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Application News

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Food Analysis / ICPMS-2030

Analysis of Trace Elements in Certified Fish Sample with ICP-MS

Introduction

Fish is one of the most important food resource that is widely consumed in many parts of the world due to its high protein content, low saturated fats and omega-3 fatty acid. However, because of industrial discharges from human activities, fish may have trace levels of heavy metals such as arsenic, cadmium, lead, mercury and nickel which are absorbed from the surrounding waters and the foods they eat. Not only do these heavy metals accumulate in organisms and circulate in food chain, they also remain in the sediments of the ecosystem in the long term [1]. The toxicity of these metals poses a concern to human health through the consumption of fish. Fish are the single largest sources of mercury and arsenic for man. Mercury is a known human toxicant and the primary sources of mercury contamination in man are through eating fish [2]. Minamata disease is a methylmercury poisoning associated with the daily consumption of large quantities of fish and shellfish heavily contaminated with the toxic chemical [3]. Here, Shimadzu ICPMS-2030, a inductively coupled plasma mass spectrometer (ICP-MS) was used to conduct a simultaneous analysis of elements in fish. In addition to being highly sensitive, the ICPMS-2030 uses a helium gas collision system that greatly reduces the spectral interference caused by argon and chlorine.

Experimental

The sample used in this analysis was Fish Protein Certified Reference Material for Trace Metals (DORM-4) from National Research Council Canada. There were two sets of preparation. The sample, 0.5 g, was placed in a digestion vessel followed by addition of 5.0 mL of concentrated nitric acid, 2.0 mL of hydrogen peroxide and 1.0 mL of water. The mixture was digested using microwave-assisted digestion system based on AOAC 999.10 procedure. After the digestion process, deionized water was added to the digested sample to a final total volume of 200.0 mL.

The calibration standards were prepared from 1000 ppm copper (Cu), iron (Fe) and zinc (Zn) standards, and 100 ppm ICP multi-element standard solution IX, which contains arsenic (As), cadmium (Cd), chromium (Cr), mercury (Hg), nickel (Ni), lead (Pb) and selenium (Se). The internal standards were prepared from 1000 ppm bismuth (Bi), germanium (Ge), indium (In) and scandium (Sc) standards and diluted to a concentration of 10 ppb with pure water. The standard solutions were purchased from Merck Millipore, Germany. Type E-1 ultra pure water with resistivity of 18 ΩW was used. Four different calibration ranges were prepared to cover the range of the elements based on the certified values. The calibration standards were acid matched to the digested samples.

Measurement was conducted using the Shimadzu ICPMS-2030 ICP-MS spectrometer, equipped with the mini-torch and helium (He) gas collision system. The mini-torch reduces running cost associated with argon gas usage. The helium gas collision system reduces spectral interference caused by polyatomic ions, for example, $^{40}Ar^{16}O$ that interferes with measurement of ^{56}Fe, $^{40}Ar^{35}Cl$ that interferes with measurement of ^{75}As, and $^{40}Ar^{38}Ar$ that interferes with measurement of ^{78}Se. The typical measurement conditions are shown in Table 1. The elements, mass, and calibration ranges used are shown in Table 2.

Table 1. Instrument and measurement conditions

Instrument	:	ICPMS-2030	
Radio Frequency Power	:	1.20	(kW)
Sampling Depth	:	6	(mm)
Plasma Gas Flow Rate	:	8.0	(L/min)
Auxiliary Gas Flow Rate	:	1.10	(L/min)
Nebulizer Gas Flow Rate	:	0.60	(L/min)
Nebulizer	:	Coaxial Nebulizer	
Spray Chamber	:	Cyclone Chamber (electronic cooling)	
Plasma Torch	:	Mini-torch	
Collision gas	:	He	

Table 2. Elements, mass and calibration ranges

Element	Mass	Calibration Range
As	75	0 to 20 ppb
Cd	111	
Cr	52	
Hg	202	
Ni	60	
Pb	208	
Se	78	
Cu	65	0 to100 ppb
Fe	56	0 to 2000 ppb
Zn	66	0 to 200 ppb

❑ Results and Discussion

The quantitative results for the trace elements were within the certification range of Fish Protein CRM DORM-4 as shown in Table 3.

The instrument detection limits (IDL) and the limits of quantitation (LOQ) are shown in Table 4. The IDL is calculated as three times the standard deviation of 10 replicate measurements of a calibration blank, and the LOQ is calculated as ten times the standard deviation of 10 replicate measurements of a calibration blank.

Table 3. Quantitation results of Fish CRM DORM-4

Element	Fish CRM DORM-4		
	Measured Value (mg/kg)	(Duplicate) Measured Value (mg/kg)	Certified Value (mg/kg)
As	6.59	6.69	6.80 ± 0.64
Cd	0.299	0.298	0.306 ± 0.015
Cr	1.88	1.89	1.87 ± 0.16
Cu	15.6	15.5	15.9 ± 0.9
Fe	326	328	341 ± 27
Hg	0.415	0.360	0.410 ± 0.055
Ni	1.31	1.29	1.36 ± 0.22
Pb	0.388	0.389	0.416 ± 0.053
Se	3.44	3.51	3.56 ± 0.34
Zn	55.0	53.3	52.2 ± 3.2

Table 4. Detection Limits

Element	IDL (ppb)	LOQ (ppb)
As	0.003	0.0097
Cd	0.001	0.0044
Cr	0.005	0.018
Cu	0.02	0.065
Fe	0.06	0.20
Hg	0.003	0.011
Ni	0.01	0.040
Pb	0.004	0.014
Se	0.01	0.045
Zn	0.03	0.091

❑ Conclusions

The ICPMS-2030 can provide a sensitive multi-element analysis of the trace elements in fish accurately, even for low concentration of Cd, Hg and Pb.

❑ References

1. Sreenivasa Rao. J., Vasudeva Rao. Y., Devindra. S and Longvah. T. (2014). Analysis of heavy metal concentrations in Indian marine fish using ICP-MS after closed vessel micro wave digestion method. International Journal of Analytical and Bioanalytical Chemistry 4(3): 67-73.
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