SNS COLLEGE OF PHARMACY AND HEALTH SCIENCES

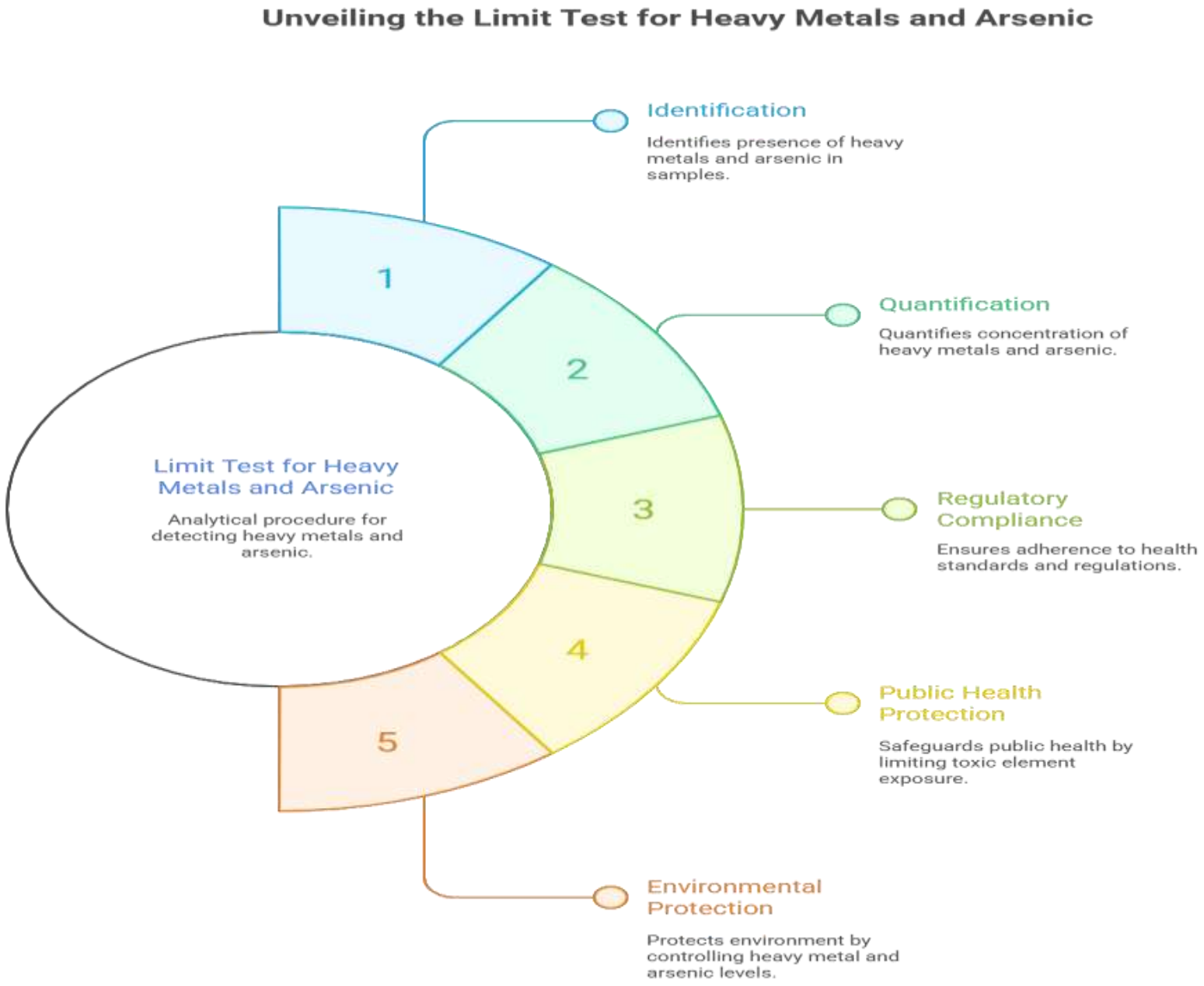
Affiliated To The Tamil Nadu Dr. MGR Medical University, Chennai
Approved by Pharmacy Council of India, New Delhi.
Coimbatore -641035

