

# UNIT 11

## UV-VISIBLE ABSORPTION SPECTROSCOPY

### Structure

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11.1 Introduction	Hypsochromic Shift
Expected learning Outcomes	Bathochromic Shift
11.2 Electromagnetic Radiations	Hyperchromic Shift
11.3 Electronic Transitions, $\lambda_{\max}$ & $\epsilon_{\max}$	Hypochromic Shift
11.4 Some important Terms Used in Electronic Spectroscopy	11.5 Summary
Chromophore	11.6 Terminal Questions
Auxochrome	11.7 Answers

### 11.1 INTRODUCTION

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In the previous block of this course you have learnt about the aromatic, heteroaromatic and active methylene compounds. This block is on “Applications of Spectroscopy to Simple Organic Molecules”.

In this unit, we will start our discussions on electromagnetic radiations. Then you will learn about ultraviolet-visible spectroscopy which is a consequence of interaction of electromagnetic radiation in the ultraviolet – visible range with the molecules constituting matter. The different types of transitions possible in the electronic spectra of polyatomic molecules,  $\lambda_{\max}$  and  $\epsilon_{\max}$  will then be discussed. The terms chromophore, auxochrome, hypsochromic and bathochromic effects, hyperchromic and hypochromic effects will be defined. In the next unit, you will actually learn how to identify small organic compounds, specially those with conjugated dienes, with the help of UV-visible spectroscopy.

### Expected Learning Outcomes

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After studying this unit and having performed the experiments, you should be able to:

- ❖ explain electromagnetic radiations and its wave nature;
- ❖ explain using diagrams the  $n - \pi^*$ ,  $n - \sigma^*$ ,  $\sigma - \sigma^*$ , and  $\pi - \pi^*$  transitions;
- ❖ define chromophore, auxochrome, hypsochromic and bathochromic effects, hyperchromic and hypochromic effects in electronic spectra;

## 11.2 ELECTROMAGNETIC RADIATIONS

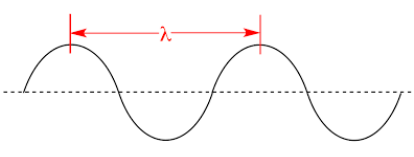
Now, the question is 'why certain substances are coloured? For example, the green colour of vegetation is due to a compound chlorophyll. Transition metal complexes, such as  $[\text{Ti}(\text{H}_2\text{O})_6]^{3+}$ , are coloured. The answer to the origin of colours in substances and information about excited states of molecules can be obtained from a study of their electronic spectra which occur in the visible (400-800 nm) and ultraviolet (200-400 nm) regions of electromagnetic spectrum. The spectra are obtained from spectroscopy which is a powerful tool to investigate the interaction of electromagnetic field with matter. It is utilized by researchers to conduct analysis in research laboratories and industries.

Spectroscopy is the study of interaction of electromagnetic radiation with matter.

An electromagnetic radiation may be defined as the radiant energy which is transmitted through space at enormous velocities. It does not require any medium for transmission. In previous courses you must have studied the characteristic of light, a form of electromagnetic radiation, and we will provide only a summary here. Gamma rays, X-rays, ultraviolet light, visible light, infrared radiation, microwaves and radio waves are all examples of **electromagnetic radiation** and part of the electromagnetic spectrum. Because electromagnetic radiation behaves as a wave travelling at the speed of light, it is described in terms of its wavelength and frequency.

**Wavelength** is the linear distance between any two consecutive equivalent points on the wave (for examples, crest to crest or trough to trough). Wavelength is given the symbol  $\lambda$  (Greek letter *lambda*) and is usually expressed in the SI base unit of meters. Other derived units commonly used to express wavelength are given in Table 11.1.

