

# **Application Data Sheet**

#### ICP-AFS

Inductively Coupled Plasma Atomic Emission Spectrometry

## Analysis of Fish Sample with ICP-AES for Trace Element Contamination

No. AD-0068

Fish is one of the most important food resource and is widely consumed in many parts of the world because of its high protein content, low saturated fats and omega-3 fatty acid. It is also rich in calcium and phosphorus. However due to industrial pollution, many fish have trace levels of contaminants such as arsenic and lead which are absorbed by surrounding waters and from foods they eat. Hence, toxic metal accumulation in fish due to toxic effluents can have an adverse effect on the health of human beings. The itai-itai disease was the documented case of mass cadmium poisoning in Toyama Prefecture, Japan, starting around 1912.

This application data sheet demonstrate the ability of ICPE-9000 simultaneous ICP atomic emission spectrometer in quantitative analysis of trace elements in fish.

#### **□Sample and Preparation**

Fish Protein Certified Reference Material for Trace Metals (DORM-4) was used as fish sample. The sample was weighed out to 0.5g into a digestion vessel. 5.0mL concentrated nitric acid, 2.0mL of hydrogen peroxide and 1.0mL of water were added. The sample was digested using microwave-assisted digestion system and the digestion procedure was based on AOAC 999.10 [1]. After the digestion process, deionized water was added to the digested sample to make up to a final total volume of 20.0mL. A duplicate sample was prepared to check the reproducibility of the method.

The calibration standards were prepared from 100ppm ICP multi-element standards and 1000ppm AAS standards from Merck, Germany, and were acid matched to the digested samples. Table 1 shows the target elements, wavelengths selected and the calibration curve standards concentration prepared for each element.

Table 1. Target elements, wavelengths and calibration curve standard concentrations prepared

| Element | Wavelength<br>(nm) | Standard (ppm) |      |      |     |
|---------|--------------------|----------------|------|------|-----|
|         |                    | 1              | 2    | 3    | 4   |
| As      | 189.042            | 0              | 0.01 | 0.05 | 0.2 |
| Cd      | 214.438            | 0              | 0.01 | 0.05 | 0.2 |
| Cr      | 205.552            | 0              | 0.01 | 0.05 | 0.2 |
| Cu      | 324.754            | 0              | 0.5  | 1    | 2   |
| Fe      | 238.204            | 0              | 5    | 10   | 20  |
| Ni      | 231.604            | 0              | 0.01 | 0.05 | 0.2 |
| Pb      | 220.353            | 0              | 0.01 | 0.05 | 0.2 |
| Se      | 196.090            | 0              | 0.01 | 0.05 | 0.2 |
| Zn      | 206.200            | 0              | 0.5  | 1    | 2   |

Table 2. ICP-AES Instrument and analytical conditions

| Instrument              | : | ICPE-9000         |  |
|-------------------------|---|-------------------|--|
| Radio Frequency Power   | : | 1.20 (kW)         |  |
| Plasma Gas Flow Rate    | : | 10.0 (L/min)      |  |
| Auxiliary Gas Flow Rate | : | 0.60 (L/min)      |  |
| Nebulizer Gas Flow Rate | : | 0.70 (L/min)      |  |
| Sample Introduction     | : | Coaxial Nebulizer |  |
| Spray Chamber           | : | Cyclone Chamber   |  |
| Plasma Torch            | : | Mini-torch        |  |
| Observation             |   | Axial             |  |

