# Organic Spectroscopic Analysis

# 有机 波谱分析

赠光盘
英文原音朗读

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唐川江 译

## Royal Society of Chemistry

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## 内 容 提 要

本书是配合高等院校化学及相关专业实施双语教学而引进的英文原版经典教材。涵盖了有机波谱分析的基本内容,包括有机波谱的一般原理、红外光谱、核磁共振波谱、质谱、光谱综合解析等。语言简洁、易懂,注重实际应用,并配有习题及答案。

本书可供高等院校化学专业、应用化学专业及相关专业师生使用,也可供化学领域技术人员阅读、参考。

# Preface /前言

The aim of this textbook is not to provide you, the reader, with large numbers of correlation tables for every form of spectroscopy used by organic chemists; nor is it designed to give you an in-depth understanding of the physical processes that occur when molecules interact with electromagnetic radiation. What we hope to do in this textbook is to provide you with a basic understanding of how each type of spectroscopy gives rise to spectra, and how these spectra can be used to determine the structure of organic molecules. This text is aimed at undergraduate students in Years 1 and 2, and is meant to provide an introduction to organic spectroscopic analysis, leading to an appreciation of the information available from each form of spectroscopy and an ability to use spectroscopic information in the identification of organic compounds.

波谱(光谱)学

电磁辐射

波谱(光谱)

有机波谱分析

We will concentrate upon the most commonly used techniques in organic structure determination: nuclear magnetic resonance (NMR), infrared (IR) and ultraviolet-visible (UV-Vis) spectroscopy, and mass spectrometry (MS). The amount of space devoted to each technique in this text is meant to be representative of their current usage for structure determination.

核磁共振/红外光谱
紫外—可见光谱/质谱学

Finally, we will try to bring all of these techniques together in an attempt to show you how to go about the structure determination of an unknown compound in a (reasonably) logical manner. Our aim has been to provide spectra to illustrate every point made, but do analyse fully each of the spectra in order to obtain the maximum information available. We hope you enjoy this text and find it useful in your studies.

R. J. Anderson, D. J. Bendell and P. W. Groundwater
*Sunderland*

# 出版前言

进入 21 世纪，各高校纷纷在提高素质教育、与国际接轨方面进行了积极的探索。其中"双语教学"成为推进素质教育的又一着陆点。教育部于 2001 年 8 月发布文件，针对本科教学工作提出 12 项措施，其中明确要求各高校在三年内开设 5% ~ 10% 的双语课题，并引进原版教材和提高师资水平。这使得高校双语教学势在必行。双语教学既是教育自身对课程设置的调整，也是社会发展对教育改革的必然要求。实施双语教学不仅可提高学生的英语水平，适应未来的发展，更重要的是提高我国综合国力和国际竞争力的需要。

通过问卷调查、座谈等方式，我们了解到双语教材的缺乏是高校实施双语教学的一大难点。作为科技出版社，我们拥有纺织、染化、服装、工美等专业人才，并和英、美等国家的专业出版社、院校、协会保持着良好的合作，因此，我们希望利用自身专业优势，与高校合作出版双语教材，使之成为高校实施双语教学的先行兵。

经过广泛的调研、深入的讨论，我们在双语教材的内容、出版形式等方面形成了较完整的思路。目前，我社出版的双语教材按专业分为五个系列：纺织工程专业双语教材、轻化工程专业双语教材、高分子材料专业双语教材、化学专业双语教材、服装专业双语教材。所选图书均为国外的经典教材，其内容与国内相关专业课程相近，便于学生在掌握专业基础知识的同时，开阔视野，提高语言应用能力。我们将力求出版形式多样化，既有原汁原味的影印本，又有中译本和中文注释版本。部分教材将配有多媒体光盘，更加丰富了教材的表现手段，有助于提高学生的英语听说能力。

可以说，这是我们在双语教材出版方面所进行的一次有意义的尝试，希望能对高校双语教学的开展起到抛砖引玉的作用，也希望读者对双语教材的出版提出建议、意见，以便我们在今后的工作中逐步改进、完善。

出版者

2007 年 4 月

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# 1
# General Principles / 一般原理

