

OUR LADY OF FATIMA UNIVERSITY

College of Pharmacy

PHAN 212

**WEEK 5:
REFRACTOMETRY
AND POLARIMETRY**



UNIT OUTCOMES

At the end of this unit, the students are expected to:

- describe the instrument and functions of the essential parts of refractometry and polarimetry
- understand the principle involved
- identify useful application of the instruments in pharmaceutical quality control



UNIT OUTLINE

- **Refractometry**
 - **Refractive Index**
 - **Instrumentation**
- **Polarimetry**
 - **Stereoisomers / Enantiomers**
 - **Instrumentation**



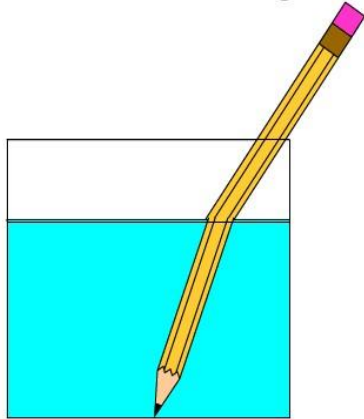
REFLECTION

REFRACTION

Refractometry

- is a technique that measures how light is refracted when it passes through a given substance (an unknown compound).
- The amount by which the light is refracted determines the **refractive index**.

Refraction of Light

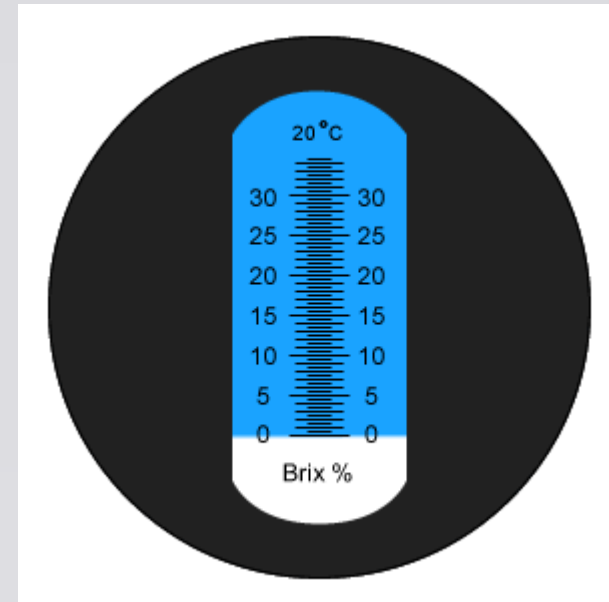


Light bends inwards because the speed of light is slower in water



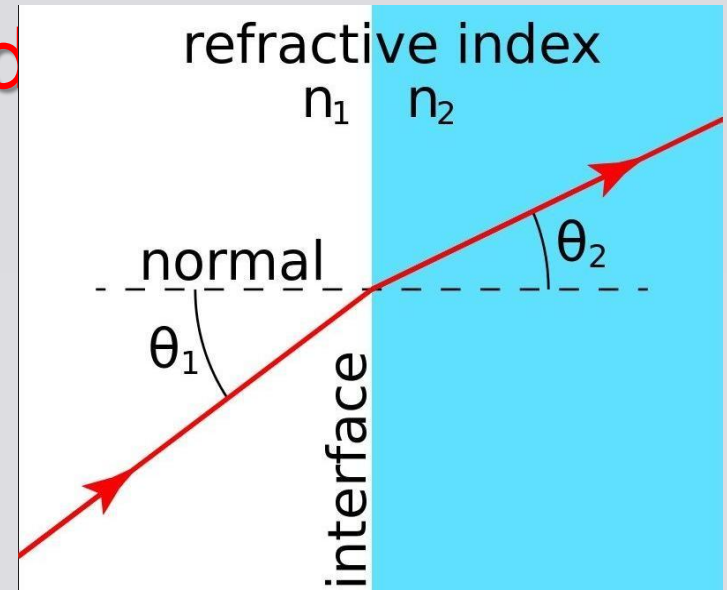
Refractometry

- Index of refraction is a physical constant frequently made use of it in the determination of the **identity and purity** of drug products.



