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Optimization of the Critical Instrumental parameters in Analysis of some Trace Elements in Sodium chloride Matrix by Flame Spectrometry

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Abstract

The optimization of the instrumental parameters of flame atomic absorption including effect of burner height, flame stoichiometry of fuel to oxidant was studied. The presence of highly interfering matrix such as sodium chloride on the absorption signals of cadmium, chromium, manganese and nickel were also investigated in order to examine the effect of matrix concentration on the absorption signal and then the reality of the absorbance values given by the instrument, hence the sensitivity and reproducibility of the instrument readings and then sensitivity and reproducibility of the analysis were tested . Improvements of analysis in presence of highly interfering matrices using some releasing agent were also studied.

Keywords: *Interferences, flame spectrometry, optimization of parameters.*

1. Introduction

The flame technique is the oldest of the AAS techniques. For many years it was the 'workhorse' for the determination of trace elements and also for main constituents, and even nowadays it is difficult to imagine a routine analytical laboratory without this technique; due to its simplicity and economy[1]. Usually in flame atomic absorption spectrometry (FAAS) 1 - 2 % salt solution are employed. Working with more concentrated salt solutions with the conventional techniques of sample introduction leads to number of related problems e,g.: blockage of nebulizer and burner , considerable background absorption , transport and chemical interferences and drop in sensitivity and precision [2]. Along with this the direct FAAS analysis of samples of high salt content has several analytical advantages over conventional FAAS: number of analytical steps is reduced and limits of detection are reduced [3],[4].The nebulization of a washing solution prior to and after each sample efficiently washes both the nebulizer and burner, which has a favorable effect on the analysis of samples of high salt content [5]. The advantages and drawbacks of the FAAS analysis of samples of high matrix content were the basis for the formulation of the purpose of the present investigation: to perform a direct quantitative FAAS analysis of trace elements (cadmium, chromium, manganese and nickel) in highly concentrated aqueous solution of NaCl [6]. The aim of this work is to compare the critical instrumental parameters such as burner height and fuel-oxidant stoichiometry during the analysis of metal ions in a standard solution and in presence of some matrices; Second step to study the effect of addition of releasing agents and some additives on the absorption signal of the metal ions in presence of some highly interfering matrices such as sodium chloride, urine and serum; Finally is to study the effect of the sample pretreatment at different temperatures before caring out instrumental analysis[7],[8].

2.Experimental

All glass wares were socked with de-ionized water and 0.2% purified nitric acid. The standard solutions were prepared from highly pure stock solutions specially made for analysis by flame atomic absorption spectrometer. A novAA350 double beam flame atomic absorption spectrometer equipped with deuterium lamp background correction was used , with computer control for the different components in the instrument like slit width , fuel flow , intensity of light by controlling lamp currant applied ,burner height , wavelength , and Stoichiometry of fuel to oxidant , In all experiments an air/acetylene flame was used; Sodium chloride solution was prepared by dissolving known Wight from NaCl in de-ionized water to produce 5, 10, 15, and 20 percent by weight.

3.Results and discussion

The effects of fuel – oxidant Stoichiometry (l/h) and burner height (mm) for chromium ion; manganese ion; cadmium ion and nickel ion in 2ppm standard solutions are shown in figures (1.a , 1.b, 2.a , 2.b, 3.a , 3.b, 4.a , 4.b).

