

## APPLICATION OF EMISSION SPECTROSCOPY

**D.FARVEZ BASHA**

*First Year-B.Sc Physics,  
Thanthai Hans Roever College,  
Perambalur (India)*

### I. INTRODUCTION

In emission spectroscopy, a sample is excited by absorbing thermal or electronic energy and the radiation emitted by the excited sample is studied for both qualitative and quantitative analysis. Most of the spectroscopic techniques are related to molecules but emission spectroscopy is related to atoms. Emission spectroscopy is regarded as the most reliable method for elemental quantitative analysis available at present. If proper precautions are taken, the method can be used for quantitative analysis of about seventy elements at concentration level as low as 1ppm.

### II. TYPES OF EMISSION SPECTRA:

When a beam of light is allowed to pass through a prism or grating, it is broken up into its constituent colours. This array of colours is known as spectrum.

#### ➤ **Continues Spectra:**

A continuous spectrum is characterised by generally uninterrupted emission over a considerable wave-length region and by the absence of sharp lines or discrete bands. This type of spectrum is obtained whenever matter in the bulk is heated. For example, incandescent solids like iron or carbon emit continuous spectra when they are heated until they glow.

#### ➤ **Band Spectra:**

A band spectrum is given out by the excited molecules. Each molecule emits bands which are characteristics of the molecule concerned and that is why we call this as molecule spectrum also. A band spectrum consists of group of lines so close together that under ordinary conditions they appear into continuous bands.

#### ➤ **Line Spectra:**

A line spectrum consists of discrete irregularly spaced lines. This type of spectrum is obtained when the light emitting substance is in the atomic state. Therefore, it is also called **atomic spectrum**. In molecules, there are vibrational and rotational energy levels which are superimposed on the electric energy levels but in atoms there are only electric energy levels. Line spectrum depends upon the types of atom. Therefore, it is of predominant type in emission spectroscopy.

### III. EXCITATION SOURCES

#### ➤ Flames

Flame is used for these molecules which do not require very high temperatures for excitation and dissociation into atoms. Different flames having different temperatures are used in emission spectroscopy. Flame temperature and flame composition have an influence on interferences that may cause erroneous reading to occur.

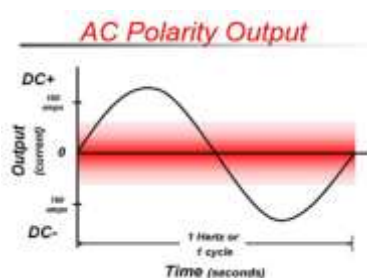
#### ➤ Direct current arc:



This is one of the most versatile excitation devices for both quantitative spectro chemical emission analysis. The function of variable resistance in the circuit  $R$  is to adjust the current whereas the inductance coil  $L$  stabilizes the flow of current. The advantages of direct current are as follows:

- i. The high temperature generated in the DC arc makes it a very sensitive excitation source.
- ii. The excitation energy provided by this arc is not electrical but mainly thermal and is thus sufficient for exciting all the metal elements.
- iii. By this arc, emission species are neutral atoms rather than ions.

#### ➤ Alternating current arc:



The essential components of a circuit for an alternating current arc. A high voltage of 2000 to 5000 V is maintained by a transformer. A current of 1 to 5A is maintained in the primary circuit by adjusting a variable inductance  $L_1$  in this circuit. One more effect of the stop and start the gap temperature is much less than with the direct current arc, with a lower sensitivity.

#### ➤ Alternating current spark:

Schematically illustrates a circuit for alternating current spark sources. In the primary circuit a voltage of 110 to 220 is maintained which is stepped up to 10,000 to 40,000V by the step up transformer. When it loses charge, it follows a damped oscillatory discharge, typically oscillating at a frequency of 10 MHz. The initial pulse of a discharge may produce temperature up to 40000 K.

#### IV.ELECTRODES



The electrodes used in emission spectroscopy are of two types: Self electrodes and graphic electrodes.

When the materials under investigation is a good conductor and can withstand high temperatures, the materials as such is used for the electrodes. Such type of electrodes are known as self electrodes. For example, analysing alloys or metal powders can be pressed into solid discs or cylinders to be used as electrodes.

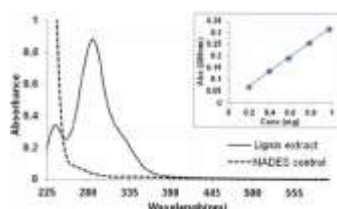
When the substance under investigation is not a good conductor and cannot withstand high temperatures, it is placed in a small cavity of the lower graphite electrode whereas the upper electrode is also made up of graphite which is brought to a point in a pencil sharpener. The centre post decreases wandering of the arc source to improve the reproducibility whereas the narrow neck improves the sensitivity.

#### V.SAMPLE HANDING

Samples for emission spectroscopy may be solids or liquids. When a sample is a solid having good conductance properties and can withstand high temperature, this can be used for making electrodes required for the electric discharge.

