Quasimeme Laboratory Performance Studies

Round60

1 January 2010 to 30 April 2010

Exercise Protocols

Version 01-02-2010

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Introduction Round60

Thank you for participating in the 2010 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round60 that you have ordered will be sent to you by courier in the week beginning 18 January 2010. 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 sent, please use the form which can be found in Annex 1 of this document to request replacement materials. The test materials will be dispatched to you within two weeks after receipt of the form.

Additional test materials may also be purchased from QUASIMEME.

This protocol covers the following studies :

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We expect to implement our new data uploading system in the beginning of February. At that time we will provide you with instructions on how to use the new system. If, for unknown reasons we are unable to achieve this operational deadline we will use continue with the old QUEST system for the very last time. Therefore we will leave the instruction on how to use the QUEST file in this protocol. The exercise template files, for use with the QUEST[1] data transfer system (Windows 9X, XP, NT or 2000), must be downloaded from your Quasimeme SharePoint Site.

All data for these studies must be uploaded to your Quasimeme SharePoint Site, no later than 30 April 2010

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

[1] QUEST (QUASIMEME Electronic Storage and Transfer) System. Information on installation and use of this software is given in Annex 2

ROUND	60	Exercise 870
AQ-1 Nutrients in Seawater		
Test materials	QNU207SW, QNU208SW, QNU209SW	

Objective

This study covers the determination of nutrients in the seawater test materials. The test materials should be analysed and one result for each nutrient in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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. An extra bottle was sent for the DOC analysis. 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
QNU207SW	Seawater (Salinity > 30 psu) spiked
QNU208SW	Seawater (Salinity > 30 psu) unspiked
QNU209SW	Seawater (Salinity > 30 psu) spiked

Precaution

Some of the elements may present a health hazard, and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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	µM	0.2—5	0.2—5	0.1	6.0%	
DOC	mg C/L	0.1—20	0.1—20	0.1	6.0%	
Nitrite	µM	0.01—2	0.01—2	0.01	6.0%	
Phosphate	µM	0.05—5	0.05—5	0.05	6.0%	
Silicate	µM	0.5—10	0.5—10	0.1	6.0%	
Total-N	µM	5—25	5—25	0.5	6.0%	
Total-P	µM	0.1—5	0.1—5	0.05	6.0%	
TOxN	µM	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. As a guide, the concentrations (µM) of the nutrients in the spiked test materials are within the ranges mentioned in the Quasimeme scheme.

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

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.

ROUND	60	Exercise 871
AQ-2 Nutrients in Estuarine and low salinity open water		
Test materials	QNU210EW, QNU211EW, QNU212EW, QNU213EW	

Objective

This study covers the determination of seven nutrients in each of the two estuarine water test materials and in the two low salinity open water test materials. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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. An extra bottle was added for determination of DOC. Each bottle contains approximately 250 ml of the test material.

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
QNU210EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU211EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU212EW	Low salinity open water (Salinity 8 - 20 psu) spiked
QNU213EW	Low salinity open water (Salinity 8 - 20 psu)

Precaution

This element presents some health hazard and is 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	µM	2—50	0.2—5	0.1	6.0%	
DOC	mg C/L	0.1—20		0.1	6.0%	
Nitrite	µM	0.5—25	0.01—2	0.01	6.0%	
Phosphate	µM	1—15	0.02—5	0.05	6.0%	
Salinity	Psu			0.001	0.1%	
Silicate	µM	5—100	0.5—20	0.1	6.0%	
Total-N	µM	10—200	2—20	0.5	6.0%	
Total-P	µM	1—20	0.02—2	0.05	6.0%	
TOxN	µM	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.

As a guide, the concentrations (µM) of the nutrients in the spiked test materials are within the ranges mentioned in the Quasimeme scheme.

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

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.

