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Direct determination of toxic trace metals in honey and sugars using inductively coupled plasma atomic emission spectrometry

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Abstract

A rapid method for the determination of Pb, Cd, Cu, Cr, Co, Ni, Mn and Zn in honey and sugars without prior digestion or ashing of the sample was developed, using inductively coupled plasma atomic emission spectrometry (ICP-AES). The critical instrumental parameters such as sample flow rate and radio frequency incident power were thoroughly optimized. The effect of matrix type and its concentration was also examined for glucose/fructose, sucrose and honey matrices. The sensitivity was investigated using calibration curves obtained in presence of the above matrices. The obtained recoveries for Cd, Cu, Cr, Co, Ni and Mn at the $\mu g\,l^{-1}$ level were satisfactory and practically independent of the matrix used for the calibration standards. The recoveries of Pb and Zn were less sufficient. Various commercial samples of honey, sugar, glucose and fructose were analyzed with respect to their toxic metal content. The method can be applied for routine analysis, quality and environmental pollution control purposes at the $\mu g\,l^{-1}$ level of concentration, after suitable dilution of the samples.

Keywords: Toxic metals; Honey; Sugars; Glucose; Inductively coupled plasma; Atomic emission spectrometry

1. Introduction

Honey is high-viscosity liquid foodstuff containing a range of nutritiously important complementary elements. It contains a mixture of carbohydrates, such as fructose (25–45% m/m), glucose (25–37% m/m), maltose (2–12% m/m), sucrose (0.5–3% m/m) with traces of many other sugars depending on the floral source and water (15–18% m/m) [1]. The mean content of mineral substances in honey has been calculated to be 0.17% m/m, although it varies within a wide range. Honey is the result of a bio-accumulation process useful for the collection of information related to the environment where the bees live. Since the forage area of the hive is very large (more than 7 km^2) and the bees come in contact not only with air but also with soil and water, the concentration of heavy metals in honey reflects their amount in the whole region. Therefore, honey has been recognized as a biological indicator of environmental pollution [2]. But nevertheless, determination of heavy metals in honey is of high interest mainly for quality control and nutritional aspect. High levels of metals are undesirable because of their known or supposed toxicity, so that, for instance a limit of 1 $mg\,kg^{-1}$ for lead is set in some countries [3].

Furthermore, sugars (such as glucose, fructose or sugar) are classified as generally recognized safe food ingredients. Because of their extended usage, high concern exists about the contribution of these ingredients to the total dietary intake of trace metals that may be present as contaminants. Specific attention has focused on lead because of increasing knowledge of adverse health effects from lead at various levels of exposure [4]. However, these ingredients also serve as potential sources of exposure to other metals, such as cadmium, copper and tin, which occur at various levels in the environment [5,6].

Metals determination in sugar-rich foodstuffs has been a challenging analytical task due to the interference arising from the matrix. Sample pre-treatment is usually required to destroy the organic matrix and to extract the metal ions bound in organic complexes. Obviously, the selection of the digestion procedure must take into account the analytes, the

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sample matrix and the time requirements of the analytical technique considered. Several techniques have been proposed for the determination of heavy metals in honey and other sweeteners, but in most cases the matrix mineralization is required [3,4,7]. However, in common mineralization procedures there are risks of contamination or analyte loss as a result of prolonged sample manipulation and heating.

Flame atomic absorption spectrometry (FAAS), due to its relatively low cost and quite good analytical performance has been widely used for determination a various metals in honey [8–11]. The conventional way to carry such determinations involves a mineralization stage to obtain a final solution suitable for introduction into the flame nebulizer [9]. The destruction of the organic matter eliminates both spectral interferences and the accumulation of residues in the burner head and spray chamber. However, a drawback is posed since the linear response range of the AAS is narrow. Difficulties associated to the high organic matter content are overcome by dissolving the samples in acidified water and then directly introducing the resulting solution in the nebulizer [10]. Electrothermal atomic absorption spectrometry (ETAAS) has been proposed as a more sensitive analytical technique compared to FAAS for the determination of toxic metals in honey and sugars [1,12].

