

Determination of Cd, Cr, Cu, Ni, Pb and Zn in compost: evaluation of different approaches for sample preparation and instrumental analysis (MP-AES as an alternative to ICP-OES)

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In the Bulgarian legislation are stated maximum acceptable concentrations for several elements (Cd, Cr, Cu, Ni, Pb and Zn) in treated biowaste such as compost. The prescribed limits correspond to the soluble fraction of the elements extracted with nitric acid or aqua regia using procedures described in EN 16173:2012 and EN 16174:2012, respectively. In the current work it was compared the elemental transfer into solution when the two eligible reagents (nitric acid or aqua regia) were used. The acid extractions were carried out under microwave irradiation in closed vessels. The soluble fractions of the elements were measured by two alternative instrumental techniques i.e. inductively coupled plasma optical emission spectrometry (ICP-OES) and microwave plasma atomic emission spectroscopy (MP-AES). The results show that MP-AES is an adequate alternative to ICP-OES which fits the analytical purpose to monitor Cd, Cr, Cu, Ni, Pb and Zn in compost. Several samples from different manufacturers were studied and it was found that the extraction of Cd, Cr, Pb and Zn was not influenced by the applied acid treatment. On the opposite, the extraction of Cr and Ni was significantly higher when aqua regia was used in comparison to the solely application of nitric acid.

Key words: elemental analysis of compost; microwave-assisted acid extraction of elements; inductively coupled plasma optical emission spectrometry (ICP-OES), microwave plasma atomic emission spectroscopy (MP-AES).

INTRODUCTION

The intensive farming and the inadequate land management leads to a reduction of the needed for agriculture organic substances in soils [1]. Possible solution of the latter problem can be found in the application of compost as a source of organic matter. Various types of compost have been obtained from resources, such as municipal solid waste, green waste, food processing waste, animal manures, sewage sludge [2, 3].

Since the compost is a product from reutilized materials it can be a source of pollutants, in particular heavy metals, which will be deposited by its use in the environment. The contamination with toxic elements is a hazard to the human health and the animal populations because these pollutants can be taken up directly by humans and animals through inhalation of dusty soil or they may enter the food chain as a consequence of absorption by plants or infiltration to the groundwater and contamination of the drinking waters [4]. For this reason, monitoring of the toxic chemical elements in agricultural soils and soil improvers, such as compost, is needed. In Bulgaria the quality requirements for compost are

prescribed in a regulatory document called “Ordinance on treatment of biowaste” [5]. In the latter document are stated maximum acceptable levels for the soluble fractions of Cd, Cr, Cu, Ni, Pb and Zn extracted from compost with nitric acid or aqua regia following procedures described in EN 16173 [6] and EN 16174 [7], respectively. No preference is given to any of the suggested sample preparation approaches. Hence the aim of the current work was to assess if there is any significant difference in the soluble fractions of the listed elements extracted with each eligible reagent – nitric acid or aqua regia.

Inductively coupled plasma optical emission spectrometry (ICP-OES) is one of the recommended instrumental techniques for elemental detection in treated biowaste [8]. A relatively new instrumental technique which can be used for multi-elemental analysis with lower operational costs than ICP-OES is microwave plasma atomic emission spectroscopy (MP-AES) [9]. To the best of our knowledge MP-AES was not previously used for determination of Cd, Cr, Cu, Ni, Pb and Zn in compost samples and this motivated us to test the applicability of this technique for the intended purpose.

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EXPERIMENTAL

The reagents used for extraction of soluble fractions of elements from compost were hydrochloric acid ($\text{HCl} \geq 37\%$, Fluka, grade p.a.) and nitric acid ($\text{HNO}_3 \geq 65\%$, Fluka, grade p.a.). Ultrapure water with $2 \mu\text{S cm}^{-1}$ electroconductivity (Ultrapure Water System Adjarov Technology Ltd.) was used throughout this work for preparation of solutions and rinsing the vessels. Standard solutions were prepared from a multi-element standard solution (ICP Multi-element Standard Solution IV 1000 mg L^{-1} - Ag, Al, B, Ba, Bi, Ca, Cd, Co, Cr, Cu, Fe, Ga, In, K, Li, Mg, Mn, Na, Ni, Pb, Sr, Tl, Zn, Merck KGaA) by dilution with acidified ultrapure water (5% w/w HNO_3).