COURSE NAME:PHARMACEUTICAL CHEMISTRY

I YEAR D PHARM

TOPIC 5: LIMIT TEST FOR IRON AND HEAVY METALS

Mind Map

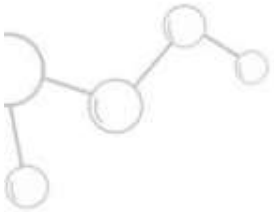

Unveiling the Limit Test for Heavy Metals and Arsenic
Limit Test for Heavy Metals and Arsenic
Analytical procedure for detecting heavy metals and arsenic.
1
2
3
4
5
Identification
Identifies presence of heavy metals and arsenic in samples.
Quantification
Quantifies concentration of heavy metals and arsenic.
Regulatory Compliance
Ensures adherence to health standards and regulations.
Public Health Protection
Safeguards public health by limiting toxic element exposure.
Environmental Protection
Protects environment by controlling heavy metal and arsenic levels.

LIMIT TEST FOR IRON

Principle

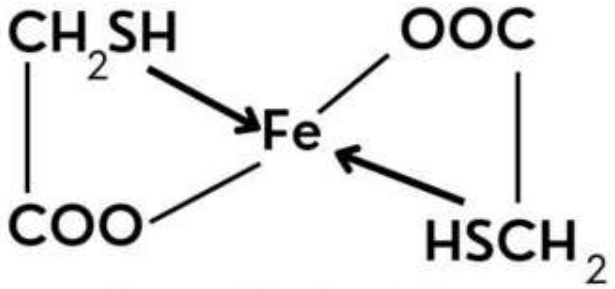
- Limit test of Iron is based on the reaction of iron in ammonical solution with thioglycollic acid in presence of citric acid to form iron thioglycolate which is pale pink to deep reddish purple in color.

LIMIT TEST FOR IRON (REACTIONS)

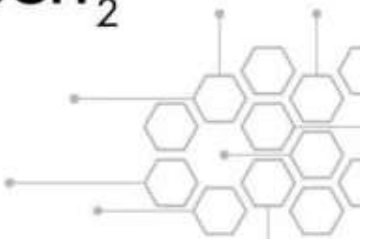


$$2\,Fe^{3+} + HSCH_2COOH \longrightarrow 2\,Fe^{2+} + Fe(HSCH_2COO)_2 + 2H^+$$

$$2\,Fe^{2+} + HSCH_2COOH \longrightarrow$$



Ferrous Thioglycolate Complex



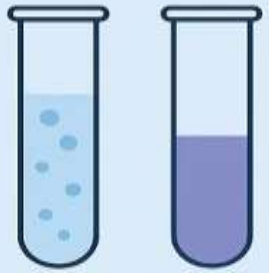
LIMIT TEST OF IRON

DEFINITION

A qualitative test that detects the presence of trace amounts of iron.

THEORY

Iron impurities react with thioglycolic acid in an acidic medium to form a purple-colored complex.



Fe^{3+} Ferrous thioglycolate

$$Fe^{3+} + \text{Thioglycolic acid} \rightarrow \text{Ferrous thioglycolate (purple complex)}$$

