Quasimeme Laboratory Performance Studies

Round 72

1 March 2013 to 1 June 2013

Exercise Protocols

Version 3 (15May 2013)

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Introduction Round 72

Thank you for participating in the 2013 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round72 that you have ordered will be sent to you by courier in the week of March 2013. 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	Exercise	Analysis Group Code		
72	996	AQ-1	Seawater	Nutrients
72	997	AQ-2	Estuarine and Low Salinity Open Water	Nutrients
72	1007	AQ-14	Seawater	DOC
72	1014	MS-1	Sediment	Trace Metals
72	1015	MS-2	Sediment	Chlorinated Organics
72	1016	MS-3	Sediment	Polycyclic Aromatic Hydrocarbons
72	1017	MS-6	Sediment	Organotins
72	1018	MS-7	Sediment	Brominated Flame Retardants
72	1008	BT-1	Fish or Shellfish	Trace Metals
72	1009	BT-2	Fish or Shellfish	Chlorinated Organics
72	1010	BT-4	Shellfish	Polycyclic Aromatic Hydrocarbons
72	1011	BT-8	Biota	Organotins
72	1012	BT-9	Fish or Shellfish	Brominated Flame Retardants
72	1013	BT-10	Fish or Shellfish	Perfluorinated Alkyl Substances (PFASs)
72	998	AQ-3	Seawater	Metals
72	999	AQ-4	Seawater	Mercury
72	1000	AQ-5	Seawater	Halogenated Organics
72	1001	AQ-6	Seawater	Volatile Organics
72	1002	AQ-7	Seawater	Pentachlorophenol
72	1003	AQ-8	Seawater	Triazines and organophosphorus compounds
72	1004	AQ-11	Seawater Filter	Chlorophyll and Pheopigments
72	1005	AQ-12	Seawater	Organotins
72	1006	AQ-13	Seawater	Polycyclic Aromatic Hydrocarbons
72 & 73	1020	BT-7	Shellfish and Solution	ASP Shellfish Toxins

72 & 73	1021	DE-10	Shellfish and Solution	DSP Shellfish Toxins
72	1022	DE-14	Shellfish and Solution	PSP Shellfish Toxins

All data for these studies must be uploaded to your Quasimeme SharePoint Site, using the data submission forms, no later than 1 June 2013

All other information should be sent to: QUASIMEME Project Office

QUASIMEME Project Office Wageningen UR Alterra CWK P.O. Box 47 6700 AA Wageningen The Netherlands	Website: http://www.Quasimeme.org Tel.: +31 (0) 317 48 65 46 Fax: +31 (0) 317 41 90 00 E-mail: Quasimeme@wur.nl

ROUND	72	Exercise 996
AQ-1 Nutrients in Seawater		
Test materials	QNU249SW, QNU250SW, QNU251SW	

Objective

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

Test Materials and storage

The test materials were prepared at the MUMM Laboratory, Ostend, Belgium, using seawater collected from the Atlantic Ocean aboard of the R.V. Belgica.

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.

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.

Code	Description
QNU249SW	Seawater (Salinity > 30 psu)
QNU250SW	Seawater (Salinity > 30 psu) spiked
QNU251SW	Seawater (Salinity > 30 psu) spiked

Precaution

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

Determinands and concentration ranges

The following nutrients should be determined:

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

Data assessment for unspiked samples will be carried out by calculating with a proportional error of 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

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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 997
AQ-2 Nutrients in Estuarine and low salinity open seawater		
Test materials	QNU252EW, QNU253EW, QNU254EW, QNU255EW	

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 MUMM Laboratory, Ostend, Belgium, using seawater collected from the Atlantic Ocean (Estuarine water samples) 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 10-15 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.

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.

Code	Description
QNU252EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU253EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU254EW	Low salinity open water (Salinity 8 - 20 psu) spiked
QNU255EW	Low salinity open water (Salinity 8 - 20 psu)

Precaution

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

Determinands and concentration ranges

The following nutrients should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Estuarine water (spiked)	Low salinity open water (spiked)	Const	Prop	
Ammonia	µmol/L	2—50	0.2—5	0.1	6.0%	
Nitrite	µmol/L	0.5—25	0.01—2	0.01	6.0%	
Phosphate	µmol/L	1—15	0.02—5	0.05	6.0%	
Salinity	psu			0.001	0.1%	
Silicate	µmol/L	5—100	0.5—20	0.1	6.0%	
Total-N	µmol/L	10—200	2—20	0.5	6.0%	
Total-P	µmol/L	1—20	0.02—2	0.05	6.0%	
TOxN	µmol/L	10—100	0.05—15	0.05	6.0%	

Data assessment for unspiked samples will be carried out by calculating with a proportional error of 12.5%.

Salinity is an indicative measurement in support of methodology.
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 forms which are placed on the sharepointsite.

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 to send you an additional data submission form.

ROUND	72	Exercise 1007
AQ-14 DOC in seawater		
Test materials	QDC017SW, QDC018SW, QDC019EW, QDC020EW	

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 MUMM Laboratory, Ostend, Belgium, using seawater collected from the Atlantic Ocean (Seawater samples)

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 10-15 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.

Code	Description
QDC017SW	Seawater (Salinity > 30 psu)
QDC018SW	Seawater (Salinity > 30 psu) spiked
QDC019EW	Estuarine water (Salinity 8 - 20 psu) spiked
QDC020EW	Estuarine water (Salinity 8 - 20 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%	

Data assessment for unspiked samples will be carried out by calculating with a proportional error of 12.5%

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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 998
AQ-3 Metals in Seawater		
Test materials	QTM187SW, QTM188SW, QTM189SW	

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 Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys.

The test materials were prepared in bulk in a 100 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.