Inductively coupled plasma-based techniques (ICP-AES and ICP-MS) have been applied as multi-elemental techniques for the determination of heavy metals in honey and other sweeteners [7,13–16]. ICP-AES is attractive for trace analysis, owing to the satisfactory sensitivity coupled with the advantage of simultaneous determinations of several metals at several spectral lines. However, the great disadvantage of sample dry or wet digestion still remains. Hence, the sample is usually digested by wet-acid or dry ashing by heating in a microwave oven or a furnace.

The aim of this study was to investigate the possibility of the simultaneous and direct measurement of heavy metals in honey and sugars using ICP-AES without any dry or wet sample dissolution. The effect of the matrix type and its concentration was examined using aqueous (AQ) matrix and three different carbohydrate matrices: a 1 + 1 mixture of glucose and fructose mixture (GF), sucrose (SU) and honey (HO). The sensitivity of the method with respect to each metal was evaluated using the resulted slope of the calibration curves. As there are not readily available standard reference materials [17,18], the recoveries of the analytes were measured at different concentrations using spiked solutions and used as estimation of the accuracy of the method. The precision was evaluated by measuring the repeatability of the method for all analytes and at different sugar matrixes. The capability of the method as a routine analysis method was estimated through the determination of the detection limits of every heavy metal studied. The proposed method was applied in a number of commercial samples of honey, sugar, glucose and fructose.

Table 1
Operating conditions and description of the ICP-AES instrument

rf generator	40 MHz, free-running
rf incident power	Optimized
Torch, injector, i.d.	Fassel type, Alumina, 2.0 mm
Argon flow rates	Auxiliary 0.5 $\mathrm{l\,min^{-1}}$; nebulizer 0.85 $\mathrm{l\,min^{-1}}$; plasma 15 $\mathrm{l\,min^{-1}}$
Air flow rate	18 $\mathrm{l\,min^{-1}}$
Spray chamber	Scott double-pass
Nebulizer	Gem tip cross flow
Sample propulsion	Peristaltic pump, three channel
Sample flow rate	Optimized
Polychromator/resolution	Echelle/0.006 nm at 200 nm
Detector	Segmented-array charge-coupled (SCD)

2. Experimental

2.1. Instrumentation

A Perkin Elmer Optima 3100 XL axial viewing inductively coupled plasma atomic emission spectrometer was used, according to the operating conditions given in Table 1. The analytical wavelengths for each analyte was, Pb: 220.353, 217.000, 261.418 nm; Cd: 214.440, 226.502, 228.802 nm; Cu: 324.752, 224.700, 327.393 nm; Cr: 283.563, 284.325, 267.716 nm; Co: 228.616, 238.892, 230.786 nm; Ni: 221.648, 232.003, 341.476 nm; Mn: 257.610, 259.372, 260.568 nm; Zn: 213.857, 202.548, 206.200 nm. For the optimization of the instrument's performance different radio frequency (rf) incident power levels and sample flow rates were investigated.

2.2. Reagents and solutions

All chemicals were of analytical reagent grade and were provided by Riedel de Haen. The chemical reagents used for preparation of matrix matched standards (glucose, fructose and sucrose) were of analytical grade, provided by Riedel de Haen. De-ionized water of MilliQ quality was used throughout. Mixed working standard solutions of the analytes (Pb, Cd, Cu, Cr, Co, Ni, Mg and Zn) were prepared by appropriate stepwise dilutions of stock standard solutions containing 1000 $\mathrm{mg\,l^{-1}}$ of each analyte in 0.5 $\mathrm{mol\,l^{-1}}$ HNO_3 (Riedel de Haen), according to the procedures described below.

