

Elemental analysis of instant soups and seasoning mixtures by ICP–OES

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Abstract

A method for the determination of Na, K, Ca, Mg, P, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn levels in high saline food products (instant soups and seasoning mixtures) using ICP–OES was developed. The results of the direct determination with multi-elemental water standards were compared with the internal standardisation, the standard addition methods and the allowable limits of the above mentioned elements in food. The method was shown to be sensitive with limits of detection: Na 1.50, K 12, Ca 0.32, Mg 0.13, P 1.1, Cd 0.16, Cr 0.21, Cu 0.32, Fe 0.30, Mn 0.17, Ni 0.42, Pb 2.1 and Zn 0.21 (in mg kg⁻¹). The method exhibited RSD 2–8%.

The proposed method was applied for the analysis of one seasoning supplement, beef and vegetable soup bases and four brands of instant soups acquired in supermarkets and suits for the determination of dietary value and nutritional assessment of food intake.

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1. Introduction

Due to the increased interest in finding the biological roles of nutrients and their function in chronic diseases, a knowledge of dietary nutrient intake is needed to optimise human health. The nutritional assessment of food intake is of permanent interest to food chemists. Dietary sources of essential elements are important for correct physiological functions of the human body. Reference daily intakes for 12 significant elements have been established: calcium (1000 mg), chloride (3400 mg), chromium (120 µg), copper (2 mg), iron (18 mg), iodine (150 µg), potassium (3500 mg), magnesium (400 mg), manganese (2 mg), molybdenum (75 µg), sodium (2400 mg), phosphorus (1000 mg), selenium (70 µg), and zinc (15 mg) (Mindel, 2000).

The frantic rhythm of modern life and the increase in the number of people that live alone have determined changes in the preparation of food and in the habits of consumption. Less time is available for a cook to make food. In this

consequence, the rapid progress of the ready-oven food technology and its products has to be mentioned. Instant soups are a wide group of dried foods which play an important role in the nutrition of people because they fulfil present and future social consumer requirements. An easy and convenient way of making a soup is to use a soup base in the form of broth cubes, granules or powder. This way, the knowledge of the mineral content of instant soups as well as another ready-oven food is important and a multi-element analysis method that is applicable to similar food products and easy to use is important from both a nutritional and a toxicological point of view.

Currently, commercial instant soups available in the market include meat, vegetables, potatoes, spices, etc. One of the most important soup components is table salt, which is used for preservation. It is usually present at percent amounts and can cause problems during analysis due to the ionisation interference of Na. Regarding common spectral and non-spectral interferences caused by the matrix, determination of trace concentrations of metals in highly saline samples using wide-spread techniques such as atomic absorption spectrometry (AAS), inductively coupled plasma optical emission or mass spectrometry

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(ICP–OES or ICP–MS) is possible only in diluted solutions (Montaser & Golightly, 1992; Welz & Sperling, 1999). The sample dilution worsens detection limits. In order to reach desired detection limits, a pre-treatment step is usually included into the analytical procedure. However, applications based on extraction and preconcentrating techniques are time and labour consuming and increase analysis cost and risk of contamination.

Ferreira et al. presented a separation and preconcentration of nickel (ng g^{-1}) from saline matrices (alkaline salts of analytical grade and table salt) before using ICP–OES (Ferreira, de Brito, Dantas, Lopo de Araújo, & Spinola Costa, 1999). Online separation and preconcentration was used before analysis of trace elements in urine and haemodialysis solution (Jiménez, Velarte, & Castillo, 2002) or sea water (Abollino, Aceto, Bruzzoniti, Mentasti, & Sarzanini, 1998; Bispo, das Graças Andrade Korn, da Boa Morte, & Teixeira, 2002; Petrov, Wibetoe, & Tsalev, 2006; Otero-Romani, Moreda-Piñeiro, Bermejo-Barrera, & Bermejo-Barrera, 2005; Batterham, Munksgaard, & Parry, 1997). Kühn et al. analysed thermal brines. The determination of Na, K, Ca, Mg, Sr, Fe, Mn, Si, B, Zn, Pb, Cd was done with ICP–OES. The minor constituents Zn, Pb, Cu, Cd, Cr, Sc, Co, Y, La, Ce, Al were enriched by trace matrix separation using the cation exchange resin Chelex100 and after an acidic elution measured using ICP–MS (Kühn, Niewöhner, Isenbeck-Schröter, & Schulz, 1998). Worden et al. investigated oilfield formation water with total dissolved salt values of up to 230 mg L^{-1} using ICP–OES and ICP–MS (Worden, Manning, & Bottrell, 2006). Vardavas et al. determined fatty acid and the salt contents of snacks in connection with child and adolescent health risk. They found a very high salt content for almost all the brands analysed from 0.8 to 3.9 g per 100 g of the product (Vardavas, Yianopoulos, Kiriakakis, Poulli, & Kafatos, 2007). Lioe et al. evaluated three types of Japanese soy sauce for the content of salt and sensory characteristics (Lioe, Wada, Aoki, & Yasuda, 2007).

