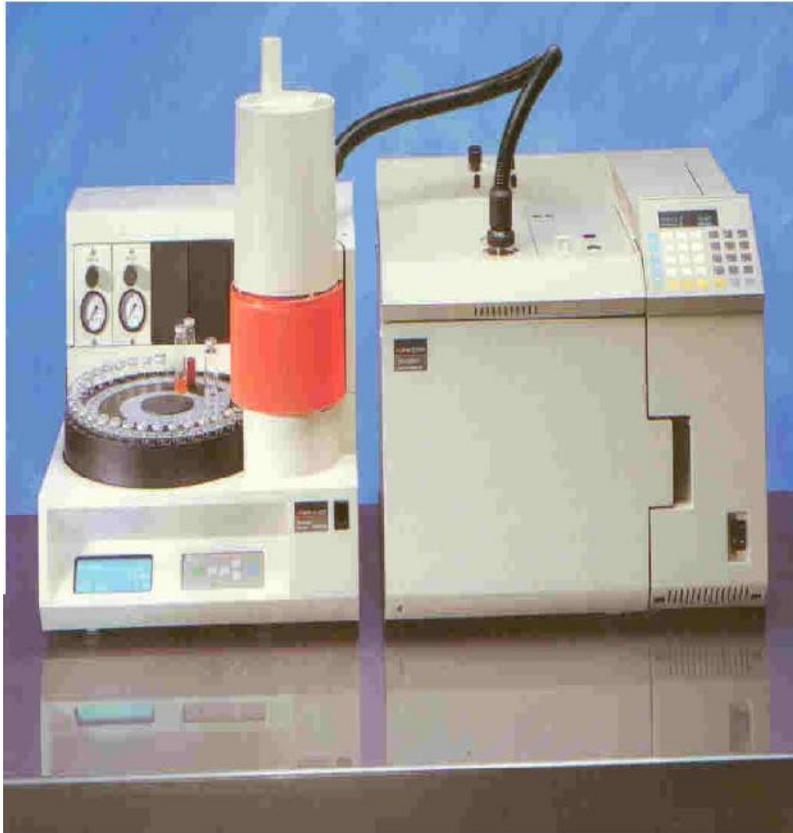


GC ANALYSIS



Detectors

Detector	Type	Support gases	Selectivity	Detectability	Dynamic range
Flame ionization (FID)	Mass flow	Hydrogen and air	Most organic cpds.	100 pg	10^7
Thermal conductivity (TCD)	Concentration	Reference	Universal	1 ng	10^7
Electron capture (ECD)	Concentration	Make-up	Halides, nitrates, nitriles, peroxides, anhydrides, organometallics	50 fg	10^5
Nitrogen-phosphorus	Mass flow	Hydrogen and air	Nitrogen, phosphorus	10 pg	10^6
Flame photometric (FPD)	Mass flow	Hydrogen and air possibly oxygen	Sulphur, phosphorus, tin, boron, arsenic, germanium, selenium, chromium	100 pg	10^3
Photo-ionization (PID)	Concentration	Make-up	Aliphatics, aromatics, ketones, esters, aldehydes, amines, heterocyclics, organosulphurs, some organometallics	2 pg	10^7
Hall electrolytic conductivity	Mass flow	Hydrogen, oxygen	Halide, nitrogen, nitrosamine, sulphur		



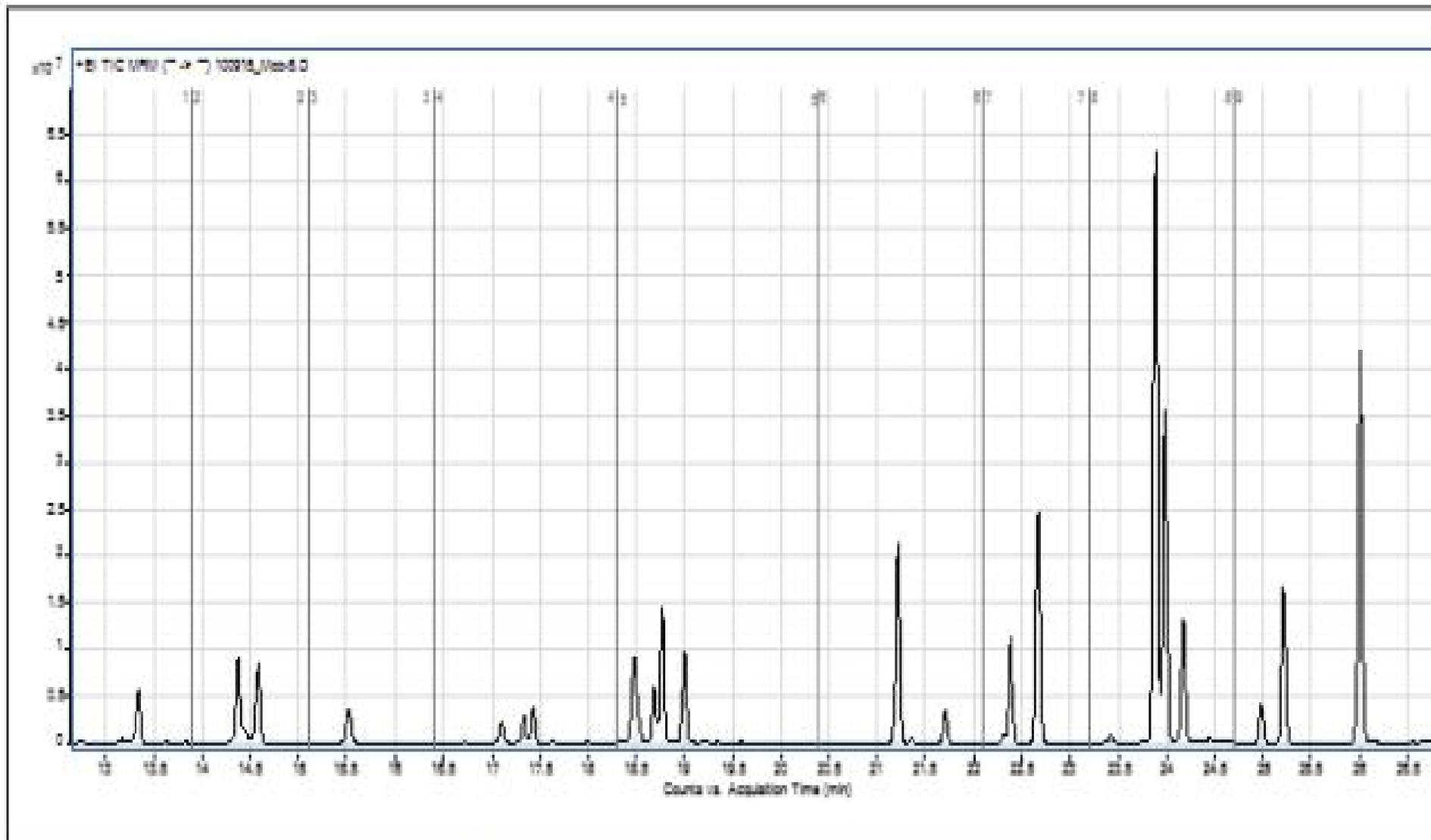


Figure 1. Extracted pesticides standard TIC.

Clip slide



GAS CHROMATOGRAPHY- MASS SPECTROMETRY (GC-MS)

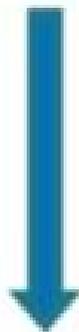
Gas chromatography



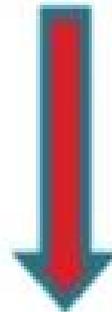
Mass spectrometry



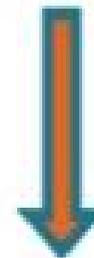
GC-MS



Separates mixture of components into individual



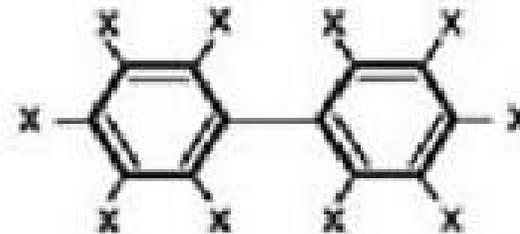
Identifies molecules based on their mass



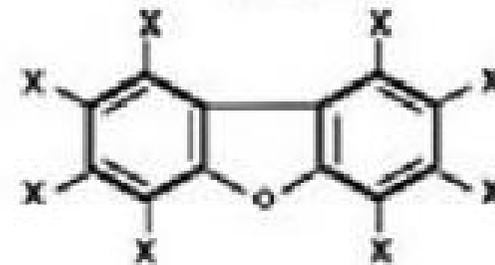
A chemical analysis technique combining two instruments to provide for powerful separation & identification.