Eight-point calibration curves were obtained using matrix matched standards prepared in aqueous (AQ), glucose/fructose in water (GF), sucrose in water (SU) and honey in water (HO) matrix. The final mixed working standard solutions had the following concentrations: 0.25, 0.50, 1.00, 2.50, 5.00, 10.0, 25.0 and 50 $\mathrm{\mu g\,l^{-1}}$ for each metal. In case of the carbohydrate matrices, a 2% m/v solution of the corresponding sugar (glucose/fructose, sucrose, or honey) was used for dilution. In order to check the overall repeatability of the ICP-AES detector two different calibration procedures were made at two different time periods. The slope of the calibration curves was used to estimate the sensitivity of the method.

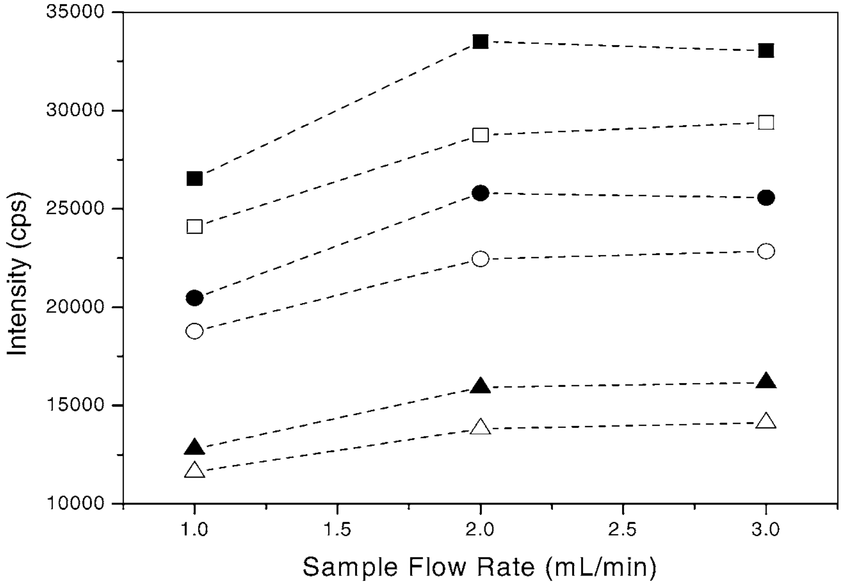

Fig. 1. Effect of the sample flow rate on the intensity of 50 μg l^{-1} Mn at GF matrix, at the following spectral lines and carbohydrate concentrations: (■) 257.610 nm–1% m/v; (□) 257.610 nm–10% m/v; (●) 259.372 nm–1% m/v; (○) 259.372 nm–10% m/v; (▲) 260.568 nm–1% m/v; (△) 260.568 nm–10% m/v.

Samples of honey, glucose and fructose are considered as not homogeneous matrices due to their very high viscosity, thus it is crucial to obtain representative sub-samples for further analysis [7,16]. In case of honey, glucose and fructose samples, the sample container was warmed to ca. 50 °C in a water-bath and sonicated for 5 min. Then an amount of 20 g of the sample was accurately weighed in a 250-ml beaker and diluted with about 80 ml of doubly de-ionized water. Finally, the mixture was transferred to a 1000 ml volumetric flask, diluted to volume with de-ionized water. The obtained solution was used as a 2% m/v honey solution. In case of sugar samples the above procedure was followed without a warming step.

3. Results and discussion

3.1. Effect of the incident rf power

In order to examine the effect of rf power on the intensity, of 50 μg l^{-1} concentration level of each analyte at two carbohydrate concentration levels (2 and 5% m/v) in GF matrix, at three different power levels (1300, 1400 and 1500 W) were tested. As it was found, by increasing the rf power the intensity is also increased, for all analytes. However, the plasma became unstable at 1400 and 1500 W, thus the rf power was adjusted to 1300 W for the rest of the experiments.

