Environmental chemistry Proficiency testing







# Programme 2024

Version 15 December 2023

WEPAL-QUASIMEME Wageningen University | Coc nr. 09215846 P.O. Box 8005 | 6700 EC Wageningen | The Netherlands Bornsesteeg 10 | 6721 NG Bennekom | The Netherlands

Website: <u>https://www.wepal.nl</u> E-mail: <u>wepalquasimeme@wur.nl</u>

# Table of Contents

What is Profic	iency testing?
Why participa	te in Proficiency testing?
What is WEPA	L-QUASIMEME?
Why choose f	or WEPAL-QUASIMEME?6
What to expe	ct from WEPAL-QUASIMEME?6
What are the	terms of participation?
How to partici	pate?
Sample handl	، ing and shipment
-	ssing
	and procedures
	neasurement
•	g Left Censored Values
	Codes
	f Data
	11 and Faisincation of Results
	v and Data Submission to Third Parties
	24
	AL-QUASIMEME 2024
	cy testing
	e materials
ISE	International Soil-analytical Exchange Programme
Freshwater se	diment22
SETOC	Sediment Exchange for Tests on Organic Contaminants
Marine sedime	ent24
MS-1	Trace Metals in Sediment25
MS-2	Chlorinated Organics in Sediment27
MS-3	Polycyclic Aromatic Hydrocarbons in Sediment
MS-6	Organotin in Sediment
MS-7	Brominated Flame Retardants in Sediment
MS-8	Perfluorinated Alkyl Substances (PFAS) in sediment
Seawater	
AQ-1	Nutrients in Seawater
AQ-2	Nutrients in Estuarine and Low Salinity Open Water
AQ-3	Metals in Seawater
AQ-4	Mercury in Seawater41
AQ-5	Halogenated Organics in Seawater42
AQ-6	Volatile Organics in Seawater

WAGENINGEN UNIVERSITY & RESEARCH



AQ-7	Pentachlorophenol in Seawater46
AQ-8	Triazines and Organophosphorus Pesticides in Seawater47
AQ-1	1 Chlorophyll and Phaeopigments in Seawater49
AQ-1	2 Organotin in Seawater50
AQ-1	3 Polycyclic Aromatic Hydrocarbons in Seawater
AQ-1	4 Dissolved Organic Carbon in Seawater53
AQ-1	5 Ocean Acidification
Freshwate	r and wastewater55
FW-1	Metals in wastewater (metalen in afvalwater)57
FW-2	Metals in freshwater (metalen in oppervlaktewater)59
FW-3	Dried residue (onopgeloste bestanddelen)61
FW-4	General parameters in freshwater (algemene parameters in oppervlaktewater)62
FW-5 afval	Charge and general parameters in wastewater (heffing- en algemene parameters in water)
Plants	
IPE	International Plant-analytical Exchange Programme66
Biota	
BT-1	Trace Metals in Biota68
BT-1 BT-2	Chlorinated Organics in Biota
BT-2 BT-4	Polycyclic Aromatic Hydrocarbons in Biota
BT-4 BT-8	Organotin in Biota
ВТ-8 ВТ-9	Brominated Flame Retardants in Biota
-	
BT-1	
Sheimshit	oxins79
BT-7	ASP Shellfish Toxins80
BT-1	
BT-1	
Manure &	compost
MAR	SEP International Manure and Refuse Sample Exchange Programme
Biomass .	
BIME	P Biomass Exchange Programme
	evelopment) exercises
DE-1	3 Passive sampling in Seawater
DE-1	
DE-1	
DE-1	
DE-1	
	materials
	on and Structure WEPAL-QUASIMEME
	of activities96
The QUAS	IMEME Scientific Advisory Board98





# What is Proficiency testing?

Proficiency testing determines the performance of individual laboratories for specific tests or measurements and is used to monitor laboratories' continuing performance. Proficiency testing is also called interlaboratory comparison. As this term implies, proficiency testing compares the measuring results obtained by the participating laboratories. Routine laboratory performance studies provide the basis of external quality assurance for institutes that make regular chemical measurements in the environment.

As a result of participating in a proficiency test it is possible to identify areas of poor performance, which would benefit from a more detailed scrutiny. An improvement study may be initiated through a workshop run at an institute with sound expertise followed by a series of development exercises to provide detailed tuition and information, with a range of test materials tailored to the specific needs of the problem.



# Why participate in Proficiency testing?

Participation in proficiency testing schemes is beneficial for method development and improvement of laboratories. It provides external quality assurance (QA) for national and/or international monitoring programmes, individual or collaborative research and for contract studies.

Participants can use the assessment of the study data to:

- Validate internal laboratory QA
- Develop and improve methods
- Support accreditation
- Support QA of environmental monitoring data
- Provide data for national or international programmes





# What is WEPAL-QUASIMEME?

WEPAL-QUASIMEME is part of the Wageningen University and is an ISO17043 accredited provider for laboratory proficiency testing. WEPAL-QUASIMEME offers worldwide proficiency testing programs in several environmental matrices (see below). WEPAL-QUASIMEME covers proficiency tests related to the terrestrial environment (soil, plants, manure, compost and biomass); aquatic environment (freshwater, wastewater and sediment) and the marine environment (seawater, marine sediment, biota and shellfish toxins). Additional to the programmes, special (development) exercises are offered to develop and improve methodology, that may result in a new proficiency test scheme on a regular base. Also, samples from our proficiency test schemes are offered to laboratories as reference material. These can be used for method development or as reference samples for internal quality control.







### Why choose for WEPAL-QUASIMEME?



WEPAL-QUASIMEME is part of Wageningen University and has over 65(!) years of experience in proficiency testing. WEPAL-QUASIMEME is accredited for the organisation of Interlaboratory Studies by the Dutch Accreditation Council RvA since April 26, 2000 (registration number R002). The RvA is co-signatory to the ILAC and IAF Multilateral Agreements. The accreditation is based on the ISO 17043 requirements. The scope can be found at: https://www.rva.nl/.

- Participation to our Proficiency test programmes is open to any laboratory.
- All materials are homogeneous and stable for the objective of the test.
- Most proficiency test programmes that WEPAL-QUASIMEME offer have two to four rounds per annum with a minimum of two test materials containing the analytes at different concentrations.
- At competitive fees, WEPAL-QUASIMEME cover a wide range of sample types/matrices, e.g. soil, sediments (freshwater and marine), aquatic (seawater, freshwater and wastewater), plants, biota, shellfish toxins, manure, compost, and biomass and has a vast number of <u>determinands</u> to choose from ranging from elements, chemical pollutants, toxins, and nutritional values to microplastics and human and animal pathogens.
- If you register as a permanent member in WEPAL-QUASIMEME programmes, you are entitled to discounts for multiple orders e.g. proficiency testing programmes, available reference materials and other services.
- Reporting of the analytical data is done directly in our database, including data check.
- Determinands can be added to the program on request.
- Your reports are published within four weeks after the submission deadline and are strictly confidential.
- Track & trace information of packages is provided.
- Regular questionnaires to investigate the needs of laboratories and to improve the quality of the services provided by WEPAL-QUASIMEME.
- The output from the marine studies is reviewed annually by a Scientific Advisory Board, which is comprised of experts in each of the main areas of the Marine Laboratory Performance studies.

### What to expect from WEPAL-QUASIMEME?

- Natural test materials of relevant matrices with determinands at realistic concentrations.
- Test materials with full evidence of homogeneity and stability for the duration of the test.
- Clear protocols how to handle the test materials.
- Full performance assessment based on robust statistics, performance summary sheets and exercise reports.
- Confidential information on own laboratory performance and anonymised results from all participants.
- Method information about the methods used by participants.
- Workshops for discussion and improvement in methodology.
- A Helpdesk, which is supported by experts in the field.
- News, workshops and conferences.





# What are the terms of participation?

- The WEPAL-QUASIMEME proficiency tests are available to any organisation for the purposes of providing support to laboratories' QA programme.
- Each test material will be provided in a suitable container, correctly labelled with the hazard warning and international codes for transport. It will be homogeneous and stable for the duration of the study period and sufficient for the determination. Additional test material can be obtained.
- Test materials for unstable determinands will be freshly prepared, shipped by courier with sameday delivery and are provided with a pre-determined date of analysis.
- Unless replicate data are requested, only one set of data will be assessed from each participant for each test material.
- Sharing test materials between participants is not recommended due to the high risk of contamination or handling effects on the sample. Each participant should request a separate set of test materials.
- It is the participant's responsibility to notify WEPAL-QUASIMEME of any problems with packages, for example if the test material received is damaged or not received at all. Damaged test material will be replaced free of charge, provided the participant provides details of the damage.
- Fees and the annual subscription are due upon receipt of the participant's application and shipment of the first set of test materials.
- No participant's subscription fee will be refunded except when a study is cancelled and the test material is not issued. Test material once issued will not be accepted for return and full payment for the material is due.
- A proficiency test round runs for approximately three months (January-March, April-June, July-September and October-December/January). Shorter times are applicable for the freshwater & wastewater programme due to shorter shelf life of the test materials. Participants can send in results during the entire period of each round. Data which arrive after the deadline may not be included in the assessment.









Participation in the WEPAL-QUASIMEME Proficiency test studies is open to all institutes and companies world-wide that make chemical measurements in environmental matrices and require external quality assurance. Participation in our proficiency testing programmes can be done via application on our website <a href="https://participants.wepal.nl/participation/index.php">https://participation/index.php</a>, or by clicking on the button below.

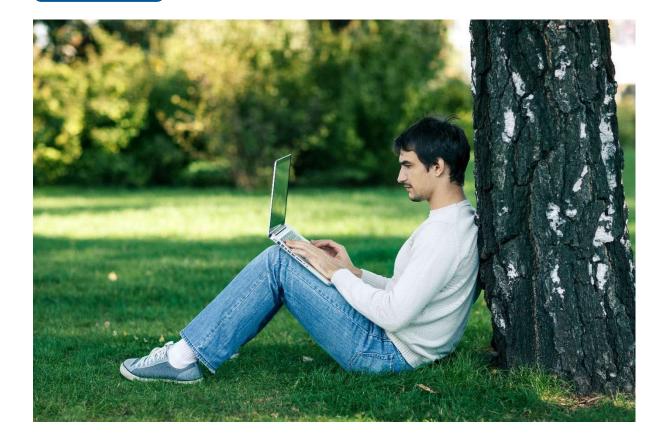
#### Participation form

It is strongly advised to join each round organised for a selected exercise in a specific year. Nevertheless, it is also possible to join a single round or a selection of different rounds.

The minimum number of participants for any study is preferably n=10. With this number, WEPAL-QUASIMEME can ensure sound statistical analysis on the data received by participants. When the number of participants is less then n=10, WEPAL-QUASIMEME will determine on case-by-case basis what to do. Important considerations are costs and the possibilities to establish reliable assigned values and thus meaningful z-scores. When a study is cancelled, participants will be notified, and no costs will be incurred.

Most Laboratory Performance studies are conducted in multiple rounds per year, with a minimum of two test materials per round. Each proficiency test round runs for approximately three months (January-March, April-June, July-September and October-December/January). Participants can send in results during the entire period of each round. The timetable for this year's programme can be found by clicking on the button or see page 14.

#### Timetable







### Sample handling and shipment

WEPAL-QUASIMEME uses the best shipment options for each test material. Test materials from the programs on soil, plant, manure & compost and biomass are normally send by post. All other test materials sent by WEPAL-QUASIMEME will be delivered by courier. Most test materials will be sent in an ambient condition, unless specifically stated (e.g. chlorophyll, shellfish toxin, freshwater and wastewater samples). For stability reasons, these samples will be sent under cooled conditions and will be packed in an EPS box accompanied with cool packs frozen at -80°C.



Most samples are at least stable for the purpose of the test and are often stable for many years. However, some freshwater and wastewater samples have limited shelf life (e.g. dried residue; FW-3) and need to be analysed on a pre-determined date. This is stated in the information sheet of that proficiency test.

All labels come with test material storage requirement advice. Please be aware that test material samples arriving at your laboratory labelled -20°C, have travelled with frozen blocks and should be placed immediately in deep freezers

Please notify WEPAL-QUASIMEME immediately on receipt of test material samples if there are any breakages, leaks or wrongly received orders. New samples will be sent the following Monday after receipt of complaint.

If Customs in your country of delivery require extra information, please inform WEPAL-QUASIMEME as this will ensure quick delivery to your laboratory.









#### Methods and procedures

Participants should use their normal validated methods and procedures to analyse the test materials.

#### Units of measurement

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. Left censored values are included in the statistical evaluation of the data, and in the reports. Please report all measured concentrations for determinands when they exceed your limit of detection.

#### Method Codes

Participants are requested to fill in method information codes (MIC) for each determinand. The MIC's are collated and included in the proficiency test study reports. This allows participants to review the range and similarity of the methodologies used. When the method used by your laboratory can not be chosen by one of the MIC options given in the Data Submission Form, please select others (option Z) and provide us with the details of the method used by your lab.

#### Return of Data

Upload all analytical data to WEPAL-QUASIMEME by using the Data Submission Forms on the Participant Site. This allows a rapid and accurate transfer of the data and quick reporting. Data should only be submitted to the WEPAL-QUASIMEME when all quality checks have been made. No data will be changed in the database UNLESS there is evidence that WEPAL-QUASIMEME or data transfer has caused an error. In such cases WEPAL-QUASIMEME will undertake a quality query to investigate the problem and inform the participant of the outcome of the Query. WEPAL-QUASIMEME reserves the right not to include data submitted after the deadline. 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.

#### Collusion and Falsification of Results

WEPAL-QUASIMEME accepts that most participants operate with professional integrity and that data returned as part of the proficiency test 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. WEPAL-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 proficiency test studies to accreditation bodies and analysts alike.

WEPAL-QUASIMEME reserves the right to withdraw participation of any institute who has submitted data following collusion or falsification. This statement is made as a formal requirement for accreditation for Laboratory Performance Studies under ISO17043.







The proficiency tests studies of WEPAL-QUASIMEME frequently give rise to datasets that have complex distributions including excessive tailing and multiple modes. Consequently, sophisticated statistical methods are required to obtain meaningful assessments. Laboratory assessment is accomplished by attributing a z'-score to each PT result  $x_i$  submitted by participants. The z'score is calculated as

$$z_{i}' = \frac{x_{i} - x_{pt}}{Total \ error} = \frac{x_{i} - x_{pt}}{\sqrt{\left(\sigma_{pt}\right)^{2} + u^{2}(x_{pt})}}$$

Laboratory results with  $|z'| \le 2$  are considered to be satisfactory, results with  $2 < |z'| \le 3$  are questionable and results with |z'| > 3 are labelled as unsatisfactory.

The term  $x_{pt}$  represents the assigned value of the exercise. The consensus value obtained with our very robust NDA model is used as assigned value when datasets have at least 8 numerical observations. The NDA method uses all data submitted, including extreme values and left censored values (LCVs). In the WEPAL-QUASIMEME programmes, less numerical data can be used to provide assigned values when specific criteria are met. This is discussed later.

The term  $u(x_{pt})$  provides the standard uncertainty in the assigned value. This uncertainty is calculated according to ISO 13528 as

$$u(x_{pt}) = 1.25 * \frac{s_{NDA}}{\sqrt{p}}.$$

In this expression,  $S_{NDA}$  is the NDA standard deviation and p the number of laboratory data in the exercise.

The standard deviation for proficiency assessment  $\sigma_{pt}$  underlies the evaluation criteria for the proficiency test. Z' scores measure the distance of laboratory results to the assigned value in units total error. The total error is dominated by the standard deviation for proficiency assessment.

The basic approach is to use the NDA standard deviation as standard deviation for proficiency assessment  $\sigma_{pt}$  when eight or more numerical data are present. A modification is applied depending when at least ten proficiency tests have been carried out in the past years for the measurand and matrix concerned. This modification makes the results from each proficiency test round less dependent on the laboratories participating in the exercise and makes the assessments more stable over the years. Currently, the modification affects primarily the MS, AQ and BT exercises. We have calculated the NDA means  $\bar{x}_{NDA}$  and NDA standard deviations  $s_{NDA}$  for numerical data in all WEPAL-QUASIMEME proficiency tests conducted between 2006 up to and including 2023. The NDA mean  $\bar{x}_{NDA}$  is used as independent variable and the NDA standard deviation  $s_{NDA}$  as dependent variable and fit the so-called uncertainty function  $F_{unc}$ ,

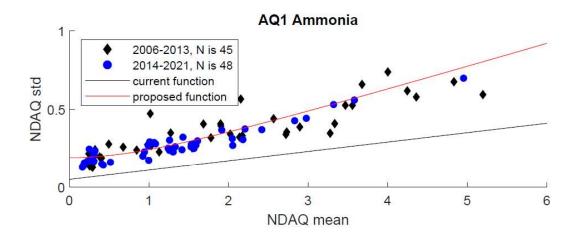
$$F_{unc} = \sqrt{(\alpha^2 + (\beta * \bar{x}_{NDA})^2)}$$

through the data.

An example is given in the following figure:







In this figure, the results of 45 PTs obtained from 2006-2013 are depicted with black triangles, the 48 results acquired in the period 2014-2021 are shown with blue circles. The black line gives the so-called fitness function that WEPAL-QUASIMEME has used to calculate the standard deviation for proficiency assessment  $\sigma_{pt}$  in the period up to 2021. This fitness has the formula  $\sigma_{pt} = a + b * \bar{x}_{NDA}$ , with the intercept a and slope *b* given by the proportional error. These constant and proportional errors are given in the annual catalogue.