电磁辐射

波谱（光谱）
分子跃迁

频率 / 波长 / 波数

等效双键数（不饱和度）

## 1.1 The Interaction of Electromagnetic Radiation with Molecules

电磁辐射与分子间的相互作用

You will already know from your studies that the energy levels of atoms and molecules are **quantized**, *i.e.* there are discrete energy levels in atoms and molecules (Figure 1.1).
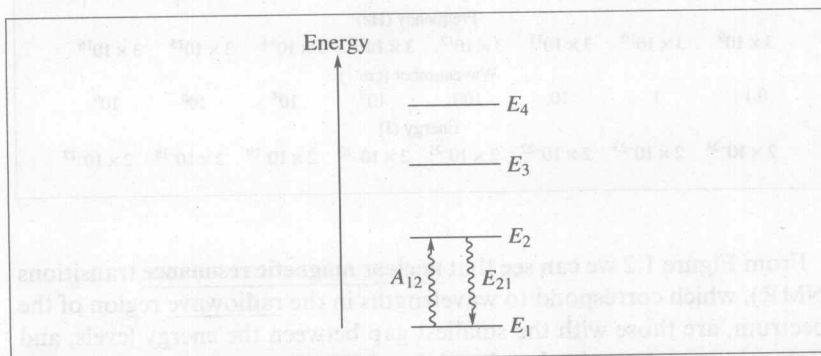
量子化的 / 不连续的能级



**Figure 1.1** Discrete energy levels of an atom or molecule

基态
激发态
差

量子

In Figure 1.1, $E_1$ corresponds to the ground state of the atom/molecule, and $E_2$, $E_3$ and $E_4$ represent excited states. Given that these are all discrete energy levels, the difference in energy between them (and so the energy required for a particular transition) will also be a discrete value, which is called a **quantum**. We can see from Figure 1.1 that in order to excite the atom/molecule from $E_1$ to $E_2$ it must absorb an amount of energy equivalent to $A_{12}$. Excited states are generally short lived and relax back to the ground state by emission of energy, in this case $E_{21}$.

频率 / 波长
电磁辐射
跃迁

The energy difference between the excited state ($E_2$) and the ground state ($E_1$) will correspond to a certain **frequency** ($v$) or **wavelength** ($\lambda$) of electromagnetic radiation, and this will depend upon the type of transition (and hence the separation between energy levels). The relationship between the energy of a transition and the frequency is given by equation (1.1):

$h$ is Planck's constant ($6.626 \times 10^{-34}$ J s).
普朗克常数

$c = \lambda v.$

$$\Delta E = hv \tag{1.1}$$

and so:

$$\Delta E = hc/\lambda \quad \text{or} \quad \Delta E = hc\bar{v} \tag{1.2}$$

The energy of a particular transition is, therefore, proportional to the frequency or wavenumber ($\bar{v} = 1/\lambda$) and inversely proportional to the wavelength (equation 1.2).

电磁波谱

The **electromagnetic spectrum** is divided into a number of regions. The names of these regions and the associated atomic/molecular transitions, together with the corresponding energies, frequencies, wavelengths and wavenumbers, are shown in Figure 1.2.

| Wavelength (m) | | | | | | | |
|---|---|---|---|---|---|---|---|
| 0.1 | $10^{-2}$ | $10^{-3}$ | $10^{-4}$ | $10^{-5}$ | $10^{-6}$ | $10^{-7}$ | $10^{-8}$ |
| Radiowave | Microwave | | Infrared | | | Visible–ultraviolet | X-ray |
| NMR | Rotational | | Vibrational | | | Electronic | |
| **Frequency (Hz)** | | | | | | | |
| $3 \times 10^9$ | $3 \times 10^{10}$ | $3 \times 10^{11}$ | $3 \times 10^{12}$ | $3 \times 10^{13}$ | $3 \times 10^{14}$ | $3 \times 10^{15}$ | $3 \times 10^{16}$ |
| **Wavenumber (cm$^{-1}$)** | | | | | | | |
| 0.1 | 1 | 10 | 100 | $10^3$ | $10^4$ | $10^5$ | $10^6$ |
| **Energy (J)** | | | | | | | |
| $2 \times 10^{-24}$ | $2 \times 10^{-23}$ | $2 \times 10^{-22}$ | $2 \times 10^{-21}$ | $2 \times 10^{-20}$ | $2 \times 10^{-19}$ | $2 \times 10^{-18}$ | $2 \times 10^{-17}$ |