ICP–OES, respectively, ICP–MS is widespread in the multi-element analysis of micro- and macronutrient and toxic elements in food (Dolan & Capar, 2002; Barnes & Debrah, 1997; Flajnik, 1995; Zhou & Liu, 1997; Ikem, Nwankwoala, Oduyungbo, Nyavor, & Egiebor, 2002; Dashti, Al-Awadi, AlKandari, Ali, & Al-Otaibi, 2004; Özcan, 2004). Kaniyas used instrumental neutron activation analysis to determine trace elements (Cs, Co, Fe, Rb, Sc, Sr, Ag and Zn) in instant and long-time soups and evaluated the content found in comparison with the daily required amount (Kaniyas, 1991).

The practical choice and usability of analytical methods for the determination of elements in soup samples depends on the concentration levels of the elements determined. ICP–OES is an efficient technique for the rapid multi-element analysis of a wide scale of various sample types. If the saline matrix is simple and known, spectral interferences are easily predicted from the spectral line library. For a higher salt concentration in the sample, the matrix

produces non-spectral interferences which in ICP–OES lead mostly to suppression of analytical signal intensity. Matrix effects are connected with significant differences in the physical properties of calibration standards and analysed samples which are related to the changes in plasma (Montaser & Golightly, 1992).

The methods commonly used for matrix effect elimination are empirical. A sample dilution is not appropriate because it decreases the ICP–OES detection ability. In the case of matrix matching, the real composition of a sample matrix is modelled in calibration standards (Pettersson, Frank, & Hoppe, 1993). The standard addition method and internal standardisation are used mostly as correction and successful techniques (Krejčová, Černohorský, & Čurdová, 2001; Hoenig, Dočekalová, & Baeten, 1998; de Boer & Veltrop, 1996). The above mentioned extraction techniques, excitation-buffering techniques or mathematical corrections can also be used (Dressler, Pozebon, & Curtius, 1998; de Boer & Veltrop, 1996; Nicolaï, Rosin, Tousset, & Nicolai, 1999), but all these techniques have limitations as discussed by Grotti, Magi, and Frache (2000).

The present work reports the result of elemental analysis of high saline food products (instant soups and spice supplements). This study has two main objectives: (i) to demonstrate the general advantages of ICP–OES in inorganic routine analysis of high saline samples; (ii) to assist oven-ready food producers in the reduction of pollution of their final products through a better quality control of raw materials, ingredients and production lines and ensure that their products meet the requirements of legislation and consumer demands of quality.

2. Materials and methods

2.1. Equipment

Measurements was carried out with a sequential, radially viewed ICP atomic emission spectrometer INTEGRA XL 2 (GBC, Dandenong Australia), equipped with a ceramic V-groove nebulizer (Glass expansion, Australia) and a glass cyclonic spray chamber (Glass expansion, Australia). The samples were decomposed in a microwave decomposition apparatus MWS-3+ (Berghof, Eningen, Germany). Demineralised water was taken from a Milli-Q Plus water-purification system (Millipore, Bedford, USA). The other equipment used included a calibrated volumetric glassware.

2.2. Reagents

The single component standards of Mg, Ca and P (each one with the content of $10\,000 \pm 30 \text{ } \mu\text{g mL}^{-1}$, CPI International, USA) and Na, K, Cr, Cu, Fe, Mn, Ni, Pb, Zn, Be, Sc and Y ($1000 \pm 3 \text{ } \mu\text{g mL}^{-1}$, CPI International, USA) were used. For the decomposition, analytical grade 65% w/v HNO_3 and 30% w/v H_2O_2 (both Lachema, Czech Republic) was used.