The red line is the uncertainty function fitted through the datapoints. From 2024 onwards, we use this function for the WEPAL and QUASIMEME programmes as new fitness function, i.e.

$$\sigma_{pt} = \sqrt{(\alpha^2 + (\beta * \bar{x}_{NDA})^2)}$$

The parameters  $\alpha$  and  $\beta$  are used after rounding off and are given in the annual catalogue.

When data from less than 10 PTs are present, for instance when a proficiency exercise in a certain area has just started, we use the basic approach, i.e. the NDA standard deviation when at least eight numerical data are available as standard deviation for proficiency assessment  $\sigma_{pt}$ . We will fit an uncertainty function and establish the fitness function after data for ten or more rounds have been obtained.

The "proportional error"  $\beta$  (N≥10) respectively the NDA std (N<10) are maximised at 25% of the assigned value. This implies, that  $\beta$  cannot be larger than 0.25. This limit is chosen on the basis of the EU directive 2009/90/EC. This directive gives technical specifications for chemical analysis and monitoring of water status. We decided to apply this limit solely to the proportional error as limiting  $\sigma_{pt}$  would give rise to too harsh criteria at low concentrations. Wo do limit, however, the total error (standard deviation for proficiency assessment, including the uncertainty) to 32,5% of the assigned value.

WEPAL-QUASIMEME has several proficiency tests with a small number of participants so that additional measures have been formulated to enable assessment. It is to be realised, however, that in such cases the assessment only signifies the degree of comparability among the participants. When the number of numerical observations is between 4 and 7, a standard deviation for proficiency assessment  $\sigma_{pt}$  is limited to 12,5% of the assigned value. This applies to all situations (n=4 tot 7), regardless of whether the total error and constant error are given or not.

Moreover, additional quality criteria for all datasets are used. When 4 to 7 numerical data are reported, at least 80% of the z'-scores should be satisfactory. When 8 to 12 data are reported, at least 70% of the z'-





scores should be satisfactory. When more than 12 data are reported at least 60% of the z'-scores should be satisfactory, with a minimum of 9 satisfactory observations. Finally, the relative uncertainty is calculated for every determinand-matrix combination. This relative uncertainty should not exceed 25%, otherwise the uncertainty in the Assigned Value is judged to be too large. No assigned value and no z'-scores are given if these conditions are not met.



### Confidentiality and Data Submission to Third Parties

WEPAL-QUASIMEME operates a fully confidential service to all participants. The results remain the property of each participant and full confidentiality is maintained. No information on individual participants' performance is disclosed to any third party. WEPAL-QUASIMEME will provide each participant with a unique code for the proficiency test studies. WEPAL-QUASIMEME provide ICES with the general reports of the QUASIMEME programmes, in which the data are published under code only. For some Swiss and UK laboratories, participation is made mandatory by the government and results are made available to the designated authorities after permission from the laboratories concerned. Data cannot therefore be traced back to individual laboratories. WEPAL-QUASIMEME will publish the evaluation and overview of the proficiency test studies in peer review journals, maintaining confidentiality. All data, however presented, will be non-attributable. The codes described above will be the only codes used in publications.

The data generated by participants is valuable to the national and/or international organisations that collate and assess environmental data for the chemical determinands analysed in the WEPAL-QUASIMEME proficiency test studies. WEPAL-QUASIMEME encourages all participants to submit their quality assurance (QA) data, including their proficiency test studies results, in the submission of environmental information to the national and/or international marine monitoring programmes. QA data submission to any third party, including submission of proficiency test studies data, is the responsibility of the individual institutes. The assessment files, in text and csv formats, will be provided electronically after the completion of each proficiency test study.



## Timetable 2024



WEPAL-QUASIMEME proficiency tests follows an annual timetable. This timetable allows all participants to incorporate the test materials into their ongoing analytical programme. This is particularly important for those participants who need to undertake their QA analysis alongside their environmental samples in the laboratory or at sea.

Samples for the schemes ISE, IPE, SETOC, MARSEP and BIMEP are sent by post a few weeks before the start date of the period. Samples for the schemes MS, AQ and BT are sent by courier in the first week of the period. Samples for the schemes FW are sent by courier on pre-determined dates. Participants that receive samples by courier will be notified of the exact date of dispatch.

The deadlines for submission of data are fixed. Any data received after the deadline may not be included in the assessment. A confidential individual laboratory report, the full study report and the electronic summary files will be provided after the deadline for the submission of data. These reports and summary files will also be provided for data received after the report is issued, but the individual laboratory report will include the statement, "Data received after the report was issued."

Period	Schemes	Start date	Deadline submission of data	Reports available
Q1	ISE, IPE, SETOC, MARSEP, BIMEP	1 January	31 March	21 April
Q2	ISE, IPE, SETOC, MS, AQ, BT, MARSEP, BIMEP	1 April	30 June	21 July
	FW-1, FW-2	20 March	10 April	1 May
	FW-3	17 April	8 May	29 May
	FW-4	24 April	22 May	12 June
Q3	ISE, IPE, SETOC, MARSEP, BIMEP	1 July	30 September	21 October
Q4	ISE, IPE, SETOC, MARSEP, BIMEP	1 October	31 December	21 January
	MS, AQ, BT	1 October	31 January	21 February
	FW-5	9 October	6 November	27 November

# Proficiency testing scheme 2024

Matrix	Code	Proficiency test	Number		Per	iod	
			of test	Q1	Q2	Q3	Q4
			materials				
Soil	ISE	International Soil-analytical	4	Х	X	Х	Х
		Exchange Programme					
Plant	IPE	International Plant-analytical	4	Х	Х	Х	X
		Exchange					
Freshwater	SETOC	Sediment Exchange for Tests on	4	Х	X	Х	x
sediment		Organic Contaminants					
Manure &	MARSEP	International Manure and Refuse	4	Х	X	Х	x
Compost		Sample Exchange Programme					
Biomass	BIMEP	Biomass Exchange Programme	4		Х		Х
Marine	MS-1	Metals	2		Х		X
sediment	MS-2	Chlorinated organics	2		Х		X
	MS-3	PAHs	2		Х		X
	MS-6	Organotin	2		Х		Х
	MS-7	Brominated Flame Retardants	2		х		Х
	MS-8	PFAS	2		Х		Х





Matrix	Code	Proficiency test	Number		Per	riod	
			of test	Q1	Q2	Q3	Q4
			materials				
Seawater	AQ-1	Nutrients (high salinity)	3		Х		Х
	AQ-2	Nutrients (estuarine and low	4		Х		X
		salinity)					
	AQ-3	Metals (high and low salinity)	4		Х		Х
	AQ-4	Mercury (high and low salinity)	4		Х		Х
	AQ-5	Halogenated organics (high and	3		Х		
		low salinity)					
	AQ-6	Volatile organics in estuarine and	2		X		
		freshwater (high salinity)					
	AQ-7	Pentachlorophenol (high salinity)	3		Х		
	AQ-8	Pesticides (high and low salinity)	3		Х		
	AQ-11	Chlorophyll and Phaeopigments	2		Х		X
		(estuarine and freshwater) (filter)					
	AQ-12	Organotin (high salinity)	2		Х		
	AQ-13	PAHs (high and low salinity)	3		Х		
	AQ-14	DOC (high salinity and estuarine)	4		Х		X
	AQ-15	Ocean acidification (total alkalinity	3		X		X
		and DIC) (high and low salinity)					
Biota	BT-1	Metals	2		Х		Х
	BT-2	Chlorinated organics	2		Х		X
	BT-4	PAHs	2		Х		Х
	BT-8	Organotin	2		Х		Х
	BT-9	Brominated Flame Retardants	2		Х		X
	BT-10	PFAS	2		Х		Х
Shellfish	BT-7	ASP shellfish toxins	3		Х		X
toxins	BT-11	Lipophilic shellfish toxins	3		Х		X
	BT-12	PSP shellfish toxins	3		Х		X
Special	DE-13	Passive samplers	2				X
exercises	DE-16	Tetrodotoxin (TTX)	2				Х
	DE-17	Microplastics	3		Х		
	DE-18	PFAS in (sea)water	3				X
	DE-19	Pharmaceuticals in (sea)water	3				X
Freshwater &	FW-1	Metals in wastewater	3		X		
wastewater		(metalen in afvalwater)					
	FW-2	Metals in freshwater	3		X		
(Oppervlakte- (metalen in oppervlaktewater)							
water & FW-3 Dried residue		5		X			
afvalwater)		(onopgeloste bestanddelen)					
	FW-4	General parameters in freshwater	3		X		
		(algemene parameters in					
		oppervlaktewater)					
	FW-5	Charge and general parameters in	3				Х
		wastewater					
		(heffing- en algemene parameters					
		in afvalwater)					







# Soil

#### Proficiency tests in Soil

ISE - International Soil-analytical Exchange Programme p19

#### General information

- The sample types/matrices are chosen to represent the materials which you normally analyse. There is no spiking or use of artificial samples.
- The soil samples are dried at max. 40°C, milled and sieved to <0.5 mm.
- The homogeneity of each batch of samples is tested on a selection of parameters.
- The dried soil samples are stable over a number of years when stored at room temperature.
- You analyse the test materials according to your normal validated methods and procedures for those elements and parameters you are interested in.
- Your results are processed at WEPAL-QUASIMEME and published every three months under confidential code names.
- The reports contain all data, statistical evaluation including Z-score plots and method information about the method used.
- The reports are available within three weeks after the submission deadline.
- New determinands can be added on request.







### ISE International Soil-analytical Exchange Programme

Year: 2024	Participants: 250 laboratories expected
Number of rounds: 4 per year	Start exercise: 1 January, 1 April, 1 July, 1 October
Number of materials: 4 per round	Sample size: 100 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of chemical analysis in soils. Participation is open to all laboratories.

#### Determinands

Concentration ranges and constant and proportional errors are available in the determinand list on the participants website.

Determinand group	Determinand*
Real totals	Ag; Al; As; B; Ba; Be; Bi; Br; C-elementary; Ca; Cd; Ce; Co; Cr; Cs; Cu; F; Fe; Ga; Ge; Hg; I; K; La; Li; Mg; Mn; Mo; N- elementary; Na; Nb; Nd; Ni; P; Pb; Pd; Pt; Rb; Rh; S; Sb; Sc; Se; Si; Sn; Sr; Te; Th; Ti; Tl; U; V; W; Y; Zn; Zr;
Acid extractable (So-called totals)	Ag; Al; As; B; Ba; Be; Bi; Br; Ca; Cd; Ce; Co; Cr; Cu; F; Fe; Ga; Hg; I; K; La; Li; Mg; Mn; Mo; N; Na; Nb; Nd; Ni; P; Pb; Pt; Rb; S; Sb; Sc; Se; Si; Sn; Sr; Te; Th; Ti; Tl; U; V; Y; Zn; Zr;
Aqua Regia (ISO 11466)	Ag; Al; As; B; Ba; Be; Bi; Br; Ca; Cd; Ce; Co; Cr; Cu; F; Fe; Ga; Hg; I; K; La; Li; Mg; Mn; Mo; Na; Nd; Ni; P; Pb; Pt; Rb; S; Sb; Sc; Se; Si; Sn; Sr; Te; Th; Ti; Tl; U; V; Y; Zn; Zr;
Extraction with boiling 2M HNO3	Cd; Co; Cr; Cu; Hg; Mo; Ni; Pb; Tl; Zn;
Extraction with 0.1M NaNO3	Cd; Cu; Ni; Pb; Zn;
Extraction with 0.01M CaCl2 1:10	Al; B; Cd; CN; Co; Cr; Cu; Fe; K; Mg; Mn; N-NH4 (as N); N-NO3 (as N); N total soluble; Na; Ni; P; Pb; SO4; Zn;
Soil characteristics	C-org others (W&B a.o.); TIC=Tot. Inorg C(as CaCO3); TOC=Total Org. C; TC=Total C (org.+inorg.); Org.matter (L.O.I.); Active Lime (as CaCO3); EC-SC (ISO 11265); pH- CaCl2; pH-H2O; pH-KCl; Fraction < 2 $\mu$ m; Fraction < 16 $\mu$ m; Fraction < 63 $\mu$ m; Fraction > 63 $\mu$ m;
Other determinations	B-Hot water; CN-Free; CN-Total; delta 13C; delta 15N; K-HCl (as K); Mg-NaCl (as Mg); <b>Moisture-content</b> ;
Fluoride (Swiss standard procedure)	F-Total;
Digestion with conc. HNO3 + conc. HCl + H2O2 (UNEP-UN/EC 91075A)	Al; As; B; Ba; Be; Ca; Cd; Co; Cr; Cu; Fe; Hg; K; Li; Mg; Mn; Mo; Na; Ni; Pb; S; Sb; Se; Si; Sn; Sr; Tl; V; Zn;
Pot. CEC using 1M NH4-acetate at pH=7	CEC; Al; Ca; K; Mg; Na;
Pot. CEC using 1M or 0.1M BaCl2- TEA at pH=8.1 (ISO 13536 OR BZE)	CEC; AI; Ca; K; Mg; Na;
Pot. CEC using 1M NH4Cl (BZE)	CEC; Al; Ca; Fe; H; K; Mg; Mn; Na;
Act. CEC using 0.01M BaCl2 (ISO 11260)	CEC; Al; Ca; Fe; H; K; Mg; Mn; Na;
Act. CEC using 0.1M BaCl2 (UNEP-UN/EC 91065A)	CEC; AI; Ca; Fe; H; K; Mg; Mn; Na;





Determinand group	Determinand*
Act. CEC using cobaltihexamine (AFNOR NFX 31 130)	CEC; AI; Ca; Fe; H; K; Mg; Mn; Na;
Mehlich-3	Al; As; B; Ca; Cd; Cr; Cu; Fe; K; Mg; Mn; Na; P; Pb; Zn;
Extraction with Ca-lactate (VDLUFA, Germany)	К; Р;
Extraction with double lactate (VDLUFA, Germany)	К; Р;
Water soluble 1:10 (w/v) (EN- 12457-4)	Br; Cl; F; N-NO3 (as N);
Extraction with 0.01M CaCl2- 0.005M DTPA 1:10 (w/v)	Cu; Fe; Mn; Zn;
Extraction with 1M KCl 1:10 (w/v)	N-NH4 (as N); N-NO3 (as N);
Phosphorus and related analysis	P-Ox; Al-Ox; Fe-Ox; P-AL (as P); P-w (as P); P-Bray (as P); P- Olsen (as P);
Extraction with 1M HCI (Polish standard)	B; Cu; Fe; Mn; Zn;
Water soluble 1:10 (w/v) (Neth standard VPR C85-06)	Br; Cl; F; SO4;
UK Soil Methods	K-NH4NO3 (1/5); Mg-NH4NO3 (1/5); P-NaHCO3 (1/20); pH-H2O (2/5);
Extraction with dilute nitric acid (0.43 Mol/I) ISO 17586	Ag; Al; As; B; Ba; Be; Br; Ca; Cd; Co; Cr; Cu; Fe; Hg; K; Mg; Mn; Mo; Na; Ni; P; Pb; S; Sb; Se; Sn; Sr; Te; Ti; Tl; V; Zn;
*Determinands not in hold are not i	in the scope of accreditation

\*Determinands not in bold are not in the scope of accreditation







## Freshwater sediment

#### Proficiency tests in Freshwater sediment

SETOC - Sediment Exchange for Tests on Organic Contaminants p23

#### **General Information**

- The sample types/matrices are chosen to represent the materials which you normally analyse. There is no spiking or use of artificial samples.
- The samples are obtained from riverbeds, lake bottoms and harbours.
- Sediments are dried and milled and sieved to <0.5 mm.
- The homogeneity of each batch of samples is tested on a selection of parameters.
- The dried sediment samples are stable over a number of years when stored at room temperature.
- You analyse the samples according to your own procedures and for those elements and parameters you are interested in.
- Your results are processed at WEPAL-QUASIMEME and published every three months under confidential code names.
- The reports contain all data, statistical evaluation including Z-score plots and method information about the method used.
- Your reports are available within three weeks after the submission deadline
- New determinands can be added on request.







## SETOC Sediment Exchange for Tests on Organic Contaminants

Year: 2024	Participants: 50 laboratories expected
Number of rounds: 4 per year	Start exercise: 1 January, 1 April, 1 July, 1 October
Number of materials: 4 per round	Sample size: 100 g

Participation form	Timetable	PT Scheme	Costs
--------------------	-----------	-----------	-------

This study covers the determination of chemical analysis in freshwater sediments.

#### Determinands and Analysis

Concentration ranges and constant and proportional errors are available in the determinand list on the participants website.

