Quasimeme Laboratory Performance Studies

Round 2017 - 2

2 October 2017 to 1 February 2018

Exercise Protocols

Version 1: 26 September 2017

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Introduction Round 2017 - 2

Thank you for participating in the 2017 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round 2017-2, that you have ordered will be sent to you by courier in the week of 2 October 2017. Please check that the contents of your package are correct and that all test materials are intact. If any test materials have been damaged in transit or if the wrong samples have been send, use the form in Annex 1 of this document to request replacement materials within two weeks after receipt of the test materials.

Additional test materials may also be purchased from QUASIMEME.

This protocol covers the following studies:

Round	Analysis Group Code	Matrix	Analytes
2017 - 2	AQ-1	Seawater	Nutrients
2017 - 2	AQ-2	Estuarine and Low Salinity Open Water	Nutrients
2017 - 2	AQ-3	Seawater	Metals
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2017 - 2	MS-3	Sediment	Polycyclic Aromatic Hydrocarbons
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2017 - 2	MS-7	Sediment	Brominated Flame Retardants
2017 - 2	BT-1	Biota	Trace Metals
2017 - 2	BT-2	Biota	Chlorinated Organics
2017 - 2	BT-4	Biota	Polycyclic Aromatic Hydrocarbons
2017 - 2	BT-8	Biota	Organotins
2017 - 2	BT-9	Biota	Brominated Flame Retardants
2017 - 2	BT-10	Biota	Perfluorinated Alkyl Substances (PFASs)
2017 - 2	BT-7	Shellfish and Solution	ASP Shellfish Toxins
2017 - 2	BT-11	Shellfish and Solution	DSP Shellfish Toxins

All data for these studies must be uploaded to your Quasimeme SharePoint Site, using the Data Submission Forms, no later than **1st February 2018**.

IMPORTANT: Please note that the Data Submission Forms are changed, report ONLY the parameters as they are asked in this protocol

All other information should be sent to: QUASIMEME Project Office

Wageningen University & Research WEPAL-QUASIMEME Project Office P.O. Box 8005 6700 EC Wageningen The Netherlands Bornesesteeg 10 6721 NG Bennekom The Netherlands	Website: http://www.Quasimeme.org Tel.: +31 (0) 317 48 65 46 Fax: +31 (0) 317 48 56 66 E-mail: quasimeme@wur.nl

ROUND	2017-2
AQ-1 Nutrients in Seawater	
Test materials	QNU312SW, QNU313SW and QNU314SW

Objective

This study covers the determination of nutrients in the seawater test materials.

Test Materials and storage

The test materials were prepared at the laboratory of RBINS (Royal Belgium Institute of Natural Sciences), Ostend, Belgium, using seawater collected from the North Sea between Belgium and the UK.

The seawater was filtered using a 0.45µm / 0.2µm double membrane filter. The pH of the filtered seawater was adjusted to ~ pH 7.2 with 0.1M hydrochloric acid and spiked to appropriate concentrations. The spiked seawater is then thoroughly mixed before being dispensed into the glass and plastic bottles. The filled bottles are then autoclaved at 110˚C, 1.5 bar for 30 minutes. The autoclaving process removes micro-organisms, which affect the stability of the nutrient test materials. It has been demonstrated that autoclaving the test materials generates an increase in pH. We have found that after autoclaving, the pH of the nutrient test materials is within the range pH 7.5 to 8.5.

The three test materials differ from each other in respect of their nutrient concentrations.

There are two bottles for each test material - one glass and one plastic. The glass bottle should only be used for the determination of TOxN, nitrite, ammonia and total-N. The plastic bottle should only be used for the determination of silicate, phosphate and total-P. Each bottle contains approximately 250 ml of the test material .

There is a separate bottle for the determination of salinity labelled Salinity ONLY (QNU312SW). Salinity should NOT be measured and/or reported for both other samples.

Each batch of material was prepared in bulk. Homogeneity testing is performed on each batch of test materials produced. The nutrient test materials are stable for the period of the test, and have also been shown to be stable for a period of some months, even after opening, if used under the correct conditions.

Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

Treat all test materials in the same manner as your routine samples.

Samplecode	Description
QNU312SW	Seawater (Salinity > 30 psu)
QNU313SW	Seawater (Salinity > 30 psu) spiked
QNU314SW	Seawater (Salinity > 30 psu) spiked

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following nutrients should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Seawater	Seawater (spiked)	Const	Prop	
Ammonia	µmol/L	0.2—5	0.2—5	0.1	6.0%	
Nitrite	µmol/L	0.01—2	0.01—2	0.01	6.0%	
Phosphate	µmol/L	0.05—5	0.05—5	0.05	6.0%	
Silicate	µmol/L	0.5—10	0.5—10	0.1	6.0%	
Total-N	µmol/L	5—25	5—25	0.5	6.0%	
Total-P	µmol/L	0.1—5	0.1—5	0.05	6.0%	
TOxN	µmol/L	0.05—15	0.05—15	0.05	6.0%	
Salinity	psu			0.01	0.1%	

Please report Salinity only for QNU312SW

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received”. The concentration of nutrients should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017 -2
AQ-2 Nutrients in Estuarine and Low Salinity Open Seawater	
Test materials	QNU315EW, QNU316EW, QNU317EW and QNU318EW

Objective

This study covers the determination of nutrients in estuarine water test materials and low salinity open water test materials.

Test Materials and storage

The test materials were prepared at the laboratory of RBINS (Royal Belgium Institute of Natural Sciences), Ostend, Belgium, using seawater collected from the North Sea between Belgium and the UK, and from the Baltic Sea (low salinity water samples)

The seawater was filtered using a 0.45µm / 0.2µm double-membrane filter and diluted with ultrapure demineralised water. The pH of the filtered seawater was adjusted to ~pH7.2 with 0.1M hydrochloric acid and spiked to appropriate concentrations. The spiked seawater is then thoroughly mixed before being dispensed into the glass and plastic bottles. The filled bottles are then autoclaved at 110°C, 1.5 bar for 30 minutes. The autoclaving process removes micro-organisms, which affect the stability of the nutrient test materials. It has been demonstrated that autoclaving the test materials generates an increase in pH. We have found that after autoclaving, the pH of the nutrient test materials is within the range pH 7.5 to 8.5. The four test materials differ from each other in respect of their nutrient concentrations and the salinity of the water. The salinity of the water will be approximately 8-20 psu. One of the samples is the unspiked sample and the other samples are spiked with nutrients.

There are two bottles for each test material - one glass and one plastic. The glass bottle should only be used for the determination of TOxN, nitrite, ammonia and total-N. The plastic bottle should only be used for the determination of silicate, phosphate and total-P.

There is a separate bottle for the determination of salinity labelled Salinity ONLY (QNU315EW). Salinity should NOT be measured and/or reported for the other samples.

Test materials should be stored in a refrigerator at +4°C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

Treat all test materials in the same manner as your routine samples.

Samplecode	Description
QNU315EW	Estuarine water (Salinity 8 - 15 psu) spiked
QNU316EW	Estuarine water (Salinity 8 - 15 psu) spiked
QNU317EW	Low salinity seawater (Salinity 10 - 20 psu) spiked
QNU318EW	Unspiked Low salinity seawater (Salinity 10 - 20 psu)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following nutrients should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Estuarine water (spiked)	Low salinity open water (spiked)	Const	Prop	
Ammonia	µmol/L	2—50	0.2—5	0.1	6.0%	
Nitrite	µmol/L	0.5—25	0.01—2	0.01	6.0%	
Phosphate	µmol/L	1—15	0.02—5	0.05	6.0%	
Salinity	psu			0.01	0.1%	
Silicate	µmol/L	5—100	0.5—20	0.1	6.0%	
Total-N	µmol/L	10—200	2—20	0.5	6.0%	
Total-P	µmol/L	1—20	0.02—2	0.05	6.0%	
TOxN	µmol/L	10—100	0.05—15	0.05	6.0%	

Please report Salinity only for QNU315EW

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result for the different nutrients per test material is required. The results should be expressed on the test material “as received”. The concentration of the nutrients should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
AQ-14 DOC in seawater	
Test materials	QDC053SW, QDC054SW, QDC055EW and QDC056EW

Objective

This study covers the determination of DOC in open seawater and estuarine water test materials.

