



Analytical Methods

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

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ABSTRACT

Analytical capabilities of ICP-*oa*-TOF-MS for rapid, simultaneous and reliable determination of more than 50 major, trace and ultra-trace elements in different food and beverages samples (milk and dairy based products, cereals, meat, offal, sugar and sugar products, potatoes, fats, baby food samples, fruit juices, alcoholic beverages), following microwave closed vessel digestion of samples, were described. Under optimum instrumental conditions, and by using Rh as an internal standard and an external calibration method, ICP-*oa*-TOF-MS enables an accurate analysis, taking about one minute per a sample for all elements and isotopes of interest even for some elements such Zn, Ni, Cu, As or Co whose assay is more difficult when using conventional quadrupole instruments. In order to verify the accuracy and precision of the proposed method, 8 commercially available reference materials representing 3 major groups of food (milk and dairy based products, meat, cereals) were analysed, yielding results in agreement with certified values and the precision below 15%. In addition, accuracy was confirmed by spiked analytical recoveries study and accurate isotopic ratio determinations with the precision typically better than 5% with 5 s data acquisition period, also for other elements of interest whose content was not certified and different sample matrices. Limits of detection (3σ) have varied from 0.04 ng g⁻¹ for Th to 1630 ng g⁻¹ for Ca.

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1. Introduction

Accurate and precise elementary analysis of food and beverages is necessary prerequisite for assessing of nutritional composition and identifying of food contamination sources and potentially toxic elements (Capar, Mindak, & Cheng, 2007). Identification of the geographical origin of food according to elemental composition has grown more important in recent years as consumers are more interested in the provenance of the food (Cubadda, Raggi, & Coni, 2006; Galgano, Favati, Caruso, Scarpa, & Palma, 2008; Joebstl, Bandoniene, Meisel, & Chatzistathis, 2010; Kelly, Heaton, & Hoogewerff, 2005; Lo Feudo, Naccarato, Sindona, & Tagarelli, 2010; Spalla et al., 2009). Inductively coupled plasma mass spectrometry is due to its high sensitivity and multi-element capabilities well established in a number of laboratories for food analysis for the purpose of determination of elements at trace and ultra-trace levels (Walczyk, 2001). Quadruple based spectrometers are currently the most widespread instruments in analytical practice owing to their low cost. However formation of spectral interferences origi-

nating from monoatomic and polyatomic ions produced in plasma from argon and matrix constituents makes the quantification of several trace elements difficult when systems with conventional low-resolution quadruple mass discrimination are used (Evans & Giglio, 1993; Reed, Cairns, Hutton, & Takaku, 1994). To attenuate the interferences to a manageable level, application of this technique thus has to be often combined with a separation/pre-concentration methods (Dressler, Pozebon, & Curtius, 1998), and/or with the use of collision/reaction cell (Cubadda et al., 2006; D'Illo et al., 2008; Dufailly, Noel, & Guerin, 2006) or e.g. mathematical correction equations should be applied (Cubadda & Raggi, 2005; Cubadda, Raggi, Testoni, & Zanasi, 2002; Nardi et al., 2009; Noel, Dufailly, Lemahieu, Vastel, & Guerin, 2005).

The use of magnetic sector high resolution (HR) spectrometers can usually overcome polyatomic interferences that cannot be avoided with Q-ICP-MS (Frazzoli, Cammarone, & Caroli, 2007; Riondato, Vanhaecke, Moens, & Dams, 2001; Martino, Sanchez, & Sanz-Medel, 2001). Further advantages of the HR-ICP-MS instruments over conventional Q-ICP-MS are the extremely low background of less than 0.2 ions s⁻¹ and their high transmission, which results in clearly lower limits of detection, typically lying within the pg L⁻¹ range for non-interfered isotopes in the low res-

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olution mode. Nevertheless because of a high cost of the instrumentation this technique is less accessible to common laboratories.

