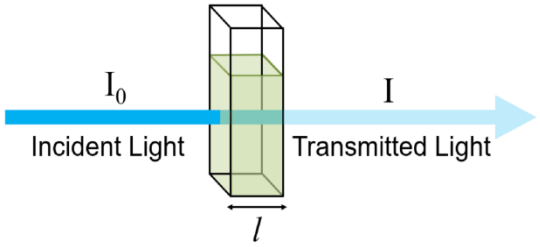
EXPERIMENT 2

AIM: Determination of the Concentration of an Unknown $KMnO_4$ Solution by colorimetrically.

THEORY: The Beer-Lambert law states that the absorbance of a solution is directly proportional to the concentration of the absorbing species in the solution and the path length. Thus, for a fixed path length (cuvette length), Visible spectroscopy (colorimetry) can be used to determine the concentration of the absorber in a solution. The absorbance changes with concentration, A higher concentration of the colored solution absorbs more light (and transmits less) than a solution of lower concentration.

According to Beer–Lambert law,



$$\log (I_o / I_t) = A = \varepsilon c l$$

where

I_o and I_t are the incident and transmitted intensities,

A = **absorbance** and

ε is a constant *i.e.* **absorptivity** (formerly called the *extinction coefficient*). If the concentration is measured in $molL^{-1}$, the absorptivity is called the *molar absorptivity* .

$$\mathbf{A = \varepsilon c l}$$

At constant length of solution

$$\mathbf{A \alpha c}$$

REQUIREMENTS

Colorimeter, cuvette, six test tubes

Two Burettes or graduated cylinders two 100 mL beakers

0.01M $KMnO_4$ solution

distilled water, test tube rack, stirring rod, tissues (preferably lint-free)

PROCEDURE

(a) Determination of λ_{max}

(b) Absorbance of different concentration solution at λ_{max}

(a) Calculation of λ_{max}

This can be taken from references (tables of molar extinction coefficients), or more accurately, determined from a calibration curve.

i. Prepare 100 ml, 0.01M **$KMnO_4$** (**Molecular weight 158.03 gm/mol**) solution (stock solution), fill it in a burette

ii. Switch on the computer **and/or** the instrument powers; wait for 30 minutes for 'warm-up' of the instrument.

iii. In the instrument one can choose % transmittance or absorbance display, wavelength range of interest, etc.

iv. Take one clean and dry glass (only for visible range scan) or quartz cuvette with a given path length (say, 1 cm). Prepare a *blank* by filling a cuvette 3/4 full with distilled water

Note: To correctly use cuvettes, remember:

- Wipe the outside of each cuvette with a lint-free tissue.
- Handle cuvettes only by the top edge of the ribbed sides.
- Dislodge any bubbles by gently tapping the cuvette on a hard surface.
- Always position the cuvette so the light passes through the clear sides.

v. By using blank cuvette (with distilled water) calibrate the colorimeter (absorbance = 0, transmittance =100 %) at **filter 1** and putout the blank cuvette.

vi. Then fill another cuvette with the stock solution and measure absorbance, note the reading in table.

vii. Repeat the step v and vi for all the filters (**wave length range**)

viii. From table we can see the **λ_{max}** *i.e.* wave length at which solution show maximum absorbance/ O.D.

Observation Table for calculating the λ_{max}

S.No	Filter	Wavelength/ Range	O.D./ Absorbance
1	1	420	
2	2	440	
3	3	490	
4	4	520	
5	5	540	
6	6	570	
7	7	600	
8	8	650	
9	9	700	

(b) Absorbance of different concentration solutions

- ✓ Obtain small volumes 0.01M $KMnO_4$ (**Molecular weight 158.03 gm/mol**) of solution and distilled water in separate beakers, fill in the separate graduated burettes
- ✓ Label five clean, dry, test tubes 1–5. Use Burettes to prepare five standard solutions according to the chart below. Thoroughly mix each solution with a stirring rod. Clean and dry the stirring rod between uses.

Concentration can be calculate by $M_1V_1 = M_2V_2$

1 2 3 4 5 6 (Unknown)

Test Tube	1	2	3	4	5	6
0.01M KMnO4. (mL)	2	4	6	8	10	
Distilled H_2O (mL)	8	6	4	2	0	
Concentration(M)	0.002	0.004	0.006	0.008	0.0100	Unknown

