

## Determination of trace elements in food reference materials by instrumental neutron activation analysis

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**Abstract:** Two biological Certified Reference Materials (CRMs), KRISS 108-04-001 (oyster tissue) and 108-05-001 (water dropwort stem), were prepared by Korea Research Institute of Standards and Science (KRISS) during FY '01. The certified values of these materials had been determined by Isotope Dilution Mass Spectrometry (IDMS) for six elements (Cd, Cr, Cu, Fe, Pb and Zn). Additional analytical works are now progressing to certify the concentrations of a number of the environmental and nutrimental elements in these CRMs. The certified values in a CRM are usually determined by using a single primary method with confirmation by other method(s) or using two independent critically-evaluated methods. Instrumental Neutron Activation Analysis (INAA) plays an important role in the determination of certified values as it can eliminate the possibility of common error sources resulting from sample dissolution. In this study INAA procedure was used in determination of 23 elements in these two biological CRMs to acquire the concentration information and the results were compared with KRISS certified values.

**Key words:** Instrumental neutron activation analysis, oyster tissue, water dropwort, trace elements, certified reference material, comparator method

### 1. Introduction

The certified values in a Certified Reference Material (CRM) are usually determined by using a single primary method with confirmation by other method(s) or using two independent critically-evaluated methods.<sup>1</sup> Instrumental Neutron Activation Analysis (INAA) has been one of the most important methods for reliable and accurate, and more or less sensitive determination of a number of elemental constituents in many complex-matrix materials. In addition, INAA require no modification of a

substance during analysis (dissolution, separation, derivatization) which may constitute breaks in the chain of traceability or create additional sources of uncertainty.<sup>2,3</sup> Therefore the use of INAA as one method can eliminate the possibility of common uncertainty sources resulting from sample dissolution.

Korea Research Institute of Standards and Science (KRISS) released the two biological CRMs, KRISS CRM 108-04-001 (oyster tissue) and 108-05-001 (water dropwort stem).<sup>4</sup> These CRMs are currently certified for six elements (Cd, Cr, Cu, Fe, Pb and Zn) by Isotope Dilution Mass Spectrometry (IDMS) as a

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single primary method. If the concentration of additional elements were known, the utility of these CRMs could be increased. Additional analytical works are now progressing to certify the concentrations of a number of the environmental and nutritional elements in these materials. This paper describes the comparator INAA procedures for acquiring the concentration information for 20 elements and the results were compared with KRISS certified values. Every attempt has been made to optimize and assess the accuracy of measurements by INAA procedures.

## 2. Experimental

### 2.1. Preparation of biological CRMs

#### 2.1.1. Oyster tissue CRM(KRISS 108-04-001)

Oyster used to prepare this CRM was collected from the west coast of Korean Peninsular. The oysters had been cultivated during 18 months and its mean size was 1.5 cm. After peeling off the shell a total of approximately 150 kg of wet oyster tissue was collected. Oyster tissue was cleaned thoroughly with tap water, filtered out water, and then freeze-dried. The dried material was pulverized by variable speed rotor mill (FRISCH pulverisette 14, Germany) with titanium balls, sieved at 250  $\mu\text{m}$  (46% passing), homogenized in a Teflon coated Y-blender, packaged in screw-cap amber glass bottles each containing approximately 15 g, and then radiation sterilized at 20 kGy ( $^{60}\text{Co}$ ) dose. A number of the packaged bottles were 330.

#### 2.1.2. Water dropwort CRM (KRISS 108-05-001)

Water dropwort used to prepare this CRM was collected from Chunbuk province of Korea. The water dropwort had been cultivated during 5 months at the water rice field. After cutting out part of root a total of approximately 160 kg of water dropwort was collected. The next preparation procedures (wash, dry, pulverization, sieving, homogenization, sterilization, etc.) were same as preparation of oyster tissue CRM. 330 bottles were produced, each contain ca. 8 g of powder.