Test Materials and storage

The test materials were prepared at the laboratory of RBINS (Royal Belgium Institute of Natural Sciences), Ostend, Belgium, using seawater collected from the North Sea between Belgium and the UK.

The seawater was filtered using a 0.45µm / 0.2µm double-membrane filter and diluted with ultrapure demineralised water for the estuarial samples. The pH of the filtered seawater was adjusted to ~pH7.2 with 0.1M hydrochloric acid and spiked to appropriate concentrations. The spiked seawater is then thoroughly mixed before being dispensed into the glass bottles. The filled bottles are then autoclaved at 110˚C, 1.5 bar for 30 minutes. The autoclaving process removes micro-organisms, which affect the stability of the DOC test materials. It has been demonstrated that autoclaving the test materials generates an increase in pH. We have found that after autoclaving, the pH of the DOC test materials is within the range pH 7.5 to 8.5. The four test materials differ from each other in respect of their DOC concentrations and the salinity of the water. The salinity of the estuarine seawater will be approximately 7 - 10 psu. One of the samples is the unspiked sample and the other samples are spiked with DOC.

Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QDC053SW	Seawater (Salinity > 30 psu)
QDC054SW	Seawater (Salinity > 30 psu) spiked
QDC055EW	Estuarine water (Salinity 7 - 10 psu) spiked
QDC056EW	Estuarine water (Salinity 7 - 10 psu) spiked

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following nutrients should be determined:

Determinand	Unit	Concentration range			Error		AA-EQS
		Seawater	Seawater (spiked)	Estuarine water (spiked)	Const	Prop	
DOC	mg C/L	0.1—20	0.1—20	0.1—20	0.1	6.0%	

Analysis

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result for DOC test material is required. The result should be expressed on the test material "as received". The concentration of DOC should be determined against your own calibration solutions.

Reporting

The result for DOC should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
AQ-3 Metals in Seawater	
Test materials	QTM255SW, QTM256SW, QTM257SW and QTM258SW

Objective

This study covers the determination of metals in seawater and low salinity seawater test materials.

Test Materials and storage

The test materials were prepared at Wageningen Environmental Research, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK.

The test materials were prepared in bulk in a 50 litre vessel. The seawater was filtered using a 0.45 µm /0.2µm double-membrane filter. Low salinity seawater test material was prepared by diluting the seawater with ultra-pure demineralised water. All test materials are preserved with 2 ml trace metal grade nitric acid per litre of seawater. Spiked test materials were prepared by adding aqueous solutions of known trace metal concentration. Approximately 1 litre of each test material is provided. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre polyethylene bottles. The test materials are stable for the purposes of the exercise.

Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

Samplecode	Description
QTM255SW	Unspiked Seawater (Salinity > 30 psu)
QTM256SW	Seawater (Salinity > 30 psu) spiked with metals
QTM257SW	Low salinity Seawater (Salinity 8 - 20 psu) spiked with metals
QTM258SW	Low salinity Seawater (Salinity 8 -20 psu) sample spiked with concentrations between 5 and 100 times higher than the indicative range (500 ml)

Precaution

Some of the substances may presents a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Analysis

Treat all test materials in the same manner as your routine samples.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result per determinand per test material is required.

Determinands and concentration ranges

The following metals should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Seawater (spiked)	Low salinity Seawater (spiked)	Const	Prop	
Arsenic	µg/L	0.05—5	0.2—10	0.5	12.5%	
Boron	µg/L	1000—5000	200—5000	0.4	12.5%	
Cadmium	µg/L	0.001—0.5	0.05—1	0.005	12.5%	0.2
Chromium	µg/L	0.01—5	0.5—10	0.1	12.5%	
Cobalt	µg/L	0.001—0.5	0.01—10	0.2	12.5%	
Copper	µg/L	0.05—10	0.2—10	0.2	12.5%	
Iron	µg/L	0.05—10	0.2—20	0.4	12.5%	
Lead	µg/L	0.0002—15	0.1—5	0.01	12.5%	7.2
Manganese	µg/L	0.02—5	0.1—5	0.4	12.5%	
Nickel	µg/L	0.2—5	0.1—5	0.2	12.5%	20
Silver	µg/L	0.02—2	0.1—5	0.2	12.5%	
Tin	µg/L	0.02—1	0.1—5	0.2	12.5%	
Vanadium	µg/L	0.1—5	0.2—5	0.2	12.5%	
Zinc	µg/L	0.5—20	0.2—20	0.4	12.5%	

QTM258SW contains concentrations of the determinands which are 5 to 50 times higher compared to the indicative range given in this protocol.

Boron is naturally occurring at higher concentrations.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
AQ-4 Mercury in Seawater	
Test materials	QTM259SW, QTM260SW, QTM261SW and QTM262SW

Objective

This study covers the determination of mercury in the seawater test materials. The test materials should be analysed and one result for mercury in each test material should be reported using the Data Submission Forms provided on the Participant Site.

Test Materials and storage

The test materials were prepared at Wageningen Environmental Research, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK.

The test materials were prepared in bulk in a 50 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. All test materials are preserved with 2 ml trace metal grade nitric acid per litre of seawater. Test materials were spiked with aqueous solutions of known mercury concentration.

Approximately 1 litre of each test material is provided.

Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre glass bottles. The test materials are stable for the purposes of the exercise. Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

Samplecode	Description
QTM259SW	Seawater (Salinity > 30 psu) spiked with mercury
QTM260SW	Seawater (Salinity > 30 psu) spiked with mercury
QTM261SW	Low salinity Seawater (Salinity 10 - 20 psu) spiked with mercury
QTM262SW	Low salinity Seawater (Salinity 10 - 20 psu) spiked sample with concentrations between 5 and 50 times higher compared to the concentrations given in this protocol

Precaution

Some of the substances may presents a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

Mercury should be determined in each test material.

Determinand	Unit	Concentration range	Error		AA-EQS
		Seawater (spiked)	Const	Prop	
Mercury	ng/L	0.1—100	0.2	12.5%	50

QTM262SW contains concentrations of the determinands which are 5 to 50 times higher compared to the indicative range given in this protocol.

Analysis

Treat all test materials in the same manner as your routine samples.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Only one result per test material is required.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site.

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
AQ-11 Chlorophyll-a in Seawater	
Test materials	QCH088SW and QCH089SW

Objective

This study covers the determination of chlorophyll a, b, c and pheopigments in filtered seawater residue test materials.

Test Materials and storage

The test materials for the analysis of chlorophyll a, b, c and pheopigments were prepared at Wageningen Environmental Research the Netherlands. Test materials were prepared unspiked freshwater (QCH088SW) and seawater (QCH089SW). For each test material, the resultant damp filter paper (Whatman GF/F) was wrapped in aluminium foil, inserted into cryovial and immediately 'flash frozen' in liquid nitrogen. The test materials were stored at -80˚C until the day of dispatch. The test materials were homogeneous for the purposes of the LP study.

The filter papers have been shipped on cool packs, and should be stored at -20˚C, or a lower temperature, immediately upon receipt, and should be analysed as soon as possible after receipt. The cool packs will thaw slowly during transit, but this does not significantly affect the stability of the material for the purpose of this study, providing the test materials themselves are frozen immediately on receipt.