ROUND	60	Exercise 872
MS-1 Trace metals in Sediment		
Test materials	QTM090MS, QTM091MS	

Objective

This study covers the determination of metals, total organic carbon and carbonate in marine sediments. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

Test Materials and storage

Test materials were prepared by the Institute for Environmental Studies, Vrije Universiteit, Amsterdam, The Netherlands and 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
QTM090MS	Sediment (open sea)
QTM091MS	Sediment (estuary)

Precaution

It should be noted that some of these elements present some health hazard and a number are biologically active. Please ensure that your analytical and handling procedures for these materials 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	%	1—10	0.1	12.5%	
Arsenic	mg/kg	2—50	1	12.5%	
Cadmium	µg/kg	10—2000	20	12.5%	
Chromium	mg/kg	10—1000	2	12.5%	
Copper	mg/kg	1—500	1	12.5%	
Inorganic-carbonate	%	0.05—10	0.05	12.5%	
Iron	%	0.5—10	0.1	12.5%	
Lead	mg/kg	5—500	2	12.5%	
Lithium	mg/kg	10—100	0.1	12.5%	
Manganese	mg/kg	100—2000	0.1	12.5%	
Mercury	µg/kg	50—2500	10	12.5%	
Nickel	mg/kg	5—100	1	12.5%	
Scandium	mg/kg	1—20	0.1	12.5%	
TOC	%	0.2—10	0.1	12.5%	
Zinc	mg/kg	20—1500	2.5	12.5%	

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. It is essential that you give your method code for each metal. If you fail to report your method codes QUASIMEME will assess the data based on a total digest 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

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.

ROUND	60	Exercise 873
MS-2 Chlorinated Organics in Sediment		
Test materials	QOR100MS, QOR101MS, QOR102MS	

Objective

This study covers the determination of chlorobiphenyls (CBs), organochlorine pesticides (OCPs) and total organic carbon in marine sediment. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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 analyzed as soon as possible after receipt.

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

Code	Description
QOR100MS	Sediment (harbour)
QOR101MS	Sediment (estuary)
QOR102MS	Sediment (open sea)

Precaution

It should be noted that some of these compounds may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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

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.

ROUND	60	Exercise 874
MS-3 Poly Aromatic Hydrocarbons in Sediment		
Test materials	QPH064MS, QPH065MS	

Objective

This study covers the determination of PAHs and total organic carbon in marine sediment. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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 analyzed as soon as possible after receipt.

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

Code	Description
QPH064MS	Sediment (open sea)
QPH065MS	Sediment (harbour)

Precaution

It should be noted that some of these compounds may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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
			Const	Prop	
Acenaphtene	µg/kg	2-500	0.1	12.5%	
Acenaphtylene	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[k]fluoranthene	µg/kg	10-1000	0.1	12.5%	
Benzo[e]pyrene	µg/kg	10-1500	0.2	12.5%	
Benzo[g,h,i]perylene	µg/kg	10-1500	0.2	12.5%	
Chrysene	µg/kg	10-1500	0.2	12.5%	
Chrysene+Triphenylene	µg/kg	10-3000	0.2	12.5%	
Triphenylene	µg/kg	20-3000	0.5	12.5%	
Dibenz[a,h]anthracene	µg/kg	5-500	0.05	12.5%	
Dibenzo[a,i]pyrene	µg/kg		0.5	12.5%	
Dibenzothiophene	µg/kg	2-200	0.1	12.5%	

Fluoranthene	µg/kg	20-3000	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%	
Naphtalene	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%	
2-Methylphenanthrene	µg/kg	5-1000	0.5	12.5%	
3,6-Dimethylphenanthrene	µg/kg	1-500	0.5	12.5%	
Pyrene	µg/kg	10-3000	0.2	12.5%	
1-Methylpyrene	µg/kg	2-500	0.5	12.5%	
TOC	%	0.2-10	0.02	12.5%	

In addition total organic carbon should be determined for both test materials.
Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

Analysis

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

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

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

Reporting

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.

ROUND	60	Exercise 875
MS-6 Organotins in Sediment		
Test materials	QSP031MS, QSP032MS	

Objective

This study covers the determination of organotin compounds in sediments.

The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

Test Materials and storage

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

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
QSP031MS	Sediment (harbour)
QSP032MS	Sediment (harbour)

Precaution

Some of the analytes may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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 new data system or, if unavailable, QUEST. 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 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

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.