2.3. Samples

One commercial seasoning supplement, two brands of soup bases in the form of broth cubes (beef and vegetable) and four brands of instant soups (two kinds of chicken, goulash and tripe) were made by Czech producers. They were purchased at a supermarket and stored at $-4\text{ }^{\circ}\text{C}$ before analysis. Instant soups mutually differ only in flavour and they usually contain basic ingredients as follows: dried vegetables, meal, eggs and spices, potato starch, wheat flour, milk proteins, vegetable fat, salt, aromatic ingredients, etc. Seasoning mixtures mainly contain a mixture of table salt, spices and root vegetables. As producers declare, the NaCl content in dry matter of instant soups varies from 3% to 14% w/w and the total dried matter is about 98%. The salt content in the vegetable soup base cube is declared to be 50%. For soup bases, each cube, with a nominal weight of 11 g, was standardised for making a soup of 500 mL. The characteristics of the samples (determined dry matter, declared fat and salt) is shown in Table 1.

2.4. Standard preparation

Aqueous standards. Single and multi-element standards were prepared containing four levels of concentration: (1) single Na 500–100–50–5 mg L^{-1} , (2) multi-element: (i) K 100, Ca, Mg, P 5 and Cr, Cu, Fe, Mn, Ni, Pb, Zn 0.1 mg L^{-1} ; (ii) the same elements with half concentrations as (i), (iii) the same elements five times diluted as (i), (iv); the same elements diluted tenfold as (i). As well as a special multi element standard for the determination of limits of detection and relative standard deviation was prepared. It contained Na 250 mg L^{-1} and other elements on concentration level (iv).

According to the matrix, three sets of calibration standards were prepared. The first set of calibration standards was stabilised with 1 mL 65% w/v $\text{HNO}_3/100\text{ mL}$ of solution. The second set was prepared with 10 mL 65% w/v $\text{HNO}_3/100\text{ mL}$ of solution, which was used for the microwave decomposition in that volume. In the third set, the internal standard Be 0.1 mg L^{-1} and Y, Sc 1 mg L^{-1} and HNO_3 were used for stabilisation.

Table 1
Description of samples analysed

Sample	Dry matter (%)	Declared fat (%)	Declared salt (%)
Seasoning mixture	98.18		
Beef soup base cube	97.26	25.2	
Vegetable soup base cube	97.08		50
Instant tripe soup	95.75	34.6	12
Instant goulash soup	91.16		9
Chicken soup	91.18		14
Chicken soup with noodles	92.54	7	3.6

Table 2
The operating conditions for ICP–OES analysis

Parameter	Value	Parameter	Value
RF power	1150 W	Nebulizer gas	0.65 L min^{-1}
View height	5 mm	Sample aspiration rate	2 mL min^{-1}
Gas	Ar 99.999%	Read	On-peak, 3 s
Plasma gas	0.7 L min^{-1}	Background correction	Fixed point
Auxiliary gas	12 L min^{-1}	Number of replicates	3

Standard additions. Approximately 0.5 g of mineralised sample and (i) 25, (ii) 10 and (iii) 5 mL of the most concentrated aqueous multi-element standard was made up to volume 50 mL with water and mixed.

The water standard blank containing: (i) 1 mL, (ii) 10 mL $\text{HNO}_3/100\text{ mL}$ and (iii) 1 mL HNO_3 together with internal standards was used. All solutions were stored in polyethylene flasks.

2.5. Sample preparation

Prior to analysis, the samples were homogenised in an agate mortar. Approximately 0.5 g of the sample was accurately weighed into an acid washed TPFPA digestion tube and 5 mL of nitric acid (65% w/v) and 2 mL H_2O_2 (30% w/v) were added. The tube was heated in a microwave oven at a power setting of 50% and 150 $^{\circ}\text{C}$ for 10 min, at 70% and 220 $^{\circ}\text{C}$ for 20 min and at 10% and 100 $^{\circ}\text{C}$ for 15 min. The maximum total output of the microwave generator was 1450 W and the maximum pressure in the digestion tube was 100 bar. The digest was transferred into a 50 mL acid washed volumetric flask and an internal standard was added and the flask was filled up with demineralised water and stored in a polypropylene container. Each sample was decomposed into six replicates. Two water blanks were run with each batch of samples.

2.6. The ICP–OES method

The measurement conditions were optimised based on the signal-to-background ratio of the least sensitive element (Ni), but it was necessary to modify them in order to set the plasma stable due to a high salt concentration. The measurement conditions and emission lines are listed in Tables 2 and 3. The analyte emission was based on taking the difference of measured emission intensity on the top of the peak and background near the peak. All detection limits given by the ICP–OES software were based on three times the standard deviation of the background counts. Including the washing time between samples, the total time for analysis was approximately 5 min.