Sample Code	Description
QCH088SW	Filtered residue from 1 litre of freshwater
QCH089SW	Filtered residue from 1 litre of seawater

Precaution

Some of the substances may presents a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following pigments should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Filtered residues	Const	Prop	
Chlorophyll-a	µg/L	0.1—20	0.05	12.5%	
Chlorophyll-b	µg/L	0.01—5	0.01	12.5%	
Chlorophyll-c	µg/L	0.02—2.5	0.01	12.5%	
Pheopigments	µg/L	0.02—2.5	0.01	12.5%	
Chlorophyll-a (HPLC)	µg/L	0.1—20	0.05	12.5%	
Chlorophyll-b (HPLC)	µg/L	0.01—5	0.01	12.5%	
Chlorophyll-c (HPLC)	µg/L	0.02—2.5	0.01	12.5%	
Chlorophyll-a (corrected)	µg/L	0.1—20	0.05	12.5%	

Chlorophyll-a (corrected) should only be used to report chlorophyll-a concentrations which are corrected for Phaeophytin.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

Treat all test materials in the same manner as your routine samples. Use your normal validated methods and procedures to analyse the test materials. Only one result per determinand per test material is required. The results of each determinand should be expressed on the test materials "as received". **Concentrations need to be calculated based on a filter prepared out of a 1 litre sample.**

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site.

Chlorophyll-a (corrected) should only be used to report chlorophyll-a concentrations which are corrected for Phaeophytin.

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

N.B. Laboratories using HPLC related methods should report their results separately as well as labs who correct their results of Chlorophyll-a for Phaeophytine.

Please report your Method information together with the results.

ROUND	2017-2
MS-1 Trace metals in Sediment	
Test materials	QTM120MS and QTM121MS

Objective

This study covers the determination of metals, total organic carbon and carbonate in marine sediment test materials.

Test Materials and storage

Test materials were prepared by WEPAL, Wageningen, The Netherlands.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the sediment was determined. All materials have been shown to be homogeneous at or below the intake mass used by the participants, and stable for the purposes of the test.

The test materials should be stored at room temperature prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QTM120MS	Sediment (river)
QTM121MS	Sediment (harbor)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following trace metals should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
Aluminium-AE	%	1—10	0.1	12.5%	
Aluminium-RT	%	1—10	0.1	12.5%	
Arsenic-AE	mg/kg	2—50	1	12.5%	
Arsenic-RT	mg/kg	2—50	1	12.5%	
Cadmium-AE	µg/kg	10—2000	20	12.5%	
Cadmium-RT	µg/kg	10—2000	20	12.5%	
Chromium-AE	mg/kg	10—1000	2	12.5%	
Chromium-RT	mg/kg	10—1000	2	12.5%	
Copper-AE	mg/kg	1—500	1	12.5%	
Copper-RT	mg/kg	1—500	1	12.5%	
Inorganic-carbonate	%	0.05—10	0.05	12.5%	
Iron-AE	%	0.5—10	0.1	12.5%	
Iron-RT	%	0.5—10	0.1	12.5%	
Lead-AE	mg/kg	5—500	2	12.5%	
Lead-RT	mg/kg	5—500	2	12.5%	
Lithium-AE	mg/kg	10—100	0.1	12.5%	
Lithium-RT	mg/kg	10—100	0.1	12.5%	
Manganese-AE	mg/kg	100—2000	0.1	12.5%	
Manganese-RT	mg/kg	100—2000	0.1	12.5%	
Mercury-AE	µg/kg	50—2500	10	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
Mercury-RT	µg/kg	50—2500	10	12.5%	
Nickel-AE	mg/kg	5—100	1	12.5%	
Nickel-RT	mg/kg	5—100	1	12.5%	
Scandium-AE	mg/kg	1—20	0.1	12.5%	
Scandium-RT	mg/kg	1—20	0.1	12.5%	
TOC	%	0.2—10	0.1	12.5%	
Zinc-AE	mg/kg	20—1500	2.5	12.5%	
Zinc-RT	mg/kg	20—1500	2.5	12.5%	

RT = Real Total destructions e.g. HF-destruction, RÖntgen-diffraction and neutron activation
AE= Acid extractable and all other methods

Total organic carbon and inorganic carbonate can be determined for both test materials. Please note that inorganic carbonate should be reported as % carbon

In addition to the parameters given in the table you will be able to report the following metals: Na, Mg, P, S, K, Ca, Ti, V, Co, Ga, Rb, Se, Sr, Mo, Sn, Cs, Ba, Ce, Ta, Tl, Th, U.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

The sediments have been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator. It is left to the discretion of the laboratory to dry the material. You are advised to check the moisture content of the sediment by drying a small portion of the sediment to constant weight at ~ 110˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material "as received".

The concentration of metals should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
MS-2 Chlorinated Organics in Sediment	
Test materials	QOR132MS and QOR133MS

Objective

This study covers the determination of chlorobiphenyls (CBs), organochlorine pesticides (OCPs) and total organic carbon in marine sediment test materials.

Test Materials and storage

Test materials were prepared by WEPAL, Wageningen, The Netherlands.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the sediment was determined. All materials have been shown to be homogeneous at or below the intake mass used by the participants, and stable for the purposes of the test.

The test materials should be stored at room temperature prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QOR132MS	Sediment (open sea)
QOR133MS	Sediment (harbor)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following Chlorinated Organics should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
a-HCH	µg/kg	0.02—1	0.02	12.5%	
b-HCH	µg/kg	0.05—2	0.025	12.5%	
CB101	µg/kg	0.2—50	0.025	12.5%	
CB105	µg/kg	0.1—50	0.025	12.5%	
CB118	µg/kg	0.1—200	0.025	12.5%	
CB138	µg/kg	0.2—50	0.025	12.5%	
CB138+CB163	µg/kg	0.2—50	0.025	12.5%	
CB153	µg/kg	0.2—50	0.025	12.5%	
CB156	µg/kg	0.05—5	0.025	12.5%	
CB180	µg/kg	0.1—50	0.025	12.5%	
CB28	µg/kg	0.1—50	0.025	12.5%	
CB31	µg/kg	0.1—50	0.025	12.5%	
CB52	µg/kg	0.1—500	0.025	12.5%	
d-HCH	µg/kg	0.05—2	0.025	12.5%	
Dieldrin	µg/kg	0.1—10	0.025	12.5%	
g-HCH	µg/kg	0.05—2	0.025	12.5%	
HCB	µg/kg	0.05—250	0.025	12.5%	
HCBD	µg/kg	0.1—10	0.025	12.5%	
op'-DDT	µg/kg	0.02—250	0.025	12.5%	
pp'-DDD	µg/kg	0.1—20	0.025	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
pp'-DDE	µg/kg	0.1—10	0.025	12.5%	
pp'-DDT	µg/kg	0.1—10	0.025	12.5%	
TOC	%	0.2—10	0.02	12.5%	
Transnonachlor	µg/kg	0.01—2	0.025	12.5%	
PN	%		0.02	12.5%	

In addition total organic carbon should be determined for both test materials.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

The sediments have been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator. It is left to the discretion of the laboratory to dry the material. You are advised to check the moisture content of the sediment by drying a small portion of the sediment to constant weight at ~ 110°C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material "as received".

The concentrations should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
MS-3 Polycyclic Aromatic Hydrocarbons in Sediment	
Test materials	QPH095MS and QPH096MS

Objective

This study covers the determination of PAHs and total organic carbon in marine sediment test materials.

Test Materials and storage

Test materials were prepared by WEPAL, Wageningen, The Netherlands.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the sediment was determined. All materials have been shown to be homogeneous at or below the intake mass used by the participants, and stable for the purposes of the test.

