Quasimeme Laboratory Performance Studies

Round 2019 - 2

1 October 2019 to 1 February 2020

Exercise Protocols

Version 1: 27 September 2019

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Introduction Round 2019 - 2

Thank you for participating in the 2019 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round 2019 - 2, that you have ordered will be sent to you by courier in the week of 30 September 2019. 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
2019 - 2	AQ-1	Seawater	Nutrients
2019 - 2	AQ-2	Estuarine and Low Salinity Open Water	Nutrients
2019 - 2	AQ-3	Seawater	Metals
2019 - 2	AQ-4	Seawater	Mercury
2019 - 2	AQ-11	Seawater	Chlorophyll and Pheopigments
2019 - 2	AQ-14	Seawater	DOC
2019 - 2	MS-1	Sediment	Trace Metals
2019 - 2	MS-2	Sediment	Chlorinated Organics
2019 - 2	MS-3	Sediment	Polycyclic Aromatic Hydrocarbons (PAHs)
2019 - 2	MS-6	Sediment	Organotins
2019 - 2	MS-7	Sediment	Brominated Flame Retardants (BFRs)
2019 – 2	MS-8	Sediment	Perfluorinated Alkyl Substances (PFASs)
2019 - 2	BT-1	Biota	Trace Metals
2019 - 2	BT-2	Biota	Chlorinated Organics
2019 - 2	BT-4	Biota	Polycyclic Aromatic Hydrocarbons (PAHs)
2019 - 2	BT-8	Biota	Organotins
2019 - 2	BT-9	Biota	Brominated Flame Retardants (BFRs)
2019 – 2	BT-10	Biota	Perfluorinated Alkyl Substances (PFASs)
2019 - 2	BT-7	Shellfish and Solution	ASP Shellfish Toxins
2019 - 2	BT-11	Shellfish and Solution	DSP Shellfish Toxins
2019 – 2	BT-12	Shellfish	PSP Shellfish Toxins
2019-2	DE-16	Shellfish and solution	Tetrodotoxin

All data for these studies must be uploaded to your Quasimeme SharePoint Site, using the Data Submission Forms, no later than 1st February 2020.

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

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ROUND	2019 - 2
AQ-1 Nutrients in Seawater	
Test materials	QNU340SW, QNU341SW and QNU342SW

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 (QNU340SW). 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
QNU340SW	Seawater (Salinity > 30 psu)
QNU341SW	Seawater (Salinity > 30 psu) spiked
QNU342SW	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	0.05—5	0.1—5	0.1	6.0%	
Nitrite	µmol/L	0.01—2	0.1—2	0.01	6.0%	
Phosphate	µmol/L	0.02—5	0.1—5	0.05	6.0%	
Silicate	µmol/L	0.2—10	0.2—10	0.1	6.0%	
Total-N	µmol/L	2.5—25	5—25	0.5	6.0%	
Total-P	µmol/L	0.1—5	0.2—5	0.05	6.0%	
TOxN	µmol/L	0.05—15	0.1—15	0.05	6.0%	
Salinity	psu			0.01	0.1%	

Please report Salinity only for QNU340SW

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	2019 -2
AQ-2 Nutrients in Estuarine and Low Salinity Open Seawater	
Test materials	QNU343EW, QNU344EW, QNU345EW and QNU346EW

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 (QNU343EW). 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
QNU343EW	Estuarine water (Salinity 5 - 10 psu) spiked
QNU344EW	Estuarine water (Salinity 5 - 10 psu) spiked
QNU345EW	Low salinity seawater (Salinity 5 - 10 psu) spiked
QNU346EW	Unspiked Low salinity seawater (Salinity 5 - 10 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	0.002—2	0.01	6.0%	
Phosphate	µmol/L	1—15	0.01—5	0.05	6.0%	
Salinity	psu			0.01	0.1%	
Silicate	µmol/L	5—100	0.2—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	0.01—15	0.05	6.0%	

Please report Salinity only for QNU343EW

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	2019 - 2
AQ-14 DOC in seawater	
Test materials	QDC069SW, QDC070SW, QDC071EW and QDC072EW

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
QDC069SW	Seawater (Salinity > 30 psu)
QDC070SW	Seawater (Salinity > 30 psu) spiked
QDC071EW	Estuarine water (Salinity 5 - 10 psu) spiked
QDC072EW	Estuarine water (Salinity 5 - 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	2019 - 2
AQ-3 Metals in Seawater	
Test materials	QTM287SW, QTM288SW, QTM289SW and QTM290SW

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 at the coastal site near deltaparc "Neeltje Jans", the Netherlands .

