Analytical Methods

Inductively coupled plasma orthogonal acceleration time-of-flight mass spectrometry (ICP-oa-TOF-MS) analysis of heavy metal content in *Indocalamus tesselatus* samples

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ABSTRACT

Indocalamus tesselatus is one of the most popular packing materials in China. Heavy metals in Chinese *I. tesselatus* samples have received great interest because they are related to health. A simple and fast method for the determination of Pb, Cd, Mn, Ni, Cr, As, Hg, Cu and Zn, by inductively coupled plasma orthogonal acceleration time-of-flight mass spectrometry (ICP-oa-TOF-MS), following microwave closed vessel digestion of samples, was proposed. The method was validated using standard reference material (GBW 07605-Tea). Samples of *I. tesselatus* from five different regions of China were analysed using the proposed method. Heavy metals contents from different regions were found at different levels. Their low contents of heavy metals showed that collection areas were not polluted and all collected *I. tesselatus* samples could be unreservedly used as food packing materials without any health risk.

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1. Introduction

Indocalamus tesselatus (family Graminales) is grown predominately in South China, including Hubei, Jiangxi, Guangdong, Guangxi and Yunnan province. It is often uncultivated in mountain slope, shady slope and semi-shady slope of cleugh at elevations no more than 1000 above sea level. In China, it is often used as packing materials. It is wrapped with glutinous rice, green bean and pork, named 'Zongzi' or 'traditional Chinese rice-pudding'. As the Dragon Boat Festival comes, Chinese will make traditional Chinese rice-pudding to commemorate ancestors. Chinese rice-pudding is also exported to America, Canada, Australia, Japan and Korea.

I. tesselatus is rich with polysaccharides and flavonoid. It is known that polysaccharides have high biological activity. Polysaccharides from *I. tesselatus* were extracted and its activity was studied. The results showed that some of them had high anticancer, antitumor and antioxidative effects (Ding, Li, Chen, Elmahadi, & Xu, 1998). *I. tesselatus* also has high antimicrobial activity, but its antimicrobial mechanism is still not clear. Just because of its antioxidative and antimicrobial activity, *I. tesselatus* can successfully prevent the stuffing deterioration for up to 7 days at room temperature. There is general understanding that packing materials should form an effective barrier between outer environment and

the food, and the concentration of all elements in packing materials should be below the safe level for human consumption (Chen et al., 1999; Gomez, Cerutti, Sombra, Silva, & Martinez, 2007; Zhang & Li, 2010). Using *I. tesselatus* as packing materials should ensure that it will not contaminate the food.

Reliable control of a range of packaging materials required analytical methods allowing the determination of traces metals in a large variety of matrices with satisfactory sensitivity and selectivity (Batista, Rodrigues, de Souza, Oliveira Souza, & Barbosa, 2011; Capar, Mindak, & Cheng, 2007; Kim et al., 2008; Llorent-Martínez, Ortega-Barrales, Fernández-de Córdova, Domínguez-Vidal, & Ruiz-Medina, 2011; Skrzydlewska, Balcerzak, & Vanhaecke, 2003). The use of both microwave digestion in closed-vessel for sample preparation and the inductively coupled plasma mass spectrometry (ICP-MS) for detection permitted an accurate determination of multi-element at trace and ultra-trace level in food samples (Millour et al., 2011; Sanchez Lopez, Gil Garcia, Sanchez Morito, & Martinez Vidal, 2003). However, some limitations occurred, including in elemental coverage when detecting rapid transient signals, and distinguishing very small differences between isotope ratios, etc.

The introduction of ICP time-of-flight mass spectrometers to the analytical practice has brought many practical advantages, e. g. enhanced sample throughput and elemental coverage, enhanced resolution, unlimited use of internal standards without performance compromises, improved isotope ratio precision capability owing to the high correlation of the noise sources for all isotopes which occur with simultaneous sampling and measurement of transient

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signals (Dick et al., 2008; Husáková, Urbanová, Šrámková et al., 2011).

Since only a small number of laboratories operated with ICP-oa-TOF-MS system, the practical experience including the analysis of food packing materials was still missing. The present study relates to analysis the presence of heavy metals (Pb, Cd, Mn, Ni, Cr, As, Hg, Cu and Zn) in *I. tesselatus* from five different regions of China, by inductively coupled plasma orthogonal acceleration time-of-flight mass spectrometry (ICP-oa-TOF-MS), following microwave closed vessel digestion of samples.