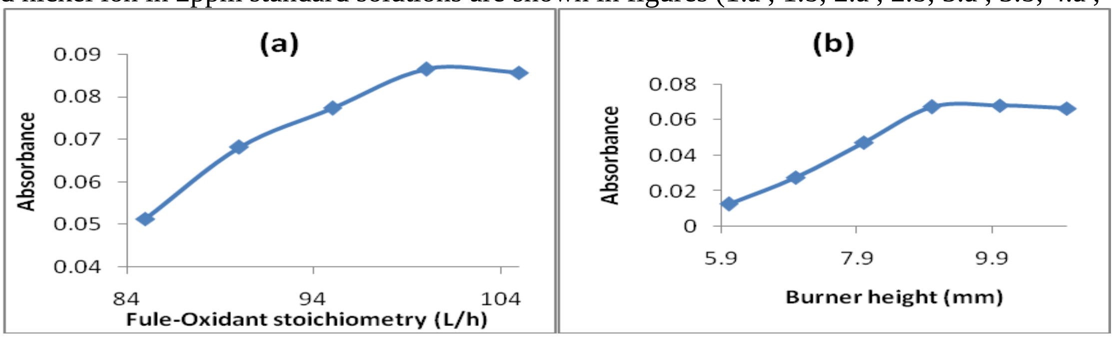

Figure (1): The effect of two parameters on absorbance signal of 2 ppm Cr (a): effect fuel-oxidant Stoichiometry at burner height 10 (b): effect of burner height at fuel-oxidant Stoichiometry 90.

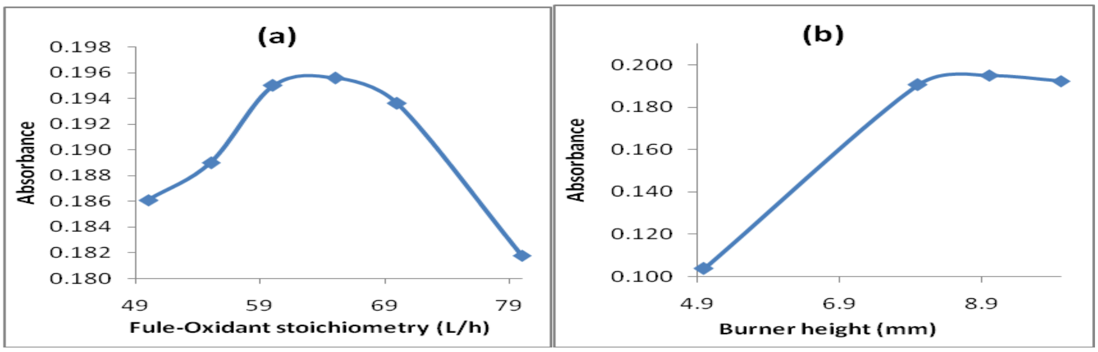

Figure (2): The effect of two parameters on absorbance signal of 2 ppm Mn (a): effect fuel-oxidant Stoichiometry at burner height 9 (b): effect of burner height at fuel-oxidant Stoichiometry 60.

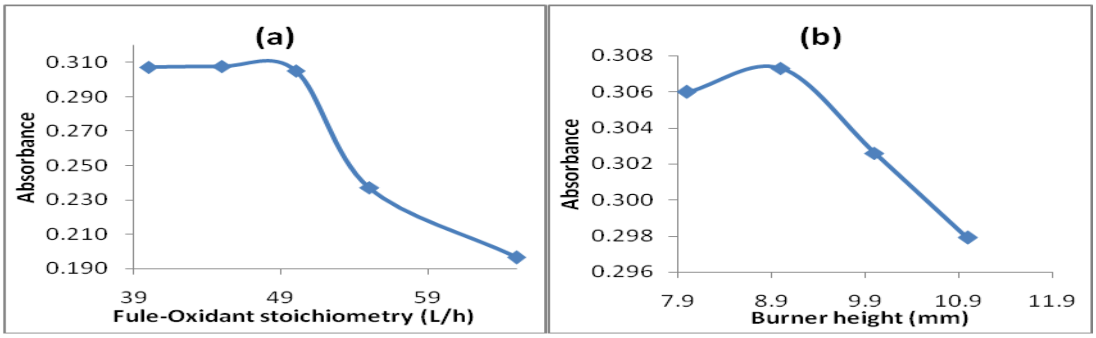

Figure (3): The effect of two parameters on absorbance signal of 2 ppm Cd (a): effect fuel-oxidant Stoichiometry at burner height 9 (b): effect of burner height at fuel-oxidant Stoichiometry 40.

