Quasimeme Laboratory Performance Studies

Round68

1 January 2012 to 30 April 2012

Exercise Protocols

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Introduction Round68

Thank you for participating in the 2012 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round68 that you have ordered will be sent to you by courier in the week beginning 9 January 2012. 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, 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 :

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All data for these studies must be uploaded to your Quasimeme SharePoint Site, using the data submission forms, no later than 30 April 2012

All other information should be sent to: QUASIMEME Project Office

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ROUND	68	Exercise 953
AQ-1 Nutrients in Seawater		
Test materials	QNU235SW, QNU236SW, QNU237SW	

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 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 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
QNU235SW	Seawater
QNU236SW	Seawater (Salinity > 30 psu) spiked
QNU237SW	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 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	68	Exercise 954
AQ-2 Nutrients in Estuarine and low salinity open water		
Test materials	QNU238EW, QNU239EW, QNU240EW, QNU241EW	

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
QNU238EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU239EW	Estuarine water (Salinity 8 - 20 psu) spiked
QNU240EW	Low salinity open water (Salinity 8 - 20 psu) spiked
QNU241EW	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	68	Exercise 955
AQ-14 DOC in seawater		
Test materials	QDC009SW, QDC010SW, QDC011EW, QDC012EW	

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 estuaria 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
QDC009SW	Seawater (unspiked)
QDC010SW	Seawater (spiked)
QDC011EW	Estuarine water (Salinity 10 -15 psu) spiked
QDC012EW	Estuarine water (Salinity 10 -15 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
		Sea water (spiked)	Estuarine water (spiked)	Const	Prop	
DOC	mg C/L	0.1 - 10	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 the DOC per test material is required. The results should be expressed on the test material "as received". The concentration of the DOC should be determined against your own calibration solutions.

Reporting

One result for DOC 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	68	Exercise 956
MS-1 Trace metals in Sediment		
Test materials	QTM098MS, QTM099MS	

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
QTM098MS	Sediment (open sea)
QTM099MS	Sediment (harbor)

Precaution

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

Determinands and concentration ranges

The following trace metals should be determined:

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

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%	
Methyl-Mercury-AE	µg/kg	50—2500	10	12.5%	
Methyl-Mercury-RT	µg/kg	50—2500	10	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.

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

Reporting of the results is splitted in reporting so called real totals methods (parameter-RT) and acid extractable methods (parameter-AE). Report only Real Total destructions e.g. HF-destructions, röntgen-diffraction and neutron activation as parameter-RT (e.g. Zinc-RT). Report acid extractable and all other methods as parameter-AE (e.g. nickel-AE).

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	68	Exercise 957
MS-2 Chlorinated Organics in Sediment		
Test materials	QOR110MS, QOR111MS	

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
QOR110MS	Sediment (harbor)
QOR111MS	Sediment (harbor)

Precaution

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

Determinands and concentration ranges

The following Chlorinated Organics should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
a-HCH	µg/kg	0.02—1	0.02	12.5%	
b-HCH	µg/kg	0.05—2	0.025	12.5%	
CB101	µg/kg	0.2—50	0.025	12.5%	
CB105	µg/kg	0.1—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 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	68	Exercise 958
MS-3 Polycyclic Aromatic Hydrocarbons in Sediment		
Test materials	QPH073MS, QPH074MS	

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
QPH073MS	Sediment (harbor)
QPH074MS	Sediment (harbor)

Precaution

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

Determinands and concentration ranges

The following 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 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	68	Exercise 959
MS-6 Organotins in Sediment		
Test materials	QSP040MS, QSP041MS	

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
QSP040MS	Sediment (harbor)
QSP041MS	Sediment (harbor)

Precaution

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

Determinands and concentration ranges

The following organotin compounds should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Sediment	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	1—5000	0.1	12.5%	
Diphenyltin(DPT)	µg Sn/kg	0.1—200	0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	1—5000	0.1	12.5%	
Monophenyltin(MPT)	µg Sn/kg	0.1—200	0.1	12.5%	
Tributyltin(TBT)	µg Sn/kg	1—5000	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 forms which are placed on the 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 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	68	Exercise 960
BT-1 Trace metals in Biota		
Test materials	QTM093BT, QTM094BT	

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
QTM093BT	Fish muscle tissue
QTM094BT	Fish liver tissue

Precaution

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

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

Determinands and concentration ranges

The following trace metals should be determined:

Determinand	Unit	Concentration range		Error		AA-EQS
		Fish muscle tissue	Fish liver tissue	Const	Prop	
Arsenic	mg/kg	1—10	1—5	0.02	12.5%	
Ash-weight	%			0.1	12.5%	
Cadmium	µg/kg	1—50	5—1000	20	12.5%	
Chromium	µg/kg	50—500	20—1000	20	12.5%	
Copper	µg/kg	100—1000	2000—10000	100	12.5%	

