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Theme:

REALIZATION AND CALIBRATION OF A UV-VIS SPECTROPHOTOMETER

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Abstract

UV-Visible spectroscopy is a widely used technique for analyzing chemical compounds through their interaction with light. In this work, a low-cost UV-Vis spectrophotometer prototype was designed and developed, then tested and evaluated using different chemical solutions and concentrations. The results were compared with those obtained with Shimadzu UV-3600 spectrophotometer and with the existing literature.

The recorded spectrums showed that the developed system was able to detect the main absorption features of the tested samples and follow the expected concentration trends.

Although the UV-3600 instrument provided more accurate measurements, the prototype produced satisfactory results and demonstrated good overall performance.

Résumé

La spectroscopie UV-Visible est une technique largement utilisée pour l'analyse des composés chimiques grâce à leur interaction avec la lumière.

Ce travail porte sur la conception et la réalisation d'un prototype de spectrophotomètre UV-Vis à faible coût. Le système réalisée a été évalué à l'aide de différentes solutions chimiques préparées à plusieurs concentrations, puis comparé au spectrophotomètre Shimadzu UV-3600 ainsi qu'aux données de la littérature.

Les résultats obtenus montrent que le prototype est capable de détecter les principales caractéristiques d'absorption des échantillons étudiés et de suivre l'évolution de l'absorbance avec la concentration. Malgré une précision inférieure à celle de l'instrument de référence, le prototype présente des performances satisfaisantes, démontrant ainsi de bonnes performances globales.

ملخص

يُعدّ مطياف الأشعة فوق البنفسجية والمرئية تقنية واسعة الاستخدام في تحليل المركبات الكيميائية اعتمادًا على تفاعلها مع الضوء. يهدف هذا العمل إلى تصميم وتطوير نموذج لمطياف منخفض التكلفة، حيث تم بناؤه واختباره وتقييمه باستخدام محاليل كيميائية مختلفة وبتراكيز متعددة. كما تمت مقارنة النتائج المتحصل عليها مع تلك التي يوفرها جهاز شيمادزو 3600 . إضافة إلى البيانات المتوفرة في المراجع العلمية . أظهرت النتائج الطيفية أن النظام المطور قادر على تحديد خصائص الامتصاص الأساسية للعينات المدروسة وتتبع العلاقة المتوقعة بين الامتصاص والتركيز. وعلى الرغم من أن جهاز شيمادزو 3600 يتميز بدقة أعلى، فإن النموذج الأولي قدّم نتائج مرضية وأظهر أداءً عامًا جيدًا .

General introduction

Spectroscopy is one of the most important techniques used in science to study the interaction between light and matter. Among the different spectroscopic methods, UV-Visible spectroscopy is widely used because it is simple, fast, and allows the analysis of various chemical compounds.

This technique measures the amount of light absorbed by a sample at different wavelengths and provides information about its composition and concentration.

With the development of electronic components and its low cost, it has become possible to design simple spectrophotometers for educational and research purposes.

These systems offer an affordable alternative to laboratory instruments while helping users better understand the principles of optical measurements and spectroscopy in general.

In this work, a UV-Vis spectrophotometer prototype was designed and tested.

Its performance was evaluated by measuring the absorption spectra of iodine (I_2) and potassium permanganate ($KMnO_4$) solutions at different concentrations and comparing the obtained results with those from a Shimadzu UV-3600 spectrophotometer and literature data. This comparison allows the evaluation of the prototype's accuracy and its ability to detect the expected spectroscopic behavior of the studied compounds.

CHAPTER 1: SPECTROSCOPY

1.1 Introduction

Spectroscopy is the study of how electromagnetic radiation interacts with matter. It examines how materials absorb, reflect, scatter, or emit light under various conditions.

This field began in the 17th century when Sir Isaac Newton did his experiments with optics. By passing white sunlight through a glass prism, he demonstrated that white light splits into a continuous spectrum of colors, proving that it is actually a combination of distinct components. For a long time, scientists focused only on the visible light, during the 19th and 20th centuries, the study of spectroscopy advanced to include the other regions of the spectrum, such as X-rays, microwaves, radio waves, and infrared radiation.

Researchers eventually discovered that every chemical element and molecule interacts with specific wavelengths in a unique way, which acts like a "fingerprint." Because of this discovery, spectroscopy evolved from a simple visual observation into a non-destructive analytical technique allowing scientists to determine the identity and the concentration of a sample without needing to break its chemical structure.

1.2 The Electromagnetic Spectrum

To understand how spectroscopy work, we need to analyze the electromagnetic spectrum. Light is considered to have a dual nature: acts both as a wave traveling through space and as a stream of small energy packets called photons. When we study light as a wave, it is described by two characteristics: wavelength and frequency. Wavelength is the distance between the peak of one wave and the next. Frequency describes how many wave cycles pass a specific point in one second.

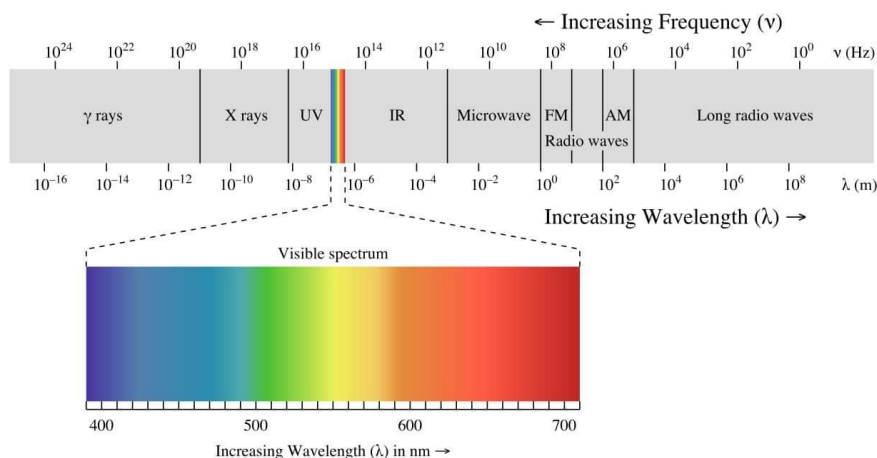


Figure 1 : the electromagnetic spectrum [15]

From an energy perspective, every photon carries a specific amount of energy that depends on its frequency. High-frequency light has short wavelengths and carries a large amount of energy, while low-frequency light has long wavelengths and carries much less energy. When light shines on an object, absorption occurs only if the energy of the incoming photon must exactly match the energy required by the target molecule.

Because of this condition, different parts of the spectrum cause unique physical changes in matter. For example, high-energy UV-Vis light has enough energy to excite electrons into higher-energy levels. Moving down to lower-energy mid-infrared (IR) light, the energy is not strong enough to move electrons, but it is enough to make covalent bonds vibrate, stretch, and bend. Even lower-energy waves, such as microwaves, simply cause polar molecules to rotate in place. [1]

1.3 Ultraviolet-Visible (UV-Vis) Spectroscopy

UV-Vis spectroscopy is a technique used to measure how much light a liquid sample absorbs as a beam passes through it. The technique focuses on two parts of the electromagnetic spectrum: the ultraviolet region (190 to 400 nm) and the visible region (400 to 800 nm), which contains the colors visible to the human eye.

When a molecule absorbs a photon, the energy excites an outer valence electron from its stable, low-energy ground state to a higher-energy, anti-bonding orbital. Because this process involves moving electrons between these energy levels, UV-Vis is often called electronic spectroscopy.

During a standard lab test, a spectrophotometer compares two light beams. It measures the intensity of light before it enters the sample (reference) I_0 and compares it to the intensity after it passes through the sample I . The results are generally shown as either Transmittance or Absorbance.

Transmittance (T): percentage representing how much light passed through the sample without being absorbed. Represented by

$$T = \frac{I}{I_0} \times 100$$

Absorbance (A): uses a logarithmic scale to measure the amount of light absorbed by the chemical. Represented by

$$A = -\log(T)$$

The chemists generally prefer using absorbance because it has a linear relationship with the chemical's concentration. This makes it easier to use in calibration graphs and mathematical equations compared to the non-linear nature of transmittance [1].

1.3.1 Solvent Effects

The liquid solvent used to dissolve a sample affects the shape, position, and height of its spectral peaks. This happens because solute molecules interact with surrounding solvent molecules through forces like dipole-dipole attractions and hydrogen bonding. These interactions stabilize either the ground state or the excited state of the solute, which changes the internal energy gap. The direction of this shift depends on the type of electronic transition [1].

Solvents also determine how detailed your peaks look:

Non-polar solvents (like hexane or cyclohexane) do not form strong bonds with the solute. This allows the details of internal molecular vibrations to appear as sharp, small sub-peaks on the graph [1].

Polar solvents (like water) form a constantly changing hydrogen-bond network. These strong interactions broaden the vibrational signals, making individual peaks overlap and appear as one broad, smooth band instead of separate peaks [1].

1.3.2 Molecular Orbitals and Transitions

1) Electronic Transitions in UV-Vis Spectroscopy

The absorption of ultraviolet and visible radiation is related to the excitation of valence electrons within a molecule. In organic molecules, these electrons occupy different types of molecular orbitals, including bonding orbitals (σ and π) and non-bonding orbitals (n). Higher-energy antibonding orbitals (σ^* and π^*) are normally unoccupied. When a molecule absorbs UV-Vis radiation, an electron can be promoted from an occupied orbital to an unoccupied antibonding orbital.

The main electronic transitions observed in UV-Vis spectroscopy are:

I. Sigma to Sigma-Star Transition ($\sigma \rightarrow \sigma^*$)

This transition involves the excitation of an electron from a sigma (σ) bonding orbital to a sigma antibonding (σ^*) orbital. Since sigma bonds are very stable, this transition requires a large amount of energy and usually occurs at wavelengths below 200 nm.

II. Non-Bonding to Sigma-Star Transition ($n \rightarrow \sigma^*$)

This transition occurs when an electron from a non-bonding orbital is moved to a sigma antibonding orbital. It is usually observed in compounds containing heteroatoms like oxygen, nitrogen, or halogens. The absorption occurs in the range of 160–260 nm.

III. Pi to Pi-Star Transition ($\pi \rightarrow \pi^*$)

In this transition, an electron is excited from a pi (π) bonding orbital to a pi antibonding (π^*) orbital. It is characteristic of unsaturated compounds containing double or triple bonds and generally appears between 200 and 500 nm.

IV. Non-Bonding to Pi-Star Transition ($n \rightarrow \pi^*$)

This transition involves the promotion of a non-bonding electron to a pi antibonding orbital. It is generally linked with functional groups containing both lone pairs and π bonds. The absorption band is typically observed in the 250–600 nm region.

Among these transitions, the $\pi \rightarrow \pi^*$ and $n \rightarrow \pi^*$ transitions are particularly important in UV-Vis spectroscopy because they involve functional groups that are frequently met in analytical applications and absorb within the operating range of most UV-Vis spectrophotometers [2]

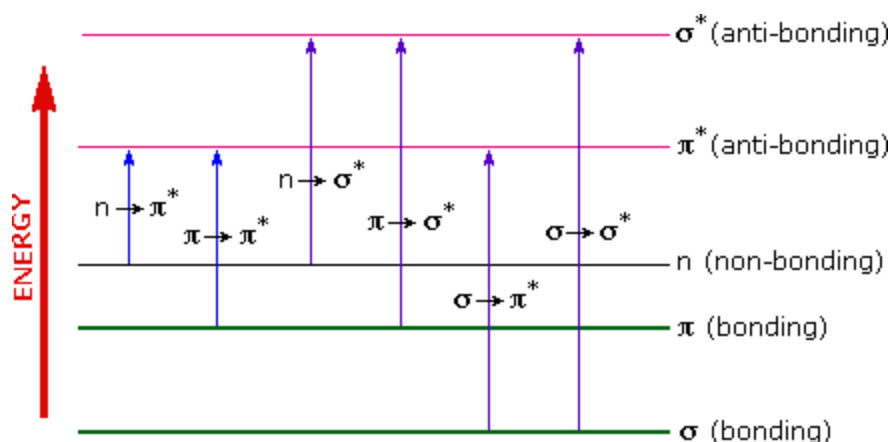


Figure 2 : Energy Level Diagram Showing Possible Electronic Transitions in UV-Vis Spectroscopy.

1.3.3 Chromophores and Shifts

The specific structure of an organic molecule determines its UV-Vis absorption spectrum. To understand how parts of a molecule change its color and light interactions, scientists use:

Chromophore: A functional group or part of a molecule responsible for absorbing light and creating a signal. Examples include isolated double bonds, nitro groups, or benzene rings [2].