**Table 11.1: Common units used to express wavelength ( $\lambda$ )**

Unit	Relation of meter	
millimeter (mm)	1 mm = $10^{-3}$ m	
Micrometer ( $\mu\text{m}$ )	1 $\mu\text{m}$ = $10^{-6}$ m	
nanometer (nm)	1 nm = $10^{-9}$ m	

The **frequency** of a wave is the number of full cycles of the wave that pass a given point in a second. Frequency is given the symbol  $\nu$  (Greek letter *nu*) and is reported in second inverse or per second,  $\text{s}^{-1}$  (also called **Hertz, Hz**). Wavelength and frequency are inversely proportional, and we can calculate one from the other using the relationship:

$$c = \nu\lambda$$

where  $\nu$  is frequency in  $\text{s}^{-1}$ ,  $c$  is the velocity of light ( $3.0 \times 10^8 \text{ m s}^{-1}$ ), and  $\lambda$  is the wavelength in meters.

The wave number is the number of waves per centimeter per unit distance. It is expressed in units of  $\bar{\nu}$  and is equal to the reciprocal of the wavelength expressed in meters (m). Thus the unit of wave number will be meter inverse ( $\text{m}^{-1}$ ). If the wavelength is expressed as cm then wave number will be  $\text{cm}^{-1}$ .

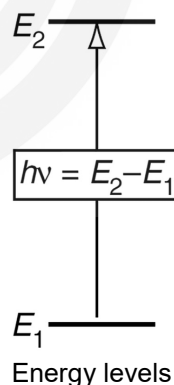
$$\bar{\nu} = \frac{1}{\lambda}$$

An alternative way to describe electromagnetic radiation is in terms of particles. We call these particles as photons. The energy of a photon and the frequency of radiation are related by the equation.

$$E = h\nu = h(c/\lambda)$$

Where  $E$  is the energy in kJ and  $h$  is Planck's constant,  $6.626 \times 10^{-34} \text{ J s}$ . This equation tells us that high-energy radiation corresponds to short wavelengths, and vice versa. Thus, ultraviolet light (higher energy) has a shorter wavelength (approximately  $10^{-7} \text{ m}$ ) than infrared radiation (lower energy), which has a wavelength of approximately  $10^{-5} \text{ m}$ .

An atom or molecule undergoes a transition from lower energy state  $E_1$  to a higher energy state  $E_2$  by irradiating it with electromagnetic radiation corresponding to the energy difference between states  $E_1$  and  $E_2$  as illustrated schematically in Fig.11.1. Absorption of photon results in electronic transition of a molecule, and electrons are promoted from ground state to higher electronic states.



**Fig. 11.1: Electromagnetic radiation.**

When the excited atom or molecule returns to the ground state, an equivalent amount of energy is emitted. When a compound is irradiated with electromagnetic radiation of various wavelengths, it absorbs energy of particular wavelengths, while the wavelengths not absorbed simply pass through or are reflected from the sample unchanged. The structure of electronic spectra, involves the change of at least three quantum numbers simultaneously, namely electronic, vibrational and rotational quantum numbers. Since it involves an electron excitation phenomenon, so, UV-visible Spectroscopy is also called as Electronic Spectroscopy.

$$1 \text{ cm} = 10^{-2} \text{ m}$$

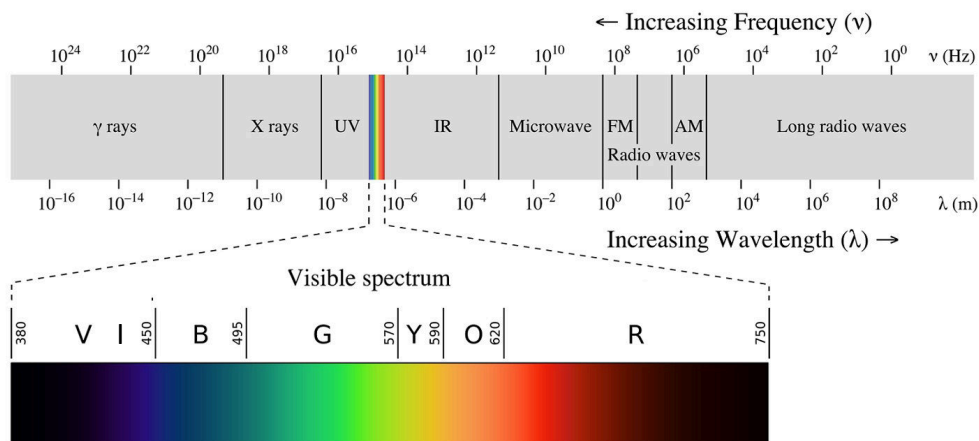
$$1 \mu\text{m} = 10^{-6} \text{ m}$$

$$1 \text{ nm} = 10^{-9} \text{ m}$$

$$1 \text{ \AA} = 10^{-10} \text{ m}$$

In UV-visible spectra, nanometer is usually used as unit for wavelength.