2. Experimental

2.1. Reagents and chemicals

All solutions were prepared with analytical reagent grade chemicals. The water used in all studies was ultrapure water obtained from a Barnstead Easy pure RF compact ultrapure water system. Nitric acid was obtained from Beijing Chemical Works (Beijing, China). All calibration standard solutions were prepared by appropriate dilutions of 1000 µg/L stock solutions (National analysis centre for iron and steel (Beijing, China)) immediately before use.

2.2. Indocalamus tesselatus samples

5 *I. tesselatus* samples were collected randomly from Guangdong (sample region 1), Hubei (sample region 2), Jiangxi (sample region 3), Guangxi (sample region 4) and Yunnan province (sample region 5), China. Samples were dried under vacuum and grinded. The best amount of sample was fixed in 0.2 g to fix the minimum amount of acids required for treatment of that sample mass and to decide the final volume of sample solutions.

2.3. Sample preparation

Samples were digested using a MARS5 microwave digestion system (CEM, American), equipped with HP-1500 PTFE (polytetrafluoroethylene) vessels. Sample digestion procedure was applied according to the method of Millour et al. (2011) with slight modifications. Before use, PTFE vessels were decontaminated in a bath of 10% of nitric acid (65%, v/v), then rinsed with ultra pure water and dried in a 40 °C oven.

0.2 g of *I. tesselatus* sample was weighed precisely in a PTFE digestion vessel and wet-oxidised with 8 mL nitric acid (65%, v/v) and 2 mL hydrogen peroxide (30%, w/w) in the microwave digestion system. The vessels were closed and samples were heated at the same time in the microwave oven. 1200 W maximum power was used for the digestion of samples and reference materials. The operating program for the sample microwave digestion system was carried out according to these conditions: 1200 W (13 min of ramp), 1200 W for 20 min and 20 min for cooling to room temperature. One randomly selected vessel was filled with reagents only and taken through the entire procedure as a blank. After cooling to room temperature, digests were transferred into vessels and Rh was added as internal standard at a 2 µg/L concentration. Then, the digested samples were diluted with ultra pure water to 50 mL in vessels for further analysis by ICP-oa-TOF-MS.

2.4. Instrumentation and analysis

The Optimass 9500 ICP-oa-TOF-MS instrument (GBC Scientific Equipment Pty. Ltd., Australia) was used throughout this study. The equipment was fitted with a nebuliser and a spray chamber for conventional nebulisation (Husáková, Urbanová, Audrlická-

Vavrušová et al., 2011). After preliminary experiments, the final instrumental condition selected for ICP-oa-TOF-MS operation was given in Table 1. The operational performance of the instrument was optimised daily with a multi-elemental solution.

2.5. Calibration

A multi-element standard working solution with the elements to be analysed at a concentration of 1000 µg/L was prepared by stock solutions. All solutions were 1% (v/v) in nitric acid. For calibration, multi-element solutions were prepared with the following concentrations: 0, 0.5, 4, 6 and 20 µg/L of Cd, Ni, As and Hg multi-element solution; 0, 5, 40, 60, 200 µg/L of Cu, Zn and Cr multi-element solution; 0, 25, 200, 300, 1000 µg/L of Mn solution and 0, 1, 8, 12, 40 µg/L of Pb solution.

2.6. Statistical analysis

The data was analysed using one-way analysis of variance (ANOVA) to examine statistical significance of differences in the mean concentration of heavy metals in samples. Results were considered significant at $p < 0.05$. Statistical analyses were conducted using SPSS version 15 statistical analysis packages.

3. Results and discussion

3.1. Operational condition for microwave digestion

The main object of microwave digestion was to accomplish complete dissolution of samples in the shortest time and with the minimum amounts of acids. Nitric acid is a strong oxidising agent and is widely used for liberating trace elements from biological and botanical matrices because it behaves ideally under microwave conditions (Wang et al., 2006). Since it is known that hydrogen peroxide has clarifying effect, the mixture of nitric acid and hydrogen peroxide had already been tested as digestion re-

Table 1
Operating condition for ICP-oa-TOF-MS.

