

AA-93 January 1990

Determination of Cu, Zn, Fe, Ca, Mg, Na and K in serum flame by atomic absorption spectroscopy

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Introduction

Several analytical methods are normally needed for the determination of Cu, Zn, Fe, Ca, Mg, Na and K in serum [1]. Even with determination of these elements by atomic absorption spectroscopy, various dilutions and some anti-interference reagents are required [2,3]. The procedures are relatively complicated and the addition of anti-interference reagents could easily lead to contamination problems. In this article, a simple method for the determination of seven elements in serum is introduced. One millilitre of serum is diluted to 10 millilitres with deionized water and the determination of seven elements is automatically performed.

Material and Method

Instrumentation

A SpectrAA-40 atomic absorption spectrometer, PSC-56 programmable sample changer, Epson LX-80 printer, and Cu, Zn, Fe, Ca, Mg, Na and K hollow cathode lamps from Varian were used in the procedure.

Parameters

The main instrument parameters for the analysis were as follows:

Element	Wavelength	Slit Width	Lamp Current	Burner Height	Acetylene Flow	Air Flow
	nm	nm	mA	mm	L/min	L/min.
Cu	324.8	0.5	4	10	1.70	13.5
Zn	213.9	1.0	4	10	2.00	13.5
Fe	248.3	0.2	8	10	2.50	13.5
Ca	422.7	0.5	3	4	2.50	13.5
Mg	202.5	1.0	3	10	2.50	13.5
Na	330.3	0.5	4	10	1.70	13.5
K	404.4	0.5	4	10	2.00	13.5

Reagents

The water used was doubly distilled and deionized. All reagents used for preparation of standard solutions and a anti-interference reagents were ultra pure grade. Standard solutions were prepared in glass bottles, previously washed with diluted nitric acid and rinsed with deionized water.

Standard Solutions

A stock mixed standard solution was prepared with the concentration of elements as follows:

Element	Cu	Zn	Fe	Ca	Mg	Na	K
Concentration							
(μg/mL)	5	5	5	200	50	5000	5000

A series of mixed calibration standards were prepared by diluting the stock standard with deionized water. The volume of stock standard and resultant concentration in each calibration standard were as follows:

Stock solution (mL)

	···-/								
0	1	2	3	4	5				
Concentration of element (µg/mL)									
0	0.10	0.20	0.30	0.40	0.50				
0	0.10	0.20	0.30	0.40	0.50				
0	0.10	0.20	0.30	0.40	0.50				
0	4.00	8.00	12.00	16.00	20.00				
0	1.00	2.00	3.00	4.00	5.00				
0	100	200	300	400	500				
0	10.0	20.0	30.0	40.0	50.0				
Total volume (mL)									
50	50	50	50	50	50				
	ntration 0 0 0 0 0 0 0 0 0 colume (r	ntration of element 0 0.10 0 0.10 0 0.10 0 0.10 0 1.00 0 1.00 0 1.00 0 10.0 volume (mL)	ntration of element (μg/mL) 0 0.10 0.20 0 0.10 0.20 0 0.10 0.20 0 0.10 0.20 0 4.00 8.00 0 1.00 2.00 0 100 200 0 10.0 20.0 rolume (mL)	ntration of element (μg/mL) 0 0.10 0.20 0.30 0 0.10 0.20 0.30 0 0.10 0.20 0.30 0 0.10 0.20 0.30 0 4.00 8.00 12.00 0 1.00 2.00 3.00 0 10.0 200 300 0 10.0 20.0 30.0 volume (mL)	ntration of element (µg/mL) 0 0.10 0.20 0.30 0.40 0 0.10 0.20 0.30 0.40 0 0.10 0.20 0.30 0.40 0 4.00 8.00 12.00 16.00 0 1.00 2.00 3.00 4.00 0 100 200 300 400 0 10.0 20.0 30.0 40.0 volume (mL)				

Procedure

1 millilitre of serum placed in a test tube is diluted to 10 millilitre with deionized water. The test tube is placed in the auto sampler carousel after mild shaking. The measurement is automatically carried out and the results are the average of two replicates according to the standard addition calibration method.

Results

Recovery Rate

The accuracy of the measurements have been expressed in terms of the recovery of spiked additions of each element as follows:

 Element
 Cu
 Zn
 Fe
 Ca
 Mg
 Na
 K

 Recovery(%)
 97-99
 92-103
 92-104
 91-94
 94-111
 96-100
 92-93

Percentage Deviation

The precision of measurements has been expressed in terms of percentage deviation as follows:

Element	Cu	Zn	Fe	Ca	Mg	Na	K
Percentage deviation (%)	2.2	2.9	3.9	1.3	1.7	8.0	3.1
No. of measurements	10	10	10	10	10	10	10

To compare the effect of different dilution ratios in the determination of Cu, Zn, Fe in serum, a mixed serum was diluted 1+4 and 1+9 respectively with deionized water, and the results were as follows:

Element	Cu (μg/mL)	Zn (μg/mL)	Fe (μg/mL)
Dilution 1+4	1.426 ±0.012	1.309 ±0.014	1.615 ±0.021
Dilution 1+9	1.564 ±0.012	1.419 ±0.036	1.715 ±0.024
Deviation (%)	+9.7	+8.4	+6.2
No of measuremen	nts 4	4	4

Two methods were compared for the determination of Ca, Mg, Na and K in serum. A mixed serum was diluted by 1+9 with deionized water and 1+49 with 0.25% SrCl₂ respectively. The results were as follows:

Element	Ca (μg/mL)	Mg (μg/mL)	Na (μg/mL)	Κ (μg/mL)
Dilution 1+4	49 83.63 ±0.60	21.61 ±0.22	3247 ±40	230.9 ±1.7
Dilution 1+9	9 95.30 ±1.00	23.51 ±0.30	3198 ±43	225.0 ±1.1
Deviation (%) +14.0	+8.8	-1.5	-2.6
No. of mea	surements 4	4	4	4

Discussion

In all methods of determining Cu, Zn Fe, Ca, Mg, Na and K in serum there are three key points: the dilution ratio, the use of anti-interference reagents and the composition of element groups. The method introduced here is simple in all three aspects. For the determination of Cu and Zn in serum, dilution ratios of 1+4 and 1+9 with deionized water have been used by other authors [2,5,6]. Our results show that serum diluted 1+4 can cause the Mark V burner to become partially clogged, but this does not occur for 1+9 dilution. Following is the comparison of uptake rates for the standard solution, and serum diluted 1+4 and 1+9 with deionized water:

Solution	Standard Solution	1+9 Dilution	1+4 Dilution
Uptake rate (mL)	4.28	4.10	3.95
Deviation percent	age (%)	-4.20	-7.70

With the determination of Fe in serum, a procedure to remove protein from the serum is often needed, [7]. However, direct dilution with deionized water shows a good recovery rate and is of course, more simple. A 1+9 dilution procedure with deionized water for the determination of Ca and Mg has a good recovery rate and can minimize contamination from anti-interference reagents [4,8].

In order to determine seven elements in the same diluted solution, less sensitive absorption lines are selected for the determination of Mg, Na and K. The procedure can be completed automatically with 150 measurements in one hour.

References

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