Auxochrome: is a functional group that does not absorb light by itself, but can influence the absorption characteristics of a chromophore when attached to it. It modifies the electron distribution in the molecule, which can affect the intensity and position of absorption bands [2].

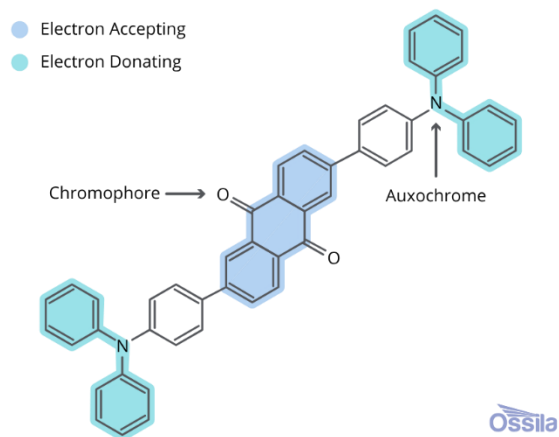


Figure 3 : Chromophore and auxochrome.

Changes in molecular structure can lead to variations in absorption spectra. These changes are generally observed as shifts in wavelength or intensity of absorption bands [2].

1.4 Analysis of the ISO 105-Z10 Standard:

The standard **ISO 105-Z10** defines an objective method for evaluating the relative color strength of a dye in liquid solution by comparing it with a standard reference sample. The color strength represents the natural ability of a dye to produce color on a material.

Traditional methods mostly depend on visual observation or reflectance measurements of dyed fabrics. However, these techniques are often influenced by various factors such as substrate quality, temperature, and chemical fixation conditions. To minimize the effect of these experimental variables, this standard uses absorption spectrophotometry in a liquid medium instead [3].

1.4.1 Application of the Beer-Lambert Law

The fundamental core of this standard relies entirely on the validation and mathematical application of the **Bouguer-Lambert-Beer law**.

The Fundamental Equation

When a monochromatic light beam travels through a homogeneous and non-scattering solution, its intensity decreases exponentially depending on the solute concentration and the optical path length. The standard describes this logarithmic relationship using the following equation:

$$A = \log_{10} \left(\frac{1}{t} \right) = \epsilon c d$$

- **Absorbance (A):** dimensionless parameter representing optical density.
- **Transmittance (t):** The ratio of transmitted light to incident light, expressed as a decimal fraction from 0 to 1,0.
- **Specific Extinction Coefficient (ε):** is the specific extinction coefficient, in square centimeters per gram.
- **Concentration (c):** is the dye concentration, in grams per cubic centimeter.
- **Optical Path Length (d):** s the cell path length, in centimeters.

1.4.2 Conditions for Law Validity

The linear relation between absorbance and concentration is not strictly valid in all states.

Which means, the standard defines strict physical conditions that must be satisfied during experimentation:

- **Absence of light scattering:** The dye solution must be completely free from turbidity, colloids, micro-precipitates, or air bubbles. Any dispersed particles can cause light scattering instead of pure absorption, that can lead to errors in the measured absorbance value.
- **Similarity of spectral profiles:** The absorption spectra of the test sample and the reference standard must be very similar, or ideally identical, across the visible range (typically 400 to 700 nm). This method cannot be reliably applied to colorants that show different spectral shapes or additional absorption peaks [3].
- **The solute concentration:** It should be very low to ensure the Beer-Lambert law validity (dilute solution).

1.4.3 Classification and Selection of Colorants

The protocol is highly flexible and can be applied to several chemical classes of dyes:

Water-soluble dyes: This category includes acid, basic (cationic), direct, and reactive dyes, which are prepared in demineralized water. The standard states that acid and basic dyes show a strong correlation between solution measurements and final substrate color, as they fully exhaust onto the fibers [3].

• **Water-insoluble dyes:** This category mainly includes disperse dyes used for synthetic fibers. As they are not soluble in water, they must first be completely dissolved in an organic solvent such as methanol, ethanol, acetone... to break down molecular aggregates, before being diluted in an aqueous medium under controlled conditions [3].

1.4.4 Experimental Conditions and Protocol

To keep experimental uncertainty within a confidence interval, the standard imposes strict laboratory conditions:

Dissolution and Chemical Stabilisation

- **Temperature Control:** Water-soluble dyes are preferably dissolved by heating to ensure complete molecular dissociation.

Chemical Additives:

- **pH adjusters:** Strong or weak acids, bases are used to control and stabilize the ionic state of the dye molecules.
- **Sequestering agents:** These are added to remove or neutralize trace metal impurities that could interfere with the measurements.
- **Surfactants / dispersing agents:** Non-ionic compounds are recommended as dispersing agents to prevent dye molecules from forming aggregate

Optimal Absorbance Window

The prepared solution must be diluted so that, at the wavelength of maximum absorption (λ_{max}), the measured transmittance remains between 10% and 60%. This operating range ensures a linear response of the detector, preventing distortion from stray light at low transmittance and poor signal-to-noise ratio at high transmittance [3].

1.4.5 Spectrophotometric Measurements

Spectrophotometric measurements must be performed immediately after solution preparation to prevent any time related degradation. Light-sensitive dyes must be manipulated in low-light environment [3].

The standard uses cuvettes with an optical path length (d) of 5 mm or 10 mm. Depending on the instrument sample holder.

Data acquisition can be carried out using:

- **Double-beam spectrophotometer:** This system automatically corrects for the solvent background by measuring a reference cell containing only the solvent at the same time as the sample cell.
- **Single-beam spectrophotometer:** This method requires calibration with a solvent blank (A_B) before measuring the sample absorbance (A_R). The actual absorbance of the sample is then obtained by subtracting the blank value:

$$A = A_R - A_B$$

The absolute difference in absorbance between the test sample and the reference dye must not exceed 30%. This requirement ensures that both measurements remain within the same linear response range of the photodiode detector [3].

1.4.6 Relative Color Strength (F_s) Calculation

The relative color strength is expressed as a percentage ratio, with the reference dye defined as the standard baseline at 100%. The main equation is:

$$F_s = \frac{f_{k2}}{f_{k1}} \times 100$$

where f_{k1} and f_{k2} represent the absolute color strength values of the reference dye and the test sample, respectively. [3]

By applying the Beer–Lambert law and introducing concentration and path length terms, the equation can be rewritten in a more practical form:

$$F_s = \frac{\varepsilon_2}{\varepsilon_1} \times 100 = \frac{A_2 \cdot c_1}{A_1 \cdot c_2} \times 100$$

where index 1 corresponds to the reference dye and index 2 corresponds to the test dye.

When both solutions are prepared at the same concentration $c_1 = c_2$, the expression simplifies to the absorbance ratio:

$$F_s = \frac{A_2}{A_1} \times 100$$

This simplified form directly relates the measured absorbance ratio to the relative commercial strength of the dye batch [3].

1.5 Colorimetry

Color is an important qualitative property of a substance, linked to its ability to absorb or reflect light.

The human eye does not see the light absorbed by a sample. Instead, we see its complementary color the remaining wavelengths of white light that pass through or reflect

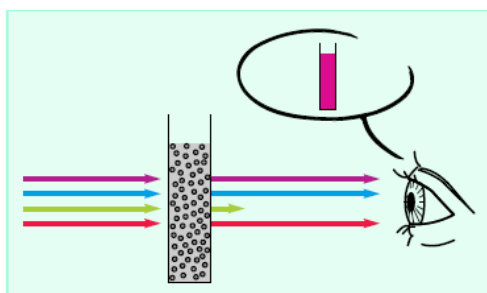


Figure 4 : Interaction of Polychromatic Light with a Sample.

1.6 Absorbance, and Complementary Colors

When white light passes through a sample, chemical compounds selectively absorb certain wavelengths depending on their molecular structure.

The wavelengths that are not absorbed are transmitted and reach the eye. Since the eye receives the combination of transmitted wavelengths, the observed color refers to the complementary color of the absorbed light.

For example, if a solution absorbs in the yellow region (around 580–595 nm), those wavelengths vanish from the transmitted light, and the solution appears blue.

This relationship between absorption and complementary color is crucial in UV-Visible spectroscopy, it help in determination of the wavelength at which a spectrophotometer should operate to accurately measure the concentration of a sample.

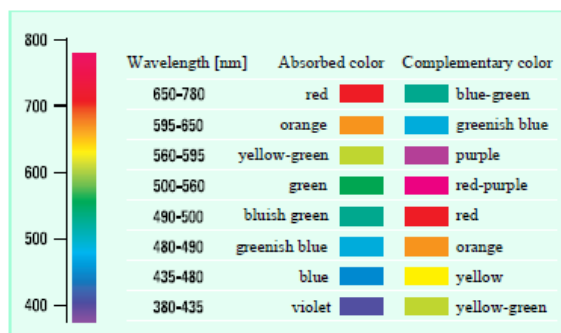


Figure 5 : Complementary Colors

1.7 Colorimetry standards:

The “Commission Internationale de l’Éclairage” CIE color system represents any visible color using three tristimulus values (X, Y, Z), derived from the human eye’s sensitivity to different wavelengths of light.

These values are then projected onto a chromaticity diagram, which maps all perceivable colors in a two-dimensional space.

The diagram shows relationships between colors, with spectral colors forming the outer boundary (the spectral locus), while mixtures of colors are located within the interior area.

This system, developed by (CIE), provides a standardized and objective method for describing color across different devices and lighting conditions [4].

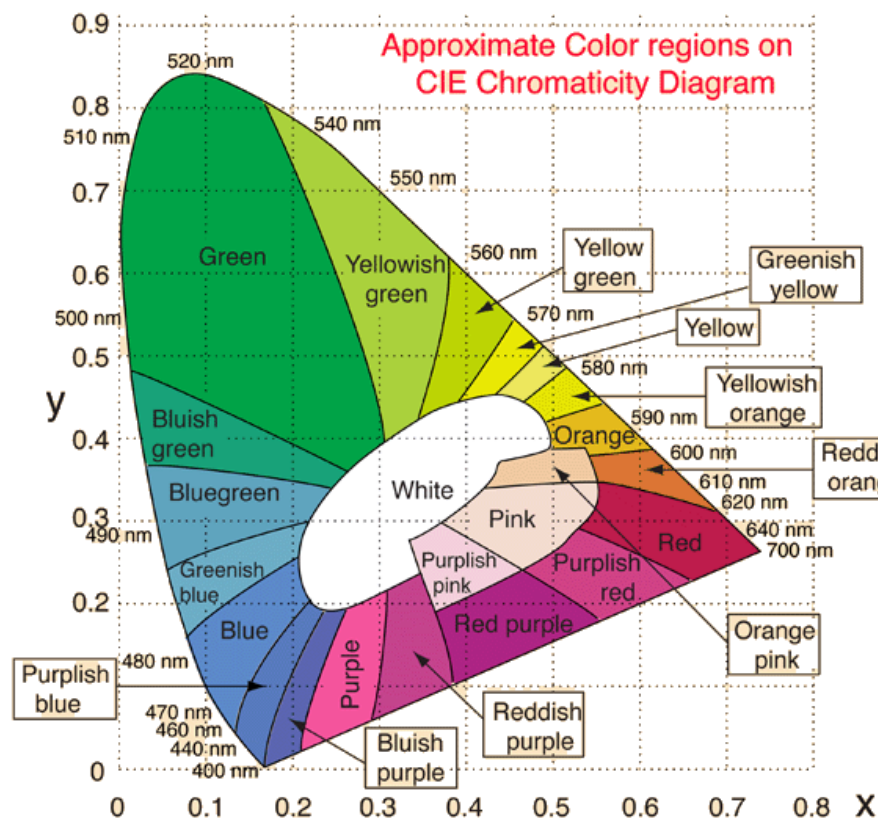


Figure 6 : CIE 1931 Chromaticity Diagram Mapping Visible Wavelengths [4].

1.8 Infrared (IR) Spectroscopy

Infrared (IR) spectroscopy focuses on the low-energy section of the electromagnetic spectrum straight after the visible red range. Unlike UV-Vis light, infrared radiation does not have enough energy to move valence electrons. Instead, it provides an enough amount of energy needed to excite vibrational transitions within chemical bonds [5,6].

The infrared spectrum is divided into three main zones:

Near-Infrared (NIR) (12,800–4,000) cm^{-1} :

This region is closest to visible light. It is characterized by, high-frequency signals and is widely used for rapid, non-destructive quality control in industries like agriculture, food, and pharmaceuticals.