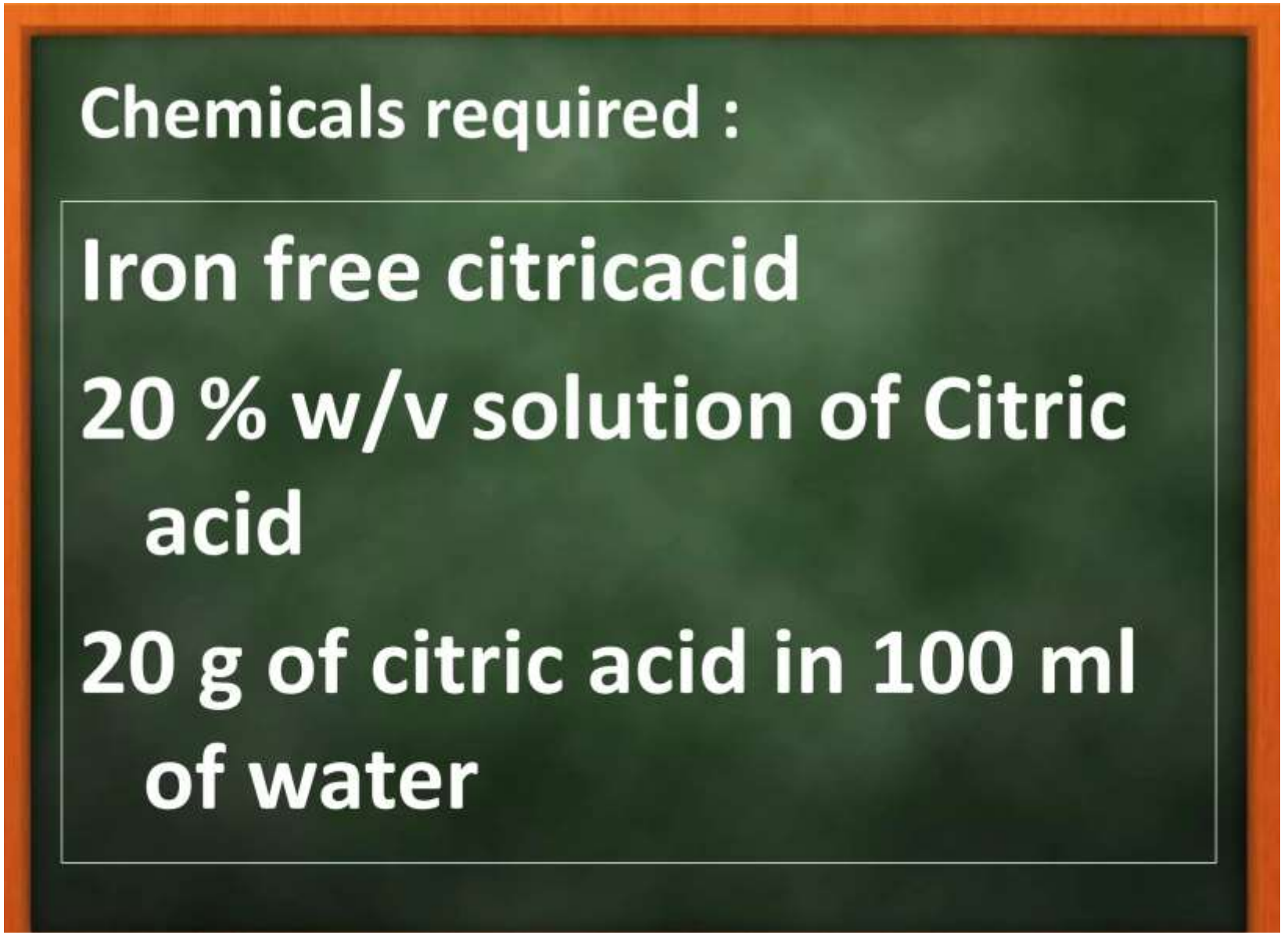
REAGENTS

- Citric acid
- Thioglycolic acid
- Ammonia solution
- Iron standard solution

APPLICATION

Used in pharmaceutical chemistry to ensure that raw materials and finished products are free from iron impurities.

Standard	Test
Pipette out 2 ml of standard Iron solution into the Nessler's cylinder marked as standard	Dissolve the specified quantity of given sample in distilled water in Nessler's cylinder marked as test
Dilute it to 40 ml with distilled water	Dilute it to 40 ml with distilled water
Add 2 ml of 20 % w/v solution of iron free citric acid	Add 2 ml of 20 % w/v solution of iron free citric acid
Add 0.1ml of thioglycolic acid.	Add 0.1ml of thioglycolic acid.
Mix and make alkaline with iron free ammonia solution and dilute to 50ml with water	Mix and make alkaline with iron free ammonia solution and dilute to 50ml with water
Immediately stir with a glass rod and allow it to stand for five minutes	Immediately stir with a glass rod and allow it to stand for five minutes


Chemicals required :
Iron free citricacid
20 % w/v solution of Citric acid
20 g of citric acid in 100 ml of water