Refractive index

- Also known as index of refraction.
- It is a measure of how fast light rays travel through a given medium.
 - Refractive index is the measure of the bending of a light ray when passing from one medium to another



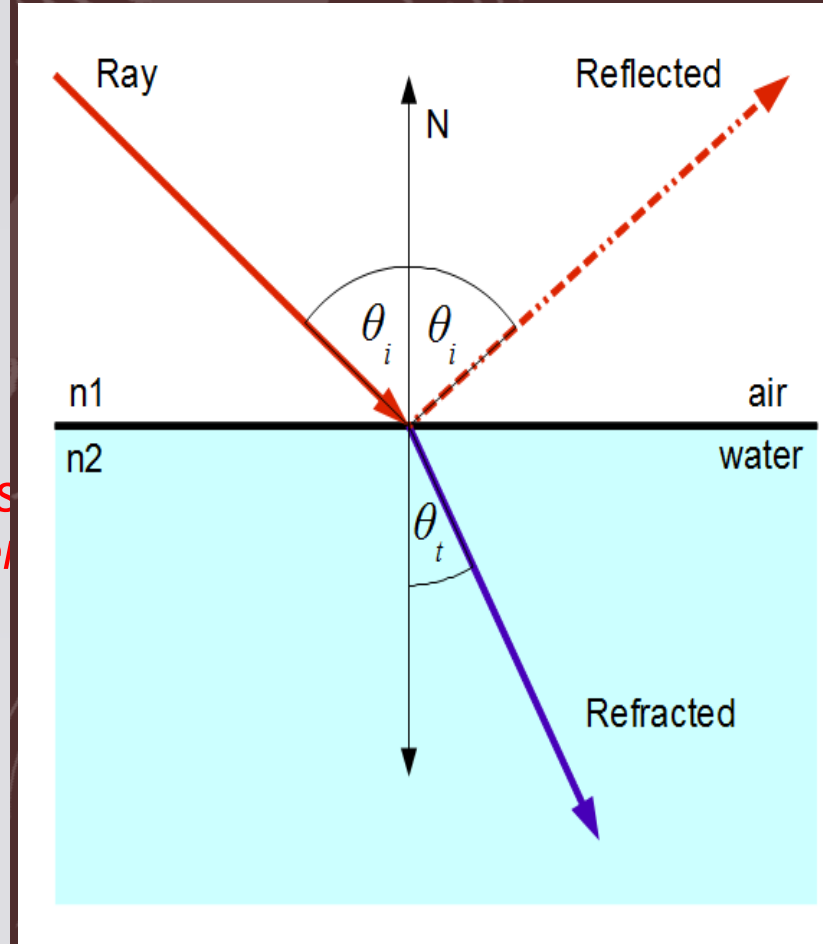
$$n = \frac{c}{v}$$

c = The speed of light in vacuum

v = The phase velocity of light in the medium

Refractive index

- can be used **to identify** an unknown liquid compound, or it can be used as a means of **measuring the purity** of a liquid compound by comparing it to literature values.
 - *The closer the refractive index is to the literature values, the purer the sample.*
- It is also defined as the ratio of the velocity of light in air to the velocity of light in the medium being measured:
$$n_D = [V \text{ air}]/(V \text{ liquid})$$





- **Refractive index** **temperature** **and** **wavelength is dependent**
- **Refractive index (n)** is determined **at** **temp of 20°C** but **Pharmacopoeial standard temp is 25°C**
 - To adjust for temperatures greater than 20°C,
Use the following formula:

$$n_D^{20} = n_D^T + 0.00045(T - 20^\circ\text{C})$$



- **Critical angle** – the maximum value of refraction
 - The angle of refraction when angle of incidence equals 90°
- **Refractive index is temperature dependent.**
 - Combining refractive index and substance density it is possible to define a quantity that is temperature independent. It is called **specific refraction**:

$$n^2 - 1$$

$$r = \frac{n^2 - 1}{d(n^2 + 2)}$$

where r - specific refraction, d - density, n - refractive index



Principle

- The main principle involved in refractometry is the refraction based on **the speed of the light that passes in the different mediums.**
 - Light enters from the lower denser medium to a higher denser medium at an angle, that is, with bent. The bent in the light ray is known as the refraction.
 - The relation between the refraction of the light between the air and the medium is given by **Snell's law**:

$$n = \frac{\sin i}{\sin r}$$

where n = refractive index; $\sin i$ = angle of incidence; $\sin r$ = angle of refraction.