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
QTM287SW	Unspiked Seawater (Salinity > 30 psu)
QTM288SW	Seawater (Salinity > 30 psu) spiked with metals
QTM289SW	Low salinity Seawater (Salinity 10 - 20 psu) spiked with metals
QTM290SW	Low salinity Seawater (Salinity 10 - 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
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
Arsenic	µg/L	Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
Boron	µg/L	0.2—10	0.05—5	0.5	12.5%	
Cadmium	µg/L	200—5000	1000—5000	0.4	12.5%	
Chromium	µg/L	0.05—1	0.001—0.5	0.005	12.5%	0.2
Cobalt	µg/L	0.5—10	0.01—5	0.1	12.5%	
Copper	µg/L	0.01—5	0.001—0.5	0.01	12.5%	
Iron	µg/L	0.2—10	0.05—10	0.2	12.5%	
Lead	µg/L	0.2—10	0.05—10	0.4	12.5%	
Manganese	µg/L	0.1—2	0.0002—15	0.01	12.5%	7.2
Nickel	µg/L	0.1—5	0.02—5	0.4	12.5%	
Silver	µg/L	0.1—2	0.2—5	0.2	12.5%	20
Tin	µg/L	0.1—2	0.02—2	0.2	12.5%	
Vanadium	µg/L	0.1—5	0.02—1	0.2	12.5%	
Zinc	µg/L	0.2—5	0.1—5	0.2	12.5%	

As the seawater is sampled at a new location, the background concentrations are not exactly known. Therefore, it is possible that the indicative range is incorrect for some of the determinands. QTM282SW 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	2019 - 2
AQ-4 Mercury in Seawater	
Test materials	QTM291SW, QTM292SW, QTM293SW and QTM294SW

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 at the coastal site near deltaparc "Neeltje Jans", the Netherlands .

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
QTM291SW	Seawater (Salinity > 30 psu) spiked with mercury
QTM292SW	Seawater (Salinity > 30 psu) spiked with mercury
QTM293SW	Low salinity Seawater (Salinity 10 - 20 psu) spiked with mercury
QTM294SW	Low salinity Seawater (Salinity 8 - 15 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
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
Mercury	ng/L	10 - 5000	0.2 -40	0.2	12.5%	50

As the seawater is sampled at a new location, the background concentrations is not exactly known. Therefore, it is possible that the indicative range is incorrect for mercury. QTM294SW 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	2019 - 2
AQ-11	Chlorophyll-a in Seawater
Test materials	QCH098SW and QCH099SW

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 seawater and freshwater. 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
QCH098SW	Filtered residue from 1 litre of freshwater
QCH099SW	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	2019 - 2
MS-1 Trace metals in Sediment	
Test materials	QTM128MS and QTM129MS

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
QTM128MS	Sediment (estuary)
QTM129MS	Sediment (estuary)

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	%	0.5—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%	
Barium-AE	mg/kg	50 - 1000	1	12.5%	
Barium-RT	mg/kg	50 - 1000	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%	
Cobalt-AE	mg/kg	1 - 50	1	12.5%	
Cobalt-RT	mg/kg	1 - 50	1	12.5%	
Copper-AE	mg/kg	1—500	1	12.5%	
Copper-RT	mg/kg	1—500	1	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%	
Magnesium-AE	mg/kg	2000 - 20000	1	12.5%	
Magnesium-RT	mg/kg	2000 - 20000	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	10—2500	10	12.5%	
Mercury-RT	µg/kg	10—2500	10	12.5%	
Molybdene-AE	mg/kg	2 - 1000	1	12.5%	
Molybdene-RT	mg/kg	2 - 1000	1	12.5%	
Nickel-AE	mg/kg	2—100	1	12.5%	
Nickel-RT	mg/kg	2—100	1	12.5%	
Phosporus-AE	mg/kg	100 - 2500	1	12.5%	
Phosphorus-RT	mg/kg	100 - 2500	1	12.5%	
Scandium-AE	mg/kg	1—20	0.1	12.5%	
Scandium-RT	mg/kg	1—20	0.1	12.5%	
Strontium-AE	mg/kg	50 - 500	1	12.5%	
Strontium-RT	mg/kg	50 - 500	1	12.5%	
Vanadium-AE	mg/kg	5 -500	1	12.5%	
Vanadium-RT	mg/kg	5 -500	1	12.5%	
Zinc-AE	mg/kg	20—1500	2.5	12.5%	
Zinc-RT	mg/kg	20—1500	2.5	12.5%	
TOC	%	0.2—10	0.1	12.5%	
Inorganic-carbonate	%	0.05—10	0.05	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	2019 - 2
MS-2 Chlorinated Organics in Sediment	
Test materials	QOR140MS and QOR141MS

Objective

This study covers the determination of chlorobiphenyls (CBs), organochlorine pesticides (OCPs) and total organic carbon in marine sediment test materials. Compared to former rounds quite some new determininands are added.