4. Limit Test for Heavy Metals

❖PRINCIPLE

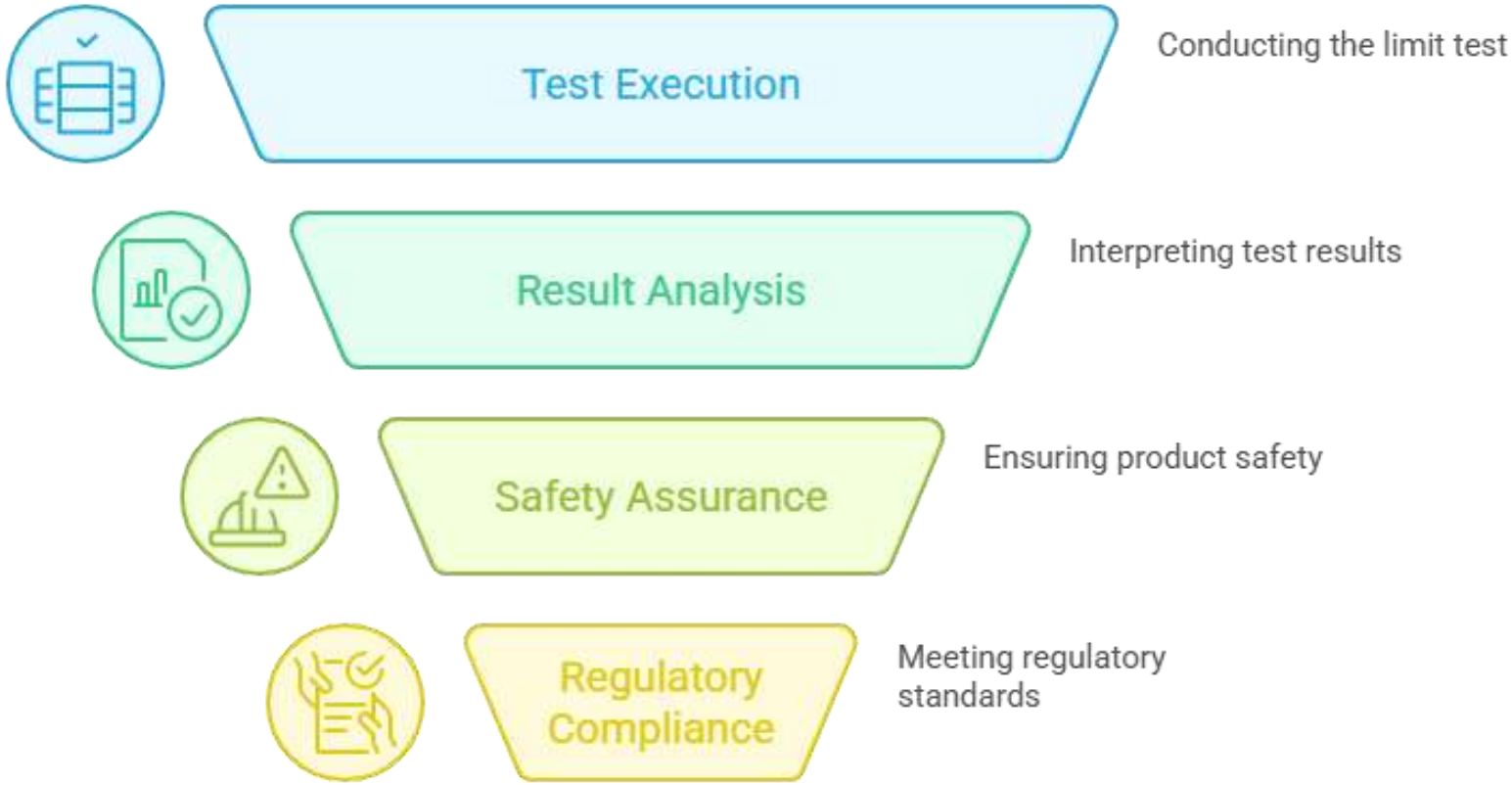
- All Metals like **Copper, Bismuth, Lead, Mercury, Arsenic, Antimony, Silver** etc (except alkali metals and alkaline earth metals) are coloured by sulphide ions (**H_2S or Na_2S**) under specified conditions.
- The limit test for detecting and limiting the impurity of Heavy metals.
- The HM are precipitated as Metal sulphides by the addition of hydrogen sulphide or sodium sulphide solution.
- Depends upon the quantity of the metal the colour varies from **brown to black**.

