WAGENINGEN EVALUATING PROGRAMMES
FOR ANALYTICAL LABORATORIES

Quasimeme Laboratory Performance Studies

Round65

1 April 2011 to 30 July 2011

Exercise Protocols

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Introduction Round65

Thank you for participating in the 2011 QUASIMEME Laboratory Performance studies.

The test materials for the exercises in Round65 that you have ordered will be sent to you by courier in the week beginning 11 April 2011. 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 July 2011

All other information should be sent to: QUASIMEME Project Office

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ROUND	65	Exercise 924
AQ-3 Metals in Seawater		
Test materials	QTM163SW, QTM164SW, QTM165SW	

Objective

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

Test Materials and storage

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

The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm /0.2µm double-membrane filter. Low salinity seawater test material was prepared by diluting the seawater with ultra-pure demineralised water. All test materials are preserved with 2 ml trace metal grade nitric acid per litre of seawater. Spiked test materials were prepared by adding aqueous solutions of known trace metal concentration. Approximately 1 litre of each test material is provided. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre polypropylene bottles. The test materials are stable for the purposes of the exercise.

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

Code	Description
QTM163SW	Seawater (Salinity > 30 psu)
QTM164SW	Seawater (Salinity > 30 psu) spiked
QTM165SW	Low salinity Seawater (Salinity 8 - 20 psu) spiked

Precaution

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

Analysis

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

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

Only one result per determinand per test material is required.

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

Determinands and concentration ranges

The following metals should be determined:

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

Boron is naturally occurring at higher concentrations.
Results should be reported for as many of these determinands as possible. Take this opportunity either to develop your methodology or check your performance on the less common determinands.

ROUND	65	Exercise 925
AQ-4 Mercury in Seawater		
Test materials	QTM166SW, QTM167SW, QTM168SW	

Objective

This study covers the determination of mercury in the seawater test materials. The test materials should be analysed and one result for mercury in each test material should be reported using the data submission forms provided on the sharepointsite.

Test Materials and storage

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

The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. All test materials are preserved with 2 ml trace metal grade nitric acid per litre of seawater. Test materials were spiked with aqueous solutions of known mercury concentration.

Approximately 1 litre of each test material is provided.

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

Code	Description
QTM166SW	Seawater (Salinity > 30 psu) spiked
QTM167SW	Seawater (Salinity > 30 psu) spiked
QTM168SW	Seawater (Salinity > 30 psu) spiked

Precaution

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

Determinands and concentration ranges

Mercury should be determined in each test material.

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

Analysis

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

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

Only one result per test material is required.

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 926
AQ-5 Halogenated Organics in Seawater		
Test materials		QOC070SS, QOC070SW, QOC071SS, QOC071SW, QOC072SS, QOC072SW

Objective

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

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Low salinity seawater test material are prepared by dilution with ultra-pure demineralised water, to a salinity of approximately 12 - 18 psu. Test materials need to be spiked with organochlorine composite solutions in methanol by the participants themselves (see Analysis section). Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre glass bottles. The test materials are stable for the purposes of the exercise. Approximately 1 litre of each test material is provided. Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

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

Code	Description
QOC070SS	Spiking solution to use for QOC070SW
QOC070SW	Seawater with Spiking solution
QOC071SS	Spiking solution to use for QOC071SW
QOC071SW	Seawater with Spiking solution
QOC072SS	Spiking solution to use for QOC072SW
QOC072SW	Low salinity Seawater with Spiking solution

Precaution

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

Determinands and concentration ranges

The following organochlorine compounds should be determined:

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
1,2,3-TCB	ng/L		0.5	12.5%	400
1,2,4-TCB	ng/L		0.5	12.5%	400
1,3,5-TCB	ng/L		0.5	12.5%	400
a-HCH	ng/L		0.2	12.5%	2
Aldrin	ng/L		0.5	12.5%	5
b-HCH	ng/L		0.2	12.5%	2
d-HCH	ng/L		0.2	12.5%	2
Dieldrin	ng/L		0.5	12.5%	5
Endosulphan-I	ng/L		0.2	12.5%	0.5
Endosulphan-II	ng/L		0.2	12.5%	0.5
Endrin	ng/L		0.5	12.5%	5

g-HCH	ng/L		0.2	12.5%	2
HCB	ng/L		0.2	12.5%	10
HCBD	ng/L		0.2	12.5%	100
Isodrin	ng/L		0.5	12.5%	5
op'-DDT	ng/L		0.5	12.5%	25
Pentachlorobenzene	ng/L		0.5	12.5%	0.7
pp'-DDD	ng/L		0.5	12.5%	25
pp'-DDE	ng/L		0.5	12.5%	25
pp'-DDT	ng/L		0.5	12.5%	10
Trifluralin	ng/L		0.5	12.5%	30

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

Analysis

Preparation and Analysis

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

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

- The spiking solution should be stabilised at 20˚C

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

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

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

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

- Analyse the test materials immediately after preparation.

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 927
AQ-6 Volatile Organics in Seawater		
Test materials	QVC049SW, QVC050SW	

Objective

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

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Test materials were spiked with the volatile organochlorine composite solution in methanol with known concentration. Flasks were completely filled with test material. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre glass bottles. The test materials are stable for the purposes of the exercise. Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

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

Code	Description
QVC049SW	Seawater (Salinity > 30 psu) spiked
QVC050SW	Seawater (Salinity > 30 psu) spiked

Precaution

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

Determinands and concentration ranges

The following analytes should be determined:

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

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

Analysis

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 928
AQ-7 Pentachlorophenol in Seawater		
Test materials		QPP045SS, QPP045SW, QPP046SS, QPP046SW, QPP047SS, QPP047SW

Objective

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

Test Materials and storage

The test materials were prepared at Alterra, Wageningen, The Netherlands. The seawater used to prepare the test materials was collected from the North Sea between Belgium and the UK, and was stored in the cold store at 7˚C in 25 litre carboys. The test materials were prepared in bulk in a 100 litre vessel. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Test materials need to be spiked with pentachlorophenol solutions in methanol by the participants themselves (see Analysis section). Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre glass bottles. The test materials are stable for the purposes of the exercise. Approximately 1 litre of each test material is provided. Test materials should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately.

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

Code	Description
QPP045SS	Spiking solution to use for QPP045SW
QPP045SW	Seawater with Spiking solution
QPP046SS	Spiking solution to use for QPP046SW
QPP046SW	Seawater with Spiking solution
QPP047SS	Spiking solution to use for QPP047SW
QPP047SW	Seawater with Spiking solution

Precaution

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

Determinands and concentration ranges

Pentachlorophenol should be determinated in each test material.

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

Analysis

Preparation and Analysis

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

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

- The spiking solution should be stabilised at 20˚C

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

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

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

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

- Analyse the test materials immediately after preparation.

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 929
AQ-8 Triazines and organophosphorus compounds in the seawater		
Test materials		QTP076SS, QTP076SW, QTP077SS, QTP077SW, QTP078SS, QTP078SW

Objective

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

Test Materials and storage

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

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

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

Homogeneity of the test materials is assumed, as they were prepared from the same bulk seawater, and the spiking solutions were also prepared in bulk. The test materials are stable for the purposes of the exercise. Test materials (seawater and spiking solutions) should be stored in a refrigerator at +4˚C, and analysed as soon as possible after receipt. Once the test materials are opened they should be analysed immediately. Treat all test materials in the same manner as your routine samples.