### 2.2. Analysis Procedure by INAA

The comparator INAA method<sup>5</sup> was used following established irradiation and counting procedures. The INAA procedure was conducted in a sequence of irradiation and counting steps. A short-time irradiation was followed by two counts for elements with short-lived nuclides; this then was followed by a long-term irradiation and the counts for elements with intermediate- and long-lived nuclides. The INAA procedure incorporated twenty-nine elements, however mass fraction values with sufficiently low uncertainties were obtained for 23 elements in oyster tissue and 21 elements in water dropwort, with the other elements not found above the limits of detection. National Institute of Standards and Technology (NIST) SRM 1566b Oyster Tissue served as control materials for this work. The procedures were as follows:

#### 2.2.1. Sample Preparation

Six test portions of nominally 100 mg were taken from each bottle of the CRMs, and two 200 mg portions from one bottle of the control material. A pellet was formed from each test portion by pressing the material in a 13 mm diameter "KBr"-die at approximately 1.0 tons. The pellet was then weighed and sealed in polypropylene(PP) as the primary sample container. For the long irradiation, these packaged samples were additionally sealed in Linear Polyethylene (LPE) film. The dry weight correction factors (wet/dry ratio) were determined at the time of sample preparation after drying in a glass desiccator containing magnesium perchlorate as desiccant for 11 days. For each bottle the determined factors were used to correct the sample mass to dry weight.

The standards were taken from the multi-element solutions and pipetted on 55 mm diameter Whatman 41 filter papers. After air-drying, the standards were also pelletized and sealed in LPE envelopes. The dimensions of the pellets closely match the size of the CRM samples.

#### 2.2.3. Irradiation

The irradiation was carried out in the RT-1

Table 1. Nuclear data and experimental parameters used in the assay of the samples.

Element/Nuclide	Half Life	$\gamma$ -Energy (keV)	Irradiation	Count / Decay
Ag / $^{110m}\text{Ag}$	249.76 d	657.76 884.68	long	8h / > 25 d
Al / $^{28}\text{Al}$	2.2406 min	1778.99	short	5 min / 2 min
As / $^{75}\text{As}$	1.097 d	559.08	long	1 h / 5 d
Ce / $^{141}\text{Ce}$	32.5 d	145.44	long	8h / > 25 d
Co / $^{60}\text{Co}$	5.271 y	1173.24 1332.50	long	8h / > 25 d
Cr / $^{51}\text{Cr}$	27.704 d	320.08	long	8h / > 25 d
Cs / $^{141}\text{Cs}$	32.501 d	145.44	long	8h / > 25 d
Cu / $^{66}\text{Cu}$	5.120 m	1039.35	short	5 min / 2 min
Fe / $^{59}\text{Fe}$	44.496 d	1099.25 1291.60	long	8h / > 25 d
Hf / $^{181}\text{Hf}$	42.39 d	482.0	long	8h / > 25 d
K / $^{42}\text{K}$	12.36 h	1524.58	short	30 min / 2 d
La / $^{140}\text{La}$	1.678 d	1596.54	long	1 h / 5 d
Mn / $^{56}\text{Mn}$	2.5785 h	1810.77 2113.15	short	10min / 20min
Mo / $^{99m}\text{Tc}$	065.94 h	142.683	long	1 h / 5 d
Na / $^{24}\text{Na}$	014.9590 h	1368.60 2753.99	short long	10min / 20min
Rb / $^{86}\text{Rb}$	18.66 d	1076.69	long	8h / > 25 d
Sb / $^{124}\text{Sb}$	60.2 d	602.73	long	8h / > 25 d
Sc / $^{46}\text{Sc}$	083.83 d	889.25 1120.51	long	8h / > 25 d
Se / $^{75}\text{Se}$	119.779 d	264.66 400.35	long	8h / > 25 d
Sm / $^{153}\text{Sm}$	001.946 d	103.18	long	1 h / 5 d
Th / $^{233}\text{Pa}$	27.0 d	312.01	long	8h / > 25 d
Ti / $^{51}\text{Ti}$	5.76 min	320.08	short	5 min / 2 min
U / $^{239}\text{Np}$	2.3565 d	106.13	long	1 h / 5 d
V / $^{52}\text{V}$	3.75 min	1434.08	short	5 min / 2 min
W / $^{187}\text{W}$	23.9 h	685.74	long	1 h / 5 d
Zn / $^{65}\text{Zn}$	244.1 d	1115.52	long	8h / > 25 d