3.2. Effect of the sample flow rate

In ICP nebulizers, the sample flow rate defines the amount of analyte in the torch and affects the sensitivity of the method. The effect of sample flow rate on the intensity was studied in the range 1.0–2.0–3.0 ml min^{-1} and the Ar flow rate in the nebulizer was adjusted to 0.80–0.85–0.90 l min^{-1}, respectively. Two matrices (GF and SU), two carbohydrate concentrations 1.0 and 10% m/v) and 50 μg l^{-1} concentration of each analyte were used through this study. In Fig. 1, the effect of the sample flow rate on the intensity of Mn is demonstrated. According to the obtained results the intensity was increased by increasing the flow rate from 1 to 2 ml min^{-1} and levelled of over 2 ml min^{-1} for all the examined analytes. Also, another trend was observed: significant depressing of the intensity, by increasing the carbohydrate concentration in the matrix from 1 to 10% m/v.

Similar behaviour was found in presence of the other examined matrix type (SU, HO). Moreover, the obtained results were statistically analyzed using the paired *t*-test at a confidence level of 95%, in order to define the optimum value of sample flow rate. The results in flow rates of 1 and 2 ml min^{-1} are statistically different and in all cases the intensity at 2 ml min^{-1} was higher. However, there is not a significant statistical difference between the results obtained at flow rate of 2 and 3 ml min^{-1}. Therefore, 2 ml min^{-1} sample flow rate was selected for the rest of the experiments.

3.3. Effect of the carbohydrate matrix

Generally, the matrix type and the carbohydrate concentration affects significantly the viscosity of the sample and consequently the performance of ICP-AES methods. Therefore, the effect of the matrix type and the carbohydrate concentration on the emission intensity was investigated using the GF, SU and HO matrices at increasing concentration (1.0, 2.0, 5.0, 10 and 25% m/v) of carbohydrates. The optimum

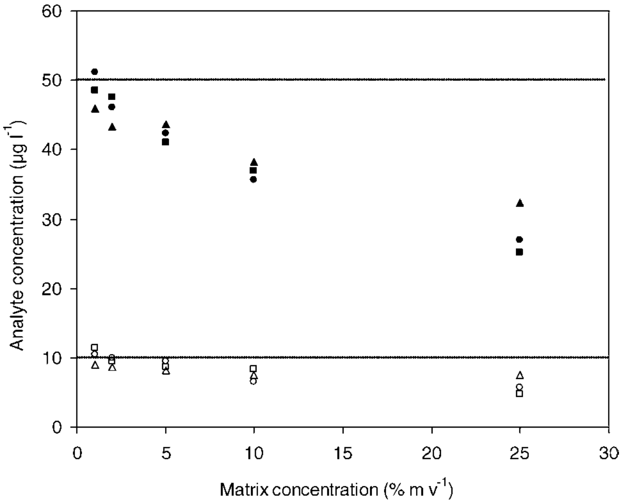

Fig. 2. Effect of matrix type and matrix concentration on the recovered analyte concentration of Co—230.786 nm. (■/□) Glucose and fructose; (●/○) sucrose; (▲/△) honey. Unfilled symbols refer to 10 µg l^{-1} analyte concentration and filled symbols to 50 µg l^{-1}. The horizontal dotted lines represent the concentration level of 10 and 50 µg l^{-1} aqueous standard solutions in absence of carbohydrate concentration.

of these concentrations defines also the dilution of the real samples, which should be used in order to be analyzed. All the studied analytes were measured at the three spectral lines and at two analyte concentrations 10 and 50 µg l^{-1}, respectively. Indicative results on the effect of the matrix type and concentration on Co at two analyte concentrations appear in Fig. 2. In all cases the recovery was almost the same for the low carbohydrate concentrations (1–2% m/v) in the solution, while at higher concentrations the recovery decreased and the baseline becomes unstable. Therefore, 2% m/v carbohydrate concentration in the solution or 2% m/v dilution of the samples was adopted for further study, as a compromise between sufficient sensitivity and low matrix influence. However, higher concentrations (up to 10% m/v) can be used for higher sensitivity but with lower precision.