If a solid not a good conductor and cannot withstand high temperatures, it is first powdered and then mixed with powdered the lower graphite electrode. After it loaded, the electrode is placed in the circuit. The function of adding powdered graphite to the powdered solid sample is to increase reproducibility, (ie) minimizing aimless wandering of the arc. Both types of liquid sample holders can be used for aqueous as well as for non-aqueous solvents. Organic solutions ignite in the discharge and may cause erratic emission. This increases considerably when the rotating disc electrode is employed.

#### VI.QUANTITATIVE ANALYSIS



In order to perform qualitative analysis, the emission spectrum of the sample is recorded on a photographic plate or film. The emission lines are then identified by comparing them with lines from the known samples. If three or more sensitive lines of the element in the unknown sample are identified definitely, this is usually a sufficient

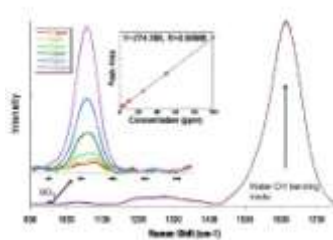
proof that this element is present in the unknown, But this is a difficult procedure because it involves identification of many lines. This can be simplified by detecting few raies ultima or RU lines.

When a metal is excited in an electric discharge, a complex spectrum is obtained. This spectrum contains many lines, some strong, some weak. If the concentration of the metal is decreased, the weaker lines disappear. If more dilution is carried out, many lines will disappear until few lines are left. The lines that are left are called the raies ultima or RU lines.

An alternative method to calculate wavelength is given by Hartman which is also an exact though more tedious interpolation method. Hartman's equation is as follow:

$$\lambda_x = \lambda_0 + c / d_x - d_0$$

## VII. QUANTITATIVE ANALYSIS

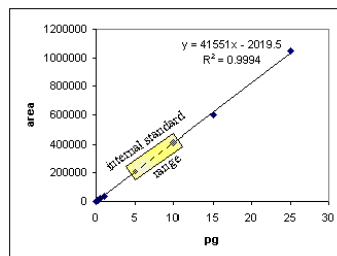


We will now discuss some of the procedures employed in quantitative spectroscopy analysis.

### ➤ Comparison sample method:

In this method, the spectra of the unknown sample and various standards are photographed alternately on the same plate under the same set of conditions. After developing the plate, one can calculate the concentration of the desired constituent by comparing the blackening of the lines of this constituents with the same lines on the standards. Visual or photometric comparison of blackening may be used.

### ➤ Internal standard method:



In this method, an internal standard is selected. Its known amount is added to the sample. It is not essential that the internal standard element should not be present in the original sample because this would not allow our knowing the total amount present.

When the sample having internal standard is excited in the electrical discharge, the sample as well as internal standard emit spectral lines. These are photographed on a photographic plate. We have to do two measurements:

i.Intensity of the sample emission line is measured.

ii.Intensity of the internal standard emission line is measured.

Another property of internal standard is that it should resemble the elements being determined to the extent that their boiling points and chemical reactivity should be similar.

➤ **Spectrographic standards:**

In addition to the choice of an internal standard, a most critical phase in the development of a spectrographic method involves the preparation or acquisition of a set of standard samples from which a calibration curve is prepared. For the ultimate inaccuracy the standards must closely approximate the samples both in chemical composition and in physical form. In some cases standards can be synthesized are most readily prepared by this method.

### VIII.SPECIFIC APPLICATIONS

Emission spectroscopy has been used in many field to provide useful information. Some of these are listed in this section.

➤ **Metals and alloys**



The iron and steel as well as the aluminium industries are based on emission spectroscopic analysis in all steps of their process. This is done to control the composition of the molten metals before further processing. For example, the presence of nickel, chromium, silicon, manganese, molybdenum, copper, aluminium, arsenic, tin, cobalt, vanadium, lead, etc.

Emission spectroscopy has also been used for the analysis of aluminium alloys, copper alloys, magnesium alloys, lead alloys and tin alloys.

➤ **Oils:**



One is interested to know trace elements present in crude oil because some of these can poison the catalysts used in the cracking process. Some of these trace elements are vanadium, copper, nickel and iron. All these have been estimated by employing automatic emission spectrograph. This gives the car designer valuable information on the parts of automobiles that need protection. This study has been possible by the use of emission spectroscopy.

➤ **Animals and Men:**

Emission spectroscopy has been used for the analysis of many elements including sodium, potassium, zinc, copper, calcium, magnesium, nickel and men. Changes in trace metal concentrations have been studied as related to the aging process.

➤ **Plants and Soils:**

Emission spectroscopy has been used to detect 40 elements in plants and soils. This has been done to diagnose deficiency problems in plants. When these are known, One can suggest the application of deficient elements through soil applications or through sprays applied to the leaves.

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