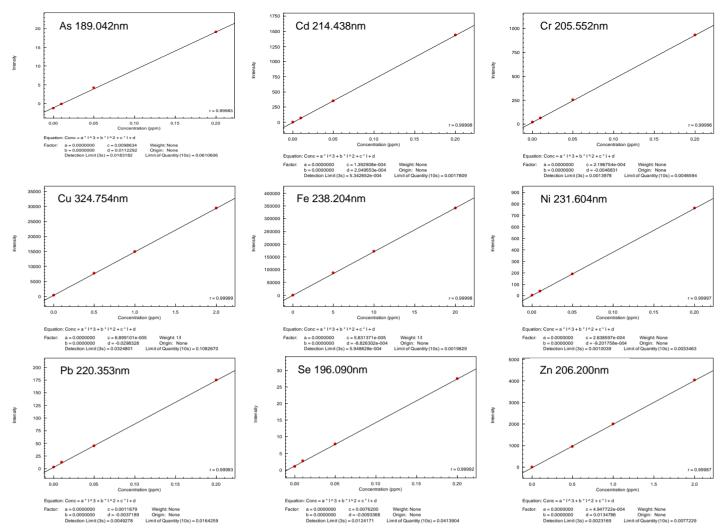


Figure 1. Quantitation calibration curves of nine elements

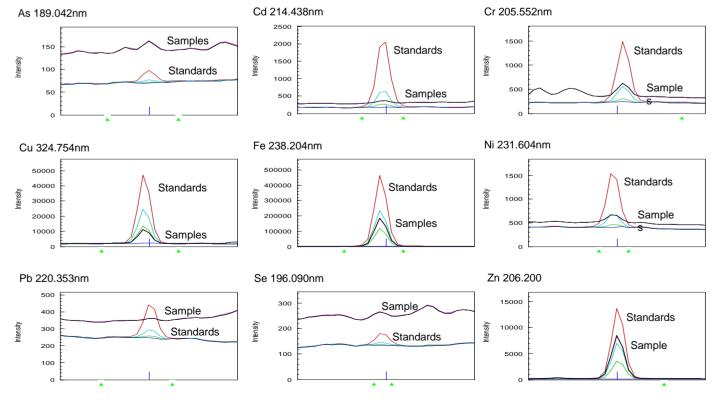


Figure 2. Peak Profiles of nine elements

Table 3. Quantitation results of Fish CRM DORM-4

|         | Fish CRM DORM-4   |       |                            |  |
|---------|---|-------|----------------------------|--|
| Element | Measured Value (Duplicate) (mg/kg) Measured Value (mg/kg) |       | Certified Value<br>(mg/kg) |  |
| As      | 7.07  | 6.83  | $6.80 \pm 0.64$            |  |
| Cd      | 0.312   | 0.312 | 0.306 ± 0.015              |  |
| Cr      | 1.74  | 1.74  | 1.87 ± 0.16                |  |
| Cu      | 15.5  | 15.6  | 15.9 ± 0.9                 |  |
| Fe      | 317   | 318   | 341 ± 27                   |  |
| Ni      | 1.17  | 1.18  | 1.36 ± 0.22                |  |
| Pb      | 0.392   | 0.439 | 0.416 ± 0.053              |  |
| Se      | 3.36  | 3.46  | $3.56 \pm 0.34$            |  |
| Zn      | 50.3  | 51.9  | 52.2 ± 3.2                 |  |

Table 4. Quantitation results and percentage recovery of CRM-TMF

|         | CRM-TMF                   |                            |                         |  |  |
|---------|---------------------------|----------------------------|-------------------------|--|--|
| Element | Measured Value<br>(mg/kg) | Certified Value<br>(mg/kg) | Percentage Recovery (%) |  |  |
| As      | 103                       | 100                        | 103                     |  |  |
| Cd      | 5.32                      | 5                          | 106                     |  |  |
| Cr      | 21.2                      | 20                         | 106                     |  |  |
| Cu      | 46.6                      | 50                         | 93                      |  |  |
| Fe      | 101                       | 100                        | 101                     |  |  |
| Ni      | 19                        | 20                         | 95                      |  |  |
| Pb      | 9.69                      | 10                         | 97                      |  |  |
| Zn      | 989                       | 1000                       | 99                      |  |  |

#### ☐ Results and Discussion

The calibration curves are displayed in Figure 1 and the peak profiles of standards and samples are displayed in Figure 2.

The quantitation results obtained matched the certified values of Fish Protein CRM DORM-4 and are shown in Table 3. Certified Reference Material-Trace Metals in Fish Solution, CRM-TMF, was diluted 1000 times and used to check the calibration curves. The quantitation results and percentage recovery are shown in Table 4.

### □ Conclusions

Shimadzu ICPE-9000 can provide a rapid method to analyze trace elements in fish simultaneously. The results show excellent correlation with the certified reference material.

#### □ Reference

1. AOAC Method 999.10