Mid-Infrared (Mid-IR) (4,000–400) cm^{-1} :

This is the most used region for chemistry laboratories. It captures the fundamental structural vibrations of almost all organic molecules, making it ideal for identifying specific functional groups.

Far-Infrared (FIR) <400 cm^{-1}

This low-energy region is primarily used to analyze the vibrations of heavy atoms [5,6].

1.8.1 Models of Bond Vibration

The Symmetrical View

we can model a covalent chemical bond by imagining two weights connected by a flexible spring. When the atoms are at rest, they stay at a stable bond length. If you pull the atoms apart or push them together, a restoring force immediately pulls them back to their original position [5,6].

This model gives us two simple rules to predict where absorption peaks will appear on an IR spectrum:

Bond Stiffness:

Stronger bonds act like stiffer springs. Since stiffer springs vibrate faster, double bonds ($\text{C}=\text{C}$) vibrate at higher frequencies than single bonds ($\text{C}-\text{C}$), and triple bonds ($\text{C}\equiv\text{C}$) vibrate at even higher frequencies. [5,6].

Atomic Mass:

Lighter atoms move much faster than heavy. Because of this, bonds involving light hydrogen atoms ($\text{C}-\text{H}$, $\text{O}-\text{H}$ or $\text{N}-\text{H}$) vibrate at much higher frequencies than bonds between heavier elements, such as carbon and chlorine ($\text{C}-\text{Cl}$) [5,6].

1.8.2 Vibrational Modes

Diatomic molecule (two atoms bonded) has only one way to vibrate: stretching the bond back and forth. larger polyatomic molecules can move in many directions [5,6].

We can determine the total number of fundamental vibrations using the total number of atoms (N):

Every atom has three "degrees of freedom" (it can move along the X, Y, and Z axes), giving a molecule $3N$ total movements.

We can resume the movements of the molecule as a whole (translation and rotation):

- **Linear molecules** (like CO_2) have 3 translational and 2 rotational modes, leaving $(3N-5)$ vibrational modes.
- **Non-linear molecules** (like H_2O) have 3 translational and 3 rotational modes, leaving $(3N-6)$ vibrational modes.

These complex movements are divided into two main categories:

Stretching Vibrations:

Symmetric Stretching: Neighboring bonds lengthen and shorten in perfect synchronization.

Asymmetric Stretching: One bond shortens while the neighbor lengthens, breaking the synchronization.

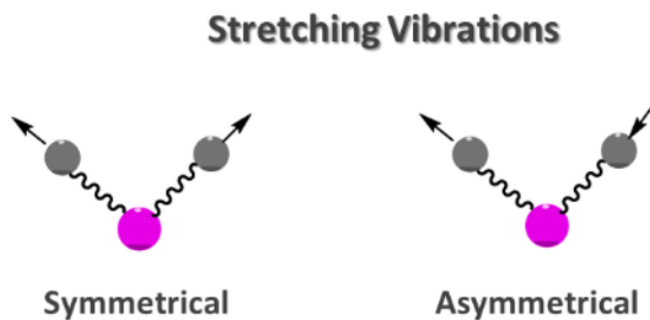


Figure 7 : stretching vibrations [19]

Bending Vibrations

Because changing a bond angle requires less energy than stretching a bond, these vibrations appear at lower positions on the graph (usually below $1,500\text{ cm}^{-1}$, in an area known as the "fingerprint region" [5,6].

" They are classified by whether the atoms stay in the same plane:

In-plane:

Scissoring: Atoms swing toward/ away from each other like scissor blades.

Rocking: Bonds swing back and forth together like a pendulum.

Out-of-plane:

Wagging: Atoms swing up and down together, above and below the molecule's plane.

Twisting: One atom swings up above the plane while its neighbor swings down below it [5,6].

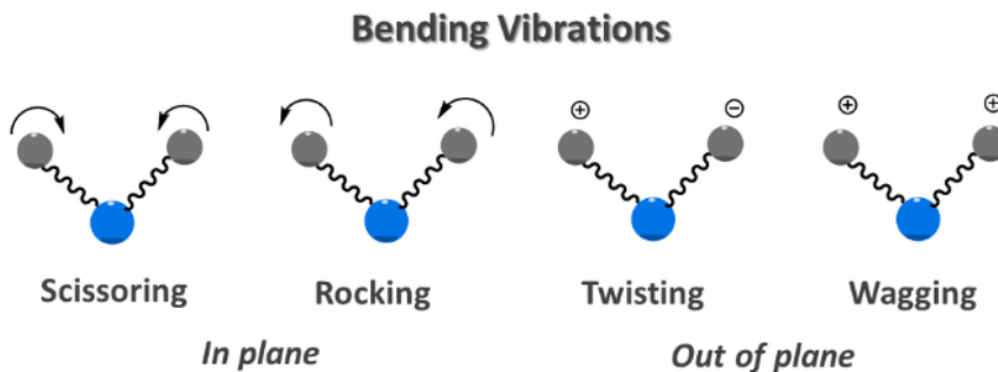


Figure 8 bending vibrations [19].

1.8.3 IR Selection

For a vibration to be “infrared active”, it must cause a change in the molecule’s **electric dipole moment** [5,6].

A dipole moment occurs when there is an uneven distribution of electrical charge, creating a partial positive side and a partial negative side.

For example, Carbon dioxide (CO₂). Being a linear molecule, it has four fundamental vibrations:

Symmetric Stretching:

Both oxygen atoms move away from the central carbon simultaneously. As a result, the molecule remains perfectly symmetrical, and the charge distributions from each side cancel one another out. Since the dipole moment does not change and remains zero, this vibration is IR inactive and therefore does not appear in the spectrum [5,6].

Asymmetric Stretching:

One oxygen moves closer to the carbon while the other moves further away. This movement breaks the symmetry, causing the electrical charge to shift creating a large, IR active absorption.

Bending Modes:

The molecule bends from its linear structure into a “V” shape. This deformation breaks the molecular symmetry and generates a temporary dipole moment, making these vibrational modes IR active [5,6].

1.9 Instrumentation

1.9.1 Architectures

Spectrophotometers are analytical instruments made to measure how much light intensity a sample absorbs. There are three primary optical designs:

1. Single-Beam Spectrophotometers

This is the simplest and oldest design. Light passes from a lamp, through a monochromator (wavelength selector), and then through the sample to the detector. To use it, you must manually measure a "blank" (pure solvent) first, then measure it for the real sample [1,7].

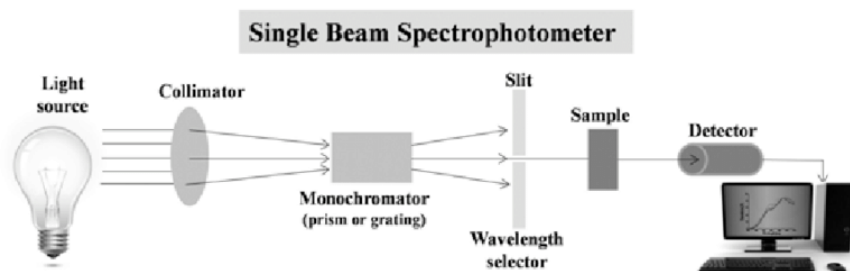


Figure 9 : Single beam spectrophotometer [20].

2. Double-Beam Spectrophotometers

To fix the issue of drift, this design splits the light beam into two paths using a fast-spinning mirror called a chopper.

One beam passes through the sample; at the same time the other beam passes through a reference cell (solvent only).

The CPU compares the sample and reference signals in real time. This process cancels out fluctuations in the light source or detector temperature, making it ideal for slow, precise scans over wide wavelength ranges [1,7].

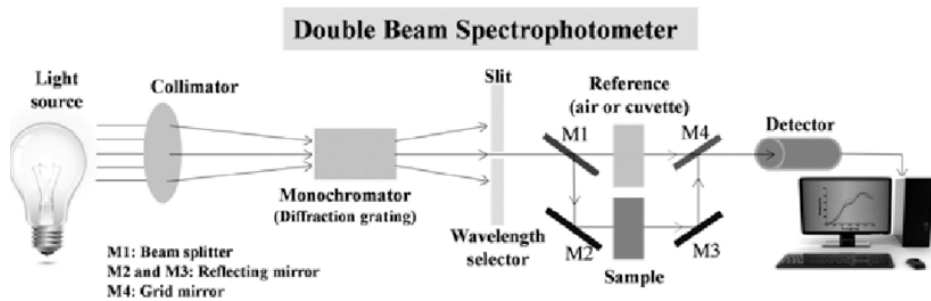


Figure 10 : Double beam spectrophotometer [20].

3. Diode-Array Spectrophotometers

Instead of filtering light before the sample, full-spectrum white light passes through the sample first.

After passing through the sample, the light hits a fixed diffraction grating, which spreads all wavelengths across a chip containing hundreds of independent light sensors called **photodiodes**.

Because there are no moving parts, these machines can capture an entire spectrum (190 to 1100 nm) in a fraction of a second. This makes them the best choice for tracking fast chemical reactions in real time [1,7].

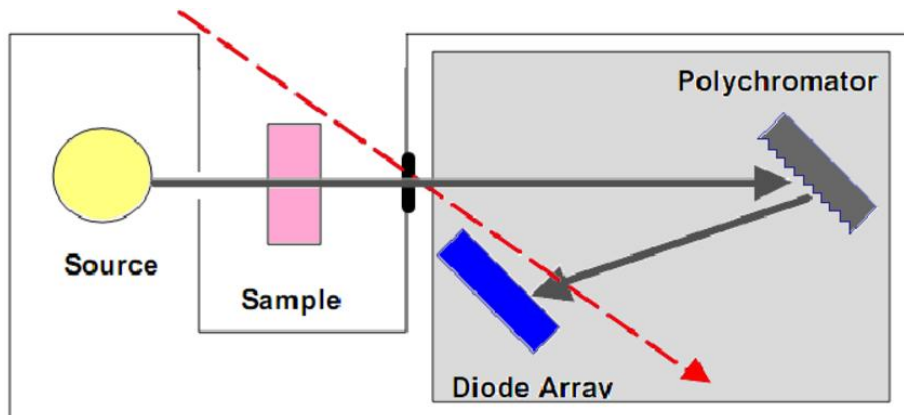


Figure 11 : Photodiode Array system.[21]

1.9.2 Radiation Sources

To ensure accurate measurements, a high-quality spectrometer requires a light source that provides a stable continuous stream of light across the spectral range. Because no single source can cover every wavelength efficiently, engineers used a combination of specialized lamps:

Deuterium Arc Lamps:

These are used specifically for the ultraviolet (UV) region (190 nm to 400 nm). They function by creating a high-voltage arc within a bulb filled with deuterium gas, producing a smooth and consistent UV light output [1,5].



Figure 12 : Deuterium lamp.

Tungsten-Halogen Lamps:

These lamps cover the **visible and near-infrared (NIR) regions** (320 nm to 2,500 nm). they heat a tungsten filament inside a halogen gas envelope to generate a broad spectrum of white light [1,5].

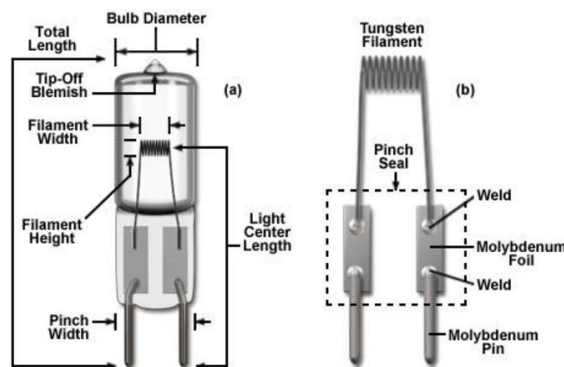


Figure 13 :Halogen lamp [22].

Globar:

This is used for the **mid-infrared (Mid-IR) region**. A Globar consists of a solid silicon carbide rod heated electrically until it glows, emitting a powerful and steady stream of thermal infrared radiation. [1,5].



Figure 14 : Silicon carbide rods



Figure 15 : Nernst glower [23]

Mercury arc lamps:

Used for the Far-IR region. A Mercury Arc Lamp is a glass tube filled with high-pressure mercury vapor. When electricity passes through it, it creates a hot, glowing plasma that acts as a powerful source of light, giving the instrument the extra energy it needs to work in the Far-IR range [1,5].