Statistical paired *t*-test was used to evaluate the differences in the values obtained using different matrices. In Table 2 results on the experimentally calculated *t*-values are listed for several analytes at two analyte concentrations, where t_{crit} denotes the critical value taken from statistical tables of the student *t*-test distribution for 14 degrees of freedom at a confidence level of 95%. The statistical analysis proved that, differences obtained between the three different studied matrices (glucose/fructose, sucrose and honey) were not of statistical significance at the 95% confidence level (except for Cu at 50 µg l^{-1} concentration).

Table 2
Test of significance (*t*-test) of the difference between carbohydrate matrices at two analyte concentrations

Analyte	Analyte concentration (µg l^{-1})	Comparison between matrices			t_{crit}
		GF–SU[a]	GF–HO[a]	SU–HO[a]	
Cd	10	0.66	0.19	0.53	2.14
Cd	50	1.82	2.04	1.22	2.14
Cu	10	0.08	1.57	1.49	2.14
Cu	50	0.19	2.60	2.37	2.14
Co	10	0.27	0.63	0.94	2.14
Co	50	0.02	1.28	0.72	2.14
Mn	10	1.92	1.54	0.70	2.14
Mn	50	1.08	0.86	0.36	2.14

[a] (GF) glucose/fructose in water; (SU) sucrose in water; and (HO) honey in water.

3.4. Regression analysis

Calibration curves were obtained for all the analytes at three spectral lines for each one in the following matrices: AQ, GF, SU, HO. The carbohydrate concentration in the GF, SU and HO was 2% m/v. The sensitivity of each calibration is expressed by the slope of the linear regression equation in the range 0–50 µg l^{-1}. The slopes (*S*) together with their corresponding standard errors (SEs) and the linear correlation coefficient (*r*), for each analyte are listed in Table 3. Among the three spectral lines of each analyte, those with the better sensitivity (*S*), reproducibility (SEs) and linearity (*r*) were given, and were used for the rest the study. As it is shown, the slope values referring to different matrices were similar and the values of the standard error of the slopes were very low. This observation confirms that the results are independent of the sugar matrix. The correlation coefficients for all calibration curves were >0.990, except Pb and

Table 3
Slope ± standard error and correlation coefficients of the calibration equations for all analytes in various matrices

Analyte spectral line	Matrix			
	Aqueous	Glucose/fructose	Sucrose	Honey
Pb—220.353	6.1 ± 1.4 (0.9770)	7.3 ± 1.4 (0.9520)	5.3 ± 2.2 (0.8111)	5.0 ± 2.2 (0.7962)
Cd—228.802	61.9 ± 0.3 (0.9999)	57.1 ± 0.2 (0.9999)	57.0 ± 1.2 (0.9994)	58.0 ± 0.2 (0.9999)
Cu—324.752	469 ± 9 (0.9995)	474 ± 15 (0.9985)	437 ± 13 (0.9986)	457 ± 31 (0.9934)
Cr—283.563	309 ± 11 (0.9981)	285 ± 7 (0.9990)	264 ± 13 (0.9960)	266 ± 9 (0.9982)
Co—230.786	36.4 ± 0.3 (0.9999)	33.9 ± 0.2 (0.9999)	33.9 ± 0.2 (0.9999)	33.0 ± 0.3 (0.9999)
Ni—341.476	40.7 ± 3.2 (0.9907)	43.9 ± 1.4 (0.9984)	37.9 ± 3.5 (0.9874)	41.5 ± 2.4 (0.9952)
Mn—257.610	1135 ± 5 (0.9999)	1068 ± 3 (0.9999)	1052 ± 6 (0.9999)	1041 ± 8 (0.9999)
Zn—213.857	511 ± 136 (0.9999)	114 ± 88 (0.8161)	15 ± 20 (0.4340)	201 ± 65 (0.8701)

Zn, illustrating the good linearity in the studied range. Very high sensitivities were obtained in the determination of Mn followed by Cu, Zn, Cr, Cd, Co and Ni. The sensitivity in the determination of Pb was rather low.