**Figure 1.2** The electromagnetic spectrum

核磁共振
无线电波

紫外—可见光区

From Figure 1.2 we can see that **nuclear magnetic resonance** transitions (NMR), which correspond to wavelengths in the radiowave region of the spectrum, are those with the smallest gap between the energy levels, and electronic transitions in the **ultraviolet-visible (UV-Vis) region** have the

largest energy gap between transition levels. The UV-Vis region is important, since absorptions in this region give rise to the colour associated with molecules; Figure 1.3 shows this region in more detail.
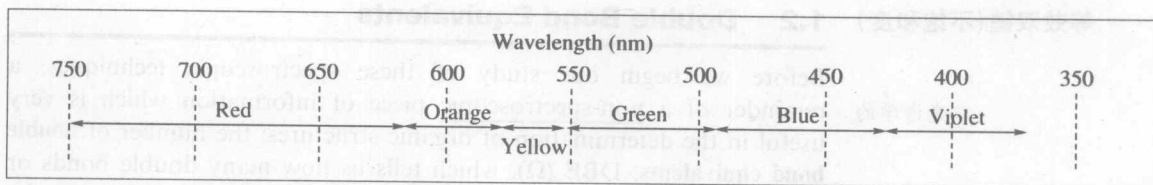
Wavelength (nm)

750   700   650   600   550   500   450   400   350

Red | Orange | Green | Blue | Viplet

Yellow

**Figure 1.3** The visible region of the electromagnetic spectrum

X-rays have such high energy that they are capable of <u>ionizing</u> atoms and molecules, but they are also important in terms of structure determination using <u>single-crystal X-ray diffraction</u>. This technique is the ultimate in structure determination since it provides a "map" of the molecule in the crystal, *e.g.* Figure 1.4, but it is highly specialized, is limited to crystals and is not routinely available for all organic chemists, so it will not be discussed any further here.

离子化的

单晶 X 射线衍射
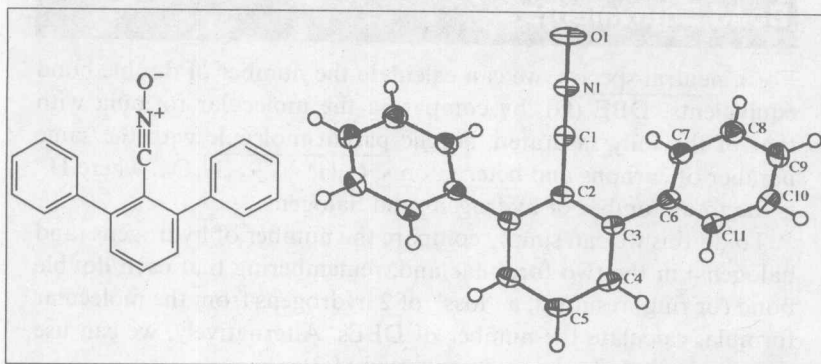
**Figure 1.4** Single-crystal X-ray structure of 2,6-diphenylbenzo-nitrile oxide

2,6 – 二苯基苯腈氧化物

Microwave (rotational) spectra are very complex, even for <u>diatomic molecules</u>, and give little useful information on organic molecules, which are relatively large. <u>Rotational transitions</u> are often responsible for the broadness of <u>infrared</u> (IR) bands, since each <u>vibrational transition</u> has a number of rotational transitions associated with it. The use of microwave spectroscopy is extremely rare in organic chemistry, and it too will be discussed no further here.

微波（转动）光谱／双原子分子

转动跃迁

红外谱带／振动跃迁

Transitions in all of the other forms of spectroscopy (UV-Vis, IR and NMR) are governed by <u>selection rules</u> that state which transitions are "**allowed**" and which are "**forbidden**" (although these latter transitions may still take place). We will mention the selection rules for each of these forms of spectroscopy when we study them in detail in Chapters 2–4.

选律,选择定则

质谱学

Finally, one technique that does not rely upon electromagnetic radiation, **mass spectrometry**, will be discussed in detail in Chapter 5.