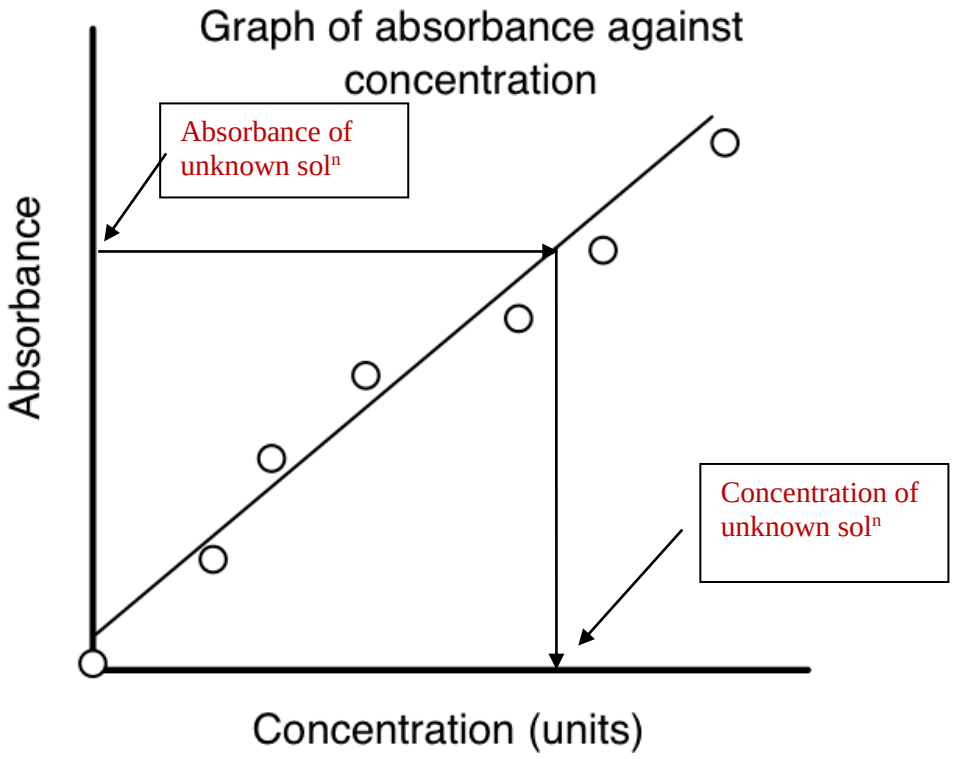
- ✓ You are now ready to collect absorbance-concentration data for the five standard solutions.
- ✓ Switch on the computer **and/or** the instrument powers; wait for 30 minutes for 'warm-up' of the instrument.
- ✓ In the instrument one can select light sources (UV and visible), choose the slit width, scan speed and % transmittance or absorbance display, wavelength range of interest, etc.
- ✓ Take two clean and dry glass (only for visible range scan) or quartz cuvettes with a given path length (say, 1 cm). Prepare a *blank* by filling a cuvette 3/4 full with distilled water and the other cuvette with aqueous KMnO4 solution with lowest concentration.
- ✓ Read the absorbance value displayed in the meter. When the displayed absorbance value stabilizes, record its value in your data table.
- ✓ Repeat the procedure for Test Tubes 2 to 6 .Similarly spectral runs are done for all the other samples starting from the lowest concentrations to next higher concentrations of $KMnO_4$. Every time one should rinse the cuvette taking a small portion of the solution to be analyzed next.
- ✓ Plot a curve between Absorbance v/s concentration.
- ✓ **Either** By locating the absorbance of the unknown on the vertical axis of the graph, the corresponding concentration can be found on the horizontal axis.

Or the concentration of the unknown can also be found using the slope of the Lambert-Beer's law curve. Slope $\varepsilon = A / c\,l$

Then, concentration of unknown = Absorbance of unknown / $\varepsilon\,l$

Data Table/ Observation Table

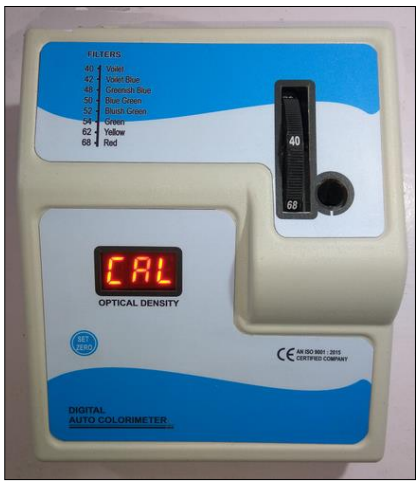
Test Tube	0.01M $KMnO_4$. (mL)	Distilled H_2O (mL)	Concentration(M)	Absorbance
	0	**10**	**0.00**	
1	2	8	0.002	
2	4	6	0.004	
3	6	4	0.006	
4	8	2	0.008	
5	~10	0	0.0100	
6			**Unknown**	



Result The concentration of unknown $KMnO_4$ solution is ……..