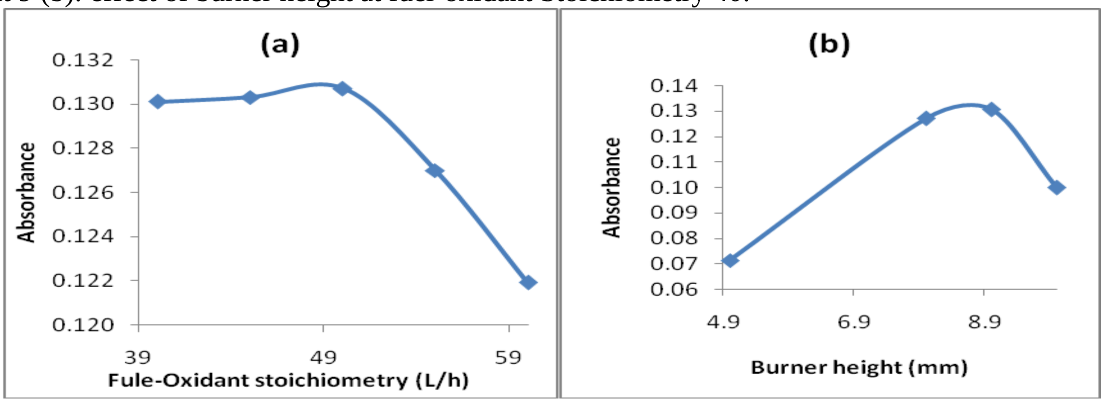

Figure (4): The effect of two parameters on absorbance signal of 2 ppm Ni (a): effect fuel-oxidant Stoichiometry at burner height 9 (b): effect of burner height at fuel-oxidant Stoichiometry 50.

The effects of different concentrations of sodium chloride matrix on the absorption signal of some metal ions compared with the absorption signal of the same metal ions in a standard solution are shown in figure (5). As can be seen from the figure (5), the absorbance value (no. of free atoms in gaseous state) is strongly reduced by 40 % in case of Cadmium in presence of 5 % NaCl (sea water) compared with stranded solution with no addition. The reduction of the signal continue with increasing sodium chloride concentration, this means that as concentration of sodium chloride increase the absorption read by the instrument is erroneous and the interference becomes more and more. Manganese and chromium are also affected almost the same as cadmium and the reduction of signal is about 50 % as compared with their standard solutions. Nickel which is known as less volatile element showed a stable absorption values at 5 % sodium chloride solution and a slight decrease in of absorption at 10 % matrix concentration.

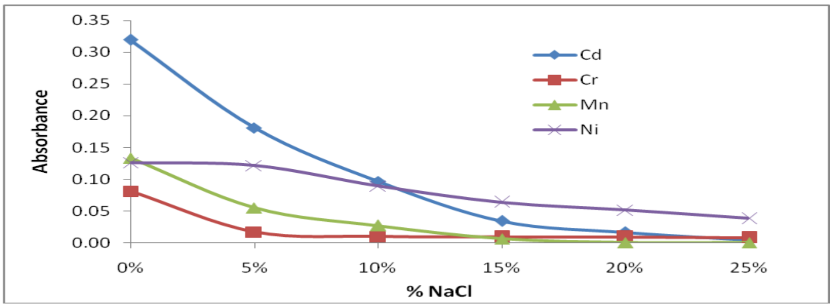

Figure (5): Effect of different NaCl concentrations matrix on the absorption signal some trace elements

Optimization of cadmium analysis in presence of sodium chloride matrix is investigated using a standard solution of 2 ppm Cd in 5 % NaCl, the results obtained for fuel-oxidant Stoichiometry and burner height are shown in figures (6.a and 6.b) respectively. Better absorption values could be obtained at 40 fuel oxidant and burner height of 7mm instead of 40 and 9 mm for cadmium in case of standard solution.