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
QOR140MS	Sediment (open sea)
QOR141MS	Sediment (estuary)

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

Determinand	Unit	Concentration Range	Error		AA-EQS
		Sediment	Const	Prop	
PCB28	µg/kg	0.1—100	0.025	12.5%	
PCB31	µg/kg	0.1—100	0.025	12.5%	
PCB52	µg/kg	0.1—500	0.025	12.5%	
PCB101	µg/kg	0.2—250	0.025	12.5%	
PCB105	µg/kg	0.1—50	0.025	12.5%	
PCB118	µg/kg	0.1—200	0.025	12.5%	
PCB138+PCB163	µg/kg	0.2—50	0.025	12.5%	
PCB138	µg/kg	0.2—100	0.025	12.5%	
PCB153	µg/kg	0.2—100	0.025	12.5%	
PCB156	µg/kg	0.05—5	0.025	12.5%	
PCB180	µg/kg	0.1—50	0.025	12.5%	
α-HCH	µg/kg	0.02—1	0.02	12.5%	
β-HCH	µg/kg	0.05—2	0.025	12.5%	
γ-HCH	µg/kg	0.05—2	0.025	12.5%	
δ-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%	
Dieldrin	µg/kg	0.1—10	0.025	12.5%	
pp'-DDD	µg/kg	0.1—25	0.025	12.5%	
pp'-DDE	µg/kg	0.1—20	0.025	12.5%	
op'-DDT	µg/kg	0.02—250	0.025	12.5%	

pp'-DDT	µg/kg	0.1—10	0.025	12.5%	
Transnonachlor	µg/kg	0.01—2	0.025	12.5%	
Heptachlor	µg/kg		0.025	12.5%	
Heptachlor-epoxide	µg/kg		0.025	12.5%	
TOC	%	0.2—10	0.02	12.5%	
PN	%		0.02	12.5%	
PCB18	µg/kg		t.b.d.	12.5%	
PCB44	µg/kg		t.b.d.	12.5%	
PCB47	µg/kg		t.b.d.	12.5%	
PCB49	µg/kg		t.b.d.	12.5%	
PCB66	µg/kg		t.b.d.	12.5%	
PCB110	µg/kg		t.b.d.	12.5%	
PCB128	µg/kg		t.b.d.	12.5%	
PCB141	µg/kg		t.b.d.	12.5%	
PCB149	µg/kg		t.b.d.	12.5%	
PCB151	µg/kg		t.b.d.	12.5%	
PCB158	µg/kg		t.b.d.	12.5%	
PCB170	µg/kg		t.b.d.	12.5%	
PCB183	µg/kg		t.b.d.	12.5%	
PCB187	µg/kg		t.b.d.	12.5%	
PCB194	µg/kg		t.b.d.	12.5%	
Emamectin	µg/kg		t.b.d.	12.5%	
Teflubenzuron	µg/kg		t.b.d.	12.5%	

t.b.d= to be decided

NB. The determinands given in blue, are new determinands added to the MS2 exercise.

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	2019 - 2
MS-3 Polycyclic Aromatic Hydrocarbons in Sediment	
Test materials	QPH103MS and QPH104MS

Objective

This study covers the determination of PAHs and total organic carbon in marine sediment test materials. Compared to former rounds quite some new determininands are added.

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
QPH103MS	Sediment (open sea)
QPH104MS	Sediment (estuary)

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

Determinand	Unit	Concentration Range	Error	
		Sediment	Const	Prop
Acenaphthene	µg/kg	2—2000	0.1	12.5%
Acenaphthylene	µg/kg	1—1000	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[k]fluoranthene	µg/kg	10—1000	0.1	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%
Chrysene	µg/kg	10—1500	0.2	12.5%
Chrysene+Triphenylene	µg/kg	10—3000	0.2	12.5%
Triphenylene	µg/kg	20—3000	0.5	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—4000	0.2	12.5%
Fluorene	µg/kg	2—1000	0.1	12.5%
Indeno[1,2,3-cd]pyrene	µg/kg	10—1500	0.2	12.5%
Naphthalene	µg/kg	10—4000	0.5	12.5%
1-methyl naphthalene	µg/kg		0.2	12.5%
2-methyl naphthalene	µg/kg		0.2	12.5%