<sup>#</sup> For dioxins and furans, a special round i	s held once yearly in the period October-December.
--	--

Determinand group	Determinand*
Polycyclic aromatic hydrocarbons	acenaphthene; acenaphthylene; anthracene; benz(a)anthracene; benzo(a)pyrene; benzo(b)fluoranthene; benzo(ghi)perylene; benzo(k)fluoranthene; chrysene; dibenz(ah)anthracene; fluoranthene; fluorene; indeno(1,2,3-cd)pyrene; naphthalene; phenanthrene; pyrene; EPA ΣPAH(16);
Polychlorobiphenyls	<b>PCB 028</b> ; PCB 031; <b>PCB 052</b> ; PCB 077; PCB 081; <b>PCB 101</b> ; PCB 105; PCB 114; <b>PCB 118</b> ; PCB 123; PCB 126; PCB 128; <b>PCB 138</b> ; PCB 149; <b>PCB 153</b> ; PCB 156; PCB 157; PCB 167; PCB 169; <b>PCB 180</b> ; PCB 189; ΣPCB(7);
Organochlorine pesticides	1,2,3 trichlorobenzene; 1,2,4 trichlorobenzene; 1,3,5 trichlorobenzene; Sum trichlorobenzenes; 1,2,3,4 tetrachlorobenzene; 1,2,3,5 tetrachlorobenzene; 1,2,4,5 tetrachlorobenzene; Sum tetrachlorobenzenes; aldrin; alpha- endosulfan; alpha-HCH; beta-endosulfan; beta-HCH; chlordane; cis- chlordane; delta-HCH; dieldrin; endosulfan; endosulfan sulfate; endrin; gamma-HCH; heptachlor; heptachlor epoxide; <b>hexachlorobenzene</b> ; hexachlorobutadiene; isodrin; o,p`-DDD; o,p`-DDE; o,p`-DDT; p,p`-DDD; <b>p,p`-DDE</b> ; p,p`-DDT; pentachlorobenzene; pentachlorophenol; telodrin; toxaphene; trans-chlordane;
Other parameters	AOX; CN-Free; <b>CN-Total</b> ; EOX; Inorganic carbon; <b>Organic carbon</b> ; <b>Mineral oil, GC</b> ; Mineral oil, IR; Particles < 2 $\mu$ m; Particles < 63 $\mu$ m; Particles > 63 $\mu$ m;
Metals	As; Ba; Cd; Co; Cr; Cu; Hg; Mo; Ni; Pb; Zn;
Dibenzo-P Dioxin <sup>#</sup>	1,2,3,4,6,7,8 CI7DD; 1,2,3,4,7,8 CI6DD; 1,2,3,6,7,8 CI6DD; 1,2,3,7,8 CI5DD; 1,2,3,7,8,9 CI6DD; 2,3,7,8 CI4DD; CI8DD;
Dibenzofuran#	1,2,3,4,6,7,8 Cl7DF; 1,2,3,4,7,8 Cl6DF; 1,2,3,4,7,8,9 Cl7DF; 1,2,3,6,7,8 Cl6DF; 1,2,3,7,8 Cl5DF; 1,2,3,7,8,9 Cl6DF; 2,3,4,6,7,8 Cl6DF; 2,3,4,7,8 Cl5DF; 2,3,7,8 Cl4DF; Cl8DF;
Brominated Flame Retardants	BDE 028; BDE 047; BDE 066; BDE 085; BDE 099; BDE 100; BDE 153; BDE 154; BDE 183; BDE 209;
Experimental	DEHP; Tributyl Tin (TBT);

\*Determinands not in bold are not in the scope of accreditation







# Marine sediment

#### Proficiency tests in Marine sediment



#### **General Information**

- Sediment samples cover a wide range of naturally contaminated sandy and silty sediments from open sea, estuaries, rivers and harbour locations.
- Sediments are dried and milled and sieved to < 0.5 mm.
- Sediments are packed in glass bottles (metals: 20 gram; others: 50 gram).
- Dried sediment samples are stable over a number of years when stored at room temperature (except organotin: -20°C).
- You use your normal validated methods and procedures to analyse the test materials.
- New determinands can be added on request.
- Reports contain all data reported under strict confidentiality, including Z-score plots and method information of methods used.







#### MS-1 Trace Metals in Sediment

Year: 2024	Participants: 45 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 20 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of metals, total organic carbon (TOC) and carbonate in marine sediments.

#### Test Materials

The test materials cover a range of natural sediments from contaminated waters from the North Sea and/or Mediterranean. Each batch of material is prepared in bulk. The level of within and between sample homogeneity for the sediment is determined. All materials show to be homogeneous and stable for the purpose of the test.

#### Determinands and concentration ranges

The metals to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

RT = Real Total destructions e.g. HF-destruction, röntgen-diffraction and neutron activation. AE= Acid extractable and all other methods.

In addition to the determinands given in this table, several additional metals are added into the dataset form on the Participant's sites. There you will find amongst others Na, S, K, Ti, Ga, Se, Sn, Cs, Ce, Ta, Tl. In case enough participants report results these additional metals will be added permanently to the proficiency test.

Determinand*	Unit	Concentration range	Error	
		Sediment	Const	Prop
Aluminium-AE	%	0.5-10	0.01	25.0
Aluminium-RT	%	1-10	0.01	12.5
Arsenic-AE	mg/kg	2-50	0.5	12.5
Arsenic-RT	mg/kg	2-50	0.5	12.5
Barium-AE	mg/kg	50-1000	20	17.5
Barium-RT	mg/kg	50-1000	15	6.0
Cadmium-AE	µg/kg	10-5000	25	7.5
Cadmium-RT	µg/kg	10-5000	35	10.0
Calcium-AE	g/kg	5-100		
Calcium-RT	g/kg	5-100		
Chromium-AE	mg/kg	10-1000	5	17.5





Determinand*	Unit	Concentration range	E	rror
		Sediment	Const	Prop
Chromium-RT	mg/kg	10-1000	7.5	10.0
Cobalt-AE	mg/kg	1-50	0.3	10.0%
Cobalt-RT	mg/kg	1-50	0.4	7.5%
Copper-AE	mg/kg	1-500	0.75	7.5%
Copper-RT	mg/kg	1-500	0.5	10.0%
Iron-AE	%	0.5-10	0.10	7.5%
Iron-RT	%	0.5-10	0.10	7.5%
Lead-AE	mg/kg	5-500	2.5	10.0%
Lead-RT	mg/kg	5-500	2.5	10.0%
Lithium-AE	mg/kg	10-100	2	12.5%
Lithium-RT	mg/kg	10-100	2	12.5%
Magnesium-AE	mg/kg	2000-20000	0.1	10.0%
Magnesium-RT	mg/kg	2000-20000		
Manganese-AE	mg/kg	100-2000	10	7.5%
Manganese-RT	mg/kg	100-2000	10	7.5%
Mercury-AE	μg/kg	10-2500	12.5	12.5%
Mercury-RT	μg/kg	10-2500	12.5	10.0%
Molybdene-AE	mg/kg	2-1000		
Molybdene-RT	mg/kg	2-1000		
Nickel-AE	mg/kg	2-100	1	12.5%
Nickel-RT	mg/kg	2-100	1	10.0%
Phosporus-AE	mg/kg	100-3500	10	10.0%
Phosphorus-RT	mg/kg	100-3500	10	10.0 /0
Rubidium-AE	μg/kg	10-50		
Rubidium-RT	μg/kg	10-100		
Scandium-AE	mg/kg	1-20		
Scandium-RT	mg/kg	1-20		
Strontium-AE	mg/kg	50-500	10	6.0%
Strontium-RT	mg/kg	50-500	1	12.5%
Thallium-AE	µg/kg	50-1000		
Thallium-RT	µg/kg	100-1500		
Uranium-AE	mg/kg	0.2-5		
Uranium-RT	mg/kg	0.5-5		
Vanadium-AE	mg/kg	5 -500	1.5	20.0%
Vanadium-RT	mg/kg	5 -500	1	10.0%
Zinc-AE	mg/kg	20-1500	2.5	7.5%
Zinc-RT	mg/kg	20-1500	5	7.5%
тос	%	0.2-10	0.1	10.0%
Inorganic-carbonate	%	0.05-10	0.25	15.0%
Loss on ignition	%	0.02-10		

\*Determinands which are not in bold are not in the scope of accreditation.





#### MS-2 Chlorinated Organics in Sediment

Year: 2024	Participants: 25 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of poly chlorinated biphenyls (PCBs), organochlorine pesticides (OCPs) and total organic carbon (TOC) in marine sediment.

#### **Test Materials**

The test materials cover a range of natural sediments from contaminated waters from the North Sea and/or Mediterranean. Each batch of material is prepared in bulk. The level of within and between sample homogeneity for the sediment is determined. All materials show to be homogeneous and stable for the purpose of the test.

#### Determinands and Concentration Ranges

The organochlorines to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Unit Concentration Range	E	rror
		Sediment	Const	Prop
PCB18	µg/kg	0.1-10		
PCB28	µg/kg	0.1-100	0.05	20.0%
PCB31	µg/kg	0.1-100	0.05	22.5%
PCB44	µg/kg	0.1-100		
PCB47	µg/kg	0.1 - 50		
PCB49	µg/kg	0.02 - 100		
PCB52	µg/kg	0.1-500	0.15	17.5%
PCB66	µg/kg	0.02 - 100		
PCB99	µg/kg			
PCB101	µg/kg	0.2-300	0.15	20.0%
PCB105	µg/kg	0.1-50	0.1	15.0%
PCB107	µg/kg			
PCB108	µg/kg			
PCB109	µg/kg			
PCB110	µg/kg	0.1-100		
PCB111	µg/kg			
PCB112	µg/kg			
PCB113	µg/kg			





Determinand*	Unit	Concentration Range	E	rror
		Sediment	Const	Prop
PCB114	µg/kg			
PCB118	µg/kg	0.1-200	0.15	15.0%
PCB128	µg/kg	0.05-5	0.05	20.0%
PCB138+PCB163	µg/kg	0.2-50	0.3	15.0%
PCB138	µg/kg	0.2-50	0.25	20.0%
PCB141	µg/kg	0.05-10		
PCB149	µg/kg	0.05-100	0.25	20.0%
PCB151	µg/kg	0.1-20		
PCB153	µg/kg	0.2-100	0.25	17.5%
PCB156	µg/kg	0.05-5	0.05	22.5%
PCB158	µg/kg	0.1-5		
PCB170	µg/kg	0.05-10	0.1	25.0%
PCB180	µg/kg	0.1-50	0.15	25.0%
PCB183	µg/kg	0.05-5		
PCB187	µg/kg			
PCB188	µg/kg	0.05-10		
PCB194	µg/kg	0.02-2		
PCB203	µg/kg			
PCB209	µg/kg			
α-HCH	µg/kg	0.02-1	0.03	25.0%
β-ΗϹΗ	µg/kg	0.02-2	0.02	25.0%
γ-ΗCΗ	µg/kg	0.02-2	0.03	25.0%
δ-ΗCΗ	µg/kg	0.02-2		
НСВ	µg/kg	0.05-250	0.04	25.0%
HCBD	µg/kg	0.02-10		
Dieldrin	µg/kg	0.1-10	0.06	25.0%
pp'-DDD	µg/kg	0.1-25	0.01	25.0%
pp'-DDE	µg/kg	0.1-20	0.01	25.0%
op'-DDT	µg/kg	0.02-250	0.2	25.0%
pp'-DDT	µg/kg	0.05-10	0.1	25.0%
Transnonachlor	µg/kg	0.01-2		
Heptachlor	µg/kg			
cis-Heptachlor epoxide	µg/kg			
Emamectin	µg/kg			
Teflubenzuron	µg/kg		1	
тос	%	0.2-10	0.1	10.0%
PN	%			

\*Determinands which are not in bold are not in the scope of accreditation.





#### MS-3 Polycyclic Aromatic Hydrocarbons in Sediment

Year: 2024	Participants: 30 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 50 g

Darticipation form	Timetable	PT Scheme	Costs
Participation form	nnetable	PT Scheme	COSIS

This study covers the determination of Polycyclic Aromatic Hydrocarbons (PAHs) and total organic carbon (TOC) in marine sediment.

#### Test Materials

The test materials cover a range of natural sediments from contaminated waters from the North Sea and/or Mediterranean. Each batch of material is prepared in bulk. The level of within and between sample homogeneity for the sediment is determined. All materials show to be homogeneous and stable for the purpose of the test.

#### Determinands and Concentration Ranges

The PAHs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range	E	ror
		Sediment	Const	Prop
Acenaphthene	µg/kg	0.5-2000	1	25.0%
Acenaphthylene	µg/kg	0.5-1000	0.2	25.0%
Anthracene	µg/kg	1-500	1.5	22.5%
Benzo[a]anthracene	µg/kg	2-1500	3	20.0%
Benzo[a]fluorene	µg/kg	2-1000		
Benzo[a]pyrene	µg/kg	2-1500	2	20.0%
Benzo[b]fluoranthene	µg/kg	5-1500	2.5	25.0%
Benzo[k]fluoranthene	µg/kg	2-1000	0.75	22.5%
Benzo[e]pyrene	µg/kg	2-1500	1	20.0%
Benzo[g,h,i]perylene	µg/kg	2-1500	0.75	22.5%
Chrysene	µg/kg	2-1500	2	22.5%
Chrysene+Triphenylene	µg/kg	2-3000	1	20.0%
Triphenylene	µg/kg	1-3000		
Dibenz[a,h]anthracene	µg/kg	0.5-500	0.25	25.0%
Dibenzo[a,i]pyrene	µg/kg			
Dibenzothiophene	µg/kg	0.5-200	1	25.0%
Fluoranthene	µg/kg	5-4000	1	20.0%
Fluorene	µg/kg	0.5-1000	1.5	25.0%





Determinand*	Unit	Unit Concentration Range		Error		
		Sediment	Const	Prop		
Indeno[1,2,3-cd]pyrene	µg/kg	2-1500	1	25.0%		
Naphthalene	µg/kg	2-4000	3	25.0%		
1-methylnaphthalene	µg/kg					
2-methylnaphthalene	µg/kg		5	25.0%		
1-methylanthracene	µg/kg					
2-methylanthracene	µg/kg					
Perylene	µg/kg	2-500	1	25.0%		
Phenanthrene	µg/kg	5-3000	3	20.0%		
1-methylphenanthrene	µg/kg					
2-Methylphenanthrene	µg/kg	1-1000	2	20.0%		
3,6-Dimethylphenanthrene	µg/kg	0.5-500	0.3	25.0%		
Pyrene	µg/kg	2-4000	2	17.5%		
1-Methylpyrene	µg/kg	0.5-500		1		
1,2-benzodiphenylene sulfide	µg/kg					
тос	%	0.2-10	0.1	10.0%		
C1-phenanthrenes/anthracenes	µg/kg		5	25.0%		
C2-phenanthrenes/anthracenes	µg/kg		5	25.0%		
C3-phenanthrenes/anthracenes	µg/kg					
C1-pyrenes/fluoranthenes	µg/kg					
C2-pyrenes/fluoranthenes	µg/kg					
C1-chrysenes	µg/kg					
C2-chrysenes	µg/kg					
C1-benzofluoranthenes	µg/kg		1	1		
C1-dibenzothiophenes	µg/kg		1	1		
C2-dibenzothiophenes	µg/kg			Ì		
C3-dibenzothiophenes	µg/kg			Ì		
C1-naphtalenes	µg/kg		1	1		
C2-naphtalenes	µg/kg		20	25.0%		
C3-naphtalenes	µg/kg		1	1		
C1-phenanthrenes	µg/kg					
Benzofluoranthenes (b+j)	µg/kg			1		
Benzofluoranthenes (a+b+j+k)	µg/kg		1	1		
Total petroleum hydrocarbons	mg/kg					
PN	%					

\*Determinands which are not in bold are not in the scope of accreditation.





#### MS-6 Organotin in Sediment

_	
Year: 2024	Participants: 25 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 50 g



This study covers the determination of organotin compounds in marine sediment.

#### **Test Materials**

The test materials cover a range of natural sediments from contaminated waters from the North Sea and/or Mediterranean. Each batch of material is prepared in bulk. The level of within and between sample homogeneity for the sediment is determined. All materials show to be homogeneous and stable for the purpose of the test.

#### Determinands and Concentration Ranges

The organotin compounds to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand	Unit	Concentration Range Error		rror
		Sediment	Const	Prop
Tributyltin (TBT)	µg Sn/kg	1-5000	0.3	25.0%
Dibutyltin (DBT)	µg Sn/kg	1-5000	0.4	25.0%
Monobutyltin (MBT)	µg Sn/kg	1-5000	0.5	25.0%
Triphenyltin (TPhT)	µg Sn/kg	0.1-200	0.2	25.0%
Diphenyltin (DPhT)	µg Sn/kg	0.1-200		
Monophenyltin (MPhT)	µg Sn/kg	0.1-200		

\*This exercise is not in the scope of accreditation.





#### MS-7 Brominated Flame Retardants in Sediment

Year: 2024	Participants: 15 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of brominated flame retardants (BFRs) in sediment.

#### **Test Materials**

The test materials cover a range of natural unspiked sediments from contaminated waters from the North Sea and/or Mediterranean. Sediments are dried and sieved to <0.5 mm before sub-sampling into glass jars for distribution. Sediment test materials have been shown to be stable over a number of years when stored at room temperature.

#### Determinands and concentration ranges

The BFRs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.

- The constant and proportional error that will be used for assessment of the results.

Unit	Concentration range Error		rror
	Sediment	Const	Prop
µg/kg	0.01-2	0.01	25.0%
µg/kg	0.1-20	0.04	22.5%
µg/kg	0.01-10	0.02	20.0%
µg/kg	0.01-10		
µg/kg	0.1-50	0.02	25.0%
µg/kg	0.01-10	0.01	25.0%
µg/kg			
µg/kg	0.02-5	0.02	25.0%
µg/kg	0.01-5	0.03	25.0%
µg/kg	0.02-2	0.02-2 0.03 25.04	
µg/kg	2-2000	1	25.0%
µg/kg			
µg/kg	0.01-20		
µg/kg	50-1000		
	µg/kg           µg/kg	Sediment           μg/kg         0.01-2           μg/kg         0.01-20           μg/kg         0.01-10           μg/kg         0.01-10           μg/kg         0.01-10           μg/kg         0.01-10           μg/kg         0.01-50           μg/kg         0.01-5           μg/kg         0.02-5           μg/kg         0.02-2           μg/kg         0.02-2	Sediment         Const           μg/kg         0.01-2         0.01           μg/kg         0.1-20         0.04           μg/kg         0.01-10         0.02           μg/kg         0.01-10         0.02           μg/kg         0.01-10         0.02           μg/kg         0.01-10         0.02           μg/kg         0.01-50         0.02           μg/kg         0.01-50         0.01           μg/kg         0.02-5         0.02           μg/kg         0.02-5         0.03           μg/kg         0.02-2         0.03           μg/kg         0.02-2         0.03           μg/kg         2-2000         1           μg/kg         2-2000         1           μg/kg         0.01-20         1

\*Determinands which are not in bold are not in the scope of accreditation.