Figure 16 : Mercury arc lamp.

1.9.3 Wavelength Selection

To perform accurate spectroscopy, an instrument must separate the broad mixture of wavelengths from the light source into individual narrow wavelength.

This task is performed by a monochromator, which uses an entry slit, a dispersing element, and an exit slit to isolate specific wavelengths [1,6].

The most important part of a modern monochromator is the **reflective diffraction grating**.

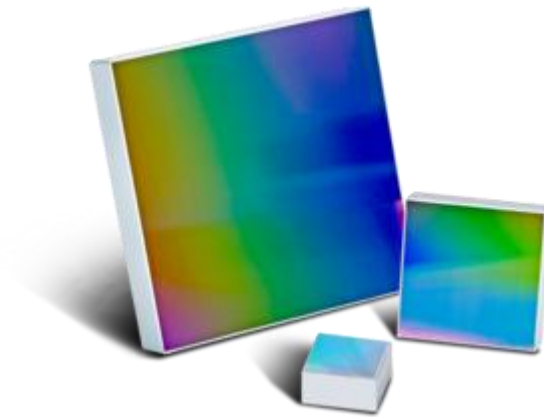


Figure 17 : diffraction-grating [24]

A diffraction grating is a highly polished metal surface engraved with thousands of microscopic, parallel grooves often more than 1,200 per millimeter. When white light hits these grooves, this process occurs:

1. **Light Scattering:** Each small groove scatters the incoming light waves in every direction.
2. **Interference:**

Because light behaves as a wave, the scattered waves undergo **constructive interference** (where waves reinforce each other) and **destructive interference** (where waves cancel each other out) as they reflect off the surface [1,6].

3. **Angle Selection:** Based on the laws of wave geometry, waves only reinforce each other at specific angles, depending on their wavelength [1,6].

Following the equation:

$$d(\sin\beta - \sin\alpha) = m\lambda$$

- d : The grating constant (distance between lines on the DVD).
- β : The angle of the diffracted light (exit angle).
- α : The angle of the incident light (entry angle, usually 45°).
- m : The diffraction order (typically 1 for this system).
- λ : The wavelength of the light being measured

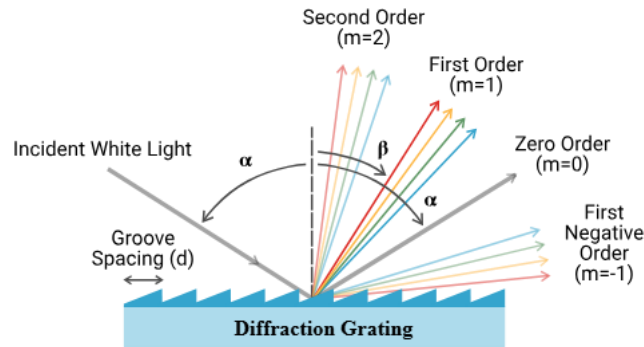


Figure 18 : Principle of Operation of a Reflection Grating [25].

By using a precise motor to rotate the diffraction grating, the instrument can control which wavelength is directed through the exit slit toward the sample. This allows the system to scan through different colors one at a time for precise analysis [1,7].

Fourier Transform Infrared (FTIR) Spectroscopy

While dispersive instruments use a monochromator to scan wavelengths sequentially,

FTIR spectrophotometers use a **Michelson interferometer** to capture the entire infrared spectrum simultaneously. In this model, the monochromator's slit and grating are replaced by an interferometer consisting of a beam splitter and two mirrors (one fixed, one moving) [1,6].

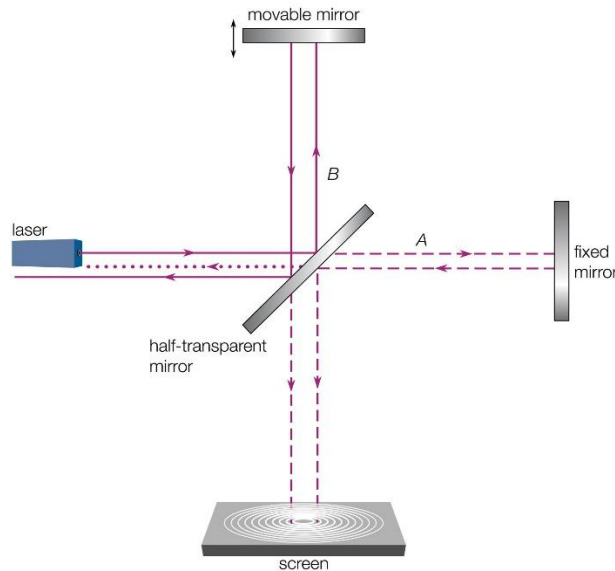


Figure 19: Michelson-interferometer principle [26]

As the mobile mirror moves, the light beams recombine to create an **interferogram**—a complex signal containing all infrared frequencies. Then the CPU performs a **Fourier Transform** to decode this signal into a spectrum. Unlike dispersive instruments that rely on a narrow slit to physically filter light (FTIR uses an **aperture** and relies on the mirror's moving distance to achieve high spectral resolution. This allows FTIR to offer superior speed, sensitivity, and precision, making it the industry standard for infrared analysis [1,6].

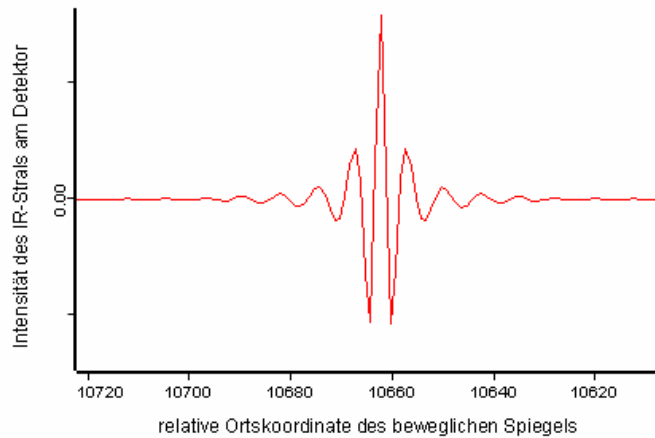


Figure 20 :Interferogram signal [27].

1.9.4 Photo-Detectors

After light passes through a sample, a detector must convert this light intensity into an electrical signal, that a computer can process. Because light spans different energy levels, specific detectors are required for different spectral ranges:

Photomultiplier Tubes (PMT):

This equipment is highly sensitive detectors used for UV-Vis light. Inside a vacuum tube, a photon hits a light-sensitive plate and knocks an electron loose. After that this electron strikes a series of charged metal plates, called **dynodes**, triggering a "cascading" effect.

A single photon impact is multiplied into a wave of millions of electrons, creating a strong electrical current. This makes PMTs ideal for detecting extremely faint light [1,5].



Figure 22 :PMT[28]

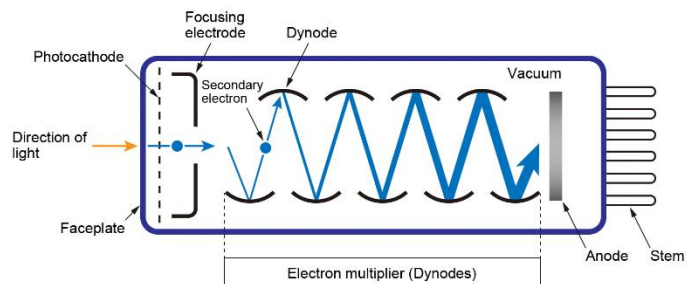


Figure 21 : photomultiplier_tubes [28]

Charge-Coupled Devices (CCD):

multi-channel detectors similar to the ones in digital cameras. A CCD chip is made of a grid of thousands of microscopic, light-sensitive pixels.

When light hits a pixel, it traps an electronic charge proportional to the light intensity. The device reads these charges line by line, allowing the spectrometer to capture an entire spread of wavelengths at once [1,5].

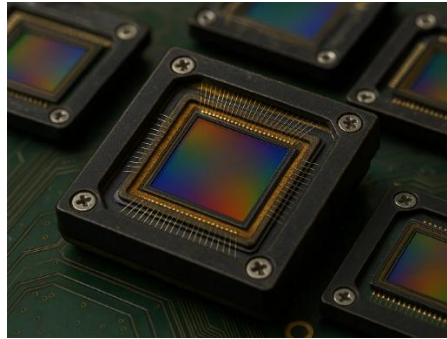


Figure 23 : Charge-coupled-devices [29]

Infrared Detectors (DTGS):

IR instruments rely on thermal detection. A widely used option is the Deuterated Triglycine Sulfate (DTGS) pyroelectric detector, which consists of a crystal placed between two electrodes. When infrared radiation strikes the crystal, it causes a slight temperature change (increase), altering its electrical polarization and producing a measurable current proportional to the radiation intensity.

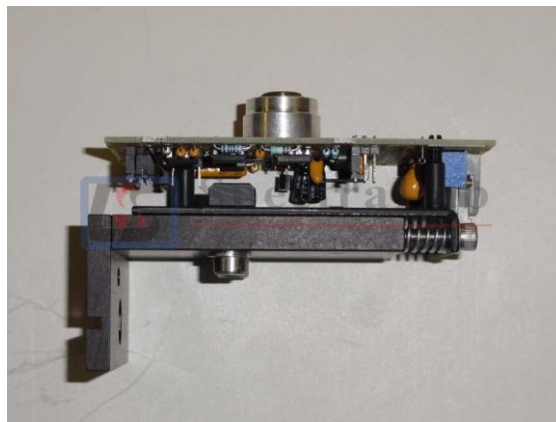


Figure 24 :DTGS detector [30].

Quality Parameters

To ensure that, a spectrometer is accurate and reliable, there are three key performance indicators:

1. Spectral Resolution and Bandwidth

Spectral resolution is the instrument's ability to separate between two close absorption peaks. This is primarily determined by the spectral bandwidth, which refers to the width of the color band passing through the monochromator's exit slit :

- If the slit is too wide, the sample is hit with a mixture of wavelengths rather than a pure color then Peaks appear artificially flattened, short, and broad, losing detail.
- For high-precision work, the bandwidth must be narrower than the natural width of the sample's absorption peaks.

2. Stray Light Percentage

Stray light consists of unwanted photons that are scattered or reflected within the instrument case and reach the detector without passing through the sample. This introduces systematic errors and causes results errors [1].

- How to measure it: by obtaining a measurement while the light source is off. Any signal registered by the detector represents the percentage of stray light leaking into the system.

3. Signal-to-Noise Ratio (SNR)

The SNR compares the strength of the experimental data (the signal) against the background noise produced by the instrument's internal electronics.

$$\text{SNR} = \frac{\text{Signal Strength}}{\text{Background Noise Level}}$$

Maintaining a high SNR is essential when working with highly diluted samples. If the SNR is low, the weak absorption signal from a dilute sample becomes hidden by the electronic noise, making accurate measurement impossible [1].

1.10 Spectroscopy Application

Spectroscopy is found in various fields due to its fast and non-destructive characteristics [8,9]:

1. Agro-Food Industry

- **Fruit and Vegetable Quality:** Measures internal parameters like moisture, sugar content (soluble solids), and ripeness levels in crops like grapes, mangos, and apples.
- **Meat and Fish Analysis:** Used to assess the conservation state and fat content of products.
- **Soil and Viticulture:** Helps analyze soil composition—such as nitrogen, organic matter, and moisture—to support farming and vineyard management.

2. Safety, Security, and Law Enforcement

Portable spectrometers are essential for rapid, on-site identification of illegal materials.

- **Threat Detection:** Used by emergency responders to identify explosives, chemical weapons, or other dangerous materials.
- **Narcotics Identification:** Law enforcement uses these tools for the immediate, on-site checking of illegal substances such as drugs.
- **Food Authenticity:** Detects food fraud to verify that products like olive oil or meat are healthy.

3. Medical and Diagnostic Applications

Spectroscopy is moving into portable and low-resource settings to improve healthcare.

- **Medical Diagnostics:** Non-invasive quantification of critical metabolites, such as glucose.
- **Pharmaceuticals:** Used for Material Identification to ensure the quality and authenticity of chemical ingredients in medicine.

4. Industrial and Environmental Monitoring

- **Material Sorting:** Quickly identifies and sorts different types of plastics, polymers, and metals for recycling
- **Environmental, Water Quality:** Detecting pollutants such as phosphates, nitrates, and heavy metals in water sources
- **Soil Analysis:** Measuring organic matter, nitrogen levels, and moisture content directly in the field to optimize fertilization

-



Figure 25 Application of spectroscopy for soil analysis [9].