Fig. 11.2 summarises the wavelengths and frequencies of some regions of the electromagnetic spectrum.



**Fig. 11.2: The wavelengths and frequencies of some regions of the electromagnetic spectrum.**

If you look at the electromagnetic spectrum as in Fig. 11.2 you can see that the spectrum is divided into  $\gamma$ -rays, x-rays, ultraviolet, visible, infrared, microwave and radio waves, etc. as the wavelength increases.

We can sense only the visible region of the spectrum with the help of human eye. Before going to the next section, try to answer the following SAQ.

### SAQ 1

Which region of the electromagnetic region is the UV-visible region?

## 11.3 ELECTRONIC TRANSITIONS

In an atom, various electronic states can arise from a given electronic configuration due to coupling of orbital angular momentum and spin angular momentum of electron. The atomic spectra arise due to transition between different electronic energy states. Similarly for molecules various electronic states can arise from a given electronic configuration of a molecule. The electronic configuration of a molecule can be derived on the basis of molecular orbital theory which you studied in Unit 9 of the BCHCT131 course. Please recapitulate the Molecular Orbital Treatment of molecules in that unit, especially the LCAO method. You also have to remember that the rules of filling up electrons in molecular orbitals are same as the filling of atomic orbitals. That is

- the electrons occupy the available molecular orbitals one at a time, the lowest energy orbital being filled first (**aufbau principle**),
- each molecular orbital can accommodate a maximum of two electrons, provided their spins are opposite (**Pauli's exclusion principle**),

- iii) no pairing of electrons in orbitals of equal energy will take place unless there is at least one electron in each of them (**Hund's rule of maximum multiplicity**).

The electronic structure of the absorbing species, like, atoms, molecules, ions or complexes, which absorb in the UV-visible region of the spectrum, influence the absorption of radiation.

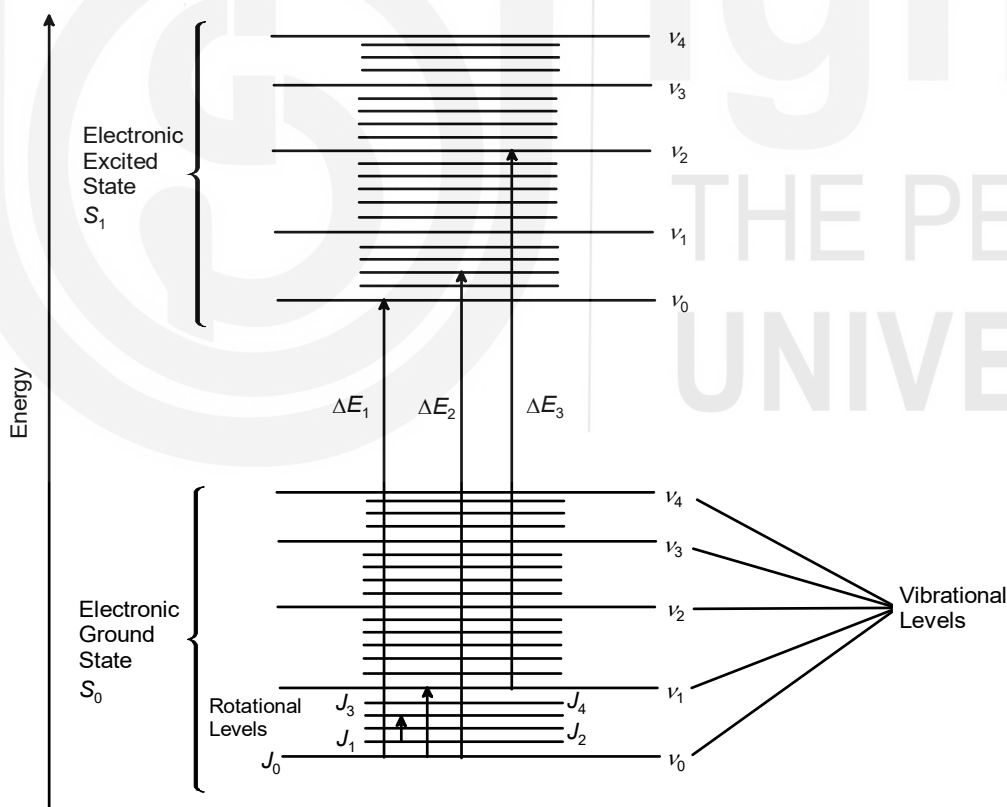
The structure of electronic spectra, involves the change of at least three quantum numbers simultaneously, namely electronic, vibrational and rotational quantum numbers. This follows the Born-Oppenheimer approximation that rotational ( $E_R$ ), vibrational ( $E_V$ ) and electronic ( $E_e$ ) energy levels are independent of one another. The total energy  $E$  is written as

