Quasimeme Laboratory Performance Studies

Round61

1 April 2010 to 30 July 2010

Exercise Protocols

Version 1.1 (01 June 2010)

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Introduction Round61

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

The test materials for the exercises in Round61 that you have ordered will be sent to you by courier in the week beginning 12 April 2010. Please check that the contents of your package are correct and that all test materials are intact. If any test materials have been damaged in transit or if the wrong samples have been send, use the form in Annex 1 of this document to request replacement materials within two weeks after receipt of the test materials.

Additional test materials may also be purchased from QUASIMEME.

This protocol covers the following studies :

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

All other information should be sent to: QUASIMEME Project Office

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

ROUND	61	Exercise 882
AQ-3 Metals in Seawater		
Test materials	QTM151SW, QTM152SW, QTM153SW	

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 50 litre aspirators. The seawater was filtered using a 0.45 µm /0.2µm double-membrane filter. Low salinity seawater test material was prepared by diluting the seawate 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
QTM151SW	Seawater (Salinity > 30 psu) blank
QTM152SW	Seawater (Salinity > 30 psu) spiked
QTM153SW	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.

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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 883
AQ-4 Mercury in Seawater		
Test materials	QTM154SW, QTM155SW, QTM156SW	

Objective

This study covers the determination of mercury 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 in bulk in 50 litre aspirators. 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
QTM154SW	Seawater (Salinity > 30 psu) spiked
QTM155SW	Seawater (Salinity > 30 psu) spiked
QTM156SW	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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 884
AQ-5 Halogenated Organics in Seawater		
Test materials		QOC067SS, QOC067SW, QOC068SS, QOC068SW, QOC069SS, QOC069SW

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 25 litre aspirators. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filte Low salinity seawater test material are prepared by dilution with ultra-pure demineralised water, to a salinity of approximately 12 - 18 psu. Methanol solutions containing known concentrations of halogenated organic compounds 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
QOC067SS	Spiking solution to use for QOC067SW
QOC067SW	Seawater with Spiking solution
QOC068SS	Spiking solution to use for QOC068SW
QOC068SW	Seawater with Spiking solution
QOC069SS	Spiking solution to use for QOC069SW
QOC069SW	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 compunds 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
Pentabromodiphenylether	ng/L		0.5	12.5%	0.2
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 is recommended. In case you use a syringe note that the density of the spike 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 980 gram with the seawater provided and mix thoroughly. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Please calculate the volume of seawater used with a density of the seawater of 1.025 (kg/L). So 980 gram of seawater is equal to 956 ml of seawater.

- 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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 885
AQ-6 Volatile Organics in Seawater		
Test materials	QVC047SW, QVC048SW	

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 25 litre aspirators. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filter. Test material were spiked with the volatile organochlorine composite solution in methanol with known concentration. Glass beads were added to the test materials to raise the headspace in order to prevent volatilisation of the spiking solution. 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.

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

Code	Description
QVC047SW	Seawater (Salinity > 30 psu) spiked
QVC048SW	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 volatile organochlorines should be determined against your own calibration solutions.

Reporting

One result for each determinand in each test material should be reported using the data submission form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 886
AQ-7 Pentachlorophenol in Seawater		
Test materials	QPP042SS, QPP042SW, QPP043SS, QPP043SW, QPP044SW	

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 25 litre aspirators. The seawater was filtered using a 0.45 µm / 0.2 µm double-membrane filte Methanol solutions containing known concentrations of pentachlorophenol 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
QPP042SS	Spiking solution to use forQPP042SW
QPP042SW	Seawater with spiking solution
QPP043SS	Spiking solution to use for QPP043SW
QPP043SW	Seawater with spiking solution
QPP044SW	Blank 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

Pentachlorophenol should be determinated in each test material.

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

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 is recommended. In case you use a syringe note that the density of the spike 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 980 gram with the seawater provided and mix thoroughly. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Please calculate the volume of seawater used with a density of the seawater of 1.025 (kg/L). So 980 gram of seawater is equal to 956 ml of seawater.

- 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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 887
AQ-8 Triazines and organophosphorus compounds in the seawater		
Test materials		QTP073SS, QTP073SW, QTP074SS, QTP074SW, QTP075SS, QTP075SW

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 25 litre aspirators. 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
QTP073SS	Spiking solution to use for QTP073SW
QTP073SW	Seawater with Spiking solution
QTP074SS	Spiking solution to use for QTP074SW
QTP074SW	Low salinity Seawater with Spiking solution
QTP075SS	Spiking solution to use for QTP075SW
QTP075SW	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 is recommended. In case you use a syringe note that the density of the spike 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 980 gram with the seawater provided and mix thoroughly. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Please calculate the volume of seawater used with a density of the seawater of 1.025 (kg/L). So 980 gram of seawater is equal to 956 ml of seawater.