The analysed compost samples were produced by three different manufacturers reutilizing green waste from Bulgaria. Samples were dried in an oven at 40°C for 16 hours, after that they were quartered to provide about 200 g sub-sample which was homogenized with grinder for 2 minutes.

Acid extractions were accomplished according to EN 16173 (reagent - nitric acid) [6] and EN 16174 (reagent - aqua regia) [7]. Sample with mass 0.5 g was weighed into microwave vessels and 10 mL nitric acid or 8 mL aqua regia (6 mL hydrochloric acid and 2 mL nitric acid) were added. The following microwave program for heating was used: 20 min ramp to $175 \pm 5^\circ\text{C}$, hold on $175 \pm 5^\circ\text{C}$ for 10 min. After cooling to room temperature the digests were filtered through filter paper and diluted with ultrapure water to 50 g. Blank samples were proceeded for each extraction procedure.

A Microwave Digestion System (MARS 6, CEM Corporation) with closed vessels was used to perform the microwave-assisted extraction.

Inductively coupled plasma optical emission spectrometer ICP-OES Agilent 720 and microwave plasma atomic emission spectrometer MP-AES Agilent 4200 were used for elemental detection with operating parameters indicated on Table 1.

RESULTS AND DISCUSSION

Compost samples from three different Bulgarian manufacturers (represented in the text as *Manufacturer 1*, *Manufacturer 2* and *Manufacturer 3*) were studied in the current work. Samples with diverse origin were included in the research aiming to subject to analysis varying compost matrices with different analyte concentrations.

Inductively coupled plasma optical emission spectrometry (ICP-OES) is a well-established instrumental method for elemental analysis of sludge, treated biowaste and soils [8]. For the

reason it was used as a primary technique for detection of Cd, Cr, Cu, Ni, Pb and Zn in compost. A second instrumental method for analysis which applicability for the intended purpose has been checked was the microwave plasma atomic emission spectroscopy (MP-AES). Both element specific detection techniques were tested in view of the arising spectral and non-spectral interferences when sample solutions after microwave-assisted digestion with nitric acid or aqua regia were analysed.

When an instrumental technique based on emission spectrometry is used for elemental analysis several issues should be considered for selection of analyte lines which will be measured. In the current study the set criteria for spectra registration in priority order (for both ICP-OES and MP-AES) were as follows: i) analyte lines free from spectral interferences, ii) low background noise and iii) high sensitivity of the measured lines. The studied emission lines (nm) in compost matrices for ICP-OES were Cd II 214.439, Cd II 226.502, Cd I 228.802, Cr II 267.716, Cr II 283.563, Cr I 357.868, Cu II 224.700, Cu I 324.754, Cu I 327.395, Ni II 221.648, Ni II 230.299, Ni II 231.604, Ni I 341.476, Pb II 220.353, Pb I 283.305, Zn II 202.548, Zn II 206.200 and Zn I 213.857. The respective ones for MP-AES were Cd II 214.439, Cd II 226.502, Cd I 228.802, Cr I 357.868, Cr I 425.433, Cr I 520.844, Cu I 324.754, Cu I 327.395, Cu I 510.554, Ni I 231.096, Ni I 341.476, Pb I 283.305, Pb I 405.781, Zn II 202.548, Zn II 206.200, Zn I 213.857 and Zn I 481.053. The selected emission lines and the corresponding instrumental conditions for measurements are presented on Table 1.

For all compost samples analysed by ICP-OES, spectral interferences were registered for the lines Cd II 214.439, Cd II 226.502 and Pb I 283.305. When ICP-OES was used for analysis of the compost produced by *Manufacturer 3* spectral interference from Fe I 213.859 over Zn I 213.857 was also found due to the high concentration of iron (*ca.* 0.8%) in this sample. For this reason when MP-AES was used for measurements the most sensitive zinc line (Zn I 213.857) was also not preferred.

In order to compensate for the effect of non-spectral interferences the method of standard addition was used as calibration strategy in any combination between final solution media (nitric acid or aqua regia) and used detection technique (ICP-OES or MP AES).

The obtained methodological detection limits when using ICP-OES and MP-AES are given on Table 2.