CHAPTER 2:

Development and Experimental Validation

2.1 Introduction

In the previous chapter, we presented the basic principles of spectroscopy and the operation principle of spectroscopic instruments. These concepts provide the theoretical foundation required to understand how light can be used to analyze chemical substances.

The objective of the current chapter is to develop a low-cost spectrophotometer capable of measuring and recording absorption spectra mainly in the visible region of the electromagnetic spectrum. To achieve this goal, we designed a system based on simple optical and electronic components combined with dedicated software for spectrum acquisition and analysis.

In this chapter, we present the design of the developed device and explain the role of each component in the measurement process. We also describe the calibration procedure used to obtain reliable wavelength measurements. Finally, we evaluate the performance of the spectrophotometer through a series of experiments using different colorant solutions and analyze the obtained results.

2.2 Design of the prototype

Components:

As presented in the previous chapter, a spectrophotometer is made of several components, from the light source to the detector.

For our system, we chose to follow a design similar to the diode-array spectrophotometer design, because of its simple configuration and its ability to acquire an entire spectrum simultaneously.

The light source:

The light source used in this system is a tungsten lamp. Its role is to provide a continuous beam of visible light that passes through the sample before entering the optical system.



Figure 26 : tungsten lamp

The slit:

The slit is positioned after the sample and is made using two parallel razor blades placed close to each other to ensure a narrow width below 1 mm, a narrow slit improves the quality of the emerging beam of light.



Figure 27: razor blades for the slit

Diffraction Grating

The diffraction grating is the component responsible for separating light into its different wavelengths. In this system, we used a piece of DVD as a diffraction grating because its surface contains thousands of closely spaced microscopic grooves, the DVD is tilted at 30° from the detector. When the light reaches these grooves, it diffracts and get separated into different colors, forming a spectrum that can be captured by the detector.



Figure 28: DVD piece containing microscopic grooves.

We selected the DVD because it offers a good compromise between performance, availability, and cost. Compared to a CD, a DVD has a higher track (groove) density (1350 track per mm), which allows better wavelength separation and produces a spectrum with higher resolution.

Although professional diffraction gratings provide better accuracy but they are considerably more expensive and since the objective of this project was to develop a low-cost spectrophotometer, the DVD represented a practical and effective alternative.



Figure 29: DVD

Detector

We put a webcam at the end of the optical path to do the role of the detector. It functions by capturing the entire spectrum diffracted by the diffraction grating and convert it into a digital image. The camera sensor translates each wavelength in the spectrum into a pixel with a specific intensity.

We selected a standard webcam for this project due to its cost, accessibility, and ability to capture the entire visible spectrum simultaneously.



Figure 30: camera as the light detector

The camera is equipped with an OV7670 CMOS sensor, which is suitable for spectroscopic application. Its VGA resolution offers enough pixels to capture a clear spectrum, the sensor is sensitive enough to detect weak signals, making it an affordable and effective choice for the design of our spectrophotometer prototype.

The main specifications of the sensor are listed in Table [12]

Parameter	Specification
Sensor type	CMOS OV7670
Optical format	1/6 inch
Resolution	640 × 480 pixels (VGA)
Pixel size	3.6 μm × 3.6 μm
Sensitivity	1.1 V/lux·s
Image area	2.36 mm × 1.76 mm

2.3 Prototype Design and Dimensions

The components were assembled and placed in a rectangular box of 15 cm length and 8cm height and 10 cm width.

we painted the interior surface of the box with black color to reduce stray light and unwanted reflections that may affect the quality of the obtained spectra.

We determined the design of our optical components through experimentation, with the goal to achieve a clear, defined spectrum while keeping the overall design compact.

The tungsten lamp was placed at 1 cm from the sample holder to ensure the maximum incident light intensity. In addition, a distance of 1 cm between the sample holder and the slit was chosen in order to minimize light loss and maintain a high intensity of the light entering our optical system. A spacing distance of 6 cm between the slit and the DVD diffraction grating ensures an optimal wavelengths diffraction before reaching the detector.

We positioned the DVD at an angle of approximately 30° relative to the camera to ensure the capture of the diffracted spectrum, then we set the distance between the diffraction grating and the

detector to about 3 cm, which provided the best projection of the entire spectrum onto the camera sensor.



Figure 31: prototype interior

2.4 Theremino Spectrometer Software

We used the Theremino Spectrometer as the software for spectrum acquisition of the developed prototype. It is designed to function with the webcam-based spectrometers, allowing direct acquisition of images from the camera and their conversion into spectral data.

The software analyzes the distribution of light intensity along the spectrum and displays the results in real time, making it possible to observe spectral variations almost instantly. [12]

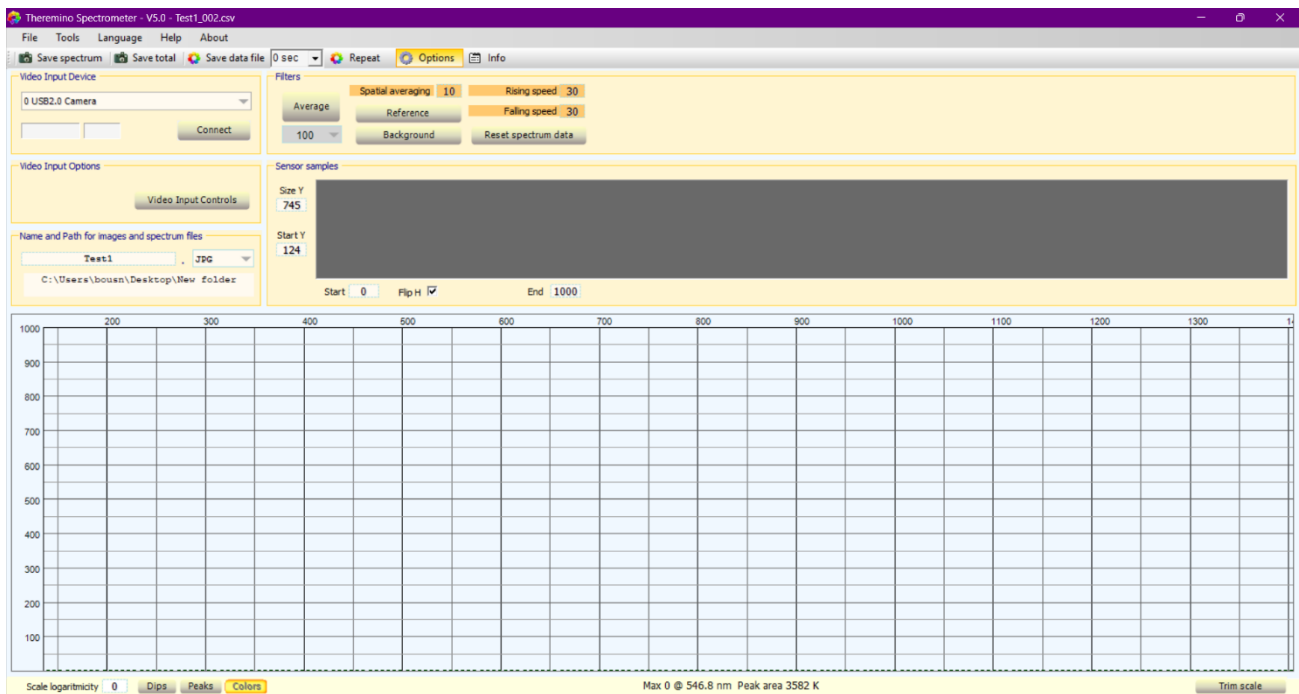


Figure 32: thermino software interface for data acquisition

For webcam applications, Theremino offers several tools like: exposure control, brightness settings, image orientation, and selection of the region of interest used for spectral analysis.

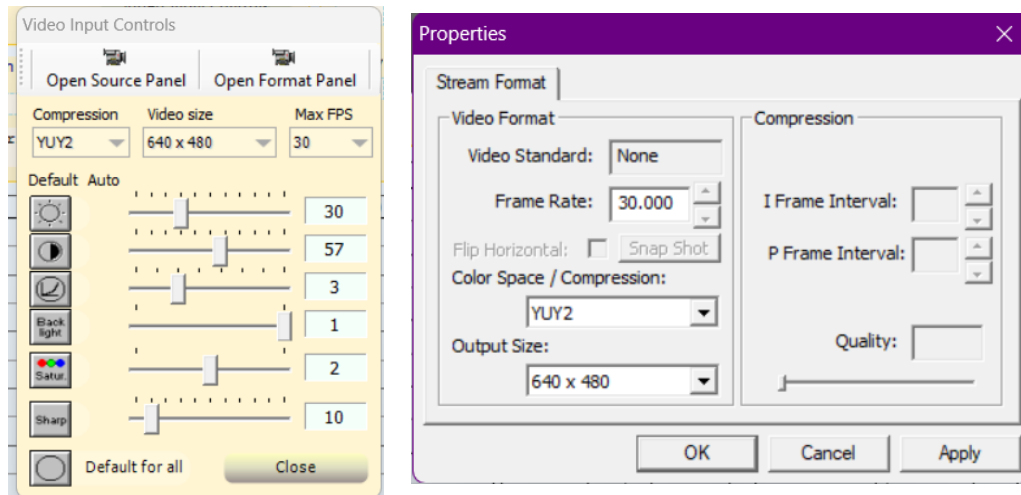


Figure 33 : Tools in thermino software

The software also averages multiple pixel rows of the captured image, which improves sensitivity and reduces noise while minimizing the effects of slight optical misalignments.

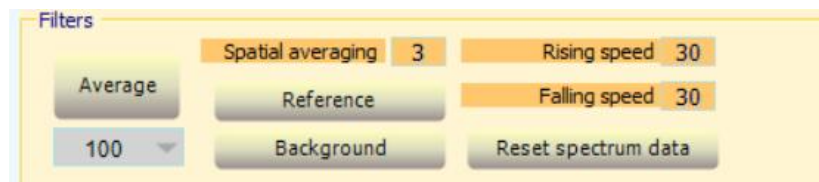


Figure 34 : Options to control the spectrum in thermino

In addition to spectrum acquisition, the software offers several processing and visualization tools like colors, peaks ...

Thermino spectrometer option offers two methods for saving the acquired data. By saving the results as image files for visualization and documentation, or export the data in spreadsheet format (excel) for more processing and analysis.

2.5 The prototype principle of working

The process begins with the tungsten lamp, which emits a continuous spectrum of visible light. This light passes through the sample, where some wavelengths are absorbed and others are transmitted.

The amount of absorbed light depends on the nature and concentration of the sample.

The transmitted light then passes through the slit. The objective of the slit is to narrow the beam of light before it reaches the diffraction grating and helps enhancing the quality of the spectrum

After the slit, the light reaches the DVD diffraction grating. The surface of the DVD contains a large number of microscopic grooves (1350 per 1mm) that diffract the incident light, then different wavelengths are separated and spread out, forming a visible spectrum.

The spectrum is then projected to the webcam sensor. Each wavelength reaches a different position on the sensor (pixels), allowing it to capture the intensity of each part.

Finally, the data is captured and processed using the Theremino Spectrometer software. It converts the recorded data into a graph representing light intensity as a function of wavelengths. After calibration, the spectrum can be used to measure absorption and study the optical properties of the sample.

2.6 Calibration

After assembling the spectrophotometer, and assuring the presence of a proper signal, the next step is the calibration of the system.

The Theremino Spectrometer software includes a built-in wavelength calibration feature called trim scale. This tool allowed us to define reference points on the displayed spectrum and assign their corresponding wavelength values.

We used a compact fluorescent lamp (CFL) as a reference light source. The mercury vapor in the lamp emits lines at known wavelengths (peaks), which we used as reference points to calibrate the wavelength scale in the software [13]



Figure 35: Reference CFL lamp

We used several emission points of the lamp as calibration points, as shown in the figure below. By linking these wavelengths with the corresponding peaks in the spectrum, the wavelength axis of the spectrophotometer was calibrated.