$$E = E_e + E_v + E_R$$

and therefore the change in the total energy as a result of electronic transition, in a molecule is given by

$$\Delta E = \Delta E_e + \Delta E_v + \Delta E_R$$

Thus each electronic level comprises a number of vibrational levels and each vibrational level consists of several rotational levels, as is shown in Fig. 11.3.



**Fig. 11.3: Rotational, vibrational and electronic energy levels in a molecule.**

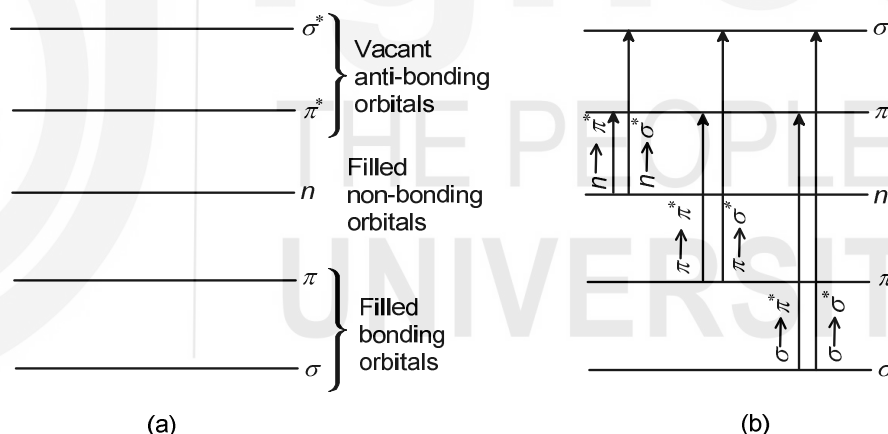
The UV-visible radiations cause transition of electrons from the low energy electronic states to the higher energy states. But IR (discussed in Units 13 and 14) radiations cause transitions among vibrational and rotational energy levels.

You see that every electronic level has several vibrational levels. Again, each vibrational level is associated with several rotational energy levels. So there

are several vibrational and rotational transitions associated in the transitions caused by UV-visible radiation. From these values of relative order of energies, as we see in Fig. 11.3, we find that the vibrational changes give a 'coarse structure' and the smaller rotational changes give a 'fine structure' to the electronic spectra. Since rotational energy changes are minimum, these energy changes are neglected and electronic band system is considered in terms of transitions between electronic levels each consisting of a series of vibronic levels of the same kind. So the molecular spectra becomes very complex due to such large number of transitions. This results in band spectrum due to electronic absorptions. The bands for molecules are found to be quite broad.

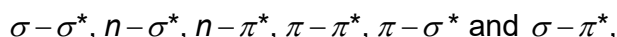
Absorption of ultraviolet and visible radiation in organic molecules is restricted to certain functional groups (chromophores) that contain valence electrons of low excitation energy.