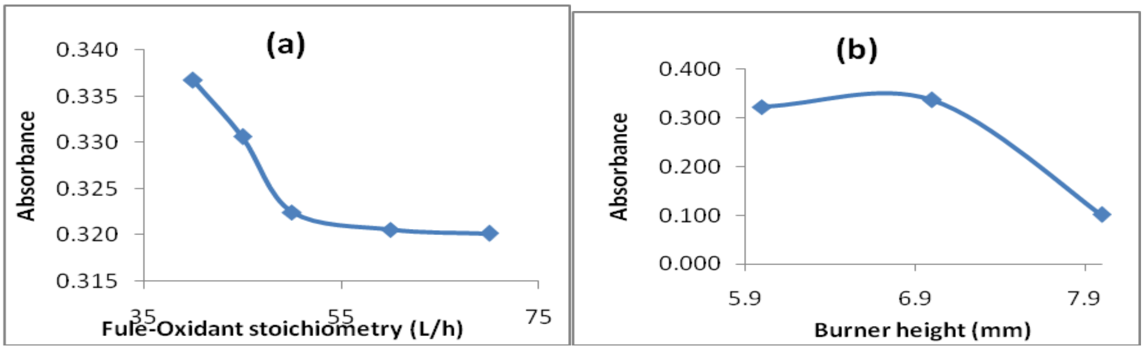

Figure (6): The effect of two parameters on absorbance signal of cadmium analysis in presence of 5% sodium chloride (a): effect of fuel-oxidant stoichiometry at burner height 7 (b): effect of burner height at 40 fuel-oxidant stoichiometry.

The effects of some additives such as releasing agents on the absorption signal of cadmium in sodium chloride matrix and compared to that value in case of standard solution at different temperatures are investigated. Results obtained at different temperature are shown in figure (7). As can be seen from the figure below that sodium chloride reduces the absorption signal of cadmium, slight enhancement for absorption signal in case of adding EDTA as releasing agent, a very good effect is shown by addition of ethanol at 80°C and more reduction in signal is obtained in case of citric acid addition.

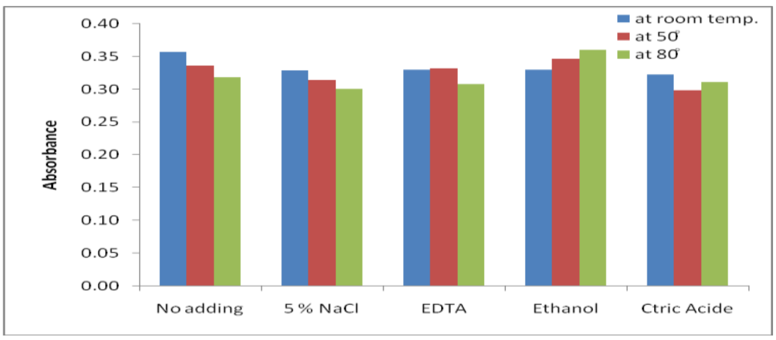

Figure (7): Absorbance of 2ppm Cd in 5% NaCl matrix with different additives at different temperatures.

Optimization of Nickel analysis in presence of sodium chloride matrix is investigated using a standard solution of 2 ppm Ni in 5 % NaCl. The results obtained for fuel-oxidant Stoichiometry and burner height are shown in figures (8.a

and 8.b) respectively; Better absorption values could be obtained at 60 fuel oxidant burner and 8 mm height of instead of 50 and 9 mm for Nickel in case of standard solution.