PCBs

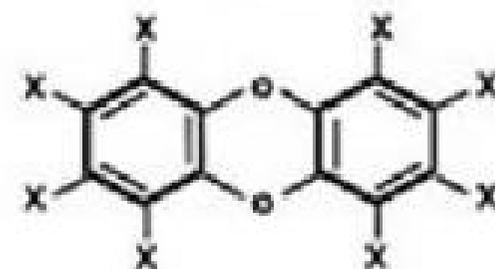
- No known natural sources
- Belong to class of halogenated aromatic hydrocarbons
- Family of chemicals with biphenyl nucleus and varying number of chlorine atoms (1 – 10)
- 209 different congeners – chemical and toxicologic properties vary
- Toxicity varies with degree of chlorination and position of chlorine atoms



POLYCHLORINATED BIPHENYLS
(PCBs)



POLYCHLORINATED DIBENZOFURANS
(Furans)



POLYCHLORINATED DIBENZODIOXINS
(Dioxins)

X = Chlorine or Hydrogen

Toxic Effects Noted

- PCBs
 - Wide ranging and varying with species!
 - Endocrine disruption (Brouwer et al. 1989; Subramanian et al. 1987; Boon et al. 1992)
 - Immune suppression (Lahvis et al. 1995; Jepson et al. 1999)
 - Reproductive failure (Reijnders 1986)

Table 1. The 51 Compounds Analyzed Using the GC/MS/MS Method

1,2,4-Trichlorobenzene	p,p'-DDE	PCB 28
Hexachlorobutadiene	Dieldrin	PCB 52
Dichlobenil	o,p'-TDE	PCB 101
alpha-HCH	Endrin	PCB 118
beta-HCH	beta-Endosulphan	PCB 153
Hexachlorobenzene	p,p'-TDE	PCB 138
gamma-HCH	o,p'-DDT	PCB 180
delta-HCH	p,p'-DDT	Cyfluthrin
Chlorothalonil	Methoxychlor	Cypermethrin
Heptachlor	Captan	Fenvalerate
Aldrin	EPTC	Deltamethrin
Isodrin	Tecnazene	Phorate
cis-Heptachlor Epoxide	Trifluralin	Tri-allate
trans-Heptachlor Epoxide	Disulphoton	Chlorpyritos-methyl
o,p'-DDE	Fenitrothion	Parathion-ethyl
alpha-Chlordane	cis-Permethrin	Chlorpyrifos-ethyl
alpha-Endosulphan	trans-Permethrin	Carbophenothion

**2016
GCMS-TQ8050**



**S/N \geq 40,000
IDL \leq 0.5 fg**

**2014
GCMS-TQ8040**



**S/N \geq 8,000
IDL \leq 4 fg**

**2012
GCMS-TQ8030**



**S/N \geq 5,000
IDL \leq 6 fg**

INTRODUCTION



Polycyclic aromatic hydrocarbons (PAHs) are widely distributed and relocated in the environment as a result of the incomplete combustion of organic matter. Many PAHs and their epoxides are highly toxic, mutagenic and/or carcinogenic to microorganisms as well as to higher systems including humans. Although various physicochemical methods have been used to remove these compounds from our environment, they have many limitations.



Trihalomethane

Trihalomethanes (THM) are a group of four chemicals that are formed along with other disinfection by products when chlorine or other disinfectants used to control microbial contaminants in **drinking water** react with naturally occurring organic and inorganic matter in **water**.

Chlorination



- the process of adding chlorine (Cl_2) or hypochlorite to water. This method is used to kill certain bacteria and other microbes in tap water as chlorine is highly toxic. In particular, chlorination is used to prevent the spread of waterborne diseases such as cholera, dysentery, and typhoid.

Chlorination



1- The process is economical and cheap .

2- It is harmless to human beings

3- It is reliable and effective .

4- Residual chlorine can be maintained in the water.

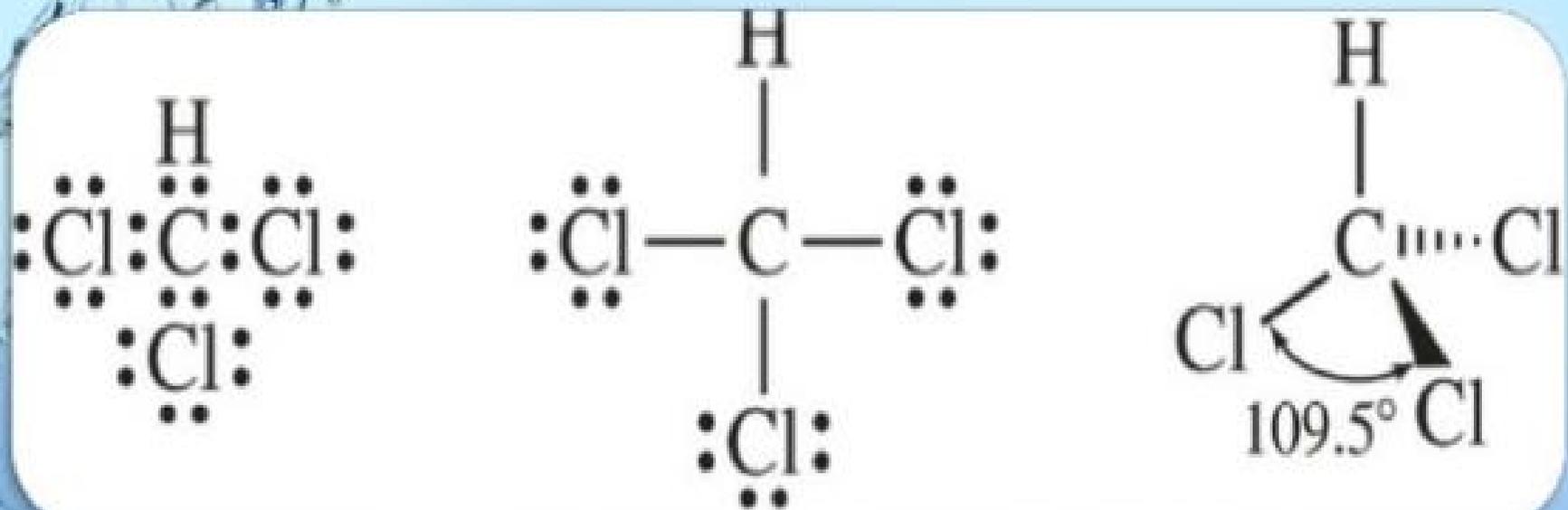
5- Easy to apply and ease to measure .

The THMs forming

When chlorine is added to water with organic material, such as algae, river weeds, and decaying leaves, THMs are formed. Residual chlorine molecules react with this harmless organic material to form a group of chlorinated chemical compounds, THMs. They are tasteless and odorless, but harmful and potentially toxic. The quantity of byproducts formed is determined by several factors, such as the amount and type of organic material present in water, temperature, pH, chlorine dosage, contact time available for chlorine, and bromide concentration in the water.

