SPECTROSCOPIC METHOD USING PHOTOELECTRIC RECORDING TECHNIQUE FOR DETERMINATION OF TRACE ELEMENTS

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بسم الله الرحمن الرحيم

" سبحانك لا علم لنا إلا ما علمتنا
إنك أنت العليم الحكيم "

صدق الله العظيم

بسم الله الرحمن الرحيم

SPECTROSCOPIC METHOD USING PHOTOELECTRIC RECORDING TECHNIQUE FOR DETERMINATION OF TRACE ELEMENTS

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SUMMARY

The present work has been devoted for the development of an atomic emission spectrometric technique for the determination of trace elements using the wall-stabilized plasma arc as an excitation source. The thesis in hand introduces the experience of the photoelectric technique for the measurements of spectral intensity in the present laboratory, where the photographic technique was the only method used in the field of atomic emission spectroscopy. The excitation source used is a home-made wall-stabilized plasma arc which proved to be a favourable source for the excitation of solution samples. A considerable part of the present work has been devoted for the application of the plasma diagnostic techniques for the investigation of the arc plasma. Various methods has been used for the measurement of temperature and electron density.

The second part of the experimental work deals with the application of the photoelectric sequential recording technique in conjunction with the wall-stabilized arc for the determination of trace elements. For this investigation the group of the rare earth elements has been chosen. This choice is based on the importance of this group of elements in the various technological fields. For instance,

they are used in manufacturing high quality alloys and special types of glasses as that which transmits infrared and absorps UV. They become also of great importance in nuclear technology as efficient neutron absorbers and in many other fields.

The present thesis is divided into seven chapters The first chapter, the introduction, discusses in general, the atomic emission spectroscopy and the physical quantities that are measured in the plasma of the excitation source. These quantities are the intensity and the width of the spectral lines. Also, a comparison is made between the photographic and the photoelectric techniques for recording and measuring the spectra.

The second chapter gives a review for the development of the plasma sources used in optical emission spectroscopy pecially the d.c. plasma sources. These sources fall in two categories. The first one contains those in which the measurements are carried out in the plume of the arc. These are called plasma jets. The second category includes plasma arc in which measurements are carried out in the current carrying portion of the plasma. The present source falls in this category. The characteristics of these sources and its application in field of spectral analysis are discussed.

Chapter III gives a brief account on the important theories of radiation emission from plasmas and the different models describing the plasma. The theoritical models are: the complete thermal equilibrium, the local thermal equilibrium, the partial local thermal equilibrium and the corona model. This chapter gives also the important relations between the intensity of spectral lines and the plasma parameters. At the end of this chapter the width of spectral lines and the different broadening mechanims are briefly reviwed.

Chapters IV, V and VI describe the experimental work and the results obtained. Chapter IV describes the construction of the wall_stabilized arc and the method of sample introduction into its plasma as well as the experimental arrangement for the photoelectric recording and measuring of spectra. The light from the analytical gap of the arc illuminates a 1m Mc Pherson scanner monochromator whose dispersive element is a plane grating of 1200 g mm^{-1}. As a detector an EMI 9558 Q end_on photomultiplier tube is used. The photo current is measured in the linear mode of the Mc Pherson logarithmic ratiometor type 782 whose measuring range lies between 10^{-8} to 10^{-5} amperes in four steps. The current can be measured directly from scale or recorded on a strip chart recorder. In order to allow for the comparison of spectral line intensity in different wavelength region, the spectral response curve of the whole system including the monochromator and the photomultiplier was measured. This measurement was carried out using a standarized tungsten lamp and covers the wavelength range from 300 nm to 600 nm.

In chapter V the different spectroscopic plasma diagnostic techniques are applied for the determination of the parameters of the present plasma. The atom excitation temperature is determined using the Boltzmann plot with a group of FeI lines in the wavelength region between 371.99 nm to 376.3 nm. The obtained plots are straight lines with the experimental points well-fitted to them which indicates that the population of the excited levels follows Boltzmann distribution law. The obtained excitation temperatures lies between $4011^{o}k$ to $5035^{o}k$ for arc currents between 14 A to 22 A. Ti II spectral lines in the wavelength region from 321.7 nm to 324.2 nm are used for the determination of ion-excitation temperature. The results revealed also that the population of the excited levels of ions follows Boltzmann distribution law and is governed by nearly the same temperature as that of the atomic states. The resultant ion - excitation temperature lies between 4121^{o}k to 5075^{o}k for arc current between 14 A to 22 A. The ionization temperature is measured using an expression resulting from the combination of Boltzmann and Saha equations. This expression gives the intensity ratio of a atom /ion line pair of a element as a function of the ionization temperature and the electron density and hence enables the determination of both simultaneously. In this experiment the intensity ratios of atom/ion line pairs of Ba, Ca, Sr and Ti are used. The accurate determination of these ratios was made possible with the help of the measured spectral response curve of the optical system. From the obtained plots, the ionization temperature as well as the electron density were determined. The obtained values of ionization temperature lies between 4708^{o}k to 6697^{o}k for arc currents between 14 and 22 A. The corresponding electron density values are 2.06×10^{20} m^{-3} to 6.21×10^{21} m^{-3}. It is to be mantioned here that the values of the electron density obtained in this way depend on the values of the transition probabilities inserted in the Boltzmann-Saha expression as well as on the excitation temperature. For this reason it was advisable to determine the electron density using another method. The Stark broadening of the hydrogen line H_β, one of the most widely methods for the determination of the electron density, is used for this purpose. This method has the advantage that it does not depend on any assumption concerning thermal equilibrium and can be applied for any type of plasma. In order to ensure the accurate determination of the Stark half-width of H_β., the apparaties half-width was determined using a He-Ne Laser and the Doppler half-width was calculated at the values of the obtained excitation temperatures. The two half widths were convoluted and the resultant half_width was removed from the observed one, using tables of Voigt function, to yield the pure Stark half_width. The electron density values obtained in this way were found to be between $3.10 \times 10^{20}\ m^{-3}$ to $5.81 \times 10^{20}\ m^{-3}$ and are lower than those obtained from the Boltzmann plot. These values are considered more accurated and were used to calculate the ionization temperature. The resultant values of the ionization temperature range between $4995^{o}k$ to $5186^{o}k$ and are in agreement with those of the excitation temperatures. From the results obtained in this chapter it could be concluded that the plasma of this source is close to the state of local thermal equilibrium.