Code	Description
QTM187SW	Seawater (Salinity > 30 psu)
QTM188SW	Seawater (Salinity > 30 psu) spiked
QTM189SW	Low salinity Seawater (Salinity 8 - 20 psu) spiked

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.

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

Determinands and concentration ranges

The following metals should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Seawater (spiked)	Low salinity Seawater (spiked)	Const	Prop	
Arsenic	µg/L	0.05—5	0.2—10	0.5	12.5%	
Boron	µg/L	1000—5000	200—5000	0.4	12.5%	
Cadmium	µg/L	0.001—0.5	0.05—1	0.005	12.5%	0.08
Chromium	µg/L	0.01—5	0.5—10	0.1	12.5%	
Cobalt	µg/L	0.001—0.5	0.01—10	0.2	12.5%	
Copper	µg/L	0.05—5	0.2—10	0.2	12.5%	
Iron	µg/L	0.05—10	0.2—20	0.4	12.5%	
Lead	µg/L	0.0002—15	0.1—5	0.01	12.5%	7.2
Manganese	µg/L	0.02—2	0.1—5	0.4	12.5%	
Nickel	µg/L	0.2—5	0.1—5	0.2	12.5%	20
Silver	µg/L	0.02—2	0.1—5	0.2	12.5%	
Tin	µg/L	0.02—1	0.1—5	0.2	12.5%	
Vanadium	µg/L	0.1—5	0.2—5	0.2	12.5%	
Zinc	µg/L	0.5—20	0.2—10	0.4	12.5%	

Note that the indicative range for some determinands in the spiked low salinity sample are higher compared to the range given in the Quasimeme guide.
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.

ROUND	72	Exercise 999
AQ-4 Mercury in Seawater		
Test materials	QTM190SW, QTM191SW, QTM192SW	

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 sharepointsite.

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys.

The test materials were prepared in bulk in a 100 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.

Code	Description
QTM190SW	Seawater (Salinity > 30 psu) spiked
QTM191SW	Seawater (Salinity > 30 psu) spiked
QTM192SW	Seawater (Salinity > 30 psu) spiked

Precaution

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

Determinands and concentration ranges

Mercury should be determined in each test material.

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

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 sharepointsite

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.

ROUND	72	Exercise 1000
AQ-5 Halogenated Organics in Seawater		
Test materials	QOC076SS, QOC076SW, QOC077SS, QOC077SW, QOC078SS, QOC078SW	

Objective

This study covers the determination of Halogenated organics in seawater test materials.

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Low salinity seawater test material are prepared by dilution with ultra-pure demineralised water, to a salinity of approximately 12 - 18 psu. Test materials need to be spiked with organochlorine composite solutions in methanol by the participants themselves (see Analysis section). 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. Approximately 1 litre of each test material is provided. 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.

Code	Description
QOC076SS	Spiking solution to use for QOC076SW
QOC076SW	Seawater with Spiking solution
QOC077SS	Spiking solution to use for QOC077SW
QOC077SW	Seawater with Spiking solution
QOC078SS	Spiking solution to use for QOC078SW
QOC078SW	Low salinity Seawater with Spiking solution

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 organochlorine compounds should be determined:

Determinand	Unit	Concentration Range		Error		AA-EQS
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
α-HCH	ng/L	2—50	0.2—20	0.2	12.5%	2
β-HCH	ng/L	1—50	0.2—20	0.2	12.5%	2
γ-HCH	ng/L	2—50	0.5—20	0.2	12.5%	2
δ-HCH	ng/L	1—50	0.2—20	0.2	12.5%	2
HCB	ng/L	0.5—20	0.1—10	0.2	12.5%	10
HCBD	ng/L	2—50	0.2—20	0.2	12.5%	100
Aldrin	ng/L	2—200	1—20	0.5	12.5%	5
Dieldrin	ng/L	2—100	1—20	0.5	12.5%	5
Endrin	ng/L	2—200	1—20	0.5	12.5%	5
Isodrin	ng/L	2—200	1—20	0.5	12.5%	5

pp'-DDD	ng/L	1—50	0.1—10	0.5	12.5%	25
pp'-DDE	ng/L	1—50	0.2—10	0.5	12.5%	25
op'-DDT	ng/L	1—50	0.2—20	0.5	12.5%	25
pp'-DDT	ng/L	1—50	0.2—20	0.5	12.5%	10
Endosulphan-I	ng/L	1—20	0.2—10	0.2	12.5%	0.5
Endosulphan-II	ng/L	0.5—20	0.1—10	0.2	12.5%	0.5
Pentachlorobenzene	ng/L	2—100	0.2—5	0.5	12.5%	0.7
1,2,3-TCB	ng/L	2—50	1—20	0.5	12.5%	400
1,2,4-TCB	ng/L	5—100	1—20	0.5	12.5%	400
1,3,5-TCB	ng/L	2—50	0.5—20	0.5	12.5%	400
Trifluralin	ng/L	2—50	0.5—20	0.5	12.5%	30

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

Preparation and Analysis

A 2000-times (approximately) dilution of the spiking solutions is required, using the seawater test materials to produce the spiked seawater. The dilution procedure is given below:

Seawater test material should be used to dilute spiking solution with the corresponding number.

- The spiking solution should be stabilised at 20°C

- Weigh 0.5 ml of spiking solution prior to dilution. The use of a positive displacement pipette or syringe is recommended. Note that the density of the spiking solution is approximately 0.79 kg/L.

- Weigh an empty 1l volumetric flask. Weight an aliquot of the seawater provided, in the flask.

- Add 0.5 ml of the spiking solution to the flask. Make up to 1000 gram with the seawater provided and mix thoroughly. A mass of 1000 gram of water is equal to 975 ml of seawater. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Measure the final weight of the diluted solution prior to extraction.

