Challenge

Determination of Pb, Fe, Zn and Se in dietary supplements

Solution

Application of High-Resolution Continuum Source solid AA for determination of Pb, Fe, Zn and Se

Determination of Metals in Dietary Supplements Using HR-CS Solid AAS

Introduction

The consumption of dietary supplements is widely spread and on the rise. These dietary supplements are generally used without prescriptions, proper counseling or any awareness of their potential health risks. In order to ensure the safety of these products and increase the awareness of the citizen to benefit from these dietary supplements it is of great importance to perform diligent analysis for (toxic) metals present in dietary supplements. While some metals are essential for living organisms because of their responsibility of maintaining the vital body functions, they have fatal effects when they are taken in excess. Research studies have even linked brain disorder symptoms to over dosage. On the other hand, heavy metals such as Pb and Cd are toxic at much lower levels and are known to induce serious diseases.

Atomic absorption spectrometry (AAS) is a robust, reliable and sensitive analytical technique which is well suited for the determination of heavy metals. This analytical technique is therefore widely employed for this purpose.

Instrumentation

The analysis was performed using the High-Resolution Continuum Source Graphite Furnace AAS contrAA 800 along with the solid sampler SSA 600L equipped with a liquid dosing unit.

Table 1: Configuration of the method and the instrument

Parameter	Specification
Temperature	20°C (room temperature)
Spectral observation width	200 pixel
Signal evaluation	area
Evaluation pixels	3 (5 for Se)
Baseline fit	IBC (iterative background correction)
Auto sampler	SSA 600L
Tube type	solid tube

Table 2: Method and evaluation parameters

Element / Wavelength	$T_{\text{Pyrolysis}}$ [°C]	$T_{\text{Atomization}}$ [°C]	Ramp [°C/s]	Time [s]	Modifier	Spectral observation width [nm]
Pb 283.306 nm	1050	2350	1500	4	5 µL $Pd/Mg(NO_3)_2$	0.32
Fe 304.266 nm	1150	2450	1300	6	-	0.32
Se 207.479 nm	500*/1100	2400	1500	5	5 µL $Pd/Mg(NO_3)_2$	0.26
Zn 307.590 nm	400	1600	1400	5	-	0.33

* oxygen ashing step with air 20 s, subsequently 30 s Ar purge to remove remaining oxygen

Samples and Reagents

Mineral tablets were finely ground and homogenized using a ball mill. Ginseng powder and the content of a capsule were analyzed as received without further sample treatment. A necessary dilution for the determination of Zn was obtained by the addition of spectral carbon powder. For that purpose, 0.04 g of the tablet were homogenized with 1 g of graphite powder using a ball mill resulting in a dilution factor of 25.

The modifier solution was prepared with 0.1% TritonX 100 to ensure wetting of the sample by the modifier.

Results and Discussion

The spectral environment of Fe and Pb shows additional atomic lines of Fe in the respective spectra. However, with a spectral resolution of approximately 2 pm at 200 nm, an interference-free analysis is performed without spectral overlap of other atomic lines. The correction of distinct molecular structures with partial or direct overlap of the analyte line is performed by applying the least squares background correction (LSBC). In this case correction spectra of the molecules that are responsible for the background are recorded, stored in the method and if present subtracted from the sample spectrum (Table 3).

A very insensitive absorption line is required for the analysis of Zn because of high contents in the sample. The available secondary line at 307.590 nm is useful after sample dilution with spectral carbon powder followed by a thorough homogenization.

The solid AA technology allows to directly analyze homogenized solid samples and waives a time-consuming and elaborate sample preparation procedure. Additionally, there is no inherent sample dilution as it is the case for digestion procedures. Analysis time is shortened and sources of contamination i.e. due to chemicals involved are reduced to a minimum.

Using the solid AA technology higher RSD values are obtained in comparison to the liquid technique. This effect can be mainly ascribed to inhomogeneities in the samples and, in this regard, it is recommended to perform the measurements with an increased number of replicates per sample. Inhomogeneity was observed in particular for the gel-like content of the capsules. Smoke during pyrolysis indicates that there is a high carbon content present in the samples. The implementation of an oxygen ashing step into the temperature-time program facilitates removal of the organic matrix and avoids residues on the sample carrier after the measurement.