等效双键(不饱和度)

## 1.2   Double Bond Equivalents

非光谱学的

卤素

饱和的 / 无环的

Before we begin our study of these spectroscopic techniques, a reminder of a non-spectroscopic piece of information which is very useful in the determination of organic structures: the number of **double bond equivalents**, DBE ($\Omega$), which tells us how many double bonds or rings are present in a molecule. Each double bond or ring reduces the number of hydrogens (or halogens) in a molecule by 2, so when we calculate the number of DBEs we simply compare the number of hydrogens which would be present in the fully saturated, acyclic compound with the number actually present, and divide by 2 to give the number of DBEs.

中性分子

母体分子
杂原子

OH

NH2

**1.1**

4 - 氨基苯酚

### Box 1.1   Calculation of the Number of Double Bond Equivalents

For a neutral species, we can calculate the number of double bond equivalents, DBE ($\Omega$), by comparing the molecular formula with that of the fully saturated, acyclic parent molecule with the same number of carbons and heteroatoms, $C_nH^*_{2n+2+y}N_yO_w$, where $H^*$ is the total number of hydrogens and halogens.

To do this we can simply compare the number of hydrogens (and halogens) in the two formulae and, remembering that each double bond (or ring) results in a "loss" of 2 hydrogens from the molecular formula, calculate the number of DBEs. Alternatively, we can use the simple formula shown in equation (1.3):

$$\Omega = (C + 1) - [(H^* - N)/2] \qquad (1.3)$$

where C = number of C atoms, $H^*$ = number of hydrogens or halogens, and N = number of nitrogens.

For example, the molecular formula for 4-aminophenol (**1.1**) is $C_6H_7NO$. The formula of the fully saturated, acyclic parent structure [$C_nH^*_{2n+2+y}N_yO_w$, where $H^*$ is the total number of hydrogens and halogens] would therefore be $C_6H_{(12+2+1)}NO$, *i.e.* $C_6H_{15}NO$. The difference in the number of hydrogens is therefore $15 - 7 = 8$, so there are 4 DBEs.
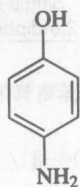
Alternatively, using equation (1.3):

$$\Omega = (6+1) - [(7-1)/2] = 7 - 3 = 4$$

Looking at the structure of 4-aminophenol, we can see that formally it has 3 double bonds, and the ring itself makes up the total of 4 DBEs.

## Worked Problem 1.1

**Q**  Calculate (a) the wavelength, (b) the frequency and (c) the energy associated with a transition with a wavenumber of 2260 cm$^{-1}$.

**A**  (a) $\bar{v} = 1/\lambda$, so $\lambda = 1/\bar{v} = 1/2600 = 4.42 \times 10^{-4}$ cm

$$= 4.42 \times 10^{-6} \text{ m } (4.42 \ \mu m).$$

(b) $c = v\lambda$, so $v = c/\lambda = 3 \times 10^8 \text{ ms}^{-1}/4.42 \times 10^{-6} \text{ m}$

$$= 6.79 \times 10^{13} \text{ s}^{-1} (6.79 \times 10^{13} \text{ Hz}).$$

(c) $E = hv = 6.626 \times 10^{-34} \text{ J s} \times 6.79 \times 10^{13} \text{ s}^{-1} = 4.5 \times 10^{-20} \text{ J}.$

## Worked Problem 1.2

**Q**  The local <u>anaesthetic</u> <u>benzocaine</u> has a molecular formula of $C_9H_{11}NO_2$. (a) Calculate the number of double bond equivalents; (b) identify all of the double bond equivalents in benzocaine (**1.2**).

麻醉剂 / 苯佐卡因

**A**  (a) The formula of the fully saturated, <u>acyclic parent structure</u>[①] $[C_nH^*_{2n+2+y}N_yO_w$, where H* is the total number of hydrogens and halogens] would therefore be $C_9H_{(18+2+1)}NO_2$, *i.e.* $C_9H_{21}NO_2$. The difference in the number of hydrogens is therefore $21-11 = 10$, corresponding to 5 DBEs. Alternatively, $\Omega = (9+1) - [(11-1)/2] = 10 - 5 = 5$.

(b) <u>Benzene ring</u> $\equiv$ 4 DBEs ($3 \times C=C$ and 1 ring) and $C=O$.

