

Pharmacopoeia the word derives from the ancient Greek pharmakopoia from (pharmako-) "drug", followed by the verb-stem (poi-) "make" and finally the abstract noun ending -ia (- ia). These three elements together can be rendered as "drug-mak-ing" or "to make a drug".

General requirements may also be given in the pharmacopoeia on important subjects related to medicines quality, such as analytical methods, microbiological purity, dissolution testing, stability

Indian Pharmacopoeia Commission (IPC) is an autonomous institution of the Ministry of Health and Family Welfare which sets standards for all drugs that are manufactured, sold and consumed in India

Pharmacopoeia (IP) which has been modelled over and historically follows from the British Pharmacopoeia. The standards that are in effect since 1 December 2010 is the Indian Pharmacopoeia 2010 (IP 2010). The Pharmacopoeia 2014 was released by Health Minister Ghulam Nabi Azad on 4 November 2013

History of Pharmacopoeia The actual process of publishing the first Pharmacopoeia started in the year 1944 under the chairmanship of Col. the I. P. list was first published in the year 1946 and was put forth for approval. The titles are suffixed with the respective years of publication, e.g. IP 1996. The following table describes the publication history of the Indian Pharmacopoeia.

1st Edition 1955 Supplement 1960

2nd Edition 1966 Supplement 1975

3rd Edition 1985 Addendum 1989

4th 1996 Addendum 2000, Addendum 2002, Addendum 2005

5th Edition 2007 Addendum 2008

6th Edition 2010 Addendum 2012

7th Edition 2014 Addendum 2015,2016

8th Edition 2018 Addendum 2019

9th Indian Pharmacopoeia (IP) 2022, Addendum 2024, released on January 10, 2024

Indian Pharmacopoeia Commission (IPC) is an autonomous institution of the [Ministry of Health and Family Welfare](#) which sets standards for all drugs that are manufactured, sold and consumed in India.^[3] The set of standards are published under the title **Indian Pharmacopoeia (IP)**

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Addendum 2024 to IP 2022 is described as light orange



Preparing and Standardizing Oxalic Acid Solutions

Step 1: Understanding Oxalic Acid

- Oxalic acid is a pure substance that can be used to make standard solutions directly.
- Its chemical formula is $(COOH)_2$, and it often comes as a hydrated form ($C_2H_2O_4 \cdot 2H_2O$).

Step 2: Calculating the Equivalent Weight

- The equivalent weight (Eq. wt.) of hydrated oxalic acid is 63.

Step 3: Preparing Solutions

- To prepare a 0.1 N (N/10) solution:
 - Weigh 6.3 grams of oxalic acid.
 - Dissolve it in water and make the volume up to 1 liter.
- To prepare a 0.05 N (N/20) solution:
 - Weigh 3.15 grams of oxalic acid.
 - Dissolve it in water and make the volume up to 1 liter.

Alternative Method: Standardizing HCl Against Standardized NaOH

- If you have a standardized 0.1 N NaOH solution (previously standardized against 0.1 N oxalic acid), you can use it.
- Use phenolphthalein as an indicator.
- Titrate the HCl solution against the standardized NaOH solution until the color changes.
- Calculate the exact normality of the HCl solution based on the titration results.

1. Oxalic Acid ($H_2C_2O_4 \cdot 2H_2O$)

- **Preparation (0.1 N):** Weigh ~6.3035 g and dissolve in water; make up to 1 L
- **Standardization:**
 - **Method A:** Titrate with NaOH using phenolphthalein (pink endpoint)

- Normality (N)= $2 \times$ Molarity (M) \Rightarrow
- M=N/2 = $0.1/2 = 0.05\text{mol}$
- Required mass= $0.05\text{mol/L} \times 126.07\text{g/mol} = 6.3035\text{g}$
- **Method B:** Titrate KMnO_4 in acidic medium (heat to $\sim 70^\circ\text{C}$); oxalic acid oxidized— KMnO_4 acts as its own indicator (purple disappears to

Prepare 1 liter of 0.1 M oxalic acid solution.