Code	Description
QTP076SS	Spiking solution to use for QTP076SW
QTP076SW	Seawater with Spiking solution
QTP077SS	Spiking solution to use for QTP077SW
QTP077SW	Seawater with Spiking solution
QTP078SS	Spiking solution to use for QTP078SW
QTP078SW	Low salinity Seawater with Spiking solution

Precaution

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

Determinands and concentration ranges

The following analytes should be determined:

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

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

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

Analysis

Preparation and Analysis

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

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

- The spiking solution should be stabilised at 20˚C

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

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

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

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

- Analyse the test materials immediately after preparation.

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

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 930
AQ-11	Chlorophyll-a in Seawater	
Test materials	QCH050SW, QCH051SW and QCH052SW	

Objective

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

Test Materials and storage

The test materials for the analysis of chlorophyll a, b, c and pheopigments were prepared at Alterra, Wageningen the Netherlands. Test materials were prepared from cultures of Chaetocheros + Pyramimonas (QCH050SW) and Nanochloropsis and Pavlova lutheri (QCH052SW) grown at Wageningen IMARES, Yerseke the Netherlands and sub-sampled onto Whatman GF/F, 47 mm filter papers. The QCH051SW sample was a blank seawater sample diluted with tapwater and spiked with algae collected from a freshwater lake, simulating an estuarine chlorophyll sample. For each test material, the resultant damp filter paper was wrapped in aluminium foil, inserted into cryovial and immediately 'flash frozen' in liquid nitrogen. The test materials were stored at -80˚C until the day of dispatch. The extra sample (QCH052SW) was sent as a more difficult extractable chlorophyll sample.

The test materials were homogeneous for the purposes of the LP study.

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

Code	Description
QCH050SW	Filtered residues from 1 litre of seawater
QCH051SW	Filtered residues from 1 litre of estuarine water
QCH052SW	Filtered residues from 1 litre of seawater

Precaution

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

Determinands and concentration ranges

The following pigments should be determined:

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

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

Analysis

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

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 931
AQ-12 Organotins in Seawater		
Test materials	QSP033SS, QSP033SW, QSP034SS, QSP034SW	

Objective

This study covers the determination of organotin compounds in the seawater test materials QSP033SW and QSP034SW, which has to be spiked in your own laboratory. The samples are not preserved, as they will be spiked on your own laboratory.

Test Materials and storage

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

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

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

Code	Description
QSP033SS	Spiking solution to use for QSP033SW
QSP033SW	Seawater with Spiking solution
QSP034SS	Spiking solution to use for QSP034SW
QSP034SW	Seawater with Spiking solution

Precaution

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

Determinands and concentration ranges

The following analytes should be determined:

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

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

Determinand	Unit	Concentration range	Error		AA-EQS
		Seawater with SS	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	0.001—0.05	0.05	12.5%	0.0002
Diphenyltin(DPT)	µg Sn/kg	0.001—0.1	0.05	12.5%	
Monobutyltin(MBT)	µg Sn/kg	0.001—0.02	0.05	12.5%	0.0002
Monophenyltin(MPT)	µg Sn/kg	0.001—0.05	0.05	12.5%	
Tributyltin(TBT)	µg Sn/kg	0.001—0.1	0.05	12.5%	0.0002
Triphenyltin(TPT)	µg Sn/kg	0.001—0.2	0.05	12.5%	

Analysis

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

Only one result per determinand per test material is required.

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

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

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

- The spiking solution should be stabilised at 20˚C

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

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

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

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 932
AQ-13 Polycyclic Aromatic Hydrocarbons in Seawater		
Test materials		QPH007SS, QPH007SW, QPH008SS, QPH008SW, QPH009SW, QPH021SS

Objective

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

Test Materials and storage

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

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

Methanol solutions containing known concentrations of PAHs compounds (QPH007SS and QPH008SS) were prepared in bulk and ampouled to make the spiking solutions.

