



INTERNSHIP REPORT

PROXIMATE ANALYSIS OF COAL AND ANALYTICAL INSTRUMENTAL TECHNIQUES

FROM: SEPTEMBER 22, 2023 TO OCTOBER 04, 2023



Submitted By:

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M.Sc. CHEMISTRY (II YEAR)



SATHYABAMA

INSTITUTE OF SCIENCE AND TECHNOLOGY

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DEPARTMENT OF CHEMISTRY

This is to certify that this Internship Report is the bonafide work of **SALETH ROBIN. G, Reg.No. 42910017** who carried out the internship entitled **“PROXIMATE ANALYSIS OF COAL AND ANALYTICAL INSTRUMENTAL TECHNIQUES ”** under our supervision from **SEPTEMBER TO OCTOBER .**

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CERTIFICATE

This is to certify that **Mr. Saleth Robin G** (Roll No: 42910017) pursuing his M.Sc., Chemistry at Department of Chemistry, Sathyabama Institute of Science and Technology, Chennai has successfully completed his Internship programme in our organization from 22.09.2023 to 04.10.2023 under the guidance of Dr. Mousumi Gharai, Scientist.

I wish success in all his future endeavours.

Chennai

04.10.2023

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About CSIR



The Council of Scientific & Industrial Research (CSIR), known for its cutting edge R&D knowledgebase in diverse S&T areas, is a contemporary R&D organization. Having a pan-India presence, CSIR has a dynamic network of 38 national laboratories, 39 outreach centres, 3 Innovation Complexes and 5 units. CSIR's R&D expertise and experience is embodied in about 4600 active scientists supported by about 8000 scientific and technical personnel.

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CSIR has put in place CSIR@80: Vision & Strategy 2022 – New CSIR for New India. CSIR's mission is “to build a new CSIR for a new India” and CSIR's vision is to “Pursue science which strives for global impact, the technology that enables innovation-driven industry and nurtures trans-disciplinary leadership thereby catalyzing inclusive economic development for the people of India”.

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About NML- Jamshedpur



CSIR-National Metallurgical Laboratory (CSIR-NML) is a premier Indian research organisation dedicated to various facets of Minerals, Metals and Materials science, technology, industrial services and human resource development. Since inception, CSIR- NML has diversified its research areas ranging from extractive metallurgy, alloy development and import substitution, refractory material development, corrosion studies, mathematical and physical modeling of metallurgical processes, mineral research, advanced materials and materials tailoring, integrity evaluation of critical industrial components, surface engineering and cleaner and sustainable metals production. The Laboratory has made notable contributions in the areas of mineral beneficiation and agglomeration, ferrous and nonferrous metallurgy, alloy development and processing, materials science & engineering and, resource conservation & environment. CSIR-NML is also carrying out major activities for creating awareness among the common masses on issues relating to health, environment, rural technology and sustainable development. The Laboratory has kept pace with changing research scenarios and needs of the country. In the last few years, greater emphasis is given to industry sponsored research and, alignment with government program; namely, Make in India, Innovate in India, Strategic sector needs, Swatch Bharat, Societal and skill India, etc. The activities of the Laboratory touch upon several major sectors relevant to the growth of India, including iron and steel, power and energy, oil and gas, automotive, railways, strategic, societal and others.

About NML- Madras



CSIR National Metallurgical Laboratory (NML) is a premier laboratory within the ambit of Council of Scientific and Industrial Research (CSIR), New Delhi, a statutory body under Government of India. CSIR National Metallurgical Laboratory Madras Centre (NML-MC) an extension Centre of CSIR NML, over the years has undertaken several R&D projects in the areas of mineral beneficiation, micro-structural characterization and failure analysis of various industrial components. The Centre has developed flow sheets for the beneficiation of ferrous and non-ferrous, industrial minerals which include limestone, feldspar, barites etc. In recent years, the Centre has made significant contributions to the development of column flotation technology for Indian mineral industry. Commercial flotation columns were commissioned for the beneficiation of sillimanite and barites. The principle R& D's of the department includes

- Process development in mineral beneficiation (barites, beach sand minerals, iron ore, limestone, graphite & coal)
- Investigations on failure analysis of metallurgical components, root cause analysis and remedial measures

- Development of value added products from mineral/mines wastes/rejects.