### MS-8 Perfluorinated Alkyl Substances (PFAS) in sediment

Year: 2024	Participants: 10 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of perfluorinated alkyl substances (PFAS) in sediment. This exercise can only be joined in both rounds as relatively low number of participants are expected.

#### **Test Materials**

The test materials cover a range of natural sediments from contaminated waters from the North Sea and/or Mediterranean. Each batch of material is prepared in bulk. The level of within and between sample homogeneity for the sediment is determined. All materials show to be homogeneous and stable for the purpose of the test.

#### Determinands and concentration ranges

The PFAS to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand	Unit	Concentration Range	Erro	or	CAS number
		Sediment	Const	Prop	
n-PFOS	µg/kg	0.05-2			1763-23-1
PFBA	µg/kg				375-22-4
PFPeA	µg/kg				2706-90-3
PFHxA	µg/kg				307-24-4
PFHxDA	µg/kg				67905-19-5
PFHpA	µg/kg				375-85-9
PFOA	µg/kg				335-67-1
PFNA	µg/kg				375-95-1
PFDA	µg/kg				335-76-2
PFUnDA	µg/kg	0.001-1			2058-94-8
PFDoA	µg/kg	0.001-0.1			307-55-1
PFTrDA	µg/kg	0.01-0.1			72629-94-8
PFTeDA	µg/kg	0.001-1			376-06-7
L-PFBS	µg/kg				375-73-5
L-PFHxS	µg/kg			1	355-46-4
L-PFHpS	µg/kg			1	375-92-8
PFOSA	µg/kg	0.01-1			754-91-6
PFDS	µg/kg				335-77-3





Determinand	Unit	Concentration Range	Error		CAS number
		Sediment	Const	Prop	
PFODA	µg/kg				16517-11-6
Total-PFOS	µg/kg	0.05-2			1763-23-1
GenX	µg/kg				62037-80-3
F-53B	µg/kg			1	73606-19-6
PFBSA	µg/kg			1	30334-69-1
PFHxSA	µg/kg			1	41997-13-1
PFPeS	µg/kg				2706-91-4
NMeFOSAA	µg/kg				909405-48-7
NEtFOSAA	µg/kg				2991-50-6
ADONA	µg/kg				958445-44-8
6:2 FTOH	µg/kg				647-42-7
8:2 FTOH	µg/kg				678-39-7
C604	µg/kg				1190931-41-9
Total-PFBS**	µg/kg				373-73-5
Total-PFHxS**	µg/kg				355-46-4
Total-PFHpS**	µg/kg				375-92-8

\* This exercise is not in the scope of accreditation.







# Proficiency tests in Seawater

AQ-1	Nutrients in Seawater	p37	AQ-8	Triazines and Organophosphorus Compounds in Seawater
AQ-2	Nutrients in Estuarine and Low Salinity Open Water	p38	AQ-11	Chlorophyll and Phaeopigments in Seawater
AQ-3	Metals in Seawater	p39	AQ-12	Organotin in Seawater p50
AQ-4	Mercury in Seawater	p41	AQ-13	Polycyclic Aromatic Hydrocarbons in p51 Seawater
AQ-5	Halogenated Organics in Seawater	p42	AQ-14	Dissolved organic Carbon in Seawate p53
AQ-6	Volatile Organics in Seawater	p44	AQ-15	Ocean acidification (Total alkalinity and DIC in Seawater)
AQ-7	Pentachlorophenol in Seawater	p46		





#### General Information

- Seawater samples are mainly prepared from seawater originating from the North Sea, Atlantic Ocean, the Baltic Sea and estuarine water to cover water samples with varying salinity. For chlorophyll samples sometimes freshwater samples are used.
- Water samples are filtered using a  $0.45\mu m / 0.2\mu m$  double membrane filter.
- The water samples are stable for the period of the test when stored properly.
- All seawater samples are prepared in bulk and tested for its homogeneity where relevant.
- Use your normal validated methods and procedures to analyse the test materials.
- Some of the test samples (AQ5; AQ7; AQ8 and AQ13) need to be spiked in your own laboratory because of stability issues.
- New determinands can be added on request.
- Reports contain all data reported under strict confidentiality, including Z-score plots and method information of methods used.







#### AQ-1 Nutrients in Seawater

Year: 2024	Participants: 55 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 3 per round	Sample size: 250 ml

Dauticipation form	Timetable	DT Cohomo	Casta
Participation form	Timetable	PT Scheme	Costs

This study covers the determination of nutrients in seawater test materials. The test materials are prepared in bulk, following the well-defined methods of A. Aminot and R. Kerouel (Analytical Chimica Acta 248(1991), pp.277-283 and Marine Chemistry 49(1995) pp.221-232).

#### Test Materials

Low nutrient seawater (LNSW), collected from the Eastern Atlantic Ocean during the late spring and summer months after the main plankton bloom, is used to prepare the test materials. This seawater is filtered to remove bacteria and particles. The pH of the seawater is adjusted to pH~7.2 using 0.1M hydrochloric acid. The seawater is spiked, mixed thoroughly and dispensed into appropriate 250 ml bottles for distribution. The dispensed materials are sterilised by autoclaving.

Homogeneity testing is performed on each batch of test materials produced. The nutrient test materials are stable for the period of the test and have also been shown to be stable for a period of some months even after opening but used under the correct conditions following the storage instructions.

#### Determinands and Concentration Ranges

The nutrients to be determined are given in the table below. The nitrogen species should be analysed in the distributed glass bottle and the silica and phosphorus species in the distributed plastic bottle. The table below also shows:

- The expected concentration range for the determinands in the spiked seawater materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range		Error	
		Seawater	Seawater (spiked)	Const	Prop
Ammonia	µmol/L	0.05-5	0.1-10	0.2	15.0%
Nitrate	µmol/L	0.05-15	0.1-25	0.1	7.5%
Nitrite	µmol/L	0.01-2	0.1-5	0.02	5.0%
Phosphate	µmol/L	0.02-5	0.1-10	0.04	5.0%
Silicate	µmol/L	0.2-20	0.2-50	0.2	6.0%
Total-N	µmol/L	2.5-25	5-50	1.5	10.0%
Total-P	µmol/L	0.05-5	0.2-10	0.08	10.0%
TOxN	µmol/L	0.05-15	0.1-25	0.08	6.0%
Salinity	psu			0.02	
Salinity indicative	psu			0.1	

\*Determinands which are not in bold are not in the scope of accreditation.

Constant error for salinity established by the SAB members





## AQ-2 Nutrients in Estuarine and Low Salinity Open Water

Year: 2024	Participants: 50 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 250 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of nutrients in estuarine water and low salinity open water test materials. The test materials are prepared in bulk, following the well-defined methods of A. Aminot and R. Kerouel (Analytical Chimica Acta 248(1991), pp.277-283 and Marine Chemistry 49(1995) pp.221-232).

#### Test Materials

Low nutrient seawater (LNSW), collected from the Baltic Sea during the late spring and summer months after the main plankton bloom, is used to prepare the estuarine test materials. The low salinity open water material is collected from the Baltic. These materials are filtered to remove bacteria and particles. The seawater is diluted with ultrapure demineralised water to produces the estuarine water matrix. The pH of the materials is adjusted to pH  $\sim$  7.2 using 0.1M hydrochloric acid. The materials are spiked, mixed thoroughly and dispensed into appropriate 250 mL bottles for distribution. The dispensed materials are sterilised by autoclaving.

Homogeneity testing is performed on each batch of test materials produced. The nutrient test materials are stable for the period of the test and have also been shown to be stable for a period of some months even after opening but used under the correct conditions following the storage instructions.

#### Determinands and Concentration Ranges

The nutrients to be determined are given in the table below. The nitrogen species should be analysed in the distributed glass bottle and the silica and phosphorus species in the distributed plastic bottle. The table below also shows:

- The expected concentration range for the determinands in the spiked seawater materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range		Error	
		Estuarine water (spiked)	Low salinity open water (spiked)	Const	Prop
Ammonia	µmol/L	2-50	0.2-5	0.25	10.0%
Nitrate	µmol/L	10-100	0.01-15	0.1	7.5%
Nitrite	µmol/L	0.5-25	0.002-2	0.02	5.0%
Phosphate	µmol/L	1-15	0.01-5	0.04	4.0%
Silicate	µmol/L	5-100	0.2-40	0.2	5.0%
Total-N	µmol/L	10-200	2-40	1.5	5.0%
Total-P	µmol/L	1-20	0.02-2	0.07	5.0%
TOxN	µmol/L	10-100	0.01-15	0.07	5.0%
Salinity	Psu			0.02	
Salinity indicative	Psu			0.1	

\*Determinands which are not in bold are not in the scope of accreditation.





#### AQ-3 Metals in Seawater

Year: 2024	Participants: 30 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 1000 ml

Participation form	Timetable	PT Scheme	Costs

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

#### **Test Materials**

The test materials are prepared in bulk from filtered seawater. Low salinity seawater test material is prepared by dilution with ultra-pure demineralised water. All test materials are preserved with 2 ml trace metal analysis grade nitric acid per litre of test material. Normally one spiked seawater, one unspiked seawater and one spiked low salinity seawater are supplied for each exercise.

N.B. In addition to the test materials mentioned above, we are intending to send one extra bottle with much higher concentrations ( $\pm 20$  times higher indicated). This bottle will be clearly indicated as high contaminated.

Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre polypropylene bottles for distribution. The test materials are stable for the purposes of the exercise.

#### Determinands and Concentration Ranges

The trace metals to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked seawater materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range		Error	
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop
Arsenic	µg/L	0.2-15	0.05-10	0.5	7.5%
Boron	µg/L	200-5000	1000-5000	125	10.0%
Cadmium	µg/L	0.05-1	0.001-1	0.025	10.0%
Chromium	µg/L	0.5-10	0.01-10	0.15	10.0%
Cobalt	µg/L	0.01-5	0.001-1	0.03	10.0%
Copper	µg/L	0.2-10	0.05-10	0.4	10.0%
Iron	µg/L	0.2-10	0.05-10	1.2	10.0%
Lead	µg/L	0.01-20	0.0002-15	0.06	10.0%
Magnesium	µg/L	100-50000	100-50000		
Manganese	µg/L	0.1-10	0.02-10	0.15	10.0%
Nickel	µg/L	0.1-40	0.2-40	0.15	10.0%
Silver	µg/L	0.1-2	0.02-2	0.05	10.0%
Strontium	µg/L	100-10000	100-10000		
Thallium	µg/L	0.01-2	0.001-0.5		
Tin	µg/L	0.1-5	0.02-5	0.2	12.5%





Determinand*	Unit	Concentrati	Error		
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop
Uranium	µg/L	0.01-2	0.001-0.5		
Vanadium	µg/L	0.2-10	0.1-10	0.25	10.0%
Zinc	µg/L	0.2-25	0.5-25	0.75	12.5%

\*Determinands which are not in bold are not in the scope of accreditation





# AQ-4 Mercury in Seawater

Year: 2024	Participants: 30 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 1000 ml

Participation form	Timetable	PT Scheme	Costs

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

## **Test Materials**

The test materials are prepared in bulk from filtered seawater. All test materials are preserved with 2 ml trace metal analysis grade nitric acid per litre of test material. Normally three spiked seawater test materials are supplied for each exercise.

*N.B.* In addition to the test materials mentioned, we are intending to send one extra bottle with much higher concentrations ( $\pm 20$  times higher indicated). This bottle will be clearly indicated as high contaminated.

Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into one-litre glass bottles for distribution. The test materials are stable for the purposes of the exercise.

#### Determinands and concentration ranges

Mercury should be determined in each test material. The table shows:

- The expected concentration range in the spiked test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration I	E	Frror	
		Low Salinity Seawater	Seawater	Const	Prop
		(spiked)	(spiked)		
Mercury	ng/L	1-5000	0.2 -40	1.75	12.5%





AQ-5 Halogenated Organics in Seawater						
Year: 2024			Participants: 12 laboratories expected			
Number of rounds: 1 per year		Start exercise: 1 April				
Number of materials: 3 per round		Sample size: 1000 ml				
Participation form	Timetable	e	PT Scheme	Costs		

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

## **Test Materials**

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The low salinity test material is prepared by dilution with ultra-pure demineralised water. The test materials are thoroughly mixed and dispensed into one-litre glass bottles. The participants are asked to dilute the supplied standard solutions using the supplied seawater test materials to produce the spiked test materials.

Homogeneity of the test materials is assumed, as they are spiked to the same concentration level. The test materials are stable for the purposes of the exercise.

## Determinands and Concentration Ranges

The organochlorines to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked test materials.
- The constant and proportional error that will be used for assessment of the results.

AA-EQS for HCH's is indicated as the sum for those determinands.

AA-EQS for aldrin, dieldrin, endrin and isodrin is indicated as the sum for those determinands. AA-EQS for pp'-DDD, pp'-DDE and op'-DDT is indicated as the sum for those determinands and pp'-DDT. AA-EQS for 1,2,3-TCB, 1,2,4-TCB and 1,3,5-TCB is indicated as the sum for those determinands. AA-EQS for Endosulphan-I and II are indicated as the sum of both isomers.

Determinand*	Unit	Concentration Range		Error	
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop
α-HCH	ng/L	2-500	0.2-20	0.5	15.0%
β-ΗϹΗ	ng/L	1-500	0.2-20	0.5	17.5%
γ-ΗCΗ	ng/L	2-500	0.5-20	0.5	20.0%
δ-ΗϹΗ	ng/L	1-500	0.2-20	0.5	25.0%
НСВ	ng/L	0.5-200	0.1-10	0.15	25.0%
HCBD	ng/L	2-500	0.2-20	0.75	25.0%
Aldrin	ng/L	2-1000	1-20	0.01	25.0%
Dieldrin	ng/L	2-1000	1-20	0.75	20.0%
Endrin	ng/L	2-1000	1-20	0.5	25.0%
Isodrin	ng/L	2-1000	1-20	0.75	25.0%





Determinand*	Unit	Concentration	Range	E	rror
		Low Salinity Seawater (spiked)	Seawater (spiked)	Const	Prop
pp'-DDD	ng/L	1-500	0.1-10	0.5	25.0%
pp'-DDE	ng/L	1-500	0.2-10	0.01	25.0%
op'-DDT	ng/L	1-500	0.2-20	0.3	25.0%
pp'-DDT	ng/L	1-500	0.2-20	0.2	25.0%
Endosulphan-I	ng/L	1-200	0.2-10	0.5	25.0%
Endosulphan-II	ng/L	0.5-200	0.1-10	0.5	25.0%
Pentachlorobenzene	ng/L	2-1000	0.2-10	0.15	25.0%
1,2,3-TCB	ng/L	2-500	1-20		1
1,2,4-TCB	ng/L	5-1000	1-20		1
1,3,5-TCB	ng/L	2-500	0.5-20		1
Trifluralin	ng/L	2-500	0.5-20	1.25	25.0%
PCB28	ng/L	2-500	0.5-20	0.2	25.0%
PCB31	ng/L	2-500	0.5-20		
PCB52	ng/L	2-500	0.5-20	0.1	22.5%
PCB101	ng/L	2-500	0.5-20	0.25	25.0%
PCB105	ng/L	2-500	0.5-20		
PCB118	ng/L	2-500	0.5-20	0.5	22.5%
PCB138	ng/L	2-500	0.5-20	0.01	25.0%
PCB138+PCB163	ng/L	2-500	0.5-20		
PCB153	ng/L	2-500	0.5-20	0.5	22.5%
PCB156	ng/L	2-500	0.5-20		
PCB180	ng/L	2-500	0.5-20	0.25	25.0%
Heptachlor	ng/L	2-500	0.5-20		
Cis-Heptachlorepoxide	ng/L	2-500	0.5-20		





#### AQ-6 Volatile Organics in Seawater

Year: 2024	Participants: 10 laboratories expected
Number of rounds: 1 per year	Start exercise: 1 April
Number of materials: 2 per round	Sample size: 1000 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of volatile organochlorine compounds (VOCs) in seawater test materials.

## Test Materials

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The test materials are thoroughly mixed and dispensed into one-litre glass bottles. These bottles are individually spiked with methanol solutions containing the volatile organic compounds (VOCs) to be analysed.

Glass beads are added to the spiked test materials to reduce the headspace volume in order to prevent volatilisation of the added VOCs.

Homogeneity of the test materials is assumed, as they were spiked to the same concentration level. The test materials are stable for the purposes of the exercise.