Table 1. Operating parameters for ICP-OES and MP-AES

	Agilent 720 ICP-OES	Agilent 4200 MP-AES
Incident power, kW	1	1
Plasma gas, L min ⁻¹	15	20
Auxiliary gas, L min ⁻¹	1.5	1.5
Nebulizer Flow	200 kPa	0.55 - 1.0, L min ⁻¹ ^a
Nebulizer type	OneNeb®	OneNeb®
Sample flow rate, mL min ⁻¹	1.2	0.9
Optics viewing position	axial	axial
Integration time, s	0.05	3
Number of readings	3	3
Selected analyte lines for measurement, nm	Cd I 228.802, Cr II 267.716, Cu I 324.754, Ni I 341.476, Pb II 220.353, Zn II 202.548	Cd I 228.802, Cr I 357.868, Cu I 324.754, Ni I 341.476, Pb I 405.781, Zn I 481.053

^a varying for the different analyte lines (integrated software optimization was applied)

Table 2. Methodological detection limits obtained by ICP-OES and MP-AES for analysis of compost after digestion with different acid mixtures (dilution factor of 100 was applied)

Acid media	ICP-OES		MP-AES	
	Measured line	MLOD mg kg ⁻¹	Measured line	MLOD mg kg ⁻¹
Nitric Acid	Cd I 228.802	0.14	Cd I 228.802	0.30
	Cr II 267.716	0.20	Cr I 357.868	0.46
	Cu I 324.754	0.21	Cu I 324.754	0.21
	Ni I 341.476	0.47	Ni I 341.476	0.67
	Pb II 220.353	1.1	Pb I 405.781	2.0
Aqua Regia	Zn II 202.548	0.27	Zn I 481.053	39
	Cd I 228.802	0.14	Cd I 228.802	0.30
	Cr II 267.716	0.21	Cr I 357.868	0.44
	Cu I 324.754	0.22	Cu I 324.754	0.21
	Ni I 341.476	0.50	Ni I 341.476	0.69
	Pb II 220.353	1.1	Pb I 405.781	2.0
	Zn II 202.548	0.29	Zn I 481.053	41

It should be emphasized that independently from the applied instrumental technique the achieved detection limits for the final solutions containing nitric acid or aqua regia were comparable. A clear advantage of ICP-OES over MP-AES are the lower detection limits of the analytes which can be reached by the former method. However, looking at the maximum acceptable levels for the target elements in compost, according to the Bulgarian legislation (Figure 1), it is obvious that for all elements (except Cd) the achieved methodological detection limits by MP-AES are at least 10 times lower than the regulated levels (the lower ones corresponding to the post transitional period). The latter fact reveals the potential of MP-AES to be applied for control analysis of Cr, Cu, Ni, Pb and Zn in compost samples digested and diluted with factor of 100. In order to lower the methodological LOD of Cd when using MP-AES an analysis with

dilution factor of 50 was also carried out. This was possible due to the utilization of the OneNeb® nebulizer which allows high dissolved solids to be introduced into the spectrometer without clogging. Under these conditions the loss of sensitivity for Cd due to non-spectral interference was *ca.* 20% and no substantial detonation of the background noise was observed. As a result, the methodological LOD of Cd for MP-AES was lowered to 0.18 mg kg⁻¹ with corresponding limit of quantification 0.60 mg kg⁻¹. The last value is more than two times lower than the prescribed limit of 1.3 mg kg⁻¹Cd.

A comparison of the results between ICP-OES and MP-AES was carried out for the compost sample produced by *Manufacturer 1*. To eliminate any artifact from sample heterogeneity six replicate samples were dissolved and each solution (DF = 100) was measured by both ICP-OES and MP-AES. For each individual replicate it was calculated the difference $\langle \text{Diff.} = C_{MP-AES} - C_{ICP-OES} \rangle$ and the corresponding relative difference $\langle \text{Rel.} = (C_{MP-AES} - C_{ICP-OES}) / C_{ICP-OES} \rangle$ between the concentrations obtained by MP-AES and ICP-OES as an assessment of the compliance of the two instrumental methods. On Table 3 are presented the calculated mean difference and the related standard deviation from the analysis of the six replicate samples when the compost sample was digested with nitric acid and on Table 4 the ones corresponding to the analysis of the soluble fraction of elements in aqua regia. The concentrations of the analysed elements in the compost sample produced by *Manufacturer 1* can be seen on Figure 1.