- Analyse the test materials immediately after preparation.

- Record the weights in the data submission form along with the spike / sample weight ratio. These results will not be used for statistical analysis but will be used for control purposes by the Quasimeme Project team. This information will assist QUASIMEME in identifying any manipulation errors in the sample preparation prior to the analysis. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1001
AQ-6 Volatile Organics in Seawater		
Test materials	QVC053SW, QVC054SW	

Objective

This study covers the determination of volatile organochlorine compounds in seawater testmaterials.

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Test materials were spiked with the volatile organochlorine composite solution in methanol with known concentration. Flasks were completely filled with test material. 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.

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

Code	Description
QVC053SW	Seawater (Salinity > 30 psu) spiked
QVC054SW	Seawater (Salinity > 30 psu) spiked

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 analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Seawater (spiked)	Const	Prop	
1,1,1-Trichloroethane	µg/L	0.2—10	0.1	12.5%	
1,1,2-Trichloroethane	µg/L	1—20	0.1	12.5%	
1,2-Dichloroethane	µg/L	1—10	0.1	12.5%	10
Benzene	µg/L	0.2—50	0.1	12.5%	8
Carbontetrachloride	µg/L	0.2—10	0.1	12.5%	12
Chloroform	µg/L	0.5—20	0.1	12.5%	2.5
Dichloromethane	µg/L	0.2—20	0.1	12.5%	20
Tetrachloroethene	µg/L	0.2—10	0.1	12.5%	10
Trichloroethene	µg/L	0.2—10	0.1	12.5%	10

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

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 the volatiles 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 forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1002
AQ-7 Pentachlorophenol in Seawater		
Test materials		QPP051SS, QPP051SW, QPP052SS, QPP052SW, QPP053SS, QPP053SW

Objective

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

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Test materials need to be spiked with pentachlorophenol solutions in methanol by the participants themselves (see Analysis section). 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. Approximately 1 litre of each test material is provided. 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.

Code	Description
QPP051SS	Spiking solution to use for QPP051SW
QPP051SW	Seawater with Spiking solution
QPP052SS	Spiking solution to use for QPP052SW
QPP052SW	Seawater with Spiking solution
QPP053SS	Spiking solution to use for QPP053SW
QPP053SW	Seawater with Spiking solution

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

Pentachlorophenol should be determinated in each test material.

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
Pentachlorophenol	ng/L	20-2000	10	12.5%	400

Analysis

Preparation and Analysis

A 2000-times (approximately) dilution of the spiking solutions is required, using the seawater test materials to produce the spiked seawater. The dilution procedure is given below:

Seawater test material should be used to dilute spiking solution with the corresponding number.

- The spiking solution should be stabilised at 20˚C

- Weigh 0.5 ml of spiking solution prior to dilution. The use of a positive displacement pipette or syringe is recommended. Note that the density of the spiking solution is approximately 0.79 kg/L.

- Weigh an empty 1l volumetric flask. Weight an aliquot of the seawater provided, in the flask.

- Add 0.5 ml of the spiking solution to the flask. Make up to 1000 gram with the seawater provided and mix thoroughly. A mass of 1000 gram of water is equal to 975 ml of seawater. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Measure the final weight of the diluted solution prior to extraction.

- Analyse the test materials immediately after preparation.

- Record the weights in the data submission form along with the spike / sample weight ratio. These results will not be used for statistical analysis but will be used for control purposes by the Quasimeme Project team. This information will assist QUASIMEME in identifying any manipulation errors in the sample preparation prior to the analysis. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Reporting

One result for each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1003
AQ-8 Triazines and organophosphorus compounds in the seawater		
Test materials	QTP082SS, QTP082SW, QTP083SS, QTP083SW, QTP084SS, QTP084SW	

Objective

This study covers the determination of triazines and organophosphorus compounds in the seawater.

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter.

Methanol solutions containing known concentrations of organophosphorus compounds and triazines were prepared in bulk and ampouled to make the spiking solutions.

For each test material, approximately 1 litre of filtered seawater and an ampoule of spiking solution is provided.

Homogeneity of the test materials is assumed, as they were prepared from the same bulk seawater, and the spiking solutions were also prepared in bulk. The test materials are stable for the purposes of the exercise. Test materials (seawater and spiking solutions) 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.

Code	Description
QTP082SS	Spiking solution to use for QTP082SW
QTP082SW	Seawater with Spiking solution
QTP083SS	Spiking solution to use for QTP083SW
QTP083SW	Seawater with Spiking solution
QTP084SS	Spiking solution to use for QTP084SW
QTP084SW	Low salinity Seawater with Spiking solution

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 analytes should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Seawater with SS	Low salinity Seawater with SS	Const	Prop	
Alachlor	ng/L	2—200	20—500	1	12.5%	300
Atrazine	ng/L	5—200	20—500	1	12.5%	600
Azinphos-ethyl	ng/L	5—200	20—500	1	12.5%	
Azinphos-methyl	ng/L	5—200	20—500	1	12.5%	
Chlorfenvinphos	ng/L	5—200	20—500	1	12.5%	100
Chlorpyriphos	ng/L	2—200	20—500	1	12.5%	30
Coumaphos	ng/L	2—100	20—500	1	12.5%	
Demeton	ng/L	5—200	50—500	1	12.5%	