The Centre is actively working towards developing value added products from mineral/mine wastes and also provides services for characterization of tiles, paver blocks and natural building stones. The esteemed notable clients of the department includes Kerala Mines and Minerals Ltd., English India Clay Ltd., RAMCO Cements Ltd., TATA Steel, JSW, Hinduja Foundries, NTPC , Hinustan Zinc Ltd. Indian Rare Earths Ltd and etc.

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COAL

1.1. INTRODUCTION

1.1.1. COAL

One of the most abundant and widely distributed fossil fuel is coal. Coal is a carbon rich material that occurs in the stratified sedimentary deposits. Coal is a heterogeneous material composed of organic and inorganic substances. It is formed from vegetation, that was consolidated between rock strata and converted by the combined effects of pressure and heat over millions of years to form coal. However in recent years, due to the volatility in oil and increase in natural gas cost; coal has gained more interest and is being used as the primary source of raw material for chemical industry.

It is commercially mined in more than 50 countries. On an average the annual world coal consumption is about 5800 million tons of which about 75% is used for electricity production. The significant uses of coal include steel production, cement manufacture and as a liquid fuel. The Carboniferous period marks the period of formation of coal which is also known as the first coal age. But the major coal deposits were formed 270-350 million years ago (i.e. Upper Carboniferous period). The geochemical process that transformed plant debris to coal is called coalification and is expressed as follows.

Peat → Lignite → Subbituminous Coal → Bituminous Coal → Anthracite

1.1.2 COMPONENTS OF COAL

The components of coal include volatile matter which is composed of aliphatic carbon atoms or aromatic hydrocarbons and mineral matter. Ash consists of inorganic matter from the earth's crust like limestone, iron, aluminum, clay, silica, and trace elements (concentrations of less than 1000 ppm [$<0.1\%$ of a rock's composition] of zinc, copper, boron, lead, arsenic, cadmium, chromium, selenium)

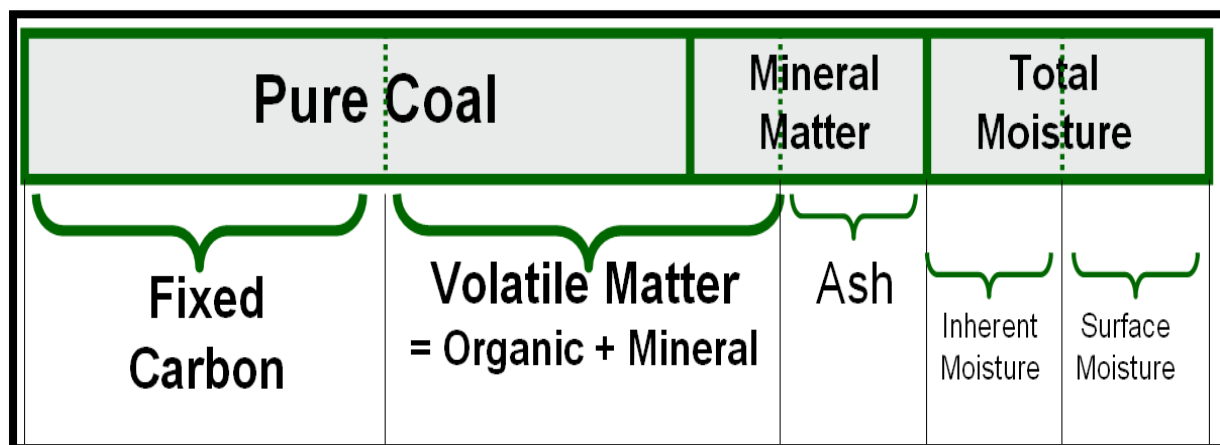


Fig.1.1. Composition of coal

1.1.3 TYPES OF COAL

Coal can be classified on basis of geological processes of decomposition of dead biotic material over a period of time as follows-

Lignite: Lignite is a low grade brown coal, which has high moisture content and lowest energy content. Lignite coal is a young coal deposits that were not subjected to extreme heat or pressure. The principal lignite reserves are in Neyveli in Tamil Nadu and are used for generation of electricity.

Subbituminous coal: It has a higher heating value than lignite. Subbituminous coal typically contains 35-45 %carbon, compared to 25-35 % for lignite.

Bituminous Coal: The deep buried coal subjected to increased temperatures is bituminous coal. It is the most popularly utilized coal in commercial use. Metallurgical coal is high grade bituminous coal which has a special value for smelting iron in blast furnaces.

Anthracite: Anthracite is the highest quality hard coal. In India coal occurs in rock series of two main geological ages, namely Gondwana, which ages 200 million years and in tertiary deposits which are only about 55 million years old.