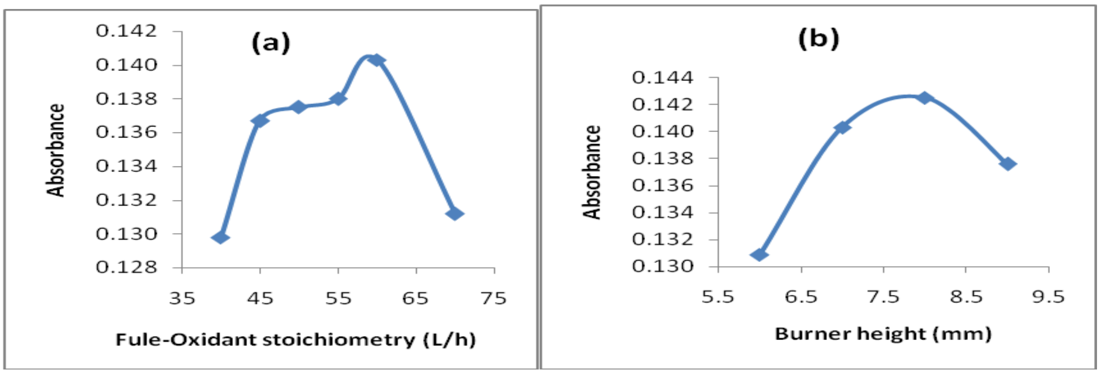

Figure (8): The effect of two parameters on absorbance signal of nickel analysis in presence of 5% sodium chloride (a): effect of fuel-oxidant stoichiometry at burner height 7 (b): effect of burner height at 60 fuel-oxidant stoichiometry.

The effects of some additives such as releasing agents on the absorption signal of Nickel in sodium chloride matrix are investigated and compared that of standard solution at different temperatures. Results obtained at different temperature are shown in figure (9). As can be seen from the figure below sodium chloride reduces the absorption signal of cadmium, more reduction in signal is obtained in case of adding EDTA as releasing agent, a very good effect is shown by addition of ethanol at 50 °and 80 °C,and more reduction in signal is obtained in case of citric acid addition.

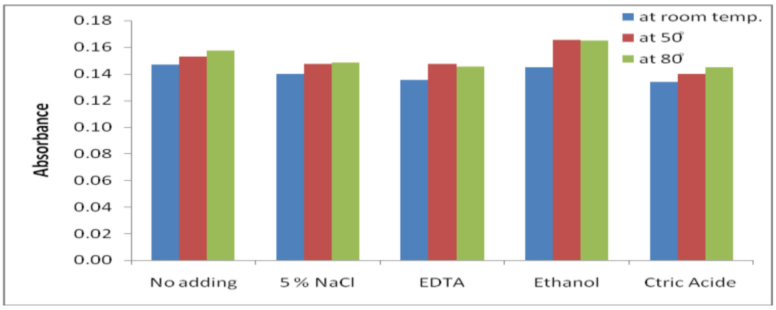

Figure (9): The absorbance of 2ppm Ni in 5 % NaCl with different additives at different temperatures.

Optimization of Chromium analysis in presence of sodium chloride matrix is investigated using 2 ppm Cr in 5 % NaCl, the results obtained for fuel-oxidant Stoichiometry and burner height are shown in figures (10.a and 10.b) respectively. Better absorption values could be obtained at 80 fuel oxidant and 9 mm burner height instead of 100 and 10 mm for Nickel in case of standard solution.