ROUND	60	Exercise 876
BT-1 Trace metals in Biota		
Test materials	QTM085BT, QTM086BT	

Objective

This study covers the determination of ten trace metals, ash weight, dry weight and total lipid in each of two biota test materials. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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 fish 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. 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
QTM085BT	Shellfish tissue (mussel)
QTM086BT	Fish liver tissue

Precaution

It should be noted that some of these elements may present a health hazard and a number are biologically active. Please ensure that your analytical and handling procedures for these materials 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 liver tissue	Const	Prop	
Arsenic	mg/kg	0.2—10	1—5	0.02	12.5%	
Ash-weight	%			0.1	12.5%	
Cadmium	µg/kg	10—500	5—1000	20	12.5%	
Chromium	µg/kg	10—5000	20—1000	20	12.5%	
Copper	µg/kg	50—10000	2000—10000	100	12.5%	
Dry-weight	%			0.1	12.5%	
Extractable-Lipid	%			0.1	12.5%	
Lead	µg/kg	10—1000	10—1000	5	12.5%	
Mercury	µg/kg	5—500	20—100	20	12.5%	

Nickel	µg/kg	10—2000	20—1000	20	12.5%	
Selenium	µg/kg	200—1000	200—5000	10	12.5%	
Silver	µg/kg	1—500	20—1000	5	12.5%	
Total-Lipid	%			0.1	12.5%	
Zinc	mg/kg	2—200	10—50	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

The containers are filled almost to the brim, so should be opened carefully to ensure no spillage of tissue or moisture. Containers are filled in this manner to ensure the elimination of air as much possible. This minimises any degradation of the test materials in transit and storage prior to opening.

We recommend wearing latex gloves when opening the jars of biota, to prevent the jar slipping.

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 jar, we recommend transferring the material to a larger container (e.g. glass or plastic beaker) 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.

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)

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

Reporting

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.

ROUND	60	Exercise 877
BT-2 Chlorinated Organics in Biota		
Test materials	QOR102BT, QOR103BT	

Objective

This study covers the determination chlorobiphenyls (CBs), organochlorine pesticides (OCPs) and total lipid in each of two biota test materials. The test materials should be analysed and one result for each determinand in each test material should be reported using the new data system or, if unavailable,the QUEST system with the QUASIMEME exercise template file.

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 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
QOR102BT	Shellfish tissue (mussel)
QOR103BT	Fish muscle tissue

Precaution

It should be noted that some of these compounds may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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
		Shellfish tissue	Fish muscle tissue	Const	Prop	
a-HCH	µg/kg	0.05—5	0.05—5	0.02	12.5%	
b-HCH	µg/kg	0.05—5	0.05—5	0.025	12.5%	
CB101	µg/kg	0.1—20	0.1—20	0.025	12.5%	
CB105	µg/kg	0.05—10	0.05—10	0.025	12.5%	
CB118	µg/kg	0.2—20	0.2—20	0.025	12.5%	
CB138	µg/kg	0.3—30	0.3—30	0.025	12.5%	
CB138+CB163	µg/kg	0.3—30	0.3—30	0.025	12.5%	
CB153	µg/kg	0.4—40	0.4—40	0.025	12.5%	
CB156	µg/kg	0.03—10	0.03—10	0.025	12.5%	
CB180	µg/kg	0.05—5	0.05—5	0.025	12.5%	

CB28	µg/kg	0.05—5	0.05—5	0.025	12.5%	
CB31	µg/kg	0.03—3	0.03—3	0.025	12.5%	
CB52	µg/kg	0.05—5	0.05—5	0.025	12.5%	
d-HCH	µg/kg	0.05—5	0.05—5	0.025	12.5%	
Dieldrin	µg/kg	0.2—20	0.2—20	0.025	12.5%	
Extractable-Lipid	%			0.1	12.5%	
g-HCH	µg/kg	0.05—5	0.05—5	0.025	12.5%	
HCB	µg/kg	0.02—5	0.02—5	0.025	12.5%	
HCBD	µg/kg			0.025	12.5%	
op'-DDT	µg/kg	0.01—1	0.01—1	0.025	12.5%	
pp'-DDD	µg/kg	0.1—10	0.1—10	0.025	12.5%	
pp'-DDE	µg/kg	0.3—30	0.3—30	0.025	12.5%	
pp'-DDT	µg/kg	0.1—10	0.1—10	0.025	12.5%	
Total-Lipid	%			0.1	12.5%	
Transnonachlor	µg/kg	0.02—10	0.02—10	0.025	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.