Spectra of small organic molecules provides important information about electronic structure. We make use of molecular orbital theory to understand theoretical aspects of spectra of these. In electronic transitions of such molecules, we encounter three types of molecular orbitals:  $\sigma$  and  $\sigma^*$ ,  $\pi$  and  $\pi^*$ , and  $n$  (nonbonding) orbitals. Orbitals without \* are bonding orbitals and those with \* are antibonding orbitals. The energy levels of these molecular orbitals, in increasing order of energy, are shown in Fig.11.4.

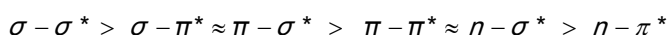


**Fig. 11.4: Schematic diagram of (a) order of molecular orbital energies (b) possible electronic transitions.**

When a molecule absorbs energy in the UV or visible region, an electron from a specific MO is excited to another MO of higher energy. The possible transitions of the electron between the MOs are:



The order of decreasing energy for these transitions is as follows:



Of all the possible transitions, the last three are responsible for absorptions in the region 200-800 nm, whereas others require much higher energy.

**$\sigma - \sigma^*$  transition:** Fig. 11.4 reveals that this transition requires very high energy. The absorption spectra obtained appears in the far ultraviolet region

(< 200 nm). Molecules which give this type of spectrum are saturated hydrocarbons and other compounds in which all valence electrons are involved in single bond formation. Since a spectrometer generally cannot measure below 185 nm, the region involving  $\sigma - \sigma^*$  transitions is relatively of little importance for chemical analysis.

**$n - \sigma^*$  transition:** The spectra corresponding to  $n - \sigma^*$  transition appears in the near UV or visible region. Compounds containing non-bonding or lone-pair electrons show this transition. For example, methyl alcohol vapour shows an absorption maximum at 183 nm. Methyl chloride and methyl amine show absorption maxima at 173 nm and 213 nm, respectively.

**$n - \pi^*$  transition:** This type of transition requires least energy and is exhibited by unsaturated molecules containing non bonding electrons. Certain organic groups like  $\text{>C-N-}$ ,  $-\text{N=O}$ ,  $\text{>C=O}$  show this type of transition and we observe absorption maxima for these systems at wavelengths greater than 280 nm. Groups such as  $\text{>C-N-}$ ,  $-\text{N=O}$ ,  $\text{>C=O}$ ,  $-\text{COOH}$  causing absorption at wavelengths greater than 175 nm are referred to as chromophores. More information on chromophores will be presented a little later.

**$\pi - \pi^*$  transition:** Bands due to  $\pi - \pi^*$  transition appear in the spectra of compounds containing  $\text{>C-C<}$ ,  $-\text{C}\equiv\text{C}-$ ,  $\text{>C=O}$ , and  $\text{>C-N-}$  functional groups. Ethylene and acetone exhibit  $\pi - \pi^*$  transition at 165 and 150 nm, respectively. The  $\pi - \pi^*$  transition is highly affected by conjugation.

You have already done experiment (Job's Method) based on UV-visible spectroscopy in the previous laboratory course BCHCL138. Recapitulating some concepts here is also very important. The absorption bands in ultraviolet and visible spectra are characterised by two main parameters which are

- i)  $\lambda_{\text{max}}$  Value: The value of the wavelength at which absorption maximum occurs is called the  $\lambda_{\text{max}}$  value. This corresponds to the wavelength of the radiation whose energy is equal to that required for an electronic transition. As different transitions require different energies, their  $\lambda_{\text{max}}$  values are different.
- ii)  $\epsilon_{\text{max}}$  Value :  $\epsilon$  value, which is known as molar absorptivity or molar extinction coefficient, is a measure of extent of absorption or intensity of absorption. The  $\epsilon$  value is characteristic of a particular compound at a given wavelength. Usually for the wavelength of maximum absorption ( $\lambda_{\text{max}}$ ), molar absorptivity is expressed as  $\epsilon_{\text{max}}$ .