Diazinon	ng/L	5—200	20—500	1	12.5%	
Dichlorvos	ng/L	2—200	20—500	1	12.5%	
Dimethoate	ng/L	5—100	20—500	1	12.5%	
Diuron	ng/L	5—200	50—500	1	12.5%	200
Fenchlorphos	ng/L	2—200	20—500	1	12.5%	
Fenitrothion	ng/L	2—200	20—500	1	12.5%	
Fenthion	ng/L	5—200	20—500	1	12.5%	
Irgarol-1051	ng/L	2—200	50—500	1	12.5%	
Isoproturon	ng/L	2—200	20—500	1	12.5%	300
Malathion	ng/L	5—200	20—500	1	12.5%	
Omethoate	ng/L	5—200	50—500	1	12.5%	
Parathion-ethyl	ng/L	5—200	20—500	1	12.5%	
Parathion-methyl	ng/L	5—200	20—500	1	12.5%	
Simazine	ng/L	5—200	20—500	1	12.5%	1000
Triazophos	ng/L	10—500	50—500	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

Preparation and Analysis

A 2000-times (approximately) dilution of the spiking solutions is required, using the seawater test materials to produce the spiked seawater. The dilution procedure is given below:

Seawater test material should be used to dilute spiking solution with the corresponding number.

- The spiking solution should be stabilised at 20°C

- Weigh 0.5 ml of spiking solution prior to dilution. The use of a positive displacement pipette or syringe is recommended. Note that the density of the spiking solution is approximately 0.79 kg/L.

- Weigh an empty 1l volumetric flask. Weight an aliquot of the seawater provided, in the flask.

- Add 0.5 ml of the spiking solution to the flask. Make up to 1000 gram with the seawater provided and mix thoroughly. A mass of 1000 gram of water is equal to 975 ml of seawater. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Measure the final weight of the diluted solution prior to extraction.

- Analyse the test materials immediately after preparation.

- Record the weights in the data submission form along with the spike / sample weight ratio. These results will not be used for statistical analysis but will be used for control purposes by the Quasimeme Project team. This information will assist QUASIMEME in identifying any manipulation errors in the sample preparation prior to the analysis. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

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

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1004
AQ-11	Chlorophyll-a in Seawater	
Test materials	QCH060SW and QCH061SW	

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 Alterra, Wageningen the Netherlands. Test materials were prepared from cultures of Isochrysis + Chaetocheros + Pyramimonas (QCH061SW) The QCH060SW sample is a blank seawater sample spiked with algae culture neochloris oleoabundans. 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.

Code	Description
QCH060SW	Filtered residues from 1 litre of seawater spiked with algae culture
QCH061SW	Filtered residues 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%	

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.**

Whilst you should use your normal validated methods and procedures to analyse the test materials in this study, previous QUASIMEME development exercises have shown that the best between laboratory agreement was obtained with either the Trichromatic method (Jeffrey and Humphrey 1975) or the Monochromatic method (Lorenzen 1967).

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1005
AQ-12 Organotins in Seawater		
Test materials	QSP037SW, QSP038SW and QSP038SS	

Objective

This study covers the determination of organotin compounds in the seawater test materials QSP037SW and QSP038SW. Testmaterial QSP038SW has to be spiked in your own laboratory. The sample QSP038SW sample is not preserved, as it will be spiked in your own laboratory. The QSP037SW sample is preserved with hydrochloric acid.

Test Materials and storage

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

It is important that the vial containing the spike solution is used or opened only when you are ready to complete the analysis. Please check the vial to ensure it has not been damaged during transit.

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

Code	Description
QSP037SW	Seawater (Spiked)
QSP038SW	Seawater
QSP038SS	Spiking solution to use for QSP038SW

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 analytes should be determined:

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.

As a guide, the concentrations of the organotin compounds in the spiked test materials are within the following ranges:

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
Dibutyltin(DBT)	ng Sn/kg	0.001—0.05	0.05	12.5%	0.2
Diphenyltin(DPT)	ng Sn/kg	0.001—0.1	0.05	12.5%	
Monobutyltin(MBT)	ng Sn/kg	0.001—0.02	0.05	12.5%	0.2
Monophenyltin(MPT)	ng Sn/kg	0.001—0.05	0.05	12.5%	
Tributyltin(TBT)	ng Sn/kg	0.001—0.1	0.05	12.5%	0.2
Triphenyltin(TPT)	ng Sn/kg	0.001—0.2	0.05	12.5%	

Analysis

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 forms on the sharepoinsite. 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.

All results should be reported as **ng Sn / kg seawater**. The concentration of organotins should be determined against your own calibration solutions.

- A 2000-times (approximately) dilution of the spiking solutions is required, using the seawater test materials to produce the spiked seawater. The dilution procedure is given below:

Seawater test material should be used to dilute spiking solution with the corresponding number.

- The spiking solution should be stabilised at 20°C

- Weigh 0.5 ml of spiking solution prior to dilution. The use of a positive displacement pipette or syringe is recommended. Note that the density of the spiking solution is approximately 0.79 kg/L.

- Weigh an empty 1l volumetric flask. Weight an aliquot of the seawater provided, in the flask.

- Add 0.5 ml of the spiking solution to the flask. Make up to 1000 gram with the seawater provided and mix thoroughly. A mass of 1000 gram of water is equal to 975 ml of seawater. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Record the weights in the data submission form along with the spike / sample weight ratio. These results will not be used for statistical analysis but will be used for control purposes by the Quasimeme Project team. This information will assist QUASIMEME in identifying any manipulation errors in the sample preparation prior to the analysis. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1006
AQ-13 Polycyclic Aromatic Hydrocarbons in Seawater		
Test materials		QPH014SW, QPH014SS, QPH015SW, QPH015SS, QPH016SW, QPH017SS

Objective

This study covers the determination of PAHs in the seawater test materials

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys.

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter.

Acetonitrile solutions containing PAHs compounds (QPH014SS and QPH015SS) were prepared in bulk and ampouled to make the spiking solutions.