The test material QPH009SW was prepared in bulk in a 5 litre flask. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. The seawater (4 litre) used to prepare test material QPH009SW was spiked with approximately 1,2 gram highly contaminated (with PAHs) colloidal milled harbour sediment. The flask with seawater and fine sediment was shaken intensively on a shaking apparatus for two hours. Following a stagnant period of 30 minutes most of the waterlayer was decanted in a 5 litre flask. Stirring the content of this flask, a subsample of 120 ml was transferred into each sample flask and these flasks were filled up to a volume of 0.750 litre with blank seawater. This sample should be analysed as a so called total water sample.

Standard solution QPH021SS was prepared by diluting a commercial standard solution containing several PAHs into acetonitril.

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
QPH007SS	Spiking solution to use for QPH071SW
QPH007SW	Seawater with Spiking solution
QPH008SS	Spiking solution to use for QPH072SW
QPH008SW	Seawater with Spiking solution
QPH009SW	Seawater (Salinity > 30 psu) spiked using Sediment
QPH021SS	Standard Solution

Precaution

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

Determinands and concentration ranges

The following trace PAHs should be determined and the indicative concentrations are given. These indicative concentrations sometimes differ from the indication ranges given in the Quasimeme guide.

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

Determinand	Unit	Concentration range		Error		AA-EQS
		Standard Solution	Seawater (sediment spiked)	Const	Prop	
Acenaphthene	µg/L	50—2000	0.2—20	0.01	12.5%	
Acenaphthylene	µg/L	50—2000	0.01—1	0.01	12.5%	
Anthracene	µg/L	50—2000	0.2—20	0.01	12.5%	0.1
Benzo[a]pyrene	µg/L	50—2000	0.1—10	0.01	12.5%	0.05
Benzo[b]fluoranthene	µg/L	50—2000	0.1—10	0.01	12.5%	0.03
Benzo[g,h,i]perylene	µg/L	50—2000	0.02—2	0.01	12.5%	0.002
Benzo[k]fluoranthene	µg/L	50—2000	0.1—10	0.01	12.5%	0.03
Fluoranthene	µg/L	50—2000	0.4—40	0.01	12.5%	0.1
Indeno(1,2,3-cd)pyrene	µg/L	50—2000	0.4—40	0.01	12.5%	0.002
Naphtalene	µg/L	50—2000	0.1—10	0.01	12.5%	1.2
Phenanthrene	µg/L	50—2000	0.5—50	0.01	12.5%	

Analysis

Preparation and Analysis of the seawater

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

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

- The spiking solution should be stabilised at 20˚C

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

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

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

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

- Analyse the test materials immediately after preparation.

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 933
BT-7 ASP Shellfish Toxins		
Test materials	QST105SS, QST106BT, QST115BT	

Objective

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

Test Materials and storage

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

- QST105SS is a domoic acid standard solution.

- QST106BT is a Clam tissue homogenate supplied in a plastic vial.

- QST115BT is a Mussel tissue homogenate supplied in a plastic vial.

For QST106BT and QST115BT, each vial contains sufficient material for one-shot analysis of domoic and epidomoic acid.

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

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

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

The Standard solution QST105SS should be stored in the refrigerator at ca 4˚C immediately upon receipt, until analysis

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

Code	Description
QST105SS	Standard Solution
QST106BT	Shellfish tissue
QST115BT	Shellfish tissue

Precaution

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

Determinands and concentration ranges

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

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

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

Analysis

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

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

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

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

ROUND	65	Exercise 934
DE-10 DSP Shellfish Toxins		
Test materials	QST107SS, QST108BT, QST109BT and QST110BT	

Objective

This study covers the determination of DSP and AZP toxins in shellfish tissue.

Test Materials and storage

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

Each vial contains sufficient material for one-shot analysis of OA, DTX1, DTX2 and their esters AZA-1, AZA-2 and AZA-3.

