EDC WFD

Metrology for monitoring endocrine disrupting compounds under the Water Framework Directive

EMPIR EURAMET

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

EDC WFD

AIM

Natural and synthetic 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.

⇒ Main Objective: Develop reliable and harmonized measurement methods for estrogens, to comply with the WFD requirements

⇒Outcomes: to be disseminated to CEN/ TC 230 and ISO/ TC 147 to be fed into the documentary standards they develop

Start date: 1st September 2019

Duration: 36 months + 6 months extension due to Covid-19 pandemy

Budget: 800 K€

OBJECTIVES

- *The overall objective of this project is*

- ✓ *to develop **traceable measurement methods** for endocrine disrupting chemicals, with a specific focus **on three estrogens of the first watch list (17-beta-estradiol (17βE2), 17-alpha-ethinylestradiol (17αEE2), and estrone (E1)**). Estrogens 17-alpha-estradiol (17αE2) and estriol (E3) will be included to demonstrate the reliability of the developed methods, and*

- ✓ *to support the requirements of Directive 2013/39/EC, Directive 2009/90/EC and Commission Implementation Decision (EU) 2018/840,*

- ✓ *hence **improving the comparability and compatibility of measurement results within Europe.***

The specific objectives of the project are to:

1. **Optimize and validate traceable aqueous reference Mass Spectrometry-based methods for the analysis of 5 estrogenic** compounds prioritizing 3 selected estrogenic compounds 17βE2, 17αEE2, 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 ≤50% at EQS.**

2. **Evaluate the interaction and partitioning** of 5 estrogenic compounds prioritizing 3 selected estrogenic compounds 17βE2, 17αEE2, and E1 **between water samples and suspended particulate matter (SPM**) and the **capability of developed methods to address the different fractions of matrix** (whole water and dissolved concentrations of estrogens).

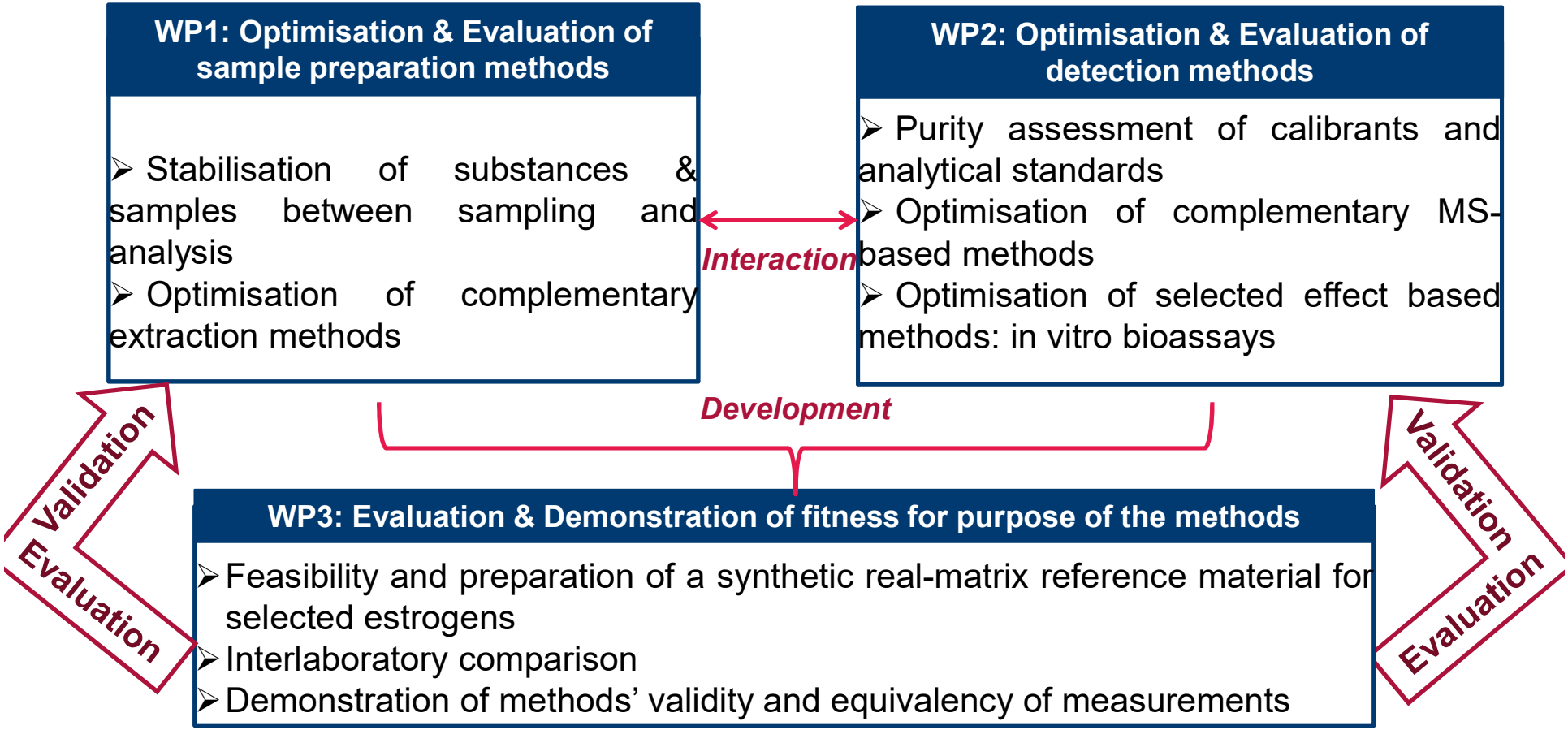
3. **Develop production methods for aqueous reference materials (RM),** which are as close as possible to real water samples, with proven homogeneity, short- and long-term stability.