The paired t-test was applied as formal statistical tool to detect any significant difference (p -value < 0.05) between the concentrations measured by MP-AES and ICP-OES.

Even though in many cases the paired t-test resulted in statistically significant deviation, generally the relative difference of the results was in the range from -5% to 5% which can be accepted

as good compliance between the instrumental techniques. The highest relative difference was observed for the analysis of Cd which can be explained with its low concentrations in the sample solution and inevitably deteriorated repeatability of the instrumental measurement under these conditions. An emphasis can also be put on the analysis of Cr for which the results obtained by MP-AES are averagely elevated with 6.5% in comparison to ICP-OES for solutions containing only nitric acid (Table 3). On the opposite when Cr was measured in the aqua regia soluble fraction the MP-AES results were lower than the ones registered by ICP-OES (Table 4). Summarizing the results from Tables 2, 3 and 4 it can be concluded that MP-AES can be used as an acceptable alternative to ICP-OES for analysis of Cr, Cu, Ni, Pb and Zn in compost samples. The analysis of Cd by MP-AES is problematic due to the insufficient detection power of the instrumental technique. The following analysis of compost samples produced by Manufacturer 2 and 3 was accomplished by final measurement with ICP-OES.

According to the Bulgarian legislation for each target element are prescribed two maximum acceptable concentrations in compost i.e. Cd – 1.30

and 2.00 mg kg⁻¹; Cr – 60 and 100 mg kg⁻¹; Cu – 200 and 250 mg kg⁻¹; Ni – 10 and 80 mg kg⁻¹; Pb – 130 and 180 mg kg⁻¹; Zn – 600 and 800 mg kg⁻¹[5]. The higher limit is a requirement to the compost production for the first 7 years from the launch of a new manufacturing plant (transition period) and the lower more rigorous limit corresponds to the next ongoing time of biowaste treatment. The legal limits are in regard to the soluble fraction of elements extracted from the compost samples by two eligible procedures i.e. extraction with nitric acid (EN 16173:2012) or aqua regia (EN 16174:2012). Since in the official regulatory document [5] no preference is given to any of the sample preparation approaches it is supposed that both extraction schemes should be accomplished and the obtained results in each case should be compared to the maximum acceptable concentrations.

In our study the solid compost sample was subjected to preliminary homogenization, before acid treatment, which efficiency was tested by analysis of several separately dissolved replicates. On Table 5 are presented the results obtained from the analysis of 11 replicates of compost (*Manufacturer 3*) treated with nitric acid.

Table 3. Comparison of the results obtained by ICP-OES and MP-AES for compost (*Manufacturer 1*) dissolved with nitric acid (DF = 100)

	Cd		Cr		Cu		Ni		Pb		Zn	
	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %
Mean ^c	0.01	0.7	3.8	6.5	9.2	5.9	0.5	1.6	0.4	0.3	36	5.2
SD ^d	0.16	11.5	1.5	2.6	4.1	2.6	1.2	4.3	2.9	2.2	7	0.9
p-value ^e	0.90		0.002		0.003		0.39		0.74		<0.001	

^a Difference between the concentrations obtained by MP-AES and ICP-OES;

^b Relative difference between the concentrations obtained by MP-AES and ICP-OES normalized to the result achieved by ICP-OES;

^c Mean value of the differences obtained from 6 replicate samples;

^d Standard deviation of the differences obtained from 6 replicate samples;

^e Obtained by the paired t-test.

Table 4. Comparison of the results obtained by ICP-OES and MP-AES for compost (*Manufacturer 1*) dissolved with aqua regia (DF = 100)

	Cd		Cr		Cu		Ni		Pb		Zn	
	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %	Diff. ^a , mg kg ⁻¹	Rel. ^b , %
Mean ^c	-0.11	-8.5	-3.7	-4.0	6.6	4.2	0.1	0.2	-4.3	-3.4	32	4.7
SD ^d	0.25	18.5	1.1	1.6	0.6	0.3	0.4	1.0	0.6	0.4	4	0.8
p-value ^e	0.32		<0.001		<0.001		0.54		<0.001		<0.001	

^a Difference between the concentrations obtained by MP-AES and ICP-OES;

^b Relative difference between the concentrations obtained by MP-AES and ICP-OES normalized to the result achieved by ICP-OES;

^c Mean value of the differences obtained from 6 replicate samples;