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

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

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

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

Code	Description
QST107SS	AZA Standard Solution
QST108BT	DSP/AZP extract
QST109BT	Shellfish tissue (mussel)
QST110BT	Shellfish tissue (mussel)

Precaution

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

Determinands and concentration ranges

Determinands

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

For the standard solution QST107SS report concentrations for the azaspiracids AZA-1, AZA-2 and AZA-3 individually and their sum. For the mussel crude extract and the mussel tissues, report concentrations for OA, DTX1, DTX2 as free toxins (pre-hydrolysis), separately and their sum, and for total OA, DTX1 and DTX2 (post-hydrolysis), and the sum of the total toxins post-hydrolysis (hy-OA + hy-DTX1 + hy-DTX2). This means there is no result reported for the ester-forms themselves, only for free toxins and the sum of free toxins plus esters. Also report the azaspiracids AZA-1, AZA-2 and AZA-3 individually and their sum.

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

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

Please report concentrations of OA, DTX-1, DTX-2 and their esters as well as the TEQ values. Calculating the TEQ values, use the TEF factors used in your own laboratory or use the TEF factors recommended by the EFSA . Report only TEQ values for the azaspiracids AZA-1, AZA-2 and AZA-3.

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
AZA-1	µg/kg		0.1	12.5%	
AZA-1 TEQ	TEQ		0.1	12.5%	
AZA-2	µg/kg		0.1	12.5%	
AZA-2 TEQ	TEQ		0.1	12.5%	
AZA-3	µg/kg		0.1	12.5%	
AZA-3 TEQ	TEQ		0.1	12.5%	
AZA-total	µg/kg		0.1	12.5%	
AZA-total TEQ	TEQ		0.1	12.5%	
Free-DTX1	µg/kg		0.1	12.5%	
Free-DTX1 TEQ	TEQ		0.1	12.5%	
Free-DTX2	µg/kg		0.1	12.5%	
Free-DTX2 TEQ	TEQ		0.1	12.5%	
Free-Okadaic-Acid	µg/kg		0.1	12.5%	
Free-Okadaic-Acid TEQ	TEQ		0.1	12.5%	
Total-DTX1	µg/kg		0.1	12.5%	
Total-DTX1 TEQ	TEQ		0.1	12.5%	
Total-DTX2	µg/kg		0.1	12.5%	
Total-DTX2 TEQ	TEQ		0.1	12.5%	
Total-Free-OA+DTX1+DTX2	µg/kg		0.1	12.5%	
Total-Free-OA+DTX1+DTX2 TEQ	TEQ		0.1	12.5%	
Total-hy-OA+DTX1+DTX2	µg/kg		0.1	12.5%	
Total-hy-OA+DTX1+DTX2 TEQ	TEQ		0.1	12.5%	
Total-Okadaic-Acid	µg/kg		0.1	12.5%	
Total-Okadaic-Acid TEQ	TEQ		0.1	12.5%	

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

Analysis

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

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

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

You should inform the QUASIMEME office staff, who will arrange to prepare an additional data submission form. It is not possible to report two sets of data using different methods on the same exercise template. Please use the View Methods function in the data submission forms to report your analytical method for each test material.

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

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

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

All results should be reported in µg/kg on the basis of wet weight of the test material as provided. The density of the standard solution is 0.7918 g/ml (MeOH) and the density of the DSP/AZP extract is 0.834g/ml. For the mussel tissues, the weight of material should be determined prior to analysis.

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

Reporting

One result for each determinand in each test material should be reported using the data submission forms which are placed on the 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	65	Exercise 935
DE-14 PSP Shellfish Toxins		
Test materials	QST111BT, QST112BT, QST113BT and QST114BT	

Objective

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

Test Materials and storage

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

Shellfish tissue test materials are supplied in a plastic 5ml vial, each vial contains sufficient material for one-shot analysis of the paralytic shellfish toxins.

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

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

The shellfish tissue test materials should be stored at -20˚C, or a lower temperature, immediately upon receipt, until analysis. The test materials have been shipped on cool packs. The cool packs will thaw slowly during transit, but this does not significantly affect the stability of the material for the purpose of this study, providing the test materials themselves are stored as directed, immediately on receipt.

