INSTYTUT CHEMII I TECHNIKI JĄDROWEJ

POLISH CERTIFIED REFERENCE MATERIAL

FOR MULTIELEMENT TRACE ANALYSIS

MIXED POLISH HERBS (INCT-MPH-2)

GENERAL INFORMATION

INTENDED USE:	CHECKING THE ACCURACY OF ANALYTICAL WORK OF THE LABORATORIES ENGAGED IN THE DETERMINATION OF TRACE ELEMENTS IN BIOLOGICAL SAMPLES. CALIBRATION OF APPARATUS AND METHODS
ELEMENTS FOR WHICH CERTIFIED CONCENTRATIONS WERE ESTABLISHED:	Al, As, Ba, Br, Ca, Cd, Ce, Cl, Co, Cr, Cs, Cu, Eu, Hf, Hg, K, La, Lu, Mg, Mn, Nd, Ni, Pb, Rb, S, Sb, Sc, Sm, Sr, Ta, Tb, Th, V, Yb, Zn
ELEMENTS FOR WHICH NON-CERTIFIED "INFORMATION" VALUES ARE AVAILABLE:	Fe, Mo, Na, P, Ti, Tl, U, W

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ORIGIN, PREPARATION AND TESTING

The reference material Mixed Polish Herbs (INCT-MPH-2) was prepared by the Institute of Nuclear Chemistry and Technology (INCT) from the mixture of herbs collected in a non contaminated rural area and used for drugs production. The mixture was supplied by HERBAPOL S.A. (Poland) and consists of nine varieties of herbs grown in Poland. The material was ground using agate ball mill, sieved through the 67 μm nylon sieves and stored in a polyethylene (PE) bag. The fraction retained on the sieve was ground and sieved again. All above operations were performed in a special laboratory in the Polish Geological Institute and special care was taken to prevent any contamination with metals. Approximately 40 kg of ground and sieved Mixed Polish Herbs was finally obtained, which was then homogenized by mixing for 20 hours in a 110 dm^3 PE drum rotated in three directions. After this time the preliminary homogeneity testing was performed by determining content of K, Ca, Mn and Fe by X-ray fluorescence method in five 6 g subsamples taken from the drum. The results did not reveal any inhomogeneity and the next step was distribution of the material in 50 g portions (future certified reference material - CRM) and 10 g portions (intercomparison sample) into 150 cm^3 dark glass and 80 cm^3 polypropylene (PP) bottles with a screw-on cap, respectively. In order to assure the long-term stability, all containers with INCT-MPH-2 were sterilized by electron beam radiation (energy 10 MeV, dose approximately 30 kGy) from the linear accelerator LAE-13/9. Final confirmation of homogeneity of the Mixed Polish Herbs was performed by instrumental neutron activation analysis (INAA) analyzing 6 subsamples taken from different containers chosen at random and comparing the results with those obtained for 6 subsamples taken from the one (seventh, randomly chosen as well) container. Statistical evaluation using Fisher's and t-Student's tests for the sample size 100 mg proved good homogeneity in case of all examined elements i.e. Co, Cr, Fe, K, Mn, Na and Rb. The analogous determinations were performed for much lower nominal sample mass amounting to 5 mg. Statistical criteria were fulfilled for the most of the elements studied. These results prove potential suitability of the Mixed Polish Herbs for analytical techniques utilizing sample masses significantly lower than 100 mg. Long term stability was checked by comparing results obtained for two bottles (randomly chosen) stored under different controlled conditions: the first in the air-conditioned room at 20°C (normal storage) and the second in the CO_2 incubator (ASAB) at 37°C, 100% humidity and 5% CO_2. Samples of the CRM (ca. 100 mg) were taken from each bottle after 0, 4, 14, 15 and 18.5 months of storage and concentration of six selected elements (Ce, Co, Fe, Rb, Sc and Zn) was determined by INAA method. Statistical evaluation of the obtained results using t-Student's test indicates that there are no significant differences between the results obtained in both experimental conditions examined and that no significant trends can be observed. Consequently, it can be stated that the material is stable in time. The test is being continued and stability of INCT-MPH-2 will be monitored during all storage. Microscopic examination of the Mixed Polish Herbs has shown that most particles had Martin's diameter (arithmetic mean of the maximum distance between opposite sides of a particle and the distance in perpendicular direction) lower than 25 μm.

Determination of moisture content

In order to express the concentrations of elements on a dry-mass basis, moisture content should be determined on a separate subsample (not that taken for analysis) by drying at 70°C for 30 hours.

CERTIFICATION

Assigning of certified values was done on the basis of worldwide interlaboratory comparison in which 109 laboratories from 19 countries participated providing 1489 laboratory averages (6956 individual determinations) for 71 elements.

