

National Institute of Advanced Industrial Science and Technology

National Metrology Institute of Japan



Reference Material Certificate

NMIJ CRM 7520-a
No. +++

Diarrhetic Shellfish Toxins in Scallop Midgut Gland

This certified reference material (CRM) was produced in accordance with the NMIJ's management system and in compliance with ISO 17034 and ISO/IEC 17025. This CRM is intended for use in the accuracy control of analyses and the validation of analytical techniques for the determination of diarrhetic shellfish toxins in scallop midgut gland and similar materials in accordance with Notice issued by the Ministry of Health, Labour and Welfare, Japan (Shoku-An-Ki 030603 and Shoku-An-Kan 030601, 2015).

Certified Values

The certified values of this CRM are given in the table below. The uncertainty of the certified value is the expanded uncertainty obtained by multiplying the combined standard uncertainty by a coverage factor (k) of 2, and it is the half-width of an interval estimated to have a level of confidence of approximately 95 %.

Compounds	CAS No.	Certified value Mass fraction (mg/kg)	Expanded uncertainty Mass fraction (mg/kg)
Okadaic acid	78111-17-8	0.205	0.080
Dinophysistoxin-1 (DTX1)	81720-10-7	0.45	0.16

Analysis

The certified values of this CRM were obtained based on the results of the analyses conducted by the nine participants (listed in Note) of the interlaboratory certification study. Each participant employed an analytical method, following the performance criteria stipulated in Appendix of Notice issued by the Ministry of Health, Labour and Welfare, Japan (Shoku-An-Ki 030603 and Shoku-An-Kan 030601, 2015), and quantified okadaic acid and DTX1 by using a standard addition method approach.

The outline of the analytical method exemplified in Annex 2 of this Notice is as follows:

[Preparation] extraction using methanol and 90 % methanol, hydrolysis by adding sodium hydroxide, washing with hexane, and clean-up by solid-phase extraction using an octadecylsilyl-silica gel (ODS) cartridge

[Liquid chromatography-tandem mass spectrometric conditions] separation column: ODS column; ionization method: electrospray ionization; ion mode: negative, detection: selected reaction monitoring

Metrological Traceability

The certified values of this CRM are the mass fractions of okadaic acid and DTX1 quantified by the analytical method described in Analysis. The standard solution used for the interlaboratory certification study was prepared gravimetrically from an okadaic acid solution and a DTX1 solution; these solutions were calibrated with NMIJ CRM 6206-a (Okadaic Acid Standard Solution) and NMIJ CRM 6207-a (Dinophysistoxin-1 (DTX1) Standard Solution), respectively. The certified values, therefore, are traceable to the International System of Units (SI).

Expiration of Certification

This certificate is valid for six months from the date of shipment, provided that the CRM remains unopened and is stored in accordance with the instructions given in this certificate.

Description of the Material

This CRM is the midgut glands of boiled scallops in the form of ocher paste at room temperature, and 10 g of this CRM in net volume is packaged in a plastic vial. The vial is then sealed in an aluminum-laminated plastic bag.

Instructions for Storage

This CRM should be stored at a temperature of $-30\text{ }^{\circ}\text{C}$ to $-20\text{ }^{\circ}\text{C}$ and protected from sunlight.

Instructions for Use

A vial of this CRM should be allowed to warm to room temperature of $15\text{ }^{\circ}\text{C}$ to $25\text{ }^{\circ}\text{C}$ for 8 to 15 hours before opening to thaw the CRM completely. This CRM should be used promptly once a vial is opened. This CRM should be well mixed for approximately 1 min, by using a spatula for instance, before it is sampled. More than 0.5 g of the material should be sampled for each analysis.

Precautions for Handling

A protective mask, gloves, and other protective gears should be used for safety when this CRM is used. Refer to the safety data sheet (SDS) on this CRM before use. This CRM must be disposed of in accordance with all relevant laws regarding waste handling and management.

Preparation

The midgut glands used as the raw material of this CRM were obtained from both toxic and non-toxic boiled scallops. These raw materials were processed to be homogenized. After the spiking of okadaic acid, they were mixed, bottled, and sterilized by the γ -ray irradiation (20 kGy). The okadaic acid used was prepared by National Research Institute of Fisheries Science (NRIFS), using a large culture of the toxic dinoflagellate *Prorocentrum lima*. Part of the preparation of the CRM was performed by the General Environmental Technos Co., Ltd under the contract with NMIJ.

NMIJ Analysts

The technical manager and production manager for this CRM is YARITA T., and the analysts are YARITA T., MIYAMOTO A., INAGAKI S., YAMAZAKI T., KAWAGUCHI M., SAKAMOTO T., and AOYAGI Y.

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 provided by Cross-ministerial Strategic Innovation Promotion Program (SIP), “Technologies for creating next-generation agriculture, forestry and fisheries” (the funding agency: Bio-oriented Technology Research Advancement Institution).

The following institutes participated in the interlaboratory certification study: National Metrology Institute of Japan (NMIJ), National Research Institute of Fisheries Science (NRIFS), National Institute of Health Sciences (NIHS), Food and Agricultural Materials Inspection Center (FAMIC), Kanagawa Prefectural Institute of Public Health, Japan Food Research Laboratories (JFRL), Japan Frozen Foods Inspection Corporation (JFFIC), Aomori Pharmaceutical Association Sanitary Inspection Center, and Shimadzu Corporation (listed in random order).

The midgut glands of scallops used as the raw material of this CRM were kindly provided by Aomori Prefecture Scallop Marketing Promotion Association Japan.

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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Revision history

Mar. 23, 2022: Uncertainty of “Certified value” of Okadaic acid and DTX1 were changed.