The test material QPH016SW was prepared in bulk in a 5 litre flask. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. The seawater (4 litre) used to prepare test material QPH016SW was spiked with approximately 1,2 gram highly contaminated (with PAHs) colloidal milled harbour sediment. The flask with seawater and fine sediment was shaken intensively on a shaking apparatus for two hours. Following a stagnant period of 30 minutes most of the waterlayer was decanted in a 5 litre flask. Stirring the content of this flask, a subsample of 120 ml was transferred into each sample flask and these flasks were filled up to a volume of 0.750 litre with blank seawater. This sample should be analysed as a so called total water sample.

Standard solution QPH017SS was prepared by diluting a commercial standard solution containing several PAHs into acetonitrile.

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.

Code	Description
QPH014SS	Spiking solution to use for QPH014SW
QPH014SW	Seawater with Spiking solution
QPH015SS	Spiking solution to use for QPH015SW
QPH015SW	Seawater with Spiking solution
QPH016SW	Seawater (Salinity > 30 psu) spiked using Sediment
QPH017SS	Standard Solution

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 trace PAHs should be determined and the indicative concentrations are given. These indicative concentrations sometimes differ from the indication ranges given in the Quasimeme guide. In relation to the Seawater spiked with sediment the indicative range is an indication only.

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.

Determinand	Unit	Concentration range			Error		AA-EQS
		Seawater (Sediment Spiked)	Seawater (Spiked)	Standard Solution	Const	Prop	
Acenaphthene	µg/L	0.02—200	0.2—200	50—2000	0.01	12.5%	
Acenaphthylene	µg/L	0.001—10	0.2—200	50—2000	0.01	12.5%	
Anthracene	µg/L	0.02—200	0.02-—20	50—2000	0.01	12.5%	0.1
Benzo[a]pyrene	µg/L	0.01—100	0.001—1	50—2000	0.01	12.5%	0.05
Benzo[b]fluoranthene	µg/L	0.01—100	0.001—1	50—2000	0.01	12.5%	0.03
Benzo[k]fluoranthene	µg/L	0.01—100	0.001—1	50—2000	0.01	12.5%	0.03
Benzo[g,h,i]perylene	µg/L	0.002—20	0.001—1	50—2000	0.01	12.5%	0.002
Fluoranthene	µg/L	0.04—400	0.02—20	50—2000	0.01	12.5%	0.1
Indeno(1,2,3-cd)pyrene	µg/L	0.04—400	0.01—10	50—2000	0.01	12.5%	0.002
Naphthalene	µg/L	0.01—100	0.2—200	50—2000	0.01	12.5%	1.2
Phenanthrene	µg/L	0.05—500	0.02—20	50—2000	0.01	12.5%	

Analysis

Preparation and Analysis of the seawater

A 2000-times (approximately) dilution of the spiking solutions is required, using the seawater test materials to produce the spiked seawater. The dilution procedure is given below:

Seawater test material should be used to dilute spiking solution with the corresponding number.

- The spiking solution should be stabilised at 20°C

- Weigh 0.5 ml of spiking solution prior to dilution. The use of a positive displacement pipette or syringe is recommended. Note that the density of the spiking solution is approximately 0.79 kg/L.

- Weigh an empty 1l volumetric flask. Weight an aliquot of the seawater provided, in the flask.

- Add 0.5 ml of the spiking solution to the flask. Make up to 1000 gram with the seawater provided and mix thoroughly. A mass of 1000 gram of water is equal to 975 ml of seawater. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Measure the final weight of the diluted solution prior to extraction.

- Analyse the test materials immediately after preparation.

- Record the weights in the data submission form along with the spike / sample weight ratio. These results will not be used for statistical analysis but will be used for control purposes by the Quasimeme Project team. This information will assist QUASIMEME in identifying any manipulation errors in the sample preparation prior to the analysis. Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery.

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1014
MS-1 Trace metals in Sediment		
Test materials	QTM102MS, QTM103MS	

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.

Code	Description
QTM102MS	Sediment (open sea)
QTM103MS	Sediment (estuarine)

Precaution

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

Determinands and concentration ranges

The following trace metals should be determined:

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

Mercury-RT	µg/kg	50—2500	10	12.5%	
Nickel-AE	mg/kg	5—100	1	12.5%	
Nickel-RT	mg/kg	5—100	1	12.5%	
Scandium-AE	mg/kg	1—20	0.1	12.5%	
Scandium-RT	mg/kg	1—20	0.1	12.5%	
TOC	%	0.2—10	0.1	12.5%	
Zinc-AE	mg/kg	20—1500	2.5	12.5%	
Zinc-RT	mg/kg	20—1500	2.5	12.5%	

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

In addition, total organic carbon and inorganic carbonate should be determined for both test materials. Please note that inorganic carbonate should be reported as % carbon.
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.

Aluminium (Al) should be determined by a total digest or non-destructive method.