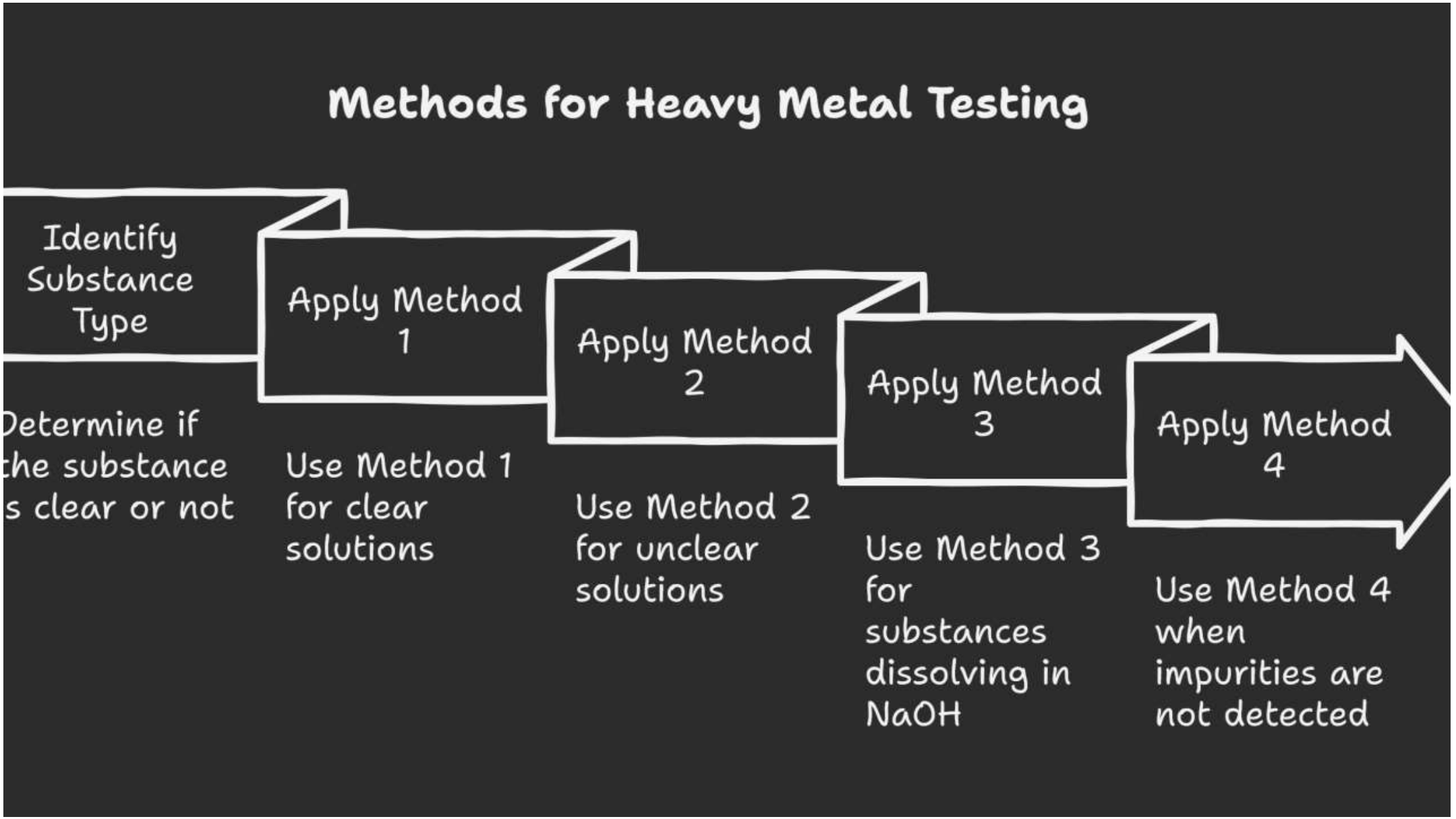
PROCEDURE

"TAKE TWO 50 ML NESSLER CYLINDER AND MARK ONE AS TEST AND OTHER AS STANDARD"

