

QUASIMEME Interlaboratory Study on the Analysis of Chlorinated Paraffins in Environmental Matrices

Chlorinated paraffins (CPs), also known as Polychlorinated alkanes (PCAs), are complex mixtures of chlorinated n-alkanes with carbon chain lengths of 10 to 30 and a chlorination degree between 30% and 70% by mass. Characterization of CPs is performed by their alkane chain lengths. They are divided in three groups: short-chain (C₁₀₋₁₃) (SCCP), medium-chain (C₁₄₋₁₇) (MCCP) and long chain (C₁₈₋₃₂) (LCCP) CPs.

CPs are used in several industrial applications like flame-retardants in the rubber industry, as high temperature lubricants and cutting fluids in the metalworking industry and as additives in liquids, paints and textiles. The analysis of CPs is highly complicated. There are tens of thousands of congeners which make separation by GC and even by GCxGC hardly possible. Alternative methods are scarce and may suffer from false positive data. The data reported on CPs to now, deduce very high uncertainties (100% or more).

Chlorinated paraffins are currently of utmost importance to the environmental analyst. They are being produced in high volumes, are under discussion in the United Nations Environmental Programme (UNEP) to ascertain whether or not they will be defined as persistent organic pollutant (POP). CPs are a mandatory determinant in the European Water Framework Directive. This said however, the analysis is far from validation, as the paraffins (CPs) are extremely complex. In March this year, QUASIMEME organised a workshop on the analysis of chlorinated paraffines in Ostend, Belgium. A number of critical issues in the analysis of these CPs were discussed. It was generally agreed that there was a clear need for an interlaboratory study (accompanied with expert comments) to be designed in a step-wise way with meetings being organised for participants in between each step. Quasimeme welcomes your participation in such a study.

Design of the Study

The study will consist of four steps. Each step will be at the participants pace. If corrective actions or a repeat of one step is required this will be accommodated. It is hoped that on completion of the entire study the CPs could be included in the routine proficiency testing scheme of QUASIMEME (www.quasimeme.org).

Study Design

Round 1

The first round of the study would focus on the analysis of CPs in a solution with an undisclosed concentration. Standards will be provided.

Materials:

- *Standard solution.* An ampoule with a standard solution containing CPs, with a known concentration.
- *Unknown solution.* An ampoule with a standard solution containing CPs, with an undisclosed concentration.

The results will be documented in a report. This report will describe the preparation of the materials, the methods of analysis used and their results. Advice will also be documented to the participating laboratories together with conclusions.

Round 2

The second round of the study would focus on the analysis of CPs in a clean extract of a sediment or fish. The extracts will be analysed and quantified with a known standard solution.

Materials:

- *Standard solution.* An ampoule with a standard solution containing CPs, with a known concentration.
- *Clean fish or sediment extract.* An ampoule with a cleaned fish or sediment extract.

The results will be documented in a report. This report will describe the preparation of the materials, the methods of analysis used and their results. Advice will also be documented to the participating laboratories together with conclusions.

Round 3

The third round of the study would focus on the analysis of CPs in clean and uncleaned extracts of a sediment or fish. The ampouled extracts will be analysed and quantified with a known standard solution.

Materials:

- *Standard solution.* An ampoule with a standard solution containing CPs, with a known concentration.
- *Clean fish extract.* An ampoule with a cleaned fish or sediment extract.
- *Uncleaned extract.* An ampoule with a fish or sediment extract, which needs to be cleaned by the labs prior to analyzing.

Round 4

The fourth round of the study focuses on the entire method: extraction, clean up and analysis of environmental samples. A fish sample and a sediment sample will be analysed and quantified with a known standard solution.

Materials:

- *Standard solution*. An ampoule with a standard solution containing CPs, with a known concentration.
- *Fish sample*
- *Sediment sample*

The results will be documented in a report. This report will describe the preparation of the materials, the methods of analysis used and their results. Advice will also be documented to the participating laboratories together with conclusions.

Evaluation meeting

An evaluation meeting will be planned after the third round.

Time Table

The tentative scheme for this study is:

Round 1

1 November 2010	Announcement and invitation
15 February 2010	Deadline for registration
1 April 2011	Shipment of samples
1 June 2011	Deadline for returning results
15 August 2011	Draft report
1 October 2011	Final report

Round 2

1 September 2011	Announcement and invitation
15 October 2011	Deadline for registration
15 November 2011	Shipment of samples
15 February 2012	Deadline for returning results
1 May 2012	Draft report
15 July 2012	Final report

Round 3

1 September 2012	Announcement and invitation
15 October 2012	Deadline for registration
15 November 2012	Shipment of samples
15 February 2013	Deadline for returning results

1 May 2013	Draft report
15 July 2013	Final report
November 2013	Evaluation meeting

Round 4

1 September 2013	Announcement and invitation
15 October 2013	Deadline for registration
15 November 2013	Shipment of samples
15 February 2014	Deadline for returning results
1 May 2014	Draft report
15 July 2014	Final report

Coordination

This study will be coordinated by Ms. Ike van der Veen and Prof.dr. Jacob de Boer, IVM, VU University, Amsterdam, The Netherlands. Both coordinators are highly experienced in the organisation of large, international interlaboratory studies.

Participation Fee

The fee for participation in this study will be 880 Euro per round. In case a pre-payment is made for all four rounds, the fee will be 3200 Euro in total for all four rounds. The samples will be dispatched after receipt of the fee.

Registration

Participants should register before 15 February 2011. To register, please send an email to the Quasimeme office, mentioning confirmation CP ILS participation with your full postal address, tel. no. and email. Suggestions with regard to the design of the study and the type of test materials are also welcome and can be added to your email. Upon receipt of your email you will receive a confirmation of your participation and an invoice for the first round (or for all 4 rounds at reduced fee, if so indicated).

QUASIMEME and co-organisers

QUASIMEME (Quality Assurance of Information in Marine Environmental Monitoring in Europe) operates a series of Proficiency Testing Studies for institutes making chemical measurements worldwide. As part of the improvement programme, QUASIMEME co-operates with centres of excellence to provide workshops for discussion, and “hands on” experience to compliment the development programmes in the Laboratory Performance Studies.

The Institute for Environmental Studies (IVM) of the VU University in Amsterdam, The Netherlands, acts as a centre of excellence for QUASIMEME. It contributes to

biological test material testing for proficiency tests on organic contaminants. In addition, scientific advice is given to the annual QUASIMEME programmes through the Scientific Assessment Group. IVM assists in organising workshops on specific analytical topics and in the organisation of specific interlaboratory studies (learning exercises). IVM combines knowledge on analytical chemistry and toxicology to address a broad range of environmental issues, with a focus on contaminants. More information can be found at www.vu.nl/ivm.