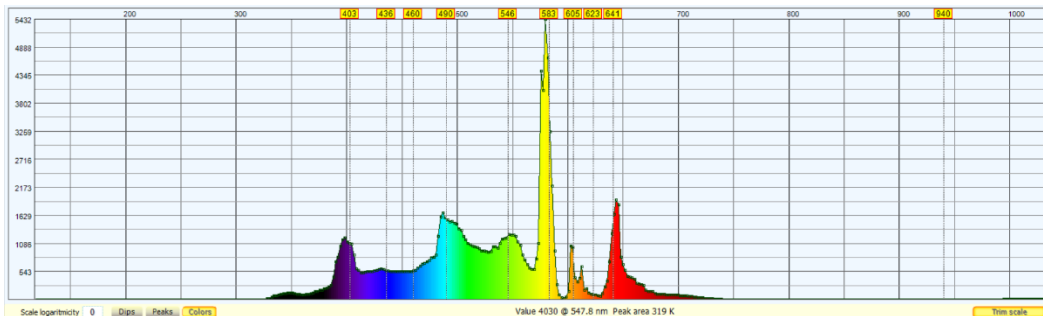


Figure 36: CFL lamp reference spectrum

For the NIR region we used an infrared LED to calibrate the point 940nm. Once the calibration is completed, the prototype is ready for measurements.

2.7 Experimental Methodology and Procedures

1. Preparation of Standard Solutions

To evaluate the performance of the developed spectrophotometer prototype, we prepared standard solutions of potassium permanganate (KMnO_4) and iodine (I_2) at different concentrations.

2. Preparation of Mother Solutions

First, we prepared the mother solutions with a concentration of **$0.001 \text{ mol}\cdot\text{L}^{-1}$** and a final volume of **1000 mL** for each compound.

The needed mass of each solute was calculated using the equation:

$$m = c \times V \times M$$

where:

- m : the mass of the solute (g),
- c : the desired concentration ($\text{mol}\cdot\text{L}^{-1}$),
- V : the solution volume (L),
- M : the molar mass ($\text{g}\cdot\text{mol}^{-1}$).

For KMnO_4 :

$$m = 0.001 \times 1 \times 158.04$$

$$m = 0.1580g$$

For I_2 :

$$m = 0.001 \times 1 \times 253.8$$

$$m = 0.2538g$$

Using this approach, we weighed:

- **0.1580 g of KMnO_4**
- **0.2581 g of I_2**



Figure 37: mass weighing

We dissolved potassium permanganate in distilled water, for the iodine we dissolved it in ethanol due to its limited solubility in water.

We then diluted each solution to a final volume of 1000 mL to obtain the desired concentration of $0.001 \text{ mol} \cdot \text{L}^{-1}$



Figure 38 : Mother solution of KMnO_4

3. Preparation of Daughter Solutions

After preparing the mother solutions, we made diluted (daughter) solutions with target concentrations of **$0.0005 \text{ mol}\cdot\text{L}^{-1}$** and **$0.0001 \text{ mol}\cdot\text{L}^{-1}$** .

The dilution process was based on the volume given by the equation:

$$C_1V_1 = C_2V_2$$

where:

- C_1 is the initial concentration (mother solution)
- V_1 is the volume taken from the mother solution
- C_2 is the final concentration (daughter solution),
- V_2 is the final volume after dilution (daughter solution).

Following the equation we obtained:

$$C_1V_1 = C_2V_2$$

$$V_1 = \frac{C_2V_2}{C_1}$$

For the first concentration **$0.0005 \text{ mol}\cdot\text{L}^{-1}$**

$$V_1 = \frac{0.0005 \times 0.1}{0.001} = 0.05L = 50ml$$

For the second concentration **$0.0001 \text{ mol}\cdot\text{L}^{-1}$**

$$V_1 = \frac{0.0001 \times 0.1}{0.001} = 0.01L = 10ml$$

To prepare the solutions we used :

- **50 mL** of the mother solution to obtain the **$0.0005 \text{ mol}\cdot\text{L}^{-1}$** solution,
- **10 mL** of the mother solution to obtain the **$0.0001 \text{ mol}\cdot\text{L}^{-1}$** solution,

We diluted the calculated volumes in a 100ml of each solvent, then transferred the prepared solutions into labeled containers for storage and further measurements.



Figure 39 : Mother solution and daughter solutions of I_2



Figure 40 : The prepared solutions

2.7.1 Spectrophotometric Measurements

Once the solutions were prepared, we conducted absorbance measurements using both the developed spectrophotometer prototype and a Shimadzu 3600 spectrophotometer available in the Laboratory of Thin Films Physics and Materials for Electronics.

Shimadzu 3600:

We used the **Shimadzu UV-3600**, a UV–VIS–NIR spectrophotometer. It measures absorbance, transmittance, and reflectance of liquid, gas, and solid samples over a wide wavelength range.



Figure 41: Shimadzu UV-3600

1. Detector System

The UV-3600 has a **three-detector system**, which improves accuracy and sensitivity across different wavelength regions. Unlike normal spectrophotometers that use only two detectors, this system uses three:

- **Photomultiplier Tube (PMT):** Used for UV and visible light measurements (about 165–1000 nm).
- **InGaAs Detector:** Used in the near-infrared region (about 700–1800 nm) and helps reduce the noise during detector changes.
- **Cooled PbS Detector:** Used for the far near-infrared region (about 1600–3300 nm) [14].

2. Optical System and Specifications

The instrument works with a **double-beam system** and a **double monochromator**, which improves measurement stability and reduces errors) [14].

Main characteristics of the spectrophotometer are:

- **Wavelength range:** 185 to 3300 nm
- **Resolution:** 0.1 nm
- **Monochromator design:** Pre-monochromator and Czerny–Turner monochromator
- **Light sources:** halogen lamp and deuterium (D₂) lamp

3. Software

The instrument is controlled using **UVProbe software**, which is used for data collection and analysis.

The software allows us to set measurement conditions, record spectra, and process the obtained data. It also includes different modes for analysis such as spectrum mode, photometric mode, and kinetics mode. In addition, UVProbe has data saving and user control features which help organize and compare results easily) [14].

2.7.2 Tests and data Collection

We recorded the absorbance values obtained from both instruments for all prepared concentrations of KMnO₄ and I₂ following these methods:

1. The prototype:

Measurement Procedure Using the Prototype

The measurements using the developed spectrophotometer prototype were obtained following the steps below:

Step 1: System setup

We connected the prototype to the Thermion spectrometer software, which we used to control the system and record the measurements.

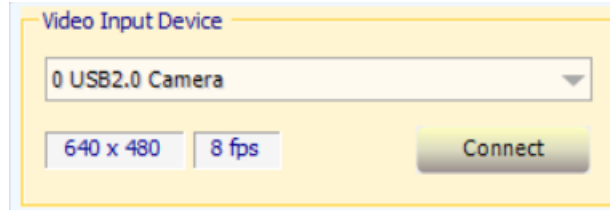


Figure 42: : System setup

Step2: Camera adjustment

We adjusted the input parameters, to ensure a stable and clear signal.

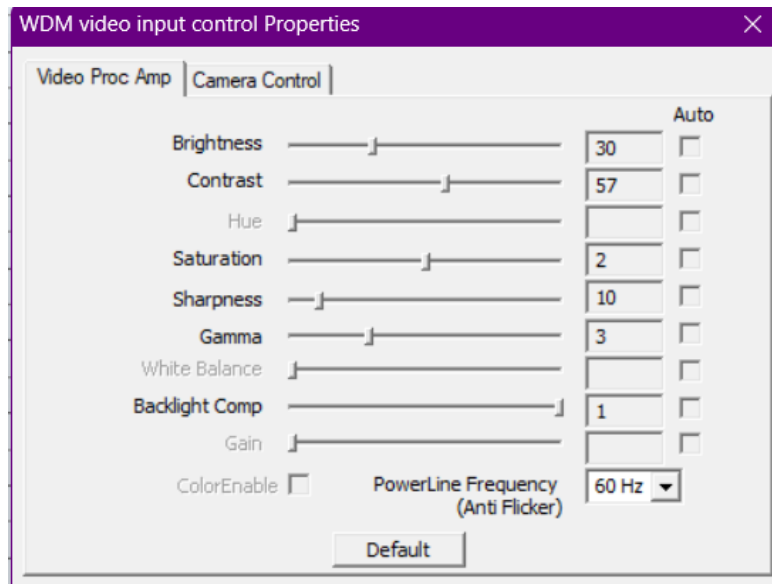


Figure 43 :Camera settings adjustment

Step 3: Reference measurement

We placed a cuvette filled with solvent in the holder, and we recorded the reference spectrum.

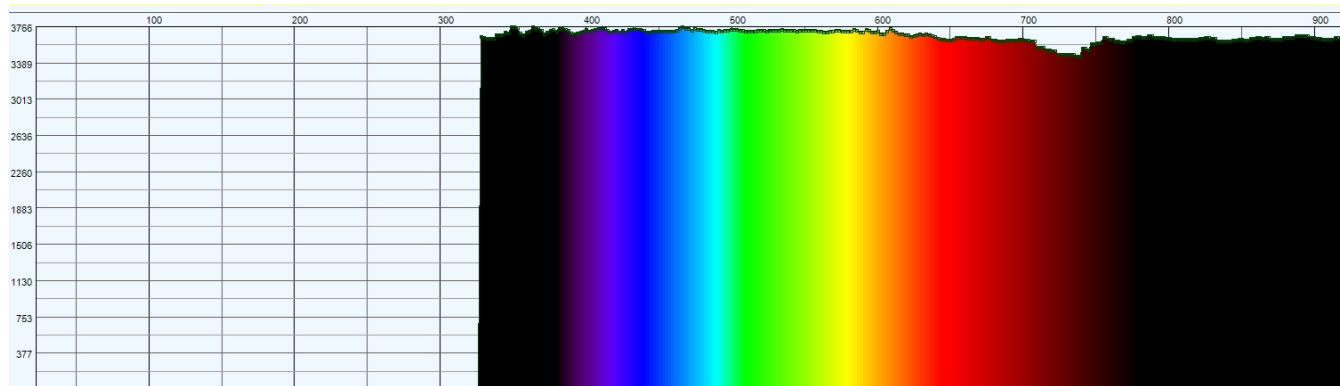


Figure 44: Reference spectrum for KMnO_4

Step 4: Sample measurement

We removed the reference cuvette and placed the cuvette containing the prepared solution in the holder,
then recorded the absorbance spectrum of the sample under the same conditions and obtained the following results:

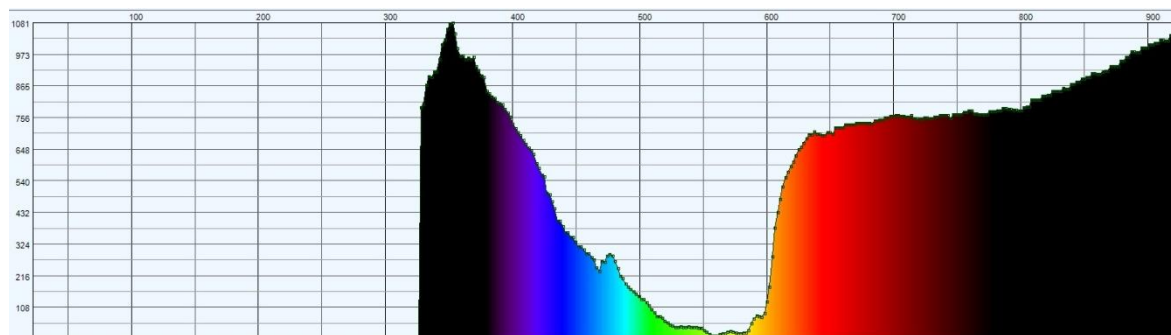


Figure 45 : the 0.001mol/L spectrum for KMnO_4

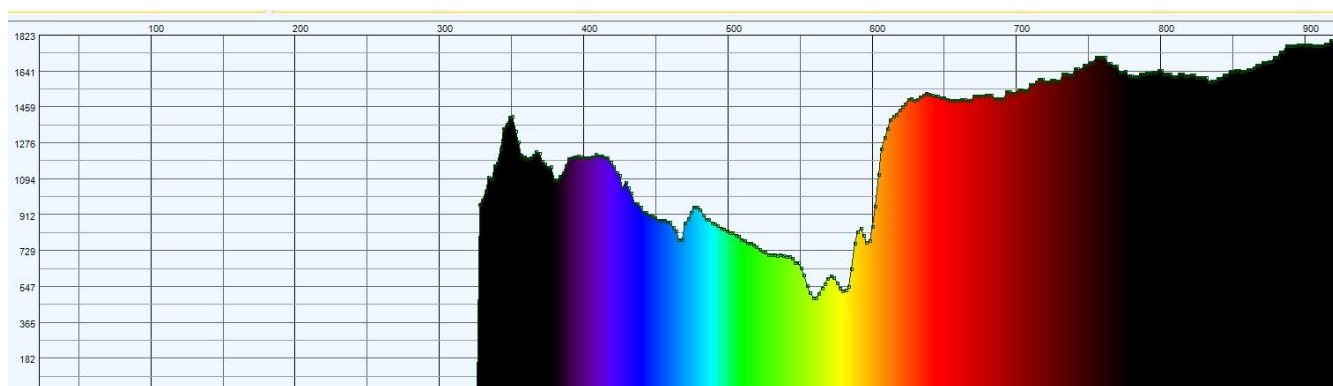


Figure 46 : the 0.0005mol/L spectrum for KMnO_4

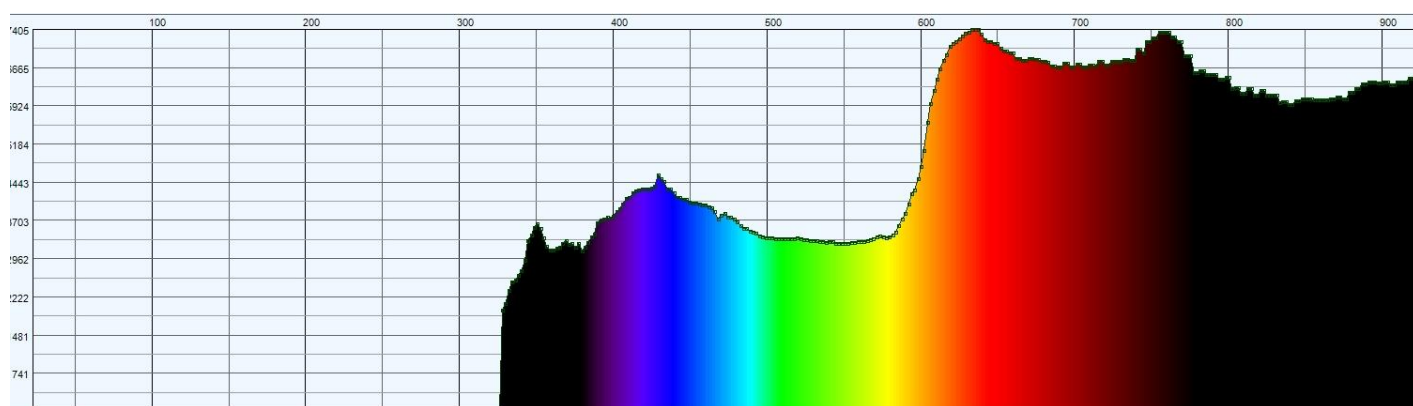


Figure 47 : the 0.0001mol/L spectrum for KMnO_4

The Thermion software records the data as intensity in function of wavelength then saves it as output files. then we exported these files to Excel for absorbance calculations.

Since the absorbance equation is written in comparison of both intensities, the reference and the measurement, we had to manually do these calculations following the beer-lambert's law, following this equation:

$$A = \text{Log} \left(\frac{I_0}{I} \right)$$

2. Measurement Procedure Using the Shimadzu Spectrophotometer

The measurements using the Shimadzu UV-3600 spectrophotometer following the steps below:

Step 1: Instrument and software startup

Turn on the Shimadzu instrument and start the UVProbe software.

Step 2: Selection of measurement mode

Select the absorbance mode in the software for all measurements.

Step 3: Baseline correction

Place the reference cuvette in the instrument and record the baseline to correct the system.



Figure 48: Sample and reference compartment

Step 4: Sample measurement

Place the different concentration samples one by one in the instrument and press start to begin scanning. The instrument recorded the absorbance spectra automatically.

Step 5: Data export

The UVProbe software export the data automatically in encrypted files. We re-exported the data into .txt format to allow further analysis.

The sample holder:

For both measurements we used standard spectrophotometer cuvettes with a fixed path length of 1 cm to hold the solutions during measurements.

The cuvettes were carefully filled and handled to avoid bubbles, fingerprints, or contamination, as these could affect the absorbance results. Each cuvette was placed in the same orientation inside the instrument to ensure consistent and reliable measurements.



Figure 49: Sample holder (cuvette).

2.7.3 Data Processing

For data processing, we used OriginPro software to visualize and analyze the data obtained from the experiments.

First, we imported the data and organized it in the software:



The screenshot shows a window titled 'absKM_F0001 *' with a data table. The table has columns: Long Name, A(X), B(Y), C(Y), and D(Y). The data is organized as follows:

	A(X)	B(Y)	C(Y)	D(Y)
Long Name	Wavelength	Abs.		
Units				
Comments		0.001	0.0005	0.0001
F(x)=				
1	200	0.719	-0.073	0.276
2	201	0.765	-0.199	0.296
3	202	0.767	-0.169	0.305
4	203	0.736	-0.158	0.317
5	204	0.778	-0.046	0.321
6	205	0.763	1.115	0.293
7	206	0.702	6	0.275
8	207	0.684	0.632	0.254
9	208	0.718	0.521	0.233
10	209	0.752	0.484	0.222

Figure 50: data table in origins pro

Then used the plot function to obtain the curves:

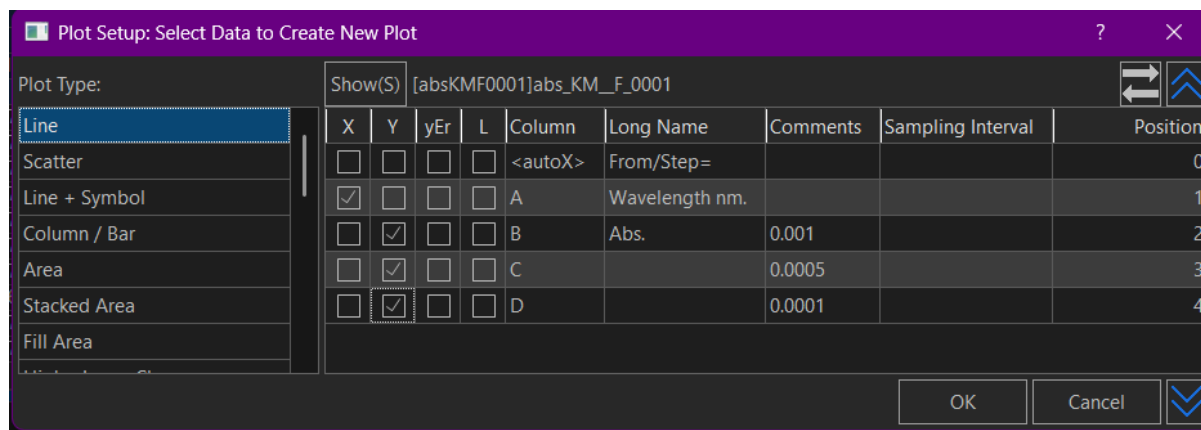


Figure 51: plot tool in origins pro

2.7.4 Results and discussion

After completing the tests, we exported the recorded data and plotted them to obtain the curves, which facilitated the visualization and analysis of the absorbance of the samples. As presented in Figures 52 and 53 for I_2 solutions:

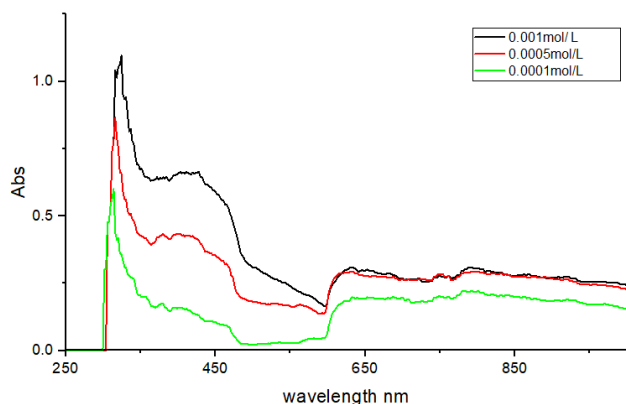


Figure 53: prototype results I_2

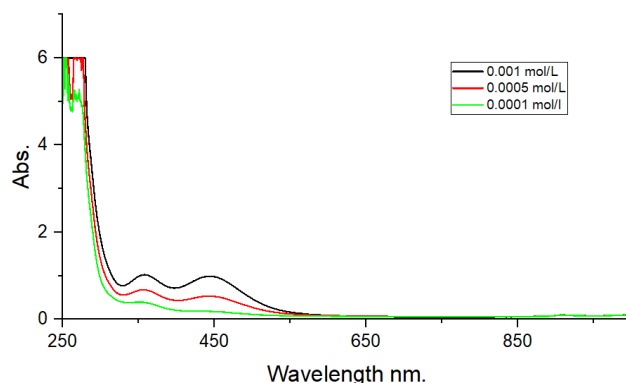


Figure 52: Shimadzu 3600 results for I_2

For Iodine (I_2) dissolved in Ethanol.

For Shimadzu 3600 analysis:

The absorption spectra of iodine (I_2) in ethanol show absorbance mainly between 250 nm and 600 nm. In the UV region below 300 nm, the absorbance is very high for all concentrations, and at the highest concentration the signal reaches saturation, so the peaks look flat.

In the visible region between 400 nm and 500 nm, we observe a broad absorption band that can appear as two close peaks at 357 nm and 443 nm, and the general shape stays as it is for all samples.

We also clearly see the effect of concentration on absorbance. When the concentration decreases from 0.001 mol/L to 0.0001 mol/L, the absorbance also decreases, while the shape of the spectrum does not change.

For the prototype:

The absorption spectra recorded by the prototype show signals mainly between 250 nm and 600 nm. In the UV region below 300 nm, the absorbance is very high for all concentrations.

In the visible region between 350 nm and 500 nm, we see a broad absorption band. It can appear as two close peaks at 320 nm and 424 nm, but the general shape is the same for all samples.

For KMnO_4 absorbance results are shown in Figures 54 and 55:

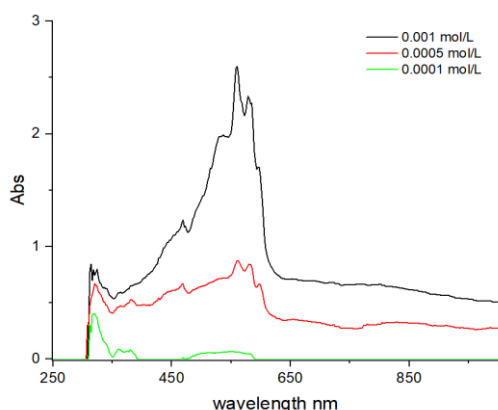


Figure 54: prototype results of KMnO_4 ,

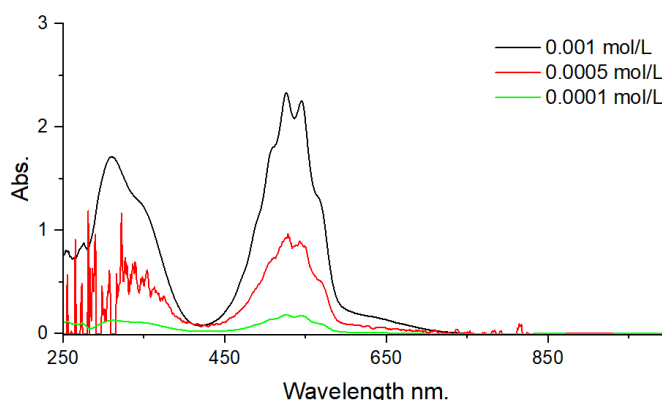


Figure 54 : uv3600 results of KMnO_4

For the Shimadzu 3600:

The absorption spectra of potassium permanganate (KMnO_4), show clear peaks in both the UV and visible regions at 527nm and 545nm.

In the visible range between 450 nm and 600 nm, a strong absorption band is observed, especially for higher concentrations. The highest absorbance is around 2.3 for the 0.001 mol/L solution at 527nm, and it decreases as the concentration becomes lower. A smaller peak is also seen in the UV region for the concentration 0.0005mol/L at 370nm.

for the prototype:

The absorption spectra of (KMnO_4) recorded by the prototype, show an absorption band in the visible region between 450 nm and 600 nm. This band is the main feature of the spectrum, and its intensity decreases as the concentration of the solution decreases.

In the UV region below 400 nm, we observe one absorption peak at 318 nm.

These measurements show some fluctuations in the UV region called noise. This noise can be caused by many reasons such as interference or light misalignment in the optical system. It can also come from impurities in the sample, calibration issues, or changes in environmental conditions ...

Comparison to the literature spectra:

To evaluate our results, we compare them with the available literature data to check their accuracy and reliability.