The test materials should be stored at room temperature prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QPH095MS	Sediment (estuarine)
QPH096MS	Sediment (open sea)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following PAHs and alkylated PAHs should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
1-Methylpyrene	µg/kg	2—500	0.5	12.5%	
2-Methylphenanthrene	µg/kg	5—1000	0.5	12.5%	
3,6-Dimethylphenanthrene	µg/kg	1—500	0.5	12.5%	
Acenaphthene	µg/kg	2—500	0.1	12.5%	
Acenaphthylene	µg/kg	1—100	0.2	12.5%	
Anthracene	µg/kg	2—500	0.1	12.5%	
Benzo[a]anthracene	µg/kg	10—1500	0.1	12.5%	
Benzo[a]fluorene	µg/kg	10—1000	0.5	12.5%	
Benzo[a]pyrene	µg/kg	10—1500	0.1	12.5%	
Benzo[b]fluoranthene	µg/kg	10—1500	0.5	12.5%	
Benzo[e]pyrene	µg/kg	10—1500	0.2	12.5%	
Benzo[g,h,i]perylene	µg/kg	10—1500	0.2	12.5%	
Benzo[k]fluoranthene	µg/kg	10—1000	0.1	12.5%	
Benzofluoranthenes (a+b+j+k)	µg/kg	10—2000	0.1	12.5%	
Chrysene	µg/kg	10—1500	0.2	12.5%	
Chrysene+Triphenylene	µg/kg	10—3000	0.2	12.5%	
Dibenz[a,h]anthracene	µg/kg	5—500	0.05	12.5%	
Dibenzo[a,i]pyrene	µg/kg		0.5	12.5%	
Dibenzothiophene	µg/kg	2—200	0.1	12.5%	
Fluoranthene	µg/kg	20—3000	0.2	12.5%	
Fluorene	µg/kg	2—300	0.1	12.5%	
Indeno[1,2,3-cd]pyrene	µg/kg	10—1500	0.2	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
Naphthalene	µg/kg	10—1500	0.5	12.5%	
Perylene	µg/kg	10—500	0.2	12.5%	
Phenanthrene	µg/kg	10—2000	0.5	12.5%	
Pyrene	µg/kg	10—3000	0.2	12.5%	
TOC	%	0.2—10	0.02	12.5%	
Triphenylene	µg/kg	20—3000	0.5	12.5%	
C1-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C2-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C3-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C1-pyrenes/fluoranthenes	µg/kg		0.5	12.5%	
C2-pyrenes/fluoranthenes	µg/kg		0.5	12.5%	
C1-chrysenes	µg/kg		0.5	12.5%	
C2-chrysenes	µg/kg		0.5	12.5%	
C1-benzofluoranthenes	µg/kg		0.5	12.5%	
PN	%		0.02	12.5%	

In addition total organic carbon should be determined for both test materials.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

The sediments have been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator. It is left to the discretion of the laboratory to dry the material. You are advised to check the moisture content of the sediment by drying a small portion of the sediment to constant weight at ~ 110˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. You may use any method with the appropriate extraction and clean-up. The final determination may be made using GC, GC-MS, HPLC etc. The method codes should be entered fully on the exercise template.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received”.

The concentrations should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017 – 2
MS-6 Organotins in Sediment	
Test materials	QSP062MS and QSP063MS

Objective

This study covers the determination of organotin compounds in sediment test materials.

Test Materials and storage

The sediment test materials were supplied by WEPAL, Wageningen.

The dry sediment test materials should be stored at room temperature, in a dry place, prior to analysis, and analysed as soon as possible after receipt.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the sediment was determined. All materials have been shown to be homogeneous at or below the intake mass used by the participants, and stable for the purposes of the test.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QSP062MS	Sediment (estuarine)
QSP063MS	Sediment (open sea)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following organotin compounds should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	1—500	0.1	12.5%	
Diphenyltin(DPT)	µg Sn/kg	0.1—200	0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	1—500	0.1	12.5%	
Monophenyltin(MPT)	µg Sn/kg	0.1—200	0.1	12.5%	
Tributyltin(TBT)	µg Sn/kg	1—500	0.1	12.5%	
Triphenyltin(TPT)	µg Sn/kg	0.1—200	0.1	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

The sediments have been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Report your method codes using the Data Submission Form on the Participant Site. Please check each of your method codes and update where necessary. Advise QUASIMEME of additional codes that would better describe your methodology.

Only one result per determinand per test material is required.

The results of each determinand should be expressed as Sn on the test materials “as received”. All results should be reported as µg Sn /kg weight of sediment as received

The concentration of organotins should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
MS-7 Brominated flame retardants in Sediment	
Test materials	QBC052MS and QBC053MS

Objective

This study covers the determination of brominated compounds in sediment test material.

Test Materials and storage

The test materials were supplied by WEPAL, Wageningen, The Netherlands.

The Sediment has been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator. It is left to the discretion of the laboratory to dry the material. You are advised to check the moisture content of the sediment by drying a small portion of the sediment to constant weight at ~ 110˚C.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored in a dry place at room temperature in the dark, prior to analysis, and analysed as soon as possible after receipt.

Sample Code	Description
QBC052MS	Sediment (estuarine)
QBC053MS	Sediment (estuarine)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
a-HBCD	µg/kg		0.05	12.5%	
BDE100	µg/kg	0.01—10	0.05	12.5%	
BDE153	µg/kg	0.1—5	0.05	12.5%	
BDE154	µg/kg	0.01—5	0.05	12.5%	
BDE183	µg/kg	0.1—2	0.05	12.5%	
BDE209	µg/kg	20—200	0.05	12.5%	
BDE28	µg/kg	0.01—2	0.05	12.5%	
BDE47	µg/kg	0.1—20	0.05	12.5%	
BDE66	µg/kg	0.01—10	0.05	12.5%	
BDE85	µg/kg	0.01—10	0.05	12.5%	
BDE99	µg/kg	0.1—50	0.05	12.5%	
b-HBCD	µg/kg		0.05	12.5%	
Dimethyl-TBBP-A	µg/kg		0.05	12.5%	
g-HBCD	µg/kg		0.05	12.5%	
TBBP-A	µg/kg		0.05	12.5%	
Total-HBCD	µg/kg	50—200	0.05	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

The sediments have been dried and sieved to < 0.5 mm. Before sub-sampling, shake the bottle for at least two minutes. Repeat this re-homogenisation each time the test material is used. It is recommended that the sediments be kept in a desiccator.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Report your method codes using the Data Submission Form on the Participant Site. Please check each of your method codes and update where necessary. Advise QUASIMEME of additional codes that would better describe your methodology.

Only one result per determinand per test material is required.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
BT-1 Trace metals in Biota	
Test materials	QTM116BT and QTM117BT

Objective

This study covers the determination of ten trace metals, ash weight, dry weight and total lipid in biological tissue test materials.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QTM116BT	Mussel tissue
QTM117BT	Salmon fillet

Precaution

The jars with biological tissue test materials are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The jars are filled in this way in order to eliminate as much air as possible. This minimises any degradation of the test materials in transit and storage prior to opening. The jars of biota should be cooled for a few hours at ca. 4˚C prior to opening, to eliminate possible overpressure.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following trace metals should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Shellfish tissue	Fish muscle tissue	Const	Prop	
Arsenic	mg/kg	0.2—10	1—10	0.02	12.5%	
Ash-weight	%			0.1	12.5%	
Cadmium	µg/kg	10—500	1—50	20	12.5%	
Chromium	µg/kg	10—5000	50—500	20	12.5%	
Copper	µg/kg	50—10000	100—1000	100	12.5%	
Dry-weight	%			0.1	12.5%	
Extractable-Lipid	%			0.1	12.5%	
Lead	µg/kg	10—1000	10—50	5	12.5%	
Mercury	µg/kg	5—500	20—1000	20	12.5%	
Nickel	µg/kg	10—2000	10—200	20	12.5%	
Selenium	µg/kg	200—1000	200—2000	10	12.5%	

Determinand	Unit	Concentration range		Error		AA-EQS
		Shellfish tissue	Fish muscle tissue	Const	Prop	
Silver	µg/kg	1—500	0.5—50	5	12.5%	
Total-Lipid	%			0.1	12.5%	
Zinc	mg/kg	2—200	2—10	2	12.5%	

