

# Preparation of fish material for interlaboratory study on PFCs

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## Introduction

Institute for Environmental Studies, Vrije Universiteit (IVM) has requested Wageningen IMARES for the preparation of fish material for use in inter-laboratory performance study on analysis of perfluorinated compounds (PFCs). It was requested that the material should be prepared from fillet of flounder caught in Western Scheldt and packed in glass jars. This document provides report on the material preparation.

## Assignment

In accordance with the order 07.029, the following activities were performed by Wageningen IMARES:

- Purchase of packing material
- Blank testing of packing material:
  - 3 blanks on glass jars and lids
  - 3 blanks on glass jars only
  - 3 blanks on lids only
  - 2 blanks on solvent used
- Purchase of flounder caught in Western Scheldt
- Filleting of fish
- Mincing of fillets and homogenization (for at least 1 hour)
- Packing the material into glass jars
- Sterilization of glass jars
- Reporting on material preparation.

## Quality Assurance

IMARES utilises an ISO 9001:2000 certified quality management system (certificate number: 08602-2004-AQ-ROT-RvA). This certificate is valid until 15 December 2009. The organisation has been certified since 27 February 2001. The certification was issued by DNV Certification B.V. The last certification inspection was held the 16-22 of May 2007. Furthermore, the chemical laboratory of the Environmental Division has NEN-AND-ISO/IEC 17025:2000 accreditation for test laboratories with number L097. This accreditation is valid until 27 March 2009 and was first issued on 27 March 1997. Accreditation was granted by the Council for Accreditation, with the last inspection being held on the 12<sup>th</sup> of June 2007.

# Materials and Methods

## *Blank testing of the packing material*

In order to test the packing material for blanks, three glass jars and three lids were thoroughly rinsed with 15 mL of methanol. The rinsing methanol was collected, then concentrated to 0.5 mL and the levels of PFCs were measured in the concentrate by LC-MS.

In addition, 24-hour exposure of closed glass jar to methanol was evaluated. Three glass jars were filled with methanol, closed by lid and shaken for 24 hours by shaker. One of the three glass jars was placed into the shaker upside down to ensure that methanol comes into contact with the lid for 24-hour. After the exposure, methanol from each jar was concentrated to 0.5 mL and the levels of PFCs were measured in the concentrate by LC-MS.

To test the PFC blank in solvent used, 15 mL of methanol was concentrated to 0.5 mL and the levels of PFCs were measured in the concentrate by LC-MS. The solvent blank test was performed in duplicate.

## *LC-MS measurement*

Each concentrate for blank tests was analysed by LC-MS twice using different settings for the sulfonates and the carboxylic acids. Next to the blanks a low level standard was analysed to provide retention time characteristic of the PFCs and one-point calibration for reporting quantitative data if any PFCs would be detected.

## *Purchase of flounder*

About 120 kg of flounder originating from the Western Scheldt (51°28'N – 03°30'E) was purchased (ship BR7) on March 21, 2007. After delivery to Wageningen IMARES, the fish was stored frozen at -20°C till the preparation.

## *Material preparation*

Filleting of the fish was performed on April 3, 2007. After the filleting, the material was minced using a mincer (Finis Machinefabriek, Uft) in combination with a Fryma mill equipped with toothed rotary knives (Fryma Maschinen AG, Rheinfelden, Switzerland) to a final size of 3.5 mm<sup>2</sup>. Subsequently, ca 25 kg sample was homogenized for 1 hour, after adding 0.02% butylhydroxytoluene(BHT), in a water-cooled Stephan cutter (Stephan Machines, Almelo, The Netherlands, type UMM/SK25, made in 1979). Approximately 100 g of homogenized material was placed into the glass bottle and frozen at -20°C without sterilization. The rest of the material was filled into the glass jars and closed with the lid (ca. 60 g of material per jar). Special attention was paid to fill the glass jars to the top to minimize head space area. Starting with jar number 10, every thirty jar was marked to be used for the homogeneity test. The jars were then sterilised in a Muvero-Mat sterilizer (type 90E) for 45 minutes at 122 °C (pressure 1.4 bar, heating-time: 90 minutes, cooling time: 20 minutes). Each glass jar was then packed into the non-transparent (black) plastic back, placed into the paper box and dispatched to IVM.

## Results and discussion

### *Blank tests*

Although all methanol concentrates from the lid test and from the 24-hour exposure test were slightly clouded, no PFCs have been detected in the jars and lids (<2.5 ng/jar). It was concluded that the jars and lids are suitable packaging of the material and will not cause any contamination of the material by PFCs.

### *Material preparation*

- One glass bottle containing ca. 100 g of homogenized, not sterilized and frozen material was prepared and delivered to IVM
- 301 glass jars of homogenized and sterilized material including marked jars for homogeneity testing were prepared and delivered to IVM.

## Conclusions

301 glass jars of homogenized and sterilized flounder from the Western Scheldt has been prepared for and delivered to the Institute for Environmental Studies (IVM), Free University, Amsterdam to be used in inter-laboratory performance study on analysis of perfluorinated compounds (PFCs). In addition, one glass bottle containing ca. 100 g of homogenized, not sterilized and frozen material was prepared and delivered. The packing material used (glass jars and lids) was tested for presence of PFCs and no PFCs were detected (<2.5 ng/jar).

# Referees and Authors

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This report has been professionally prepared by Wageningen IMARES. The scientific validity of this report has been internally tested and verified by another researcher and evaluated by the Scientific Team at Wageningen IMARES.

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