3. Results and discussion

3.1. Analytical characteristics of the process

All limits of detection given by the ICP–OES software were calculated as the concentration equivalent to three

Table 3
Analytical characteristics of ICP–OES method

	λ (nm)	LOD (mg kg ⁻¹)	RSD (%)
Na	588.995	1.50	4.6
K	769.896	12.2	7.2
Ca	422.673	0.318	5.4
Mg	285.213	0.129	6.2
P	178.284	1.05	6.3
Cd	228.802	0.156	4.9
Cr	267.716	0.214	2.1
Cu	324.754	0.316	5.0
Fe	259.940	0.301	4.5
Mn	257.610	0.173	5.4
Ni	221.647	0.421	6.7
Pb	220.353	2.13	8.2
Zn	213.856	0.214	3.1

LOD, limit of detection; RSD, relative standard deviation.

times the standard deviation of the background counts (3σ , $\mu\text{g L}^{-1}$). The procedural limits of detection (LOD, in mg g^{-1}) were worked out as $\text{LOD} = f_{\text{dilution}}^* 3\sigma$. The dilution factor f_{dilution} takes into account the dilution of the sample during the preparation step. The 3σ was determined by 10 repetitive measurements of the special multi-elemental calibration standard in the presence of Na. The concentration of Na was based on the highest content of Na in the samples analysed. In the case of Mg and Ca, it was necessary to reduce the photo-multiplier voltage to let down their signal intensities and the real ICP–OES detection ability for Mg and Ca is far better. These are summarised in Table 3.

The Czech Ministry of Health Service promulgation (Czech Collection of Laws 298/1997, pages 5553–5570) enjoins the maximal allowable content of elements in food-stuff. In Fig. 1, the comparison of procedural LODs and allowable limits for Cr, Cd, Cu, Fe, Ni, Pb and Zn in food categories “A” (ready-oven food belongs in that category) and “B” (spices) is displayed. Excepting Cd and Cr in food “A”, the procedural LODs are lower than allowable limits for other elements. In the case of Cd and Cr in food “A”, it is possible to bring down the total volume of mineralised

sample and dilution factor twice and also lower their limits of detection. For Pb, the allowable limit for food “A” is not presented in the above mentioned promulgation. The method also suits for the determination of Na, K, Ca, Mg, P and Mn for dietary value and nutritional assessment of food intakes.

3.2. Evaluation of matrix effect

Before analysis of samples, standard additions were carried out in order to find out real concentrations of elements analysed and evaluate a matrix effect of table salt. Excepting Cd, Cr, Cu, Ni and Pd, other elements were found and their amounts were counted up to spiked amounts. Without any matrix effect correction, the direct analysis of Na, K, Ca, Mg, P, Cd, Cr, Cu, Fe, Mn, Pb, and Zn was carried out for samples with standard additions. In addition, internal standards were used: Be 313.107 nm, Y 322.789 nm, Sc 356.770 nm.

From the results given in Fig. 2, it is clear that the determination using only water standard does not provide good results because the recoveries were 82–95% depending on the element analysed and matrices. The same addition of HNO_3 to calibration standards as presented in the solutions of mineralised samples improved the recoveries (87–96%). The internal standard Be gave a satisfactory recovery from 95% to 102%. For Sc, the recoveries varied from 93% to 103% and for Y from 92% to 107%. Based on the comparison of results of the internal standards, Be was recommended and used for matrix effect correction.

3.3. Analysis of samples

Using the standard addition method and internal standardisation with Be, the samples of seasoning supplement, soup base cubes and instant soups were analysed for Na, K, Ca, Mg, P, Cd, Cr, Cu, Fe, Mn, Ni, Pb, and Zn. The above mentioned procedures were also compared with