#### Determinands and Concentration Ranges

The VOCs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand* Unit		Concentration Range	E	rror
	1	Seawater (spiked)	Const	Prop
Benzene	µg/L	0.2-50	0.15	12.5%
Carbontetrachloride	µg/L	0.2-10	0.1	20.0%
Chloroform	µg/L	0.5-20	0.01	17.5%
1,2-Dichloroethane	µg/L	0.2-10	0.05	17.5%
Dichloromethane	µg/L	0.2-20	0.2	17.5%
Trichloroethene	µg/L	0.2-10	0.1	20.0%
1,1,1-Trichloroethane	µg/L	0.2-10	0.05	15.0%
1,1,2-Trichloroethane	µg/L	1-20	0.3	10.0%
Tetrachloroethene	µg/L	0.2-10	0.1	25.0%
Styrene	µg/L	0.1-50		
2-chlorotoluene	µg/L	0.1-10		
4-chlorotoluene	µg/L	0.1-10		
1,1-dichloroethane	µg/L	0.1-10	0.01	17.5%





Determinand*	Unit	Concentration Range	E	rror
		Seawater (spiked)	Const	Prop
1,1-dichloroethene	µg/L	0.1-10		
1,2-dichloropropane	µg/L	0.1-10		
1,2-dichlorobenzene	µg/L	0.1-10		
1,3-dichlorobenzene	µg/L	0.1-10		
1,4-dichlorobenzene	µg/L	0.1-10		
1,3,5-trimethylbenzene	µg/L	0.1-10		
1,1,1,2-tetrachloroethane	µg/L	0.1-10		
Chlorobenzene	µg/L	0.1-10		
cis-1,2-dichloroethene	µg/L	0.1-10		
trans-1,2-dichloroethene	µg/L	0.1-10		
Toluene	µg/L	0.1-10		
Ethylbenzene	µg/L	0.1-10		
o-xylene	µg/L	0.1-10		
m+p-xylene	µg/L	0.1-10		
Isopropylbenzene	µg/L	0.1-10		
n-propylbenzene	µg/L	0.1-10		
tert-butylbenzene	µg/L	0.1-10		





# AQ-7 Pentachlorophenol in Seawater

Year: 2024	Participants: 5 laboratories expected
Number of rounds: 1 per year	Start exercise: 1 April
Number of materials: 3 per round	Sample size: 1000 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of pentachlorophenol (PCP) in seawater test materials. As PCP is usually determined by a special method this exercise is offered separately.

# Test Materials

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The test materials are thoroughly mixed and dispensed into one-litre glass bottles. These bottles are individually spiked with methanol solutions containing PCP.

Homogeneity of the test materials is assumed, as they were spiked to the same concentration level. The test materials are stable for the purposes of the exercise.

# Determinands and Concentration Ranges

The table shows:

- The expected concentration range for the determinand in the spiked test materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range	Error	
		Seawater (spiked)	Const	Prop
Pentachlorophenol	ng/L	20-2000	5	15.0%





Q-8 Triazines and Organophosphorus Pesticides in Seawater
---

Year: 2024	Participants: 10 laboratories expected
Number of rounds: 1 per year	Start exercise: 1 April
Number of materials: 3 per round	Sample size: 1000 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of triazines and organophosphorus pesticides in seawater and low salinity seawater test materials.

## Test Materials

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The low salinity test material is prepared by dilution with ultra-pure demineralised water. The test materials are thoroughly mixed and dispensed into one-litre glass bottles. These bottles are distributed together with methanol standard solutions containing the compounds to be analysed. The participants are asked to dilute the supplied standard solutions using the supplied seawater test materials to produce the spiked test materials.

Homogeneity of the test materials is assumed, as they are spiked to the same concentration level. The test materials are stable for the purposes of the exercise.

## Determinands and Concentration Ranges

The triazines and organophosphorus compounds to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked test materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range		E	Error	
		Low salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
Aclonifen	ng/L	20-2000	2-200			
Alachlor	ng/L	20-2000	2-200	0.5	20.0%	
Atrazine	ng/L	20-2000	5-200	0.5	17.5%	
Atrazine- desethyl	ng/L	20-2000	5-200			
Azinphos-ethyl	ng/L	20-2000	5-200	5	25.0%	
Azinphos-methyl	ng/L	20-2000	5-200	10	25.0%	
Bifenox	ng/L	20-2000	5-200			
Bifenthrin	ng/L	20-2000	5-200			
Chlorfenvinphos	ng/L	20-2000	5-200	5	20.0%	
Chlorpyriphos	ng/L	20-2000	5-200	2	17.5%	
Chlotianidin	ng/L	20-2000	5-200			
Coumaphos	ng/L	20-2000	5-200			

Back to index





Determinand*	Unit	Concentration range		6	Error	
		Low salinity Seawater (spiked)	Seawater (spiked)	Const	Prop	
Cypermethrin	ng/L	20-2000	5-200			
Deltamethrin	ng/L	20-2000	5-200			
Demeton	ng/L	20-2000	5-200			
Diazinon	ng/L	20-2000	5-200	2	20.0%	
Dichlorvos	ng/L	20-2000	5-200	1	22.5%	
Dicofol	ng/L	20-2000	5-200			
Dimethoate	ng/L	20-2000	5-200	2.5	25.0%	
Diuron	ng/L	20-2000	5-200	1	15.0%	
Esfenvalerate	ng/L	20-2000	5-200			
Fenchlorphos	ng/L	20-2000	5-200			
Fenitrothion	ng/L	20-2000	5-200	5	25.0%	
Fenthion	ng/L	20-2000	5-200			
Glyphosate	ng/L	20-2000	5-200			
Imidacloprid	ng/L	20-2000	5-200			
Irgarol-1051	ng/L	20-2000	5-200	3	10.0%	
Isoproturon	ng/L	20-2000	5-200	0.01	17.5%	
Malathion	ng/L	20-2000	5-200	1	20.0%	
Nicosulfuron	ng/L	20-2000	5-200			
Omethoate	ng/L	20-2000	5-200			
Parathion-ethyl	ng/L	20-2000	5-200	2	25.0%	
Parathion- methyl	ng/L	20-2000	5-200	5	25.0%	
Permethrin	ng/L	20-2000	5-200			
Quinoxyfen	ng/L	20-2000	5-200			
Simazine	ng/L	20-2000	5-200	0.5	15.0%	
Terbutryn	ng/L	20-2000	5-200			
Terbuthylazine	ng/L	20-2000	5-200			
Thiacloprid	ng/L	20-2000	5-200			
Thiamethoxam	ng/L	20-2000	5-200			
Triazophos	ng/L	20-2000	5-200			
Triclosan	ng/L	20-2000	5-200			





# AQ-11 Chlorophyll and Phaeopigments in Seawater

Year: 2024	Participants: 45 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: filter

	The state		Casta
Participation form	Timetable	PT Scheme	Costs

This study covers the determination of chlorophyll and phaeopigments in seawater and estuarine water. Normally, filtered residues are prepared from seawater or estuarine water. Occasionally, filtered residues are prepared from freshwater.

# Test Materials

Test materials are prepared from seawater or estuarine water and sub-sampled onto Whatman GF/F, 47 mm filter papers each test material is immediately 'flash frozen' in liquid nitrogen. Selected filters at regular intervals are chosen for homogeneity testing. The test materials are homogeneous for the purposes of the LP study.

# Determinands and Concentration Ranges

The pigments to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range	E	rror
		Filtered Residues	Const	Prop
Chlorophyll-a	µg/L	0.1-50	0.2	17.5%
Chlorophyll-b	µg/L	0.01-10	0.15	20.0%
Chlorophyll-c	µg/L	0.02-5	0.3	20.0%
Phaeopigments	µg/L	0.02-2.5	0.10	25.0%
Chlorophyll-a (HPLC)	µg/L	0.1-50	0.01	25.0%
Chlorophyll-b (HPLC)	µg/L	0.01-10		
Chlorophyll-c (HPLC)	µg/L	0.02-5		
Chlorophyll-a (corrected)	µg/L	0.1-50	0.15	17.5%

\*Determinands which are not in bold are not in the scope of accreditation.





# AQ-12 Organotin in Seawater

Year: 2024	Participants: 20 laboratories expected	
Number of rounds: 1 per year	Start exercise: 1 April	
Number of materials: 2 per round	Sample size: 750-1000 ml	

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of organotin compounds in seawater test materials.

## **Test Materials**

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The test materials are spiked, thoroughly mixed and dispensed into 1 litre glass bottles for distribution.

Homogeneity of the test materials is assumed, as they are produced in bulk. The test materials are stable for the purposes of the exercise.

#### Determinands and concentration ranges

The organotin compounds to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked test materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range	Error	
		Seawater (spiked)	Const	Prop
Tributyltin (TBT)	ng Sn/L	1-200	0.03	25.0%
Dibutyltin (DBT)	ng Sn/L	1-100	0.03	25.0%
Monobutyltin (MBT)	ng Sn/L	1-200	0.01	25.0%
Triphenyltin (TPhT)	ng Sn/L	1-200	0.02	25.0%
Diphenyltin (DPhT)	ng Sn/L	1-100	0.01	25.0%
Monophenyltin (MPhT)	ng Sn/L	1-50	0.01	25.0%





# AQ-13 Polycyclic Aromatic Hydrocarbons in Seawater

Year: 2024	Participants: 15 laboratories expected
Number of rounds: 1 per year	Start exercise: 1 April
Number of materials: 3 per round	Sample size: 750-1000 ml

Participation form	Timetable	PT Scheme	Costs

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

## **Test Materials**

The seawater for this study is collected from the Eastern Atlantic Ocean and is filtered to remove bacteria and particles. The test materials are spiked, thoroughly mixed and dispensed into glass bottles for distribution.

Homogeneity of the test materials is assumed, as they are produced in bulk. The test materials are stable for the purposes of the exercise.

## Determinands and Concentration Ranges

The PAHs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the spiked test materials.

- The constant and proportional error that will be used for assessment of the results.

AA-EQS for benzo[g,h,i]perylene and indeno(1,2,3-cd)pyrene is indicated as the sum of those determinands.

AA-EQS for benzo[b]fluoranthene and benzo[k]fluoranthene is indicated as the sum of those determinands.

Determinand*	Unit	Cor	Concentration range			rror
		Seawater (sediment spiked)	Seawater (spiked)	Low Salinity Seawater (spiked)	Const	Prop
Acenaphthene	µg/L	0.02-20	0.2-5	0.5-20	0.02	20.0%
Acenaphthylene	µg/L	0.01-1	0.5-10	0.5-20	0.07	20.0%
Anthracene	µg/L	0.2-20	0.05-2	0.5-10	0.05	20.0%
Benzo[a]anthracene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Benzo[a]pyrene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Benzo[b]fluoranthene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Benzo[e]pyrene	µg/L	0.1-10	0.001-0.1	0.01- 0.5		
Benzo[k]fluoranthene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Benzo[g,h,i]perylene	µg/L	0.02-2	0.001-0.1	0.01- 0.5	0.01	25.0%
Chrysene	µg/L	0.1-10	0.001-0.1	0.01- 0.5		
Dibenzo[ah]anthracene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Fluorene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	20.0%





Determinand*	Unit	Concentration range			E	rror
		Seawater (sediment spiked)	Seawater (spiked)	Low Salinity Seawater (spiked)	Const	Prop
Fluoranthene	µg/L	0.4-40	0.05-2	0.1-10	0.01	25.0%
Indeno(1,2,3-cd)pyrene	µg/L	0.2-40	0.02-1	0.1-5	0.01	25.0%
Naphthalene	µg/L	0.1-10	0.5-10	1-50	0.1	22.5%
Phenanthrene	µg/L	0.2-50	0.05-2	0.5-10	0.1	17.5%
Pyrene	µg/L	0.1-10	0.001-0.1	0.01- 0.5	0.01	25.0%
Total Petroleum- Hydrocarbons	µg/L	0.1-10	0.001-0.1	0.01- 5		





# AQ-14 Dissolved Organic Carbon in Seawater

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 250 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of dissolved organic carbon in seawater test materials. The test materials are prepared in bulk, following the well-defined methods of A. Aminot and R. Kerouel (Analytical Chimica Acta 248(1991), pp.277-283 and Marine Chemistry 49(1995) pp.221-232).

## Test Materials

Low nutrient seawater (LNSW), collected from the Eastern Atlantic Ocean during the late spring and summer months after the main plankton bloom, is used to prepare the test materials. This seawater is filtered to remove bacteria and particles.

Homogeneity testing is performed on each batch of test materials produced. The test materials are stable for the period of the test and have also been shown to be stable for a period of some months even after opening but used under the correct conditions following the storage instructions.

# Determinands and Concentration Ranges

The dissolved organics carbon (DOC) content should be analysed in the distributed glass bottles. The table below also shows:

- The expected concentration range for DOC in the spiked seawater materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range	Error	
		Seawater (spiked)	Const	Prop
DOC	mg C/L	0.5-20	0.25	6.0%

\*This determinand is in the scope of accreditation.





# AQ-15 Ocean Acidification

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 1 per year	Start exercise: 1 April
Number of materials: 3 per round	Sample size: 500 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of total alkalinity and dissolved inorganic carbon in the seawater test materials. The test materials are prepared in bulk.

# Test Materials

Low nutrient seawater (LNSW), collected from the Eastern Atlantic Ocean and Baltic Sea during the late spring and summer months after the main plankton bloom, is used to prepare the test materials. This seawater is filtered to remove bacteria and particles.

Homogeneity testing is performed on each batch of test materials produced. The test materials are stable for the period of the test under the correct conditions following the storage instructions.

# Determinands and Concentration Ranges

The dissolved inorganic carbon (DIC) content and Total Alkalinity should be analysed in the distributed glass bottles. The table below also shows:

- The expected concentration range for DIC in the spiked seawater materials.

- The constant and proportional error that will be used for assessment of the results.

The proportional and constant error is established by the SAB members based on the "Weather goal"

Determinand*	Unit	Concentration range	Error	
		Seawater (spiked)	Const	Prop
DIC	µmol/kg	10-5000	1	0.5
Total Alkalinity	µmol/kg	100-5000	1	0.5
рН			0.04	

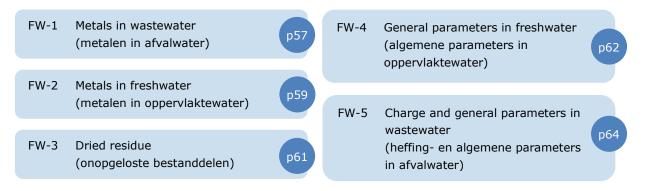






# Freshwater and wastewater

## Proficiency tests in freshwater and wastewater







# General information

The proficiency tests in the freshwater and wastewater programme are a continuation of the proficiency tests previously organised by Rijkswaterstaat (RWS-CIV) in the Netherlands. Rijkswaterstaat is the implementing agency of the Dutch Ministry of Infrastructure and Water Management and has organised proficiency tests in freshwater and wastewater until 2022 to support Dutch and Belgian laboratories involved in national monitoring. WEPAL-QUASIMEME was already the supplier of the test materials used in these proficiency tests. From 2023, WEPAL-QUASIMEME offers the full organisation of these proficiency tests and will offer five new proficiency tests for freshwater and wastewater.

Participation is open to all laboratories worldwide, however, some determinands are only suitable for local laboratories as analysis needs to be performed on fresh samples. Preservation of samples is not applicable for all types of determinands.





# FW-1 Metals in wastewater (metalen in afvalwater)

Year: 2024	Participants: 12 laboratories expected
Number of rounds: 1 per year	Period exercise: 20 March – 10 April
Number of materials: 3 per round	Sample size: 1000 ml



This study covers the determination of trace metals in wastewater test materials and participation is open for all laboratories world-wide. A request to participate can be made by sending an e-mail to wepalquasimeme@wur.nl.

#### Test Materials

The test materials are prepared in bulk from filtered (1 mm) wastewater. All test materials are preserved with 2 ml trace metal analysis grade nitric acid per litre of test material. Normally one blank wastewater and two spiked wastewater samples are supplied for each exercise.

Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre polypropylene bottles for distribution. The test materials are stable for the purposes of the exercise.

# Determinands and Concentration Ranges

Determinand*	Unit	Concentration Range	Err	or
			Const	Prop
Al - Aluminium	mg/l	20-200		
Ag - Silver	µg/l	25–250		
As - Arsenic	µg/l	10-50		
Ba - Barium	µg/l			
Be - Beryllium	µg/l	5–50		
Ca - Calcium	mg/l	>50		
Cd - Cadmium	µg/l	5–50		
Ce - Cerium	µg/l			
Co - Cobalt	µg/l	5–50		
Cr - Chromium	µg/l	100-1000		
Cu - Copper	µg/l	100-1000		
Fe - Iron	mg/l	20-200		
Hg - Mercury	µg/l	2–20		
K - Kalium	mg/l	>50		
Mg – Magnesium	mg/l	>25		
Mn - Manganese	mg/l	20-200		
Mo - Molybdene	µg/l	100-1000		
Na - Sodium	mg/l	>50		
Ni - Nickel	µg/l	100-1000		
Pb - Lead	µg/l	100-1000		





Determinand*	Unit	Concentration Range	Err	or
			Const	Prop
S – Sulfur	mg/l			
Sb - Antimony	µg/l	5–50		
Se - Selenium	µg/l	500-5000		
Sn – Stannum	µg/l	100-1000		
Sr – Strontium	µg/l			
Te – Tellurium	µg/l			
Ti - Titanium	µg/l	100-1000		
TI _ Thallium	µg/l			
U – Uranium	µg/l			
V - Vanadium	µg/l	100-1000		
W - Tungsten	µg/l			
Zn – Zinc	µg/l	100-1000		





# FW-2 Metals in freshwater (metalen in oppervlaktewater)

Year: 2024	Participants: 12 laboratories expected
Number of rounds: 1 per year	Period exercise: 20 March – 10 April
Number of materials: 3 per round	Sample size: 1000 ml



This study covers the determination of trace metals in freshwater test materials and participation is open for all laboratories worldwide. A request to participate can be made by sending an e-mail to wepalquasimeme@wur.nl.