^d Standard deviation of the differences obtained from 6 replicate samples;

^e Obtained by the paired t-test

Table 5. Assessment of sample homogeneity - results for compost from *Manufacturer 3*

Element	Min	Max	Mean	SD	mg kg ⁻¹ ^a			RSD, %
					Percentiles			
					25-th	50-th	75-th	
Cd	0.53	0.83	0.71	0.08	0.69	0.72	0.75	10.8
Cr	38	45	41	2	40	42	43	4.8
Cu	98	113	106	4	105	108	108	3.9
Ni	22	25	24	1	23	24	24	4.4
Pb	101	119	109	5	106	108	111	5.1
Zn	393	438	423	17	418	432	435	3.9

^a 11 replicate samples were analyzed after extraction with nitric acid.

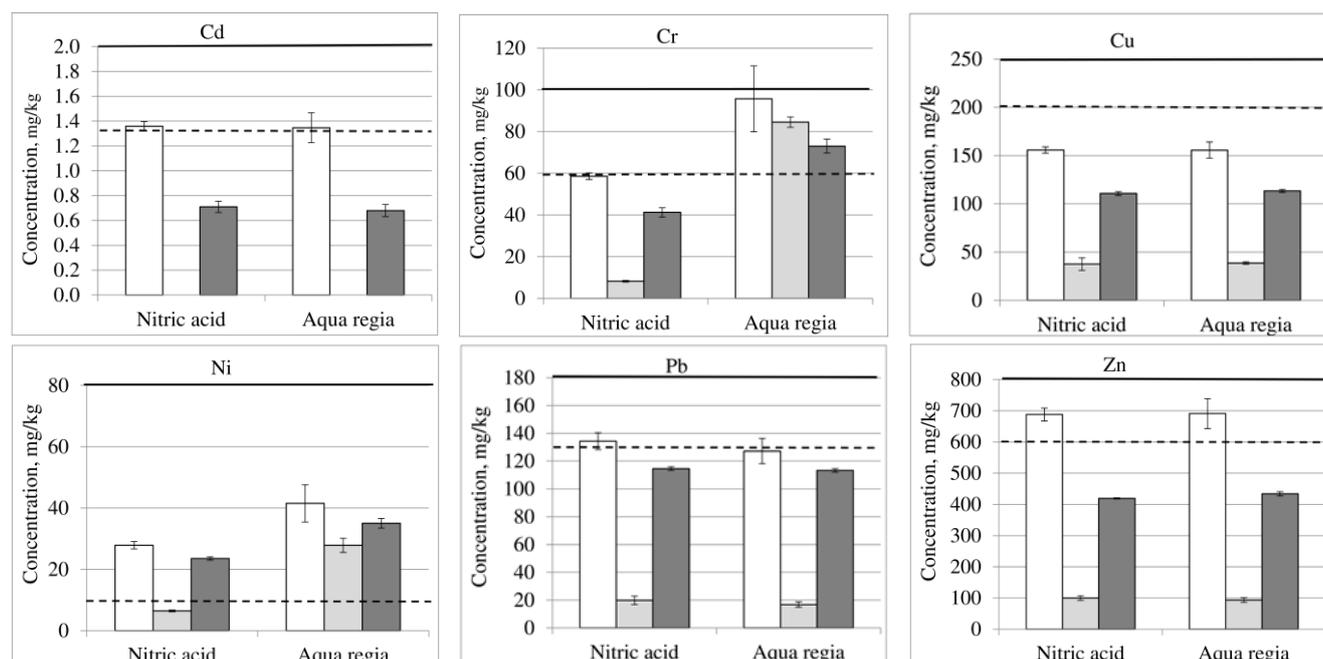


Figure 1. Comparison of the extracted amounts of elements with nitric acid and aqua regia from three different compost samples i.e. white bars - *Manufacturer 1* (6 replicates), light gray bars - *Manufacturer 2* (3 replicates) and dark gray bars - *Manufacturer 3* (3 replicates). The visualized uncertainties correspond to the standard deviation calculated from the analysis of several replicate samples. With solid line is presented the maximum acceptable concentration for the first seven years of compost manufacturing and with dashed line the one which is prescribed for the ongoing years

From the data given in Table 5 it can be derived that the used homogenization procedure is adequate. Only for Cd the relative spread of the concentrations exceeded 5%. However, since the concentration of Cd is much lower than the ones of the other elements, the higher variation range of the Cd content is most likely to be due to the deteriorated reproducibility of the instrumental measurement ($RSD\% > 8\%$) and is less probable to be a consequence of the sample heterogeneity.