- 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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 888
AQ-11	Chlorophyll-a in Seawater	
Test materials	QCH046SW, QCH047SW	

Objective

This study covers the determination of chlorophyll a, b, c and pheopigments in two 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 grown at Wageningen IMARES, Yerseke the Netherlands and sub-sampled onto Whatman GF/F, 47 mm filter papers. For each test material, the resultant damp filter paper was wrapped in aluminium foil, inserted into a numbered cryovial and immediately 'flash frozen' in liquid nitrogen. The test materials were stored at -80˚C until the day of dispatch.

Samples were selected for homogeneity testing. 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
QCH046SW	Filtered residues from 1 litre of seawater
QCH047SW	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%	

Concentrations need to be calculated based on a filter prepared out of a 1 litre sample.
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). For those who wish to use these methods, the detailed "Protocol for the Spectrophotometric Determination of Chlorophyll-a and Pheopigments" is provided in the email as "Chlorophyll Spectrophotometric Protocol.pdf". This paper is a condensed version of the original ICES TIMES document No. 31, and contains details of the Trichromatic Method (Jeffery and Humphrey 1975) and the Monochromatic Method with acidification (Lorenzen 1967). This protocol only covers spectrophotometric methods.

Reporting

One result for each determinand in each test material should be reported using the data submission form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 889
AQ-12 Organotins in Seawater		
Test materials	QSP031SS, QSP031SW, QSP032SS, QSP032SW	

Objective

This study covers the determination of organotin compounds 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.

The solution of organotins have been prepared under dry conditions and a nitrogen atmosphere. The main cause of degradation of the tin solutions is the presence of water. It is important that the ampoule containing this solution is used or opened only when you are ready to complete the analysis. Please check the ampoule to ensure it has not been damaged during transit.

The test materials were prepared in bulk. All material provided for the test period were prepared with an assured level of homogeneity for a given mass of sample, and stability for the purposes of the test.

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
QSP031SS	Spiking solution to use for QSP031SW
QSP031SW	Seawater with spiking solution
QSP032SS	Spiking solution to use for QSP032SW
QSP032SW	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 (spiked)	Seawater with SS	Const	Prop	
Dibutyltin(DBT)	µg Sn/kg	0.001—0.05	0.001—0.05	0.05	12.5%	0.0002
Diphenyltin(DPT)	µg Sn/kg	0.001—0.1	0.001—0.1	0.05	12.5%	
Monobutyltin(MBT)	µg Sn/kg	0.001—0.02	0.001—0.02	0.05	12.5%	0.0002
Monophenyltin(MPT)	µg Sn/kg	0.001—0.05	0.001—0.05	0.05	12.5%	
Tributyltin(TBT)	µg Sn/kg	0.001—0.2	0.001—0.1	0.05	12.5%	0.0002
Triphenyltin(TPT)	µg Sn/kg	0.001—0.2	0.001—0.2	0.05	12.5%	

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

Analysis

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 is recommended. In case you use a syringe note that the density of the spike 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 980 gram with the seawater provided and mix thoroughly. 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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 890
AQ-13 Polycyclic Aromatic Hydrocarbons in Seawater		
Test materials	QPH004SS, QPH004SW, QPH005SS, QPH005SW, QPH006SW	

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 material spiking solutions were prepared by diluting a certified reference material with different PAHs in acetonitril as well as in dichloromethane for both LC and GC applications. The test material QPH002SW was prepared by spiking 750 ml of filtered (0.45 µm/0.20 µm double membrane filter) seawater with a known amount of PAHs in methanol solution. Due to possible adsorption of PAHs onto glass we ask you kindly to analyse the flask with the watersample as a whole.

The sediment spiked test material 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 prepar this test materil was spiked with approximately 1,5 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 150 ml was transferred into each sample flask and these flasks were filled up to a volume of 1 litre with blank seawater. This sample should be analysed as a so called total water sample. Conservation of the testsamples was carried out by adding mercurychloride at a concentration of 12,5 mg/L to the testsamples.

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
QPH004SS	Spiking solution to use for QPH004SW
QPH004SW	Seawater with spiking solution
QPH005SS	Spiking solution to use for QPH005SW
QPH005SW	Seawater with spiking solution
QPH006SW	Seawater spiked using Sediment

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%	

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 is recommended. In case you use a syringe note that the density of the spike 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 980 gram with the seawater provided and mix thoroughly. Do not add the spiking solution to the bottle of seawater, as the bottle contains approximately 1 litre of water.

- Please calculate the volume of seawater used with a density of the seawater of 1.025 (kg/L). So 980 gram of seawater is equal to 956 ml of seawater.

- 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.

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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 891
BT-7 ASP Shellfish Toxins		
Test materials	QST085SS, QST086BT, QST087BT	

Objective

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

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 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 7ml plastic vials) should be stored at -20˚C, or a lower temperature, immediately upon receipt, until analysis.