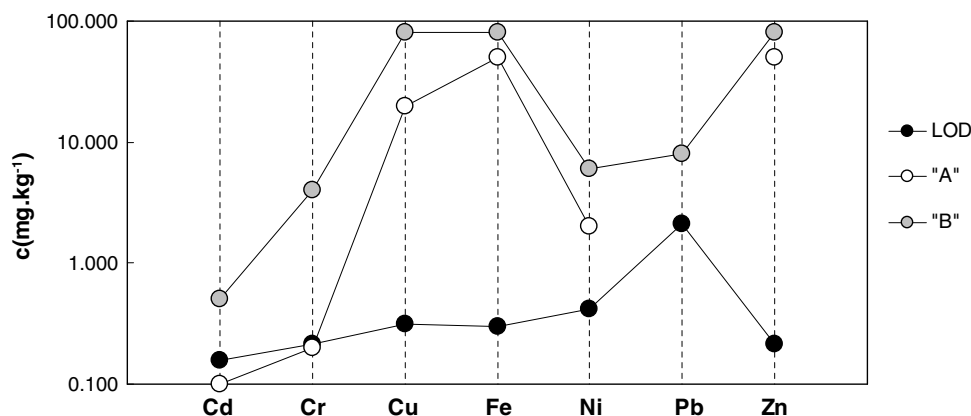


Fig. 1. Comparison of the procedural limits of detection with allowable limits for Cr, Cd, Cu, Fe, Ni, Pb and Zn in food categories: “A” (ready-oven food) and “B” (spices).

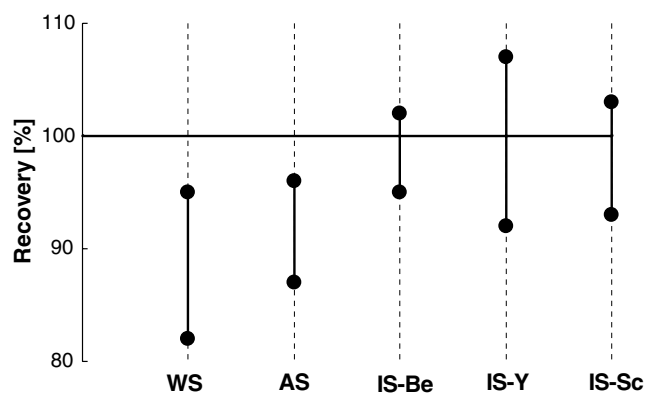


Fig. 2. Analysis of spiked soup base samples. Recoveries for the determination without matrix effect correction and for the internal standardisation with Be, Y and Sc are compared. WS, calibration standards only stabilised with HNO_3 ; AS, calibration standards in 10% HNO_3 ; IS-Be, internal standard Be; IS-Y, internal standard Y; IS-Sc, internal standard Sc.

direct determination without correction of matrix effect. All results (mean \pm SD, six replicates) are summarised in Table 4. In the case of Cd, Cr, Cu and Pb, no detectable amount was found. All results obtained were subjected to a one-way analysis of variance (ANOVA) to ascertain (at 95% confidence level) the homogeneity across the analytical procedures. The ANOVA results proved that there was not a significant variation between the single analytical procedures employed.

4. Conclusion

A simple internal standardisation technique has been utilised for multi-element analysis of Na, K, Ca, Mg, P, Cd, Cr, Cu, Fe, Mn, Ni, Pb and Zn in high saline food products – one seasoning supplement, beef and vegetable soup bases in the form of broth cubes and the four brands of two kinds of chicken, goulash and tripe instant soups using ICP–OES. In comparison with the more time consuming standard addition method, the results obtained for the internal standard Be are compatible. As expected, the results obtained for the ICP–OES analysis without any correction of the saline matrix effect are significantly lower (tens of percent) and unusable for routine analysis. In all cases of analysed samples, Cd, Cr, Cu and Pb were not detected. The method is also suited for the determination of Na, K, Ca, Mg, P and Mn for dietary value and nutritional assessment of food intakes. Ni was found only in one sample of the seasoning mixture (1.5 mg kg^{-1}). The content of Na varied from 50 to 270, K 1920 to 6200, Mg 80 to 630, Ca 170 to 700, P 300 to 2000, Fe 19 to 46, Mn 1.3 to 24, and Zn 8 to 18, all in mg kg^{-1} . The ANOVA procedure proved that there was no statistically significant difference between both analytical procedures employed.

The proposed analytical method offers an interesting perspective for elemental analysis of impurities in similar highly saline matrices. It enables the avoidance of time and labour consuming separation pre-treatment step, decreases analysis cost and risk of contamination and sim-

Table 4
Results of ICP–OES analysis of a seasoning mixture, soup base cubes and instant soups