**1.2**
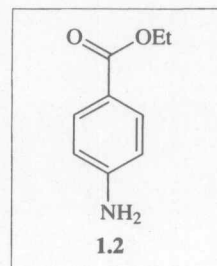
苯环

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① 无环母体结构

能级

无线电波

振动跃迁

无环母体化合物

## Summary of Key Points

1.  The energy levels of atoms and molecules (and so the difference in energy between these levels) have discrete values (quanta).

2.  The wavenumber of a transition is inversely proportional to the wavelength ($\bar{v} = 1/\lambda$).

3.  The relationship between the energy of a transition and the frequency is given by $\Delta E = hv$ or $\Delta E = hc/\lambda$ or $\Delta E = hc\bar{v}$, where $h$ is Planck's constant. The energy of a particular transition is, therefore, proportional to the frequency or wavenumber, and inversely proportional to the wavelength.

4.  NMR transitions correspond to wavelengths in the radiowave region of the spectrum, vibrational transitions correspond to wavelengths in the IR region, and electronic transitions to the UV-Vis region.

5.  The number of double bond equivalents corresponds to the difference between the molecular formula and that for the saturated acyclic parent compound. Each DBE (double bond or ring) results in the subtraction of 2 hydrogens or halogens from the molecular formula of this parent structure.

## Problems

**1.1.**  Calculate the energy associated with transitions with the following frequencies, wavelength or wavenumber. What type of molecular transition is associated with each transition? (a) $v = 3 \times 10^8$ Hz; (b) $\lambda = 254$ nm ($254 \times 10^{-9}$ m); (c) $\lambda = 1.0$ cm; (d) $\bar{v} = 2600$ cm$^{-1}$; (e) $v = 4.1 \times 10^{14}$ Hz; (f) $\bar{v} = 2 \times 10^8$ m$^{-1}$.

**1.2.**  How many double bond equivalents are there in each of the following molecules: (a) $C_6H_{12}O_6$; (b) $C_8H_9NO_2$; (c) $C_{27}H_{46}O$; (d) $C_5H_5N_5$; (e) $C_6H_3Cl_3O$; (f) $C_{46}H_{58}N_4O_9$.
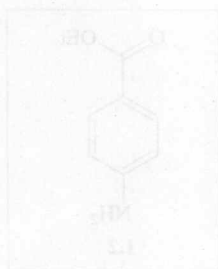
# 2

# Ultraviolet–Visible (UV-Vis) Spectroscopy / 紫外—可见光谱

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## Aims

This chapter describes how ultraviolet–visible (UV-Vis) spectroscopy is used in organic chemistry. After you have studied this chapter, you should be able to:

- Describe briefly how the absorbance of a UV-absorbing compound is obtained
- Explain the relationship between the absorption wavelength and the energy difference between the energy levels involved
- Calculate the molar absorptivity of a molecule from its absorbance, concentration and the path length
- Recognize chromophores, and explain how conjugation and aromatic substituents can contribute to chromophores
- Explain how pH can affect the chromophore and the UV-Vis spectra of acidic and basic compounds
- Predict the absorbance maximum for unsaturated compounds using the Woodward–Fieser rules
- Summarize some practical applications of UV-Vis spectroscopy

摩尔吸光系数
吸光度 / 浓度 / 光程长度
发色团（生色团）/ 共轭作用
芳香族取代基

酸性的 / 碱性的
不饱和化合物

## 2.1    Instrumentation

仪器设备

There are several different types of UV-Vis spectrometers, with the usual instrument met in an undergraduate laboratory being a double (or dual) beam spectrophotometer, which consists of a UV-visible light source, two cells through which the light passes, and a detector (usually a photomultiplier) to measure the amount of light passing through the cells. There are basic spectrometers that measure the absorbance at a specific wavelength, set by the user, and others that can scan the entire

分光光度计
双光束分光光度计
光源
吸收池 / 检测器
光电倍增器

特定波长

分光光度计

校正图

单光束紫外—可见分光光度计

二极管阵列检测器

基团

真空装置

双光束紫外—可见分光光度计

溶剂

乙醇

UV-Vis range. Newer spectrometers are usually computer controlled and allow the user greater flexibility, *e.g.* in overlaying spectra of a reaction mixture over time, or constructing a calibration graph to determine the concentration of an unknown.