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 tins of biota 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. Opening the bottom of the tin, instead of the top, may also help to prevent spillage of moisture.

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 (e.g. glass or plastic beaker) 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.

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)

The concentrations should be determined against your own calibration solutions.

Reporting

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.

ROUND	60	Exercise 878
BT-3 Non ortho CBs, PCDDs and PCDFs in Biota		
Test materials	QPL030BT, QPL031BT	

Objective

This study covers the determination of non-ortho Chlorobiphenyls (CBs), polychlorinated dibenzo-p-dioxins (PCDDs), polychlorinated dibenzofurans (PCDFs), total TEQ and total lipid in two marine biota 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 fish 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.

Code	Description
QPL030BT	Fish muscle tissue
QPL031BT	Shellfish tissue (mussel)

Precaution

The tins of biota 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. 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 analytes should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Fish muscle tissue	Shellfish tissue	Const	Prop	
1,2,3,4,6,7,8-HpCDD	ng/kg	0.05—5	0.05—5	0.02	12.5%	
1,2,3,4,6,7,8-HpCDF	ng/kg	0.02—0.5	0.02—0.5	0.02	12.5%	
1,2,3,4,7,8,9-HpCDF	ng/kg			0.02	12.5%	
1,2,3,4,7,8-HxCDD	ng/kg	0.02—0.2	0.02—0.2	0.02	12.5%	
1,2,3,4,7,8-HxCDF	ng/kg	0.01—0.2	0.01—0.2	0.02	12.5%	

1,2,3,6,7,8-HxCDD	ng/kg	0.03—1	0.03—1	0.02	12.5%	
1,2,3,6,7,8-HxCDF	ng/kg	0.01—0.2	0.01—0.2	0.02	12.5%	
1,2,3,7,8,9-HxCDD	ng/kg	0.02—0.5	0.02—0.5	0.02	12.5%	
1,2,3,7,8,9-HxCDF	ng/kg			0.02	12.5%	
1,2,3,7,8-PeCDD	ng/kg	0.02—1	0.02—1	0.02	12.5%	
1,2,3,7,8-PeCDF	ng/kg	0.03—1	0.03—1	0.02	12.5%	
2,3,4,6,7,8-HxCDF	ng/kg	0.01—0.5	0.01—0.5	0.02	12.5%	
2,3,4,7,8-PeCDF	ng/kg	0.05—3	0.05—3	0.02	12.5%	
2,3,7,8-TCDD	ng/kg	0.01—0.5	0.01—0.5	0.02	12.5%	
2,3,7,8-TCDF	ng/kg	0.03—5	0.03—5	0.02	12.5%	
CB126	ng/kg	1—20	1—20	0.02	12.5%	
CB169	ng/kg	0.2—5	0.2—5	0.02	12.5%	
CB77	ng/kg	10—100	10—100	0.02	12.5%	
Extractable-Lipid	%			0.1	12.5%	
OCDD	ng/kg	0.05—5	0.05—5	0.02	12.5%	
OCDF	ng/kg	0.02—0.5	0.02—0.5	0.02	12.5%	
Total-Lipid	%			0.1	12.5%	
Total-TEQ(Dr.CALUX)	ng/kg			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

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

After Carefully 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 (e.g. glass or plastic beaker) 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.

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.

Reporting

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.

ROUND	60	Exercise 879
BT-4 Poly Aromatic Hydrocarbons in Biota		
Test materials	QPH057BT, QPH058BT	

Objective

This study covers the determination of Poly Aromatic Hydrocarbons (PAHs) and total lipid in

two marine biota 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 fish 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.

