National Institute for Environmental Studies

Certified Reference Material No.22
"Fish Otolith"

The National Institute for Environmental Studies (NIES) announces the availability of NIES Certified Reference Material No. 22, Fish Otolith.

This Certified Reference Material (CRM) is intended for use in the quality assurance of the analysis of selected major, minor, and trace elements in fish otolith and similar marine aragonite matrices. One unit of this CRM contains approximately 3 g of otolith powder. This CRM was produced by National Institute for Environmental Studies (NIES), Japan and Western Australian Marine Research Laboratories (WAMRL), Australia, and is distributed by NIES.

Preparation of the CRM

The starting material for this CRM was a stock (1.4 Kg) of sagittal otoliths removed from *Lutjanus sebae* collected from the northwest coast of Western Australia. The otoliths were washed with purified water, dried, pulverized to pass a 105-μm nylon screen and blended to ensure sample homogeneity.

Homogeneity

Homogeneity of this CRM was assessed by measuring the concentrations of major, minor and trace constituents in five 100 mg-subsamples from each of 5 randomly selected vials by inductively coupled plasma atomic emission spectrometry, atomic absorption spectrometry and inductively coupled plasma mass spectrometry after acid dissolution. No significant within- and between-vial variation was detected.

Stability

This CRM is assumed to be stable under the appropriate storage condition, specified below, though the stability has not been rigorously assessed. NIES will monitor the stability of this CRM.

Certified and Reference values

Certified values were determined for Na, Mg, K, Ca, Sr and Ba based on a collaborative analysis involving NIES and 5 other laboratories. Certified values are listed in the Table. Means of the acceptable mean values from the collaborating laboratories were assigned as certified value and their 95% confidence intervals represent the uncertainty ranges attached to the certified values.

Reference values are given for Cu, Zn, Cd and Pb based on values derived from two independent analytical methods including isotope dilution mass spectrometry. Note that reference values were based on the analytical values obtained in a single laboratory (NIES) and may include analytical bias.

Certified and reference values are expressed on a dry weight basis. Analytical values should be corrected by the moisture content to relate to the certified and reference values. See *Instructions* for the drying procedure.

Instruction for Use

Storage

The vessel should be tightly capped and stored at room temperature in a clean, dry environment. Storage in a desiccator is recommended.

Use

Shack the vessel before weighing to ensure homogeneity. Wait until the fine powder settles before opening the cap. Care should be taken to avoid inhalation of the material when handling. Precautions for calcium carbonate handling should be followed.

Drying

This CRM contains 0.2%-0.4 % moisture at the time of certification. As the moisture content may depend on the storage conditions, moisture content should be measured prior to analysis. Approximately 500 mg of the material to be accurately weighed into a dry glass or metal vessel of known weight. The vessel with material to be heated in an electric oven at 85℃ for 4 hours. Then the vessel should be placed in a desiccator with silica gel for 30 min. to cool. Reweigh the vessel with the material and the weight loss to be assigned as moisture content.

Sample decomposition

The decomposition of this CRM is accomplished either by acid digestion (e.g., concentrated HNO_3) at the boiling point of the acid or by dilute acid (e.g., 1M) dissolution at room temperature. Compensation for the interference resulting from undigested organic matter may be necessary in element determinations if the sample solution is prepared by the acid dissolution technique. Care should be taken to compensate for the interference from the calcium matrix in both decomposition techniques. A minimum sample weight of 100mg is recommended for the analysis.

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Certified and reference values for selected elements in NIES/WAMRL fish otolith CRM

	unit	certified value[1,2]	analytical methods used for the certification[4]
Na	%	0.223±0.010	ICP,AAS,FES,INAA
Mg	mg/kg	21± 1	ICP,AAS,ICP-HRMS
K	mg/kg	282± 8	ICP,AAS,FES,INAA
Ca	%	38.8± 0.5	ICP,AAS,INAA,titration
Sr	%	0.236±0.005	ICP,AAS,INAA,ID-ICP-MS
Ba	mg/kg	2.89±0.08	INAA,ICP-MS,ID-ICP-MS
		reference value[1,3]	
Cu	mg/kg	0.74	
Zn	mg/kg	0.47	
Cd	mg/kg	0.0028	
Pb	mg/kg	0.023	

1 Certified and Reference values are expressed on a dry weight basis. Analytical values should be corrected according to the moisture content. See text for the method for moisture content measurement.

2 Certified values are determined based on the mean of the acceptable mean values obtained in the collaborative analysis. Uncertainty is represented by the 95% confidence interval of the mean. The minimum sample on which the certified values are based was 100mg.

3 Reference value were based on analytical values obtained in a single laboratory and are therefore of limited reliability.

4 Abbreviations; ICP, inductively coupled plasma atomic emission spectrometry; AAS, flame atomic absorption spectrometry; FES, flame emission spectrometry; INAA, instrumental neutron activation analysis; ICP-MS, inductively coupled plasma mass spectrometry; ID-ICP-MS, isotope dilution ICP-MS; ICP-HIRMS, ICP high resolution MS

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