Sample	Method	Na	K	Mg	Ca	P	Fe	Mn	Zn	Ni
		<i>c</i> (mg kg^{-1})								
Seasoning mixture	WS	252 ± 12	1820 ± 210	111 ± 5.6	299 ± 11	281 ± 13	37.9 ± 3.1	2.05 ± 0.11	12.0 ± 0.47	1.45 ± 0.10
	SA	264 ± 12	1920 ± 72	117 ± 3.1	317 ± 15	298 ± 22	46.2 ± 2.9	2.28 ± 0.19	13.5 ± 0.50	1.52 ± 0.13
	IS-Be	252 ± 19	1830 ± 81	115 ± 4.7	321 ± 16	291 ± 22	45.8 ± 2.7	2.32 ± 0.20	13.0 ± 0.34	1.45 ± 0.13
Beef soup base cube	WS	172 ± 10	2420 ± 160	74.0 ± 2.9	150 ± 12	1030 ± 57	19.7 ± 1.4	1.21 ± 0.11	8.21 ± 0.38	<LOD
	SA	189 ± 9.4	2720 ± 270	79.5 ± 2.1	172 ± 10	1110 ± 73	22.2 ± 1.6	1.35 ± 0.075	8.81 ± 0.54	
	IS-Be	187 ± 12	2740 ± 140	78.0 ± 5.6	166 ± 9.8	1130 ± 69	21.2 ± 1.1	1.33 ± 0.074	8.48 ± 0.23	
Vegetable soup base cube	WS	163 ± 10	3130 ± 110	169 ± 8.3	568 ± 23	536 ± 16	18.3 ± 0.37	3.43 ± 0.24	65.6 ± 0.23	<LOD
	SA	173 ± 10	3660 ± 130	192 ± 4.4	699 ± 33	644 ± 19	20.4 ± 0.41	3.59 ± 0.26	7.74 ± 0.28	
	IS-Be	176 ± 5.3	3800 ± 240	188 ± 3.8	702 ± 23	656 ± 23	20.5 ± 0.75	3.49 ± 0.25	7.67 ± 0.37	
Instant tripe soup	WS	46.4 ± 3.2	2190 ± 80	280 ± 7.8	491 ± 19	830 ± 58	18.9 ± 1.1	3.78 ± 0.26	16.8 ± 1.1	<LOD
	SA	51.8 ± 2.4	2550 ± 300	306 ± 17	523 ± 20	1000 ± 47	20.9 ± 1.7	4.35 ± 0.23	18.0 ± 0.71	
	IS-Be	50.4 ± 2.7	2610 ± 120	297 ± 14	515 ± 19	1010 ± 40	19.9 ± 1.3	4.20 ± 0.22	17.8 ± 1.0	
Instant goulash soup	WS	52.2 ± 2.7	5860 ± 580	541 ± 15	639 ± 28	1770 ± 120	29.8 ± 2.1	10.1 ± 0.56	15.4 ± 0.75	<LOD
	SA	62.6 ± 3.7	6160 ± 360	603 ± 24	682 ± 25	1990 ± 110	33.6 ± 2.3	11.4 ± 0.72	17.1 ± 0.79	
	IS-Be	63.2 ± 3.5	6170 ± 360	579 ± 30	695 ± 36	1901 ± 88	32.2 ± 1.8	11.1 ± 0.92	16.3 ± 0.63	
Instant chicken soup	WS	66.3 ± 5.5	2970 ± 120	602 ± 19	877 ± 45	1594 ± 53	20.0 ± 1.2	7.95 ± 0.41	14.9 ± 0.45	<LOD
	SA	78.4 ± 5.4	3390 ± 120	632 ± 18	924 ± 52	1861 ± 130	23.9 ± 1.4	9.24 ± 0.63	18.0 ± 1.3	
	IS-Be	78.9 ± 5.5	3290 ± 230	639 ± 16	937 ± 53	1854 ± 140	15.6 ± 0.85	9.01 ± 0.62	17.9 ± 0.50	
Instant chicken soup with noodles	WS	65.4 ± 3.0	4020 ± 150	298 ± 7.9	349 ± 17	1216 ± 61	18.2 ± 0.74	3.97 ± 0.27	11.9 ± 0.90	<LOD
	SA	68.6 ± 5.7	4730 ± 190	338 ± 28	407 ± 21	1442 ± 48	19.4 ± 1.2	4.63 ± 0.24	12.5 ± 0.38	
	IS-Be	67.5 ± 2.2	4850 ± 400	327 ± 20	388 ± 20	1429 ± 44	19.0 ± 0.76	4.73 ± 0.24	12.3 ± 1.9	

WS, calibration standards only with stabilising HNO_3 ; IS-Be, the internal standard Be 313.107 nm; SA, the standard addition method.

plifies laboratory work. It can assist ready-oven food producers in the reduction of pollution of their final products through a better quality control of raw materials, ingredients, and production lines, and ensure that their products meet the requirements of legislation and consumers' demands for quality.

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