1.1.4 GRADING OF COAL

According to the Indian Ministry of Coal, the gradation of non-coking coal is based on Gross Calorific Value (GCV), the gradation of coking coal is based on ash content and for semi coking /weakly coking coal it is based on ash and moisture content(Source: Ministry Of Coal, Government of India).

A. Grades of Coking Coal

Table 1.1 Grading of coking coal

GRADE	ASH CONTENT
Steel Grade -I	Not exceeding 15%
Steel Grade- II	15% - 18%
Washery Grade-I	18% - 21%
Washery Grade -II	21% - 24%
Washery Grade -III	24% -28%
Washery Grade –IV	28% - 35%
Washery Grade –V	35% - 42%
Washery Grade –VI	42% - 49%

B. Grades of Semi-coking and Weakly Coking Coal

Table 1.2 Grading of semi coking coal

GRADE	ASH+ MOISTURE
Semi coking grade –I	Not exceeding 19%
Semi coking grade -II	19%-24%

C. Grades of Non-coking Coal

Table 1.3 Grading of non-coking coal

GCV BAND (K.Cal./Kg.)	GRADE
Exceeding 7000	G-1
6700-7000	G-2
6400-6700	G-3
6100-6400	G-4
5800-6100	G-5
5500-5800	G-6
5200-5500	G-7
4900-5200	G-8
4600-4900	G-9
4300-4600	G-10
4000-4300	G-11
3700-4000	G-12
3400-3700	G-13
3100-3400	G-14
2800-3100	G-15
2500-3100	G-16
2200-2500	G-17

1.1.5 APPLICATIONS OF COAL

The uses of coal in various sectors are as follows:

❖ Electricity Generation

Power generation is the primary use for coal worldwide. Here, coal is burnt to create steam that drives turbines and generators for the production of electricity.

❖ Metal Production

Metallurgical (coking) coal is a key ingredient in steelmaking. Coal converted to coke is used to produce around 70% of the world's steel. Coal is also widely used in the production of other metals including aluminium and copper.

❖ Cement Production

Coal is used as a key energy source in cement production. By-products of coal combustion such as fly ash also play an important role in cement manufacture and the wider construction industry.

❖ Chemical And Pharmaceutical Industries

Many chemical products are manufactured from the by-products of coal. Refined coal tar is used to make chemicals such as creosote oil, naphthalene, phenol and benzene.

❖ Other Industries

Other major users of coal include the paper, textile and glass industries. Coal is also used in the manufacture of carbon fibre and specialist ingredients such as silicon metals, which are used to produce ingredients for the household and personal care sectors.

1.1.7 COAL ANALYSIS METHODOLOGIES

The chemical and physical composition of coal can be analyzed according to International and national standards coal analyses guidelines i.e. American Society for Testing and Materials standards for coal analyses (ASTM). The Quality of coal can be determined by two ways

- i) Proximity analysis
- ii) Ultimate analysis

i. Proximity Analysis Of Coal

The proximity analysis gives the amount of gross impurities present in the coal sample . This method is used to determine moisture content, volatile matter, ash content and fixed carbon of coal (J. G. Speight, 2005).

a) Moisture Content

The heating of coal at a temperature slightly above the boiling point of water causes a loss of weight from the sample that is defined as moisture. The moisture content of coal ranges from about 5% to almost 70%. Moisture is an undesirable constituent of coals because it reduces the heating value .The moisture of air-dried, finely powdered coal sample was determined by heating accurately weighed quantity of a sample in a silica crucible at 105⁰ – 110°C for 1 hour in an electric oven. The loss in the weight of sample is found out by the difference in weights. The percentage of moisture was calculated as

$$\text{Percentage of moisture} = \frac{\text{Loss in weight of the sample}}{\text{Weight of air-dried sample}} \times 100$$

b) Volatile Matter

Volatile matter (VM) in coal refers to the thermally decomposed product which liberates upon heating coal at high temperature in the absence of air. The percentage of volatile matter is determined by heating an accurately weighed air-

dried coal sample in a silica crucible covered with lid. The heating is carried out at $925 \pm 5^\circ\text{C}$ in a furnace for exactly seven minutes in the absence of air.

$$\text{Percentage of volatile matter} = \frac{\text{Loss in weight of the sample}}{\text{Weight of air-dried sample}} \times 100\% - \text{moisture \%}$$

c) Ash content

Ash is residue obtained after complete combustion of coal, which consists of CaO, MgO, SiO₂, Al₂O₃ and etc. The coal sample whose volatile matter was found was then heated in a furnace at $725 - 750^\circ\text{C}$ without lid for 1 hour. Cooled and weighed. Process of ignition, cooling and weighing was repeated till combustion is complete which is indicated by constant weight of the residue.