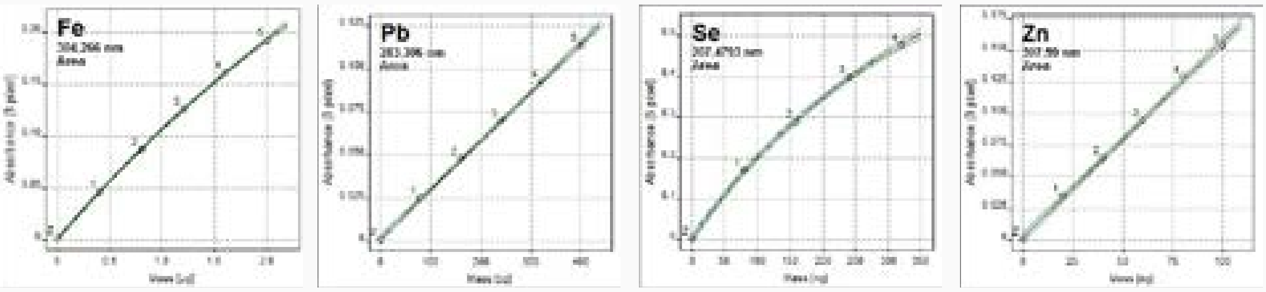


Figure 1: Calibration curves

Table 3: Least squares background correction (LSBC)

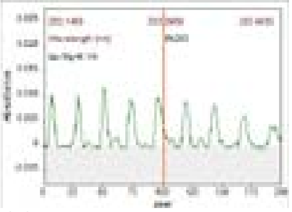
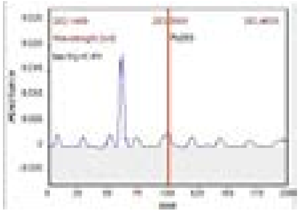
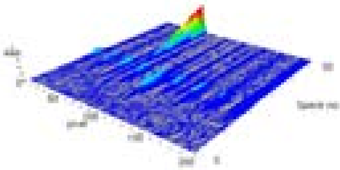
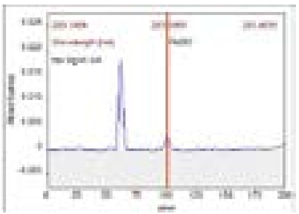
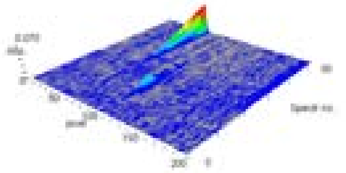
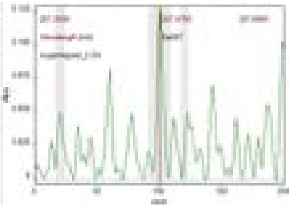
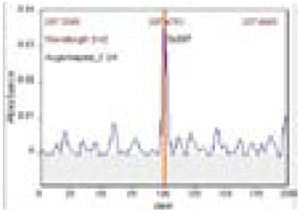
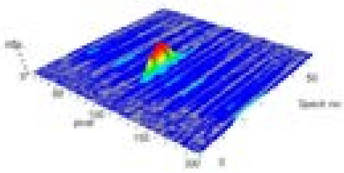
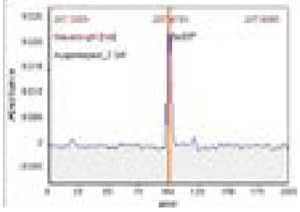
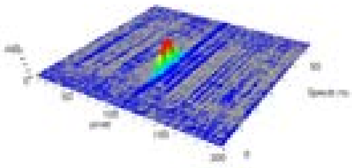
Element	Molecule spectrum	Sample spectrum without LSBC	Sample spectrum with LSBC
Pb	 SiO_2	 	 
Se	 PO (ca. 4 mg $NH_4H_2PO_4$)	 	 

Table 4: Results from measurement of dietary supplements samples on contrAA 800

Sample	Element / Line	Weight of sample [mg]	Weight of tablet/ capsule [g]	Concentration		RSD (n=4) [%]
				[mg/kg]	content per tablet or capsule	
mineral-tablet	Pb 283.306 nm	0.8 – 1.5	1.9689	0.093 ± 0.006	0.183 µg	5.9
	Fe 304.266 nm	0.4 – 0.7	1.9689	2205 ± 18.3	4.34 mg	8.9
	Zn 307.590 nm	0.2 – 0.4	1.9689	2818 ± 47.4	4.55 mg	10.1
ginseng powder	Pb 283.306 nm	2.0 – 2.8	-	< LOQ **	-	-
capsule #1	Se 207.479 nm	1.8 – 4.0	0.3345	76.5 ± 6.16	25.6 µg	18.2
capsule #2	Se 207.479 nm	1.8 – 4.0	0.3275	82.4 ± 6.0	27.0 µg	16.5

**Limit of detection (LOD): 4,84 pg Pb (corresponds to 1,94 µg/kg using a sample weight of 2,5 mg)
Limit of quantification (LOQ): 14,5 pg Pb (corresponds to 5,80 µg/kg using a sample weight of 2,5 mg)
Determined by measuring 11 times the blank (empty sample carrier) with modifier against the calibration curve

Conclusion

With its ability of applying correction algorithms for background as well as spectral interferences, contrAA 800 provides not only a robust and sensitive instrument solution, but furthermore allows for simple yet reliable analysis of trace metal contents in any kind of sample. solid AA reduces sample preparation to a minimum avoiding measurement errors and guaranteeing efficient routine operation.

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Version 1.0 | Author: AnJu
en · 12/2020