Calculation:

Required mass=Molarity \times Molar mass \times Volume (L)= $0.1\text{ mol/L} \times 126.07\text{ g/mol} \times 1\text{ L} = 12.607\text{ g}$

Procedure:

1. Weigh **12.61 g** (rounded) of oxalic acid dihydrate.
2. Dissolve it in a beaker with $\sim 800\text{ mL}$ of **distilled water**.
3. Transfer the solution to a 1-liter volumetric flask.
4. Make up the volume to the mark with distilled water.
5. Mix thoroughly.

Sodium Hydroxide (NaOH)

- **Preparation (0.1 N):** Weigh 4 g, dissolve in water, and dilute to 1 L
- **Standardization:** Titrate against standard oxalic acid with phenolphthalein as indicator (pink endpoint)

Hydrochloric Acid (HCl)

- **Preparation (0.1 N):** Dilute $\sim 8.33\text{ mL}$ of $\sim 12\text{ M}$ concentrated HCl to 1 L
- **Standardization:** Titrate with sodium carbonate (Na_2CO_3) using methyl orange indicator (orange endpoint)

Sulfuric Acid (H_2SO_4)

- **Preparation (0.1 N):** Dilute $\sim 5.56\text{ mL}$ of $\sim 18\text{ M}$ concentrated H_2SO_4 to 1 L (add acid to water)
- **Standardization:** Titrate with sodium carbonate using methyl orange (orange endpoint)

Potassium Permanganate (KMnO_4)

- **Preparation (0.1 N):** Weigh $\sim 3.1606\text{ g}$, dissolve, dilute to 1 L; decompose over time so needs standardization
- **Standardization:** Titrate against standard oxalic acid in acidic medium (H_2SO_4), usually heated to $\sim 70^\circ\text{C}$; KMnO_4 serves as self-indicator (purple fades to pale pink)

Solution	Preparation (Concentration)	Standardization Method	Indicator / Endpoint
Oxalic Acid (0.1 M)	12.6 g → 1 L	Titrate with NaOH or KMnO ₄	Phenolphthalein / pink
NaOH (0.1 N)	4 g → 1 L	Titrate with oxalic acid	Phenolphthalein / pink
HCl (0.1 N)	~8.33 mL (12 M) → 1 L	Titrate with Na ₂ CO ₃	Methyl orange / orange
H ₂ SO ₄ (0.1 N)	~5.56 mL (18 M) → 1 L	Titrate with Na ₂ CO ₃	Methyl orange / orange
Na ₂ S ₂ O ₃ (0.1 N)	24.818 g → 1 L	Titrate with K ₂ Cr ₂ O ₇ / KI + starch	Disappearance of blue
KMnO ₄ (0.1 N)	3.1606 g → 1 L	Titrate with oxalic acid in acidic heat	Self-indicator (pink)
Ceric Ammonium Sulphate (0.1 M)	65–67 g + H ₂ SO ₄ + water → 1 L	Titrate with As ₂ O ₃ or oxalate	Ferroin / red to pale blue

Errors in Measurement

Error is the deviation of the observed or measured value from the true or accepted value.

Error=Measured Value–True Value **Sources of Errors**

- **Instrumental Errors:** Due to faulty or improperly calibrated instruments.
- **Personal Errors:** Due to human mistakes in reading or recording.
- **Environmental Errors:** Caused by temperature, pressure, humidity, etc.
- **Observational Errors:** Due to parallax or wrong use of instrument.
- **Procedural Errors:** Due to incorrect experimental methods.

INSTRUMENTAL ERRORS

CAUSED BY FAULTY OR UNCALIBRATED INSTRUMENTS.

EXAMPLES:

- **UNCALIBRATED BALANCE**
- **FAULTY THERMOMETER**
- **LEAKY BURETTE**

b. Personal (Human) Errors

Errors due to mistakes in observation or judgment.

Examples:

- Misreading the meniscus
- Parallax error
- Delayed stopwatch readings

c. Environmental Errors

Caused by uncontrolled environmental conditions.