pneumatic facility of the NIST research reactor at a reactor power of 20 MW. The irradiations for short-lived nuclides were done for 30 s. Each sample was individually irradiated together with one standard and one flux monitor foil, packaged at the center of the irradiation container with the pellets oriented perpendicular to the axis of the irradiation container. For the long irradiation, two similar sets of sample and standard pellets were packaged in two irradiation containers. Irradiation time, spaced 24 h apart, was 3+3 h with a 180-

degree inversion of the irradiation capsule after the first 3 h for flux homogeneity. The third irradiation with two water dropwort samples was added under the same condition to explore the determination of As.

#### 2.2.4. Gamma-Ray Spectrometry

After irradiation, the outer PP film were removed, the samples, standards, flux monitor foils, and blanks counted using the four high purity Ge detectors with active volumes of 60 to 66 cm<sup>3</sup>. Table 1 lists the

relevant nuclear and experimental parameters for gamma-ray assay of each element. For the assay of short-lived nuclides, two counts were carried out after 2 min decay (count A) and after 15 min decay (count B) for samples; the standards and flux monitors were counted after or before the sample counts. For the long irradiations, the first count (count C) was 1 h after 5 d decay, the second count (count D) was 8 h after at least 25 d decay. The gamma energy range was 40 keV to 3500 keV for the count A and count B gamma-ray spectrometers, 40 keV to 2900 keV for the count C and count D spectrometer. All spectrometers were equipped with analog shaping amplifiers (Ortec 672), loss free counting (LFC) modules (ND/Canberra 599) for count A, B, and C, 16 k channel fixed conversion time analog to digital converter (ADC, ND/Canberra 581) and network data acquisition interfaces (AIM, ND/Canberra 566). The amplifier shaping time constant was 3  $\mu$ s for as high as feasible throughput during counts A to C. To achieve better resolution, the time constant was set to 6  $\mu$ s for counts D. Dead time and pile-up correction was achieved with the virtual pulser method incorporated in the LFC, for decaying sources in counts A, B and C via the "loss-free" method, for counts D via the live-time extension method. The counts C and D spectrometers are served by a sample changer which is controlled by the DEC Alphaserber 800 computer (GROUCH) used for networked data acquisition. The two water dropwort samples from the third irradiation were first counted on various spectrometers and then submitted to the same count D.

### 2.2.5. Spectrum and Data Evaluation

Gamma spectrum data were converted to mass fraction values for the identified elements using the Canberra/Nuclear Data VMS software routines installed on the GROUCH computer. Count rates of the elements in the samples are compared to those of the standards for calculation of concentrations. Again, statistical uncertainties are propagated to a final calculated random uncertainty for each element concentration. Of course, all count rates are corrected

for radioactive decay, spectral interferences, and pile-up.

### 2.2.6. Calculation of recommended mass fractions

Weighted mean values are used since varying counting conditions make it necessary to compare results with different statistical uncertainty

### 2.2.7. Quality Assurance

Measures for quality assurance of the results include the review of nuclear interferences, proper consideration of counting losses, and a review of consistency of trace element results determined in the different counts and of different gamma lines as well as, in the case of antimony, also based on different nuclides.

Counting losses due to system dead time and pile-up are corrected by the LFC mode and/or the system live time. Extensive tests of the virtual pulser method have demonstrated long-term reliability of the equipment.<sup>6</sup> The adjustable pulse evolution time (PET) monostable calibration was checked before and after each use.

The use of multiple standards of similar preparation as well as a common element in different standard preparations provides data for estimates on the reproducibility of the standard preparations and the flux variations during irradiation. Metal foil flux monitors were used to partially validate the pipetted multi-element standards. Metal contents in Al, Cu, and Fe foils were determined to be 100%.

