University of Basrah College of Education for Pure Sciences Department of Chemistry



# Determination of Some Anions and Cations by Home-made Ion Chromatography System

#### A Thesis

Submitted to the College of Education for Pure Sciences/University of Basra in Partial Fulfillment of the Requirements for the Degree of Doctor of Philosophy in science- Analytical chemistry

### BY

## Khawla Salman Abdul Rassol Al-Myahy

(M. Sc. Analytical chemistry 2000)

Asst. Prof . Supervisor Dr. Kamil H. Al-Sowdani

## **Summary**

The work in this study consists of three chapters. The first one described a literature review about theoretical and development of ion chromatography.

The experimental part, which includes optimum conditions for analysis of anions and cations were presented in the second chapter. Also, a part of this chapter focused on the build - up and construction of the home – made Ultra Violate (UV )- Flow Injection (FI) and UV - IC system.

Chapter three includes four parts. The first part, was designed to develop a simple, selective, fast and validated UV - FI method for the analyzing of  $NO_3^-$ ,  $Br^-$ , and  $NO_2^-$  ions in different water samples.

The linearity was 0.5-6, 2-8, and  $0.5-8~\mu g~ml^{-1}$  for  $NO_3^-$ ,  $Br^-$ , and  $NO_2^-$  respectively. The detection limits were 0.1, 0.5,  $0.25~\mu g~ml^{-1}$  for  $NO_2^-$ ,  $Br^-$ , and  $NO_3^-$  respectively with correlation coefficient which were 0.9970, 0.9980 and 0.9965 for  $NO_2^-$ ,  $Br^-$ , and  $NO_3^-$  also respectively.

Total nitrogen in the range  $0.1-6~\mu g~ml^{-1}$  and  $0.5-6~\mu g~ml^{-1}$  can be easily determined by incorporating a 6 W UV – Lamp in the UV –

system and using digestion method with persulphite respectively. The sample through put was 15 sample/h.

The second part includes a validated UV – IC method for simultaneous determination of  $NO_3^-$ ,  $Br^-$ , and  $NO_2^-$  ions. The retention times for each ion, separately , and in mixtures were determined , which are used to identify each, and the peak height corresponding the concentration of each ion in standards and water samples solutions .The range of linearity , detection of limit and correlation coefficient were  $1.5-120~\mu g~ml^{-1}$ ,  $0.07-1~\mu g~ml^{-1}$  and 0.9980-0.9970 respectively.

IC system equipped with conductivity detector was used to the analysis of anions  $(NO_3^-, Br^-, and NO_2^-)$  and cations  $(Na^+, NH_4^+, and K^+)$  were described in the second part.

The third part includes the main aim of this study to simultaneous analysis of anions and cations by single injection with isocratic home – made IC with conductivity detector.

Six ions  $(NO_3^-, Br^-, and NO_2^-, Na^+, NH_4^+, and K^+)$  can be easily analyzed by using saulphosalycilic acid and crown ether as eluent and using IC home – made equipped with conductivity detector.

The ranges of linearity were  $5-150~\mu g~ml^{-1}$  and  $5-140~\mu g~ml^{-1}$  for anions and cations respectively, detection of limit 0.07 -  $1~\mu g~ml^{-1}$  and  $0.1-0.25~\mu g~ml^{-1}$  for anions and cations respectively. Also, the sample through put was 21~sample / h.

Finally, recovery studies were conducted in orders to a evaluated the home – made system by using the standard additions methods. The results were in the range 99.90 – 100.40 % for anions and 100.22 – 100.50 % for cations which is satisfactory. Many water samples and pharmaceutical preparations were run for analyzing some anions and cations by the Home – made ion chromatograph system.