The specific objectives of the project are to:

4. **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.

5. **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.

6. **Contribute to the work of key European and international standardization organizations e.g. CEN TC 230 and ISO TC 147 ensuring that the outputs of the project are aligned with their needs, communicated quickly to those developing the standards and to those who will use them** to support the implementation of directives, and in a form that can be incorporated into the standards at the earliest opportunity.


WP1: Optimisation & Evaluation of sample preparation methods
➢ Stabilisation of substances & samples between sampling and analysis
➢ Optimisation of complementary extraction methods
Interaction
WP2: Optimisation & Evaluation of detection methods
➢ Purity assessment of calibrants and analytical standards
➢ Optimisation of complementary MS-based methods
➢ Optimisation of selected effect based methods: in vitro bioassays
Development
Validation
Evaluation
WP3: Evaluation & Demonstration of fitness for purpose of the methods
➢ Feasibility and preparation of a synthetic real-matrix reference material for selected estrogens
➢ Interlaboratory comparison
➢ Demonstration of methods' validity and equivalency of measurements
Validation
Evaluation

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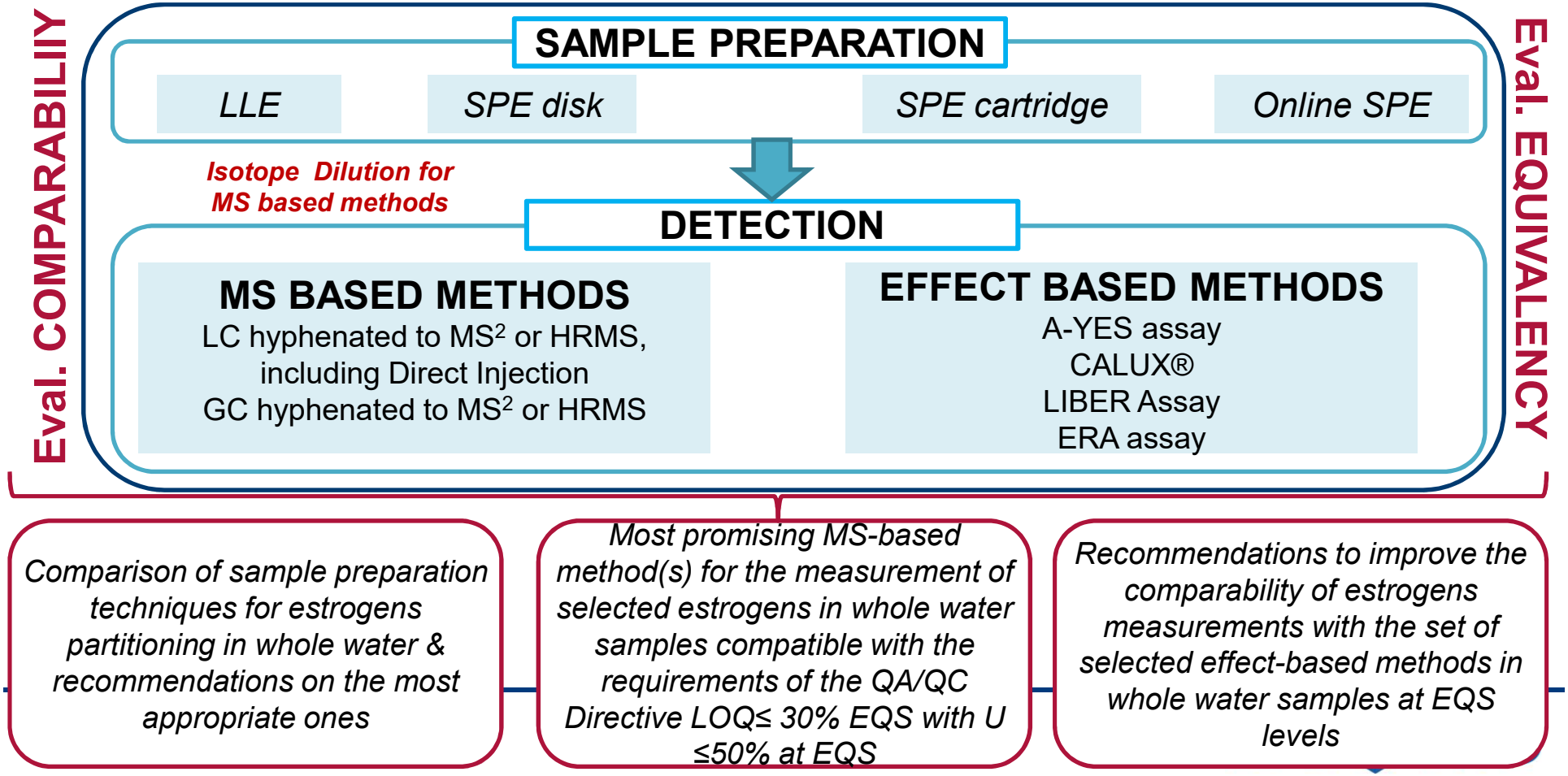
METHOD OPTIMIZATION



METHOD VALIDATION

❑ Validation will be performed according to the following standards:

Identification
ISO 21253-1

+

Performance characteristics
FprCEN/TS 16800: 2019
Level 1

+

Uncertainty
EN ISO 11352
GUM

❑ With respects to their intended scope:

	MS based methods	Effect Based methods
Measurand	E1, 17αE2, 17βE2, 17αEE2, E3	E2 eq
Unit	µg/L	µg E2 eg /L
Matrix	Surface water and ground water Options: Some partners will try to investigate drinking water and marine water	
Fraction of the matrix	Whole water up to 50 mg/L of SPM	
Concentration range	1/3 EQS to tens of ng/L	

METHOD VALIDATION

❑ 6 samples representative of the scope

- 3 synthetics:
  - Evian DOC 1mg/L
  - Evian DOC 7mg/L
  - Evian 7mg/L + TSS 50mg/L

+

- 3 naturals (choice of laboratory)
  characterized with TSS, DOC, ionic strength

❑ 3 levels of concentrations will be tested:

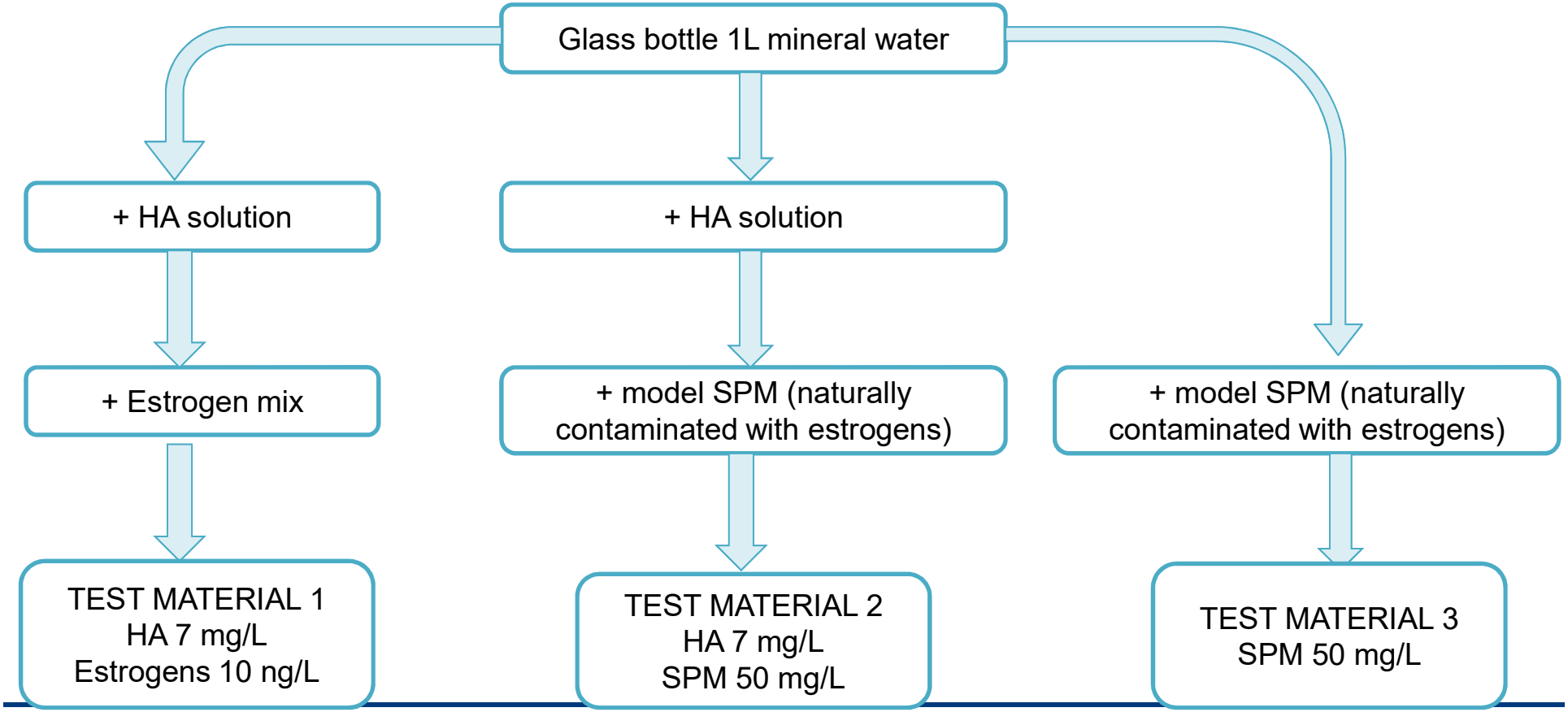
- 1/3 EQS or PNEC, for some compounds possibly below
- EQS or PNEC
- P95 European WL survey

TRAINING WORKSHOP

- Provide information on the developed protocols for measurements of estrogens and give practical advice
- 2 days meeting
- Hosted by EPOC-LPTC, Université of Bordeaux (France)
- T2 2022

First announcement in T2 2021

DEVELOPMENT OF REFERENCE MATERIAL



DEVELOPMENT OF REFERENCE MATERIAL

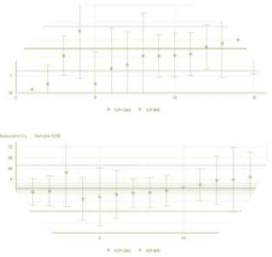
- Stability and homogeneity tests for the candidate reference material
  - Homogeneity
  - Short term stability
  - Long-term stability

- According to ISO 35:2017

INTERLABORATORY COMPARISON

- ❑ Reference materials satisfying to homogeneity/stability requirements

- ❑ Open to participants outside the project
- ❑ Implementation of the methods developed within the project and/or home laboratory



- ➢ **For the project: Demonstration of method validity and equivalency of measurements**
- ➢ **For participants: Z score and/or zeta score**

First announcement in T1 2022

THE CONSORTIUM

8 partners:

- Bringing together scientific excellence in research and experience in ultra-trace measurements of micropollutants.
- Offering a balance of expertise in method development and validation, development and certification of Reference Materials, proficiency tests / interlaboratory comparison design and standardisation activities,

to address the objectives of EDC-WFD.

Coordinator:

- **If you will to collaborate, participate to our programme of activities : CIL, dissemination, trainings, or**

- **For more information**

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Follow us on our website:
http://projects.lne.eu/jrp-edc-wfd/

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