Test	Standard
Place 25ml of solution prepared for the test as directed in the individual monograph or dissolve the specified quality of the substance under examination in sufficient water to produce 25ml.	Pipette 1.0ml of lead standard solution (20 ppm Pb) and dilute with water to produce 25 ml.
Adjust PH between 3 to 4 with Dil. Acetic acid or Dil. Ammonia solution and dilute with water to produce 35ml and mix.	Adjust PH between 3 to 4 with Dil. Acetic acid or Dil. Ammonia solution and dilute with water to produce 35ml and mix.
Add 10ml of freshly prepared Hydrogen sulphide solution, mix and dilute to 50ml with water. Allow to stand for 5 minute.	Add 10ml of freshly prepared hydrogen sulphide solution,mix and dilute to 50ml with water. Allow to stand for 5 minute.

Ensuring Safety Through Limit Testing




Methods for Heavy Metal Testing
Identify Substance Type
Apply Method 1
Apply Method 2
Apply Method 3
Apply Method 4
Determine if the substance is clear or not
Use Method 1 for clear solutions
Use Method 2 for unclear solutions
Use Method 3 for substances dissolving in NaOH
Use Method 4 when impurities are not detected

$$Pb + H_2S \longrightarrow PbS\downarrow + 2H^+$$

colour (brown)