Dry-weight	%			0.1	12.5%	
Extractable-Lipid	%			0.1	12.5%	
Lead	µg/kg	10—50	10—1000	5	12.5%	
Mercury	µg/kg	20—1000	20—100	20	12.5%	
Nickel	µg/kg	10—200	20—1000	20	12.5%	
Selenium	µg/kg	200—2000	200—5000	10	12.5%	
Silver	µg/kg	0.5—50	20—1000	5	12.5%	
Total-Lipid	%			0.1	12.5%	
Zinc	mg/kg	2—10	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

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 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	68	Exercise 961
BT-2 Chlorinated Organics in Biota		
Test materials	QOR110BT, QOR111BT	

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 or jars 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
QOR110BT	mussel tissue
QOR111BT	Freshwater fish muscle tissue

Precaution

The tins or 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 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
		Fish muscle tissue	Shellfish tissue	Const	Prop	
a-HCH	µg/kg	0.5 - 5	0.05—5	0.02	12.5%	
b-HCH	µg/kg	0.5 – 5	0.05—5	0.025	12.5%	
CB101	µg/kg	30 – 300	0.1—20	0.025	12.5%	
CB105	µg/kg	10 – 100	0.05—10	0.025	12.5%	
CB118	µg/kg	30 – 300	0.2—20	0.025	12.5%	
CB138	µg/kg	60 – 600	0.3—30	0.025	12.5%	
CB138+CB163	µg/kg	60 – 600	0.3—30	0.025	12.5%	
CB153	µg/kg	100 – 1000	0.4—40	0.025	12.5%	
CB156	µg/kg	3 – 40	0.03—10	0.025	12.5%	
CB180	µg/kg	20 – 200	0.05—5	0.025	12.5%	
CB28	µg/kg	5 – 50	0.05—5	0.025	12.5%	
CB31	µg/kg	1 – 10	0.03—3	0.025	12.5%	
CB52	µg/kg	10 – 100	0.05—5	0.025	12.5%	
d-HCH	µg/kg		0.05—5	0.025	12.5%	
Dieldrin	µg/kg	10 – 100	0.2—20	0.025	12.5%	
Extractable-Lipid	%			0.1	12.5%	
g-HCH	µg/kg	0.2 – 5	0.05—5	0.025	12.5%	
HCB	µg/kg	5 – 50	0.02—5	0.025	12.5%	
HCBD	µg/kg	0.1 – 50		0.025	12.5%	
op'-DDT	µg/kg	0.1 – 2	0.01—1	0.025	12.5%	
pp'-DDD	µg/kg	10 – 100	0.1—10	0.025	12.5%	
pp'-DDE	µg/kg	10 – 100	0.3—30	0.025	12.5%	
pp'-DDT	µg/kg	0.3 – 10	0.1—10	0.025	12.5%	
Total-Lipid	%			0.1	12.5%	
Transnonachlor	µg/kg	3 - 40	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.

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 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	68	Exercise 963
BT-4 Polycyclic Aromatic Hydrocarbons in Biota		
Test materials	QPH065BT, QPH066BT	

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 or jars 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
QPH065BT	Shellfish tissue
QPH066BT	Shellfish tissue

Precaution

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

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

Determinands and concentration ranges

The following 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%	
1-methyl naphthalene	µg/kg		0.2	12.5%	
2-methyl naphthalene	µg/kg		0.2	12.5%	
2-methyl anthracene	µg/kg		0.2	12.5%	
C1-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C2-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C3-phenanthrenes/anthracenes	µg/kg		0.2	12.5%	
C1-pyrenes/fluoranthenes	µg/kg		0.2	12.5%	
C2-pyrenes/fluoranthenes	µg/kg		0.2	12.5%	
C1-Chrysenes	µg/kg		0.2	12.5%	
C2-Chrysenes	µg/kg		0.2	12.5%	
C1-benzofluoranthenes	µg/kg		0.2	12.5%	

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 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	68	Exercise 964
BT-8 Organotins in Biota		
Test materials	QSP040BT, QSP041BT	

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 or jars 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
QSP040BT	Shrimp tissue
QSP041BT	Mussel tissue

Precaution

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

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

Determinands and concentration ranges

The following analytes should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
		Biota	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	0.1—10	0.1	12.5%	
Diphenyltin(DPT)	µg Sn/kg		0.1	12.5%	
Monobutyltin(MBT)	µg Sn/kg	0.5—30	0.1	12.5%	
Monophenyltin(MPT)	µg Sn/kg		0.1	12.5%	
Tributyltin(TBT)	µg Sn/kg	0.2—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 (µg 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 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	68	Exercise 965
BT-9 (Formerly BS-1) Brominated flame retardants in Biota		
Test materials	QBC032BT, QBC033BT	

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
QBC032BT	Fish tissue (bream)
QBC033BT	Fish tissue (flounder)

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.

$$\% \text{ dry weight} = 100 - [(\text{wet weight} - \text{dry weight}) * 100]/(\text{wet weight})$$

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.

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 30 October 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:

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 30 December 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, 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 in capitals

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.