Proving that the studied compost samples are homogenous is needed in order to distinguish if there is any difference in the concentration of the soluble fractions of Cd, Cr, Cu, Ni, Pb and Zn when the compost was treated alternatively with nitric acid or aqua regia. The obtained results for all tested compost samples (*Manufacturer 1*, *2* and *3*) are shown on Figure 1.

The concentration of Cd in the compost produced by *Manufacturer 2* is not presented on Figure 1 since it was below the methodological limit of quantification of ICP-OES (0.44 mg kg^{-1}).

It was found that the soluble fractions of Cd, Cu, Pb and Zn were statistically identical when nitric acid and aqua regia were used for sample treatment. This conclusion is valid for the three studied compost matrices. On the opposite for all compost samples it was found that the extracted amounts of Cr and Ni were significantly higher when aqua regia was applied for extraction compared to the treatment with nitric acid. A caution should be paid on this fact since in some samples the concentrations of Ni and Cr extracted with nitric acid are below the maximum acceptable levels but the content of these elements in the soluble

fractions in aqua regia is above the prescribed limits (Fig.1).

CONCLUSIONS

Concerning the sample preparation step for monitoring of elemental impurities in compost it can be concluded that the soluble fractions of Ni and Cr in aqua regia may be significantly higher than the ones extracted with nitric acid. No such difference was observed for Cd, Cu, Ni and Zn. The obtained results by ICP-OES and MP-AES were in good accordance and the achieved methodological limits of quantification with MP-AES were sufficiently low to allow the application of this instrumental technique to test the compliance with the maximum acceptable concentrations in compost for Cr, Cu, Ni, Pb and Zn. MP-AES can also be an adequate technique for monitoring the accordance of Cd concentration with its regulatory limit if sample dilution factor is decreased down to 50.

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MP-AES at Faculty of Chemistry, University of Plovdiv.

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ОПРЕДЕЛЯНЕ НА Cd, Cr, Cu, Ni, Pb И Zn В КОМПОСТ: ОЦЕНКА НА РАЗЛИЧНИ ПОДХОДИ ЗА ПРЕДВАРИТЕЛНА ПОДГОТОВКА НА ПРОБИТЕ И ИНСТРУМЕНТАЛЕН АНАЛИЗ (MP-AES КАТО АЛТЕРНАТИВА НА ICP-OES)

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(Резюме)

Съгласно българското законодателство са постулирани пределно допустими концентрации за набор от елементи (Cd, Cr, Cu, Ni, Pb и Zn) в третирани биоотпадъци като компост. Зададените граници са по отношение на разтворимите фракции на елементите, екстрахирани с азотна киселина или царска вода, съгласно процедури описани в БДС EN 16173:2012 и БДС EN 16174:2012. В настоящата работа бе сравнено количеството на извлечените елементи в разтвор при използване на препоръчаните реагенти (азотна киселина или царска вода). Киселинните екстракции са проведени в микровълнова система, ползваща затворени съдове за разтваряне. Разтворимите фракции на елементи са анализирани посредством две инструментални техники - оптико-емисионна спектрометрия с индуктивно свързана плазма (ICP-OES) и атомно-емисионна спектрометрия с микровълново генерирана плазма (MP-AES). Получените резултати показват, че MP-AES може да бъде приложен за мониторинг на Cd, Cr, Cu, Ni, Pb и Zn в компост и методът е адекватна алтернатива на ICP-OES. При изследването на набор от проби компост от различни производители бе установено, че екстракцията на Cd, Cr, Pb и Zn е статистически идентична за различните подходи на киселинно третиране. Заключениеето за Cr и Ni бе противоположно – екстрахираните количества от тези елементи с царска вода са значително по-големи спрямо извлечените при самостоятелната употреба на азотна киселина.

Ключови думи: елементен анализ на компост; микровълново-подпомогната киселинна екстракция на елементи; оптико-емисионна спектрометрия с индуктивно свързана плазма (ICP-OES), атомно-емисионна спектрометрия с микровълново генерирана плазма (MP-AES).