3.5. *Accuracy and precision of the method*

Usually, in order to assess the accuracy of a method, standard reference materials are analyzed, the obtained results are compared with the certified ones and the absolute or the relative error is calculated. However, an SRM material with certified metals concentration is not available for honey, as already mentioned by Caroli et al. [17,18]. These authors ascribed the lack of a honey-based certified reference material due to its practical problems of transformation into a stable, homogeneous mass, adequate for a certification project. Therefore, the accuracy of the proposed method was evaluated by determining the recoveries of the analytes at various concentrations (1, 5, 25 and 50 µg l^{-1}), using calibration curves at different matrices. Results from the recovery experiments appear in Table 4. Quantitative recoveries were obtained at high metal concentrations (25 and 50 µg l^{-1}) and they were independent of the matrix used. As it is shown, the recoveries obtained for all analyte concentrations were ranged between 93 and 110%. The recoveries of Cd and Mn were very good, while those of Cu, Cr, Co and Ni were adequate at the 25 and 50 µg l^{-1} levels. On the other hand Pb and Zn recoveries were not satisfactory (<80%).

The precision of the method was evaluated using the relative standard deviation, s_{r}, of repeated determination of the analytes at 25 µg l^{-1} concentration level, in presence of 2% m/v carbohydrate in the solution. For Cd, Cu, Cr, Co, Ni, and Mn the s_{r} ranged between 0.3 and 17% as listed in Table 5. The variation of s_{r} is similar for all matrices tested. However, the repeatability of the method was not adequate in the case of Pb and Zn, and in some cases it was higher than 50%.

Table 4
Recovery of analytes at aqueous and carbohydrate matrices

Matrix	Added	Recovery (%)					
		Cd	Cu	Cr	Co	Ni	Mn
AQ	50 µg l^{-1}	94	104	88	93	91	93
GF		102	103	95	99	86	98
SU		102	112	103	99	94	100
AQ	25 µg l^{-1}	94	115	97	93	102	93
GF		102	113	106	100	103	98
SU		102	123	114	100	121	100
AQ	5 µg l^{-1}	101	200	123	96	52	96
GF		109	200	134	103	49	102
SU		110	217	144	103	54	103
AQ	1 µg l^{-1}	99	n.q.	n.q.	n.q.	n.q.	107
GF		106	n.q.	n.q.	n.q.	n.q.	113
SU		107	n.q.	n.q.	n.q.	n.q.	115

n.q.: not quantified.

Table 5
Precision of the method at various matrices (25 µg l^{-1} of the analyte, relative standard deviation s_{r} %, $n = 10$)

Analyte	Matrix			
	Aqueous	Glucose/fructose	Sucrose	Honey
Pb	6.2	19	25	>50
Cd	1.0	0.7	1.1	1.3
Cu	2.8	2.2	2.5	1.0
Cr	7.9	6.6	6.9	1.9
Co	1.0	1.7	3.2	1.6
Ni	3.7	17	5.6	15
Mn	0.3	0.6	0.5	0.6
Zn	17	>50	>50	>50

Table 6
Detection limits (µg l^{-1}) obtained for all analytes in aqueous and carbohydrate matrices

Analyte	Aqueous	Glucose/fructose	Sucrose	Honey
Pb	6.7	4.5	9.7	8.6
Cd	0.7	1.2	0.9	1.4
Cu	1.5	1.0	4.8	1.0
Cr	17	6.8	15	5.7
Co	0.4	0.8	2.0	1.8
Ni	7.3	4.3	16	3.1
Mn	0.2	0.2	1.0	0.2
Zn	48	139	309	29

The detection limit (c_{L}) was calculated according to the 3 s criterion (three times the standard deviation of 10 blank measurements). The results are listed in Table 6. The lower detection limits were obtained for Mn, Cd, Co and Cu, moderate for Cr, Ni and Pb and higher for Zn. Consequently, the detectability of the proposed method is considered satisfactory for routine analysis and quality control. Similar detection limits for Mn, Cd, Co and Cu have been also obtained using ICP-AES after a preliminary mineralization stage [15]. The ion chromatographic and the voltammetric determination of the elements Pb, Cd, Cu, Co and Ni gave much higher detection limits (40, 15, 3, 2 and 4 µg l^{-1}, respectively) [3] after laborious mineralization stage. On the other hand, lower detection limits for Pb, Cd, Cu and Zn were reported only in case of using the ETAAS technique [1].

