

National Institute of Advanced Industrial Science and Technology (AIST)  
Japan Fisheries Research and Education Agency (FRA)

**Development of certified reference materials for shellfish toxins that cause diarrhetic shellfish poisoning  
- Contribution to highly reliable shellfish-poison testing using analytical instruments -**

- We developed and will start providing certified reference materials for liquid chromatography-mass spectrometric analysis of shellfish toxins that cause diarrhetic shellfish poisoning.
- We developed a method for determining the concentration of okadaic acids using quantitative nuclear magnetic resonance analysis.
- The method will contribute to the implementation and widespread use of reliable and instrument-based shellfish-poison testing.

#### Overview

The National Institute of Advanced Industrial Science and Technology (AIST), in collaboration with the Japan Fisheries Research and Education Agency (FRA), developed certified reference materials for okadaic acid group (okadaic acid and dinophysistoxin-1) to be used in analysis of shellfish toxins using liquid chromatography-mass spectrometry instruments.

These certified reference materials were developed by preparing highly purified okadaic acid and dinophysistoxin-1 using the technologies for purifying shellfish poisons and for mass culture of algae producing such poisons currently being developed by FRA. The AIST prepared solutions using highly purified okadaic acids. The concentration of the solutions was accurately determined using a quantitative analysis method for organic compounds based on nuclear magnetic resonance (quantitative NMR analysis), which was developed and being promoted for widespread use primarily by AIST. The newly developed certified reference materials can be used as standard solutions for accurately determining concentrations based on analytical instrument signals in the testing for okadaic acids causing diarrhetic shellfish poisoning

The use of analytical instruments in testing for okadaic acid groups causing diarrhetic shellfish poisoning is also being promoted in other countries. In Japan, the Ministry of Health, Labour and Welfare has announced the use of analytical instruments for okadaic acid testing. The availability of certified reference materials for okadaic acid and dinophysistoxin-1 at highly accurate concentrations is expected to promote the implementation and widespread use of reliable, instrument-based shellfish poison testing within Japan.

These certified reference materials will be provided through a subcontractor starting April 6, 2016 at the following specifications:

Name of standard substance	Okadaic acid standard solution	Dinophysistoxin-1 standard solution
Standard substance number	NMIJ CRM 6206-a	NMIJ CRM 6207-a
Concentration	0.909 $\mu\text{g/mL} \pm 0.073 \mu\text{g/mL}$	1.079 $\mu\text{g/mL} \pm 0.078 \mu\text{g/mL}$
Solvent	Ethanol-containing methanol (0.5% ethanol by volume)	
Volume	1 mL	
Container	Brown-colored glass ampule	



Figure 1. Certified reference materials for shellfish-poison testing  
Okadaic acid standard (left), Dinophysistoxin-1 standard (right) solutions