Sample introduction and plasma	
ICP-oa-TOF-MS	Optimass 9500
Generator set power	1200 W
Plasma gas flow rate	10.0 L min^{-1}
Auxiliary gas flow rate	0.50 L min^{-1}
Nebulizer gas flow rate	0.63 L min^{-1}
Mass spectrometer	
Ion optics	
Skimmer	−1450 V
Extraction	−1400 V
Z1	−1000 V
Y mean	−200 V
Y deflection	0 V
Z lens mean	−1200 V
Z lens deflection	0 V
Lens body	−160 V
Pulse shaping	
Fill	−35.00 V
Fill bias	−0.75 V
Fill grid	−22 V
Push-out grid	−400 V
Push-out plate	520 V
Blanker	200 V
Reflectron	550 V
Detection	
Multiplier gain	3300 V
Ion threshold	8.4 mV
Integration window	Auto
Measurement mode	Pulse counting/analogue

Table 2
Equations and correlation coefficient for calibration curves.

Element	Equations	Correlation coefficient
Pb	$Y = 7.685 \times 10^2 x + 4.141 \times 10^3$	0.9998
Cd	$Y = 2.421 \times 10^2 x + 4.093 \times 10^3$	0.9996
Mn	$Y = 5.682 \times 10^2 x + 8.626 \times 10^3$	0.9994
Ni	$Y = 5.348 \times 10^2 x + 7.709 \times 10^3$	0.9993
Cr	$Y = 2.672 \times 10^2 x + 1.596 \times 10^4$	0.9999
As	$Y = 6.826 \times 10 x + 1.146 \times 10^3$	0.9956
Hg	$Y = 4.132 \times 10^2 x + 3.691 \times 10^3$	0.9997
Cu	$Y = 4.724 \times 10^2 x + 3.781 \times 10^3$	0.9994
Zn	$Y = 2.192 \times 10^2 x + 4.221 \times 10^3$	0.9992

Table 3
The results for reference standard material ($N = 3$).

Element	GBW 07605-Tea (µg/g) Certified[a]	Found[a]	Recovery (%)	U_Δ	Δ_m	p
Pb	4.4 ± 0.3	4.6 ± 0.3	104.5	0.458	0.2	>0.05
Cd	0.057 ± 0.010	0.057 ± 0.06	100.0	0.071	0	>0.05
Mn	1240 ± 70	1180 ± 17	95.2	72.70	60	>0.05
Ni	4.6 ± 0.5	4.8 ± 0.4	104.3	0.681	0.2	>0.05
Cr	0.80 ± 0.03	0.79 ± 0.02	98.7	0.038	0.01	>0.05
As	0.28 ± 0.04	0.27 ± 0.05	96.4	0.070	0.01	>0.05
Hg	(0.013)	0.018	–	–	–	–
Cu	17.3 ± 1.8	16.8 ± 0.6	97.1	1.928	0.5	>0.05
Zn	26.3 ± 2.0	26.5 ± 1.3	100.8	2.501	0.2	>0.05

[a] Mean ± standard deviation.

agents by other authors (Sanz-Segundo, HernaÂndez-Artiga, Hidalgo-Hidalgo de Cisneros, Bellido-Milla, & Naranjo-Rodriguez, 1999). Employing 8 mL nitric acid (65%, v/v) and 2 mL hydrogen peroxide (30%, w/w) led to a total and simultaneous dissolution of samples in a short time. If a small amount of hydrogen peroxide was used, it would allow greater sample masses to be digested adequately. However, too much hydrogen peroxide used would slow down the digestion speed and a charred residue was obtained (Eilola & Perämäki, 2001).

3.2. Selection of isotopes

The development of a method for the determination of heavy metals (Pb, Cd, Mn, Ni, Cr, As, Hg, Cu and Zn) entailed careful selection of isotopes for monitoring their concentrations in samples. When the element of interest had two or more isotope masses, at least two isotopes per element were monitored (Ataro, McCrindle, Botha, McCrindle, & Ndibewu, 2008). Signal intensities were corrected for the detector dead time and the isobaric interference before calculating the isotope ratios of element (Lam, Chan, Yip, Tong, & Sin, 2010). The isotopes ^{206}Pb, ^{207}Pb, ^{208}Pb, ^{110}Cd, ^{111}Cd, ^{112}Cd, ^{113}Cd, ^{114}Cd, ^{55}Mn, ^{58}Ni, ^{60}Ni, ^{52}Cr, ^{75}As, ^{199}Hg, ^{200}Hg, ^{202}Hg, ^{65}Cu, ^{63}Cu, ^{64}Zn, ^{66}Zn, ^{68}Zn were selected as analytical mass in ICP-oa-TOF-MS standard mode. For six elements (Pb, Cd, Ni, Hg, Cu and Zn), several specific isotopes were considered. Interference correction formulas were included in the software of the ICP-oa-TOF-MS instrument (Larcher, Nicolini, & Pangrazzi, 2003).

