

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 6206-a
No. +++

Okadaic Acid Standard Solution

This certified reference material (CRM), okadaic acid solution (dissolved in methanol containing 0.5% (volume fraction) ethanol), is produced in accordance with the NMIJ's management system and is in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in the preparation of standard solutions for the determination of okadaic acid in diarrhetic shellfish toxin testing.

Certified Value

The certified value for the mass concentration of okadaic acid at 25 °C is given in the table below. The uncertainty of the certified value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Compound	CAS No.	Certified value, Mass concentration ($\mu\text{g/mL}$)	Expanded uncertainty Mass concentration ($\mu\text{g/mL}$)
Okadaic Acid	78111-17-8	0.909	0.073

Analysis

This CRM was prepared by the gravimetric blending of the solvent (0.5 % (volume fraction) ethanol in methanol) and okadaic acid solution, in which the mass fraction of okadaic acid was determined by a quantitative NMR method. The mass concentration is obtained from the mass fraction of okadaic acid and the density of the diluted solution. The standard uncertainties due to NMR measurement, dilution, homogeneity, stability and the temperature-related variation in density are combined. The uncertainty of the quantitative NMR method was estimated from a collaborative study, and is included in the uncertainty of NMR measurement.

Metrological Traceability

In the quantitative NMR method, a 1,4-bis(trimethylsilyl)benzene solution in methanol, calibrated using 3,5-bis(trifluoromethyl)benzoic acid (certified reference material (NMIJ CRM 4601-a)), was used as an internal standard. A Japan Calibration Service System (JCSS) calibrated balance was used for gravimetric dilution of the okadaic acid solution. The certified value is traceable to the International System of Units (SI).

Indicative Values

The indicative values for mass fraction and amount of substance concentration of okadaic acid at 25 °C are given in the table below. The uncertainty of the indicative value is the half-width of the expanded uncertainty interval calculated using a coverage factor (k) of 2, which gives a level of confidence of approximately 95 %.

Compound		Indicative value	Expanded uncertainty
Okadaic Acid	Mass fraction	1.155 $\mu\text{g/g}$	0.093 $\mu\text{g/g}$
	Amount of substance concentration	1.129 $\mu\text{mol/L}$	0.091 $\mu\text{mol/L}$

Expiration of Certification

This certificate is valid for one year from the date of shipment, provided that the material remains unopened and is stored in

accordance with the instructions given in this certificate.

Sample Form

This CRM is in the form of a colorless clear liquid. This CRM of ca. 1 mL in net volume is kept in an amber ampule.

Homogeneity

The homogeneity of this CRM was determined by analyzing 10 ampules selected from among 522 ampules by stratified random sampling in order of subdivision. The homogeneity of this CRM was evaluated from the peak area of okadaic acid by HPLC-UV. The homogeneity is reflected in the uncertainty of the certified value.

Instructions for Storage

This CRM should be stored below $-20\text{ }^{\circ}\text{C}$ and shielded from light.

Instructions for Use

This CRM should be used between $20\text{ }^{\circ}\text{C}$ and $30\text{ }^{\circ}\text{C}$. The ampule of this CRM should be warmed to between $20\text{ }^{\circ}\text{C}$ and $30\text{ }^{\circ}\text{C}$ before opening and used promptly once the ampule is opened. Evaporation of methanol should be avoided. This CRM is for laboratory use only.

Precautions for Handling

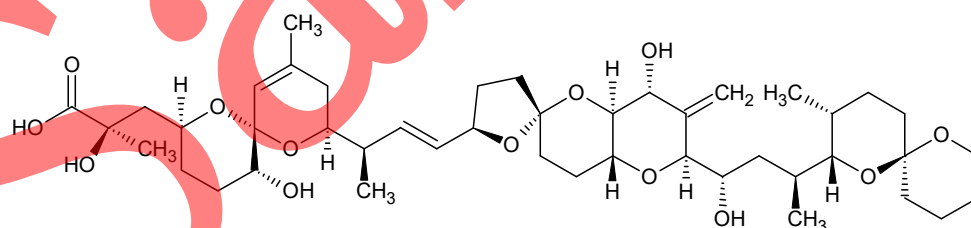
Wear a mask, gloves, and other protective equipment during handling. Be careful to avoid injury when opening the glass ampoule. Follow local rules or regulations for the disposal of this CRM or diluted solutions. Refer to the safety data sheet (SDS) on this CRM before use.

Preparation

Okadaic acid was produced by a large culture of the toxic dinoflagellate *Prorocentrum lima*, and isolated by liquid-liquid partitioning and several column chromatography steps at National Research Institute of Fisheries Science (NRIFS). The CRM solution was prepared by dissolving purified okadaic acid in methanol containing ethanol and dispensed into amber glass ampoules at NMIJ.

Technical Information

The density of this CRM is 0.7871 g/cm^3 at $25\text{ }^{\circ}\text{C}$ and it will change by approximately 0.6 % for a $5\text{ }^{\circ}\text{C}$ change in temperature. The molar mass of okadaic acid is 805.00 g/mol . The structure of okadaic acid is shown below.



NMIJ Analysts

The technical and production managers for this CRM are TAKATSU A.; and the analysts are YAMAZAKI T., KAWAGUCHI M. and EYAMA S.

Information

If substantive technical changes occur that affect the certification before the expiration of this certificate, NMIJ will notify the registered customer. Customer registration on the NMIJ Website (given below) will facilitate notification. Technical reports regarding this CRM can be obtained from the contact details given below.

Reproduction of Certificate

In reproducing this certificate, it should be clearly indicated that the document is a copy.

Note

This CRM was developed in collaboration with National Research Institute of Fisheries Science (NRIFS), with the support of a grant from Cross-ministerial Strategic Innovation Promotion Program (SIP), “Technologies for creating next-generation agriculture, forestry and fisheries” (funding agency: Bio-oriented Technology Research Advancement Institution).

Production of this CRM was based on the notable research achievements made by Prof. YASUMOTO Takeshi in the discovery of diarrhetic shellfish toxins and the production of their standards.

April 1, 2020

ISHIMURA Kazuhiko
President

National Institute of Advanced Industrial Science and Technology

If you have any questions about this CRM, please contact:
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