- Samples with different refractive index will produce different angles of refraction.
 - *This helps in the assessment of the compounds' composition and the purity of the compounds.*

Refractometry in Pharmaceutical Analysis

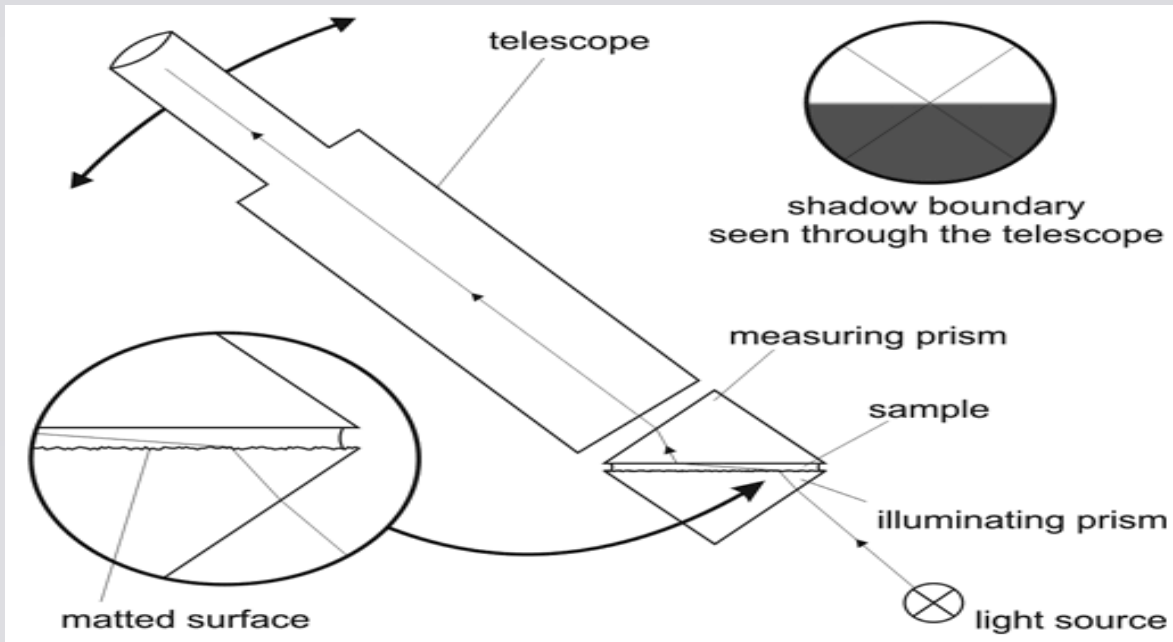
- It may be used to determine quantitatively the **strength and purity** of solutions or the portions in which liquids are mixed.

e.g., the percentage of alcohol in water



Instrumentatio

- **Abbe refractometer** has the following parts:



- Calibration is done using **water** as standard with n value of **1.3330 at 20°** and **1.3325 at 25°C**

Instrumentation

- The instrument used for the determination of the refractive index is known as the **refractometer**.
 - **Abbe refractometer**
 - This is a benchtop refractometer, designed by Ernst Abbe, which provides high accuracy. In this refractometer, the sample is held between the illuminating prism and the refracting prism. A light source is allowed through the illuminating prism and the detector is placed behind the refracting prism.
 - **Handheld Refractometer**
 - The main principle involved in this refractometer is the measurement of the critical angle. It is comprised of the lenses and prisms to project the black line on the glass when the sample is placed between the measuring prism and the plate.





Sample Problems

1. Calculate the speed of light for the following mediums:

- Water
- Diamond
- Whale oil

| material | n | material | n |
|---------------|--------|-------------------|-------|
| Vacuum | 1 | Crown Glass | 1.52 |
| Air | 1.0003 | Salt | 1.54 |
| Water | 1.33 | Asphalt | 1.635 |
| Ethyl Alcohol | 1.36 | Heavy Flint Glass | 1.65 |
| Fused Quartz | 1.4585 | Diamond | 2.42 |
| Whale Oil | 1.460 | Lead | 2.6 |



Sample Problems

2. Optical fibers are generally composed of silica, with an index of refraction around 1.44. How fast does light travel in a silica fiber?



Sample Problems

3. Calculate the refractive index for a substance if the speed of light in that medium is

a. 2.10×10^8 m/s

b. 1.50×10^8 m/s



Sample Problems

4. The speed of light in an unknown medium is measured to be 2.76×10^8 m/s.

(a) What is the index of refraction of the medium?



