



***Metrology for monitoring endocrine
disrupting compounds under the Water
Framework Directive_EDC-WFD***

CONCLUSION

THE OBJECTIVES

1. **Optimize and validate traceable aqueous reference Mass Spectrometry-based methods for the analysis of 5 estrogenic compounds** prioritizing $17\beta\text{E}2$, $17\alpha\text{EE}2$, and E1 in **whole water samples** at environmental quality standard (EQS) levels. **Methods will have limit of quantification (LOQ) not exceeding 30% EQS with a measurement uncertainty of $\leq 50\%$ at EQS**

Partners developed and validated Ms and Effect based methods : deliverables :

- **D1 : Report on the comparison of sample preparation techniques for estrogens partitioning in whole water and recommendations on the most appropriate methods**
- **D2 : Report and recommendations to improve the short and long-term stability of selected estrogens in complex water matrix**
- **D3 : SOP/method description for the most promising MS-based measurement method(s) of estrogens in whole water samples compatible with the requirements of the QA/QC Directive**

2. **Develop production methods for aqueous reference materials (RM)**, which are as close as possible to real water samples, with proven homogeneity, short- and long-term stability

Two reference materials : pure compounds and kit

- **D5 : Recommended production methods for aqueous reference materials, which are as close as possible to real water samples, with proven homogeneity and short- and long-term stability**

THE OBJECTIVES

3. **Improve the comparability of estrogen measurements with selected Effect-Based Methods (EBM)** in whole water samples at EQS level. Methods will have been correctly calibrated and information on uncertainty will be provided

Partners developed and validated Effect based methods :

- **D4 : SOP/method description for the most promising Effect-based measurement method(s) of estrogens in whole water samples compatible with the requirements of the QA/QC Directive**

4. **Organize and perform an interlaboratory comparison (ILC)** to demonstrate the performance of the developed methods using the reference material (RM) for the selected estrogen substances

Interlaboratory comparison

- **D6 : Interlaboratory comparison of chemical MS-based methods and Effect-Based Methods (EBMs bioassays) for estrogens measurements**

5. **Contribute to the work of key European and international standardization organizations e.g. CEN TC 230 and ISO TC 14**

Standard draft

- **D7 : Standard draft provided to CEN/TC230 "Water quality" and ISO/TC 147 " Water analysis"**

ALL DELIVERABLES :

<https://projects.lne.eu/jrp-edc-wfd/>

A LNE Joint Research Project

Home

Project Overview

Workpackages

Project Events

Partners

Tools / publications

Member Space

EDC WFD

Metrology for monitoring endocrine disrupting compounds under the Water Framework Directive



Natural and pharmaceutical estrogens are key Endocrine Disrupting Chemicals (EDC) which are monitored differently depending on the country, and for which standardised reference methods are currently not available. The overall objective of the project is to develop reliable and harmonised measurement methods for estrogens, to comply with the Water Framework Directive

EMPIR  

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States

PROJECT OUTCOMES

No „real“ water sample available which can be provided to all the project partners which fits the EQS levels given by the EU-WFD (long term stability for the material is needed e.g., 3 years of project time)

Synthetic real water matrix with distinct composition and defined estrogen level

This RM kit can be adapted to other analytes

Method development: different methods due to the synergy partnership:

- SPE disk is strongly recommended especially for high SPM content ($> 50 \text{ mg L}^{-1}$)
- Purification of extracts is recommended even for LC-MS (ion suppression)
- IDMS is needed (preferably for each analyte an isotopically labelled IST)
- Uncertainty estimation associated to EBMs is also possible/evaluated according ISO 11352

PROJECT OUTCOMES

- Both LC- and GC-MS/MS reached the old EQS level for all five estrogens while LC-MS/MS reached also the new EQS level for EE2*
- LC-HRMS is not sufficiently sensitive. For GC-MS/MS and GC-HRMS comparable sensitivity could be reached (A question of budget)*

Method validation: same samples and experimental design which is fit for purpose for MS-based and EB-methods

Standardisation:

- Draft is under review for chemical measurements
- Proposition of ISO EBM standard

*this is only valid for equipment which was used within the project

PROJECT OUTCOMES

ILC:

- The general accurate application of the reconstitution step positively influenced the very good agreement of the measurements with the reference concentration of the tested samples
- For EBM need new interlaboratory trails
- The EDC-WFD ILC adds useful information about application of EBMs, in a future perspective of implementation within the framework of environmental waters monitoring activities in Europe, together with chemical measurement

AKNOWLEDGMENTS



“This project 18NRM01 EDC-WFD has received funding from the EMPIR programme co-financed by the Participating States and from the European Union’s Horizon 2020 research and innovation programme”

THANKS

Coordinator 1 : Sophie LARDY-FONTAN

Advisory group

- Carere Mario, *Istituto Superiore di Sanità*
- Marina RICCI, *Joint Research Centre (JRC)*,
- Ulrich Borchers, *IWW Water Centre*
- PERCEVAL Olivier, *Office français de la biodiversité*
- STAUB Pierre-François, *Office français de la biodiversité*

All the stakeholders

CEN, Ministère environnement français, ASLAE (french laboratories association), French standardisation body AFNOR (Arnaud), JRC, AFB (french agency of biodiversity), HELCOM, SQUAREF, Centre of economic development, Transport and environment of southeast Finland, ARPA(s), ANSES, Umwelt Bundesamt, Ministry of environment Finland, Ministry of environment and spatial planning Slovenia, Slovenian association for water protection, Institute of Environmental Technology Berlin, Central Waste water Treatment Plant Domzale-Kamnik



E. HEATH, A. KOVACIC



T. GÖKCEN, I. UN



P. De ZORZI, M. POTALIVO, C. ASCENZI, E. CALABRETTA, S. BARBIZZI, G. MOLTEDO



S. LARDY-FONTAN, V. LE DIOURON, C. FALLOT,
B. LALERE & our LC/MS² (critical member of the team)



K. LE MENACH, P. PARDON, H. BUDZINSKI



C. PIECHOTTA, L. STEINHÄUSER, T. WESTPHALEN,
U.-A. KLYK-SEITZ, K. KAMINSKI, S. KLUGE



T. NÄYKKI, J. VIIDANOJA