Code	Description
QST111BT	Shellfish tissue
QST112BT	Shellfish tissue
QST113BT	Shellfish tissue
QST114BT	Shellfish tissue

Precaution

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

Determinands and concentration ranges

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

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

Determinand	Unit	Concentration range	Error		AA-EQS
			Const	Prop	
11-OH-STX	µmol/kg		0.1	12.5%	
C1	µmol/kg		0.1	12.5%	
C2	µmol/kg		0.1	12.5%	
C3	µmol/kg		0.1	12.5%	
C4	µmol/kg		0.1	12.5%	
dc-GTX1	µmol/kg		0.1	12.5%	
dc-GTX2	µmol/kg		0.1	12.5%	
dc-GTX3	µmol/kg		0.1	12.5%	
dc-GTX4	µmol/kg		0.1	12.5%	
dc-NEO	µmol/kg		0.1	12.5%	
dc-STX	µmol/kg		0.1	12.5%	
GTX1	µmol/kg		0.1	12.5%	
GTX2	µmol/kg		0.1	12.5%	
GTX3	µmol/kg		0.1	12.5%	
GTX4	µmol/kg		0.1	12.5%	
GTX5	µmol/kg		0.1	12.5%	
GTX-6	µmol/kg		0.1	12.5%	
NEO	µmol/kg		0.1	12.5%	
STX	µmol/kg		0.1	12.5%	
Total toxicity	µgSTXdiHCl-eq/kg		2	12.5%	

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

TEFs recommended by the EFSA

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

Analysis

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

Each vial contains sufficient quantity of homogenate for one analysis. The whole transferable contents of each vial should be extracted, and one result reported for the sum of the two isomers (If participants are using the AOAC 2005.06 method then those toxins that co-elute (eg GTX1 and GTX-4, GTX-2 and GTX-3, dcGTX-2 and dcGTX-3 or C-1 and C-2) must be reported using the higher toxicity factor of the two isomers. For example if participants find the presence of GTX-1,4 (co-eluting) in the sample then they should report the sum of the two isomers in the GTX-1 column in the reporting

template as GTX-1 has the higher toxicity factor). To transfer the contents into a preweighed or tared extraction tube, the vial should be fully defrosted, vortex-mixed and the contents poured into the desired container.

All results should be reported in TEQ values on the basis of wet weight of the test material as provided. The weight of the shellfish tissue test material should be determined prior to analysis.

Reporting

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

It is not possible to report two sets of data using different methods in the same data submission forms.

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

Reporting of Results and Analytical Methods

Units

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

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

Reporting Left Censored Values

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

Method Codes

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

Return of Data

Upload all analytical data to the QUASIMEME SharePoint site only using 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 the data submission forms can be included in the assessment. Return the results to the QUASIMEME Project Office in Wageningen no later than 30 July 2011. 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 September 2011. Background information on the data assessment will be provided with the reports.

Collusion and Falsification of Results

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

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

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

QUASIMEME reserves the right to withdraw participation of any institute who, in the opinion of the Scientific Assessment Group, has submitted data following collusion or falsification. This statement is made as a formal requirement for accreditation for Laboratory Performance Studies under G13: 2000 3.9.

ANNEX 1 Notification of damaged test materials.

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

Laboratory Code : .....

Damaged container number : .....

Loss of weight container number : .....

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

Date : .....

Signature :.....

Name of participant :.....

Name and address of institute : .....

.....

.....

Telephone number :.....

Fax number :.....

Return this form to :

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

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

ANNEX 2 Instructions for login into sharepointsite

Login to http://www.quasimeme.org

Select sharepointsite

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

Password: your specific password

Ask the Quasimeme project office when the login information is unknown

Select the correct year

Select the correct round

Select the correct exercise

Enter your results and method information into the data submission form

Lower than results will be automatically transferred into – values.

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