The Standard solution 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
QST085SS	Standard Solution
QST086BT	Shellfish tissue
QST087BT	Shellfish tissue

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

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 shelfish 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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 892
DE-10 DSP Shellfish Toxins		
Test materials	QST088SS, QST089SS, QST090BT, QST091BT, QST092BT	

Objective

This study covers the determination of diarrhetic 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.

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 7ml 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
QST088SS	Standard Solution (okadaic acid)
QST089SS	Standard Solution (AZA)
QST090BT	DSP/AZP extract
QST091BT	Shellfish tissue
QST092BT	Shellfish tissue

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

Determinands

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

For the standard solution QST088SS, report concentrations for OA, DTX1, DTX2 as free toxins (without hydrolysis), individual analogues separately and their sum. For the standard solution QST089SS report concentrations for the azaspiracids AZA1, AZA2 and AZA3 individually and their sumb).

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

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

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

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 AZA1, AZA2 and AZA3.

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 send you an additional exercise template file. It is not possible to report two sets of data using different methods on the same exercise template. Please use the View Methods function in the QUEST program 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 solutions is 0.7918 g/ml (MeOH). For thwe 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 form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

ROUND	61	Exercise 893
DE-14 PSP Shellfish Toxins		
Test materials	QST093BT, QST094BT, QST095BT, QST096BT	

Objective

This study covers the determination of paralytic shellfish toxins (STX analogues) in four tissue samples QST093BT, QST094BT, QST095BT and QST096BT. The test materials should be analysed and where applicable the individual analogues as well as the sum of the STX analogues (STX, NEO, GTX1, GTX2, GTX3, GTX4, GTX5, GTX6, C1, C2, C3, C4, dc-STX, dc-NEO, dc-GTX1, dc-GTX2, dc-GTX3, dc-GTX4 and 11-OH-STX) in each test material should be reported using data submission form on the Quasimeme Sharepoint Site.

Test Materials and storage

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

Shelfish 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
QST093BT	Shellfish tissue
QST094BT	Shellfish tissue
QST095BT	Shellfish tissue
QST096BT	Shellfish tissue

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 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 (Oshima 1995) of the PSP toxins that appear in “Supplemental Information for PSP toxin CRMs” supplied with National Research Council (NRC) CRMs (see below).

TEF values (Oshima 1995)

Determinand	TEF
GTX-5, B1	0.0644
GTX-6, B2	0.0644
C1	0.0060
C2	0.0963
C3	0.0133
C4	0.0576
dcGTX-2	0.1538
dcGTX-3	0.3766
dcSTX	0.5131
GTX-1	0.9940
GTX-2	0.3592
GTX-3	0.6379
GTX-4	0.7261
11-hydroxy-STX	0.3186
NEO	0.9243
STX	1.0000

Analysis

3.3.1.1 k; 3.4; 3.5.1.5

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 and the TEFs given above. This may include correcting for blanks and for recovery.
The test materials are supplied in 5g aliquots and participants using MBA or ELISA test methods should scale down their extractions to incorporate this smaller aliquot size.
A result 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. 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.
All participants should report the final (total toxicity) result for each test material in µg STX dihydrochloride equivalents/kg. Participants using HPLC methods should also report each PSP analogue identified and give individual analogue concentrations in µmol/kg sample.
To transfer the contents into a preweighed or tared extraction tube, the vial should be fully defrosted, vortex-mixed and the contents poured into the desired container.

Reporting

One result for each determinand in each test material should be reported using the data submission form on your sharepointsite.

It is not possible to report two sets of data using different methods on the same exercise data submission form.

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

For participants using AOAC 2005.06 HPLC method please report toxins that are determined together (dcGTX-2,3; GTX-1,4; GTX-2,3 and C-1,2) in the columns labelled dcGTX-2,3; GTX-1,4; GTX-2,3 and C-1,2. For those participants using chemical methods where epimeric separation is possible, please report individual epimer concentrations in the appropriate columns as well as the sum of the epimers in the dcGTX-2,3; GTX-1,4; GTX-2,3 and C-1,2 columns to facilitate comparison of both methods."

Reporting of Results and Analytical Methods

Enter your analytical data and the method codes using the data submission form on the sharepoint site conform the instructions given in Annex 2.

Units

The units of measurement are given in data submission form. Ensure that the concentration of each determinant is reported in the units given. This may differ from your normal units for reporting; it is essential that all data reported are comparable.

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

Reporting Left Censored Values

If the concentration of a determinant is below the detection limit of your method, you may wish to report the value as less than the detection limit. To do this, you should report your detection limit, either as a negative number or preceded by the "less-than" symbol, <. I.e. to report a value less than a detection limit of 10, report either "-10" or "<10". The QUEST 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 form. 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 using the data submission form. 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.

Return the results to the QUASIMEME Project Office in Wageningen no later than 30 July 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.

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 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 uploading results with data submission form

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

Lower than results will be automatically transferred into – values.

Click on the save button to store your data in 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 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.