Single-beam UV-Vis spectrometers work on the same general principles, but measure the absorption of the reference first, followed by the sample. They can scan across the entire UV-Vis range or can be used at a single wavelength. Detector technology has improved recently: the diode array detector enables simultaneous detection over the entire range to be achieved, allowing rapid quantification of absorbing species.

The term UV-Vis normally applies to radiation with a wavelength in the range 200–800 nm. There are many groups that absorb below 200 nm, but this part of the spectrum is difficult to examine (as oxygen absorbs UV radiation below 200 nm) unless the spectra are recorded in a vacuum (vacuum UV-Vis).

In the **double-beam UV-Vis spectrophotometer** the light is split into two parallel beams, each of which passes through a cell; one cell contains the sample dissolved in solvent and the other cell contains the solvent alone. The detector measures the intensity of the light transmitted through the solvent alone ($I_0$) and compares it to the intensity of light transmitted through the sample cell ($I$). The absorbance, $A$, is then calculated from the relationship shown in equation (2.1):

$$A = \log_{10} \frac{I_0}{I} \qquad (2.1)$$

Ethanol is transparent to UV above 200 nm and is, therefore, commonly used as the solvent in UV-Vis spectroscopy.

选律和比尔—朗伯(吸收)定律

We can calculate the energy of a particular wavelength using equation (2.2):

$$E(\text{kJ mol}^{-1}) = \frac{1.19 \times 10^5}{\lambda(\text{nm})} \qquad (2.2)$$

As the relationship between $E$ and $\lambda$ is a reciprocal one, we can see that short wavelength radiation corresponds to high energy and long wavelengths correspond to low energy.

振动 / 转动

红外线的 / 微波的

## 2.2   Selection Rules and the Beer–Lambert Law

As you will have seen in Chapter 1, light at the short wavelength end of the electromagnetic spectrum has enough energy to promote electronic transitions in organic molecules, such that absorption of UV light (200–400 nm; 595–299 kJ mol$^{-1}$) or Vis light (400–800 nm; 299–149 kJ mol$^{-1}$) can result in the promotion of outer electrons from one electronic energy level to a higher one.

The difference between electronic energy levels is greater than the difference between any other molecular energy levels, so these transitions require the higher energy of short wavelength radiation. Promotion between other energy levels, *i.e.* vibrational or rotational, requires only the lower energy infrared (vibrational) or microwave (rotational) radiation (Figure 2.1).

Vibrational energy levels　振动能级

$E_2$

Energy

Electronic transitions　电子跃迁

Rotational energy levels　转动能级

$E_1$
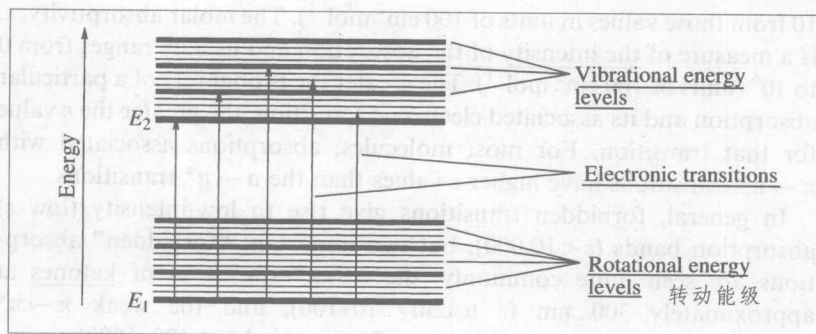
**Figure 2.1** Schematic diagram showing possible molecular electronic transitions, and vibrational and rotational energy levels

At room temperature, the majority of molecules are in the lowest vibrational state of the lowest electronic energy level, the "**ground state**" （振动态 / 基态） ($E_1$). Absorption of UV or visible light leads to promotion of an electron from $E_1$ to the excited electronic level, $E_2$. The electronic transition is accompanied by vibrational and rotational transitions, so that the promotion of an electron can occur from the ground state electronic energy level of $E_1$ to any of the vibrational or rotational energy levels of $E_2$. This explains why UV-Vis absorption bands are characteristically （吸收带） broad, although energy transitions between rotational and vibrational energy levels within one electronic energy level can show fine structure （精细结构） (*e.g.* see Chapter 3 for many examples of sharp absorbances). Fine structure in UV spectra can sometimes be achieved by using a solvent that has little interaction with the sample molecules.

