

Flow Injection Analysis Determination of Sodium Using Flame Photometric Method of Detection

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ABSTRACT

A flame photometric (FP) determination of sodium in a variety of solvents was performed. Water and 12 organic liquids (8 alcohols and 4 ketones) were used as solvents in steady state determination of sodium. A 3-fold enhancement of signal was obtained when methanol was used as a solvent.

Water and 8 alcohols were used both as sample solvents and as liquid carriers in flow injection analysis (FIA) using flame photometric detection for sodium determination. The best solvent was methanol and the best carrier was n-butanol. A 5.8-fold enhancement of the signal was achieved when methanol was used as solvent and n-butanol as carrier.

INTRODUCTION

The most widely used high sensitivity methods for the determination of alkali elements are the spectral methods: atomic emission (flame) and atomic absorption spectrometry.⁽¹⁾ Of the most important analyses carried out in any clinical laboratory are sodium and potassium.⁽²⁾ The flame photometric determination was the most used method⁽³⁾ until the advent of the use of ion-selective electrodes, but flame photometry continues to be used in many routine clinical chemistry laboratories.⁽²⁾

Recent reviews on flame injection analysis (FIA) show the rapid growth of FIA publications in the last ten years. (4,5,6) Such a large growth is due in part to the great promise FIA techniques have in clinical, environmental and industrial monitoring since they are easily adaptable for simultaneous determination of two or more parameters in the same sample. FIA has the advantages also of high frequency of sample analysis, the use of microliter sample volumes, simplicity and capability of accomodating different types of detectors. (7,8,9)

Inspite of the increased number of FIA papers published, no reports about flow injection analysis with flame photometric detection for the determination of alkali metals were found, although FIA with atomic absorption (FIA-AA) represents a major part of the flow injection analysis papers published. (4,5) The use of FIA-AA system with non-aqueous solvents and carriers was also demonstrated. (10,11,12) It was shown that a signal enhancement is achieved if an organic solvent is used either as solvent or as carrier or both. (10,11,12) In determination of copper and zinc, the FIA-AA signal was enhanced by 10.5- and 9.5 folds respectively, if a nonaqueous solvent is used. (10,11) Organic solvents burn efficiently in the flame and are more efficiently vaporized due to lower viscosity and surface tension. (13) In this study twelve organic solvents were used as sample solvents in steady state determination of sodium and an enhancement of the signal was obtained, and eight organic solvents were used, both as sample solvents and liquid carriers, in a flow injection analysis with flame photometry. A total of 81 combinations of sample solvent-liquid carrier pairs are reported. The sensitivity is improved by 5.8-fold with the use of optimum sample solvent-liquid carrier combination.

EXPERIMENTAL

REAGENTS:

A stock 1000 ppm sodium solution was prepared from sodium chloride dried at 140°C for 48h before its use. Distilled deionized water was used to prepare all aqueous solutions. Methanol, ethanol and diethylketone were obtained from E. Merck Darmstadt; 1-butanol, 1-propanol and methylisobutylketone (MIBK) were obtained from Cambrian Chemicals, Beddington, Farm Road, England; butanone, 1-pentanol and 3-pentanol were obtained from Koch-Light, Ltd. England; and acetone, isopropanol and 2-butanol were obtained from BDH Chemicals, Ltd.

APPARATUS:

A corning Flame Photometer with a natural gas flame and a Bryans chart recorder were used throughout this study. Teflon tubing 1-mm i.d. from Beckman Altex was used in the flow system. An injector from Valco Instrument Co., Huston, Texas, with a 52.5ul volume sample loop, was used to introduce the samples into the flow system. No pump was used; instead the negative pressure from the nebulizer was used to draw the carrier solvents from the reservoirs to the injector and to the burner of the flame photometer.

PROCEDURE:

Sodium standards in different solvents were prepared by diluting the sodium stock solution in the appropriate solvent. Except for water, methanol, ethanol and acetone, a few drops of ethanol were added to help dissolve the water droplets from the stock solutions in the other organic solvents. The same volume of water as that of stock solution was dissolved in each solvent, and in the same manner, to be used as blank. Five parts per million sodium standard solutions in all solvents were prepared and used for com-

paring the relative sodium signal in the different solvent-carrier combinations. The steady state signals in different solvents were obtained by immersing the tube in the appropriate solvent. The sample is aspirated to the nebulizer of the flame photometer. The FIA signals with different carriers were obtained by immersing the pumping tube in the appropriate carrier and injecting sodium sample solutions in the different solvents using the injection loop. The liquid carrier carries the sample plug to the port of the nebulizer of the flame photometer, where it is introduced to the flame. All the solvents were investigated as sample solvents and carriers except ketones which were avoided because they cause damage to the rubber o-ring of the injection valve. The flow rates were as given in table I.