2- methyl anthracene	µg/kg		0.2	12.5%
Perylene	µg/kg	10—500	0.2	12.5%
Phenanthrene	µg/kg	10—3000	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%
Pyrene	µg/kg	10—4000	0.2	12.5%
1-Methylpyrene	µg/kg	2—500	0.5	12.5%
TOC	%	0.2—10	0.02	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%
1-methylphenanthrene	µg/kg		t.b.d.	12.5%
1-methylanthracene	µg/kg		t.b.d.	12.5%
2-methylanthracene	µg/kg		t.b.d.	12.5%
1,2-benzodiphenylene sulfide	µg/kg		t.b.d.	12.5%
C1-dibenzothiophene	µg/kg		t.b.d.	12.5%
C2-dibenzothiophene	µg/kg		t.b.d.	12.5%
C3-dibenzothiophene	µg/kg		t.b.d.	12.5%
C1-Naphtalenes	µg/kg		t.b.d.	12.5%
C2-Naphtalenes	µg/kg		t.b.d.	12.5%
C3-Naphtalenes	µg/kg		t.b.d.	12.5%
Total petroleum hydrocarbons	µg/kg		t.b.d.	12.5%

t.b.d= to be decided

NB. The determinands given in blue, are new determinands added to the MS3 exercise.

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	2019 – 2
MS-6 Organotins in Sediment	
Test materials	QSP070MS and QSP071MS

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
QSP070MS	Sediment (river)
QSP071MS	Sediment (estuary)

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(DPhT)	µg Sn/kg	0.1—200	0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	1—500	0.1	12.5%	
Monophenyltin(MPhT)	µg Sn/kg	0.1—200	0.1	12.5%	
Tributyltin(TBT)	µg Sn/kg	1—500	0.1	12.5%	
Triphenyltin(TPhT)	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	2019 - 2
MS-7 Brominated flame retardants in Sediment	
Test materials	QBC060MS and QBC061MS

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
QBC060MS	Sediment (river)
QBC061MS	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

Determinand	Unit	Concentration range	Error	
		Sediment	Const	Prop
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%
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	2—2000	0.05	12.5%
TBBP-A	µg/kg		0.05	12.5%
Dimethyl-TBBP-A	µg/kg		0.05	12.5%
a-HBCD	µg/kg		0.05	12.5%
b-HBCD	µg/kg		0.05	12.5%
g-HBCD	µg/kg	0.01 - 20	0.05	12.5%
Total-HBCD	µg/kg	50—1000	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	2018 - 2
MS-8 PFASs in Sediment	
Test materials	QPF007MS and QPF008MS

Objective

This study covers the determination of perfluorinated alkyl substances (PFASs) 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
QPF007MS	Sediment (harbor sediment)
QPF008MS	Sediment (estuary sediment)

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	
PFOS	µg/kg		0.005	12.5%	
PFBA	µg/kg		0.005	12.5%	
PFPeA	µg/kg		0.005	12.5%	
PFHxA	µg/kg		0.005	12.5%	
PFHpA	µg/kg		0.005	12.5%	
PFOA	µg/kg		0.005	12.5%	
PFNA	µg/kg		0.005	12.5%	
PFUnA	µg/kg		0.005	12.5%	
PFDoA	µg/kg		0.005	12.5%	
PFTrA	µg/kg		0.005	12.5%	
PFTeA	µg/kg		0.005	12.5%	
L-PFBS	µg/kg		0.005	12.5%	
L-PFHxS	µg/kg		0.005	12.5%	
L-PFHps	µg/kg		0.005	12.5%	
PFOSA	µg/kg		0.005	12.5%	
PFDS	µg/kg		0.005	12.5%	
PFODA	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

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	2019 - 2
BT-1 Trace metals in Biota	
Test materials	QTM124BT and QTM125BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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
QTM124BT	Fish tissue (5-15% fat)
QTM125BT	Mussel tissue

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		EQS
		Fish Liver Tissue	Fish Muscle Tissue	Shellfish Tissue	Const	Prop	
Aluminium	mg/kg	1 – 100	0.5 – 10	2 – 50	0.2	12.5%	
Arsenic	mg/kg	1 - 5	1 - 10	0.2 - 10	0.02	12.5%	
Barium	µg/kg	5 – 500	5 – 500	200 – 10000	0.2	12.5%	
Cadmium	µg/kg	5—1000	1—50	10—500	20	12.5%	
Calcium	mg/kg	20 – 1000	50 – 5000	50 – 2000	10	12.5%	
Chromium	µg/kg	20—1000	50—500	10—5000	20	12.5%	
Cobalt	µg/kg	10 – 500	1 – 100	10 – 500	0.2	12.5%	
Copper	µg/kg	2000—10000	100—1000	50—10000	100	12.5%	
Iron	mg/kg	10 - 500	5 – 200	5 - 200	0.2	12.5%	