Trihalomethanes (THMs)

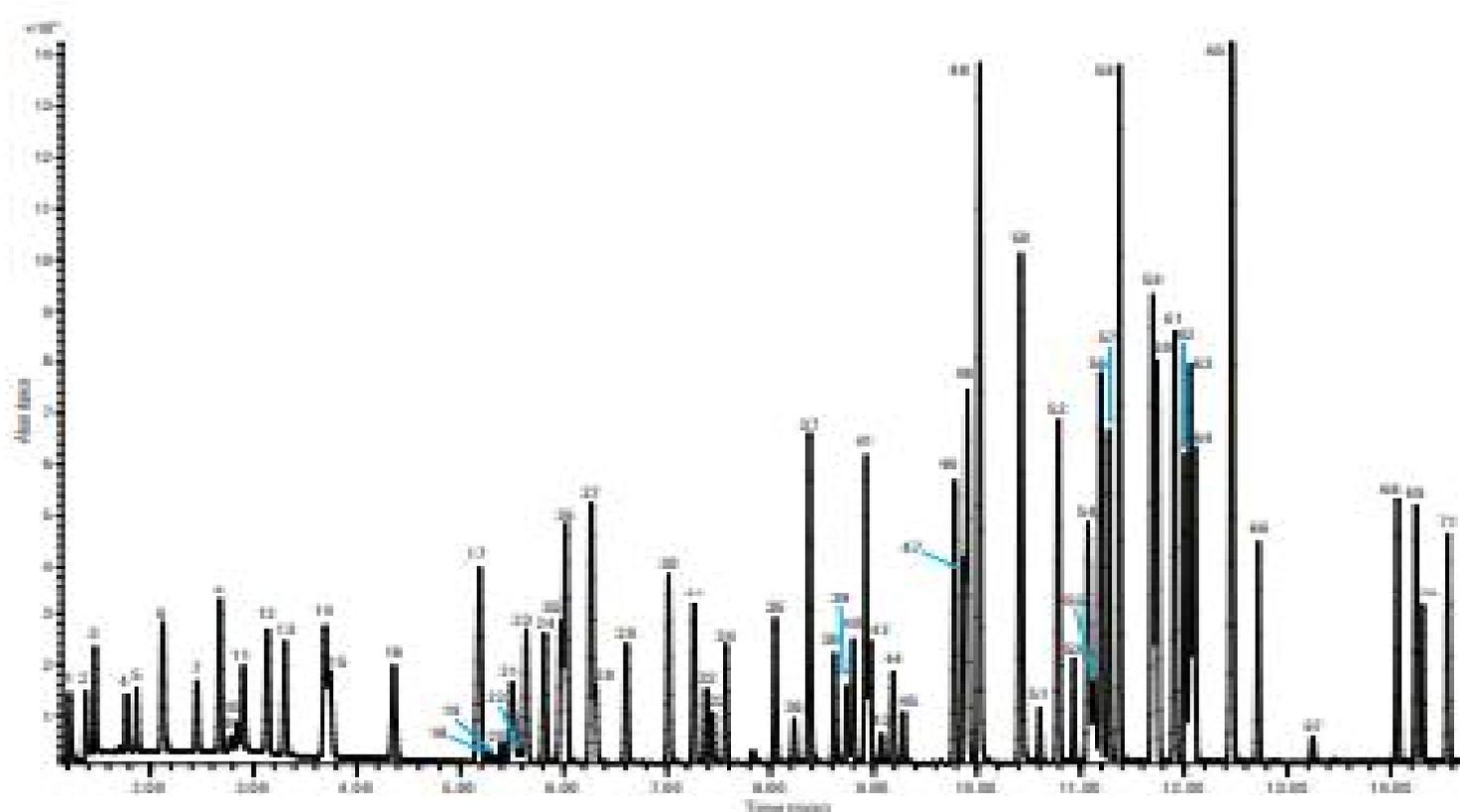
THMs are a class of chemical compounds derived from methane (CH_4) in which 3 of the 4 H atoms have been replaced by halogens Possibilities in drinking water include CHCl_2Br , CHClBr_2 and CHBr_3 . But the most prevalent is chloroform, CHCl_3 .



The Health Effects of THMs

	Compound	Adverse effects
THMS	CHCl ₃	Cancer, liver, kidney and adversely affect the process of reproduction.
	CHClBr ₂	The kidney affects the nervous system, liver, kidney and reproduction.
	CHCl ₂ Br	Cancer, liver, kidney and affects the process of reproduction
	CHBr ₂	Cancer, nervous system, liver, kidney





- 1. Dichlorodifluoromethane
- 2. Chloromethane
- 3. Vinyl chloride
- 4. Bromomethane
- 5. Chloroethane
- 6. Trichlorofluoromethane
- 7. Diethyl ether
- 8. 1,1-Dichloroethane
- 9. Acetone
- 10. Iodomethane
- 11. Carbon disulfide
- 12. Allyl chloride
- 13. Methylene chloride
- 14. Acrylonitrile
- 15. trans-1,2-dichloroethane
- 16. Methyl-tert-butyl ether (MTBE)
- 16. 1,1-Dichloroethane
- 17. 2,2-Dichloropropane
- 18. cis-1,2-Dichloroethane
- 19. 2-Butanone (MEK)
- 20. Propionitrile

- 20. Methylacrylate
- 21. Bromochloromethane
- 22. Methylacrylonitrile
- 22. TBAF
- 23. Chloroform
- 24. 1,1,1-Trichloroethane
- 25. 1-Chlorobutane
- 26. Carbon tetrachloride
- 27. 1,1-Dichloro-1-propene
- 27. Benzene
- 28. 1,2-Dichloroethane
- 29. Fluorobenzene
- 30. Trichloroethene
- 31. 1,2-Dichloropropane
- 32. Dibromomethane
- 33. Methylmethacrylate
- 34. Bromodichloromethane
- 35. cis-1,2-Dichloropropane
- 36. 1,1-Dichloropropane
- 37. Nitropropane
- 38. 4-methyl-3-pentanone (MIBK)

- 37. Toluene
- 38. trans-1,3-Dichloropropane
- 39. Ethylmethacrylate
- 40. 1,1,2-Trichloroethane
- 41. Tetrachloroethane
- 42. 1,2-Dichloropropane
- 43. 2-Hexanone
- 44. Dibromochloromethane
- 45. 1,2-Dibromomethane
- 46. Chlorobenzene
- 47. 1,1,1,3-Tetrachloroethane
- 48. Ethylbenzene
- 48. m-p-Xylene
- 49. o-Xylene, Styrene
- 51. Bromoform
- 52. Isopropylbenzene
- 53. Bromofluorobenzene
- 54. Bromobenzene
- 55. 1,1,1,2-Tetrachloroethane
- 56. 1,2,2-Trichloropropane
- 57. trans-1,4-Dichloro-2-butene

- 56. n-Propylbenzene
- 57. 2-Chlorotoluene
- 58. 1,2,5-Trimethylbenzene
- 59. 4-Chlorotoluene
- 60. tert-Butylbenzene
- 61. 1,2,4-Trimethylbenzene
- 61. sec-Butylbenzene
- 62. 1,3-Dichlorobenzene
- 63. p-tolylchloride
- 64. 1,4-Dichlorobenzene
- 65. 1,2-Dichlorobenzene-d4
- 66. 1,3-Dichlorobenzene
- 67. n-Butylbenzene
- 68. Hexachloroethane
- 69. 1,2-Dibromo-3-chloropropane (DBCP)
- 70. 1,2,4-Trichlorobenzene
- 71. Hexachlorocyclopentadiene
- 72. Naphthalene
- 73. 1,2,3-Trichlorobenzene

Radiological aspects:

Radioactivity should be as low as possible

Guideline values-

Gross alpha activity-0.5 Bq /L

Gross beta activity- 1.0 Bq /L

1Bq= 1 disintegration per second



Effects of radioactivity on water



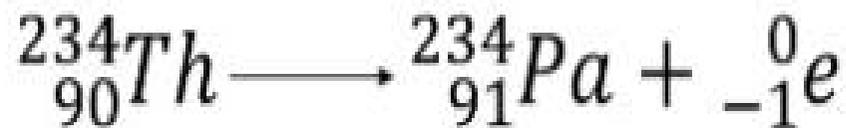
Risks



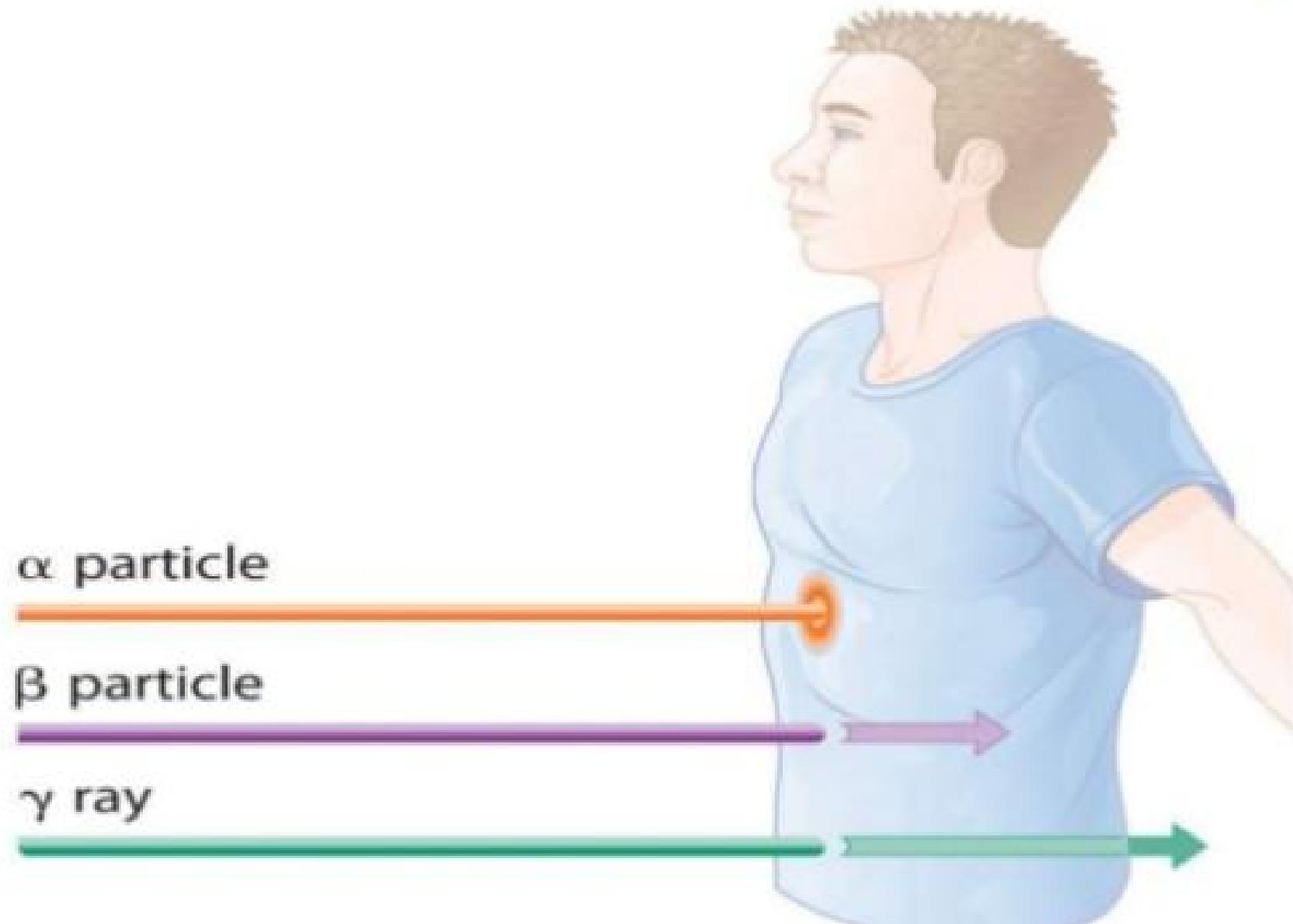
- **Damage to tissues**
- **Cells**
- **DNA and other vital molecules**
- **Cancer**
- **birth defects**
- **Abnormalities**
- **Death**

Natural radioactivity

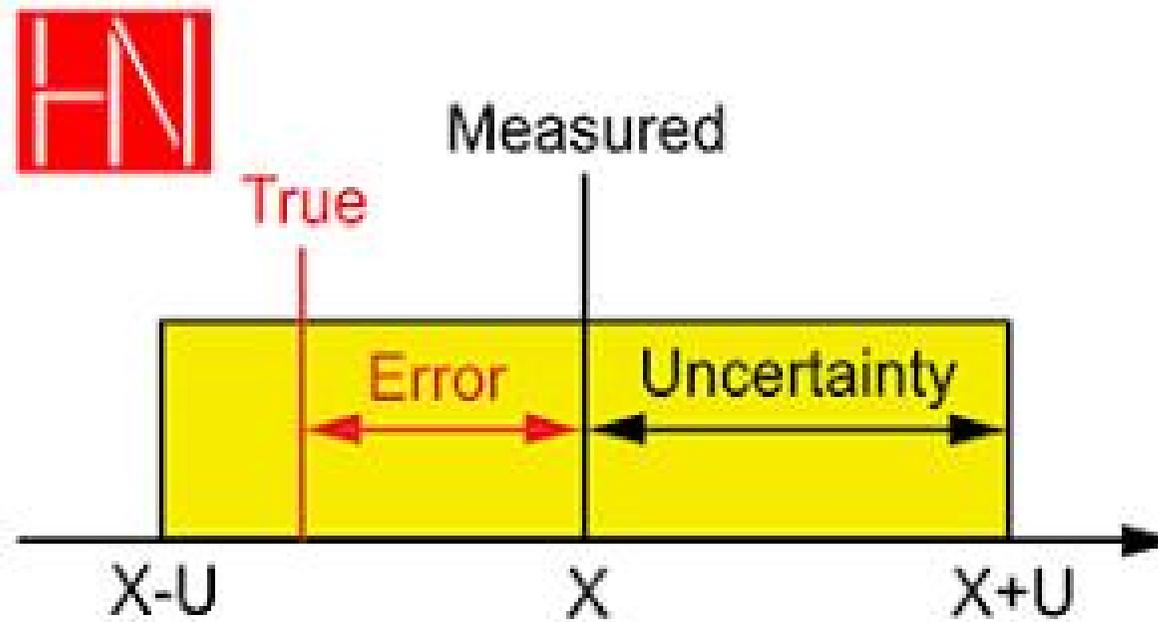
- The element whose nuclei spontaneously disintegrate are called *radioactive element*.
- Example of natural radioactivity are:-



