



NOTE

Determination of Trace Elements in *Hucho taimen Pallas* from Ganhe River Simultaneously by CRC-ICP-MS

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The trace elements Cu, Pb, Zn, Cd, Ca, Na, Fe, Mn, Cr and Ni in *Hucho taimen Pallas* muscle from Ganhe river were determined by inductively coupled-plasma mass spectrometry with collision reaction cell (CRC-ICP-MS). The ICP-MS operating parameters and the pre-treatment conditions were optimized in the same time. The standard curve correlation coefficient range from 0.9992-0.9999 and the relative standard deviation (RSD) was from 2.31-4.85 % and the recovery range from 94.21-104.5 %. The data showed that CRC-ICP-MS is an accurate and precise method to determine the content of trace elements in *Hucho taimen Pallas* muscle, *Hucho taimen Pallas* muscle contains rich trace elements and heavy metals meet industry standards for pollution-free agriculture of aquatic requirements.

Key Words: Coupled-plasma mass spectrometry with collision reaction cell, *Hucho taimen Pallas*, Trace elements.

Hucho salmon also named as *Hucho taimen Pallas*, belonging to Salmoniformes head, Salmonidae, Salmoninae subfamily, *Hucho* genus, is one kind of rare cold-water fish living in northern China. *Hucho salmo*'s natural resource has come to the verge of extinction and has been included in the "China Red Data Book of Endangered Animals Fish"¹. As the population of rare salmon *Hucho* is small, it is difficult to collecting samples, so researches on *Hucho* salmon have been rarely reported. The samples come from Ganhe river which is one of *Hucho* salmon origins, a tributary of Nen river in north-eastern China.

Contents of trace elements in fish muscle tissue are comprehensive evaluation of their nutritional value, food safety and environmental pollution where fishes live²⁻⁵. Today there are many detection methods of trace elements, commonly used method such as atomic absorption method⁶, inductively coupled plasma atomic emission spectrometry⁷⁻⁹, colorimetric and fluorescence method, etc. In this paper, 13 trace elements in *Hucho* salmon muscle were determined simultaneously by inductively coupled-plasma mass spectrometry¹⁰⁻¹⁶ and eliminating the interference with collision reaction cell method.

Digestion of samples: The samples are wild fishes caught from the Ganhe river in November 2009, which are robust and fresh, body weight was 1.5 ± 0.2 kg. Firstly washing the surface of fish cleanly with deionized water and sampling its muscle after peeled, chopped and stir adequately. Weigh 0.5 g muscle minced in nitric acid 8 mL, adding PTFE digestion

tank, the microwave digestion conditions was shown in Table-1. After cooling the digestion tank, adding volume to 25 mL for detection.

TABLE-1
MICROWAVE DIGESTION TEMPERATURE
CONTROL PROGRAM

Step	Maximum power (W)	Climb time (min)	Pressure (psi)	Temp. (°C)	Hold time (min)
1	1200	5	300	120	5
2	1200	10	300	190	10

Instrument parameters: RF power has greater influence on the ratio of instrument signal to noise. According to the principle of covering all detected mass number, 10 ng mL⁻¹ of Cu and Pb, 100 ng mL⁻¹ of Na and Ca were taken for investigating signal strength under different power, the four elements obtained the best signal strength at RF power 1100, 1200, 1200 and 1250 W, respectively. But taking into account the too high RF power could damage torch, so we selected 1200 W as detection RF power. The operating conditions were showed in Table-2.

Standard curve: Multi-element standard solution was diluted to 0, 10.0, 20.0, 50.0, 100.0, 200.0 µg/L with nitric acid, drawing standard curve for determining concentrations of Cu, Pb, Zn, Cd, Ca, Na, Fe, Mn, Cr and Ni in *Hucho* salmon samples (concentrations of Na, Ca standard working solution are 100 times of other elements). Table-3 shows that the linear

TABLE-2
OPERATING PARAMETERS OF ICP-MS

Parameters	RF power (W)	Carrier gas flow (L min ⁻¹)	Cooling gas flow (L min ⁻¹)	Atomization temperature (°C)	Sampling depth (mm)	Repeats
Value	1200	1.25	15.0	2.0	6	3

TABLE-3
STANDARD CURVE, CORRELATION COEFFICIENT, LIMITS OF DETECTION AND RELATIVE STANDARD DEVIATIONS (RSD) OF DETECTED ELEMENTS

Elements	Linear range (ng mL ⁻¹)	Curve equation	Correlation coefficient	Recovery (%)	Limits of detection (ng mL ⁻¹)	RSD (%)
Cu	0-200	Y = 3.582X - 0.613	0.9992	96.68	0.029	2.35
Pb	0-200	Y = 0.8362X + 0.026	0.9996	104.4	0.091	4.56
Zn	0-200	Y = 0.759X + 0.7348	0.9993	96.59	0.004	4.63
Cd	0-200	Y = 0.0951X + 0.031	0.9997	94.21	0.005	3.21
Ca	0-20000	Y = 0.002X + 0.198	0.9997	94.82	0.009	2.89
Na	0-20000	Y = 0.859X + 24.83	0.9996	104.5	0.041	4.62
Fe	0-20000	Y = 0.0243X + 1.25	0.9999	97.67	0.019	4.29
Mn	0-200	Y = 1.217X + 0.226	0.9995	95.49	0.015	4.85
Cr	0-200	Y = 0.112X + 0.039	0.9999	100.2	0.005	3.96
Ni	0-200	Y = 0.231X + 0.032	0.9993	97.91	0.006	2.31

ranges were wider and standard curve slopes range from 0.9993-0.9999, which full achieved the testing requirements.

Determination of detection limit: Cod fish samples were selected for recoveries of test samples to test the accuracy of this method. This sample was consecutively determined 11 times, three-fold of the standard deviation of all elements were the detection limits, just as in Table-3.

Detection of samples: From the data (Table-4), Hucho salmon from Ganhe river contains many and much trace elements, such as copper, zinc and chromium, which are essential trace elements for human health. When excessive intake of copper, zinc, chromium and other heavy metals, human body will be threaten. Present research showed that all the limited hazardous elements meets to agricultural industry standards of aquatic products NY5073-2006¹⁷ and FAO standard¹⁸. So it is concluded that Hucho salmon from Ganhe river is nutritional and safe according to trace elements and heavy metals.

TABLE-4
CONCENTRATIONS OF TRACE ELEMENTS IN
Hucho taimen Pallas mg kg⁻¹

Elements	Results			Mean
	Parallel sample 1	Parallel sample 2	Parallel sample 3	
Na	519.4	523.6	518.7	520.6
Ca	169.9	171.3	168.8	170.0
Cr	0.05	0.052	0.049	0.050
Mn	0.123	0.121	0.124	0.123
Fe	4.400	4.300	4.500	4.400
Ni	0.023	0.022	0.021	0.022
Cu	0.382	0.376	0.378	0.379
Zn	4.760	4.750	4.780	4.760
Cd	0.005	0.005	0.006	0.005
Pb	0.016	0.019	0.018	0.018

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