The intensity of absorption can be expressed as transmittance ( $T$ ), which is defined as the ratio of the intensity of the radiation transmitted from the sample ( $I$ ) to that of the radiation incident on the sample ( $I_0$ ), i.e.,

$$T = I / I_0$$

Intensity of absorption is more conveniently expressed in terms of absorbance ( $A$ ), which is the logarithm of reciprocal of transmittance ( $T$ ), i.e.,

$$A = \log_{10} (1/T) = \log_{10} (I_0/I)$$

Absorbance of a band is related to the sample thickness and the concentration of the absorbing species. The relationship is expressed in the form of Beer-Lambert law as shown below:

$$A = \varepsilon cl = \log_{10} (I_0/I)$$

or 
$$\varepsilon = \frac{A}{cl}$$

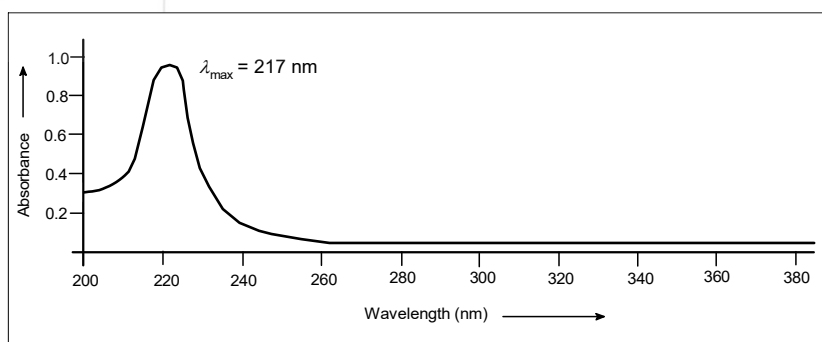
where  $\varepsilon$  = molar absorptivity or molar absorption constant;  $c$  = concentration of solute;  $l$  = sample thickness or path length through the sample

Absorbance is a dimensionless quantity. Concentration ( $c$ ) is usually expressed in  $\text{mol dm}^{-3}$  and path length ( $l$ ) in cm, hence  $\varepsilon$  has the units of  $\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$ . If we use SI units of  $\text{mol m}^{-3}$  for concentration and m for path length, the units of  $\varepsilon$  will be  $\text{m}^2 \text{mol}^{-1}$ . We can obtain the values of  $\varepsilon$  in  $\text{m}^2 \text{mol}^{-1}$  units from those in  $\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$  units in the following manner:  $\varepsilon = \text{dm}^3 \text{mol}^{-1} \text{cm}^{-1} = 10^{-3} \text{m}^3 \text{mol}^{-1} (10^{-2} \text{m})^{-1} = 10^{-1} \text{m}^2 \text{mol}^{-1}$ .

Values of  $\varepsilon$  in SI units can, therefore, easily be obtained from published values in  $\text{dm}^3 \text{mol}^{-1} \text{cm}^{-1}$  by dividing the numerical quantity in latter units by 10.

Absorption bands with  $\varepsilon_{\text{max}}$  value  $> 10^3 \text{m}^2 \text{mol}^{-1}$  are considered to be high intensity or strong bands, whereas those with  $\varepsilon_{\text{max}}$  values  $< 10^2 \text{m}^2 \text{mol}^{-1}$  are known as low intensity or weak bands.

The process of scanning is said to be done when a single wavelength (in the UV-visible region) is employed on the sample. This way the UV-visible spectrum is obtained over a range of wavelength. The amount of radiation absorbed at each wavelength is measured and plotted against the wavelength or frequency (in nm) in the x-axis and the intensity of absorption (absorbance) in the y axis. Such UV-visible spectroscopy is used extensively in teaching, research and analytical laboratories for the quantitative analysis of molecules that absorb ultraviolet and visible electromagnetic radiation and therefore it is important for you to know the basics of this powerful tool. The kind of data that we generally see in UV-visible spectroscopy is shown in the following Fig.11.5:



**Fig. 11.5: Sample graph obtained in UV-visible spectroscopy.**

Now try out the following SAQ before moving to the next section.

### SAQ 2

Which are the two main parameters on which the absorption bands in ultraviolet and visible spectra are measured?