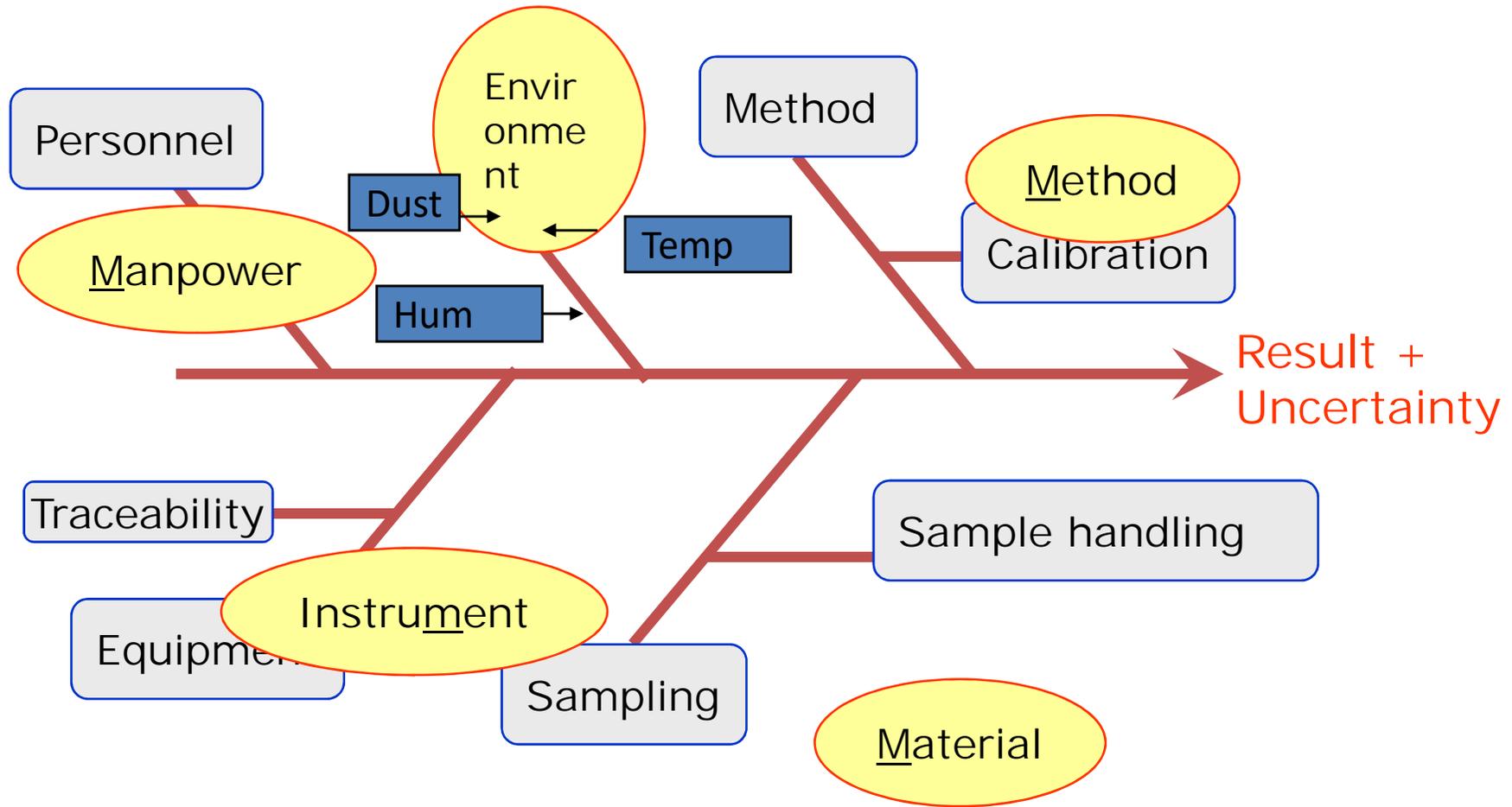
Radiation Penetration Ability



Evaluation of measurement uncertainty



Sources of uncertainty in analysis



ii) MICROBIOLOGICAL ASPECTS

- ❖ Bacteriological indicators
- ❖ Virological aspects
- ❖ Biological aspects



Bacteriological quality of drinking water

ORGANISMS	GUIDELINE VALUE
All water intended for drinking	Must not be detectable in any 100ml sample
Treated water entering distribution system (E.coli ,total coliform count)	Must not be detectable in any 100ml sample
Treated water entering distribution system (E.coli ,total coliform count)	Must not be detectable in any 100ml sample In c/o large supplies, must not be present in 95% of samples taken throughout any 12month period

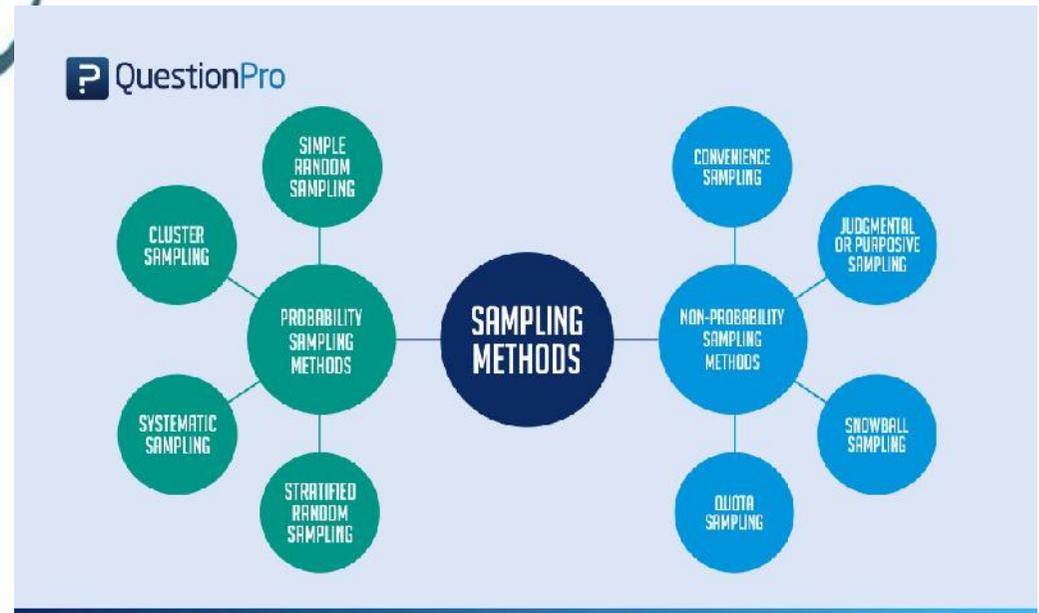
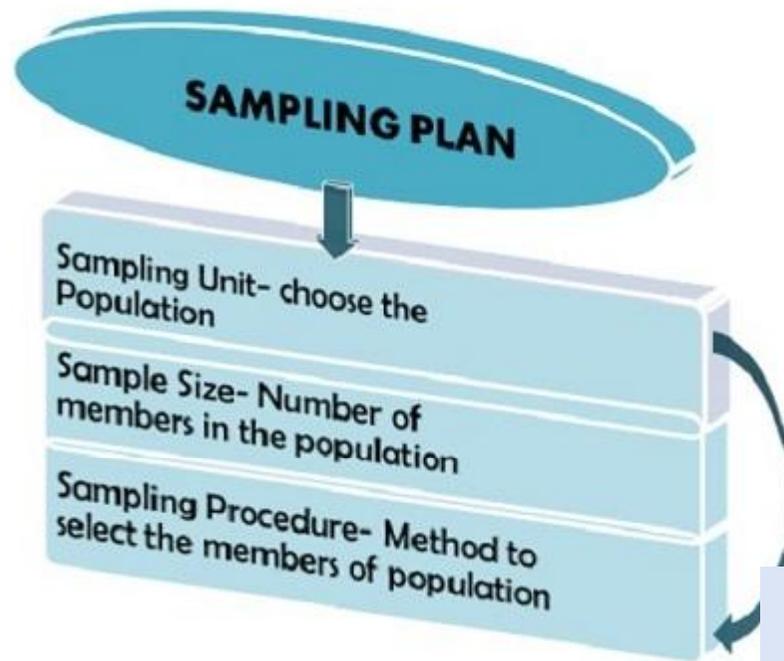
6.Sampling



**Systematic
Sampling**



Sampling plan & methods



Laboratory involved in sampling must have:

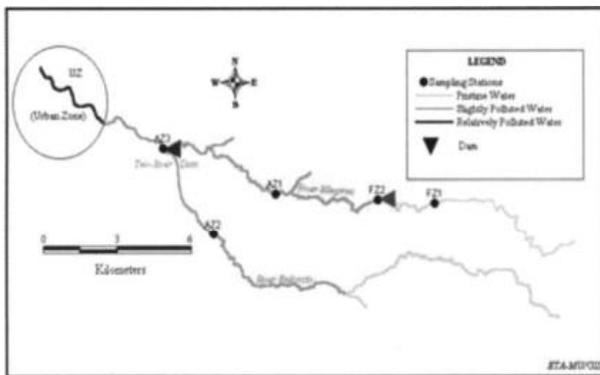
- ❖ Sampling Plan.
- ❖ SOP for sampling.
- ❖ Work instruction for sampling.
- ❖ Field data sheets.
- ❖ Necessary PPE.
- ❖ Sampling tools.
- ❖ Specific pre washed Containers.
- ❖ Preservatives.
- ❖ Equipments and chemical for on site testing.
- ❖ Sample storage facility.
- ❖ Vehicle for transport of samples to the laboratory.

sampling method

a) selection of samples or sites

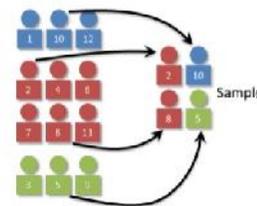
b) sampling plan

c) preparation & treatment of samples

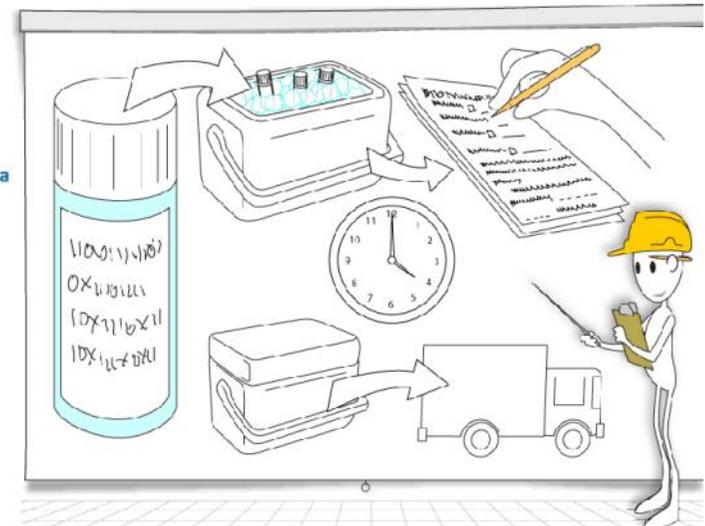


Stratified Sampling

- Population with some distinct categories can be organized into separate "stratum" which can be sampled as an independent sub-population, out of which individual elements can be randomly selected.