$$\text{Percentage of Ash} = \frac{\text{Weight of ash(residue)}}{\text{Weight of dried sample}} \times 100$$

d) Fixed Carbon

The Fixed carbon in coal is the amount of carbon that remains in the coal sample after volatile matter is driven off. The fixed carbon in the coal can be calculated as follows

$$\text{Percentage of fixed carbon} = 100 - (\text{moisture\%} + \text{volatile matter\%} + \text{ash\%})$$

Quality of coal depends on Percentage of fixed carbon; it should be as high as possible. Fixed carbon varies inversely with volatile matter. More the percentage of fixed carbon, higher is the calorific value.

1.2 PROCEDURE

1.2.1 DETERMINATION OF MOISTURE

- * Take the given sample (Raw coal-Head sample).
- * Take the empty weight of the crucible and note the value.
- * The given sample (Raw coal-Head sample) of 0.5000-0.5050g is taken.
- * Note the value by weighing the sample approximately.
- * And the sample is placed in the oven for 1 hour at 100°C.
- * After heating the sample cool for few minutes and then note the sample weight.

1.2.2 DETERMINATION OF ASH

- * Take the given sample (Raw coal-Head sample).
- * Take the empty weight of the crucible and note the value.
- * The given sample (Raw coal-Head sample) of 0.5000-0.5050g is taken.
- * Note the value by weighing the sample approximately.
- * And the sample is placed in the Furnace for 2 1/2 hour at 900°C.
- * After heating the sample cool for few minutes and then note the sample weight.

1.2.3 DETERMINATION OF VOLATILE MATTER

- * Take the given sample (Raw coal-Head sample).
- * Take the empty weight of the crucible and note the value.
- * The given sample (Raw Coal-Head sample) of 0.5000-0.5050g is taken.
- * Note the value by weighing the sample approximately.
- * And the sample is placed in the Furnace for 7 minutes at 900°C.
- * After heating the sample cool for few minutes and then note the sample weight.

CALCULATIONS

1. $A-B/C \times 100$ $A = 18.1547$

MOISTURE %

1. $A-B/C \times 100$ $A = 18.1547$
 $B = 18.1501$

$A-B = 0.0046$ $C = 0.5022$

$A-B/C = 0.0091$

$A-B/C \times 100 = 0.9159$

2. $A-B/C \times 100$

$A = 25.3391$

$A-B = 0.0058$

$B = 25.3333$

$A-B/C = 0.0115$

$C = 0.5031$

$A-B/C \times 100 = 1.1528$

ASH %

1. $D-C/A \times 100$ $D = 25.4426$

$C = 25.2989$

$D-C = 0.1437$

$A = 0.5004$

$D-C/A = 0.2871$

$D-C/A \times 100 = 28.7170$

2. $D-C/A \times 100$

$D = 24.2882$

$C = 24.1438$

$D-C = 0.1444$

$A = 0.5034$

$D-C/A = 0.2868$

$D-C/A \times 100 = 28.6849$

VOLATILE MATTER %

$$1. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M$$

$$M1 = 15.1107$$

$$M2 = 16.112$$

$$M3 = 15.8927$$

$$(M) \% = 0.9159$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 = 21.8715$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 20.9841$$

$$2. \frac{[M2 - M3]}{[M2 - M1]} \times 100 - M$$

$$M1 = 15.0054$$

$$M2 = 16.0101$$

$$M3 = 15.7917$$

$$(M) \% = 1.1528$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 = 21.7378$$

$$\frac{[M2 - M3]}{[M2 - M1]} \times 100 - M = 20.5850$$

Fixed carbon %

$$100 - (\text{Ash \%} + \text{Moisture \%} + \text{Volatile matter \%})$$

1. $100 - (28.7170 + 0.9159 + 20.9856)$

$$100 - 50.6185 = 49.3815$$

$$\text{Fixed carbon} = 49.3815 \%$$

2. $100 - (\text{Ash \%} + \text{Moisture \%} + \text{Volatile matter \%})$

$$100 - (28.6894 + 1.1528 + 20.5850)$$

$$100 - 50.4227 = 49.5773$$

$$\text{Fixed carbon} = 49.5773 \%$$

1.4 RESULTS AND DISCUSSIONS

Moisture %

Sample Name: (Raw Sample- Head Sample)