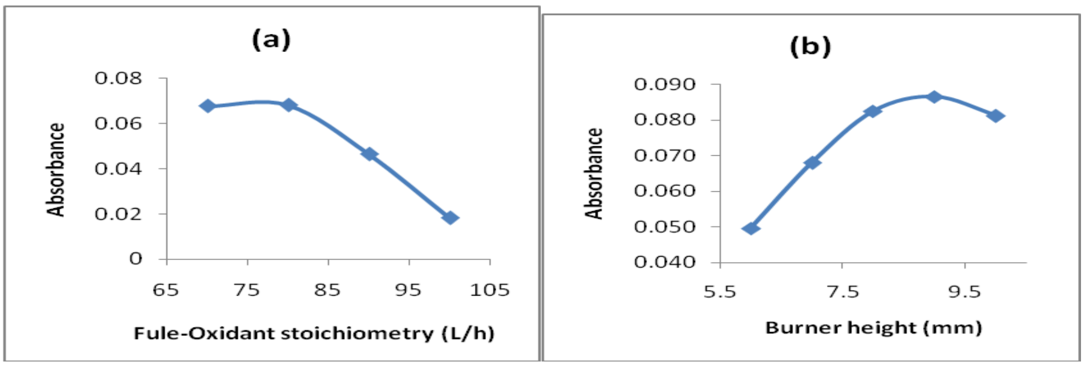

Figure (10): The effect of two parameters on absorbance signal of chromium analysis in presence of 5% sodium chloride (a): effect of fuel-oxidant stoichiometry at burner height 7 (b): effect of burner height at 80 fuel-oxidant stoichiometry.

The effects of some additives such as releasing agents on the absorption signal of Chromium in sodium chloride matrix are investigated and compared with that of standard solution at different temperatures. Results obtained at different temperatures are shown in figure (11). As can be seen from the figure below that sodium chloride reduces the absorption signal of Chromium, and the signal becomes slightly better in case of adding EDTA as releasing agent but the signal reduction at hgier temperatures, better effect can be shown by addition of ethanol but the signal reduction at hgier temperatures, and better effect can be shown by addition of citric acid addition at all temperatures.

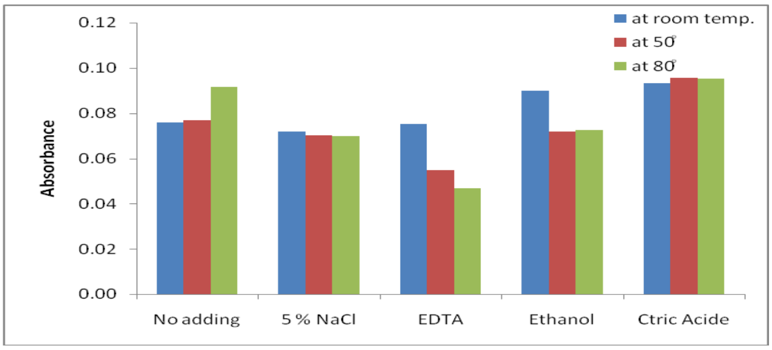

Figure (11): The absorbance of 2ppm Cr in 5 % NaCl with different additives at different temperatures.

Optimization of Manganese analysis in presence of sodium chloride matrix is investigated using a standard solution of 2 ppm Mn in 5 % NaCl. The results obtained for fuel-oxidant Stoichiometry and burner height are shown in figures (11.a and 11.b) respectively. Better absorption values are obtained at 65 fuel oxidant and 10 mm burner height instead of 60 and 9 mm for Manganese in case of standard solution.

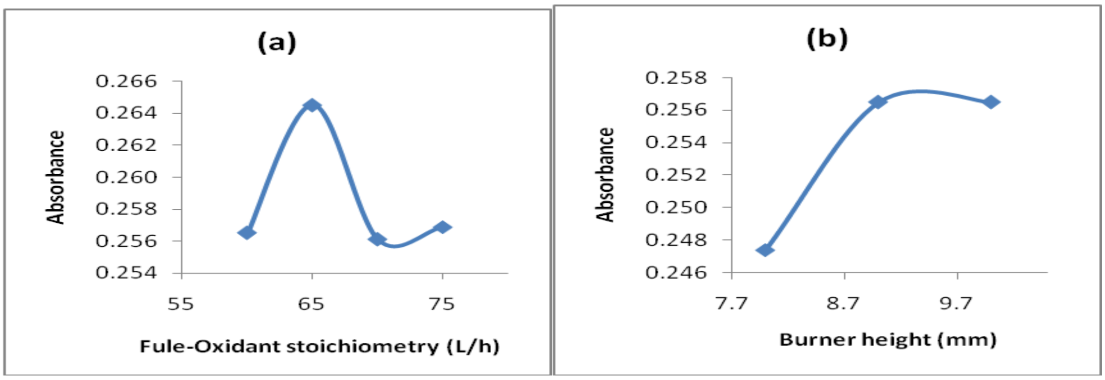

Figure (12): The effect of two parameters on absorbance signal of manganese analysis in presence of 5% sodium chloride (a): effect of fuel-oxidant stoichiometry at burner height 9 (b): effect of burner height at 65 fuel-oxidant stoichiometry.