Code	Description
QPH057BT	Shrimp
QPH058BT	Shellfish tissue (mussel)

Precaution

The tins of biota 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. 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 analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
Acenaphtene	µg/kg	0.5-100	0.2	12.5%	
Acenaphtylene	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[k]fluoranthene	µg/kg	0.2-5	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%	
Chrysene	µg/kg	0.2-20	0.2	12.5%	
Chrysene+Triphenylene	µg/kg	0.2-20	0.2	12.5%	
Triphenylene	µg/kg		5	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%	
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%	
Naphtalene	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%	
2-Methylphenanthrene	µg/kg	0.2-5	2	12.5%	
3,6-Dimethylphenanthrene	µg/kg	0.2-2	0.5	12.5%	
Pyrene	µg/kg	1-20	0.2	12.5%	
1-Methylpyrene	µg/kg		2	12.5%	
Total-lipid	%		0.1	12.5%	
Extractable-lipid	%		0.1	12.5%	

Analysis

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

After carefully 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 (e.g. glass or plastic beaker) for homogenizstion 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 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.

Reporting

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.

ROUND	60	Exercise 880
BT-8 Organotins in Biota		
Test materials	QSP032BT, QSP033BT	

Objective

This study covers the determination of organotin compounds in biota test materials. The test materials should be analysed and one result for each analyte in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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.

Each batch of material was prepared in bulk. The level of within and between sample homogeneity for the biota was determined by measuring selected PCBs. 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.

butylhydroxytoluene (BHT) has been added as an antioxidant.

The biota test materials should be stored at room temperature, in a dry place, prior to analysis, and analyzed 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
QSP032BT	Shrimp
QSP033BT	Mussel (mussel)

Precaution

The containers are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. Containers 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.

Some of the analytes may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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

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 new data system or, if unavailable, QUEST. 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 on the test materials "as received". All results should be reported as µg Sn / kg wet weight of tissue. The concentration of organotins should be determined against your own calibration solutions.

Some of the analytes may present a health hazard and some are biologically active. Please ensure that your analytical and handling procedures for these materials have been fully assessed for safety and that all specified precautions are taken.

The containers are filled almost to the brim, so should be opened carefully so that no tissue or moisture is spilt. Containers 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.

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 jar or tin, we recommend transferring the material to a larger container (e.g. glass or plastic beaker) for homogenisation and sub-sampling. Aliquots should be taken immediately after homogenising, to prevent liquid lipids from separating from the solid tissue particles, causing in homogeneity again.

After sub-sampling the remaining tissue should be immediately transferred to a clean glass jar, sealed and stored at -20°C.

Reporting

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.

ROUND	60	Exercise 881
BS-1 Brominated flame retardants in Biota and Sediment		
Test materials	QBC026BT, QBC027MS	

Objective

This study covers the determination of brominated flame retardants in sediment and biological tissue. The test materials should be analysed and one result for each analyte in each test material should be reported using the new data system or, if unavailable, the QUEST system with the QUASIMEME exercise template file.

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.

Sediment

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.

Biota

The tin 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 in a dry place at room temperature in the dark, prior to analysis, and analysed as soon as possible after receipt.

Code	Description
QBC026BT	Shrimp
QBC027MS	Sediment (harbour)

Precaution

Care should be taken when opening all test materials.

The tin of biota test material has been sterilised and as a result a positive pressure may be present in the tin. The tin should be cooled to ca 4°C for a few hours prior to opening.

Some of these compounds may present a health hazard, and some are biologically active. Please ensure that your analytical and handling procedures for these materials 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	Sediment	Const	Prop	
a-HBCD	µg/kg			0.05	12.5%	
BDE100	µg/kg	0.005—2	0.01—10	0.05	12.5%	
BDE153	µg/kg	0.01—1	0.1—5	0.05	12.5%	
BDE154	µg/kg	0.001—1	0.01—5	0.05	12.5%	
BDE183	µg/kg	0.001—0.1	0.1—2	0.05	12.5%	
BDE209	µg/kg	0.01—0.1	20—200	0.05	12.5%	
BDE28	µg/kg	0.001—1	0.01—2	0.05	12.5%	
BDE47	µg/kg	0.05—20	0.1—20	0.05	12.5%	
BDE66	µg/kg	0.01—10	0.01—10	0.05	12.5%	
BDE85	µg/kg	0.01—10	0.01—10	0.05	12.5%	
BDE99	µg/kg	0.01—10	0.1—50	0.05	12.5%	
b-HBCD	µg/kg			0.05	12.5%	
Dimethyl-TBBP-A	µg/kg			0.05	12.5%	
g-HBCD	µg/kg			0.05	12.5%	
TBBP-A	µg/kg			0.05	12.5%	
Total-HBCD	µg/kg		50—200	0.05	12.5%	