Lead	µg/kg	10—1000	5—50	10—1000	5	12.5%	
Magnesium	mg/kg	50 – 1000	50 - 1000	100 – 2000	10	12.5%	
Manganese	µg/kg	200 – 5000	50 – 5000	500 – 5000	0.2	12.5%	
Mercury	µg/kg	20—100	10—1000	2—500	2	12.5%	20
Molybdene	µg/kg	20 – 500	2 – 200	10 – 500	0.2	12.5%	
Nickel	µg/kg	20—1000	10—200	10—2000	20	12.5%	
Potassium	mg/kg	500 – 5000	500 – 5000	500 - 5000	10	12.5%	
Selenium	µg/kg	200—5000	50—2000	200—1000	10	12.5%	
Silver	µg/kg	20—1000	0.5—50	1—500	5	12.5%	
Sodium	mg/kg	200 - 5000	200 – 5000	1000 - 10000	10	12.5%	
Uranium	µg/kg	0.2 – 50	0.2 – 50	2 - 100	0.2	12.5%	
Vanadium	µg/kg	5 – 200	5 – 200	50 – 5000	0.2	12.5%	
Zinc	mg/kg	10—50	2—10	2—200	2	12.5%	
Ash-weight	%				0.1	12.5%	
Dry-weight	%				0.1	12.5%	
Total-Lipid	%				0.1	12.5%	
Extractable-Lipid	%				0.1	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, P, S, Sc, Ti, Ga, Rb, Sr, Y, Zr, Pd, Sn, Sb, Te, Cs, La, Ce, Nd, Ta, W, Pt, Au, Tl, Bi, Th. 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	2019 - 2
BT-2 Chlorinated Organics in Biota	
Test materials	QOR140BT and QOR141BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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 jars 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
QOR140BT	Turbot liver tissue
QOR141BT	Mussel tissue

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

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 analytes should be determined:

Determinand	Unit	Concentration range			Error		EQS
		Fish Liver tissue and Freshwater Fish	Fish Muscle Tissue	Shellfish Tissue	Const	Prop	
PCB28	µg/kg	0.5—50	0.05—5	0.05—5	0.025	12.5%	
PCB31	µg/kg	0.5—10	0.03—3	0.03—3	0.025	12.5%	
PCB52	µg/kg	2—100	0.05—20	0.05—5	0.025	12.5%	
PCB101	µg/kg	10—300	0.1—50	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	10—300	0.2—30	0.2—20	0.025	12.5%	
PCB138+PCB163	µg/kg	20—600	0.3—70	0.3—30	0.025	12.5%	
PCB138	µg/kg	20—600	0.3—70	0.3—30	0.025	12.5%	
PCB153	µg/kg	20—1000	0.4—100	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	5—200	0.05—20	0.05—5	0.025	12.5%	
α-HCH	µg/kg	0.05—5	0.05—5	0.05—5	0.02	12.5%	
β-HCH	µg/kg	0.1—5	0.05—5	0.05—5	0.025	12.5%	
γ-HCH	µg/kg	0.05—5	0.05—5	0.05—5	0.025	12.5%	
δ-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%	10
HCBD	µg/kg	0.05—5			0.025	12.5%	55
Dieldrin	µg/kg	0.5—100	0.2—20	0.2—20	0.025	12.5%	
pp'-DDD	µg/kg	0.5—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%	
Heptachlor	µg/kg				0.025	12.5%	0.0067
Heptachlor-epoxide	µg/kg				0.025	12.5%	0.0067
Total-Lipid	%				0.1	12.5%	
Extractable-Lipid	%				0.1	12.5%	
Cis-chlordane	µg/kg				t.b.d.	12.5%	
Trans-chlordane	µg/kg				t.b.d.	12.5%	
Oxychlordane	µg/kg				t.b.d.	12.5%	
Dicofol	µg/kg				t.b.d.	12.5%	

t.b.d= to be decided

NB. The determinands given in blue, are new determinands added to the BT2 exercise.

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	2019 - 2
BT-4 Polycyclic Aromatic Hydrocarbons in Biota	
Test materials	QPH095BT and QPH096BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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
QPH095BT	Shellfish tissue (mussel)
QPH096BT	Shellfish tissue (mussel)

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

Precaution

The jars with biological tissue 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 minimizes 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. 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	
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 - 20	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%	5
Benzo[b]fluoranthene	µg/kg	0.2 - 10	0.2	12.5%	