#### **Test Materials**

The test materials are prepared in bulk from filtered (1 mm) freshwater. All test materials are preserved with 2 ml trace metal analysis grade nitric acid per litre of test material. Normally one blank freshwater and two spiked freshwater samples are supplied for each exercise. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre polypropylene bottles for distribution. The test materials are stable for the purposes of the exercise.

# Determinands and Concentration Ranges

Determinand*	Unit	Concentration Range	Er	ror
			Const	Prop
Ag – Silver	µg/l			
Al - Aluminium	µg/l			
As – Arsenic	µg/l	1–20		
B – Boron	µg/l	1-100		
Ba -Barium	µg/l	1-200		
Be – Beryllium	µg/l	1–5		
Ca - Calcium	mg/l	>50		
Cd – Cadmium	µg/l	1–5		
Co – Cobalt	µg/l	1–20		
Cr – Chromium	µg/l	1–50		
Cu – Copper	µg/l	1–50		
Fe – Iron	mg/l	1–5		
Hg – Mercury	µg/l	0.1-1		
K – Kalium	mg/l	>10		
Mg – Magnesium	mg/l	>25		
Mn - Manganese	µg/l	1-200		
Mo – Molybdene	µg/l	1–20		
Na - Sodium	mg/l	>50		
Ni – Nickel	µg/l	1–50		
Pb - Lead	µg/l	1-50		
S - Sulfur	mg/l			
Sb - Antimony	µg/l	1–20		





Determinand*	Unit	Concentration Range	Err	or
			Const	Prop
Se – Selenium	µg/l	1–20		
Sn – Stannum	µg/l	1–200		
Sr - Strontium	µg/l			
Te - Tellurium	µg/l			
TI – Thallium	µg/l			
U – Uranium	µg/l			
V – Vanadium	µg/l	1-100		
W – Tungsten	µg/l			
Zn – Zinc	µg/l	1–50		





# FW-3 Dried residue (onopgeloste bestanddelen)

Year: 2024	Participants: 12 laboratories expected
Number of rounds: 1 per year	Period exercise: 17 April – 8 May
Number of materials: 5 per round	Sample size: 1000 ml



This study covers the determination of dried residue in freshwater and wastewater test materials. Participation is only applicable for local laboratories as analysis needs to be performed on fresh samples. A request to participate can be made by sending an e-mail to <u>wepalquasimeme@wur.nl</u>.

## Test Materials

The test materials are prepared in bulk from filtered (1 mm) freshwater and wastewater. In addition, a standard sample is prepared from Silica in tapwater. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into 1 litre glass bottles for distribution. Samples are freshly prepared a day in advance to the pre-determined date and sent cooled by courier with same-day delivery. Samples need to be kept cooled until analysis. **The pre-determined date of analysis of these samples is 19<sup>th</sup> of April 2024**. These samples cannot be distributed worldwide.

# Determinands and Concentration Ranges

Determinand*	Matrix	Unit	Concentration Range	Error	
				Const	Prop
Dried residue	Freshwater	mg/l	1-100		
Loss on ignition	Freshwater	mg/l	1-100		
Dried residue	Wastewater	mg/l	1-100		
Loss on ignition	Wastewater	mg/l	1-100		
Dried residue	Standard	mg/l	1-100		





# FW-4 General parameters in freshwater (algemene parameters in oppervlaktewater)

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 1 per year	Period exercise: 24 April – 22 May
Number of materials: 3 per round	Sample size: 500-1150 ml



This study covers the determination of general parameters in freshwater test materials. Participation of acidified samples is open for world-wide laboratories. Participation of determinands where conservation is 'completely filled' is only applicable for local laboratories as analysis needs to be performed on fresh samples. A request to participate can be made by sending an e-mail to <u>wepalquasimeme@wur.nl</u>.

# Test Materials

The test materials are prepared in bulk from filtered (1 mm) freshwater. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into glass or plastic bottles for distribution.

Acidified samples with conservation 'HCl pH<2' or 'H<sub>2</sub>SO<sub>4</sub> pH<2' are stable for the purpose of the exercise and can be send worldwide. Samples with conservation 'completely filled' are freshly prepared a day in advance to the pre-determined date and sent cooled by courier with same-day delivery. Samples need to be kept cooled until analysis. **The pre-determined date of analysis of the 'completely filled' samples is 26<sup>th</sup> of April 2024**. Therefore, these samples cannot be distributed worldwide.

# Determinands and Concentration Ranges

Determinand*	Unit	Concentration Range	Conservation	Sample size	En	Error	
				SIZE	Const	Prop	
Mineral oil (minerale olie)	mg/l	1-5	HCI pH<2	800 ml			
BOD-5 (BZV-5)	mg/l	5-50	Completely filled	~1150 ml			
тос	mg/l	1-20					
DOC	mg/l	1-20					
Cl - Chloride	mg/l	100-300	Completely filled	~1150 ml			
EC (EGV) (25°C)	mS/m	-					
NO2 (nitrite)	mg/l	0.1-1					
NO3 (nitrate)	mg/l	1-5					
PO4 (phosphate)	mg/l	0.1-1					
SiO2 (silicon oxide)	mg/l	5-20					
SO4 (sulfate)	mg/l	100-300					
pН	-	-					
F (fluoride)	mg/l	1-50					
Br (bromide)	mg/l	1-50					
KjN (kjeldahl nitrogen)	mg/l	1-5	H₂SO₄ pH<2	500 ml			
NH4 (ammonium)	mg/l	1-5					
tN (total nitrogen)	mg/l	1-5					

Back to index





Determinand*	Unit	Concentration Range	Conservation	Sample size	Err	or
					Const	Prop
tP (total phosphorus)	mg/l	0.1-2				
COD (CZV)	mg/l	3-30				





# FW-5 Charge and general parameters in wastewater (heffing- en algemene parameters in afvalwater)

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 1 per year	Period exercise: 2 October – 30 October
Number of materials: 3 per round	Sample size: 500-1150 ml



This study covers the determination of general parameters in wastewater test materials. Participation of acidified samples is open for world-wide laboratories. Participation of determinands where conservation is 'completely filled' is only applicable for local laboratories as analysis needs to be performed on fresh samples. A request to participate can be made by sending an e-mail to <u>wepalquasimeme@wur.nl</u>.

## Test Materials

The test materials are prepared in bulk from filtered (1 mm) freshwater. Homogeneity of the test materials is assumed, as they were prepared in bulk and thoroughly mixed, before being dispensed into glass or plastic bottles for distribution. Acidified samples with conservation  $H_2SO_4$  pH<2' are stable for the purpose of the exercise and can be send worldwide. Samples with conservation 'completely filled' are freshly prepared a day in advance to the pre-determined date and sent cooled by courier with same-day delivery. Samples need to be kept cooled until analysis. **The pre-determined date of analysis of the BOD-5 samples is 4<sup>th</sup> October 2024.** Therefore, these samples cannot be distributed worldwide.

# Determinands and Concentration Ranges

Determinand*	Unit	Concentration Range	Conservation	Sample size	En	Error	
				5120	Const	Prop	
Mineral oil (Minerale olie)	mg/l	1-20	H <sub>2</sub> SO <sub>4</sub> pH<2	750 ml			
BOD-5 (BZV-5)	mg/l	10-50	Completely filled	~550 ml			
COD (CZV)	mg/l	100-1000	H₂SO₄ pH<2	500 ml			
тос	mg/l	4-20					
Kj-N (Kjeldahl nitrogen)	mg/l	10-20					
NH4 (ammonium)	mg/l	5-10					
tP (total phosphorus)	mg/l	5-10					
tN (total nitrogen)	mg/l	10-20					
F (fluoride)	mg/l	1-50	Completely filled	~1150 ml			
Br (bromide)	mg/l	1-50					
Cl (chloride)	mg/L	>50					
NO2 (nitrite)	mg/l	1-5					
NO3 (nitrate)	mg/l	1-25					
o-PO4 (ortho-phosphate)	mg/l	1-10					
SO4 (sulfate)	mg/l	10-200					
рН	-						
EC (EGV) (25°C)	mS/m						







# Plants

# Proficiency tests in Plants

IPE - International Plant-analytical Exchange Programme



## **General Information**

- The sample types/matrices are chosen to represent the materials which you normally analyse.
- The plant samples are dried at max. 70°C, milled and sieved to <0.5 mm.
- The homogeneity of each batch of samples is tested on a selection of parameters.
- The dried plant samples are stored at -20°C and are stable over a number of years.
- You analyse the test materials according to your normal validated methods and procedures for those elements and parameters you are interested in.
- Your results are processed at WEPAL-QUASIMEME and published every three months under confidential code names.
- The reports contain all data, statistical evaluation including Z-score plots and method information about the method used.
- Your reports are available within three weeks after the submission deadline.
- New determinands can be added on request.







# IPE International Plant-analytical Exchange Programme

Year: 2024	Participants: 170 laboratories expected
Number of rounds: 4 per year	Start exercise: 1 January, 1 April, 1 July, 1 October
Number of materials: 4 per round	Sample size: 20 g



This study covers the determination of chemical analysis in plant materials. Participation is open to all laboratories.

## Determinands

Concentration ranges and constant and proportional errors are available in the determinand list in the participants website.

Determinand group	Determinand*			
Inorganic Chemical Composition	Ag; As; B; Ba; Be; Bi; Br; Ca; Cd; Cl (as Cl); Co; Cr; Cs; Cu; F; Fe; Ga; Hg; I; K; Li; Mg; Mn; Mo; N-Kjeldahl (as N); N-NH4 (as N); N-NO3 (as N); Na; Ni; P (as P); Pb; Pd; Pt; Rb; Rh; S (as S); Sb; Se; Sn; SO4 (as SO4); Sr; Ti; V; Zn;			
Real totals	Al; C-elementary; N-elementary; Si;			
Acid extractable (So-called totals)	Al; Si;			
Other determinations	delta 13C; delta 15N;			
Nutritional values	ADF-ash-free; <b>Crude fibre</b> ; NDF-ash-containing; NDF-ash-free; Polysaccharides (starch); <b>Total ash</b> ; Total Disaccharides; Total fat; Total monosaccharides;			

\*Determinands not in bold are not in the scope of accreditation







# Biota

## Proficiency tests in Biota

BT-1	Trace Metals in Biota	p68	BT-8	Organotin in Biota	p74
BT-2	Chlorinated Organics in Biota	p70	BT-9	Brominated Flame Retardants in Biota	p75
BT-4	Polycyclic Aromatic Hydrocarbons in Biota	p72	BT-10	Perfluorinated Alkyl Substances (PFAS) in Biota	p77

#### General information

- Biota test materials are collected from contaminated waters, seawater and coastal sites in Europe like the North Sea, Baltic Sea and the Mediterranean Sea.
- Depending on the type of exercise, biota samples are prepared from mussel tissue, fish tissue, fish liver or shrimp and contain ±50 gram of homogenized tissue.
- Biota samples are either natural contaminated or spiked with a selection of determinands where relevant.
- Biota samples are autoclaved and are stable over a number of years when stored at room temperature.
- Reports contain all data reported under strict confidentiality, including Z-score plots and method information of the methods used.







#### BT-1 Trace Metals in Biota

Year: 2024	Participants: 50 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 30-50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of trace metals, ash weight, dry weight and total and extractable lipid in biota test materials.

# **Test Materials**

The test materials cover a range of natural biota species from contaminated waters from the North Sea and/or Mediterranean. The supplied biota test materials can consist of fish muscle, fish liver and shellfish tissue. Wet biota test materials are homogenised and sterilised by autoclaving. These biota test materials have been shown to be stable over a number of years when stored at room temperature.

## Determinands and concentration ranges

The trace metals to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

In addition to the parameters given in this table, we will add several additional metals into the dataset form on the Participant's sites. There you will find e.g. Li, Be, S, Sc, Rb, Sr, Y, Zr, Pd, Sb, Te, Cs, La, Ce, Nd, Ta, W, Pt, Au, Tl, Bi, Th and MeHg. In case enough participants report results these additional metals will be added permanently to the programme.

Determinand*	Unit	C	oncentration Rang	e	E	rror
		Fish Liver	Fish Muscle	Shellfish	Const	Prop
		Tissue	Tissue	Tissue		
Aluminium	mg/kg	1-100	0.5-10	2-50	0.6	25.0%
Antimony	µg/kg				0.5	25.0%
Arsenic	mg/kg	1-5	1-10	0.2-10	0.02	10.0%
Barium	µg/kg	5-500	5-500	100-10000	35	15.0%
Cadmium	µg/kg	5-1000	0.5-50	10-500	2	10.0%
Calcium	mg/kg	20-1000	50-5000	50-2000		
Chromium	µg/kg	20-1000	25-500	10-5000	20	22.5%
Cobalt	µg/kg	10-500	1-100	10-500	1	10.0%
Copper	µg/kg	2000-10000	100-1500	50-10000	40	10.0%
Iron	mg/kg	10-500	2.5-200	5-200	1	12.5%
Lead	µg/kg	10-1000	2.5-50	10-1000	5	10.0%
Magnesium	mg/kg	50-1000	50-1000	100-2000	12.5	7.5%
Manganese	µg/kg	200-5000	50-5000	500-5000	25	12.5%





Determinand*	Unit	Concentration Range				rror
		Fish Liver	Fish Muscle	Shellfish	Const	Prop
		Tissue	Tissue	Tissue		
Mercury	µg/kg	20-100	10-1000	2-500	1	12.5%
Molybdenum	µg/kg	20-500	2-200	10-500	5	15.0%
Nickel	µg/kg	20-1000	10-200	10-2000	15	12.5%
Phosphorus	mg/kg	2000-3000	2000-5000	2000-5000		
Potassium	mg/kg	500-5000	500-5000	500-5000		
Selenium	µg/kg	200-5000	50-2000	200-1000	30	12.5%
Silver	µg/kg	20-1000	0.5-50	1-500	1	20.0%
Sodium	mg/kg	200-5000	200-5000	1000-10000	0.01	10.0%
Tin	µg/kg	10-1000	10-1000	10-1000	15	25.0%
Titanium	µg/kg	50-2000	50-2000	50-2000		
Uranium	µg/kg	0.2-50	0.2-50	2-100	0.4	12.5%
Vanadium	µg/kg	5-200	2-200	50-5000	6	17.5%
Zinc	mg/kg	10-50	2-20	2-200	0.4	10.0%
Ash-weight	%					
Dry-weight	%				0.25	3.0%
Total-Lipid	%				0.4	7.5%
Extractable- Lipid	%					

\*Determinands which are not in bold are not in the scope of accreditation





# BT-2 Chlorinated Organics in Biota

Year: 2024	Participants: 40 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 30-50 g

Participation form	Timetable	PT Scheme	Costs
•			

This study covers the determination poly chlorinated biphenyls (PCBs), organochlorine pesticides (OCPs), total and extractable lipid in biota test materials.

# **Test Materials**

The test materials cover a range of natural biota species from contaminated waters from the North Sea and/or Mediterranean. The supplied biota test materials can consist of fish muscle, fish liver and shellfish tissue. Wet biota test materials are homogenised and sterilised by autoclaving. These biota test materials have been shown to be stable over a number of years when stored at room temperature.

# Determinands and Concentration Ranges

The organochlorines to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

EQS<sub>biota</sub> for heptachlor and heptachlor-epoxide is indicated as the sum of those determinands.

Determinand*	Unit	Concentration range			Error	
		Fish Liver tissue and	Fish Muscle	Shellfish	Const	Prop
		Freshwater Fish	Tissue	Tissue		
PCB28	µg/kg	0.5-50	0.05-5	0.05-5	0.02	25.0%
PCB31	µg/kg	0.2-10	0.03-3	0.03-3	0.04	25.0%
PCB52	µg/kg	1-100	0.05-20	0.05-5	0.1	17.5%
PCB99	µg/kg					
PCB101	µg/kg	5-300	0.1-50	0.1-20	0.15	15.0%
PCB105	µg/kg	2-100	0.05-10	0.05-10	0.05	17.5%
PCB107	µg/kg					
PCB108	µg/kg					
PCB109	µg/kg					
PCB110	µg/kg					
PCB111	µg/kg					
PCB112	µg/kg					
PCB113	µg/kg					
PCB114	µg/kg					
PCB118	µg/kg	5-300	0.2-30	0.2-20	0.1	15.0%





Determinand*	Unit	Concentration range			Error	
		Fish Liver tissue and Freshwater Fish	Fish Muscle Tissue	Shellfish Tissue	Const	Prop
PCB128	µg/kg					
PCB138+PCB163	µg/kg	10-600	0.3-70	0.3-30	0.3	17.5%
PCB138	µg/kg	10-600	0.3-70	0.3-30	0.1	17.5%
PCB153	µg/kg	20-1000	0.4-100	0.4-40	0.1	17.5%
PCB156	µg/kg	0.5-40	0.03-10	0.03-10	0.05	17.5%
PCB170	µg/kg					
PCB180	µg/kg	2-200	0.05-20	0.05-5	0.03	17.5%
PCB183	µg/kg					
PCB187	µg/kg					
PCB194	µg/kg					
PCB203	µg/kg					
PCB209	µg/kg			-		
a-HCH	µg/kg	0.05-5	0.05-5	0.05-5	0.03	25.0%
β-НСН	µg/kg	0.1-5	0.05-5	0.05-5	0.03	25.0%
ү-НСН	µg/kg	0.05-5	0.05-5	0.05-5	0.05	25.0%
δ-НСН	µg/kg	0.05-5	0.05-5	0.05-5		
НСВ	µg/kg	1-50	0.02-5	0.02-5	0.05	22.5%
HCBD	µg/kg	0.05-5				
Dieldrin	µg/kg	0.5-100	0.2-20	0.2-20	0.15	25.0%
pp'-DDD	µg/kg	0.5-100	0.1-10	0.1-10	0.01	25.0%
pp'-DDE	µg/kg	10-500	0.3-30	0.3-30	0.1	20.0%
op'-DDT	µg/kg	0.1-2	0.01-1	0.01-1	0.15	25.0%
pp'-DDT	µg/kg	0.1-10	0.1-10	0.1-10	0.1	25.0%
Transnonachlor	µg/kg	0.05-40	0.02-10	0.02-10	0.03	15.0%
Heptachlor	µg/kg					
cis-Heptachlor epoxide	µg/kg					
Cis-chlordane	µg/kg					
Trans-chlordane	µg/kg					
Oxychlordane	µg/kg					
Dicofol	µg/kg					
Total-Lipid	%				0.4	7.5%
Extractable-Lipid	%				0.4	7.5%

\*Determinands which are not in bold are not in the scope of accreditation





# BT-4 Polycyclic Aromatic Hydrocarbons in Biota

Year: 2024	Participants: 35 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 30-50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of Polycyclic Aromatic Hydrocarbons (PAHs) and total and extractable lipid in shellfish tissue test materials.