An introduction of ICP time-of-flight mass spectrometers (ICP-TOF-MS) to the analytical practice has brought many practical advantages which were well demonstrated before now (Sturgeon, Lam, & Saint, 2000). Amongst others, e.g. enhanced sample throughput and elemental coverage wherein a complete mass spectrum within the range from 1 to 260 amu is generated from each ion-gating event, enhanced resolution, unlimited use of internal standards without performance compromises or improved isotope ratio precision capability owing to the high correlation of noise sources for all isotopes which occur with simultaneous sampling can be mentioned (Sturgeon et al., 2000). Limits of detection (LODs) equivalent to those common at quadrupole instruments can be achieved for amu 1–260 within several seconds. No reduction of LOD values is observed with increasing number of analysed elements as in the case of instruments with Q or HR analysers. High sample throughput enables to tolerate higher salinity of samples, which positively impacts/lowers risk of blockage of injector and interface cone. Only very low volume of sample is necessary for analysis. It seems that just these advantages open new opportunities for ICP-MS analysis also in the field of food analysis because of the growing trend toward increasing consumer awareness.

Currently only the small number of laboratories operates with ICP-TOF-MS systems and the practical experiences including the analysis of food are still missing. In order to show the analytical capabilities of this technique for this purpose, the determination of more than 50 elements (Li, Be, B, Mg, Al, Ca, Sc, Ti, V, Cr, Fe, Mn, Ni, Co, Cu, Zn, As, Se, Rb, Sr, Y, Zr, Mo, Pd, Ag, Cd, In, Sn, Sb, Te, Cs, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Tb, Dy, Ho, Er, Tm, Yb, Lu, Hf, Ta, W, Hg, Tl, Pb, Th, U) being used for the purpose of obtaining nutritional information, toxicological assessments or geographical origin in different kinds of widely consumed foods and beverages (milk and dairy based products, cereals and cereal products, meat and meat products, offal, sugar and sugar products, starchy roots or potatoes, fats, baby food samples, fruit juices, alcoholic beverages) was undertaken.

2. Experimental

2.1. Reagents and internal standards

High purity sub-boiling distilled nitric acid (65%, m/v) and hydrogen peroxide (30%, m/v) of Selectipur quality (Fluka, Buchs, Switzerland) were used for the digestion of samples. Sub-boiling distilled HNO₃ (Lach-Ner, Neratovice, Czech Republic) prepared using the BSB system 939IR (Berghof, Germany) was also used in the preparation of standards. All solutions were prepared using deionised water purified using the UltraClear (SG, Germany) pure water system to 0.05 µS cm⁻¹.

Sodium chloride, magnesium chloride, calcium chloride, potassium chloride, sulphuric acid (98%, w/w), hydrochloric acid (36%, w/w) used for interference studies were TraceSelect quality, obtained from Fluka (Fluka, Buchs, Switzerland). Rhodium internal standard solution was prepared from 1 g L⁻¹ Rh solution obtained from SCP Science (Canada). Multi-element stock solution "A" containing 10 mg L⁻¹ of Li, B, Al, Ti, V, Cr, Mn, Ni, Co, Cu, Ga, Ge, As, Se, Rb, Sr, Zr, Mo, Ru, Pd, Ag, Cd, Sn, Sb, Te, Ba, Ta, W, Re, Pt, Hg, Tl, Pb, Bi, Th, U, Cs and Hf and was prepared from single element standards of 1 g L⁻¹ obtained from Analytika Ltd. (Prague, Czech Republic) or SCP Science. Multi-element solution "B" containing 100 mg L⁻¹ of La, Ce, Pr, Nd ("B1") and 20 mg L⁻¹ of Sc, Y, Tb, Ho, Yb, Sm, Eu, Gd, Er, Tm, Lu and Dy ("B2") was obtained from Analytika Ltd. Multielement solution "C" containing 50 mg L⁻¹ of Mg,

Ca, Fe and Zn was prepared from single element standards of 1 g L⁻¹ obtained from Analytika Ltd. Carbon reference solutions were prepared from 10 g L⁻¹ of C stock solution prepared from urea of TraceSelect quality (Fluka).