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

Analysis

The test materials have been extensively tested for homogeneity and have been found to be suitable for the exercise. However, during the sterilisation some moisture may be separated from the bulk of the material. Consequently, it is essential to re-homogenise all of the test material prior to sub-sampling. As it is difficult to homogenise the sample in the jar or tin, we recommend transferring the material to a larger container (e.g. glass or plastic beaker) 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.

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 material "as received".

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 also be reported. The results should be reported on a weight / total weight basis (µg/kg) for all test materials. The method codes for extraction, clean-up and GC conditions should be reported using the new data system or, if unavailable, the*.qxt exercise template file. Some advice on how to recognise and avoid possible sources of error is given in Annex III.

The results reported should be corrected for recovery. Recovery values of over 100% should not be used to correct the data.

Reporting

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.

Reporting of Results and Analytical Methods

Enter your analytical data and the method codes using the new data system or, if unavailable, the *.qxt files. Instructions for installing the QUEST software if the new data system is unavailable, to report analytical data and methods are given in Annex 2.

Units

The units of measurement are given in the template files. Ensure that the concentration of each determinant 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 exercise template after the allocated deadline.

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 determinant 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 d 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 template files. When you use the new data system or, if unavailable,the QUEST programme, the exercise template includes your latest method code selection set as default. 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 under data entry in the exercise template. 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 template files can be included in the assessment. Return the results to the QUASIMEME Project Office in Wageningen no later than 30 April 2010. 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:

- Make sure that you have saved your data, and keep a copy of the data for your own records, before uploading the files to the Quasimeme SharePoint site.
- **Do not rename the data files** – use the existing file names. The file name contains your laboratory code and the exercise number, which are essential identifiers for returned data.
- Make sure your system is virus free before returning data.

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 data submitted late will receive a certificate with 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 30 June 2010. 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 repeated analyses and submitting mean data, or collaboration with colleagues undertaking the same study.

QUASIMEME checks for evidence of collusion and declares 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 :..... due to : .....

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 Installing and Using QUEST Version 2.1 if the new datasystem is unavailable

QUEST : QUASIMEME ELECTRONIC STORAGE AND TRANSFER SYSTEM

QUEST provides a Windows environment for the transfer of data for the QUASIMEME Laboratory Performance studies.

Installing QUEST 2.1

Version 2.1 of the QUEST program (for Windows 9x, NT4, 2000 or XP) has been provided to all participants. Either you received It on the Quasimeme CD (before 2007) or it can be downloaded from the QUASIMEME website. If you are not able to download the program from the website, please contact the QUASIMEME Project Office.

QUEST 2.1 from website:

On the SharePoint site you can find an instruction (General documents / Instruction Quest from website) how to download and install QUEST on your computer. It also gives you the instruction how to download QUEST files (templates) and upload your results from and onto the SharePoint site.

QUEST 2.1 on CD

QUEST 2.1 provided on the QUASIMEME CD. To install QUEST 2.1 please follow these instructions:

- Copy the files from the QUEST directory on the CD to an empty temporary directory on the hard drive of your computer (e.g. c:\temp2).
- Select <Start>, <Run>, <Browse> then select setup.exe from the temporary directory containing the QUEST 2.1 files and click <OK> to install QUEST 2.1. Follow the instructions on screen during the installation.

The QUEST program will install in the C:\Program Files\ directory unless you have specified another directory during the setup procedure.

Using QUEST 2.1

Use QUEST to open your exercise template and enter your data. All of the exercises for the same laboratory are provided in one e-mail message. The files are arranged in directories using the variable laboratory code for the round and exercise numbers as unique identifiers e.g. AD479.639 The file names to be used with the QUEST program are structured:

<exercise number><variable laboratory code>.qxt, e.g. 639AD479.qxt

To start the QUEST Program, select <Start> <Programs><Quest>. The QUEST Logo screen will appear while the program is loading and then, initially, an empty data sheet will appear. Once the program has been used to save data, the last data sheet will normally appear by default.