# **Test Materials**

The test materials consist of natural shellfish species from contaminated waters from the North Sea and/or Mediterranean. The supplied wet shellfish tissues are homogenised and sterilised by autoclaving. These test materials have shown to be stable over a number of years when stored at room temperature.

# Determinands and Concentration Ranges

The PAHs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range	Error	
		Shellfish Tissue	Const	Prop
Acenaphthene	µg/kg	0.5-100	0.5	25.0%
Acenaphthylene	µg/kg	0.2-5	0.4	25.0%
Anthracene	µg/kg	0.2-10	0.3	25.0%
Benzo[a]anthracene	µg/kg	0.2-20	0.15	20.0%
Benzo[a]fluorene	µg/kg			
Benzo[a]pyrene	µg/kg	0.2-5	0.1	25.0%
Benzo[b]fluoranthene	µg/kg	0.2-10	0.25	25.0%
Benzo[k]fluoranthene	µg/kg	0.2-10	0.1	25.0%
Benzo[e]pyrene	µg/kg	0.2-10	0.2	25.0%
Benzo[g,h,i]perylene	µg/kg	0.2-10	0.15	20.0%
Chrysene	µg/kg	0.2-20	0.15	22.5%
Chrysene+Triphenylene	µg/kg	0.2-20	0.1	25.0%
Triphenylene	µg/kg	0.1-10		
Dibenz[a,h]anthracene	µg/kg	0.2-5	0.1	17.5%
Dibenzo[a,i]pyrene	µg/kg			
Dibenzothiophene	µg/kg	0.2-5	0.25	25.0%
Fluoranthene	µg/kg	5-50	0.4	20.0%
Fluorene	µg/kg	1-50	0.3	25.0%
Indeno[1,2,3-cd]pyrene	µg/kg	0.2-5	0.2	25.0%





Determinand*	Unit Concentration range		Error	
		Shellfish Tissue	Const	Prop
Naphthalene	µg/kg	1-100	0.6	25.0%
1-methylnaphthalene	µg/kg			
2-methylnaphthalene	µg/kg			
1-methylanthracene	µg/kg			
2- methylanthracene	µg/kg			
1 methylphenanthrene	µg/kg			
Perylene	µg/kg	0.1-5	0.5	20.0%
Phenanthrene	µg/kg	2-50	1	25.0%
2-Methylphenanthrene	µg/kg	0.2-20	1.2	10.0%
3,6-Dimethylphenanthrene	µg/kg	0.2-10		
1,2-benzodiphenylene sulfide	µg/kg			
Pyrene	µg/kg	1-50	0.4	20.0%
1-Methylpyrene	µg/kg			
Benzo Fluoranthenes (a+b+j+k)	µg/kg			
Total-Lipid	%		0.4	7.5%
Extractable-Lipid	%			
C1-dibenzothiophenes	µg/kg			
C2-dibenzothiophenes	µg/kg			
C3-dibenzothiophenes	µg/kg			
C1-phenanthrenes/anthracenes	µg/kg			
C2-phenanthrenes/anthracenes	µg/kg			
C3-phenanthrenes/anthracenes	µg/kg			
C1-pyrenes/fluoranthenes	µg/kg			
C2-pyrenes/fluoranthenes	µg/kg			
C1-chrysenes	µg/kg			
C2-chrysenes	µg/kg			
C1-benzofluoranthenes	µg/kg			
Total petroleum hydrocarbons	µg/kg	0.1-50		1

\* Determinands which are not in bold are not in the scope of accreditation





## BT-8 Organotin in Biota

_	
Year: 2024	Participants: 15 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 30-50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of organotin compounds in biota test materials. As we expect relatively low number of participants, this exercise can only be joined for both rounds

# **Test Materials**

The test materials cover a range of natural biota species from contaminated waters from the North Sea and/or Mediterranean. The supplied wet biota test materials are homogenised and sterilised by autoclaving. These biota test materials have been shown to be stable over a number of years when stored at room temperature.

# Determinands and Concentration Ranges

The organotin compounds to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range	Error	
		Biota	Const	Prop
Tributyltin (TBT)	µg Sn/kg	0.2-50	0.01	25.0%
Dibutyltin (DBT)	µg Sn/kg	0.1-10	0.01	25.0%
Monobutyltin (MBT)	µg Sn/kg	0.5-30	0.3	25.0%
Triphenyltin (TPhT)	µg Sn/kg	0.1-10		
Diphenyltin (DPhT)	µg Sn/kg	0.1-5		
Monophenyltin (MPhT)	µg Sn/kg	0.1-5		





# BT-9 Brominated Flame Retardants in Biota

Year: 2024	Participants: 25 laboratories expected	
Number of rounds: 2 per year	Start exercise: 1 April, 1 October	
Number of materials: 2 per round	Sample size: 30-50 g	

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of brominated flame retardants (BFRs) in biota.

# **Test Materials**

The test materials cover a range of natural unspiked biota types. Wet biota test materials are homogenised and sterilised by autoclaving. Biota test materials have been shown to be stable over a number of years when stored at room temperature.

## Determinands and concentration ranges

The BFRs to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.

- The constant and proportional error that will be used for assessment of the results.

The EQS $_{\text{biota}}$  for BDE28, BDE47, BDE99, BDE100, BDE153 and BDE154 is given as the sum for these congeners.

Determinand*	Unit	Concentration range	Error	
	1	Biota	Const	Prop
BDE28	µg/kg	0.001-1	0.01	20.0%
BDE47	µg/kg	0.05-40	0.05	15.0%
BDE49	µg/kg			
BDE66	µg/kg	0.01-10	0.02	20.0%
BDE85	µg/kg	0.01-10		
BDE99	µg/kg	0.01-10	0.02	20.0%
BDE126	µg/kg			
BDE100	µg/kg	0.005-10	0.02	25.0%
BDE153	µg/kg	0.01-2	0.02	22.5%
BDE154	µg/kg	0.001-5	0.01	20.0%
BDE183	µg/kg	0.001-1	0.03	25.0%
BDE209	µg/kg	0.01-1		
TBBP-A	µg/kg	0.01-1		
Dimethyl-TBBP-A	µg/kg			
α-HBCD	µg/kg	0.01-1		
β-HBCD	µg/kg	0.01-1		
γ-HBCD	µg/kg	0.01-1		





Determinand*	Unit	Concentration range	Error	
		Biota	Const	Prop
Total-HBCD	µg/kg	0.01-2		
BTBPE	µg/kg			
DBDPE	µg/kg			
HBBz	µg/kg			
Total lipid	%		0.4	7.5%

\* Determinands which are not in bold are not in the scope of accreditation





# BT-10 Perfluorinated Alkyl Substances (PFAS) in Biota

Year: 2024	Participants: 10 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 2 per round	Sample size: 30-50 g

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of perfluorinated alkyl substances (PFAS) in biota.

# **Test Materials**

The test materials cover a range of natural unspiked biota types. Wet biota test materials are homogenised and sterilised by autoclaving. Biota test materials have been shown to be stable over a number of years when stored at room temperature.

## Determinands and concentration ranges

The PFAS to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range Error		rror
		Biota	Const	Prop
n-PFOS	µg/kg	0.1-1000	0.15	25.0%
PFBA	µg/kg	0.01-2		
PFPeA	µg/kg	0.01-2		
PFHxA	µg/kg	0.01-2		
PFHpA	µg/kg	0.01-2		
PFOA	µg/kg	0.01-5		
PFNA	µg/kg	0.01-5	0.05	15.0%
PFDA	µg/kg	0.01-10	0.1	15.0%
PFUnDA	µg/kg	0.01-10	0.15	15.0%
PFDoA	µg/kg	0.01-5	0.1	20.0%
PFTrDA	µg/kg	0.01-5		
PFTeDA	µg/kg	0.01-5		
L-PFBS	µg/kg	0.01-10		
L-PFHxS	µg/kg	0.01-5		
L-PFHpS	µg/kg	0.01-5		
PFOSA	µg/kg	0.01-50		
PFDS	µg/kg			
PFODA	µg/kg			
Total-PFOS	µg/kg	0.1-1000	0.3	25.0%





Determinand*	Unit	Concentration Range Error		rror
		Biota	Const	Prop
GenX	µg/kg			
F-53B	µg/kg			
PFBSA	µg/kg			
PFHxSA	µg/kg			
NMeFOSAA	µg/kg			
NEtFOSAA	µg/kg			
Total-PFBS	µg/kg	0.01-10		
Total-PFHxS	µg/kg	0.01-5		
Total-PFHpS	µg/kg	0.01-5		

\* This exercise is not in the scope of accreditation.

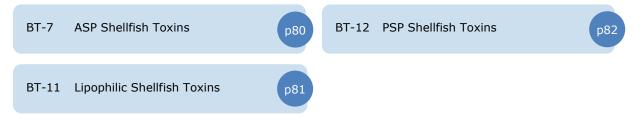






# Shellfish toxins

#### Proficiency tests in Biota



## General information

- Shellfish test materials are collected from contaminated coastal sites e.g. the North Sea, Mediterranean Sea and from Pacific Ocean.
- Depending on the type of exercise, shellfish samples are prepared from e.g. mussel, oyster, scallop, clam, or cockle tissue.
- Shellfish toxin samples are either natural contaminated or mixed with contaminated shellfish tissue where relevant.
- Shellfish tissue samples contain ±5 gram of homogenized tissue. Occasionally, extracts of shellfish tissue are distributed when relevant.
- Shellfish tissue samples are autoclaved and are stable over a number of years when stored in the freezer.
- Shellfish tissue samples are dispatched under cooled conditions.
- Reports contain all data reported under strict confidentiality, including Z-score plots and method information of the methods used.







## BT-7 ASP Shellfish Toxins

Year: 2024	Participants: 45 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 5 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of amnesic shellfish toxins (ASP) in shellfish tissue test materials.

## **Test Materials**

The supplied test materials consist of a standard solution and shellfish tissues, sufficient for one-shot analysis. Each batch of test materials is prepared in bulk, dispensed in 5 mL plastic vials and frozen at - 20°C.

The level of within and between sample homogeneity and stability is determined. All materials show to be homogeneous and stable for the purpose of the study.

### Determinands and concentration ranges

The toxins to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.

- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration Range	Error	
		Shellfish Tissue	Const	Prop
Domoic+Epidomoic	mg/kg	0.2-100	1	10.0%

\* This determinand is in the scope of accreditation.





# BT-11 Lipophilic Shellfish Toxins

Year: 2024	Participants: 40 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 5 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of organotin compounds in biota test materials. As we expect relatively low number of participants, this exercise can only be joined for both rounds

# **Test Materials**

The test materials cover a range of natural biota species from contaminated waters from the North Sea and/or Mediterranean. The supplied wet biota test materials are homogenised and sterilised by autoclaving. These biota test materials have been shown to be stable over a number of years when stored at room temperature.

# Determinands and Concentration Ranges

The lipophilic shellfish toxins to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

Determinand*	Unit	Concentration range	E	Error
			Const	Prop
Free-Okadaic-Acid	µg/kg	0.5-500	2.5	20.0%
Free-DTX1 (dinophysistoxin)	µg/kg	0.2-500	1	25.0%
Free-DTX2	µg/kg	0.5-1000	1.25	20.0%
Total-Free-OA+DTX1+DTX2	µg OA eq./kg	0.5-1000	5	20.0%
Total-Okadaic-Acid	µg/kg	0.5-500	5	20.0%
Total-DTX1	µg/kg	0.5-1000	1.5	25.0%
Total-DTX2	µg/kg	0.5-1000	3	20.0%
Total-hy-OA+DTX1+DTX2	µg OA eq./kg	0.5-1000	8	20.0%
PTX-1 (Pectenotoxin)	µg/kg	0.5-20		
PTX-2	µg/kg	0.2-50	0.3	25.0%
Total OA group and PTX group	µg OA eq./kg	0.5-1000	10	17.5%
AZA-1 (Azaspiracide)	µg/kg	0.5-1500	1.5	20.0%
AZA-2	µg/kg	0.5-500	1	20.0%
AZA-3	µg/kg	0.5-500	1.5	20.0%
AZA-total	µg AZA eq./kg	0.5-5000	5	17.5%
YTX (Yessotoxin)	mg/kg	0.01-2	0.01	22.5%
homo-YTX	mg/kg	0.5-5	0.01	22.5%
45-OH-homo-YTX	mg/kg	0.5-5	0.05	25.0%
45-OH-YTX	mg/kg	0.02-2	0.02	25.0%
YTX-total	mg YTX eq./kg	0.01-10	0.02	20.0%

\* These determinands are not in the scope of accreditation.





# BT-12 PSP Shellfish Toxins

Year: 2024	Participants: 40 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 5 ml

Participation form	Timetable	PT Scheme	Costs

This study covers the determination of brominated flame retardants (BFRs) in biota.

# **Test Materials**

The test materials cover a range of natural unspiked biota types. Wet biota test materials are homogenised and sterilised by autoclaving. Biota test materials have been shown to be stable over a number of years when stored at room temperature.

## Determinands and concentration ranges

The PSP shellfish to be determined are given in the table below. The table also shows:

- The expected concentration range for the determinands in the test materials.
- The constant and proportional error that will be used for assessment of the results.

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	E	rror
			Const	Prop
11-OH-STX (11-hydroxy- Saxitoxin)	µmol/kg			
C1	µmol/kg	0.01-5		
C1,2	µmol/kg	0.01-5	0.25	25.0%
C2	µmol/kg	0.01-1		
C3	µmol/kg			
C3,4	µmol/kg			
C4	µmol/kg			
dc-GTX1 (Gonyautoxin)	µmol/kg			
dc-GTX1,4	µmol/kg			
dc-GTX2	µmol/kg	0.01-1		
dc-GTX2,3	µmol/kg		0.1	25.0%
dc-GTX3	µmol/kg			
dc-GTX4	µmol/kg			
dc-NEO (Neo saxitoxin)	µmol/kg	0.01-2		
dc-STX	µmol/kg	0.01-5	0.05	25.0%
GTX-1	µmol/kg	0.01-1	0.15	12.5%
GTX-2	µmol/kg	0.01-10	0.2	20.0%
GTX-3	µmol/kg	0.01-2	0.04	17.5%

#### C1 N-sulfocarbamoyl toxins C1 (equal for C2, C3 and C4)





Determinand	Unit	Concentration range	Error	
			Const	Prop
GTX-4	µmol/kg	0.02-1		
GTX-5	µmol/kg	0.05-5	0.04	25.0%
GTX-6	µmol/kg			
NEO	µmol/kg	0.02-1	0.15	25.0%
STX	µmol/kg	0.05-5	0.1	20.0%
Total toxicity	µgSTXdiHCl-eq/kg	50-3000	65	25.0%
GTX-2,3	µmol/kg	0.05-10	0.01	25.0%
GTX-1,4	µmol/kg	0.01-2	0.4	25.0%

\* Determinands which are not in bold are not in the scope of accreditation







# Manure & compost

## Proficiency tests in Soil

MARSEP - International Manure and Refuse Exchange Programme p85

#### General information

- The sample types/matrices are chosen to represent the materials which you normally analyse.
- The samples included in this programme are compost, organic fertilizer and sewage sludge.
- The samples are dried, milled and sieved to <0.5 mm.
- The homogeneity of each batch of samples is tested on a selection of parameters.
- The dried samples are stable over a number of years when stored at room temperature.
- You analyse the test materials according to your normal validated methods and procedures for those elements and parameters you are interested in.
- Your results are processed at WEPAL-QUASIMEME and published every three months under confidential code names.
- The reports contain all data, statistical evaluation including Z-score plots and method information about the method used.
- Your reports are available within three weeks after deadline
- New determinands can be added on request.





# MARSEP International Manure and Refuse Sample Exchange Programme

Year: 2024	Participants: 40 laboratories expected
Number of rounds: 4 per year	Start exercise: 1 January, 1 April, 1 July, 1 October
Number of materials: 4 per round	Sample size: 20 g

Participation form	Timetable	PT Scheme	Costs
--------------------	-----------	-----------	-------

#### Determinands

Concentration ranges and constant and proportional errors are available in the determinand list on the participants website.