Ash weight, dry weight and total lipid should also be determined. In addition to the parameters given in the table, also the following metals can be reported: Li, Be, Na, Mg, Al, P, S, K, Ca, Sc, Ti, V, Mn, Fe, Co, Ga, Rb, Sr, Y, Zr, Mo, Pd, Sn, Sb, Te, Cs, Ba, La, Ce, Nd, Ta, W, Pt, Au, Tl, Bi, Th, U. If you normally measure extractable lipid, there is a field in the template for reporting this measurement. This has been added following the request from a number of participants. However, we would encourage you to also report total lipid. A successful QUASH study has found no significant difference between the Smedes lipid method and the Bligh and Dyer method for total lipid determination. We therefore recommend that wherever possible you use the Smedes lipid method, as it gives better reproducibility and does not involve the use of chlorinated solvents. A copy of the Smedes lipid method protocol is included in Annex 3.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the tin, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

The concentrations should be determined against your own calibration solutions.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
BT-2 Chlorinated Organics in Biota	
Test materials	QOR132BT and QOR133BT

Objective

This study covers the determination chlorobiphenyls (CBs), organochlorine pesticides (OCPs) and total lipid in biological tissue test materials.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, in a dry place, prior to analysis, and analysed as soon as possible after receipt. Although the tins have a protective coating, moisture can cause corrosion of the surface of the tins. Apart from the moisture, temperatures below 0°C will not negatively affect the quality in the material, and we do not expect the contaminants to be affected at these temperatures. Temperatures above 30°C may be disadvantageous for the material. The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QOR132BT	Mussel tissue
QOR133BT	Sprat (whole fish) tissue

Shellfish tissues might be spiked with organochlorines, so patterns measured can differ from natural patterns

Precaution

The tins with biological tissue test materials are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The tins are filled in this way in order to eliminate as much air as possible. This minimises any degradation of the test materials in transit and storage prior to opening. The tins of biota should be cooled for a few hours at ca. 4°C prior to opening, to eliminate possible overpressure.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range			Error		AA-EQS
		Fish Liver tissue and Freshwater Fish	Fish Muscle Tissue	Shellfish Tissue	Const	Prop	
PCB28	µg/kg	1—50	0.05—5	0.05—5	0.025	12.5%	
PCB31	µg/kg	1—10	0.03—3	0.03—3	0.025	12.5%	
PCB52	µg/kg	10—100	0.05—5	0.05—5	0.025	12.5%	
PCB101	µg/kg	30—300	0.1—20	0.1—20	0.025	12.5%	
PCB105	µg/kg	2—100	0.05—10	0.05—10	0.025	12.5%	
PCB118	µg/kg	20—300	0.2—20	0.2—20	0.025	12.5%	
PCB138+PCB163	µg/kg	20—600	0.3—30	0.3—30	0.025	12.5%	
PCB138	µg/kg	20—600	0.3—30	0.3—30	0.025	12.5%	
PCB153	µg/kg	50—1000	0.4—40	0.4—40	0.025	12.5%	
PCB156	µg/kg	1—40	0.03—10	0.03—10	0.025	12.5%	
PCB180	µg/kg	10—200	0.05—5	0.05—5	0.025	12.5%	
□HCH	µg/kg	0.05—5	0.05—5	0.05—5	0.02	12.5%	
□HCH	µg/kg	0.1—5	0.05—5	0.05—5	0.025	12.5%	
□HCH	µg/kg	0.05—5	0.05—5	0.05—5	0.025	12.5%	
□HCH	µg/kg	0.05—5	0.05—5	0.05—5	0.025	12.5%	
HCB	µg/kg	2—50	0.02—5	0.02—5	0.025	12.5%	
HCBD	µg/kg	0.05—5			0.025	12.5%	
Dieldrin	µg/kg	0.5—100	0.2—20	0.2—20	0.025	12.5%	
pp'-DDD	µg/kg	5—100	0.1—10	0.1—10	0.025	12.5%	
pp'-DDE	µg/kg	10—500	0.3—30	0.3—30	0.025	12.5%	
op'-DDT	µg/kg	0.1—2	0.01—1	0.01—1	0.025	12.5%	
pp'-DDT	µg/kg	0.1—10	0.1—10	0.1—10	0.025	12.5%	
Transnonachlor	µg/kg	0.2—40	0.02—10	0.02—10	0.025	12.5%	
Total-Lipid	%				0.1	12.5%	
Extractable-Lipid	%				0.1	12.5%	

In addition total lipid should be determined. If you normally measure extractable lipid, there is a field in the template for reporting this measurement. This has been added following the request from a number of participants. However, we would encourage you to also report total lipid. A successful QUASH study has found no significant difference between the Smedes lipid method and the Bligh and Dyer method for total lipid determination. We therefore recommend that wherever possible you use the Smedes lipid method, as it gives better reproducibility and does not involve the use of chlorinated solvents. A copy of the Smedes lipid method protocol is included in Annex 3.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the tin, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

The concentrations should be determined against your own calibration solutions.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
BT-4 Polycyclic Aromatic Hydrocarbons in Biota	
Test materials	QPH087BT and QPH088BT

Objective

This study covers the determination of PAHs and total lipid in biological tissue test materials.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands and the Institute for Environmental Studies, Free University, Amsterdam, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QPH087BT	Shrimp tissue
QPH088BT	Shellfish tissue (mussel)

Shellfish tissues might be spiked with PAHs, so patterns measured can differ from natural patterns

Precaution

The tins with biological tissue are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The tins are filled in this way in order to eliminate as much air as possible. This minimizes any degradation of the test materials in transit and storage prior to opening. The tins of biota should be cooled for a few hours at ca. 4˚C prior to opening, to eliminate possible overpressure. Opening the bottom of the tin, instead of the top, may also help to prevent spillage of moisture.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following PAHs should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Shellfish tissue	Const	Prop	
1-Methylpyrene	µg/kg		2	12.5%	
2-Methylphenanthrene	µg/kg	0.2—5	2	12.5%	
3,6-Dimethylphenanthrene	µg/kg	0.2—2	0.5	12.5%	
Acenaphthene	µg/kg	0.5—100	0.2	12.5%	
Acenaphthylene	µg/kg	0.2—5	0.2	12.5%	
Anthracene	µg/kg	0.2—10	0.2	12.5%	
Benzo[a]anthracene	µg/kg	0.2—10	0.2	12.5%	
Benzo[a]fluorene	µg/kg		0.5	12.5%	
Benzo[a]pyrene	µg/kg	0.2—5	0.2	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Shellfish tissue	Const	Prop	
Benzo[b]fluoranthene	µg/kg	0.2—10	0.2	12.5%	
Benzo[e]pyrene	µg/kg	0.2—10	0.2	12.5%	
Benzo[g,h,i]perylene	µg/kg	0.2—5	0.2	12.5%	
Benzo[k]fluoranthene	µg/kg	0.2—5	0.2	12.5%	
Benzofluoranthenes (a+b+j+k)	µg/kg	0.2—20	0.2	12.5%	
Chrysene	µg/kg	0.2—20	0.2	12.5%	
Chrysene+Triphenylene	µg/kg	0.2—20	0.2	12.5%	
Dibenz[a,h]anthracene	µg/kg	0.2—2	0.1	12.5%	
Dibenzo[a,i]pyrene	µg/kg		0.5	12.5%	
Dibenzothiophene	µg/kg	0.2—5	0.5	12.5%	
Extractable-Lipid	%		0.1	12.5%	
Fluoranthene	µg/kg	5—50	0.2	12.5%	
Fluorene	µg/kg	1—50	0.2	12.5%	
Indeno[1,2,3-cd]pyrene	µg/kg	0.2—5	0.2	12.5%	
Naphthalene	µg/kg	1—100	0.2	12.5%	
Perylene	µg/kg	0.1—5	0.5	12.5%	
Phenanthrene	µg/kg	2—50	0.2	12.5%	
Pyrene	µg/kg	1—20	0.2	12.5%	
Total-Lipid	%		0.1	12.5%	
Triphenylene	µg/kg		5	12.5%	

In addition total lipid should be determined. If you normally measure extractable lipid, there is a field in the template for reporting this measurement. This has been added following the request from a number of participants. However, we would encourage you to also report total lipid. A successful QUASH study has found no significant difference between the Smedes lipid method and the Bligh and Dyer method for total lipid determination. We therefore recommend that wherever possible you use the Smedes lipid method, as it gives better reproducibility and does not involve the use of chlorinated solvents. A copy of the Smedes lipid method protocol is included in Annex 3.