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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1015
MS-2 Chlorinated Organics in Sediment		
Test materials	QOR114MS, QOR115MS	

Objective

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

Test Materials and storage

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

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

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

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

Code	Description
QOR114MS	Sediment (harbour)
QOR115MS	Sediment (harbour)

Precaution

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

Determinands and concentration ranges

The following Chlorinated Organics should be determined:

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

pp'-DDE	µg/kg	0.1—10	0.025	12.5%	
pp'-DDT	µg/kg	0.1—10	0.025	12.5%	
TOC	%	0.2—10	0.02	12.5%	
Transnonachlor	µg/kg	0.01—2	0.025	12.5%	

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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 10163
MS-3 Polycyclic Aromatic Hydrocarbons in Sediment		
Test materials	QPH077MS, QPH078MS	

Objective

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

Test Materials and storage

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

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

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

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

Code	Description
QPH077MS	Sediment (harbour)
QPH078MS	Sediment (harbour)

Precaution

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

Determinands and concentration ranges

The following PAHs and alkylated PAHs should be determined:

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

Indeno[1,2,3-cd]pyrene	µg/kg	10—1500	0.2	12.5%	
Naphthalene	µg/kg	10—1500	0.5	12.5%	
Perylene	µg/kg	10—500	0.2	12.5%	
Phenanthrene	µg/kg	10—2000	0.5	12.5%	
Pyrene	µg/kg	10—3000	0.2	12.5%	
TOC	%	0.2—10	0.02	12.5%	
Triphenylene	µg/kg	20—3000	0.5	12.5%	
C1-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C2-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C3-phenanthrenes/anthracenes	µg/kg		0.5	12.5%	
C1-pyrenes/fluoranthenes	µg/kg		0.5	12.5%	
C2-pyrenes/fluoranthenes	µg/kg		0.5	12.5%	
C1-chrysenes	µg/kg		0.5	12.5%	
C2-chrysenes	µg/kg		0.5	12.5%	
C1-benzofluoranthenes	µg/kg		0.5	12.5%	

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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1017
MS-6 Organotins in Sediment		
Test materials	QSP044MS and QSP045MS	

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.

Code	Description
QSP044MS	Sediment (harbour)
QSP045MS	Sediment (harbour)

Precaution

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

Determinands and concentration ranges

The following organotin compounds should be determined:

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

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

Analysis

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

Use your normal validated methods and procedures to analyse the test materials. This may include correcting for blanks and for recovery. Report your method codes using the Data Submission Form on your sharepoint 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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1018
MS-7 Brominated flame retardants in Sediment		
Test materials	QBC034MS, QBC035MS	

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.

Homogeneity, Stability and Storage

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.

Code	Description
QBC034MS	Sediment (harbour)
QBC035MS	Sediment (harbour)

Precaution

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

Determinands and concentration ranges

The following analytes should be determined:

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

Data-assessment for biological tissue test materials will be carried out by calculating with a constant error of 0.005 µg/kg.
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 your sharepoint 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 your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1008
BT-1 Trace metals in Biota		
Test materials	QTM097BT, QTM098BT	

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 IMARES, Institute for Marine Resources and Ecosystem Studies, IJmuiden, The Netherlands and the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands.

The jars contain approximately 50g of minced sterilised biological tissue material, to which butylhydroxytoluene (BHT) has been added as an antioxidant.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the biological tissue test materials 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, in a dry place, prior to analysis, and analysed as soon as possible after receipt. 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.

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

Code	Description
QTM097BT	mussel tissue
QTM098BT	Fish fillet tissue (seafish)

Precaution

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

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

Determinands and concentration ranges

The following trace metals should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Shellfish tissue	Fish muscle tissue	Const	Prop	
Arsenic	mg/kg	0.2—10	1—10	0.02	12.5%	
Ash-weight	%			0.1	12.5%	
Cadmium	µg/kg	10—500	1—50	20	12.5%	
Chromium	µg/kg	10—5000	50—500	20	12.5%	
Copper	µg/kg	50—10000	100—1000	100	12.5%	

Dry-weight	%			0.1	12.5%	
Extractable-Lipid	%			0.1	12.5%	
Lead	µg/kg	10—1000	10—50	5	12.5%	
Mercury	µg/kg	5—500	20—1000	20	12.5%	
Nickel	µg/kg	10—2000	10—200	20	12.5%	
Selenium	µg/kg	200—1000	200—2000	10	12.5%	
Silver	µg/kg	1—500	0.5—50	5	12.5%	
Total-Lipid	%			0.1	12.5%	
Zinc	mg/kg	2—200	2—10	2	12.5%	

Ash weight, dry weight and total lipid should also be determined. 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 III. 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 and the final result calculated as wet weight using the dry weight determination. In this case the % dry weight should be reported.

% dry weight = 100 - [(wet weight - dry weight) □ 100]/(wet weight)

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1009
BT-2 Chlorinated Organics in Biota		
Test materials	QOR114BT, QOR115BT	

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 IMARES, Institute for Marine Resources and Ecosystem Studies, IJmuiden, The Netherlands and the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands. The tins with biological tissue test material contain approximately 70g of minced sterilised material, to which butylhydroxytoluene (BHT) has been added as an antioxidant.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the biota 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, in a dry place, prior to analysis, and analysed as soon as possible after receipt. Although the tins have a protective coating, moisture can cause corrosion of the surface of the tins. Apart from the moisture, temperatures below 0˚C will not negatively affect the quality in the material, and we do not expect the contaminants to be affected at these temperatures. Temperatures above 30˚C may be disadvantageous for the material.

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

Code	Description
QOR114BT	Fish fillet tissue (seafish)
QOR115BT	Mussel tissue

Precaution

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

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

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range			Error		AA-EQS
		Fish Liver tissue and Freshwater Fish	Fish Muscle Tissue	Shellfish Tissue	Const	Prop	
PCB28	µg/kg	1—50	0.05—5	0.05—5	0.025	12.5%	
PCB31	µg/kg	1—10	0.03—3	0.03—3	0.025	12.5%	
PCB52	µg/kg	10—100	0.05—5	0.05—5	0.025	12.5%	
PCB101	µg/kg	30—300	0.1—20	0.1—20	0.025	12.5%	