Empty weight	Sample weight(C)	Total weight before (A)	Total weight after (B)	Moisture % $A-B/C \times 100$
1. 17.6525	0.5022	18.1547	18.1501	0.9159
2. 24.8360	0.5031	25.3391	25.3333	1.1528

Ash %

Empty weight(C)	Sample weight(A)	Total weight before	Total weight after (D)	Ash % $D-C/A \times 100$
1. 25.2989	0.5004	25.7993	25.4426	28.7170
2. 24.1438	0.5034	24.6472	24.2882	28.6894

volatile matter %

Empty weight	sample weight	Total weight before (M2)	Total weight after (M3)	Volatile matter % $\frac{M2-M3}{M2-M1} \times 100$
1. 15.1107	1.0013	16.1124	15.8927	20.9841
2, 15.0054	1.0047	16.0101	15.7917	20.5850

Fixed carbon %

Ash %	Moisture %	volatile matter %	Fixed carbon 100-(Ash %+Moisture % +volatile matter %)
1. 28.7170	0.9159	20.9856	49.3815
2. 28. 6849	1.1528	20.5850	49.5773

1.5 CONCLUSION

* From the given sample the moisture percentage obtained = (I)0.9159%
(II)1.1528%

* From the given sample the ash percentage obtained = (I)28.7170%
(II) 28.6849%

* From the given sample the volatile matter percentage obtained =(I) 20.9856%
(II) 20.5850%

* From the given sample the fixed carbon percentage obtained =(I) 49.3815%
(II) 49.5773%

CHAPTER 2

INSTRUMENTAL TECHNIQUES

2.1. UV-VISIBLE SPECTROPHOTOMETER (ABSORPTION SPECTROSCOPY):

The principle of UV-Visible spectrophotometer is that the greater the number of molecules capable of absorbing light of a given wavelength, the greater the extent of light absorption. Furthermore, the more effectively a molecule absorbs light of a given wavelength, the greater the extent of light absorption. From these guiding ideas, the following empirical expression, known as the Beer–Lambert Law, may be formulated.

$$A = \log(I_0/I) = ecl$$

Where,

A = absorbance

I_0 = intensity of light incident upon sample cell

I = intensity of light leaving sample cell

c = molar concentration of solute

l = length of sample cell (cm)

e = molar absorptivity

The term $\log(I_0/I)$ is also known as the absorbance (or the optical density in older literature) and may be represented by A. The molar absorptivity (formerly known as the molar extinction coefficient) is a property of the molecule undergoing an electronic transition and is not a function of the variable parameters involved in preparing a solution. The size of the absorbing system and the probability that the electronic transition will take place control the absorptivity, which ranges from 0 to 10^6 . Values above 10^4 are termed high-

intensity absorptions, while values below 10^3 are low-intensity absorptions. Forbidden transitions have absorptivities in the range from 0 to 1000.

The Beer–Lambert Law is rigorously obeyed when a single species gives rise to the observed absorption. The law may not be obeyed, however, when different forms of the absorbing molecule are in equilibrium, when solute and solvent form complexes through some sort of association, when thermal equilibrium exists between the ground electronic state and a low-lying excited state, or when fluorescent compounds or compounds changed by irradiation are present.

2.1.2. WORKING PRINCIPLE

The typical ultraviolet–visible spectrophotometer consists of a light source, a monochromator and a detector. The light source is usually a deuterium lamp, which emits electromagnetic radiation in the ultraviolet region of the spectrum. A second light source, a tungsten lamp, is used for wavelengths in the visible region of the spectrum. The monochromator is a diffraction grating and its role is to spread the beam of light into its component wavelengths. A system of slits focuses the desired wavelength on the sample cell. The light that passes through the sample cell reaches the detector, which records the intensity of the transmitted light I . The detector is generally a photomultiplier tube, although in modern instruments photodiodes are also used. In a typical double-beam instrument, the light emanating from the light source is split into two beams, the sample beam and the reference beam. When there is no sample cell in the reference beam, the detected light is taken to be equal to the intensity of light entering the sample I_0 .



Fig.2.1 UV-Visible Spectrophotometer

Model: UV-160A, Manufacturer: Shimadzu Corporation

The sample cell must be constructed of a material that is transparent to the electromagnetic radiation being used in the experiment. For spectra in the visible range of the spectrum, cells composed of glass or plastic are generally suitable. For measurements in the ultraviolet region of the spectrum, however, glass and plastic cannot be used because they absorb ultraviolet radiation. Instead, cells made of quartz must be used since quartz does not absorb radiation in this region.