## 3. Results and Discussion

The results for the control material SRM 1566b by INAA procedures are listed in Table 2. The mean elemental mass fraction values and their uncertainties for two biological KRIS CRMs as evaluated for the INAA procedures are listed with certified values in Tables 3 and 4. The reporting follows the ISO/NIST Guide to the Expression of uncertainty in Measurements.<sup>7</sup> The estimates for Type A uncertainties have been derived from the

observed standard deviation of the measured samples and the comparator standards, which includes counting statistics and unknown heterogeneity of the materials. The estimates for Type A uncertainties are based on known factors for irradiation and counting geometries of samples and standards as well as uncertainties for the preparation of multi-element standards. The uncertainty of each value is an expanded uncertainty (U) with coverage factor 2 (approximately 95% confidence).

A comparison of the concentrations of 16 elements (Ag, Al, As, Ca, Cd, Co, Cu, Fe, K, Mn, Na, Rb, Se, Th, V, and Zn) determined in this work and their

certified values of SRM 1566b can be found Table 2. Both sets of values for most of elements are consistent within uncertainties of the certified value. Therefore the recommended values for these elements of KRISS oyster and dropwort CRMs are validated through measured values in SRM 1566b which are in agreement with the certified value. Although no direct validation was obtained in measurements of Au, Ce, Cr, La, Hf, and Sc, since these elements are not certified in SRM 1566b, the reported values can serve as a reference. The more detailed discussion of individual elemental determination follows:

Ag: A relatively high mass fraction value was

Table 2. Comparison of elemental mass fractions and uncertainties in NIST SRM 1566b with certified values.<sup>a</sup>

Element	$\gamma$ -Energy keV	INAA Results		Certified values <sup>b</sup>	
		Average	SD	Average	U
Ag	657.76	0.648	0.002	0.666	0.009
	884.650	0.611	0.012		
Al	1778.830	193.7	0.6	197.2	6
As	559.100	7.63	0.19	7.65	0.65
Au	411.8	0.096	0.001		
Ca	3084.54	828	9	838	2
Cd	336.210	2.51	0.33	2.48	0.08
Ce	145.446	0.296	0.011		
Co	1173.240	0.361	0.002	0.371	0.009
	1332.530	0.364	0.004		
Cr	320.1	0.387	0.011		
Cu	1039.35	71.4	6.8	71.6	1.6
Fe	1099.3	201.82	0.16	205.8	6.8
	1291.6	202.81	4.23		
La	1596.4	0.163	0.010		
K(%)	1524.58	0.661	0.027	0.652	0.009
Hf	132.94	0.022	0.004		
Mn	482	0.026	0.006	18.5	0.2
	846.81	18.6	0.4		
Na(%)	2754.1	0.3199	0.0026	0.3297	0.0053
Rb	1076.63	3.308	0.191	3.262	0.145
Sb	1691.04			0.011	0.002
Sc	889.277	0.0632	0.0007		
	1120.545	0.0693	0.0007		
Se	264.5	2.078	0.034	2.06	0.15
	400.66	2.069	0.028		
Th	311.9	0.0411	0.0041	0.0367	0.0043
V	1434.07	0.580	0.021	0.577	0.023
Zn	1115.52	1421	12.3	1424	46

<sup>a</sup>Concentration, mg/kg unless % indicated

<sup>b</sup>Certificate of Analysis, 2001

Table 3. Comparison of elemental mass fractions and uncertainties in KRISS CRM 108-04-001 Oyster tissue with certified values<sup>a</sup>.