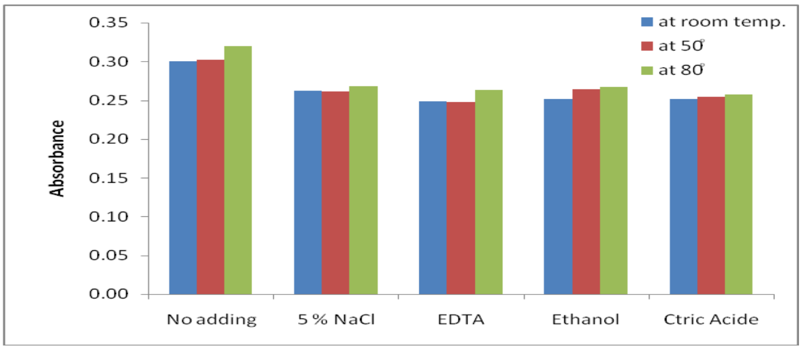

Figure (13): The absorbance of 2ppm Mn in 5 % NaCl with different additives at different temperatures.

The effects of some additives such as releasing agents on the absorption signal of Manganese in sodium chloride matrix are investigated and compared with that of standard solution at different temperatures. Results obtained at different temperatures are shown in figure (13). As can be seen from the figure below, sodium chloride reduces the absorption signal of Manganese, and more reduction in signal is obtained in case of adding EDTA as releasing agent, and reduction in case of addition of ethanol and citric acid addition.

4. Conclusion

During the analysis of trace elements in presence of highly interfering matrices such as sea water,very high attention should be paid in order to get real absorbance values and then real concentration values. Dilution of the sample may be carried out if the trace element high enough to be detected, otherwise addition of releasing agents. More accurate techniques should be used.

References

[1] Bernhard Welz," *Atomic Absorption spectrometry* ", Weinheim - New York: Wiley, , 3rd edition (1999) 149-208,477-572.

[2] John Edward Cantle, "*Atomic Absorption spectrometry*", Amsterdam - Oxford - New York : elsevier scientific publishing company, (1982), 95-200,1st edition.

[3] Richard D. Beaty and Jack D. Kerber, "*Concepts, Instrumentation and Techniques in Atomic Absorption Spectrometry*", Norwalk, CT, U.S.A: The Perkin-Elmer Corporation, (1990), 37-57, 2nd edition.

[4] The Perkin-Elmer Corporation, *"Analytical Methods for Atomic Absorption Spectroscopy"*, Printed in the United States of America: The Perkin-Elmer Corporation, (1996), 3-51.

[5] Bekjarov, G.; Kmetov, V.; Futekov, L., Trace analysis in highly concentrated salt solutions of sodium chloride and ammonium fluoride using flame atomic absorption spectrometry. *Z. Anal. Chem.*8, (1989), *335*, 971-974

[6] Diaz F.J. Determination of cyanocobalamin by atomic-absorption spectrophotometry with a pre-mix air-acetylene flame, Anal Chim Acta., Feb;58(2), (1972), 455-8. .

FJ, D., Determination of cyanocobalamin by atomic-absorption spectrophotometry with a pre-mix air-acetylene flame.

[7] Cresser, M. S.; Armstrong, J.; Cook, J.; Dean, J. R.; Watkins, P.; Cave, M., Atomic Spectrometry Update-Environmental Analysis. *Journal of Analytical Atomic Spectrometry* **,** 8, (1),(1993), 1R-44R.

[8] Yanagisawa, M.; Suzuki, M.; Takeuchi, T., Cationic interferences in the atomic absorption spectrophotometry of chromium. *Analytica Chimica Acta, 52* (2), (1970)**,** 386-389.