Benzo[k]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 - 10	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%	
Triphenylene	µg/kg	0.1 - 10	0.5	12.5%	
Dibenz[a,h]anthracene	µg/kg	0.2 - 5	0.1	12.5%	
Dibenzo[a,i]pyrene	µg/kg		0.5	12.5%	
Dibenzothiophene	µg/kg	0.2 - 5	0.5	12.5%	
Fluoranthene	µg/kg	5 - 50	0.2	12.5%	30
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%	
1-methyl naphthalene	µg/kg		0.2	12.5%	
2-methyl naphthalene	µg/kg		0.2	12.5%	
2- methyl anthracene	µg/kg		0.2	12.5%	
Perylene	µg/kg	0.1 - 5	0.5	12.5%	
Phenanthrene	µg/kg	2 - 50	0.2	12.5%	
2-Methylphenanthrene	µg/kg	0.2 - 20	2	12.5%	
3,6-Dimethylphenanthrene	µg/kg	0.2 - 10	0.5	12.5%	
Pyrene	µg/kg	1 - 50	0.2	12.5%	
1-Methylpyrene	µg/kg		2	12.5%	
Benzo Fluoranthenes (a+b+j+k)	µg/kg		0.2	12.5%	
Total-Lipid	%		0.1	12.5%	
Extractable-Lipid	%		0.1	12.5%	
C1-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C2-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C3-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C1-pyrenes/fluoranthenes	µg/kg		0.2	12.5%	
C2-pyrenes/fluoranthenes	µg/kg		0.2	12.5%	
C1-chrysenes	µg/kg		0.2	12.5%	
C2-chrysenes	µg/kg		0.2	12.5%	
C1-benzofluoranthenes	µg/kg		0.2	12.5%	
1-methylphenanthrene	µg/kg		t.b.d.	12.5%	
1-methylanthracene	µg/kg		t.b.d.	12.5%	
1,2-benzodiphenylene sulfide	µg/kg		t.b.d.	12.5%	
C1-dibenzothiophene	µg/kg		t.b.d.	12.5%	
C2-dibenzothiophene	µg/kg		t.b.d.	12.5%	
C3-dibenzothiophene	µg/kg		t.b.d.	12.5%	
Total petroleum hydrocarbons	µg/kg		t.b.d.	12.5%	

t.b.d= to be decided

NB. The determinands given in blue, are new determinands added to the BT4 exercise.

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	2019 - 2
BT-8 Organotins in Biota	
Test materials	QSP071BT and QSP072BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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
QSP071BT	Shellfish tissue (mussel)
QSP072BT	Shellfish tissue (mussel)

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

Precaution

The jars with biological tissue 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 minimizes 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 analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
Tributyltin(TBT)	µg Sn/kg	0.2 - 50	0.1	12.5%	
Dibutyltin(DBT)	µg Sn/kg	0.1 - 10	0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	0.5 - 30	0.1	12.5%	
Triphenyltin(TPT)	µg Sn/kg	0.1 – 10	0.1	12.5%	
Diphenyltin(DPT)	µg Sn/kg	0.1 – 5	0.1	12.5%	
Monophenyltin(MPT)	µg Sn/kg	0.1 - 5	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	2019 - 2
BT-9 Brominated Flame Retardants in Biota	
Test materials	QBC061BT and QBC062BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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
QBC061BT	Mussel tissue
QBC062BT	Freshwater fish tissue

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

Precaution

The jars with biological tissue are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. The 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 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%	0.0085
BDE47	µg/kg	0.05 - 40	0.005	12.5%	0.0085
BDE49	µg/kg		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%	0.0085
BDE100	µg/kg	0.005 - 10	0.005	12.5%	0.0085
BDE153	µg/kg	0.01 - 2	0.005	12.5%	0.0085
BDE154	µg/kg	0.001 - 5	0.005	12.5%	0.0085
BDE183	µg/kg	0.001 – 1	0.005	12.5%	
BDE209	µg/kg	0.01 - 1	0.005	12.5%	
TBBP-A	µg/kg	0.01 - 1	0.005	12.5%	
Dimethyl-TBBP-A	µg/kg		0.005	12.5%	
α-HBCD	µg/kg	0.01 – 1	0.005	12.5%	
β-HBCD	µg/kg	0.01 – 1	0.005	12.5%	
γ-HBCD	µg/kg	0.01 – 1	0.005	12.5%	
Total-HBCD	µg/kg	0.01 – 2	0.005	12.5%	167
BTBPE	µg/kg		t.b.d.	12.5%	
DBDPE	µg/kg		t.b.d.	12.5%	
HBBz	µg/kg		t.b.d.	12.5%	

t.b.d= to be decided

NB. The determinands given in blue, are new determinands added to the BT4 exercise.