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the tin, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

The concentrations should be determined against your own calibration solutions.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form. Please report your Method information together with the results.

ROUND	2017-2
BT-8 Organotins in Biota	
Test materials	QSP062BT and QSP063BT

Objective

This study covers the determination of organotin compounds in biological tissue test materials.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands and the Institute for Environmental Studies, Free University, Amsterdam, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Treat all test materials in the same manner as your routine samples.

Sample Code	Description
QSP062BT	Shellfish tissue (mussel)
QSP063BT	Shellfish tissue (mussel)

Shellfish tissues might be spiked with organotins, so patterns measured can differ from natural patterns

Precaution

The tins or jars with biological tissue are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The tins or jars are filled in this way in order to eliminate as much air as possible. This minimizes any degradation of the test materials in transit and storage prior to opening. The tins or jars of biota should be cooled for a few hours at ca. 4°C prior to opening, to eliminate possible overpressure.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	1—100	0.1	12.5%	
Diphenyltin(DPT)	µg Sn/kg		0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	5—30	0.1	12.5%	
Monophenyltin(MPT)	µg Sn/kg		0.1	12.5%	
Tributyltin(TBT)	µg Sn/kg	2—50	0.1	12.5%	
Triphenyltin(TPT)	µg Sn/kg		0.1	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the tin, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

The concentrations should be determined against your own calibration solutions.

Only one result per determinand per test material is required. The results of each determinand should be expressed as Sn on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on the Participant Site. It is not possible to report two sets of data using different methods on the same Data Submission Form.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form

Please report your Method information together with the results.

ROUND	2017-2
BT-9 Brominated Flame Retardants in Biota	
Test materials	QBC052BT and QBC053BT

Objective

This study covers the determination of brominated compounds in biota.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands and the Institute for Environmental Studies, Free University, Amsterdam, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Sample Code	Description
QBC052BT	Freshwater fish tissue
QBC053BT	Turbot liver tissue

Shellfish tissues might be spiked with BFRs, so patterns measured can differ from natural patterns

Precaution

The tins or jars with biological tissue are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The tins or jars are filled in this way in order to eliminate as much air as possible. This minimises any degradation of the test materials in transit and storage prior to opening. The tins or jars of biota should be cooled for a few hours at ca. 4°C prior to opening, to eliminate possible overpressure.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
BDE28	µg/kg	0.001—1	0.005	12.5%	
BDE47	µg/kg	0.05—40	0.005	12.5%	
BDE66	µg/kg	0.01—10	0.005	12.5%	
BDE85	µg/kg	0.01—10	0.005	12.5%	
BDE99	µg/kg	0.01—10	0.005	12.5%	
BDE100	µg/kg	0.005—10	0.005	12.5%	
BDE153	µg/kg	0.01—2	0.005	12.5%	
BDE154	µg/kg	0.001—5	0.005	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
BDE183	µg/kg	0.001—0.1	0.005	12.5%	
BDE209	µg/kg	0.01—0.1	0.005	12.5%	
TBBP-A	µg/kg		0.005	12.5%	
Dimethyl-TBBP-A	µg/kg		0.005	12.5%	
□HBCD	µg/kg		0.005	12.5%	
□HBCD	µg/kg		0.005	12.5%	
□HBCD	µg/kg		0.005	12.5%	
Total-HBCD	µg/kg		0.005	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process of the biological tissue test material moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the jar, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Recovery values of over 100% should not be used to correct the data.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

You may wish to use two different GC columns of different polarity for the determination of the BDEs. Use your own judgement to report the best result. The column used for this result should be reported.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site.

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
BT-10 Perfluorinated Alkyl Substances (PFASs) in Biota	
Test materials	QPF007BT and QPF008BT

Objective

This study covers the determination of perfluorinated alkyl substances in biota.

Test Materials and storage

The test materials were supplied by Wageningen Marine Research (IMARES), IJmuiden, The Netherlands and the Institute for Environmental Studies, Free University, Amsterdam, The Netherlands.

The tins or jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into tins and sterilised by autoclaving. Each batch of material was prepared in bulk.

All materials have been shown to be homogeneous at or below the intake mass used by the participants, and are stable for the purposes of the test.

The test materials should be stored at room temperature, prior to analysis, and analysed as soon as possible after receipt.

Sample Code	Description
QPF007BT	Turbot liver tissue
QPF008BT	Flounder whole fish tissue

Precaution

The tins or jars with biological tissue are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The tins or jars are filled in this way in order to eliminate as much air as possible. This minimises any degradation of the test materials in transit and storage prior to opening. The tins or jars of biota should be cooled for a few hours at ca. 4˚C prior to opening, to eliminate possible overpressure.

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
PFOS	ng/kg	0.1 - 1000	0.05	12.5%	
PFBA	ng/kg		0.05	12.5%	
PFPeA	ng/kg		0.05	12.5%	
PFHxA	ng/kg		0.05	12.5%	
PFHpA	ng/kg		0.05	12.5%	
PFOA	ng/kg		0.05	12.5%	
PFNA	ng/kg		0.05	12.5%	
PFDA	ng/kg	0.1 – 10	0.05	12.5%	
PFUdA	ng/kg	0.001 - 1	0.05	12.5%	
PFDoA	ng/kg	0.001 – 0.1	0.05	12.5%	
PFTrDA	ng/kg	0.01 – 0.1	0.05	12.5%	
PFTeDA	ng/kg		0.05	12.5%	

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
L-PFBS**	ng/kg	0.1 - 10	0.05	12.5%	
L-PFHxS**	ng/kg		0.05	12.5%	
L-PFHps**	ng/kg		0.05	12.5%	
PFOSA	ng/kg	0.1 - 10	0.05	12.5%	
PFDS	ng/kg		0.05	12.5%	
PFODA	ng/kg		0.05	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

During the sterilisation process of the biological tissue test material moisture may have partly separated from the solid part of the test materials, and this may cause inhomogeneity. Therefore, prior to taking an aliquot for analysis the following treatment of the test materials is essential. After opening the test material container the complete contents should be thoroughly homogenised. As it is difficult to homogenise the sample in the jar, we recommend transferring the material to a larger container for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing inhomogeneity again. After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20˚C.

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Recovery values of over 100% should not be used to correct the data.

Only one result per determinand per test material is required. The results of each determinand should be expressed on the test material “as received” i.e. on a wet weight basis. If your normal method is to dry the test material prior to extraction then the dry weight must be determined. The final result should be recalculated and reported based on wet weight using the dry weight determination.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site.

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
BT-7 ASP Shellfish Toxins	
Test materials	QST234SS, QST235BT and QST236BT

Objective

This study covers the determination of amnesic shellfish toxins domoic acid and epidomoic acid (as a racemic mixture) in standard solution and shellfish tissue test materials.

Test Materials and storage

The test materials were supplied by the Marine Institute, Galway, Ireland.

- QST234SS is a ASP standard solution.

- QST235BT is an oyster tissue homogenate supplied in a plastic vial.

- QST236BT is an mussel tissue homogenate supplied in a plastic vial.

For QST235BT and QST236BT, each vial contains sufficient material for one-shot analysis of domoic and epidomoic acid.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity and stability was determined. All materials have been shown to be homogeneous at or below the intake mass normally used, and stable for the purposes of the test.