The instrument design just described is quite suitable for measurement at only one wavelength. If a complete spectrum is desired, this type of instrument has some deficiencies. A mechanical system is required to rotate the monochromator and provide a scan of all desired wavelengths. This type of system operates slowly, and therefore considerable time is required to record a spectrum. A modern improvement on the traditional spectrophotometer is the diode-array spectrophotometer. A diode array consists of a series of photodiode detectors positioned side by side on a silicon crystal. Each diode is designed to

record a narrow band of the spectrum. The diodes are connected so that the entire spectrum is recorded at once. This type of detector has no moving parts and can record spectra very quickly. Furthermore, its output can be passed to a computer, which can process the information and provide a variety of useful output formats. Since the number of photodiodes is limited, the speed and convenience described here are obtained at some small cost in resolution. For many applications, however, the advantages of this type of instrument outweigh the loss of resolution.

2.1.3 SAMPLE ANALYSIS

By the use of wavelength selector in the spectrophotometer, the light then passes through a sample. For all analyses, measuring a reference sample, often referred to as the "blank sample", such as a cuvette filled with a similar solvent used to prepare the sample. If an aqueous buffered solution containing the sample is used for measurements, then the aqueous buffered solution without the substance of interest is used as the reference. The reference sample signal is then later used automatically by the instrument to help obtain the true absorbance values of the analytes.

The quartz sample holders are required for UV examination because quartz is transparent to the majority of UV light. Air may also be thought of as a filter because wavelengths of light shorter than about 200 nm are absorbed by molecular oxygen in the air. A special and more expensive setup is required for measurements with wavelengths shorter than 200 nm, usually involving an optical system filled with pure argon gas. Cuvette-free systems are also available that enable the analysis of very small sample volumes, for example in DNA or RNA analyses.

2.1.4 THE ORIGIN OF UV BAND STRUCTURE

For an atom that absorbs in the ultraviolet, the absorption spectrum sometimes consists of very sharp lines, as would be expected for a quantized process occurring between two discrete energy levels. For molecules, however, the UV absorption usually occurs over a wide range of wavelengths because molecules (as opposed to atoms) normally have many excited modes of vibration and rotation at room temperature. In fact, the vibration of molecules cannot be completely “frozen out” even at absolute zero. Consequently, a collection of molecules generally has its members in many states of vibrational and rotational excitation. The energy levels for these states are quite closely spaced, corresponding to energy differences considerably smaller than those of electronic levels. The rotational and vibrational levels are thus “superimposed” on the electronic levels. A molecule may therefore undergo electronic and vibrational–rotational excitation simultaneously. Because there are so many possible transitions, each differing from the others by only a slight amount, each electronic transition consists of a vast number of lines spaced so closely that the spectrophotometer cannot resolve them. Rather, the instrument traces an “envelope” over the entire pattern. What is observed from these types of combined transitions is that the UV spectrum of a molecule usually consists of a broad band of absorption centered near the wavelength of the major transition.

2.1.5 APPLICATIONS OF UV SPECTROSCOPY

- Detection of Impurities

It is one of the best methods for the determination of impurities in organic molecules. Additional peaks can be observed due to impurities in the sample and it can be compared with that of standard raw material. By also measuring the absorbance at a specific wavelength, the impurities can be detected.

- Structure elucidation of organic compounds

It is useful in the structure elucidation of organic molecules, such as in detecting the presence or absence of unsaturation, the presence of heteroatoms.

- UV absorption spectroscopy can be used for the quantitative determination of compounds that absorb UV radiation. UV absorption spectroscopy can characterize those types of compounds that absorb UV radiation thus used in the qualitative determination of compounds. Identification is done by comparing the absorption spectrum with the spectra of known compounds. This technique is used to detect the presence or absence of a functional group in the compound. The absence of a band at a particular wavelength is regarded as evidence for the absence of particular group.
- Kinetics of reaction can also be studied using UV spectroscopy. The UV radiation is passed through the reaction cell and the absorbance changes can be observed. Many drugs are either in the form of raw material or in the form of the formulation. They can be assayed by making a suitable solution of the drug in a solvent and measuring the absorbance at a specific wavelength.
- Molecular weights of compounds can be measured spectrophotometrically by preparing the suitable derivatives of these compounds.
- UV spectrophotometer may be used as a detector for HPLC.

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