3.3. Calibration, validation studies and detection limit

Calibration graphs were obtained by analysis of multi-element standard working solution. Equations and correlation coefficient for calibration curves were given in Table 2.

The method was validated using standard reference material (GBW 07605-Tea). The measured values were compared with the certified values following a procedure described by Linsinger (2005). To evaluate method performance, the value of Δ_m was compared with the value of U_Δ. If the value of U_Δ was larger than the value of Δ_m, the measured value was not significantly different ($p > 0.05$) from the certified value.

The value of Δ_m was calculated by the following formula:

$$\Delta_m = |C_m - C_{CRM}|$$

where Δ_m –absolute difference between mean measured value and certified value;

C_m –mean measured value;

C_{CRM} –certified value.

The value of U_Δ was calculated as follow:

$$U_\Delta = 2\sqrt{u_m^2 + u_{CRM}^2}$$

where U_Δ –expanded uncertainty of difference between result and certified value;

u_m –Uncertainty of the measurement result;

u_{CRM} –Uncertainty of the certified value.

As can be seen in Table 3, the measured values of all heavy metals were not significantly different ($p > 0.05$) from the certified values and they were in good agreement with the certified values. This indicated that validity of the proposed method for analysis of *I. tesselatus* samples.

The ICP-oa-TOF-MS proposed method detection limit, was evaluated as three times the standard deviation (SD) of the average of the procedure blank solution (10 replicates) and slope of the standard calibration solution: DL = 3SD/slope. Detection limit of heavy metals was 0.011 µg/g of Pb, 0.047 µg/g of Cd, 0.016 µg/g of Mn, 0.019 µg/g of Ni, 0.036 µg/g of Cr, 0.139 µg/g of As, 0.017 µg/g of Hg, 0.013 µg/g of Cu, and 0.034 µg/g of Zn.

3.4. Determination of heavy metals in samples

Heavy metal contents in *I. tesselatus* samples were measured by ICP-oa-TOF-MS following microwave digestion. Five different *I. tesselatus* samples were collected from different regions of China. The results of the elemental analysis were summarised in Table 4.

Table 4
Heavy metals (µg/g) determined by ICP-oa-TOF-MS in *Indocalamus tesselatus* samples from five different regions ($n = 3$).

Element	Region 1	Region 2	Region 3	Region 4	Region 5
Pb	$2.66 \pm 0.15c$	$0.810 \pm 0.09a$	$1.19 \pm 0.14b$	$4.49 \pm 0.32d$	$2.92 \pm 0.10c$
Cd	$0.146 \pm 0.014a$	$0.244 \pm 0.020c$	$0.128 \pm 0.047a$	$0.205 \pm 0.027b$	$0.268 \pm 0.012c$
Mn	$43.4 \pm 1.3c$	$8.44 \pm 0.36a$	$93.0 \pm 6.1d$	$20.8 \pm 2.7b$	$305 \pm 8.8e$
Ni	$4.10 \pm 0.13b$	$4.69 \pm 0.28c$	$2.28 \pm 0.34a$	$4.24 \pm 0.33b$	$5.77 \pm 0.40d$
Cr	$0.081 \pm 0.018a$	$0.162 \pm 0.035b$	$0.172 \pm 0.021b$	$0.549 \pm 0.029c$	$0.496 \pm 0.041c$
As	$0.945 \pm 0.029a$	$1.31 \pm 0.17b$	$0.966 \pm 0.008a$	$1.31 \pm 0.11b$	$1.84 \pm 0.16c$
Hg	$0.093 \pm 0.002b$	$0.140 \pm 0.009c$	$0.025 \pm 0.001a$	$0.030 \pm 0.001a$	$0.031 \pm 0.002a$
Cu	$6.96 \pm 0.60a$	$549 \pm 35b$	$6.83 \pm 0.51a$	$7.43 \pm 0.38a$	$3.20 \pm 0.24a$
Zn	$10.7 \pm 1.3a$	$14.2 \pm 1.0b$	$15.1 \pm 0.9b$	$15.8 \pm 1.4b$	$15.5 \pm 0.8b$

a–e Mean values within the same row with different letters were significantly different ($p < 0.05$).