Begin the analysis as soon as possible, preferably within 7 days of receipt.

The shellfish tissue homogenates (5ml plastic vials) should be stored at -20˚C, or a lower temperature, immediately upon receipt, until analysis.

The standard solution QST234SS should be stored in the refrigerator at ca 4˚C immediately upon receipt, until analysis

The test materials have been shipped on cool packs. The cool packs will thaw slowly during transit, but this does not significantly affect the stability of the material for the purpose of this study, providing the test materials themselves are stored as directed, immediately on receipt.

Sample Code	Description
QST234SS	Standard Solution
QST235BT	Shellfish tissue (oyster tissue)
QST236BT	Shellfish tissue (mussel tissue)

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

Report the sum of the domoic acid and epidomoic acid as a racemic mixture.

Determinand	Unit	Concentration range	Error		AA-EQS
		Shellfish tissue	Const	Prop	
Domoic+Epidomoic	mg/kg		0.1	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

It is advisable to analyse the test materials as soon as possible after receipt. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. One result per test material is required, for the sum of domoic and epidomoic acid as a racemic mixture. The results of each determinand should be expressed on the test material "as received" i.e. on a wet weight basis. The concentrations should be determined against your own calibration solutions.

Each vial contains sufficient quantity of homogenate for one analysis. The whole transferable contents of each vial should be extracted, and one result reported for the sum of the two isomers. To transfer the contents into a preweighed or tared extraction tube, the vial should be fully defrosted, vortex-mixed and the contents poured into the desired container.

All results should be reported in mg/kg on the basis of wet weight of the test material as provided. The density of the standard solution is 0.9853 g/ml at 22°C. The weight of shellfish tissue test materials should be determined prior to analysis.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form.

Please report your Method information together with the results.

ROUND	2017-2
BT-11 Lipophilic Shellfish Toxins	
Test materials	QST237SS, QST238BT, QST239BT, QST240BT and QST241BT

Objective

This study covers the determination of lipophilic toxins in shellfish tissue.

Test Materials and storage

The test materials were supplied by the Marine Institute, Galway, Ireland.

Each vial contains sufficient material for one-shot analysis of OA, AZA, YTX and PTX-group toxins.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity and stability was determined. All materials have been shown to be homogeneous at the intake mass normally used, and stable for the purposes of the test.

Begin the analysis as soon as possible, preferably within 7 days of receipt.

All materials (contained either in ampoules or in 5ml plastic vials) should be stored at -20˚C, or a lower temperature, immediately upon receipt, until analysis.

The test materials have been shipped on cool packs. The cool packs will thaw slowly during transit, but this does not significantly affect the stability of the material for the purpose of this study, providing the test materials themselves are stored as directed, immediately on receipt.

Sample Code	Description
QST237SS	Multitoxin standard solution
QST238BT	DSP-AZP extract
QST239BT	Mussel tissue extract
QST240BT	Mussel tissue homogenate
QST241BT	Mussel tissue homogenate

Precaution

Some of the substances may present a health hazard and can be biologically active. Please ensure that your analytical and handling procedures for this material have been fully assessed for safety and that all specified precautions are taken.

Determinands and concentration ranges

Determinands

a) Methods based on chromatographic separation techniques (e.g. LC-FD, or LC-MS):

Please report lipophilic toxins (if detected) as follows:

- Free OA-group toxins, OA, DTX-1, DTX-2 (pre-hydrolysis) individually and their sum (standard solutions, extracts and tissues).
- Total OA-group toxins, OA, DTX-1, DTX-2 (post-hydrolysis) individually and their sum (extracts and tissues only).
- PTX-group toxins, PTX-1 and PTX-2 individually (standard solutions, extracts and tissues).
- Total OA-group and PTX-group toxins, sum of OA, DTX-1, DTX-2 (post-hydrolysis), PTX-1 and PTX-2 (extracts and tissues only).

- AZA-group toxins, AZA-1, AZA-2 and AZA-3 individually and their sum (standard solutions, extracts and tissues).
- YTX-group toxins, YTX, homo-YTX, 45-OH-YTX and 45-OH-homo-YTX individually and their sum (standard solutions, extracts and tissues).

Please note, for the OA-group toxins, there is no result reported for the ester-forms themselves, only for free toxins and the sum of free toxins plus esters.

b) Methods based on determination of the sum of OA-equivalents present (e.g. PP2a):

For the standard solution, report the sum of OA-equivalents as free toxins (without hydrolysis), and the sum of OA-equivalents post hydrolysis. This means there is no result reported for the esters-forms themselves, only for free toxins and the sum of free toxins plus esters.

If you do not analyse for one of the determinands, eg. DTX-1 or DTX-2, please do not report the sum of OA+DTX-1+DTX-2. Equally if you do not carry out hydrolysis or determination of DTX-1 or DTX-2 post-hydrolysis, please do not report the sum of hydrolysed results.

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
AZA-1	µg/kg		0.1	12.5%	
AZA-2	µg/kg		0.1	12.5%	
AZA-3	µg/kg		0.1	12.5%	
AZA-total	µg AZA-eq./kg		0.1	12.5%	
Free-DTX1	µg/kg		0.1	12.5%	
Free-DTX2	µg/kg		0.1	12.5%	
Free-Okadaic-Acid	µg/kg		0.1	12.5%	
Total-free-OA+DTX-1+DTX-2	µg OA-eq./kg		0.1	12.5%	
Total-DTX1	µg/kg		0.1	12.5%	
Total-DTX2	µg/kg		0.1	12.5%	
Total-Okadaic Acid	µg/kg		0.1	12.5%	
Total-hy-OA+DTX1+DTX2	µg OA-eq./kg		0.1	12.5%	
PTX-1	µg/kg		0.1	12.5%	
PTX-2	µg/kg		0.1	12.5%	
Total-OA-group and PTX-group	µg OA-eq./kg		0.1	12.5%	
YTX	mg/kg		0.1	12.5%	
homo-YTX	mg/kg		0.1	12.5%	
45-OH-YTX	mg/kg		0.1	12.5%	
45-OH-homo-YTX	mg/kg		0.1	12.5%	
Total-YTX-group	mg YTX-eq./kg		0.1	12.5%	

Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Toxicity Equivalency Factors

Total toxicity equivalence for each of the biotoxin groups should be calculated using toxicity equivalency factors (TEFs) as recommended by EFSA, which are contained in the following table:

Toxin Group	Analogue	TEF
OA-group	OA	1
	DTX-1	1
	DTX-2	0.6
AZA-group	AZA-1	1
	AZA-2	1.8

Toxin Group	Analogue	TEF
	AZA-3	1.4
PTX-group	PTX-1	1
	PTX-2	1
YTX-group	YTX	1
	homo-YTX	1
	45-OH-YTX	1
	45-OH-homo-YTX	0.5

Analysis

It is advisable to analyse the test materials as soon as possible after receipt. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

One determination of each test material is required, for each determinand. The results of each determinand should be expressed on the test material "as received" i.e. on a wet weight basis. The concentrations should be determined against your own calibration solutions.

If you routinely conduct analyses by more than one technique you may report multiple sets of data.

You should inform the QUASIMEME office staff, who will arrange an additional exercise template file. It is not possible to report two sets of data using different methods on the same exercise template.

The standard solution ampoules contain at least 500 µl, sufficient for 1 injection of a solution into a LC or 1 analyses of the solution by an assay.

Each vial contains sufficient quantity of extract or homogenate for one analysis. The whole transferable contents of each vial should be extracted. To transfer the contents into a preweighed or tared extraction tube, the vial should be fully defrosted, vortex-mixed and the contents poured into the desired container.

Please note all test materials should be stored in the freezer at ca -20°C or less between analyses.