Polarimetry

- Measure of optical rotation
 - **Optical rotation** is a property of a substance to rotate a plane of polarized light




Polarimetr

- Optical activity is a function of both the structure and concentration of a substance
- It serves as a means of establishing the **sample purity and identity**, or sometimes therapeutic activity

////////////////////

Polarimeter is a scientific instrument used to **measure the angle of rotation** caused by passing polarized light through an **optically active substance**.



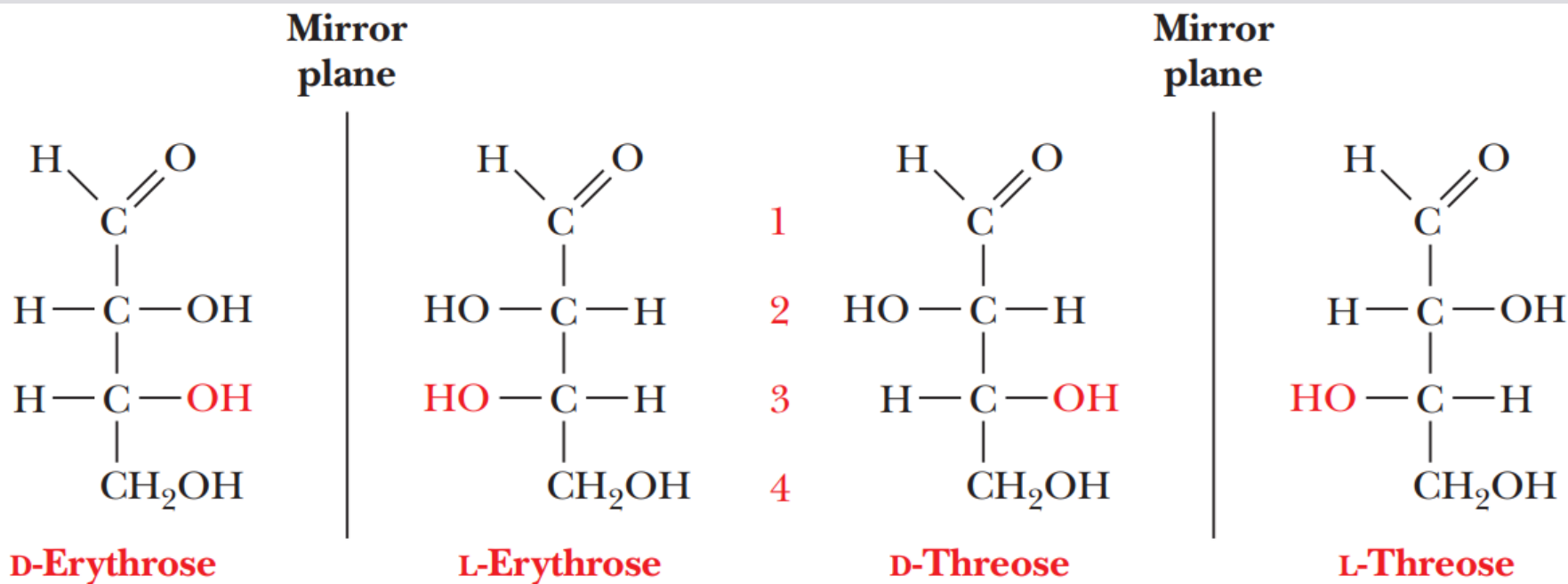


Isomers – two or more compounds having **the same molecular formula but different in the arrangement** of atoms and some properties.



Stereoisomers - isomeric molecules that have the same molecular formula and sequence of bonded atoms (constitution), but that **differ only in the three-dimensional orientations of their atoms in space**

Enantiomers - one of two stereoisomers that are mirror images of each other that are non-superimposable (not identical)

