National Institute for Environmental Studies, Japan

Date

No.

National Institute for Environmental Studies

Reference Material Information Sheet

NIES RM No. 1003 Fish Otolith

This environmental reference material (RM) was developed by the National Institute for Environmental Studies (NIES), Japan, using *Lutjanus sebae* caught off the northwest coast of Western Australia. It is intended for use in quality control and for improving the analytical precision of elemental analysis in calcium carbonate matrices, such as fish otoliths and similar marine aragonite matrices. This RM was prepared from the same raw material as NIES CRM No. 22 Fish Otolith, which was produced in 2000.

Reference Value

Element	Mass fraction			Analytical method *
	Unit	Reference value	Uncertainty	
Calcium (Ca)	%	39.0	0.6	ICP-OES, ICP-MS
Sodium (Na)	%	0.224	0.005	ICP-OES, FAAS
Strontium (Sr)	%	0.233	0.008	ICP-OES, ICP-MS
Barium (Ba)	mg/kg	2.89	0.09	ICP-MS
Magnesium (Mg)	mg/kg	20.9	0.8	ICP-OES, ICP-MS
Potassium (K)	mg/kg	281	17	ICP-OES, FAAS

The reference value was determined based on dry mass.

* FAAS, flame atomic absorption spectroscopy
ICP-MS, inductively coupled plasma-mass spectrometry
ICP-OES, inductively coupled plasma-optical emission spectrometry

Characterization

The property values of the material were statistically determined based on chemical analyses by 3 organizations (including 6 laboratories) using a wide range of methods. The uncertainty associated with the reference value is the expanded uncertainty using a coverage factor $k = 2$, corresponding to the half-width of a confidence interval of approximately 95 %. This reference value is used as a reference value since it does not meet the NIES certification criteria.

Description of the Material

The RM is supplied as fine white powder in a glass bottle.

Preparation of the RM

This RM was developed using the same raw material as NIES CRM No. 22 Fish otolith produced in 2000. The raw material for this RM 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 homogenized. The powdered samples were packed 3 g each in 375 glass bottles. Two hundred of the 375 bottles were distributed as NIES CRM No. 22, and the remaining 175 bottles were stored at room temperature (10 to 30 °C). This reference material consists of these 175 bottles.

Homogeneity

Homogeneity tests were carried out on 5 sample bottles selected by stratified random sampling. The between-bottle variation evaluated by a one-way analysis of variance (ANOVA) showed the homogeneity standard deviations between bottles for the analytes to be 2.0 % for K and less than 1 % for other elements. The material, therefore, is sufficiently homogeneous for its intended use as a reference material.

Stability

Stability tests over a period of 25 years demonstrated that any long-term variations in the values of e trace elements in the material were insignificant.

Instructions for Use

1. This RM should be kept tightly closed in its original bottle and stored in a desiccator at room temperature (≤30 °C).
2. Prior to weighing portions for analysis, the contents of the bottle should be shaken gently.
3. The mass fraction of the reference value in this RM is reported on a dry mass basis. The moisture content of this RM is approximately 0.3% (dried at 85 °C for 4 hours). Correction to dry mass should be made by measuring the moisture content after each analysis.
4. For convenient handling, a minimum sample intake of 100 mg is recommended.
5. 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.
6. Do not use for purposes other than research. When disposing of samples, adhere strictly to local laws concerning processing and disposal of waste materials.

Expiry Date

The expiry date for the reference value of this RM is June 2035, assuming that above mentioned storage conditions are adhered to. NIES will announce via its website if any changes in the contents are noticed within the term of validity.

Technical Information

Technical information and the latest reports regarding this material can be obtained from the website.
http://www.nies.go.jp/labo/crm-e/index.html

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Appendix

Information values may be useful for handling this material, though the values are not certified.

Trace elements

The trace element contents of NIES RM No.1003 Fish otolith were statistically determined based on chemical analyses by 3 organizations (including 6 laboratories). The trace element contents in NIES RM No.1003 Fish otolith were measured by inductively coupled plasma mass spectrometer (ICP-MS; Agilent 8800, Agilent Technologies) at NIES (Table A1). Acid digestion using a hot plate was employed as a pretreatment method.

Table A1 Trace element contents in NIES RM No.1003 Fish otolith

Element	Mass fraction		Analytical method *
	Unit	Content	
Cadmium (Cd)	mg/kg	0.0025	ICP-MS
Copper (Cu)	mg/kg	0.72	ICP-MS
Lead (Pb)	mg/kg	0.022	ICP-MS
Zinc (Zn)	mg/kg	0.49	ICP-MS

All values were determined based on dry mass.

* ICP-MS, inductively coupled plasma-mass spectrometry