PCB105	µg/kg	2—100	0.05—10	0.05—10	0.025	12.5%	
PCB118	µg/kg	20—300	0.2—20	0.2—20	0.025	12.5%	
PCB138+PCB163	µg/kg	20—600	0.3—30	0.3—30	0.025	12.5%	
PCB138	µg/kg	20—600	0.3—30	0.3—30	0.025	12.5%	
PCB153	µg/kg	50—1000	0.4—40	0.4—40	0.025	12.5%	
PCB156	µg/kg	1—40	0.03—10	0.03—10	0.025	12.5%	
PCB180	µg/kg	10—200	0.05—5	0.05—5	0.025	12.5%	
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%	
HCBD	µg/kg	0.05—5			0.025	12.5%	
Dieldrin	µg/kg	0.5—100	0.2—20	0.2—20	0.025	12.5%	
pp'-DDD	µg/kg	5—100	0.1—10	0.1—10	0.025	12.5%	
pp'-DDE	µg/kg	10—500	0.3—30	0.3—30	0.025	12.5%	
op'-DDT	µg/kg	0.1—2	0.01—1	0.01—1	0.025	12.5%	
pp'-DDT	µg/kg	0.1—10	0.1—10	0.1—10	0.025	12.5%	
Transnonachlor	µg/kg	0.2—40	0.02—10	0.02—10	0.025	12.5%	
Total-Lipid	%				0.1	12.5%	
Extractable-Lipid	%				0.1	12.5%	

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

% dry weight = 100 - [(wet weight - dry weight) □ 100]/(wet weight)

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1010
BT-4 Polycyclic Aromatic Hydrocarbons in Biota		
Test materials	QPH069BT, QPH070BT	

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 the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands.

The tins contain approximately 70g of minced sterilised biological tissue material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the biota 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, in a dry place, prior to analysis, and analysed as soon as possible after receipt. Although the tins have a protective coating, moisture can cause corrosion of the surface of the tins. Apart from the moisture, temperatures below 0˚C will not negatively affect the quality in the material, and we do not expect the contaminants to be affected at these temperatures. Temperatures above 30˚C may be disadvantageous for the material.

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

Code	Description
QPH069BT	Shellfish tissue (mussel)
QPH070BT	Shellfish tissue (mussel)

Precaution

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

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

Determinands and concentration ranges

The following PAHs should be determined:

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

Benzo[a]fluorene	µg/kg		0.5	12.5%	
Benzo[a]pyrene	µg/kg	0.2—5	0.2	12.5%	
Benzo[b]fluoranthene	µg/kg	0.2—10	0.2	12.5%	
Benzo[e]pyrene	µg/kg	0.2—10	0.2	12.5%	
Benzo[g,h,i]perylene	µg/kg	0.2—5	0.2	12.5%	
Benzo[k]fluoranthene	µg/kg	0.2—5	0.2	12.5%	
Chrysene	µg/kg	0.2—20	0.2	12.5%	
Chrysene+Triphenylene	µg/kg	0.2—20	0.2	12.5%	
Dibenz[a,h]anthracene	µg/kg	0.2—2	0.1	12.5%	
Dibenzo[a,i]pyrene	µg/kg		0.5	12.5%	
Dibenzothiophene	µg/kg	0.2—5	0.5	12.5%	
Extractable-Lipid	%		0.1	12.5%	
Fluoranthene	µg/kg	5—50	0.2	12.5%	
Fluorene	µg/kg	1—50	0.2	12.5%	
Indeno[1,2,3-cd]pyrene	µg/kg	0.2—5	0.2	12.5%	
Naphthalene	µg/kg	1—100	0.2	12.5%	
Perylene	µg/kg	0.1—5	0.5	12.5%	
Phenanthrene	µg/kg	2—50	0.2	12.5%	
Pyrene	µg/kg	1—20	0.2	12.5%	
Total-Lipid	%		0.1	12.5%	
Triphenylene	µg/kg		5	12.5%	

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

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 and the final result calculated as wet weight using the dry weight determination. In this case the % dry weight should be reported.

% dry weight = 100 - [(wet weight - dry weight) □ 100]/(wet weight)

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1011
BT-8 Organotins in Biota		
Test materials	QSP044BT, QSP045BT	

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 IMARES, Institute for Marine Resources and Ecosystem Studies, IJmuiden, The Netherlands and the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands.

The tins contain approximately 70g of minced sterilised biological tissue material, to which butylhydroxytoluene (BHT) has been added as an antioxidant. Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the biota 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, in a dry place, prior to analysis, and analysed as soon as possible after receipt. Although the tins have a protective coating, moisture can cause corrosion of the surface of the tins. Apart from the moisture, temperatures below 0˚C will not negatively affect the quality in the material, and we do not expect the contaminants to be affected at these temperatures. Temperatures above 30˚C may be disadvantageous for the material.

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

Code	Description
QSP044BT	Mussel
QSP045BT	Mussel

Precaution

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

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

Determinands and concentration ranges

The following analytes should be determined:

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

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

Analysis

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

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

The concentrations should be determined against your own calibration solutions.

Only one result per determinand per test material is required. The results of each determinand should be expressed as Sn on the test material "as received" i.e. on a wet weight basis (□g Sn/kg).

If your normal method is to dry the test material prior to extraction then the dry weight must be determined and the final result calculated as wet weight using the dry weight determination. In this case the % dry weight should be reported.

% dry weight = 100 - [(wet weight - dry weight) □ 100]/(wet weight)

Reporting

One result for each determinand in each test material should be reported using the Data Submission Form which is placed on your sharepoint site. It is not possible to report two sets of data using different methods on the same exercise 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 to send you an additional exercise Data Submission Form.

ROUND	72	Exercise 1012
BT-9 Brominated Flame Retardants in Biota		
Test materials	QBC034BT, QBC035BT	

Objective

This study covers the determination of brominated compounds in biota.

Test Materials and storage

The test materials were supplied the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands and by WEPAL, Wageningen, The Netherlands.