All the results should be reported in µg/kg (YTX-group toxins in mg/kg) on the basis of the wet weight of the test material as provided. The density of the standard solutions are 0.7918 g/ml (MeOH) and the density of the **lipophilic and DSP/AZP extracts** is 0.834 g/ml. For the tissues, the weight of material should be determined prior to analysis.

Please note that if your laboratory does not report on a given analogue, e.g. DTX-1, then your laboratory should not report the sum of toxins, since this will give 2 z-scores out of line and will possibly make data-analysis more difficult for the remaining laboratories which did determine this analyte.

Reporting

One result for each determinand in each test material should be reported using the Data Submission Forms which are placed on the Participant Site

It is not possible to report two sets of data using different methods in the same Data Submission Forms.

If you routinely conduct analyses by more than one technique you may report multiple sets of data. You should inform the QUASIMEME office staff, who will arrange an additional Data Submission Form. Please report your Method information together with the results.

Reporting of Results and Analytical Methods

Units

The units of measurement are given in the Data Submission Forms. Ensure that the concentration of each determinand is reported in the units given. This may differ from your normal units for reporting; it is essential that all data reported are comparable. It is not possible for you to alter the units for reporting in the Data Submission Forms.

The precision of the reported results should reflect the level of uncertainty of the measurement in your laboratory

Reporting Left Censored Values

If the concentration of a determinand is below the detection limit of your method, you may wish to report the value as less than the detection limit. To do this, you should report your detection limit, either as a negative number or preceded by the "less-than" symbol, <. I.e. to report a value less than a detection limit of 10, report either "-10" or "<10". The system will identify either of these formats as left censored ("less-than") values. Left censored values are included in the statistical evaluation of the data, and in the reports.

Method Codes

You are kindly asked to report your methods used, by the Method codes given in the Data Submission Forms. When the method used by your laboratory can not be chosen by one of the MIC (Method Information Code) options given in the Data Submission Form, please select others (option Z) and provide us with the details of the method used by your lab.

Return of Data

Upload all analytical data to the QUASIMEME site only with the Data Submission Forms on the Participant Site. This allows a rapid and accurate transfer of your data and an early report to you. Additional information and comments may be provided as attached files.

Only data submitted using the Data Submission Forms can be included in the assessment. Return the results to the WEPAL-QUASIMEME Project Office in Wageningen no later than **1 February 2018**. Data arriving after this deadline may not be entered into the database or appear in the report.

If you have further information on additional methods used or specific ways in which we can improve the data transfer, please inform the QUASIMEME Project Office (Quasimeme@wur.nl). Your co-operation is appreciated and will help the assessors in the data analysis and in providing appropriate advice in case of any analytical difficulties.

Please observe the following guidelines, to reduce the need for additional checks, replies and enquires:

Data should only be submitted to the WEPAL-QUASIMEME Project Office when all quality checks have been made. If data are submitted beyond the deadline, they might not be included in the report. Data submitted after the issue of the report will not be included in the report, and these data will also not be included as part of the consensus value. Any certificate prepared with data submitted late will include the statement "Data submitted after report issued". No data will be re-entered into the database after the report is issued. No data will be changed in the database UNLESS there is evidence that QUASIMEME or data transfer has caused an error. In such cases QUASIMEME will undertake a quality query to investigate the problem and inform the participant of the outcome of the Query.

The assigned values will be calculated based on the assessment of all data returned, using the Cofino model. The report for each study, including each laboratory's individual assessment and z-scores, will be distributed to participants no later than 10 February 2018. Background information on the data assessment will be provided with the reports.

Collusion and Falsification of Results

QUASIMEME accepts that most participants operate with professional integrity and that data returned as part of the LP studies are correct and are submitted without interference or collusion. However, in

some circumstances, data or information may be influenced by, for example, (i) repeated analyses and submitting mean data, or (ii) collaboration with colleagues undertaking the same study.

QUASIMEME checks for evidence of collusion and confirm to all participants that such activity is contrary to professional scientific conduct and will only nullify the benefits of the LP studies to accreditation bodies and analysts alike.

QUASIMEME reserves the right to withdraw participation of any institute who, in the opinion of the Scientific Assessment Group, has submitted data following collusion or falsification. This statement is made as a formal requirement for accreditation for Laboratory Performance Studies under ISO17043.

ANNEX 1 Notification of damaged test materials.

You do not need to notify QUASIMEME if the test materials arrived in good condition

Client number : .....

Damaged container number : .....

Loss of weight container number : .....

I request a new test material for :..... due to : .....

Date : .....

Signature :.....

Name of participant :.....

Name and address of institute : .....

.....

.....

Telephone number :.....

Fax number :.....

Return this form to :

WEPAL-QUASIMEME Project Office
P.O. Box 8005
6700 EC Wageningen
The Netherlands

Phone : **+31(0)317 48 65 46**
Fax: **+31(0)317 48 56 66**
E-mail : **quasimeme@wur.nl**

ANNEX 2 Instructions for login into Participant Site

Login to https://www.participants.wepal.nl or www.quasimeme.org

Type in your Username and password into the box

Ask the WEPAL-QUASIMEME Project Office when the login information is unknown

Click login

Select the correct program

Enter your results

Lower than results will be automatically transferred into – values.

Click on the save button to store your data into the database

ANNEX 3 Total Lipid Extraction According to Smedes

This method is based on research carried out by Foppe Smedes.

See : Determination of total lipid using non-chlorinated solvents

Smedes, F., Analyst 124 (1999) : 1711-1718.

Instruments and Chemicals

- Balance with a precision of 0.1 mg
- Ultra Turrax
- Centrifuge capable of holding 100 ml tubes or glass jar at a speed of 2000 rpm[1]
- Heated waterbath with condensers.
- Evaporation flasks in suitable shape and size
- Pipettes
- Deionised water
- Isopropanol
- Cyclohexane
- Solution of 13 % (w/w) isopropanol in cyclohexane.

Procedure

- Carry out a dry-weight determination on a representative portion of the test material to be analyses.
- Take a portion of wet test material, which does not contain more than 1g lipid or 8g of water.
- Weigh the test material with known moisture content in a 100ml centrifuge tube or appropriate glass jar.
- Add 18ml isopropanol and 20ml cyclohexane.
- Mix with Ultra Turrax for two minutes.
- Add W ml of water. W is calculated by :

$$W = 22 - \frac{\text{Sampleintake (g)} * \text{moisturecontent (\%)}}{100}$$

- Mix with another Ultra Turrax for minute.
- Separate the phases by centrifugation[2].
- Transfer as much as possible of the organic phase to an evaporation flask (by small pipette). Filtration is optional but makes the method more robust[3].
- Add 20 ml cyclohexane containing 13%(w/w) isopropanol and mix for one minute by Ultra Turrax.

[1] When a centrifuge is not used, the phases may take time to separate and the interface is less sharp, which can result in a low recovery of the organic phase. A check should be made to determine whether > 80% of the organic phase has been recovered (18 ml). A third extraction is recommended in the case of a lower recovery.

[2] Some tissues, like liver extracts, form an emulsion which can be prevented by replacing the water by 1 M $HClO_4$ to denature the proteins. The addition of NaCl may also help.

[3] In some cases the organic phase may contain some tissue particles when using the B & D Method. This also depends on the mixing method used (e.g. ultra sonic). When this occurs the extract should be filtered by passing the extract through a glass column plugged with ca 2cm of cottonwool which has previously been extracted with solvent.

- Centrifuge.
- Transfer the upper phase to the flask containing the first extract and evaporate the solvent.
- Quantitavely transfer the residue to a weighed wide-mouth cup by using a few ml of the cyclohexane/isopropanol mixture or diethylether.
- Evaporate in a moderately warm place to dryness (do not boil). The temperature used should be 5– 10 ˚C below the boiling point of the washing solvent. Evaporation may be assisted by a stream of nitrogen.
- Further dry the residue for one hour at 105 ˚C
- Weigh the residue and calculate the lipid content from the intake.