**Table I: Flow Rate of Different Carriers in FIA-FP
Determination of Sodium**

Solvent	Flow rate (ml/min)
Water	3.4
methanol	3.7
ethanol	2.1
n-propanol	1.4
iso-propanol	1.2
1-butanol	1.0
2-butanol	1.0
1-pentanol	0.80
3-pentanol	0.60
butanone	4.1
acetone	5.8
MIBK	3.2
3-pentanone	3.1

RESULTS AND DISCUSSION:

Table II shows the relative intensities of signals for different concentrations of sodium determined by the steady state flame photometric method. The signal of 2 ppm sodium in aqueous solution was taken as unity, and the rest were calculated relative to it. It is clear from this table that methanol is the best solvent. The signal is 3-fold enhanced compared to aqueous system. From the secondary alcohols used, isopropanol is the best since 1.7-fold enhancement is achieved when it was used as solvent, whereas a reduction in the signal height is obtained when 3-pentanol was used, as solvent, compared to aqueous solution. From the ketonic solvents used, acetone is the only solvent that enhanced the signal. A 1.4-fold enhancement is obtained when acetone was used as solvent compared to the aqueous system.

Table II: Relative Signals of Sodium Standard Solutions in Water and Different Organic Solvents.

Solvent	Concentration (ppm)				
	2	4	6	8	10
H ₂ O	1	2.14	3.21	4.11	5.27
CH ₃ OH	2.90	5.72	8.55	11.5	14.5
C ₂ H ₅ OH	2.21	4.24	6.09	8.64	10.7
n-C ₃ H ₇ OH	1.76	3.52	5.13	7.24	8.95
2-C ₃ H ₇ OH	1.67	3.32	4.94	6.88	8.55
n-C ₄ H ₉ OH	1.43	2.86	4.29	5.84	7.35
2-C ₄ H ₉ OH	1.32	3.14	4.53	6.24	7.75
n-C ₅ H ₁₁ OH	1.29	2.58	3.69	5.32	6.60
3-C ₅ H ₁₁ OH	0.429	0.904	1.30	1.77	2.13
acetone	1.38	2.80	4.08	5.72	7.05
butanone	0.524	1.10	1.59	2.21	2.72
MIBK	0.750	1.43	2.10	2.94	3.65
3-pentanone	0.381	0.714	1.16	1.57	1.89

Table III: Relative signals of 5 ppm sodium standard solutions in the different sample solvent/liquid carrier pairs.

Solvent \ Carrier	H ₂ O	CH ₃ OH	C ₂ H ₅ OH	n-C ₃ H ₇ OH	2-C ₃ H ₇ OH	n-C ₄ H ₉ OH	2-C ₄ H ₉ OH	n-C ₅ H ₁₁ OH	3-C ₅ H ₁₁ OH
H ₂ O	1	1.60	1.85	2.64	1.24	3.80	1.43	2.0	1.50
CH ₃ OH	3.04	3.97	3.82	3.31	3.86	5.76	3.04	2.69	2.57
C ₂ H ₅ OH	1.47	2.76	2.72	2.38	2.37	3.82	1.70	1.38	1.16
n-C ₃ H ₇ OH	1.90	3.98	2.29	4.06	2.09	2.95	2.29	1.57	1.86
2-C ₃ H ₇ OH	2.00	3.29	2.76	2.86	2.67	2.60	2.19	1.79	1.71
n-C ₅ H ₁₁ OH	2.67	3.55	2.34	2.38	2.33	3.04	2.22	1.90	1.86
2-C ₄ H ₉ OH	2.33	2.95	2.10	2.67	1.81	2.49	1.57	1.52	1.33
n-C ₅ H ₁₁ OH	4.03	2.71	2.19	3.14	1.81	3.14	1.10	1.14	0.76
3-C ₅ H ₁₁ OH	2.79	2.11	1.38	1.90	0.81	2.05	0.62	0.48	0.38

Table III shows the relative FIA-signals for 81 solvent/carrier combinations for 9 solvents used. The signal of water solvent/water carrier was taken as unity, and the rest were calculated relative to it. The highest relative signal was achieved when methanol was used as solvent and n-butanol as liquid carrier. The best carriers in general are ethanol, methanol, n-propanol and n-butanol. When an aqueous solution of sodium was used, the order of carriers in enhancing the signal was: n-butanol > n-propanol > n-pentanol > ethanol > methanol > 3-pentanol > 2-butanol > isopropanol.