Element	$\gamma$ -Energy keV	INAA Results				Certified values <sup>b</sup>	
		Average	Uncertainties			Average	U
			Type A	Type B	U (k=2)		
Ag	657.76	10.95	0.039	0.05	0.13		
	884.65	10.93					
Al	1778.8	325.3	2.59	0.98	5.54		
As	559.1	13.17	0.082	0.048	0.189		
Au	411.8	0.576	0.008	0.002	0.017		
Ca(%)	3084.54	0.202	0.150	0.039	0.310		
Cd	336.26	7.06	0.13	0.04	0.28	7.40	0.22
Ce	145.446	0.51	0.024	0.003	0.050		
	1173.24	0.381	0.017	0.001	0.035		
Cr	1332.5	0.386					
	320.1	0.660	0.030	0.003	0.060	0.45	0.07
Cu	1039.35	331	2.5	3.3	8.25	330.1	4.9
Fe	1099.3	351.6	4.41	1.80	9.53	354	21
	1291.6	353.7					
K(%)	1524.58	1.123	0.015	0.003	0.030		
Hf	132.94	0.036					
	482.00	0.039	0.0012	0.0003	0.0025		
La	1596.4	0.316	0.0114	0.0021	0.0232		
Mn	846.81	38.7	0.790	0.12	1.60		
Na(%)	2754.1	1.197	0.0078	0.0060	0.0195		
Rb	1076.63	4.29	0.037	0.022	0.086		
Sb	1691.04	0.022	0.0029	0.0001	0.0057		
Sc	889.277	0.0671	0.0008	0.0004	0.0018		
	1120.545	0.0702					
Se	264.5	5.938	0.122				
	400.66	5.955	0.032	0.031	0.088		
Th	311.9	0.074	0.0029	0.0005	0.0058		
V	1434.07	0.667	0.0177	0.002	0.0356		
Zn	1115.52	820.2	5.21	2.46	11.53	835.4	10.0

<sup>a</sup>Concentration, mg/kg unless % indicated<sup>b</sup>Certificate of Analysis, 2002

determined in oyster with a low uncertainty. The results for both gamma lines should be combined for a final value. In the control material the value for comparison is based on the 657 keV line since significant spectral overlap occurs with Sc at the 884 keV line. The values for dropwort in this measurement were below the detection limit of 15  $\mu\text{g}/\text{kg}$ .

As: An estimated value for dropwort was obtained through the evaluation of gamma-ray spectra of two

samples measured at very high counting rates (80 kHz) on the Axel detector system which is equipped with an Ortec digital signal processor operating in "zero dead time" mode. Arsenic could be determined in dropwort by using this technology and irradiating each sample separately 24 h apart to allow extended measurement time for each with similar decay times.

Ca: The automatic peak search of the gamma-ray spectra frequently obtains incomplete results in the

Table 4. Comparison of elemental mass fractions and uncertainties in KRISS CRM 108-05-001 water dropwort with certified values<sup>a</sup>.

Element	$\gamma$ -Energy keV	INAA Results				Certified values <sup>b</sup>	
		Average	Uncertainties			Average	U
			Type A	Type B	U (k=2)		
Al	1778.8	554	4.5	1.7	9.6		
As	559.1	0.07 <sup>c</sup>					
Au	411.8	<0.015					
Ca(%)	3084.54	0.545	0.0074	0.0069	0.0201		
Cd	336.21	<1.7				<0.066	
Ce	145.446	1.392	0.0306	0.0092	0.0639		
Co	1173.24	0.222	0.0064	0.0007	0.0128		
	1332.5	0.223					
Cr	320.1	1.001	0.055	0.0052	0.1107	(0.88)	
Cu	1039.35	21.22	2.002	0.212	4.027	22.35	0.823
Fe	1099.3	287.4	3.35	1.47	7.32	299.2	19.85
	1291.6	286.9					
K(%)	1524.58	5.949	0.0467	0.0179	0.100		
Hf	132.94	0.053					
	482	0.056	0.0061	0.0004	0.0122		
La	1596.4	0.423	0.0183	0.0029	0.0371		
Mn	846.81	76.7	0.74	0.23	1.55		
Mo	140.51	5.78	0.058	0.026	0.13		
Na(%)	2754.1	0.525	0.0022	0.0026	0.0068		
Rb	1076.63	35.78	0.118	0.183	0.437		
Sb	1691.04	0.012	0.0016	0.0001	0.0033		
Sc	889.277	0.073	0.0004	0.0005	0.0012		
	1120.545	0.074					
Se	264.5	<0.05					
Th	311.9	0.097	0.0033	0.0006	0.0067		
V	1434.07	0.527	0.0185	0.0016	0.0371		
Zn	1115.52	52.8	2.04	0.16	4.10	50.6	3.02