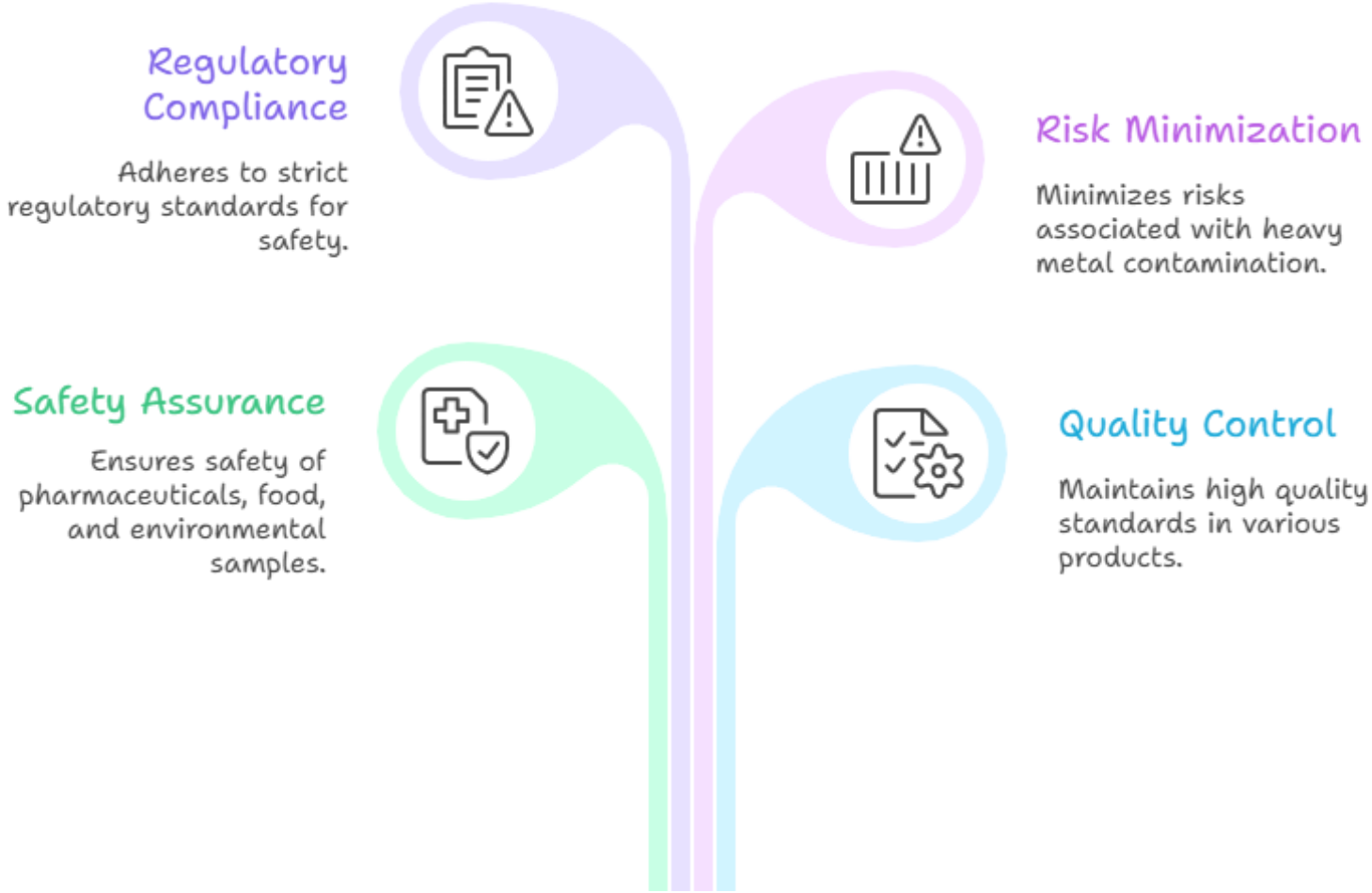

Drlogy
Heavy Metals Test
Test
Also Known As : Metal Toxicity Test
Type : Blood or Urine Test
Purpose : Detect Heavy Metal Exposure
Sample Type : Blood or Urine
Preparation : No specific prep
Fasting : Not necessary
Gender : No gender preference
Age Group : All ages
Normal Value : <25.00 µg/dL
Report Time : Few Days
Cost : 5,000 - 10,000 INR*

Heavy metals	Symbol	Maximum permissible limit (mg/kg)	References
Arsenic	As	5	(US EPA, 2015)
Barium	Ba	100	
Cadmium	Cd	1	
Chromium	Cr	5	
Lead	Pb	5	
Mercury	Hg	0.2	
Selenium	Se	1	
Silver	Ag	5	

Heavy Metals	Maximum levels (ppm)	Heavy Metals	Maximum levels (ppm)
Arsenic (As)	20	Manganese (Mn)	2000
Cadmium (Cd)	3	Lead (Pb)	50
Cobalt (Co)	50	Nickel (Ni)	100
Chromium (Cr)	100	Selenium (Se)	10
Copper (Cu)	100	Zinc (Zn)	300