## 11.4 SOME IMPORTANT TERMS USED IN ELECTRONIC SPECTROSCOPY

### Chromophore:

In a number of molecules, the absorption of a photon can be traced to the electrons of certain covalently bonded unsaturated groups. Such groups, for example, carbonyl in aldehydes or ketones which are responsible for electronic absorption, are referred to as chromophores. Other examples of chromophores are  $\text{>C=C<}$ ,  $-\text{C}\equiv\text{C}-$ ,  $-\text{COOH}$ ,  $-\text{N}=\text{O}$ ,  $-\text{NO}_2$ ,  $-\text{N}=\text{N}-$  etc. Chromophore in Greek means colour bringer and the presence of a chromophore often accounts for colours of substances. The following table lists the absorption maxima for some typical chromophores.

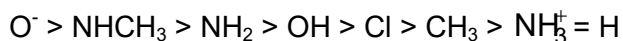
**Table 11.2: Absorption Data for Isolated Chromophores**

Chromophoric Group	System	Example	Transition	$\lambda_{\text{max}}$ nm	$\epsilon_{\text{max}}$ $\text{m}^2\text{mol}^{-1}$	Solvent
Ethylenic	$\text{RCH}=\text{CHR}$	Ethylene	$\pi \rightarrow \pi^*$	165	1500	Vapour
Acetylenic	$\text{R}-\text{C}\equiv\text{C}-\text{R}$	Acetylene	$\pi \rightarrow \pi^*$	173	6000	Vapour
Carbonyl	$\text{RR}'\text{C}=\text{O}$	Acetone	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	188 279	900 1.5	n-Hexane
Carbonyl	$\text{RHC}=\text{O}$	Acetaldehyde	$n \rightarrow \pi^*$	290	1.6	Heptane
Carbonyl	$\text{RCOOH}$	Acetic acid	$n \rightarrow \pi^*$	204	6.0	Water
Amido	$\text{RCONH}_2$	Acetamide	$n \rightarrow \pi^*$	< 208	–	–
Azomethine	$\text{>C}=\text{N}-$	Acetoxime	$\pi \rightarrow \pi^*$	190	5000	Water
Nitrile	$-\text{C}\equiv\text{N}$	Acetonitrile	$\pi \rightarrow \pi^*$	< 160	–	–
Azo	$-\text{N}=\text{N}-$	Azomethane	$n \rightarrow \pi^*$	347	0.45	Dioxane
Nitroso	$-\text{N}=\text{O}$	Nitrosobutane	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	300 665	10 2.0	Ether
Nitrate	$-\text{ONO}_2$	Ethyl nitrate	$n \rightarrow \pi^*$	270	1.2	Dioxane
Nitro	$-\text{NO}_2$	Nitromethane	$n \rightarrow \pi^*$	271	1.86	Alcohol
Nitrite	$-\text{ONO}$	Amyl nitrite	$\pi \rightarrow \pi^*$ $n \rightarrow \pi^*$	218.5 346.5	112	Petroleum ether

### Auxochrome

A saturated group with nonbonded electrons which, when attached to a chromophore, alters both the wavelength and the intensity of the absorption band is called an auxochrome, e.g., OH,  $\text{NH}_2$  and Cl. The auxochrome by itself is unable to impart colour to a compound. The auxochromic effect depends on the ability of the chemical group to donate electrons into conjugated system.

This has been most studied with aromatic systems and the band shifts of monosubstituted aromatic compounds have been correlated with electron donating power of auxochromes. The electron donating power of some common auxochromes decreases in the order



In this list, the effect of protonating the  $\text{NH}_2$  group should be noted. The proton binds to the nonbonding (lone pair) electrons on the nitrogen of the amino group and thus prevents them from interacting with the benzene  $\pi$ -electron system. Thus, the  $\lambda_{\text{max}}$  value for aniline ( $\text{C}_6\text{H}_5\text{NH}_2$ ) and the anilinium ion ( $\text{C}_6\text{H}_5\text{NH}_3^+$ ) is 230 nm and 203 nm, respectively as compared to 204 nm for benzene.

