SPECTROSCOPIC METHOD FOR DETERMINING RARE EARTHS IN LOCAL MINERALS

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بسم الله الرحمن الرحيم

وقل اعملوا فسيرى الله عملكم ورسوله والمؤمنون

«صدق الله العظيم»

الله

بسم الله الرحمن الرحيم

SPECTROSCOPIC METHOD FOR DETERMINING

RARE EARTHS IN LOCAL MINERALS

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SUMMARY

SUMMARY

The present work has been devoted for the development of a spectroscopic technique for the determination of the group of rare earth elements in local phosphate deposits. The developed technique uses a home-made plasma excitation source, that is the wall-stabilized argon arc constructed at the Spectroscopy Laboratory, National Research Centre. This arc has many features which makes it a suitable source for such a task. Its spectrum shows very low background emission and no molecular bands are observed except the OH-band spectrum at 306.4 nm. At the optimal operating conditions used for the analytical application of the arc, the intensity of the argon lines is highly reduced and depending on the current may completely disappear. Moreover at these conditions the arc emits a simple spectrum. With these properties of the present source spectral interference is at minimum. This is a favourable advantage of the arc specially for the rare earth elements with its complex spectra. For this reason a considerable part of the work has been dedicated for achieving the optimal excitation conditions and studying the effect of the matrix of the phosphate samples on the plasma temperature as well as on the intensity of the emitted spectral lines.

The thesis is divided into five chapters:-

The first chapter, the introduction, describes the requirements which should be fulfiled in an analytical technique applied for elemental determination. It gives a brief account on the efforts that had been devoted to excitation sources for optical emission spectroscopy which resulted in the development of plasma sources like inductively coupled plasma (ICP) and the direct current plasma (DCP). The introduction discusses also the importance of developing analytical techniques for the rare earth elements in geological samples specially those techniques which require no pretreatment of the samples for enrichment. At the end of this chapter the need of a spectroscopic technique for the determination of REE in local phosphate samples is discussed and therefore, the aim of the present thesis is given.

The second chapter gives a review on the physical techniques used for the determination of REE in geological materials. These techniques are, the optical emission spectroscopy using d.c. carbon arc, neutron activation, mass spectrometry, X-ray fluorescence, and the inductively coupled plasma atomic emission spectrometry. For each technique a review on the published work is also given. This chapter reviews also the previous efforts carried out for the determination of REE in Egyptian phosphates.

Chapter III is devoted to review briefly the fundamental theories related to the emission of spectral lines. It gives the important mathematical equations which relate the line intensity and the plasma temperature. This is necessary for the study of the effect of sample matrix on the plasma conditions. Also the factors influencing the line width are discussed.

Chapter IV is devoted for the experimental work and the obtained results. It is divided into four parts. The first part describes the experimental set-up, including the wall-stabilized arc which was used as an excitation source, the plane grating spectrograph PGS2 (Carl Zeiss Jena) and its illumination as well as the methods used for the emuslion calibration. The second part describes the phosphate samples and standard solutions preparation for nebulization and the selection of interference-free spectral lines for the analytical application. The effect of sample matrix on the plasma temperature and the intensity of the analysis lines is described in the third part of this chapter. For this purpose, atom-excitation temperature was measured using the intensity distribution over a group of FeI lines in the wavelength region between 371.99 nm and 376.3 nm. Also ion-excitation temperature was measured using a group of TiII lines in the wavelength region from 321.7 nm to 324.2 nm. This study revealed that the sample matrix has a slight and insignificant effect on the excitation temperatures and consequently on the intensity of spectral lines. This is explained to be due to the presence of relatively high concentration of the easily ionizable element, potassium. The last part of this chapter describes the construction of the analytical calibration function, the determination of the limits of detection for REE (which was found to be 0.008 $\mu g\ ml^{-1}$ for Yb and 0.374 $\mu g\ ml^{-1}$ for Er), as well as the precision of determination. The obtained values of the precision of the REE spectral lines was found to be 0.02 for Eu and 0.097 for Tm. Finally the result of the application of the developed technique for the determination of REE in twelve phosphate samples collected from Abu-Tartur plateau is given. The significance of these results in the field of exploration geochemistry is given as an appendix.

In the conclusion, chapter V the most important characteristics of the developed technique are summarized.

The results obtained in this work reveal that the technique is available for similar studies in any minerals or solution samples.