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	2019 - 2
BT-10 Perfluorinated Alkyl Substances (PFASs) in Biota	
Test materials	QPF016BT and QPF017BT

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 jars contains approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Representative sub samples were dispensed into jars 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
QPF016BT	Whole fish tissue
QPF017BT	Shellfish tissue (mussel)

Precaution

The jars with biological tissue 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 analytes should be determined:

Determinand	Unit	Concentration Range	Error		AA-EQS
		Biota	Const	Prop	
n-PFOS	µg/kg	0.1 - 1000	0.1	12.5%	9.1
PFBA	µg/kg	0.01 - 2	0.1	12.5%	
PFPeA	µg/kg	0.01 – 2	0.1	12.5%	
PFHxA	µg/kg	0.01 – 2	0.1	12.5%	
PFHpA	µg/kg	0.01 – 2	0.1	12.5%	
PFOA	µg/kg	0.01 – 5	0.1	12.5%	
PFNA	µg/kg	0.01 – 5	0.1	12.5%	
PFDA	µg/kg	0.01 – 10	0.1	12.5%	

PFUnA	µg/kg	0.01 - 10	0.1	12.5%	
PFDoA	µg/kg	0.01 – 5	0.1	12.5%	
PFTrA	µg/kg	0.01 - 5	0.1	12.5%	
PFTeA	µg/kg	0.01 - 5	0.1	12.5%	
L-PFBS**	µg/kg	0.01 - 10	0.1	12.5%	
L-PFHxS**	µg/kg	0.01 - 5	0.1	12.5%	
L-PFHpS**	µg/kg	0.01 – 5	0.1	12.5%	
PFOSA	µg/kg	00.1 - 50	0.1	12.5%	
PFDS	µg/kg		0.1	12.5%	
PFODA	µg/kg		0.1	12.5%	
Total-PFOS	µg/kg	0.1 – 1000	0.1	12.5%	9.1

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	2019 - 2
BT-7 ASP Shellfish Toxins	
Test materials	QST269BT, QST270BT and QST271BT

Objective

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

Test Materials and storage

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

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 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
QST269BT	Shellfish tissue (oyster tissue)
QST270BT	Shellfish tissue (scallop tissue)
QST271BT	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	0.2 - 100	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 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	2019 - 2
BT-11 Lipophilic Shellfish Toxins	
Test materials	QST272BT, QST273BT and QST274BT

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
QST272BT	Shellfish tissue (Mussel)
QST273BT	Shellfish tissue extract (Mussel)
QST274BT	Shellfish tissue (Mussel)

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	
Free-Okadaic-Acid	µg/kg	0.5 - 500	0.1	12.5%	
Free-DTX1	µg/kg	0.2 - 500	0.1	12.5%	
Free-DTX2	µg/kg	0.5 - 1000	0.1	12.5%	
Total-Free-OA+DTX1+DTX2	µg OA eq./kg	0.5 - 1000	0.1	12.5%	
Total-Okadaic-Acid	µg/kg	0.5 – 500	0.1	12.5%	
Total-DTX1	µg/kg	0.5 – 1000	0.1	12.5%	
Total-DTX2	µg/kg	0.5 – 1000	0.1	12.5%	
Total-hy-OA+DTX1+DTX2	µg OA eq./kg	0.5 – 1000	0.1	12.5%	
PTX-1	µg/kg	0.5 – 20	0.1	12.5%	
PTX-2	µg/kg	0.5 - 50	0.1	12.5%	
Total OA group and PTX group	µg OA eq./kg	0.5 - 1000	0.1	12.5%	
AZA-1	µg/kg	0.5 - 1500	0.1	12.5%	
AZA-2	µg/kg	0.5 – 500	0.1	12.5%	
AZA-3	µg/kg	0.5 - 500	0.1	12.5%	
AZA-total	µg AZA eq./kg	0.5 - 5000	0.1	12.5%	
YTX	mg/kg	0.01 – 2	0.02	12.5%	
homo-YTX	mg/kg	0.5 – 5	0.02	12.5%	
45-OH-homo-YTX	mg/kg	0.5 – 5	0.02	12.5%	
45-OH-YTX	mg/kg	0.5 – 2	0.02	12.5%	
YTX-total	mg YTX eq./kg	0.01 – 10	0.02	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

	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.

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 **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.

ROUND	2019 - 2
BT-12 PSP Shellfish Toxins	
Test materials	QST275BT, QST276BT and QST277BT

Objective

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

Test Materials and storage

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

Shellfish tissue test materials are supplied in a plastic 5ml vial, each vial contains sufficient material for one-shot analysis of the paralytic shellfish 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 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 test materials 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
QST275BT	Shellfish tissue (Mussel)
QST276BT	Shellfish tissue (Mussel)
QST277BT	Shellfish tissue (Mussel)

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 final (total toxicity) result for each test material should be reported as µg STX dihydrochloride equivalents/kg (such that HPLC, MBA and ELISA results are comparable).
Participants using HPLC methods should also report each PSP analogue identified and give individual analogue concentrations in µmol/kg sample.