Figure 1 shows the calibration curves for sodium determination in aqueous solution using methanol, ethanol, and n-butanol as carriers. 3-fold enhancement is achieved when n-butanol was used as carrier.

Figure 2 shows the calibration curves for determination of sodium in ethanol and methanol solvents using n-

propanol and n-butanol as carriers. A 5.8-fold enhancement was achieved when methanol was used as solvent and n-butanol as carrier. This enhancement of the signal obtained when organic solvents were used, as solvents and carriers, is believed to be due to the limited sample dispersion and increased overall atomization efficiency, which is caused by a variety of factors, such as rate of aspiration, finer droplets due to lower surface tension, more efficient evaporation due to volatility of the organic solvent and combustion of the solvent or carrier.⁽¹³⁾ It is shown in table III that some of the relative signals are less than unity which means that the use of some nonaqueous solvents reduced the FIA signal. This has been previously observed in the FIA-AAS of zinc determination, where some organic solvents reduced the FIA-AA signal.⁽¹¹⁾

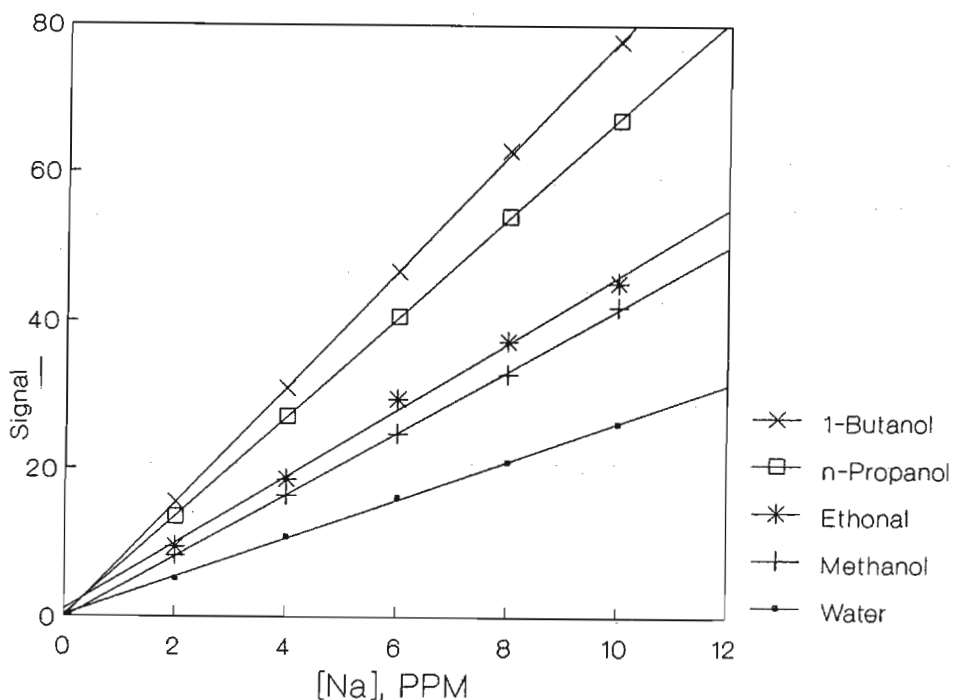


Figure 1: Calibration curves of sodium standard solutions in water using different carriers.