**Bathochromic or red shift:** A shift of the absorption maximum towards longer wavelength (lower frequency) produced by a change of medium or by the presence of an auxochrome, is called bathochromic shift or red shift. The  $\pi \rightarrow \pi^*$  transition in carbonyl compounds experiences bathochromic shift when polarity of the solvent is decreased.

**Hypsochromic shift or Blue shift:** A shift towards shorter wavelength (higher frequency) caused by a change of medium or by removal of conjugation is referred to as hypsochromic shift or blue shift. For example, the conjugation of the lone pair of electrons on the nitrogen atom of aniline with the  $\pi$ -bond system of the benzene ring is removed by protonation. Aniline absorbs at 230 nm, but in acid solution the main peak is shifted to 203 nm due to the presence of anilinium ions.

**Hypochromic effect:** An effect leading to decreased absorption intensity is called hypochromic effect.

**Hyperchromic effect:** An effect leading to increased absorption intensity is called hyperchromic effect.

You may now try to answer the following SAQ.

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### SAQ 3

Why the  $\lambda_{\text{max}}$  value for aniline ( $\text{C}_6\text{H}_5\text{NH}_2$ ) is 230 nm whereas that of benzene is 204 nm?

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## 11.5 SUMMARY

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In this unit, you have first learnt about electromagnetic radiations. Then different types of transitions possible in the electronic spectra of polyatomic molecules,  $\lambda_{\text{max}}$  and  $\epsilon_{\text{max}}$  were discussed. Lastly, you have learnt some important definitions of terms used in electronic spectroscopy. In the next unit, you will be actually studying how to apply the UV-visible spectroscopy as a tool to identify small organic compounds, specially those with conjugated dienes.

## 11.6 TERMINAL QUESTIONS

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1. How can a molecule absorb and emit electromagnetic radiation? Explain with a suitable diagram.
2. Which electronic transitions in small organic molecules are responsible for absorptions in the region 200-800 nm?
3. What are the expected transitions for amido and azomethine chromophore?
4. What information does UV-visible spectroscopy give about small organic molecules?
5. What sort of shift may be observed in chloroethylene,  $\text{CH}_2=\text{CHCl}$ ?

## 11.7 ANSWERS

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### Self-Assessment Questions

1. The visible region of the electromagnetic spectrum is 400-800 nm and ultraviolet region is 200-400 nm.
2.  $\lambda_{\text{max}}$  and  $\epsilon_{\text{max}}$
3. The auxochromes, e.g., OH,  $\text{NH}_2$  and Cl can donate electrons into conjugated system. This has been most studied with aromatic systems and the spectral shifts of monosubstituted aromatic compounds have been correlated with electron donating power of auxochromes. The electron donating power of  $\text{NH}_2$  is much more than that of H. So the observation of greater value of  $\lambda_{\text{max}}$  in aniline.

### Terminal Questions

1. We can cause an atom or molecule to undergo a transition from lower energy state to a higher energy state by irradiating it with electromagnetic radiation corresponding to the energy difference between the states (give Fig. 11.2). When the atom or molecule in excited state returns to the ground state, an equivalent amount of energy is emitted. When a compound is irradiated with electromagnetic radiation of various wavelengths, it absorbs energy of particular wavelengths, while the wavelengths not absorbed simply pass through or are reflected from the sample unchanged.
2. Of the four possible transitions given below, the last three are responsible for absorptions in the region 200-800 nm, whereas the first one requires much higher energy:  
 $\pi - \sigma^*$  transition,  $n - \sigma^*$  transition,  $n - \pi^*$  transition and  $\pi - \pi^*$  transition.
3. Please see Table 11.3.
4. UV-Visible spectroscopy involves the valance electron transitions and thus gives information about  $\pi$ -bonds and conjugated systems of small organic

molecules.

5. In chloroethylene,  $\text{CH}_2=\text{CHCl}$ ,  $\text{C}=\text{C}$  is a chromophore. Cl is an auxochrome. Substitution of a hydrogen atom in ethylene by a halogen atom causes a bathochromic shift and a hyperchromic effect.