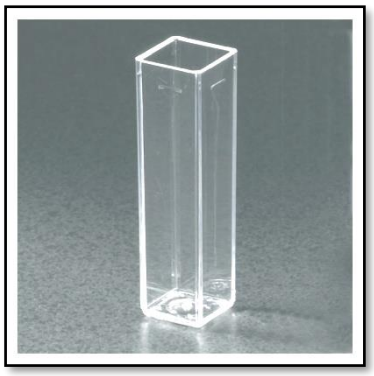
PRECAUTIONS

One should note that the Beer–Lambert law is obeyed by many substances mainly at low to moderate concentrations; therefore, dilute concentrations of the absorbing species should be measured. In practice it is advisable to measure absorbances in the range 0.1< A <1.0. Care must be taken to avoid any kind of chemical associations/dissociations of the absorbing species.

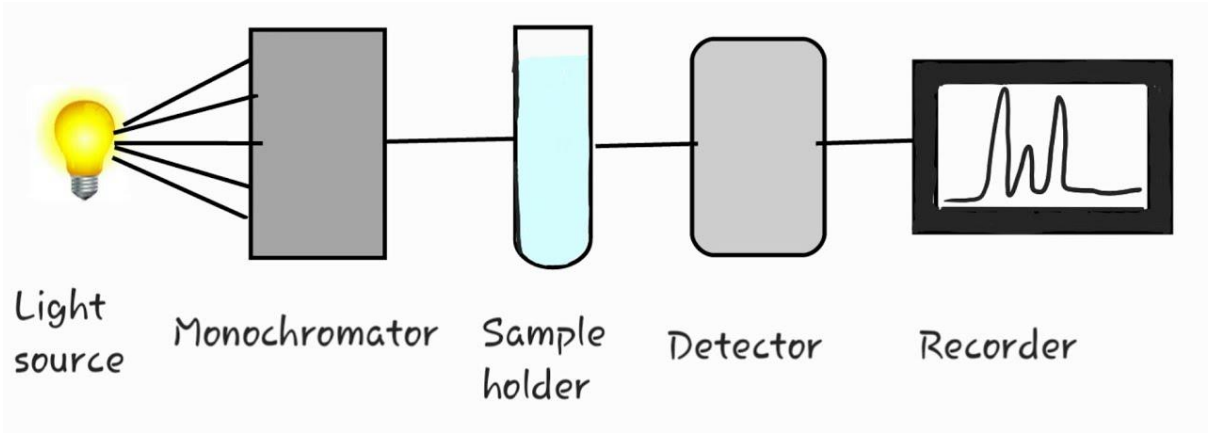


Colorimeter

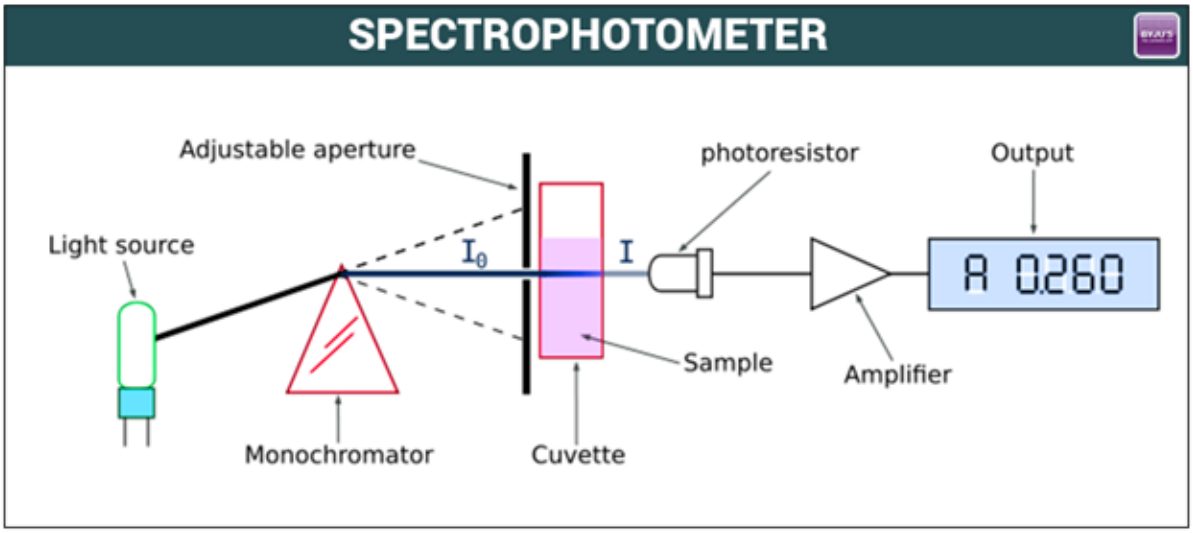
UV-Visible Spectrophotometers



Cuvette



Instrumentation


SPECTROPHOTOMETER
Adjustable aperture
photoresistor
Output
Light source
I_0
I
A 0.260
Sample
Amplifier
Monochromator
Cuvette