Examples:

- Temperature fluctuations
- Humidity affecting chemicals
- Drafts affecting balances

Procedural Errors

Examples:

- Incomplete reactions
- Impure reagents
- Wrong titration technique

e. Sampling Errors

Errors arising from non-representative or poorly mixed samples.

Examples:

- Uneven mixing in solutions
- Sampling from surface only

Systematic Errors

- Same error occurs in every measurement.
- Can be detected and corrected.
- Causes: calibration errors, procedural flaws.

Example: A balance always reads +0.5 g.

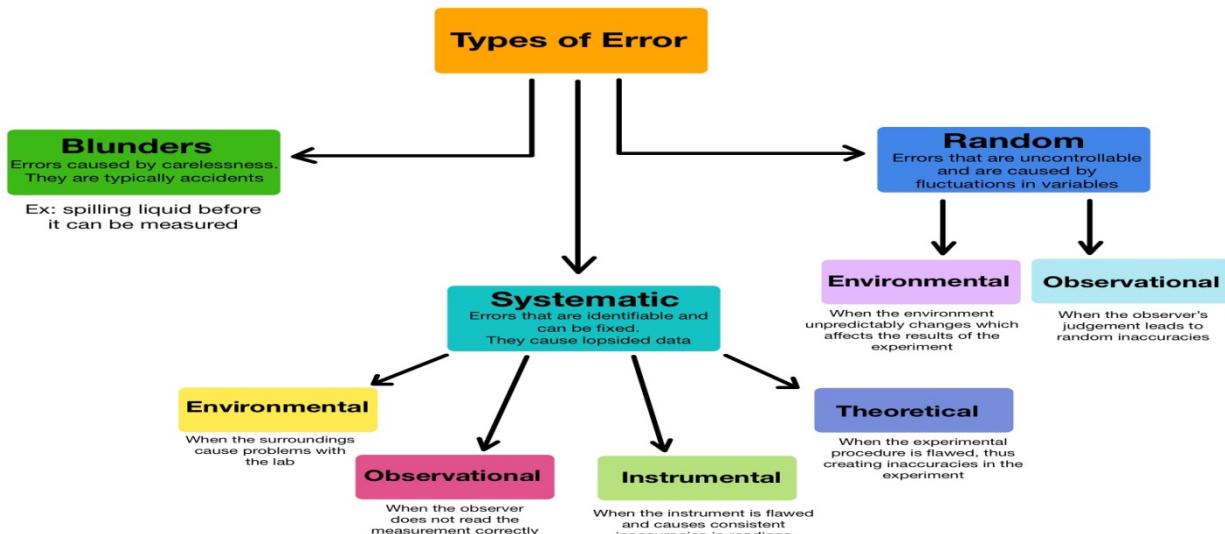
◆ b. Random Errors

- Vary unpredictably.
- Cannot be completely eliminated, but can be minimized.
- Causes: temperature, voltage fluctuations, human reaction time.

◆ c. Gross Errors

- Due to carelessness or incompetence.

- Can lead to **major deviation** from expected values.
- Must be identified and eliminated.

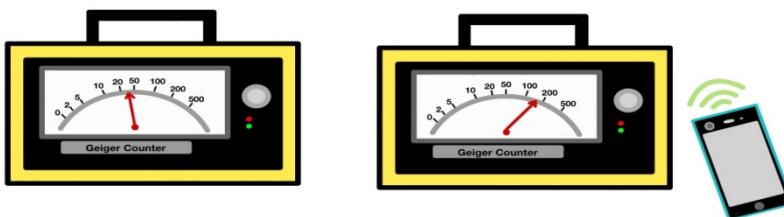


Systematic Error



Instrumental Error

The scale is improperly calibrated so it reads 2mg even with nothing on it. This causes the beaker to appear 2mg heavier than it would be, and any other measurements would read 2mg heavier than what they actually weigh



Environmental Error

The Geiger counter shows the radiation, however, when a cell phone is near the Geiger counter, the radiation is read as greater than what it actually is because the cell phone is radiating RF waves which disrupts the reading



Observational Error

The observer looks at the beaker from above eye level and write down the volume as 40mL, when in reality when looked at eye level you can see the liquid has a volume of about 42mL

Methods of minimizing Errors

- **1) Calibration of apparatus :**
- All apparatus like weights, flasks, burettes and pipettes should be calibrated.
- The appropriate corrections applied to the original measurements.
- In some case errors cannot be eliminated.
- Apply a correction for that effect.
- e.g An impurity in a weighed precipitate may be determined and its weight deducted.