Biological tissue

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

Homogeneity, Stability and Storage

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.

Code	Description
QBC034BT	Fish fillet tissue (freshwater fish)
QBC035BT	Shellfish tissue (mussel)

Precaution

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

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

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
BDE28	µg/kg	0.001—1	0.05	12.5%	
BDE47	µg/kg	0.05—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	0.01—10	0.05	12.5%	
BDE100	µg/kg	0.005—2	0.05	12.5%	
BDE153	µg/kg	0.01—1	0.05	12.5%	
BDE154	µg/kg	0.001—1	0.05	12.5%	
BDE183	µg/kg	0.001—0.1	0.05	12.5%	
BDE209	µg/kg	0.01—0.1	0.05	12.5%	
TBBP-A	µg/kg		0.05	12.5%	
Dimethyl-TBBP-A	µg/kg		0.05	12.5%	
α-HBCD	µg/kg		0.05	12.5%	
β-HBCD	µg/kg		0.05	12.5%	
δ-HBCD	µg/kg		0.05	12.5%	
Total-HBCD	µg/kg		0.05	12.5%	

Data-assessment for biological tissue test materials will be carried out by calculating with a constant error of 0.005 µg/kg.

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 and the final result calculated as wet weight using the dry weight determination. In this case the % dry weight should be reported.

% dry weight = 100 - [(wet weight - dry weight) $*$ 100]/(wet weight)

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 sharepointsite.

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 to send you an additional data submission form.

ROUND	72	Exercise 1020
BT-7 ASP Shellfish Toxins		
Test materials	QST144SS, QST145BT, QST146BT	

Objective

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

Test Materials and storage

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

- QST144SS is a domoic acid standard solution.

- QST145BT is a scallop tissue homogenate supplied in a plastic vial.

- QST146BT is a mussel tissue homogenate supplied in a plastic vial.

For QST145BT and QST146BT, each vial contains sufficient material for one-shot analysis of domoic and epidomoic acid.

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

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

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

The Standard solution QST144SS should be stored in the refrigerator at ca 4˚C immediately upon receipt, until analysis

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

Code	Description
QST144SS	Standard Solution
QST145BT	Shellfish tissue
QST146BT	Shellfish tissue

Precaution

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

Determinands and concentration ranges

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

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

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

Analysis

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

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

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

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the sharepointsite

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.

ROUND	72	Exercise 1021
DE-10 Lipophilic Shellfish Toxins		
Test materials	QST147SS, QST148SS, QST149BT, QST150BT, QST151BT	

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, Republic of 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.

Code	Description
QST147SS	AZA Standard Solution
QST148SS	DTX-1 Standard Solution
QST149BT	Shellfish tissue (mussel)
QST150BT	Shellfish tissue (scallop)
QST151BT	DSP/AZP Extract

Precaution

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

Determinands and concentration ranges

Determinands

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

Please report lipophilic toxins (if detected) as follows:

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

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

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

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

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

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

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

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

Toxicity Equivalency Factors

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

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

	AZA-2	1.8
	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 to send you an additional exercise template file. It is not possible to report two sets of data using different methods on the same exercise template.

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

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

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

All results should be reported in µg/kg (YTX-group toxins in mg/kg) on the basis of wet weight of the test material as provided. The density of the standard solutions are 0.7918 g/ml (MeOH) and the density of the lipophilic extract is 0.834g/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 at the sharepointsite

ROUND	72	Exercise 1022
DE-14 PSP Shellfish Toxins		
Test materials	QST152BT, QST153BT, QST154BT and QST155BT	

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, Republic of 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.

Code	Description
QST152BT	Mussel tissue
QST153BT	Oyster tissue
QST154BT	Mussel tissue
QST155BT	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

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%	
C2	µmol/kg		0.1	12.5%	
C3	µmol/kg		0.1	12.5%	
C4	µmol/kg		0.1	12.5%	
dc-GTX1	µmol/kg		0.1	12.5%	
dc-GTX2	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%	
GTX1	µmol/kg		0.1	12.5%	
GTX2	µmol/kg		0.1	12.5%	
GTX3	µmol/kg		0.1	12.5%	
GTX4	µmol/kg		0.1	12.5%	
GTX5	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%	

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 column in the reporting

template as GTX-1 has the higher toxicity factor). 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 sharepointsite

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.

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

Method codes are supplied as part of the data submission forms. Report all of the requested method codes. If the method codes in any section do not adequately describe your analytical method, select "Other" from the method code list, and provide additional information on your method, electronically, when you return your data.

Return of Data

Upload all analytical data to the QUASIMEME SharePoint site only with the data submission forms. 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 QUASIMEME Project Office in Wageningen no later than 1 June 2013. 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. 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 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 July 2013. 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 G13: 2000 3.9.

ANNEX 1 Notification of damaged test materials.

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

Laboratory Code : .....

Damaged container number : .....

Loss of weight container number : .....

I request a new test material for :..... Because : .....

Date : .....

Signature :.....

Name of participant :.....

Name and address of institute : .....

.....

.....

Telephone number :.....

Fax number :.....

Return this form to :

QUASIMEME Project Office
Wageningen UR
Alterra CWK
P.O. Box 47
6700 AA Wageningen
The Netherlands

Fax No : +31(0)317 486 546
E-mail : QUASIMEME@wur.nl

ANNEX 2 Instructions for login into sharepointsite

Login to http://www.quasimeme.org

Select sharepointsite

Username: wur\x……. (your specific logincode e.g. xcrum012)

Password: your specific password

Ask the Quasimeme project office when the login information is unknown

Select the correct year

Select the correct round

Select the correct exercise

Enter your results and method information into the data submission form

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.