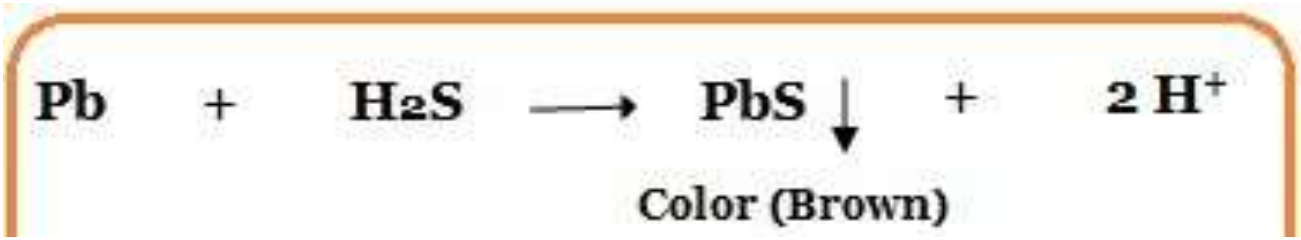
SUMMARY

Unveiling the Dimensions of Heavy Metal and Arsenic Testing



ASSESMENTS

1. Give a note on limit test for sulphate ?



2. Explain the reaction?

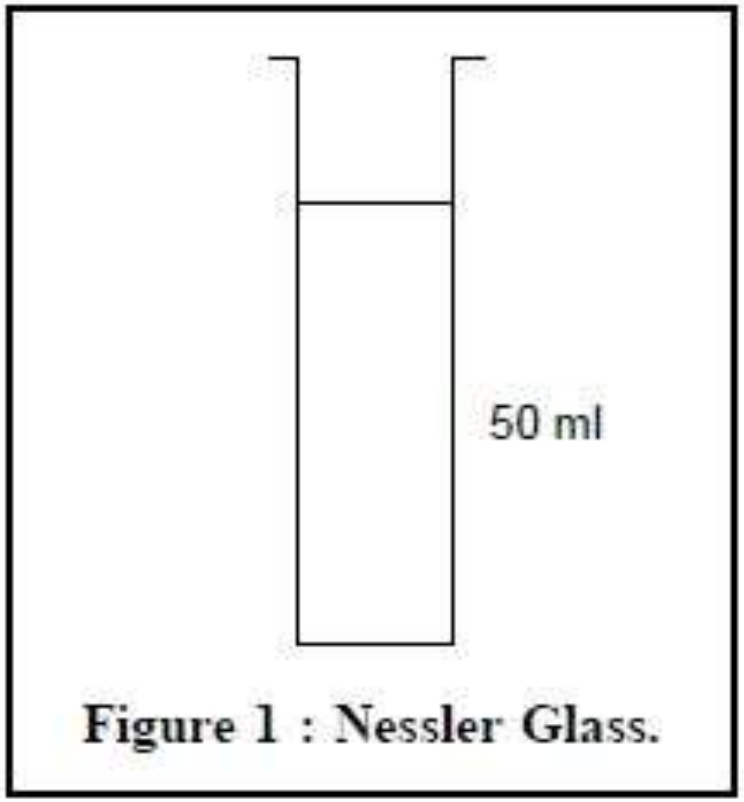
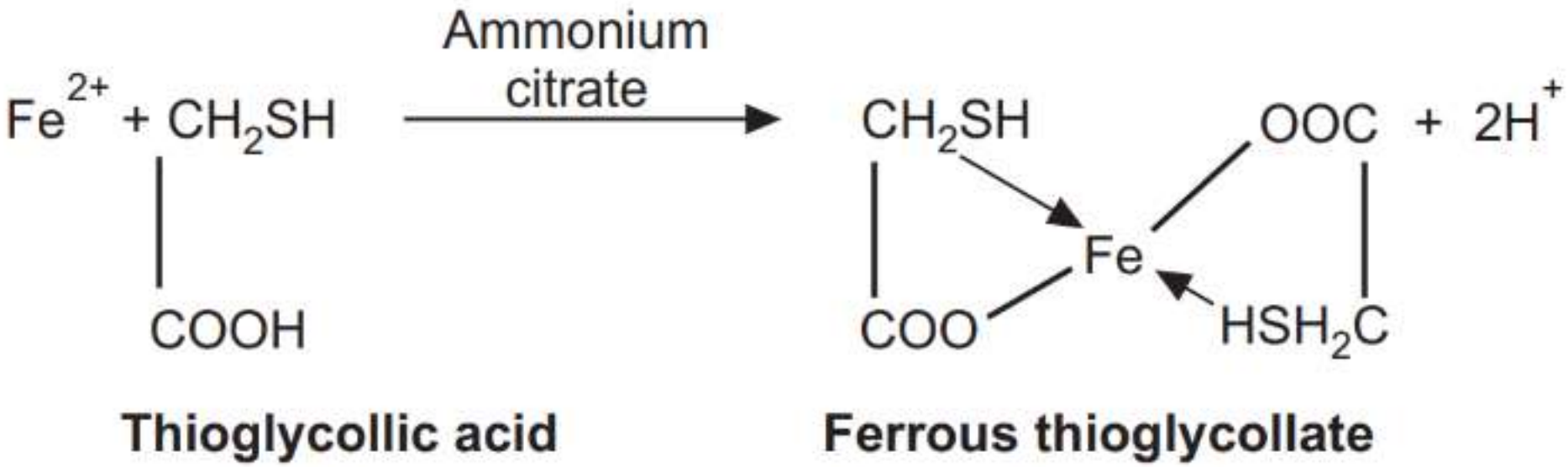


Figure 1 : Nessler Glass.

3. Name the below given apparatus?



Thioglycollic acid **Ferrous thioglycollate**

REFERENCES

•***Practical Pharmaceutical Chemistry, Part-I*** by A.H. Beckett and J.B. Stenlake: This is a widely referenced book in pharmaceutical analysis that covers various limit tests in detail.

Pharmaceutical Chemical Analysis: Methods for Identification and Limit Tests by Ole Pedersen

•***Pharmaceutical Inorganic Chemistry*** by P. Gundu Rao, and also titles by other authors such as Alagarsamy or S. Chand, R.D. Madan, and Anita Madan


Thank You