Accuracy and Precision

In everyday speech, the terms **accuracy** and **precision** are frequently used interchangeably. However, their scientific meanings are quite different. Accuracy is a measure of how close a measurement is to the correct or accepted value of the quantity being measured. Precision is a measure of how close a series of measurements are to one another. Precise measurements are highly reproducible, even if the measurements are not near the correct value. Darts thrown at a dartboard are helpful in illustrating accuracy and precision.

The ability of an instrument to measure the accurate value is known as accuracy. In other words, it is the **the closeness of the measured value to a standard or true value**. Accuracy is obtained by taking small readings. The small reading reduces the error of the calculation. The accuracy of the system is classified into three types as follows:

1)Point Accuracy

The accuracy of the instrument only at a particular point on its scale is known as point accuracy. It is important to note that this accuracy does not give any information about the general accuracy of the instrument.

2)Accuracy as Percentage of Scale Range

The uniform scale range determines the accuracy of a measurement. This can be better understood with the help of the following example:
Consider a thermometer having the scale range up to 500°C. The thermometer has an accuracy of ±0.5 percent of scale range i.e. $0.005 \times 500 = \pm 2.5^{\circ}\text{C}$. Therefore, the reading will have a maximum error of $\pm 2.5^{\circ}\text{C}$.

Precision

The closeness of two or more measurements to each other is known as the precision of a substance. If you weigh a given substance five times and get 3.2 kg each time, then your measurement is very precise but not necessarily accurate. Precision is independent of accuracy. The below examples will tell you about how you can be precise but not accurate and vice versa. Precision is sometimes separated into:

- Repeatability

The variation arising when the conditions are kept identical and repeated measurements are taken during a short time period.

- Reproducibility

The variation arises using the same measurement process among different instruments and operators, and over longer time periods.

COMPLEXOMETRIC TITRATION, OR CHELATOMETRY

It is a type of volumetric analysis that involves forming a colored complex to mark the endpoint. It's especially useful for determining the concentration of a mixture of different metal ions in a solution.

Classification of Complexometric Titrations

Complexometric titrations are broadly classified based on the method of titration:

- **Direct Titration:** The analyte (metal ion) is directly titrated with a standard solution of the chelating agent (e.g., EDTA). This method is used when the reaction is rapid and the metal-EDTA complex is stable.
 - **Reaction:** $Mn^{++} + nEDTA \rightarrow [M-EDTA]^{(n-4)+}$
 - **Example:** Estimation of water hardness using EDTA.

Back Titration: An excess but known amount of EDTA is added to the analyte solution, which is then back-titrated with a standard solution of a second metal ion. This method is suitable for metal ions that react too slowly with EDTA, or when the analyte precipitates at the required pH.

- **Reaction:** $Mn^{++} + EDTA_{excess} \rightarrow [M-EDTA]^{(n-4)+} + EDTA_{unreacted}$
- $EDTA_{unreacted} + M'm^+ \rightarrow [M'm-EDTA]$

Replacement or Substitution Titration: A less stable metal-EDTA complex is used to titrate a metal ion that doesn't react sharply with the indicator. The analyte displaces the metal ion from its complex, and the liberated ion is then titrated.

- **Reaction:** $[M'm-EDTA]^{(n-4)+} + Mn^{++} \rightarrow [M-EDTA]^{(n-4)+} + M'm^+$

Metal Ion Indicators

Metal ion indicators are dyes that change color when they bind to a metal ion. The indicator's complex with the metal ion must be less stable than the metal-EDTA complex. At the beginning of the titration, the indicator forms a complex with the metal ions, giving a specific color. As EDTA is added, it preferentially complexes with the free metal ions. At the endpoint, all the metal ions are complexed by EDTA, and the indicator is released, reverting to its original color.

