

# Quasimeme Laboratory Performance Studies

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## Round 73

1 September 2013 to 1 November 2013

Exercise Protocol AQ1 1

Version 1

**Introduction      Round 73 (AQ11)**

Thank you for participating in the 2013 QUASIMEME Laboratory Performance studies for AQ11.

The AQ11 test materials for the exercises in Round73 that you have ordered will be sent to you by courier in the week of 2 September 2013. Please check that the contents of your package are correct and that all test materials are intact. If any test materials have been damaged in transit or if the wrong samples have been 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.

All data for this study must be uploaded to your Quasimeme SharePoint Site, using the data submission forms, no later than 1 November 2013

All other information should be sent to: QUASIMEME Project Office

QUASIMEME Project Office Wageningen UR Alterra CWK P.O. Box 47 6700 AA Wageningen The Netherlands	Website: <a href="http://www.Quasimeme.org">http://www.Quasimeme.org</a> Tel.: +31 (0) 317 48 65 46 Fax: +31 (0) 317 41 90 00 E-mail: <a href="mailto:Quasimeme@wur.nl">Quasimeme@wur.nl</a>
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<b>ROUND</b>	<b>73</b>	<b>Exercise 1027</b>
<b>AQ-11 Chlorophyll-a in Seawater</b>		
<b>Test materials</b>	<b>QCH062SW, QCH063SW, QCH064SW, QCH065SW and QCH066SW</b>	

### Objective

This study covers the determination of chlorophyll a, b, c and pheopigments in filtered seawater residue test materials, in a standard solution and in an extract prepared from filtered samples.

### Test Materials and storage

The test materials for the analysis of chlorophyll a, b, c and pheopigments were prepared at Alterra, Wageningen the Netherlands. Test materials were prepared from cultures of Isochrysis + Chaetocheros + Pyramimonas (QCH066SW) The QCH065SW sample is a blank seawater sample spiked with algae culture Nanochloropsis. The QCH064SW sample is an unspiked seawater sample containing algae. For each test material, the resultant damp filter paper (Whatman GF/F) was wrapped in aluminium foil, inserted into cryovial and immediately 'flash frozen' in liquid nitrogen. The test materials were stored at -80°C until the day of dispatch. The test materials are homogeneous for the purposes of the LP study.

Sample QCH062SW is a standard solution of chlorophyll-a standard (prepared from spinach) dissolved in ethanol. QCH063SW is an extract from Nanochloropsis algae filtered on Whatman GF/F filters. The filters were extracted with acetone and the extract was filtered on 0.20µm filters.

The samples 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
QCH062SW	A standard solution of chlorophyll-a in ethanol
QCH063SW	An extract of nanochloropsis algae in acetone
QCH064SW	Filtered residues from 1 litre of unspiked seawater containing natural algae
QCH065SW	Filtered residues from 1 litre of seawater spiked with cultured nanochloropsis algae
QCH066SW	Filtered residues from 1 litre of seawater spiked with a mix of cultured algae Isochrysis+ Chaetocheros+Pyramimonas

### Precaution

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

### Determinands and concentration ranges

The following 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%	

*N.B. The indicative range for chlorophyll-a in both extracts is 50 – 5000 µg/L.*

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 of the filter samples (QCH064SW, QCH065SW and QCH066SW need to be calculated based on a filter prepared out of a 1 litre sample. The results for both the standard solution as well as the extract should be reported in µg/L as you obtained the sample without any additional calculation.**

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.

## Reporting of Results and Analytical Methods

### Units

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

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

### Reporting Left Censored Values

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

### Method Codes

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

### Return of Data

Upload all analytical data to the QUASIMEME SharePoint site only with the data submission forms. This allows a rapid and accurate transfer of your data and an early report to you. Additional information and comments may be provided as attached files.

Only data submitted using the data submission forms can be included in the assessment. Return the results to the QUASIMEME Project Office in Wageningen no later than end of October 2013. Data arriving after this deadline may not be entered into the database or appear in the report.

If you have further information on additional methods used or specific ways in which we can improve the data transfer, please inform the QUASIMEME Project Office. Your co-operation is appreciated and will help the assessors in the data analysis and in providing appropriate advice in case of any analytical difficulties.

Please observe the following guidelines, to reduce the need for additional checks, replies and enquires:

Data should only be submitted to the QUASIMEME Project Office when all quality checks have been made. If data are submitted beyond the deadline, they might not be included in the report. Data submitted after the issue of the report will not be included in the report, and these data will also not be included as part of the consensus value. Any certificate prepared with data submitted late will include the statement "Data submitted after report issued". No data will be re-entered into the database after the report is issued. No data will be changed in the database UNLESS there is evidence that QUASIMEME or data transfer has caused an error. In such cases QUASIMEME will undertake a quality query to investigate the problem and inform the participant of the outcome of the Query.

The assigned values will be calculated based on the assessment of all data returned, using the Cofino model. The report for each study, including each laboratory's individual assessment and z-scores, will be distributed on completion before the Workshop. 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 : .....

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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](mailto: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