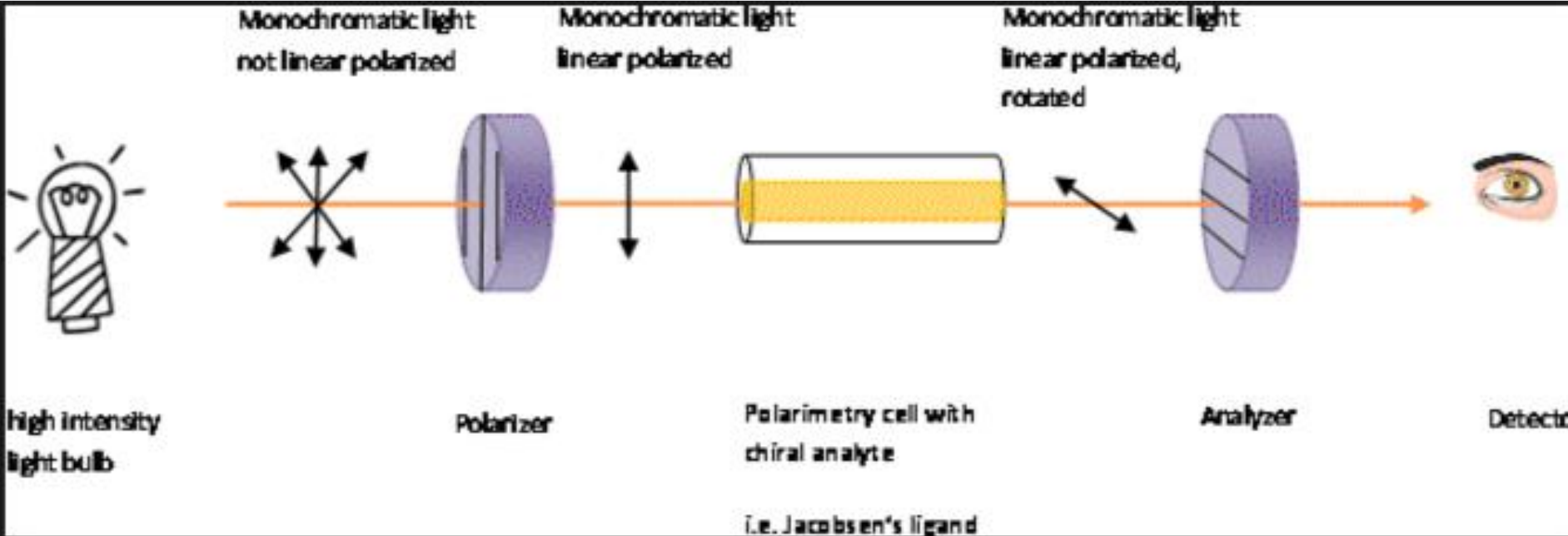




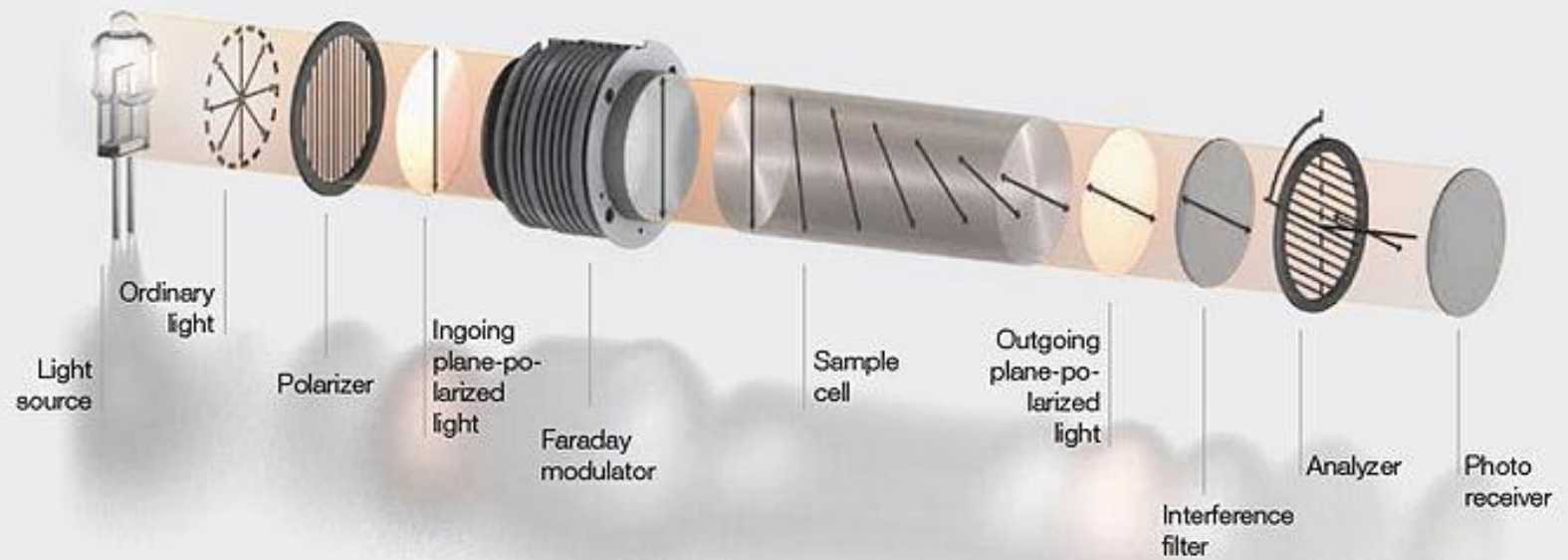
Instrumen

t

- Monochromatic light source (**sodium vapour lamp**)
- Polarizer (**calcite prism**)
- Polarimeter glass tube (20 cm)
- Analyzer (moving)
- Circular scale with vernier
- Null detector



Measuring system





Prism

- **S** Ordinary light or white light creates a transverse vibration, **to obtain a plane-polarized light prisms are used**
- Iceland spar , Nicol prism
- **Polarizer** is the prism that **polarized the light**
- **Analyzer** is the prism which **examined the light**



Rotatory power

- When emergent light reaches the analyzer, **the prism being parallel to the polarizer**, intensity of light is maximum and perpendicular to the polarized light, light is minimum.
- Overall difference in the position of the analyzer provides minimum light intensity with and without the sample in the cell is observed as **“ROTATION”** of the sample.



Specific Rotation

$$\frac{100a}{lpd} \quad \text{or} \quad \frac{100a}{lc}$$

a –observed rotation of the liquid at temperature t using sodium light(589.0 nm)

l - length of the tube,

d – sp gr of the liquid

p – conc of solution (100g soln)

c – conc of solution (100ml of solution)



Specific Rotation

- numerically equal with optical rotation value
- Angular rotation in degrees to which the plane of polarized light is rotated when passing through a 1 decimeter of a liquid, calculated on the basis of specific gravity of 1 or concentration of 1 g in 1 ml of solution.

$$[\alpha]_t^T = \frac{\alpha}{ld} \quad \text{or} \quad \frac{\alpha}{lc}$$

α –observed rotation of the liquid at temperature t using sodium light(589.0 nm)