Calibration solutions: blank, 0.1, 1, 5, 10 and 20 µg L⁻¹ of Li, B, Al, Ti, V, Cr, Mn, Ni, Co, Cu, Ga, Ge, As, Se, Rb, Sr, Zr, Mo, Ru, Pd, Ag, Cd, Sn, Sb, Te, Ba, Ta, W, Re, Pt, Hg, Tl, Pb, Bi, Th, U, Cs, Hf and La, Ce, Pr, Nd, 0.02, 0.2, 1, 2 and 4 µg L⁻¹ of Sc, Y, Tb, Ho, Yb, Sm, Eu, Gd, Er, Tm, Lu and Dy and 50, 100, 150 and 200 µg L⁻¹ of Mg, Ca, Fe and Zn, were prepared daily by appropriate dilution of multielement solutions "A" (500 µg L⁻¹), "B" (500 + 100 µg L⁻¹) and "C" (5 mg L⁻¹) in 50 ml volumetric flasks. To compensate for possible instrumental drift and matrix effects, all samples and solutions contained 0.2 µg L⁻¹ of Rh.

2.2. Samples

Beer (alcohol content 4.1% v/v, solid content of the wort before fermentation 12%), red wine (alcohol content 13.5% v/v), liquid fat milk (3.5% w/v fat), processed cheese (50% w/w fat, dry matter 43%), butter (80% w/w fat), sunflower oil (100%), oat flakes, wheat flour, liver pate (<40% w/w, fat), honey, dried mushrooms, cocoa powder, orange juice (100%), fresh potatoes, yogurt (<3% w/w fat, dry matter 20% w/w) and fruit baby formula (apple and strawberries) were obtained from local supermarkets and stored at -4 °C.

2.3. Quality assurance and quality control

The commercially supplied reference materials: NIST Reference material (RM) 8435 Whole milk powder (National Institute of Science and Technology, NIST, USA), BCR[®] Reference material (RM) No. 150 Spiked Skim Milk Powder (low level) (Institute for Reference Materials and Measurements, IRMM), BCR[®] Reference material (RM) No. 184 Bovine muscle (IRMM), BCR[®] Reference material (RM) No. 185R Bovine Liver (IRMM), GBW Certified reference material (CRM) 08503 Wheat flour (National Research Centre for Certified Reference Materials, NRCRM, China), P-WBF Reference material (RM) No. 12-2-04 Essential and Toxic Elements in Wheat Bread Flour (pb-anal Kosice, Slovakia), Reference material (RM) No. 12-2-03 P-Alfalfa Essential and toxic elements in Lucerne (pb-anal), SMU Reference material (RM) No. 12-02-01 Bovine liver (pb-anal) were analysed.

2.4. Sample preparation

In some cases (cereals, dried mushrooms) samples had to be ground in order to obtain a representative portion of homogenous materials. For this purpose the cryogenic mill (SPEX 6750) with isolated coat and own reservoir of liquid nitrogen was used. Three cycles of grinding were applied; both grinding time and cooling time between individual cycles were 2 min. Pre-cooling time before the start of grinding was 15 min, impactor frequency was 10 Hz.

In all cases samples were digested in closed-vessels with a microwave oven decomposition system (Berghof Speedwave[™] MWS-3[™]). For this purpose a 200–500 mg portion of the sample was weighed into a 100-ml pressure resistant PTFE vessel, except of the cases when the samples of beverages should be digested. For them 4 ml of sample was pipetted in general. For the purpose of a decomposition 5 ml of HNO₃ (65%, m/v) and 2 ml of H₂O₂ (30%, m/v) was added. Samples were digested according to following 5-steps program: (i) 5 min at 160 °C and 80% power (ramp 3 min), (ii) 10 min at 220 °C and 90% power (ramp 5 min), (iii) 5 min at 100 °C and 10% power (ramp 1 min). The resulting colourless solutions were diluted to 50 ml with deionised water. Three independent replicates were analysed for each sample in a total *n*

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