LightCastle Data



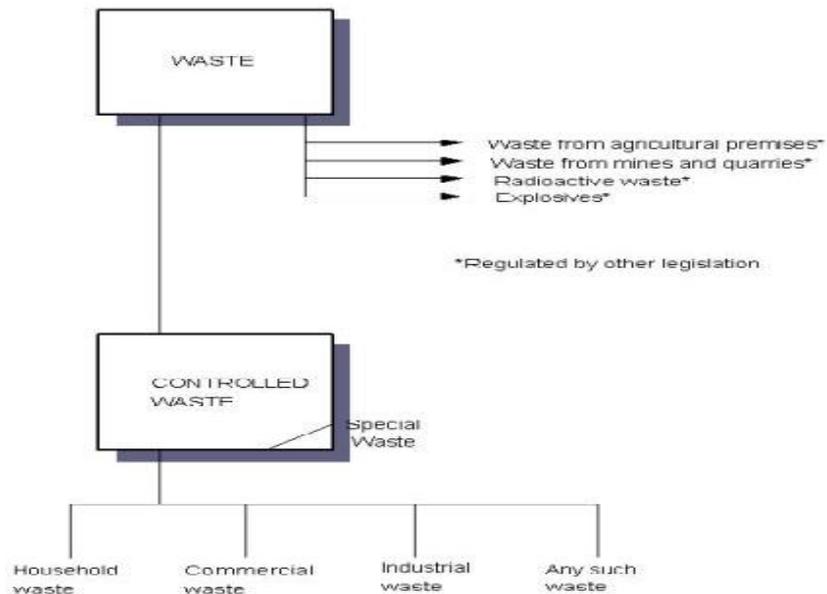
The laboratory shall retain records of sampling data that forms part of the testing or calibration that is undertaken. These records shall include, where relevant:

a	reference to the sampling method used;
b	date and time of sampling;
c	data to identify and describe the sample (e.g. number, amount, name);
d	identification of the personnel performing sampling;
e	identification of the equipment used;
f	environmental or transport conditions;
g	diagrams or other equivalent means to identify the sampling location, when appropriate;
h	deviations, additions to or exclusions from the sampling method and sampling plan.

7. Handling of Test Items

The laboratory shall have a procedure for the transportation, receipt, handling, protection, storage, retention, and disposal or return of test or calibration items, including all provisions necessary to protect the integrity of the test or calibration item,

Types of waste. A detailed explanation of the definition of waste can be found in Annex 2 of The Scottish Office Environment Department Circular 10/94.



Source: The Scottish Office Statistical Bulletin, Waste Collection, Disposal and Regulation Statistics 1993
ISBN 0-7480-4936-3



8. Ensuring the validity of results



Reliable
Not Valid



Low Validity
Low Reliability



Not Reliable
Not Valid



Both Reliable
and Valid

by Experiment-Resources.com



Everyone makes mistakes!!



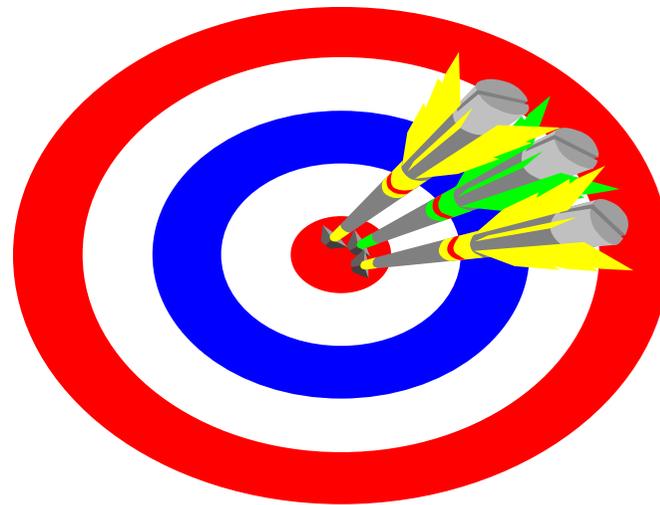
WHAT IS QUALITY CONTROL?

quality control

*a process that helps a company
make sure it creates quality
products and that staff and
management
alike make minimal mistakes*

Quality ?

- Consistency
 - Accuracy
 - Precision
- Right result
 - First time
 - Every time





For getting confidence
(Repeatability, retesting,
recovery)



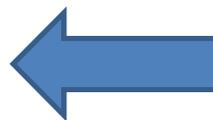
Home Examination of one
section(Intra –Laboratory
Comparison)



Examination of all sections
of a class (ILC)



CBSE Board Examination
(PT Participation)





High Accuracy
High Precision



Low Accuracy
High Precision



High Accuracy
Low Precision



Low Accuracy
Low Precision



Practice



Practice



➤ use of reference materials or quality control materials;

➤ use of alternative instrumentation that has been calibrated to provide traceable results;

➤ functional check(s) of measuring and testing equipment;

➤ use of check or working standards with control charts, where applicable;

➤ intermediate checks on measuring equipment;

➤ replicate tests or calibrations using the same or different methods;

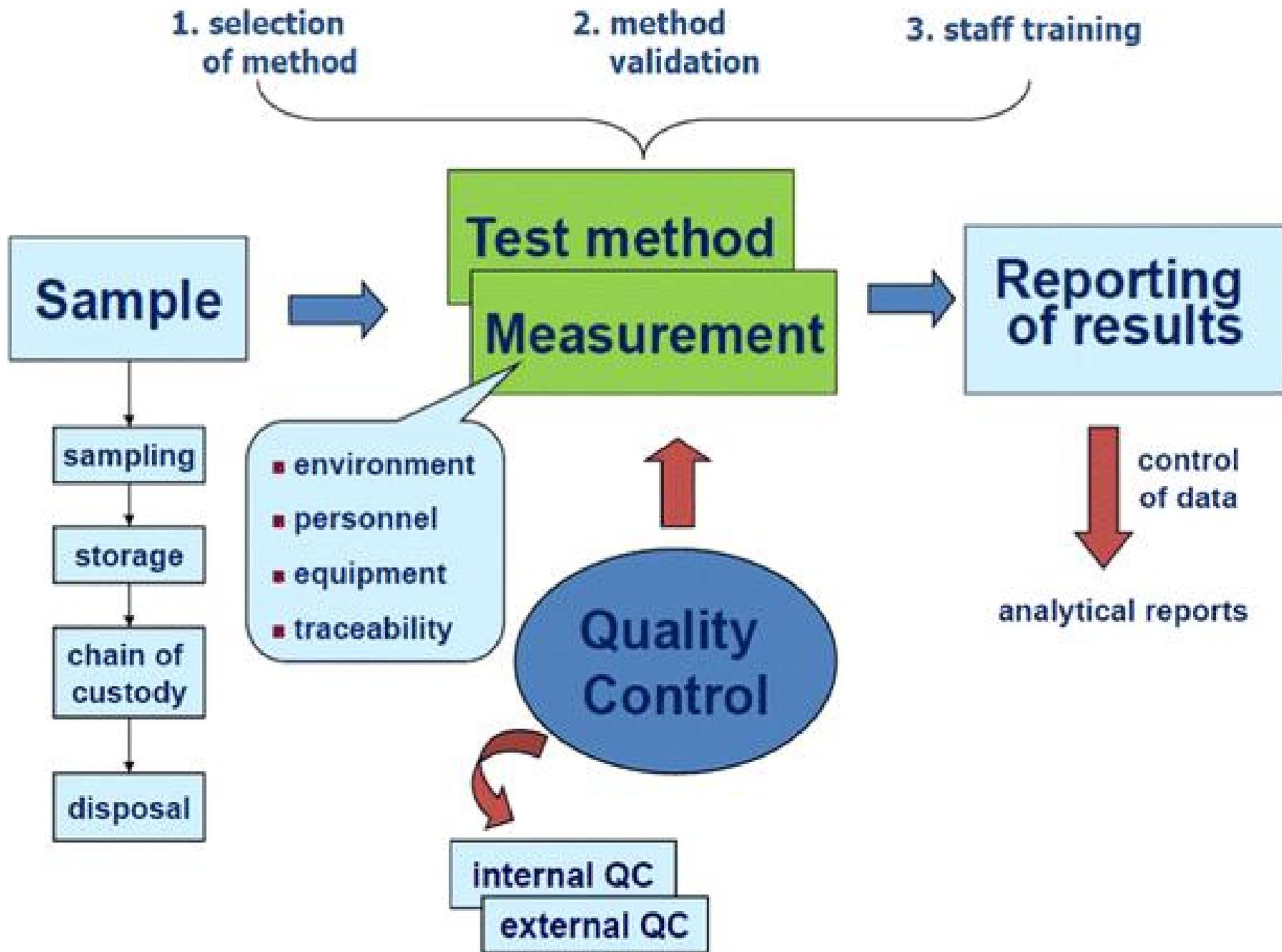
➤ retesting or recalibration of retained items;