l - length of the tube, dm

d – sp gr of the liquid

c – conc of solution g/ml



PROBLEM 1: Calculate the specific rotation of plane polarized sodium D light when it passes through an aqueous solution of lactose (13 g in 100 ml) in a 10-cm cell and gives a rotation of 7.25°



PROBLEM 2: What is the optical rotation when sodium D light passes through a solution of a compound in chloroform, containing 15 g per 100 ml, placed in a 10 cm cell and gives a specific rotation of 37.5.



PROBLEM 3: A sample of pure (S)-2-butanol was placed in a 10 cm polarimeter tube. Using the D line of sodium lamp, the observed rotation at 20°C was +104°. Determine its specific rotation if it has a density of 0.805 g/ml.



PROBLEM 4: A solution of organic compound is placed in a 20cm tube. It produces a rotation of $+38.73^\circ$ at 20°C using the d line of sodium. The solvent alone produced a rotation of -1.36 . If the specific rotation of the compound is 62.12° , what is the concentration of the solution?



Assay

s

- Acetylcysteine +21° to +27°
- Ascorbic acid +20.5° to 21.5°
- Ampicillin + 280° to 305°
- Carbidopa - 22.5° to -26.5



REFERENCES:

- Jenkins, Glenn L, Jenkins Quantitative Pharmaceutical Chemistry, N.Y, N.Y. : Mc Graw, Latest Edition



Watch these Videos:

- Abbe refractometer (The use and how it works)
 - <https://www.youtube.com/watch?v=CW9gOyH8HIA>
- Polarimetry principle and instrumentation
 - https://www.youtube.com/watch?v=T6zjiU_91g



Activities for next meeting:

- 1. Quiz No. 5: Refractometry, Polarimetry**
- 2. Lecture Topic: Compedial Requirements for Liquid Dosage Form**





THANK YOU!

Any questions?

You can find me at:

username@fatima.edu.ph

