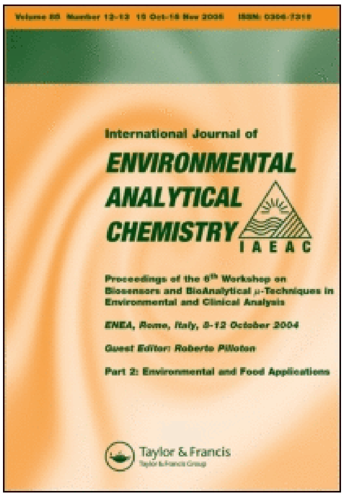

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The Determination of Trace Elements by Flame Atomic Absorption Spectrometry: Effect of the Composition of Standard Solution Matrices

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THE DETERMINATION OF TRACE ELEMENTS BY FLAME ATOMIC ABSORPTION SPECTROMETRY: EFFECT OF THE COMPOSITION OF STANDARD SOLUTION MATRICES

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The effect of using solution matrices in calibration standards different from those employed for the extraction steps in the determination of Cd, Cr, Cu, Ni and Zn were tested. Cu and Zn are little affected by matrix effects but, if the extractant is ammonium acetate, the use of other matrices may result in too high Cd, Cr and Ni values.

KEY WORDS: Cd, Cr, Cu, Ni and Zn speciation, matrix effect.

INTRODUCTION

This study was carried out as part of an interlaboratory comparison of methods for the sequential extraction of trace elements in sediments. The absence in the determination protocol of explicit instructions about the matrices to be used in the preparation of calibration standards led us to test the sensitivity of the method of determination to changes in the composition of these solutions.

MATERIAL AND METHODS

The material analyzed was a fine lake sediment sample, (IspraJRC)S7,. supplied by Ispra Joint Research Center. Trace elements (Cd, Cr, Cu, Ni and Zn) were sequentially extracted with:

A) Acetic acid 0.11 mol/l
B) Hydroxylammonium chloride 0.1 mol/l, pH 2 and
C) Ammonium acetate 0.1 mol/l, pH 5 following treatment with 30% Hydrogen peroxide
Further details on the extraction method will be published elsewhere[1]

The trace elements were determined by flame atomic absorption spectrometry (FAAS) Four sets of calibrant solutions were prepared using as matrices the extractants employed in each step of the sequential extraction procedure. Nitric acid 0.1 mol/l was included as a fourth matrix (D) because it had been used for the preparation of an internal reference solution containing the same elements. FAAS signals were compared with those of the 4 sets of calibrant solutions in each case. Five replicates of each determination were carried out. The number of data should be 60 for each element (3 extraction steps * 4 standard solution matrices * 5 replicates), but the contents of Cr and Ni were in many cases under the sensitivity threshold of the method. For this reason, only 20 Cr data, corresponding to extraction step 3, and 40 Ni data, corresponding to steps 1 and 3, were available. Because of these missing data, the effect of the matrix was separately studied by One Way Analysis of Variance within each extraction step. Pairwise mean comparisons were made using Tukey HSD test[2].

RESULTS AND DISCUSSION

The mean values obtained for each element in the different extraction steps are given in Table 1. The reference values (those in which the composition of extractant and standard solutions matrices coincide) are printed in bold. The means that differ significantly from the reference values are marked with asterisks according to the probabilities given by the Tukey HSD Test (*:$p<0.05$, **:$p<0.01$, ***:$p<0.001$). It can be observed that in extraction steps 1 and 2 the values obtained using ammonium acetate as calibrant matrix tend to be low as a rule (although the differences are not always significant) In two cases, values obtained with nitric acid are somewhat high, but in no case are the differences great. In step 3, on the contrary, Cd and Cr values obtained with standard solution matrices different from ammonium acetate are 2 to 3 times higher than the reference values. On the other hand, Ni values

Table 1 Means (in µg/g) of trace elements analyzed with various matrices: A, acetic acid 1 mol/l; B, Hydroxylammonium chloride 0.1 mol/l; C, ammonium acetate 1 mol/l; D, nitric acid 0.1 mol/l. Probabilities of the Tukey HSD test are: * $p<0.05$, ** $p<0.01$, *** $p<0.001$.

	Matrix	*Cd*	*Cr*	*Cu*	*Ni*	*Zn*
STEP 1	**A**	**7.2**		**6.0**	**13.7**	**272.0**
Extractant: A	B	7.3		5.3*	12.9	269.2
	C	6.9*		5.7	9.5***	243.4***
	D	7.8**		5.5	12.3	274.0
STEP 2	A	3.3		3.3**	7.7	141.2
Extractant:B	**B**	**3.4**		**2.6**	**7.4**	**139.4**
	C	2.9***		2.4		130.4
	D	3.7*		2.1	6.8	146.0
STEP 3	A	1.7***	147.0***	71.8	13.3**	104.1
Extractant: C	B	1.5***	118.0***	70.0	16.1***	103.0
	C	**0.7**	**69.7**	**72.1**	**11.0**	**90.4**
	D	1.9***	150.8***	71.5	15.4***	105.1*

are also high (up to 1.5 times higher than the reference ones) but Cu and Zn do not seem to be much affected by the composition of matrix solutions.

CONCLUSION

The errors derived from misinterpretation of the determination protocol as regards the composition of standard solutions do not seem to be a major source of variation in most cases, but the use of matrices other than ammonium acetate in standard solutions, when the extractant is ammonium acetate, may result in Cd, Cr and Ni values which are excessively high. Therefore, the use of standard solution matrices similar in composition to the solutions employed in the different extraction steps should be explicitly recommended in analysis protocols.

References

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2 J.W. Tukey, *Exploratory Data Analysis* (Adison Wesley, 1977).