➤ correlation of results for different characteristics of an item;

➤ review of reported results;

➤ intralaboratory comparisons;

➤ testing of blind sample(s).





Quality Assurance vs. Quality Control

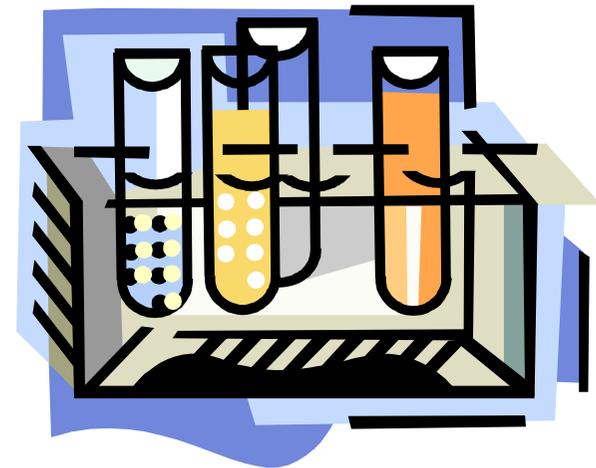
Quality Assurance

An overall management plan to guarantee the integrity of data (The “system”)

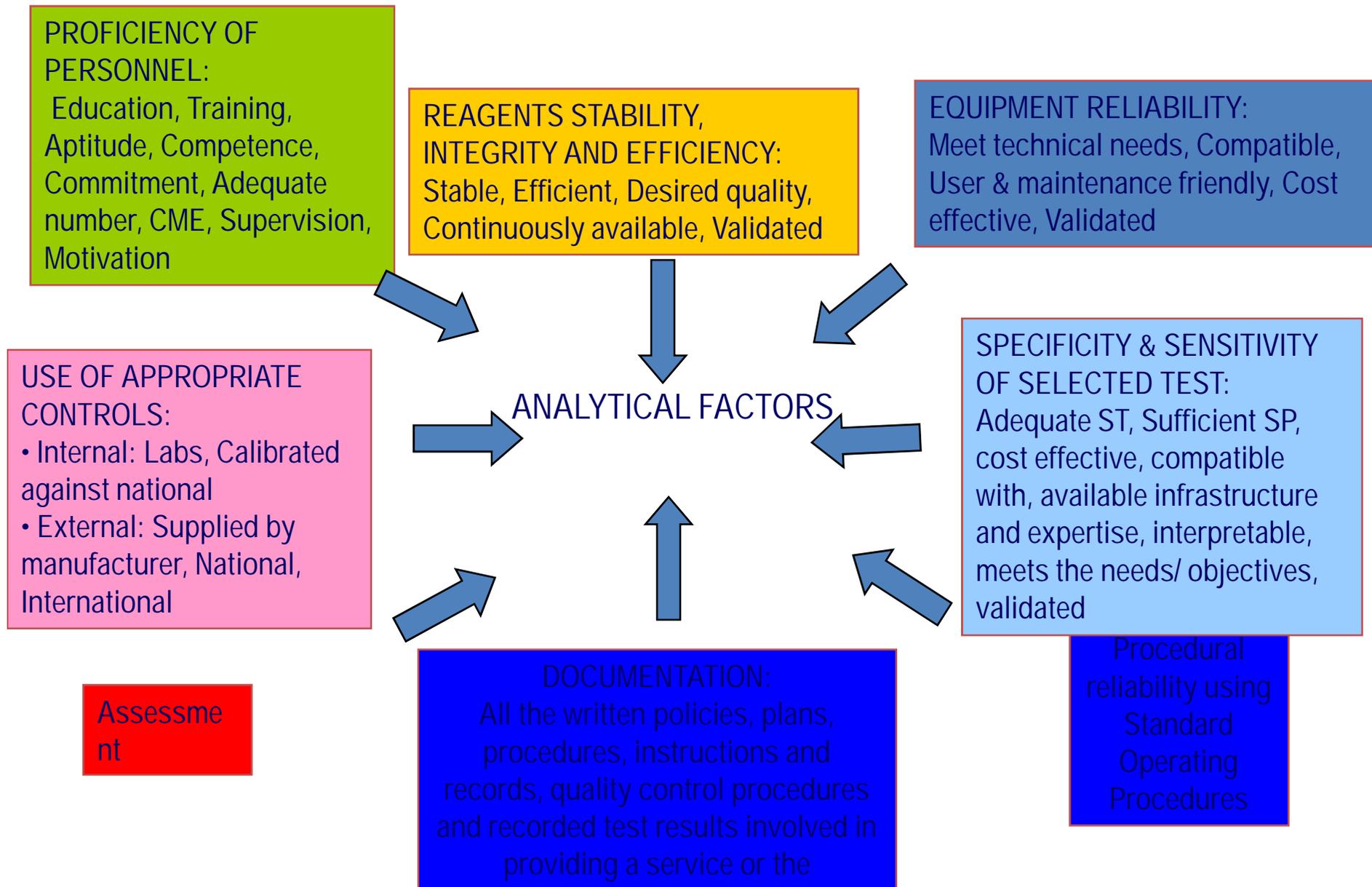


Quality Control

A series of analytical measurements used to assess the quality of the analytical data (The “tools”)



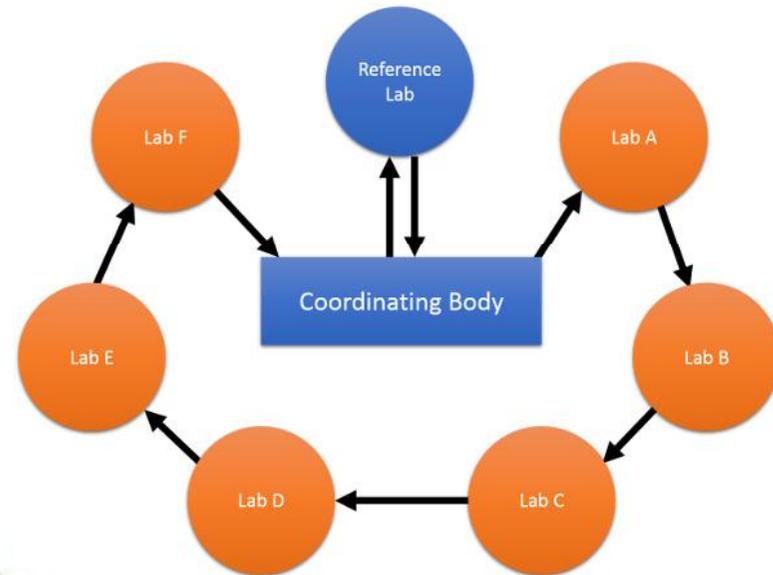
Factors influencing quality: Analytical



Monitor performance by comparison with other lab:

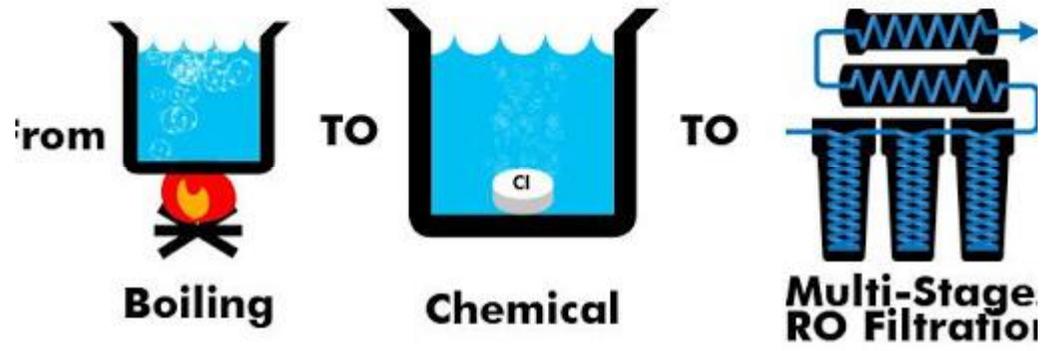
a) PT

b) Interlaboratory comparison



9. Reporting the Result





Purification on Small Scale

BOILING: for 5-10 minutes kills almost all organisms & removes temporary Hardness.

DISTILLATION: not commonly used due to higher cost, used in Labs. etc.

ADDITION OF CHEMICALS

Bleaching Powder: 5% solution is used Dose: 3-6 drops/L contact time of $\frac{1}{2}$ hour. Chlorine Tablets/Halazone Tablets. one tablet/litre.

Iodine Solution: 02 drops of 2% Soln./litre

KMnO₄: an amount that gives just pink coloration to the Water.

Alum: used for turbid water in a dose of 0.1-0.4 grains/5 litres of Water.



When sources of water are
Rivers, Streams, Lakes etc.
then water is Purified by

- Coagulation
- Sedimentation
- Filtration
- Disinfection
- Storage

When sources of water are wells, Springs, Tanks etc.

Then water is purified by the addition of

Bleaching powder/Chlorinated Lime as it is

- Cheap
- Easy to use
- Reliable and safe

. Filtration: ceramic filters –Pasteur Chamberland filter, Berkefeld filter, Katadyn filter.

Main part is candle (porcelin /infusorial earth)

In Katadyn: Surface covered with silver catalyst, bact. destroyed in contact with silver ion (oligodynamic action)

can remove bact. Not virues



1. Ultraviolet irradiation: can destroy bact. Viruses, yeast, fungi, algae, protozoa
 - Mercury vapor arc lamps emitting UV rays at a wave length of 254 nano mt
 - Water should be free from turbidity/ colloidal suspended constituents
 - Short exposure required, no foreign matter added, no taste /odour change
 - No residual effect



. Multistage Reverse osmosis purification of water:

Remove total dissolved solid, hardness, heavy metals, bacteria, viruses, protozoa, cysts.

Clarity cartridge removes suspended particles (dust/mud/sand)

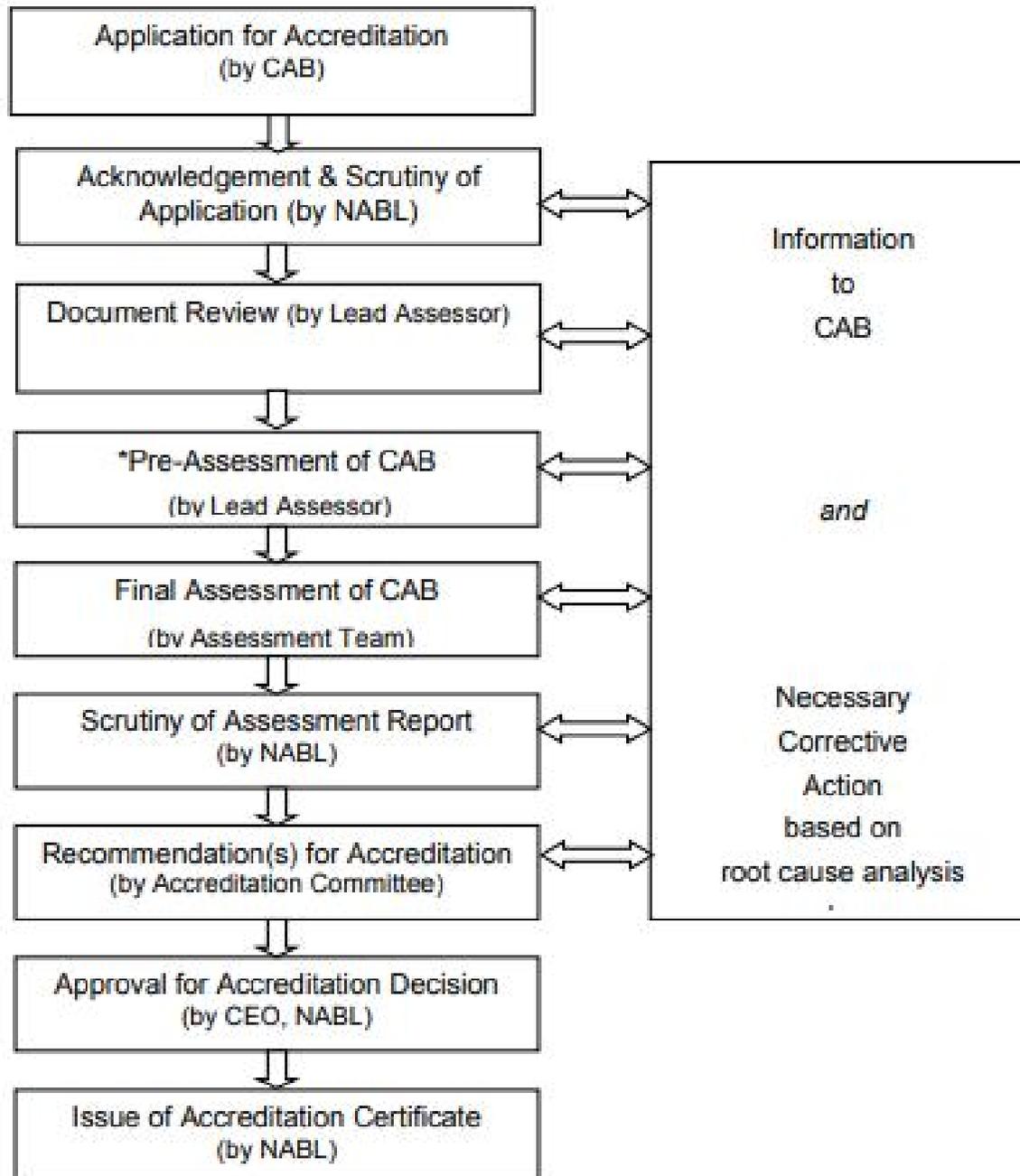
The reverse osmosis cartridge removes dissolved solid/hardness/heavy metals/micro organism.



For biological impurities like bacteria or viruses in the **water**, then an ultra violet (UV) **filter** can be used. If only chemical properties are bad — like the presence of TDS — then an **RO** is **necessary**,



ACCREDITATION PROCESS



Without a Laboratory Quality System -

too many mistakes can make analysis very costly; due to

expenses caused by wrong decisions, or



costs of analysis



- repeating analysis of samples
- investigation of problems
- revision of procedures
- loss of good reputation

Prevention is Better than cure!



'It costs less to prevent a problem than it does to correct it'

A formal quality system in the laboratory should prevent mistakes by means of:

- quality assurance measures
- quality control of the analytical results
- thorough documentation of the system
- efficient maintenance of records
- regular audits of all aspects of the system



House wife has solution of problems



Analyst has solution of chemicals





Thank you...



...



-----For your Kind attention