Participants using HPLC methods should use the specific toxicities as they appear in the EFSA Scientific Opinion of the Panel on Contaminants in the Food Chain for Marine Biotoxins in shellfish: STX group (see below).

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
11-OH-STX	µmol/kg		0.1	12.5%	
C1	µmol/kg		0.1	12.5%	
C1,2	µmol/kg		0.1	12.5%	
C2	µmol/kg		0.1	12.5%	
C3	µmol/kg		0.1	12.5%	
C3,4	µmol/kg		0.1	12.5%	
C4	µmol/kg		0.1	12.5%	
dc-GTX1	µmol/kg		0.1	12.5%	
dc-GTX1,4	µmol/kg		0.1	12.5%	
dc-GTX2	µmol/kg		0.1	12.5%	
dc-GTX2,3	µmol/kg		0.1	12.5%	
dc-GTX3	µmol/kg		0.1	12.5%	
dc-GTX4	µmol/kg		0.1	12.5%	
dc-NEO	µmol/kg		0.1	12.5%	
dc-STX	µmol/kg		0.1	12.5%	
GTX-1	µmol/kg		0.1	12.5%	
GTX-2	µmol/kg		0.1	12.5%	
GTX-3	µmol/kg		0.1	12.5%	
GTX-4	µmol/kg		0.1	12.5%	
GTX-5	µmol/kg		0.1	12.5%	
GTX-6	µmol/kg		0.1	12.5%	
NEO	µmol/kg		0.1	12.5%	
STX	µmol/kg		0.1	12.5%	
Total toxicity	µgSTXdiHCl-eq/kg		2	12.5%	
GTX-2,3	µmol/kg		0.1	12.5%	
GTX-1,4	µmol/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.

TEFs recommended by the EFSA

Determinand	TEF
STX	1
NeoSTX	1
GTX-1	1
GTX-2	0.4
GTX-3	0.6
GTX-4	0.7
GTX-5	0.1
GTX-6	0.1
C2	0.1
C4	0.1
dc-STX	1
dc-NeoSTX	0.4
dc-GTX-2	0.2
dc-GTX-3	0.4

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 STX analogues individually and as total STX-equivalents. 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 (If participants are using the AOAC 2005.06 method then those toxins that co-elute (eg GTX1 and GTX-4, GTX-2 and GTX-3, dcGTX-2 and dcGTX-3 or C-1 and C-2) must be reported using the higher toxicity factor of the two isomers. For example if participants find the presence of GTX-1,4 (co-eluting) in the sample then they should report the sum of the two isomers in the GTX-1,4 column. 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 TEQ values on the basis of wet weight of the test material as provided. The weight of the shellfish tissue test material 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	2019 - 2
DE-16 Tetrodotoxin in shellfish	
Test materials	QTT001SS; QTT002EX; QTT003BT; QTT004BT and QTT005BT

Objective

This study covers the determination of tetrodotoxin in shellfish tissue test materials.

Test Materials and storage

The test materials were supplied by the Wageningen Food Safety Research (WFSR), Wageningen, the Netherlands.

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 standard solution, shellfish extract and shellfish tissue homogenates 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.

QTT001SS	Standard solution of tetrodotoxin in ACN/MeOH/0.03M HAc (v/v 65:17.5:17.5)
QTT002EX	Extract (ACN/MeOH/0.03M HAc (v/v 65:17.5:17.5)) of shellfish tissue (mussel tissue)
QTT003BT	Shellfish tissue (oyster tissue)
QTT004BT	Shellfish tissue natural contaminated with tetrodotoxin (mussel tissue)
QTT005BT	Shellfish tissue natural contaminated with tetrodotoxin (oyster 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

Determinand	Unit	Concentration Range	Error		AA-EQS
		Shellfish Tissue	Const	Prop	
Tetrodotoxin	µg/kg	10 - 200	0.1	12.5%	

Tetrodotoxin in the standard solution and in the extract shall be reported in µg/L!!

Results should be reported for as many of these samples 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 tetrodotoxin. The results of tetrodotoxin in the shellfish tissue samples should be expressed on the test material "as received" i.e.

on a wet weight basis. The weight of shellfish tissue test materials should be determined prior to analysis. The concentrations should be determined against your own calibration solutions. The results of the tetrodotoxin in the standard solution as well as in the extract should be reported in µg/L.

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 tetrodotoxin. 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.

Reporting

One result for tetrodotoxin 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 2020**. 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 1 March 2020. 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

Fax No : +31(0)317 486 546
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 Ultra Turrax for another 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].

[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 HClO4 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.

- Add 20 ml cyclohexane containing 13%(w/w) isopropanol and mix for one minute by Ultra Turrax.
- 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.