For the (KMnO₄) :

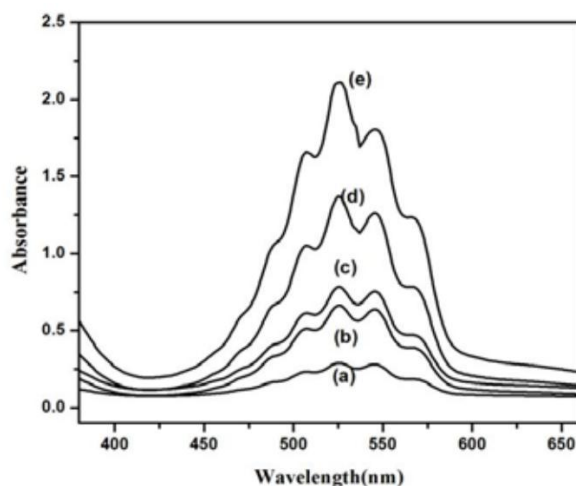


Figure 55 : Absorbance spectrum of KMnO₄ [31]

The standard absorption spectrum of potassium permanganate (KMnO₄) in the visible region (450–600 nm) shows a characteristic shape with three peaks at approximately 507 nm, 525 nm, and 545 nm, with the maximum absorbance (λ_{max}) occurring at 525 nm.

The results obtained with **the Shimadzu** instrument are in good agreement with this reference. The spectra reproduce the same multi-peaked structure in the 450–600 nm range, with a maximum absorbance observed at 527 nm for the highest concentration and the second peak at 545nm

The prototype captures the main optical behavior of the solution and is able to show the multi-peaked structure of the permanganate ion. In the visible range between 450 nm and 600 nm, the main absorption band shows several small peaks and splits, especially at higher concentrations. The highest peak is observed at around 560 nm. These peaks are slightly shifted compared to the literature, but the general pattern is still observed.

There are several factors contributing to the shift of our results such as the manual calibration of the wavelength scale, the lower resolution of the diffraction grating, slight misalignment of optical components...

For the I₂

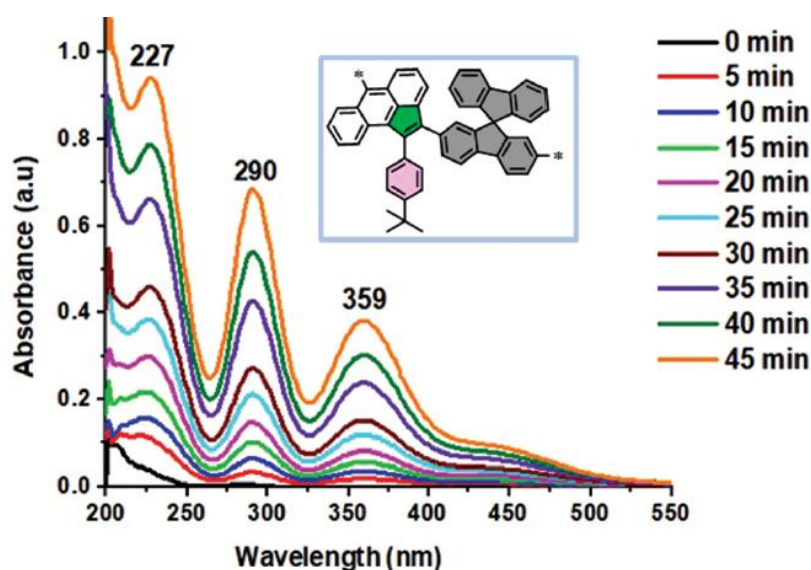


Figure 56 : Absorbance spectrum of I₂ [32]

According to the literature, dissolved iodine in ethanol shows absorption features in the near-UV and visible regions, with main peaks at 290 nm and 359 nm (and an additional peak at 227 nm). These spectral bands are commonly used to confirm the presence of iodine and to study its behavior in solution.

The Shimadzu spectrophotometer shows two main peaks at 360 nm and 443 nm. These results show a slight shift towards higher wavelengths compared to the literature, but the overall spectral features are still clearly defined.

The prototype

The recorded signal presents three peaks at approximately 324 nm, 560 nm, and 580 nm.

Compared to the literature, a larger shift towards higher wavelengths is observed.

The peaks are less defined, which is expected due to the lower resolution and limitations of the optical setup. But the prototype still shows the same general behavior, with the intensity decreasing as the concentration decreases,

2.7.5 Calculation and Validation of the Molar Extinction Coefficient:

To verify the performance of both the Shimadzu spectrophotometer and the developed prototype, we determined the molar extinction coefficient (ϵ) of the tested compounds and compared the obtained values with literature data.

Selection of the analysis wavelength and its absorbance:

We chose the wavelength that offered the clearest and most reliable measurements for the different concentrations.

	prototype		Shimadzu 3600	
	Absorbance		Absorbance	
	KMnO ₄ $\lambda=560\text{nm}$	I ₂ $\lambda=400\text{nm}$	KMnO ₄ $\lambda=525\text{nm}$	I ₂ $\lambda=443\text{nm}$
c=0.001mol/L	2.59	0.66	2.34	1
c=0.0005mol/L	0.86	0.43	0.92	0.533
c=0.0001mol/L	0.06	0.15	0.17	0.19

1. The calibration curves

- We plotted absorbance A in a function of concentration C then applied a linear fit to obtain the calibration equation and the requested result.

calibration curves of the prototype:

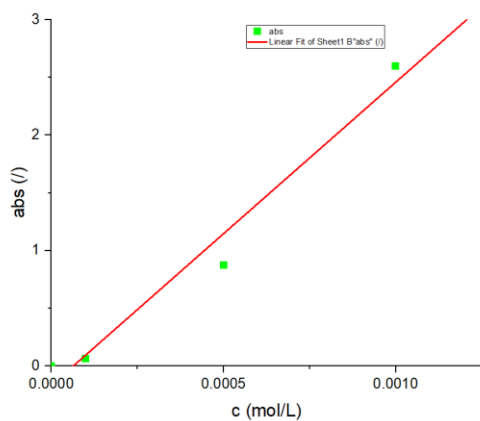


Figure 58 : prototype calibration curve of: KMnO_4

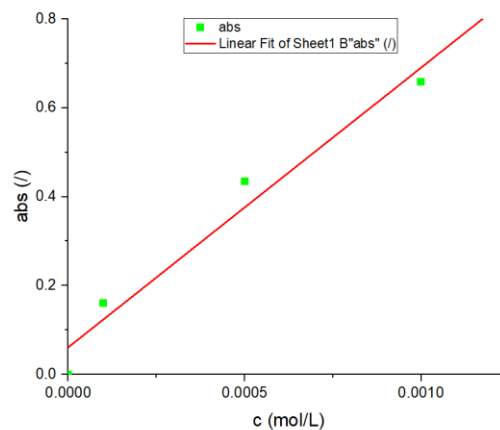


Figure 57 : prototype calibration curve of I_2

calibration curves of the Shimadzu 3600

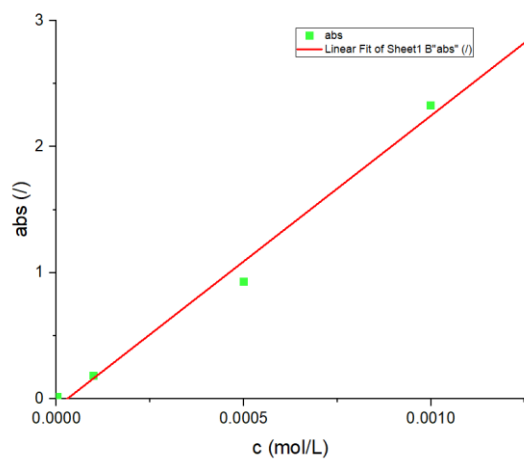


Figure 59 : calibration curves of the uv 3600 of KMnO_4

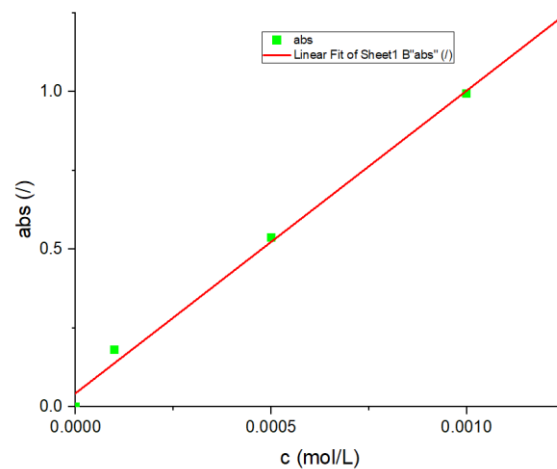


Figure 59: calibration curve of uv 3600 I_2

2. Determination of the molar extinction coefficient

$$A = \varepsilon c d$$

where:

- (A) = absorbance,
- (ε) = molar extinction coefficient ($\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$),
- (l) = optical path length (1 cm),
- (C) = concentration ($\text{mol} \cdot \text{L}^{-1}$).

Since the cuvette path length is 1 cm, the slope of the calibration curve corresponds directly to the molar extinction coefficient, as represented:

$$A = \varepsilon c$$

When plotting in origins software the equation became

$$A = mc + b$$

m = slope

b = intercept = 0

Which means

$$m = \varepsilon l$$

since

$$l = 1 \quad m = \varepsilon$$

The results obtained from the software are:

	Prototype		Simadzu 3600	
	I2	KMnO ₄	I2	KMnO ₄
ε ($\text{L} \cdot \text{mol}^{-1} \cdot \text{cm}^{-1}$)	629.35	2625	960	2312

Comparison with literature.

	Prototype		Simadzu 3600		literature	
	I ₂	KMnO ₄	I ₂	KMnO ₄	I ₂	KMnO ₄
ϵ (L·mol ⁻¹ ·cm ⁻¹)	629.35	2625	960	2312	746	2323
deviation	116.65	-302	-214	11		

In literature the extinction coefficient of each compound is:

I₂: 746 (L·mol⁻¹·cm⁻¹) [15]

KMnO₄ : 2323 (L·mol⁻¹·cm⁻¹) [16]

To better evaluate the agreement between the experimental results and the literature data, the deviations can be expressed as percentages. This allows a clearer and straight comparison of the accuracy of each instrument.

For iodine (I₂):

Prototype:

$$\text{Deviation} = \frac{|629.35 - 746|}{746} \times 100 = 15\%$$

Shimadzu

$$\text{Deviation} = \frac{|960 - 746|}{746} \times 100 = 28\%$$

For (KMnO₄) :

Prototype

$$\text{Deviation} = \frac{|2625 - 2323|}{2323} \times 100 = 13\%$$

Shimadzu

$$\text{Deviation} = \frac{|2312 - 2323|}{2323} \times 100 = 0.4\%$$

- **I₂ (Prototype): 15%**
- **I₂ (Shimadzu): 28%**
- **KMnO₄ (Prototype): 13%**
- **KMnO₄ (Shimadzu): 0.4%**

Discussion :

The percentage deviations show that the Shimadzu spectrophotometer provides the closest results to the literature, especially for KMnO₄, where the deviation is 0.4%.

The prototype shows larger deviations, around 13–15%, which can be explained by limitations in wavelength calibration and optical resolution.

Even with these differences, we can see that the obtained results remain reasonably close to the literature and confirm the ability of the prototype to reproduce the expected spectroscopic behavior.

Conclusion:

This study presented the development and evaluation of a low-cost UV-Vis spectrophotometer prototype, compared with a Shimadzu UV-3600 instrument and literature data. The results showed that the prototype is capable of detecting the main absorption features of the tested compounds and reproducing the expected spectroscopic trends.

When compared to literature values, both the prototype and the Shimadzu showed the same overall behavior, but with deviations in the absolute values. The Shimadzu results were generally closer to the reference data, while the prototype showed larger differences due to limitations in its design.

Despite these limitations, the prototype remains effective in capturing the key spectral characteristics and concentration-dependent behavior of the solutions. Overall, the system demonstrates good performance for a simplified setup and provides a cost-effective tool for educational and basic laboratory applications.

Perspectives:

- Improve the accuracy of the measurements by optimizing the optical and electronic components of the prototype.
- Reduce the noise present in the acquired signals to obtain more reliable and stable results.
- Integrate smoothing and filtering algorithm to enhance signal quality and facilitate the detection of absorption peaks.
- Extend the wavelength range of the spectrophotometer to allow the analysis of a wider variety of samples by integrating new radiation sources for each region of the spectrum.
- Improve the calibration procedure to increase the accuracy and repeatability of measurements.

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