<sup>a</sup>Concentration, mg/kg unless % indicated

<sup>b</sup>Certificate of Analysis, 2002

<sup>c</sup>two determinations achieved with a special high-rate count on the Axel spectrometer

peak areas for Ca because of peak broadening at the high gamma ray. The results consistent with user interactive evaluations were obtained after adjustment of the peak shape parameters used in the automatic peak area evaluation. NIST SRM 915a calcium carbonate was used as a standard.

Cd: Because of high background the evaluation of the gamma-ray spectra was checked by interactive peak searches and one result was adjusted based on these routines. Results for dropwort were values for

the experimental detection limit of 1.5 mg/kg.

Co: Uncertainties are based in the evaluation of the 1173 keV line; a final value should be formed considering both gamma-ray lines. However, the observed standard deviation of the results exceeds 15% relative for both materials, which may suggest some heterogeneity at the selected sample size.

Cr: The standards had been previously validated in other INAA measurements.<sup>3</sup> The observed difference between the INAA value and KRISS value for

oyster tissue and possibly for dropwort may indicate an incomplete dissolution used in IDMS measurements. And also the observed standard deviation of the results exceeds 10% relative for both materials, which may suggest some heterogeneity at the selected sample size.

Cu: The results reflect relatively poor counting statistics for the measurement of  $^{66}\text{Cu}$ . Radiochemical separation and measurement of  $^{64}\text{Cu}$  would significantly lower the uncertainty of the determinations.

Sb: High variance (35%) was found in the results for oyster and dropwort probably due to the poor counting statistics (>15%) achieved in these measurements. No values were obtained for SRM 1566b. The values should not be used as reference because of high uncertainty.

Se: Results for the 264 keV line are used for better counting statistics and lesser spectral interferences. Results for dropwort were values for the experimental detection limit of 50  $\mu\text{g}/\text{kg}$ . Long counts after extended decay times could lower this detection limit by at least an order of magnitude.

Elements not reported in Tables 2, 3 and 4 include Mo, Sm, Ti, U, and W. Only values for the detection limits were found for Ti, LD = 70 mg/kg, and W, LD = 0.3 mg/kg; no certified values are reported for SRM 1566b. Mercury has not been considered because of transparency of the irradiation packaging for mobilized Hg.

Blank values were determined for the PP packaging material by irradiating two 30  $\text{cm}^2$  samples (half sheets) together with the samples. The results are reported in ng per sample. Some oyster and dropwort samples were counted in the packaging of about  $2 \times 1.5 \text{ cm}^2$ , for all reported elements it was not necessary to correct for contributions from this blank.

On the other hand a comparison of the concentrations of 5 elements (Cd, Cr, Cu, Fe, and Zn) determined in this work and their certified values of KRISS CRMs can be found Table 3 and Table 4. The results of Cd, Cu, Fe, and Zn are in agreement with the certified value, but there is bias

between the measured value and certified one of the result of Cr. The certified values of the KRISS CRMs were determined by IDMS. This bias may have been derived from sample heterogeneity, incomplete dissolution or analyte loss in sample digestion for IDMS procedure.

## 4. Conclusion

The INAA procedure provided reliable results for all reported elements. Excellent agreement was found for the measured values with the certified values in SRM 1566b. Since the test samples were analyzed under the same conditions one can infer similar accuracy for the results on KRISS CRM 108-04-001 (oyster tissue) and CRM 108-05-001 (water dropwort stem). These two materials appear to provide a good source for control analyses in environmental and food related investigations. Caution should be exercised regarding the sample size selected for analysis. The known contaminant elements, e.g. Co and Cr, did not show a desirable repeatability of the analysis at the selected sample size of 100 mg.

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