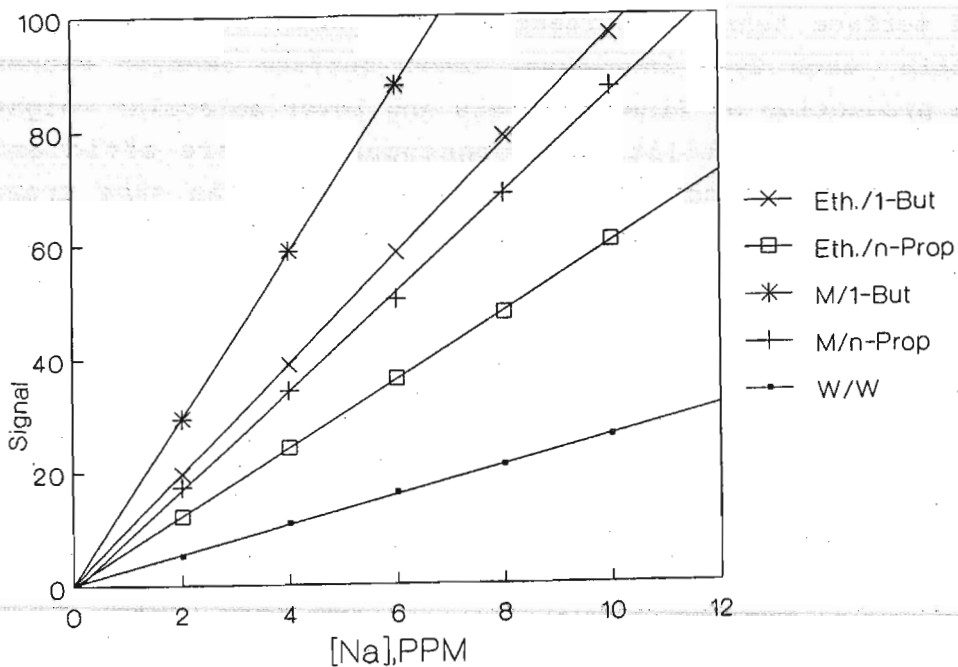


Figure 2: Calibration curves of sodium standards in different solvent/carrier systems.

Effect of Number of Carbon Atoms of Solvents in Steady State Determination of sodium:

Figure 3 shows that for the compounds of similar number of carbon atoms, the order of solvents in enhancing the atomic absorption signals is primary alcohols > secondary alcohols > ketones. It shows also that the signal of sodium decreases as the number of carbon atoms in the solvent molecule increases, in case of primary and secondary alcohols. In case of ketones, the signal decreased from acetone to 3-pentanone then increased again in the ketone (MIBK). This increase in signal with decrease in number of carbon atoms is believed to be due to different factors such as viscosity, volatility and surface tension. In case of primary and secondary alcohols, boiling point, viscosity

and surface tension increase as the number of carbon atoms within each type increases. Lower surface tension causes the production of finer droplets and lower molecular weight means more volatility and consequently, more efficient evaporation and combustion of the solvent. The same trend was also observed in copper determination using FIA-AAS. (14)

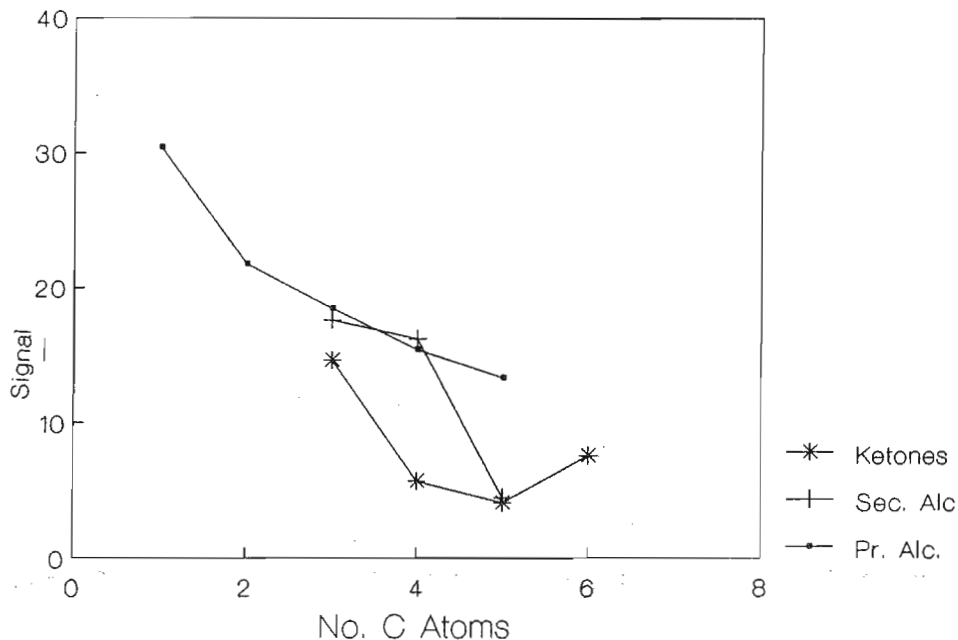


Figure 3: Effect of the number of carbon atoms of different solvents on the sodium steady state signals.

Conclusion:

It is demonstrated in this study that it is possible to perform analyses with enhanced sensitivity if the optimum solvent is used in steady state determination of sodium or if the optimum carrier-sample solvent combination is used in FIA. It is the first time that FIA is carried out using flame photometric detection. This adds new dimen-

sions to the use of flame photometer. In addition, the use of organic solvents in flame photometric determination of sodium which is an example that can be extended to other alkali elements in order to increase the sensitivity.

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