



POLISH CERTIFIED REFERENCE MATERIAL FOR MULTIELEMENT TRACE ANALYSIS

CORN FLOUR (INCT-CF-3)

It has been complied
with the requirements of ISO Guide 34

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:

B, Br, Cl, Cu, Fe, K, La, Mg, Mn, Mo, Ni, P, Rb, S, Sc, Zn

ELEMENTS FOR WHICH NON-CERTIFIED "INFORMATION" VALUES ARE AVAILABLE:

Al, As, Ba, Ca, Cd, Co, Cr, Cs, Hg, Na, Pb, Sb, Sr, Ti

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ORIGIN, PREPARATION AND TESTING

The reference material Corn Flour (INCT-CF-3) was prepared by the Institute of Nuclear Chemistry and Technology (INCT) from corn grown in Poland produced by ViVi-Tak sc. (Poland) according to Polish standard PN-A-74205:1997. The material was sieved through the 250 μm nylon sieves and stored in a polyethylene (PE) bag. Approximately 50 kg of sieved corn flour was collected. Examination by optical microscopy revealed that Martin's diameter of over 98% of particles was below 25 μm . The whole lot of corn flour 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 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 into 150 cm^3 polypropylene (PP) bottles with a screw cap in 50 g portions (future certified reference material) and in 10 g portions into 60 cm^3 polyethylene terephthalate (PET) bottles (intercomparison sample), respectively. In order to assure the long-term stability, all containers with INCT-CF-3 were sterilized by electron beam radiation (energy 10 MeV, dose 28 kGy) from the linear accelerator LAE-13/9. Final confirmation of homogeneity of Corn Flour 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, Fe, K, Mn, Rb, Sc, Br, Na and Zn. These results prove that the material can be considered as homogeneous at least for sample mass ≥ 100 mg. Long-term stability was checked by comparing results obtained for one bottle (randomly chosen) stored under controlled conditions: in the air-conditioned room at 20°C (normal storage). Samples of the CRM (ca. 100 mg) were taken from the bottle after 0, 20 and 32 months of storage and concentration of six selected elements (Co, Fe, K, Rb, Sc and Zn) was determined by INAA method. Short-term stability was examined by the determination of concentrations of the above mentioned elements in the bottle stored in the CO₂ incubator (ASAB) at 37°C, 100% humidity and 5% CO₂. 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-CF-3 will be monitored during the whole storage.

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 taken for analysis) by drying at 80°C for 24 hours.

CERTIFICATION

Assigning of certified values was done on the basis of world-wide interlaboratory comparison in which 92 laboratories from 19 countries participated providing 962 laboratory averages (4228 individual determinations) for 57 elements. Their cooperation is gratefully acknowledged. The participants along with the INCT-CF-3, 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-CF-3 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-CF-3, 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-CF-3. Each of the two databases was subjected to statistical evaluation. The 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 can not 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 Mo these data were additionally confirmed by the results obtained by the definitive method [8]. The values for Al, As, Ba, Br, Ca, Cd, Co, Cr, Cs, Cu, K, La, Mg, Mn, Ni, P, Pb, Rb, S, Sr and Zn were finally determined (certified or information values assigned) 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 and short-term stability and moisture determination are taken into account. Consequently, the combined standard uncertainty of the certified value u_c consists of four contributions:

$$u_c = \sqrt{u_i^2 + u_{ls}^2 + u_{ss}^2 + u_m^2}$$

where u_i is estimated as standard deviation of the overall mean, u_{ls} – the standard uncertainty estimated from the long-term stability studies, u_{ss} – the standard uncertainty estimated from the short-term stability studies and u_m – the standard uncertainty due to moisture determination. 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-CF-3 ($\bar{X} \pm U$)

Element	Unit	$\bar{X} \pm U$	Element	Unit	$\bar{X} \pm U$
B	mg kg ⁻¹	1.65 ± 0.33	Mn	mg kg ⁻¹	4.98 ± 0.22
Br*	mg kg ⁻¹	0.388 ± 0.046	Mo	ng g ⁻¹	152 ± 12
Cl*	mg kg ⁻¹	397 ± 33	Ni	mg kg ⁻¹	0.383 ± 0.039
Cu	mg kg ⁻¹	1.63 ± 0.13	P	mg kg ⁻¹	2831 ± 97
Fe	mg kg ⁻¹	32.0 ± 1.4	Rb	mg kg ⁻¹	0.912 ± 0.042
K	mg kg ⁻¹	3157 ± 119	S*	mg kg ⁻¹	919 ± 121
La	ng g ⁻¹	6.6 ± 1.2	Sc*	ng g ⁻¹	2.13 ± 0.32
Mg	mg kg ⁻¹	1066 ± 37	Zn	mg kg ⁻¹	20.09 ± 0.76

* certified on the basis of results by single analytical method

INFORMATION VALUES FOR INCT-CF-3

Element	Concentration	Element	Concentration
Al	12 mg kg ⁻¹	Cs	4 ng g ⁻¹
As	10 ng g ⁻¹	Hg	1.5 ng g ⁻¹
Ba	0.117 mg kg ⁻¹	Na	4.4 mg kg ⁻¹
Ca	40 mg kg ⁻¹	Pb	52 ng g ⁻¹
Cd	7 ng g ⁻¹	Sb	11 ng g ⁻¹
Co	16 ng g ⁻¹	Sr	0.103 mg kg ⁻¹
Cr	0.137 mg kg ⁻¹	Ti	0.40 mg kg ⁻¹

The shelf life of INCT-CF-3: 31 December 2015.

The preparation and certification of INCT-CF-3 certified reference material was performed by the staff of the Department of Analytical Chemistry, Institute of Nuclear Chemistry and Technology, under the direction of Dr. H. Polkowska-Motrenko. More information on the material and the certification procedure is available in the published report [9].

Warszawa, March 31, 2006

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INSTYTUT CHEMII I TECHNIKI JĄDROWEJ

It has been complied
with the requirements of ISO Guide 34

**POLISH CERTIFIED REFERENCE MATERIAL
FOR MULTIELEMENT TRACE ANALYSIS**

CORN FLOUR (INCT-CF-3)

1. **The stability of Corn Flour (INCT-CF-3) has been monitored during storage. On the basis of the obtained results, the shelf life is extended until 31 December 2024.**
2. The material is stored in an air conditioned room, in which the temperature does not exceed 22°C. Its stability is monitored at appropriate time intervals.
3. The users should store the material at room temperature [(20±5)°C] in tightly closed original containers.
4. For chemical analysis and simultaneous determination of water content, samples of the material of adequate size should be taken. Immediately after sampling, it is necessary to cover firmly the container. The unused part of samples must not be placed back in the container.

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