Determinand group	Determinand*
Inorganic Chemical Composition	Ag; Al; As; B; Ba; Be; Bi; TC =totalC (org+inorg); TOC =total org.C; OC = org.C; Ca; Cd; Ce; Cl; Co; Cr; Cu; F; Fe; Ga; Hg; I; K; Li; Mg; Mn; Mo; N; N- NH4 (as N); N-NO3 (as N); Na; Ni; P; Pb; S; S-SO4 (as S); Sb; Se; Si; Sn; Sr; Th; Ti; Tl; U; V; Zn;
Other determinations	AOX; <b>loss-on-ignition</b> ; residu-on-ignition; COD; mineral oil; <b>dry weight</b> ;

\*Determinands which are not in bold are not in the scope of accreditation.







# Biomass

## Proficiency tests in Biomass

BIMEP - Biomass Exchange Programme

## General information

- The sample types/matrices are chosen to represent the materials which you normally analyse.
- The sample types are plant materials, woods and waste (sewage sludge).
- The samples are dried, milled and sieved to <0.5 mm and are stable for over a number of years when stored at room temperature.
- The homogeneity of each batch of samples is tested on a selection of parameters.

p87

- You analyse the test materials according to your normal validated methods and procedures for those elements and parameters you are interested in.
- Your results are processed at WEPAL-QUASIMEME and published every three months under confidential code names.
- The reports contain all data, statistical evaluation including Z-score plots and method information about the method used.
- Your reports are available within three weeks after the submission deadline.
- New determinands can be added on request.







# BIMEP Biomass Exchange Programme

Year: 2024	Participants: 12 laboratories expected
Number of rounds: 2 per year	Start exercise: 1 April, 1 October
Number of materials: 4 per round	Sample size: 40 g

Participation form	Timetable	PT Scheme	Costs

#### Determinands

Concentration ranges and constant and proportional errors are available in the determinand list on the participants website.

Determinan	d group	Determinand*	
General Ana	lysis	Ash; Moisture; Calorific Value (gross); Volatile Matter;	
Elementary	Analysis	Carbon (C); Hydrogen (H); Nitrogen (N); Oxygen (O); Cl; S;	
Water Elements	Soluble	Cl; K; Na;	
Major Eleme	ents	Al; Ca; Fe; K; Mg; Na; P; Si;	
Minor Eleme	ents	As; Ba; Be; Cd; Co; Cr; Cu; F; Hg; Mn; Mo; Ni; Pb; Sb; Se; Sn; Te; Ti; Tl; V; Zn;	

\*Determinands which are not in bold are not in the scope of accreditation.

Concentration ranges and constant and proportional errors are available in the determinand list in the participants website.

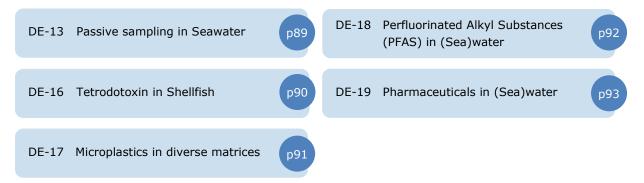






# Special (development) exercises

## Proficiency tests in development



## General information

For emerging pollutants/determinands or in case of poor comparability among laboratories analytical methodology may need to be refined. On request WEPAL-QUASIMEME offers development exercises to develop and improve the methodology, that may result in a new proficiency test scheme on a regular base.

Workshops that focus on specific problems and to discuss achievements or corrective actions can be part of these development exercises.





### DE-13 Passive sampling in Seawater

Year: 2024	Participants: 15 laboratories expected
Number of rounds: 1 per year	Start exercise: October
Number of materials: 2 per round	Sample size: samplers

Participation form	Timetable	PT Scheme	Costs

\* This exercise is not in the scope of accreditation.

Passive sampling will become an important procedure to measure concentrations of determinands, e.g. HCB, HCBD, PCBs, PAHs and Brominated flame retardants in seawater. Therefore, a development exercise will be offered in the new proficiency testing scheme.

The development exercise will be conducted in co-operation with Foppe Smedes (Deltares/Masaryk University) and Kees Booij (NIOZ/PaSOC). Following your subscription, an inventory will be held with respect to internal standards which are used in your laboratory and may conflict with PRC's to be used within the development exercise itself.





# DE-16 Tetrodotoxin in Shellfish

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 1 per year	Start exercise: October
Number of materials: 2 per round	Sample size: 40 g

Participation form	Timetable	PT Scheme	Costs
--------------------	-----------	-----------	-------

\* This exercise is not in the scope of accreditation.

This study is coordinated by Mirjam Klijnstra MSc, BU Contaminants & Toxins, WFSR, Wageningen the Netherlands

More specific information will be available on our webpage and will be communicated to participants of the shellfish toxin exercises.





# DE-17 Microplastics in diverse matrices

Year: 2024	Participants: 50 laboratories expected
Number of rounds: 1 per year	Start exercise: April
Number of materials: 3 per round	Sample size: diverse

Participation form	Timetable	PT Scheme	Costs
--------------------	-----------	-----------	-------

 $\ast$  This exercise is not in the scope of accreditation.





# DE-18 Perfluorinated Alkyl Substances (PFAS) in (Sea)water

Year: 2024	Participants: 20 laboratories expected
Number of rounds: 1 per year	Start exercise: October
Number of materials: 3 per round	Sample size: 500 ml

Participation form	Timetable	PT Scheme	Costs
·			

\* This exercise is not in the scope of accreditation.

The following PFAS determinants will at least be added to estuarine and seawater samples: PFOS, PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFDA, PFDA, PFDoA, PFDoA, PFTrDA, PFTeDA, L-PFBS, L-PFHxS, L-PFHpS, PFOSA, PFDS, PFODA, GenX, F-53B, PFBSA, PFHxSA, NMeFOSAA and NEtFOSAA. No guarantee that all individual PFAS will be spiked. This will be due to availability of the certified reference materials. A final decision about concentration levels will be discussed at the SAB meeting, but a first indication will be 0.01-10 ng/L per individual PFAS.





# DE-19 Pharmaceuticals in (Sea)water

Year: 2024	Participants: 15 laboratories expected
Number of rounds: 1 per year	Start exercise: October
Number of materials: 3 per round	Sample size: 1000 ml

Participation form Timetable PT Scheme Costs
--

\* This exercise is not in the scope of accreditation.

At least Diclofenac, Carbamazepine, Ibuprofen, Azithromycin, Clarithromycin and Erythromycin will be spiked to estuarine and seawater samples, when individual reference materials will be available for these determinands.







## General information

Samples from our proficiency test schemes are offered to laboratories as reference material. These reference materials can be used for method development or as reference samples for internal quality control. The reference materials are delivered with a certificate of analysis or summary statistics. These documents are based on results from the WEPAL-QUASIMEME proficiency testing programmes. The certificates provide information about:

- the consensus value: the mean value of the dataset obtained from the PT, calculated with robust statistics
- the standard deviation and coefficient of variation
- number of results used to calculate the consensus value
- median and MAD (Median of Absolute Deviation)

An overview of the available reference materials and how to order is given in our webshop. On request, WEPAL-QUASIMEME can prepare custom made reference materials.







# Organisation and Structure WEPAL-QUASIMEME

Roles and responsibilities of the WEPAL-QUASIMEME team are outlined in the table below. The contact details for the WEPAL-QUASIMEME Project Office are given on the first page of this document.

Name	Role	Responsibilities
Mrs. Laura Buijse	Manager WEPAL- QUASIMEME,	Manager of the WEPAL-QUASIMEME team
Mrs. Winnie van Vark	Coordinator WEPAL	Data assessment and statistics WEPAL
Mr Wim Cofino	Project advisor	Scientific responsibility of the QUASIMEME Laboratory Performance studies. Statistics
Mr. Steven Crum	Coordinator QUASIMEME	Coordination and organisation of the QUASIMEME Laboratory Performance studies Preparation of Aquatic test materials Homogeneity and stability testing Aquatic samples Test material dispatch QUASIMEME Data assessment and statistics QUASIMEME Dispatch of QUASIMEME samples
Mr. Jan Groenwold	Project assistant	Database and statistics WEPAL-QUASIMEME
Mr. Pieter Hazenberg	Quality Assurance Officer	Quality Assurance
Mrs. Ilona van de Berg	Project assistant	QUASIMEME Front Office (secretariat and subscription) Communications QUASIMEME Help desk QUASIMEME
Mrs. Minke van Veldhuizen	Project assistant	WEPAL Front Office (secretariat and subscription) WEPAL-QUASIMEME Finances Help desk WEPAL
Mrs. Laura Buijse	Project assistant	Preparation of aquatic test materials and homogeneity testing sediment and biota for organic parameters
Mrs. Arrienne Matser	Project assistant	Preparation of aquatic test materials and homogeneity testing sediment and biota for organic parameters
Mr. Dennis Penthum	Project assistant	Preparation of test materials; Processing of submitted data WEPAL; Dispatch of samples
Mr. Fred Bransen	Project assistant	Preparation of test materials
Mrs. Andrea Sneekes	Project advisor	Scientific advise, communications, website, LinkedIn
Mrs. Sabine Schnabel	Project advisor	Statistics
Mr. Maikel Verouden	Project advisor	Statistics
Mr. Patrick Roose	Project advisor	Scientific advise and chairman Scientific Advisory Board





# Overview of activities

WEPAL-QUASIMEME has a number of collaborators who prepare and provide test materials for the Laboratory Performance studies, and who analyse these test materials for homogeneity and, where appropriate, stability. All collaborators are experts in their particular field and operate to a traceable standard, which can be audited. This may include:

- Accreditation to a standard acceptable to e.g. ISO17025, ISO17043, ISO9000 series.
- National reference laboratory.
- Documented evidence of the quality of the test materials provided.

When subcontracting occurs, it is placed with a competent subcontractor and WEPAL-QUASIMEME is responsible for this work. Part of the work is done by WEPAL-QUASIMEME itself. A list of all WEPAL-QUASIMEME collaborators and their role in the provision and testing of materials for the proficiency test studies is available here.

Activity In-home	Organisation	Contact person
<ul> <li>Coordination aquatic (freshwater, wastewater and seawater), marine sediment, biota and shellfish toxins PT- scheme</li> <li>Preparation aqueous test materials</li> <li>Homogeneity testing chlorophyll</li> <li>Assessment of homogeneity tests</li> </ul>	Wageningen University WEPAL-QUASIMEME Bornsesteeg 10 6721 NG Bennekom The Netherlands	Steven Crum steven.crum@wur.nl
<ul> <li>Coordination, preparation and homogeneity testing soil, plant, freshwater sediment, manure &amp; compost and biomass PT- scheme</li> <li>Assessment of homogeneity tests</li> </ul>	Wageningen University WEPAL-QUASIMEME Bornsesteeg 10 6721 NG Bennekom The Netherlands	Winnie van Vark winnie.vanvark@wur.nl

Activity Outsourced	Organisation	Contact person
Preparation and	Royal Belgian Institute of	Marc Knockaert
homogeneity testing of	Natural Sciences,	
nutrient, DOC and Ocean	Directorate Natural	mknockaert@naturalsciences.be
acidification test	Environment,	
materials	Marine Environment	
	3e & 23e Linieregimentsplein	
	8400 Oostende	
	Belgium	
Preparation of biological	Wageningen Marine Research	Andrea Sneekes
test materials and	P.O. Box 68	
homogeneity testing	1970 AB Ijmuiden	andrea.sneekes@wur.nl
organic contaminants in	The Netherlands	
biota		





Activity Outsourced	Organisation	Contact person
<ul> <li>Preparation and homogeneity testing of shellfish toxins testmaterials (ASP, DSP and PSP)</li> </ul>	CEFAS The Nothe, Barrack road BT48UB Weymouth Dorset United Kingdom	Andrew Turner andrew.turner@cefas.co.uk
<ul> <li>Homogeneity testing of sediment and biota samples on metals</li> </ul>	Wageningen University Department of Environmental Sciences CBLB Soil chemistry Droevendaalsesteeg 3 6708 PB Wageningen The Netherlands	Anne Roepert anne.roepert@wur.nl
<ul> <li>Preparation and homogeneity testing of tetrodotoxin shellfish toxin testmaterials</li> </ul>	Wageningen Food Safety Research, BU Contaminants & Toxins Akkermaalsbos 2 6708WB Wageningen The Netherlands	Mirjam Klijnstra <u>mirjam.klijnstra@wur.nl</u>
<ul> <li>Preparation and homogeneity testing of sediment and biota samples on microplastics</li> </ul>	Dept. of Environment and Health, Faculty of Science, Vrije Universiteit De Boelelaan 1108 1081HV Amsterdam The Netherlands	Ike van der Veen ike.van.der.veen@vu.nl
Preparation and homogeneity testing of tablet samples on microplastics	NIVA Økernveien 94 0579 Oslo Norway	Bert van Bavel <u>bert.vanbavel@niva.no</u>





# The QUASIMEME Scientific Advisory Board

The QUASIMEME Scientific Advisory Board gives advice on the implementation of the scientific programme to WEPAL-QUASIMEME and oversees the data assessments and reports on the results of the Laboratory Performance (LP) studies. The Scientific Advisory Board consist of experts in the field of QA and the assessment of LP studies. The members have experience in the design and operation of LP studies and/or environmental measurements in matrices related to the marine environment. Membership of the SAB is confirmed annually. The membership of the SAB will be sufficient in number and breadth of experience to adequately cover the areas included in the QUASIMEME LP studies. The SAB may recommend specialists to the chairman to be invited to contribute to specific QUASIMEME activities as required.

Terms of Reference of the QUASIMEME SAB were agreed at the newly formed SAB Board Meeting, 26-27 September 2013 and are confirmed annually. The SAB will meet at least annually to advise and assist the QUASIMEME Project Office on:

- 1. The design of the QUASIMEME LP studies and provision of test materials and protocols.
- 2. The assessment of the LP studies and study reports.
- 3. The preparation of documentation, both printed and electronic.
- 4. Recommendations of changes in structure or content of the LP studies.
- 5. A proposed work programme for future LP studies.
- 6. The SAB will review and make recommendations to the QUASIMEME Project Office on the composition and breadth of expertise which is required to maintain the objective assessment of the programme and the results of the participants' studies. Advise on matters relating to the Quality Assurance and Quality Control requirements for the national and international marine monitoring programmes and to provide links with these programmes.
- 7. Provide information and advice on the list of determinands required for the national and international monitoring programmes, the matrices and the concentration ranges. Where lists of studies in the current LP studies are being revised, the Board shall indicate the relative priority of the studies to be undertaken.
- 8. On the level of performance required for specific monitoring programmes in terms of precision and bias for each determinand-matrix combination.
- 9. Review and revise the terms of reference of the Advisory Board, when necessary.
- 10. Advise QUASIMEME on activities to meet future needs.

QUASIMEME Scientific Advisory Board consists of representatives from organisations to which QUASIMEME participants submit environmental monitoring data. The organisations represented are responsible for nominating their member of the QUASIMEME Scientific Advisory Board.

- 1. A representative from the Oslo Commission (OSPAR) to maintain communication with OSPAR, particularly in relation to the QA requirements of the Joint Assessment and Monitoring Programme (JAMP).
- 2. A representative to maintain communication with the Helsinki Commission (HELCOM), particularly in relation to the QA requirements of the Baltic Monitoring Programme (BMP) and the Coastal Monitoring Programme (CMP).
- 3. A representative to maintain communication with the International Council for the Exploration of the Sea (ICES).
- 4. Representatives of national monitoring programmes. Two representatives from national monitoring programmes will be invited based on the national levels of participation in QUASIMEME. Representatives of other national monitoring programmes may request to attend.
- 5. The QUASIMEME Project Advisor.
- 6. A representative to maintain communication with the European Environmental Agency.
- 7. A representative to maintain communication with the Arctic Monitoring and Assessment Programme (AMAP).





Name	Institute
Dr. Patrick Roose (chairman) patrick.roose@naturalsciences.be	Royal Belgian Institute of Natural Sciences
Em. Prof. Dr. Wim Cofino (QUASIMEME Project Advisor) wim.cofino@wur.nl	WEPAL-QUASIMEME, Wageningen University
Pamela Walsham Msc. (UK NMCAG) Pamela.walsham@gov.scot	Marine Scotland Science
Dr. Martin Mork Larsen (OSPAR, ICES) mml@dmu.dk	Aarhus Universitet
Dr. Koen Parmentier (ICES) kparmentier@naturalsciences.be	Royal Belgian Institute of Natural Sciences
Dr. Michiel Kotterman (ICES) michiel.kotterman@wur.nl	Wageningen Marine Research Foundation Wageningen Research
Dr. Nicole Bandow <u>Nicole.bandow@uba.de</u>	Umweltbundesamt
Mrs. Winnie van Vark winnie.vanvark@wur.nl	WEPAL-QUASIMEME Wageningen University
Ing. Steven Crum steven.crum@wur.nl	WEPAL-QUASIMEME Wageningen University
Ing. Andrea Sneekes andrea.sneekes@wur.nl	Wageningen Marine Research Foundation Wageningen Research
Dr. Andrew Turner andrew.turner@cefas.co.uk	CEFAS
Em. Prof. Dr. Jacob de Boer jacob.de.boer@vu.nl	Vrije Universiteit
Dr. Ike van der Veen <u>Ike.van.der.veen@vu.nl</u>	Vrije Universiteit
Vacancy (HELCOM)	Helsinki Commission
Vacancy (AMAP)	Institute of Marine Research