3.6. *Analysis of commercial samples of honey and sugars*

Samples of five commercial products of honey, two of sugar, one of glucose and one of fructose were analyzed with respect to their content in heavy metals. The final concentrations of the analytes (in mg kg^{-1}) were calculated relative to the initial sample mass and corrected for any dilutions. Honey, glucose, fructose and sugar samples were pre-treated as described in Section 2. The results of mean concentrations and their standard deviation are listed in Table 7. Zn and Pb were not quantified due to their poor repeatability, as it is given in Table 5. Chromium was not

Table 7
Analytical results of commercial sample analysis (concentrations in mg kg^{-1}, mean ± standard deviation, $n = 7$)

Sample	Cd	Cu	Co	Mn	Ni
Sugar A	0.087 ± 0.013	0.16 ± 0.01	0.131 ± 0.012	n.d.	0.56 ± 0.06
Sugar B	0.084 ± 0.016	0.19 ± 0.03	0.039 ± 0.007	n.d.	0.24 ± 0.03
Glucose	0.084 ± 0.009	0.14 ± 0.02	0.036 ± 0.006	n.d.	0.09 ± 0.02
Fructose	0.088 ± 0.014	0.14 ± 0.03	0.080 ± 0.016	n.d.	0.35 ± 0.05
Honey 1	0.089 ± 0.010	0.48 ± 0.03	0.071 ± 0.010	7.22 ± 0.2	n.d.
Honey 2	0.110 ± 0.024	n.d.	0.042 ± 0.006	0.20 ± 0.03	n.d.
Honey 3	0.078 ± 0.013	n.d.	0.010 ± 0.003	0.18 ± 0.03	n.d.
Honey 4	0.097 ± 0.017	0.28 ± 0.05	0.078 ± 0.012	0.11 ± 0.02	n.d.
Honey 5	0.222 ± 0.031	0.45 ± 0.04	0.087 ± 0.015	0.12 ± 0.03	n.d.

n.d.: not detected.

detected in all samples. The concentration of Cd measured was low (0.078–0.222 mg kg^{-1}), as well as copper levels (0.14–0.48 mg kg^{-1}) and similar to those found in honey samples from various origins [13–15]. The Co concentration was very low (0.010–0.131 mg kg^{-1}) and lower than the reported values. Ni was not detected in all honey samples and its values measured in sugars were in the range 0.09–0.56 mg kg^{-1}. Finally, Mn was not detected in any of the sugars, in contrast to the honey samples where values ranged between 0.11 and 0.20 mg kg^{-1} were measured in all but the first sample, for which, the concentration of Mn was found to be very high (7.2 mg kg^{-1}). High values have been reported in the literature [13,15], which can be attributed either to the production stages of honey or to the region from where this honey has been taken.

4. Conclusions

The developed method was proved efficient and reliable for the simultaneous and direct determination of several heavy metals in routine analysis of honey and sugars using ICP-AES in the optimal instrument's parameters (2 ml min^{-1} sample flow rate and 1300 W rf power). As it was shown, the type of the carbohydrate matrices (glucose/fructose, sucrose, honey) did not have significant effect on the performance of the proposed method. Also it was shown that a 2% m/v concentration of the carbohydrate is a compromise for sufficient sensitivity and low matrix effect. Therefore, the samples were analyzed after such dilution. Furthermore, calibration curves were drawn at the aforementioned matrices, and it was proved that mixed aqueous standards can be readily employed in preparing the calibration curves. The accuracy of the method was investigated by measuring the recoveries at several metal concentrations in spiked solutions, and the precision by the relative standard deviation. Using the proposed method, the obtained recoveries were very good and the detection limits of Mn, Cd, Co, Cu and Ni were sufficient for routine analysis of commercial samples of honey, sugar, glucose and fructose.

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