- **Examples:**
 - **Eriochrome Black T (EBT):** Used for the estimation of Ca^{2+} , Mg^{2+} , and Zn^{2+} .
 - This is a very common indicator. When all the metal ions are chelated by EDTA, the free EBT appears blue.
 - **Murexide:** Used for calcium and nickel titrations. Used for the estimation of Ca^{2+} .
 - **Calcein:** Used for calcium titrations. Used for the estimation of Ca^{2+} and Mg^{2+} .

Masking and Demasking Reagents

When a sample contains multiple metal ions, one might need to determine the concentration of a specific ion. **Masking agents** are used to prevent interfering ions from reacting with the titrant or indicator. These agents form stable, non-interfering complexes with the unwanted ions. Once the targeted ion is titrated, a **demasking agent** is added to release the masked ions, allowing for their separate titration.

- **Masking Agents:**
 - **Cyanide (CN^-):** Masks ions like Zn^{2+} , Cd^{2+} , Ni^{2+} , and Co^{2+} by forming stable complexes.
 - **Triethanolamine:** Masks Al^{3+} , Fe^{3+} , and Ti^{4+} .
 - **Ammonium Fluoride:** Masks Al^{3+} , Fe^{3+} , and Ti^{4+} .
- **Demasking Agents:**
 - **Formaldehyde:** Demasks zinc from its cyanide complex.
 - **Dilute acids:** Can be used to demask certain ions by changing the pH.

Estimation of Magnesium Sulfate and Calcium Gluconate

Estimation of Magnesium Sulfate (MgSO_4)

This is a direct titration method.

1. A known amount of magnesium sulfate is dissolved.
2. A buffer solution of **ammonia and ammonium chloride** is added to maintain the pH at around 10.
3. **Eriochrome Black T** indicator is added, which forms a wine-red complex with Mg^{2+} ions.
4. The solution is titrated with a standard **EDTA** solution.
5. At the endpoint, all Mg^{2+} ions form a more stable complex with EDTA, releasing the indicator. The color changes from **wine-red to blue**.

6. The volume of EDTA used is proportional to the concentration of MgSO₄.



Estimation of Calcium Gluconate

1. Titration: Ca²⁺ is titrated with EDTA using Calmagite or Murexide as an indicator.

2. Reaction: Ca²⁺ + EDTA → Ca-EDTA complex

The direct titration of Ca²⁺ with EBT indicator provides a poor endpoint. Therefore, a substitution titration is often used.

1. A known amount of calcium gluconate is dissolved.
2. A small, known amount of **magnesium sulfate** is added. This is a crucial step.
3. A buffer solution of **ammonia and ammonium chloride** (pH 10) is added.
4. **Eriochrome Black T** indicator is added. The indicator preferentially complexes with the added Mg²⁺ ions, as the Mg-indicator complex is stronger than the Ca-indicator complex, giving a **wine-red** color.
5. The solution is titrated with a standard **EDTA** solution.
6. EDTA first complexes with all the **calcium ions**, since the Ca-EDTA complex is more stable than the Mg-EDTA complex.
7. Once all the Ca²⁺ ions are complexed, EDTA starts to complex with the Mg²⁺ ions.
8. At the endpoint, all Mg²⁺ ions are chelated, releasing the EBT indicator, which changes the color to **blue**.
9. The volume of EDTA used is corrected for the volume required to complex the added magnesium, and the remaining volume is used to calculate the concentration of calcium gluconate.

Reactions:

- Ca²⁺ + H₂Y²⁻ → CaY²⁻ + 2H⁺
- Mg²⁺ + H₂Y²⁻ → MgY²⁻ + 2H⁺
- Mg²⁺ + In²⁻ → [Mg-In]²⁻ (Wine-red)
- Ca²⁺ + H₂Y²⁻ → [Ca-EDTA]²⁻ + 2H⁺
- Mg²⁺ + H₂Y²⁻ → [Mg-EDTA]²⁻ + 2H⁺
- [Mg-In]²⁻ + H₂Y²⁻ → [Mg-EDTA]²⁻ + HIn²⁻ (Blue)