Heavy metal contents from different region were found at different levels. Pb, Cd, Mn, Ni, Cr, As, Hg, Cu and Zn were detectable in all samples, and their contents ranged from 0.810 to 4.49, 0.128 to 0.268, 8.44 to 305, 2.28 to 5.77, 0.081 to 0.549, 0.945 to 1.84, 0.025 to 0.140, 3.20 to 549, 10.7 to 15.8 µg/g, respectively. Some factors, including the environmental features of each region (water, temperature, pH and salinity), may affect metal accumulation (Rodenas de la Rocha, Sanchez-Muniz, Gomez-Juaristi, & Larrea Marin, 2009).

At present, there are still no maximum levels for heavy metals in *I. tesselatus*. Because *I. tesselatus* were used as food packing material, it would be cooked in water for several hours after packed. So, *I. tesselatus* could be applied to standard for tea established by Chinese national standard. Heavy metals values in the samples did not exceeded the limit values in tea. For safety of packing materials for food, it would be advisable to establish maximum residue levels for heavy metals in *I. tesselatus* and other plant materials.

Cu content in samples collected from region 2 presented the highest values ($p < 0.001$) at least 73 times higher than samples from region 1, 3, 4 and 5. This was possibly because that *I. tesselatus* sample was processed by immersing into copper sulphate solution, to make it returning green. Although Cu element is no longer contamination element of food by Chinese national standard in 2010, massive addition of Cu is still harmful to health.

The results (Table 4) indicated that majority values for heavy metals between different regions were significant differences ($p < 0.05$). This may be because that *I. tesselatus* samples from different regions have different chemical compositions. Majority of samples were collected from mountain slope, shady slope and semi-shady slope of cleugh, and were not processed before used for packing materials. Samples were uncultivated in rural area, heavy metal accumulation was little.

4. Conclusion

The proposed ICP-oa-TOF-MS method allows the fast determination of the content of Pb, Cd, Mn, Ni, Cr, As, Hg, Cu and Zn in Chinese *I. tesselatus* samples, following microwave digestion. The method was validated using standard reference material (GBW 07605-Tea). The study showed that the doses of heavy metals associated with *I. tesselatus* samples which could be used as food packing material under normal conditions, should not affect human health.

References

Ataro, A., McCrindle, R. I., Botha, B. M., McCrindle, C. M. E., & Ndibewu, P. P. (2008). Quantification of trace elements in raw cow's milk by inductively coupled plasma mass spectrometry (ICP-MS). *Food Chemistry, 111*, 243–248.

Batista, B. L., Rodrigues, J. L., de Souza, S. S., Oliveira Souza, V. C., & Barbosa, F. Jr., (2011). Mercury speciation in seafood samples by LC-ICP-MS with a rapid ultrasound-assisted extraction procedure: Application to the determination of mercury in Brazilian seafood samples. *Food Chemistry, 126*, 2000–2004.

Capar, S. G., Mindak, W. R., & Cheng, J. (2007). Analysis of food for toxic elements. *Analytical and Bioanalytical Chemistry, 389*, 159–169.

Chen, C., Ding, Y., Elmahadi, E. A., Zhou, J., Li, Y., & Xu, H. (1999). Study on the isolation, purification and physicochemical properties of polysaccharides from *Indocalamus tesselatus*. *Biomedical Chromatography, 13*, 11–14.

Dick, D., Wegner, A., Gabrielli, P., Ruth, U., Barbante, C., & Kriews, M. (2008). Rare earth elements determined in Antarctic ice by inductively coupled plasma-time of flight, quadrupole and sector field-mass spectrometry: An inter-comparison study. *Analytica Chimica Acta, 621*, 140–147.

Ding, Y. Q., Li, Y., Chen, C. Y., Elmahadi, E. A., & Xu, H. B. (1998). Chromatographic analysis of polysaccharides extracted from Chinese *Indocalamus Tesselatus*. *Biomedical Chromatography, 12*, 86–88.

Eilola, K., & Perämäki, P. (2001). Microwave heated vapor-phase digestion method for biological sample materials. *Fresenius' Journal of Analytical Chemistry, 369*, 107–112.

