

Control Material CS-S-1

As, Cd, Cr, Cu, Fe, Hg, Mn, Mo, Pb, Se and Zn in dried mixed spices

General Information

Intended use	Checking the performance of analytical laboratories engaged in the determination of As, Cd, Cr, Cu, Fe, Hg, Mn, Mo, Pb, Se and Zn in food and other biological samples
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Institute of Nuclear Chemistry and Technology, Dorodna 16 03-195 Warsaw, Poland	LGC STANDARDS Sp. z o.o. ul. Konopnickiej 1 05-092 Łomianki, Poland

Description of material

As the raw material, the spices and herb spices were bought at the food market in Poland. Four types of spices were selected: bay leaves, ginger, cumin and chili (1:1:1:1). The spices were milled in a mill made of stainless steel and sieved through a stainless sieves. The fraction of particles with diameter $d \le 1,0$ mm was collected. The obtained material was further homogenized by mixing in a plastic drum rotated in three directions, distributed into amber glass bottles in portion of ca. 25 g and firmly covered. Care was taken to avoid contamination. The material was then sterilized by electron beam radiation from linear accelerator with dose of 28 kGy. Homogeneity was examined for the sample size of 250 mg for each of element certified i.e. As, Cr, Cd, Cu, Fe, Hg, Mn, Mo, Pb, Se and Zn. Statistical evaluation has been performed following ISO 13528:2015 standard [1] recommendations. Good homogeneity of the material was confirmed for sample masses ≥ 250 mg.

Determination of moisture content

In order to express the concentration of elements on a dry-weight basis, moisture content should be determined on a separate subsample by drying in an oven at 80°C for 24 hours.

Assigning of reference values

Reference values were assigned on the basis of the results provided by several reference laboratories. The elements have to be determined by at least two methods in order to assign reference value. The results were obtained by AAS, ICP-MS and NAA methods. A reference value was calculated as the robust mean value [1] of the laboratory means, an uncertainty was

evaluated according to ISO 13528:2015 [1], ISO GUM [2] and IUPAC harmonized protocol [3].

Element	unit	$\begin{array}{c} \textbf{Reference value } \pm \textbf{expanded} \\ \textbf{uncertainty (k=2)} \\ X_{ref} \pm U \end{array}$
As	mg/kg d.m.	0.148 ± 0.030
Cd	mg/kg d.m.	0.116 ± 0.013
Cr	mg/kg d.m.	2.03 ±0.22
Cu	mg/kg d.m.	7.94 ± 0.39
Fe	mg/kg d.m.	264 ± 28
Hg	µg/kg d.m.	8.1 ± 1.5
Mn	mg/kg d.m.	86.7 ± 8.7
Mo	mg/kg d.m.	0.475 ± 0.025
Pb	mg/kg d.m.	0.515 ± 0.088
Se	µg/kg d.m.	58.2 ± 9.6
Zn	mg/kg d.m.	31.2 ± 2.4

Reference values for Dried Mixed Spices Powder

Long-time stability is monitored during storage. The shelf life of the control material CS-S-1 has been established to be **31 December 2025**.

References

- 1. ISO 13528:2015 Statistical methods for the use in proficiency testing by interlaboratory comparison. Geneva. 2015
- 2. International Organization for Standardization (ISO). Guide to the Expression of Uncertainty in Measurement. ISO. Geneva. 1993 (corrected and reprinted 1995)
- 3. M. Thompson. S.L.R. Ellison. R. Wood. The International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report). Pure Appl. Chem. 78 (2006) 145

Reference laboratories:

Institute of Nuclear Chemistry and Technology. Warsaw Institute of Agricultural and Food Biotechnology. Warsaw Biological and Chemical Reasearch center, University of Warsaw