It is also possible to configure the computer to start the QUEST program from the exercise file. To do this, open the Windows Explorer and go to the directory containing the Exercise Template files. The file names for use with QUEST are structured: <exercise number><variable laboratory code>.qxt, e.g. 639AD479.qxt

Double click on any *.qxt file to bring up the Open With dialogue box. Click on the button marked Other... , and select quest.exe from the C:\Program Files\ QUEST directory. QUEST will be added to the program list. Check the box marked “Always use this program to open this file”. Click <OK>. This sets the Windows Explorer to open QUEST by double clicking on any file with the extension <qxt>. Copies of the Exercise Template directories and files can be made if multiple users wish to enter data for different exercises.

The *.qxt files can be used from floppy disk or copied into a directory of your choice on the hard drive and used from there.

Please note :

If you are running Windows NT4 with User and Administrator privilege access you should install the QUEST program with administrator access but only use the QUEST program as a user.

Operating Procedures in QUEST 2.1

QUEST has been developed using the standard Windows nomenclature.

File menu : The file menu contains the standard Windows file options. This can be selected by clicking on File or <Alt>+F

Open (<Ctrl>+O) opens the file by bringing up the Explorer Directory structure. To open the file, highlight the required file, double click on the Open button using the mouse, or use the keyboard shortcut <Ctrl>+O.

Save (<Ctrl>+S) saves the open file to its directory. Once the file has been saved to the hard drive, it is possible to also save the data to disk. A dialogue box asks "Files saved to disk. Do you want to save a copy in A?". If "yes" then insert a disk in the A:\ drive and click on the "yes" button or press <Enter>. If you do not want a copy on disk, click on the "no" button.

The default option will always save the file to the root directory and not in the exercise directory on the A:\ drive. To save the data to the Exercise directory use the normal Windows save options.

Save As... Brings up the Save as... dialogue box. Select the required directory, enter the file name, if you require a different name, and click on the "Save" button or type <Alt>+S. Please do NOT use Save as to change the file name of the Exercise Template files that you return to the Project Office. The file name contains your laboratory code and the exercise number, which are essential identifiers for returned data.

Close (<Ctrl>+W) closes the file. If the file has been edited a dialogue box will appear. "Do you want to save the edits you have made?" Click on "Yes" or press <Enter> to save the data. Click on "No" to close the file without saving the data. "Cancel" allows you to return to the file without it closing.

Print (<Ctrl>+P). The print option takes the default setting of the Windows system. In the Data view a print out of the whole data table is provided. In the Methods view a printout of the Methods Codes is obtained for the determinand which is highlighted only. This saves replicate printouts for other determinands, which, in many cases, will have the same codes.

Edit The edit functions of Cut (<Ctrl>+X), Copy (<Ctrl>+C) and Paste (<Ctrl>+V) are similar to the standard Windows functions. In the View Values window, cut, copy and paste can only be done one cell at a time. Although the window gives the appearance of a spreadsheet, it is still a database entry.

Paste to All is only available in the Methods View and allows the highlighted method to be copied to all other determinands in that specific exercise.

View

The upper Window bar contains the Title of the Exercise.

In the Values View the lower Windows bar contains the data value in the highlighted cell, the directory of the file that is opened, and the current time.

In the Methods view the lower Windows bar contains the determinand/matrix name of the methods that are currently highlighted.

Values. The values window contains the analytical data. Column 1 lists the matrices, column 2 the determinands and column 3 the units for each of the determinands. These first three columns are not available for edit.

The value columns provide cells with the required number of observation for each determinand. For many of the routine measurements this will be a single column. Data may be entered into any of these cells. To enter the data highlight the required cell by clicking on the cell and enter the value.

To blank the cell, highlight the cell and press <delete>.

All the data are held in memory. To save the data to hard disc, use the Save or Save As... facility.

If the file has been edited and you try to exit the program, you will be asked if you want to save the edits that you have made. This is a standard function when closing any file after editing the data.

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[2]
- 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[3].
- 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[4].

[2] 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.

[3] 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.

[4] 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.