Gomez, M. R., Cerutti, S., Sombra, L. L., Silva, M. F., & Martinez, L. D. (2007). Determination of heavy metals for the quality control in Argentinian herbal medicines by ETAAS and ICP-OES. *Food and Chemical Toxicology, 45*, 1060–1064.

Husáková, L., Urbanová, I., Audrlická-Vavrušová, L., Šrámková, J., Černohorský, T., Bednaříková, M., et al. (2011). Multi-element analysis of urine by inductively coupled plasma orthogonal acceleration time-of-flight mass spectrometry. *Microchimica Acta, 173*, 173–181.

Husáková, L., Urbanová, I., Šrámková, J., Černohorský, T., Krejcová, A., Bednaříková, M., et al. (2011). Analytical capabilities of inductively coupled plasma orthogonal acceleration time-of-flight mass spectrometry (ICP-oa-TOF-MS) for multi-element analysis of food and beverages. *Food Chemistry, 129*, 1287–1296.

Kim, K. C., Park, Y. B., Lee, M. Jin, Kim, J. B., Huh, J. W., Kim, D. H., Lee, J. B., et al. (2008). Levels of heavy metals in candy packages and candies likely to be consumed by small children. *Food Research International, 41*, 411–418.

Lam, J. C., Chan, K., Yip, Y., Tong, W., & Sin, D. W. (2010). Accurate determination of lead in Chinese herbs using isotope dilution inductively coupled plasma mass spectrometry (ID-ICP-MS). *Food Chemistry, 121*, 552–560.

Larcher, R., Nicolini, G., & Pangrazzi, P. (2003). Isotope ratios of lead in Italian wines by inductively coupled plasma mass spectrometry. *Journal of Agriculture and Food Chemistry, 51*, 5956–5961.

Linsinger, T. (2005). Comparison of measurement result with the certified value, Application Note 1. http://www.erm-crm.org/ERM_products/ application_notes/application_note_1/Pages/index.aspx.

Llorent-Martínez, E. J., Ortega-Barrales, P., Fernández-de Córdova, M. L., Domínguez-Vidal, A., & Ruiz-Medina, A. (2011). Investigation by ICP-MS of trace element levels in vegetable edible oils produced in Spain. *Food Chemistry, 127*, 1257–1262.

Millour, S., Noe, L., Kadar, A., Chekri, R., Vastel, C., & Guerin, T. (2011). Simultaneous analysis of 21 elements in foodstuffs by ICP-MS after closed-vessel microwave digestion: Method validation. *Journal of Food Composition and Analysis, 24*, 111–120.

Rodenas de la Rocha, S., Sanchez-Muniz, F. J., Gomez-Juaristi, M., & Larrea Marin, M. T. (2009). Trace elements determination in edible seaweeds by an optimized and validated ICP-MS method. *Journal of Food Composition and Analysis, 22*, 330–336.

Sanchez Lopez, F. J., Gil Garcia, M. D., Sanchez Morito, N. P., & Martinez Vidal, J. L. (2003). Determination of heavy metals in crayfish by ICP-MS with a microwave-assisted digestion treatment. *Ecotoxicology and Environmental Safety, 54*, 223–228.

Sanz-Segundo, C., HernaÂndez-Artiga, M. P., Hidalgo-Hidalgo de Cisneros, J. L., Bellido-Milla, D., & Naranjo-Rodriguez, I. (1999). Determination of wear metals in marine lubricating oils by microwave digestion and atomic absorption spectrometry. *Mikrochimica Acta, 132*, 89–94.

Skrzydlewska, E., Balcerzak, M., & Vanhaecke, F. (2003). Determination of chromium, cadmium and lead in food-packaging materials by axial inductively coupled plasma time-of-flight mass spectrometry. *Analytica Chimica Acta, 479*, 191–202.

Wang, J., Nakazato, T., Sakanishi, K., Yamada, O., Tao, H., & Saito, I. (2006). Single-step microwave digestion with HNO_3 alone for determination of trace elements in coal by ICP spectrometry. *Talanta, 68*, 1584–1590.

Zhang, Y. X., & Li, D. Z. (2010). Taxonomic notes on *Metasasa* and *Indocalamus nananicus* (*Poaceae: Bambusoideae*). *Nordic Journal of Botany, 28*, 493–495.