Their cooperation is gratefully acknowledged. The participants along with the INCT-MPH-2, analyzed also the CRM provided by the organizers the identity of which was unknown to them. This enabled to create two databases. The first called "original" contained all results for all elements in INCT-MPH-2 sent in by participants. The second database called hereafter "alternative" was created from "original" database for these elements for which certified values in the CRM, sent to participants and analyzed by them along with INCT-MPH-2, were available. The alternative database was created as follows: For every laboratory its results obtained for a given element in the CRM were examined. If the confidence limits of the laboratory results at a significance level $\alpha = 0.05$ did not overlap with confidence limit of the CRM, the results of this laboratory for the element in question were removed from the original population of results for INCT-MPH-2. Each of the two databases was subjected to statistical evaluation. Statistical evaluation of results for individual elements was based on the method published previously [1] with some later modifications [2-7]. This approach is being based on outlier's rejection procedure which uses concurrently four statistical criteria (namely those of Dixon (D), Grubbs (G), Skewness (S) and Kurtosis (K)) at the significance level of 0.05, followed by calculation of the overall means of results remaining after outlier rejection, standard deviations, standard errors, confidence intervals etc.
The criteria used to decide whether the overall mean can be given the status of certified value were as follows:
1. The ratio of the one-sided confidence interval and the overall mean:

$$\frac{SD \cdot t_{0.05}}{\bar{X} \cdot \sqrt{N}} \begin{cases} \leq 20\% \text{ (trace elements)} \\ \leq 10\% \text{ (major elements)} \end{cases}$$

or relative standard deviation:

$$\frac{SD}{\bar{X}} \begin{cases} \leq 25\% \text{ (trace elements)} \\ \leq 15\% \text{ (major elements)} \end{cases}$$

For the purpose of this work elements with concentration exceeding 5000 mg/kg (ppm) were considered to be the major elements.
2. The overall mean was calculated on the basis of at least four "accepted" laboratory means ($N \geq 4$) obtained by more than one analytical technique. If results from only one analytical technique are available the number of "accepted" laboratory averages used for the calculation of the overall mean cannot be smaller than five ($N \geq 5$).
3. If the conditions (1) and (2) are fulfilled but the number of outliers exceeds 50%, the additional procedure is activated which repeats the process of outlier rejection from the beginning checking simultaneously the changes of the mean and standard deviation accompanying successive rejections. The process of rejecting of outliers is then stopped when the successive change in both the mean and standard deviation becomes lower or equal to 15%. The condition (1) is then rechecked.
4. If the above criteria are met but there are indications that after outlier rejection performed on the whole population, the remaining populations of results obtained by various analytical techniques differ significantly the assignment of certified value is suspended.
"Information" values were assigned to those elements for which the results while not fulfilling simultaneously the conditions (1)-(4) still fulfilled the following condition:

$$\frac{SD \cdot t_{0.05}}{\bar{X} \cdot \sqrt{N}} \begin{cases} \leq 50\% \text{ (trace elements)} \\ \leq 30\% \text{ (major elements)} \end{cases}$$

calculated on the basis of at least three "accepted" laboratory averages, and are quoted as numbers only, i.e. without confidence intervals. The elements for which the data did not fulfill the above criterion were considered to be outside of any classification. Evaluation of data from the original and alternative databases by the method described above gave in most cases similar values of the overall mean and the confidence interval. For some elements (Cd, Co) these data were additionally confirmed by the results obtained by the definitive method. The values for Al, As, Ba, Br, Ca, Cd, Ce, Co, Cr, Cs, Cu, Eu, K, La, Mg, Mn, Ni, Pb, Rb, S, Sm, Sr, Tb, Th, V, and Zn were finally determined using the alternative database. So, in certain sense these data are traceable to the existing CRM. The values for other elements were derived from the original database.
According to recent trends and recommendations for CRM producers, in addition to analytical uncertainties also the uncertainty due to the long term stability is taken into account. Consequently, the combined standard uncertainty of the certified value u_c consists of two contributions:

$$u_c = \sqrt{u_i^2 + u_s^2}$$

where u_i is estimated as standard deviation of the overall mean and u_s the standard uncertainty estimated from the long term stability studies. The expanded uncertainty (U) is obtained by multiplying u_c by a coverage factor k = 2 corresponding to 95% confidence level.

CERTIFIED VALUES FOR INCT-MPH-2 ($\bar{X} \pm U$)

Major and Minor Elements

Element	Concentration wt%
Ca	1.08 ± 0.07
Cl	0.284 ± 0.020
K	1.91 ± 0.12
Mg	0.292 ± 0.018
S	0.241 ± 0.014

Trace Elements

Element	Concentration mg kg^{-1}	Element	Concentration ng g^{-1}
Al	670 ± 111	As	191 ± 23
Ba	32.5 ± 2.5	Cd	199 ± 15
Br	7.71 ± 0.61	Co	210 ± 25
Ce	1.12 ± 0.10	Cs	76.0 ± 7.0
Cr	1.69 ± 0.13	Eu	15.7 ± 1.8
Cu	7.77 ± 0.53	Hf *)	236 ± 20
Mn	191 ± 12	Hg*)	17.6 ± 1.6
Ni	1.57 ± 0.16	La	571 ± 46
Pb	2.16 ± 0.23	Lu	9.0 ± 1.5
Rb	10.7 ± 0.7	Nd	457 ± 91
Sr	37.6 ± 2.7	Sb	65.5 ± 9.1
Zn	33.5 ± 2.1	Sc*)	123 ± 9
		Sm	94.4 ± 8.2
		Ta*)	18.6 ± 2.3
		Tb	13.5 ± 1.1
		Th	154 ± 13
		V	952 ± 163
		Yb	52.7 ± 6.6

*) certified on the basis of results by single analytical method

INFORMATION VALUES FOR INCT-MPH-2

Element	Concentration mg kg^{-1}	Element	Concentration mg kg^{-1}
Fe	460	Ti	34
Mo	0.520	Tl	0.029
Na	350	U	0.049
P	2500	W	0.035

The preparation and certification of INCT-MPH-2 certified reference material was performed by the staff of the Department of Analytical Chemistry, Institute of Nuclear Chemistry and Technology, under the direction of Prof. dr hab. R. Dybczyński. More information on the material and the certification procedure is available in the published Report [8].

Warszawa, October 31, 2002

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