The fact that there are many electronic transitions possible, however, does not mean that they can or will occur. There are complex selection rules based on the symmetry of the ground and excited states of the molecule under examination. Basically, electronic transitions are allowed if the orientation （取向） of the electron spin does not change during the transition and if the symmetry of the initial and final functions is different; these are called the **spin** and **symmetry selection rules**, respectively. （自旋 / 对称选律） However, the so-called "forbidden" transitions can still occur, but give （禁阻跃迁） rise to weak absorptions.

According to the **Beer–Lambert law**, the **absorbance**, $A$, of a solution is （吸光度） directly proportional to the **path length** ($l$, length of the cell containing the （光程长度） solution, in cm) and the **concentration** of the absorbing molecule ($c$, in （浓度） moles per litre), according to equation (2.3):

$$A = \varepsilon c l \qquad (2.3)$$

where $\varepsilon$ = the **molar absorptivity** of the absorbing molecule, some- times called the molar extinction coefficient, and is characteristic of （摩尔消光系数）

吸收强度

n denotes non-bonding and * an anti-bonding orbital (excited state).

丙酮

苯环

发色团(生色团)

非共轭的

饱和的

烷基 / 孤对(电子)

分子轨道图
乙烯

成键电子

10 from those values in units of $100 \, cm^2 \, mol^{-1}$). The molar absorptivity, $\varepsilon$, is a measure of the intensity of the absorption and usually ranges from 0 to $10^6$ (units of $100 \, cm^2 \, mol^{-1}$). The greater the probability of a particular absorption and its associated electronic transition, the greater the $\varepsilon$ value for that transition. For most molecules, absorptions associated with $\pi \rightarrow \pi^*$ transitions have higher $\varepsilon$ values than the $n \rightarrow \pi^*$ transitions.

In general, forbidden transitions give rise to low-intensity (low $\varepsilon$) absorption bands ($\varepsilon < 10,000$), but two important "forbidden" absorptions are seen quite commonly: the $n \rightarrow \pi^*$ transition of ketones at approximately 300 nm ($\varepsilon$ usually 10–100), and the weak $\pi \rightarrow \pi^*$ absorption of benzene rings at about 260 nm ($\varepsilon$ about 100–1000).

## 2.3   Chromophores

The part of the molecule containing the electrons involved in the electronic transition which gives rise to an absorption is called the **chromophore**. The wavelength of the maximum of the broad absorption is labelled $\lambda_{max}$. Most of the simple, non-conjugated chromophores give rise to high-energy (low-wavelength) absorptions with low-intensity ($\varepsilon$) values, but the majority of these absorptions are lost in atmospheric oxygen absorptions (Table 2.1).

**Table 2.1**  Absorption wavelengths of simple, non-conjugated chromophores

| $\lambda_{max}$/nm | Chromophore | Transition causing absorption |
|---|---|---|
| ~150 | C—C or C—H<br>$\sigma$-bonded electrons | $\sigma \rightarrow \sigma^*$ |
| ~185–195 | —X: (X = O, N, S)<br>Lone pair electrons | $n \rightarrow \sigma^*$ |
| ~300 | C=O:<br>Lone pair electrons | $n \rightarrow \pi^*$ |
| ~190 | Lone pair electrons | $n \rightarrow \sigma^*$ |
| ~190 | C=C (isolated)<br>$\pi$-bonded electrons | $\pi \rightarrow \pi^*$ |

With the exception of the high-energy $\sigma \rightarrow \sigma^*$ transition of saturated alkyl systems, these transitions require either a lone pair or a $\pi$-bond from which the electron can be promoted. In fact, UV spectra are generally only of interest if the system is unsaturated; chromophores with the greatest degree of unsaturation give rise to the most intense absorptions at longest wavelength.

We can show electronic transitions using molecular orbital diagrams, *e.g.* the $\pi \rightarrow \pi^*$ transition of ethene, $CH_2$=$CH_2$, is shown in Figure 2.2. Here we can see that UV radiation at 190 nm provides the required energy ($626.3 \, kJ \, mol^{-1}$) to promote a bonding electron from the $\pi$-bonding