

# Progress in the harmonization of sediment sampling and pretreatment protocols for sediment quality assessment in Switzerland

Michel Wildi<sup>1</sup>, M. Carmen Casado-Martinez<sup>1</sup>, Benoit J.D. Ferrari<sup>1</sup>, Inge Werner<sup>2</sup>

<sup>1</sup> Swiss Centre for Applied Ecotoxicology Eawag-EPFL, EPFL-ENAC-IIE-GE, Station 2, CH-1015 Lausanne, Switzerland

Phone: +41-(0)-2169 30896

E-mail: carmen.casado@centreecotox.ch

<sup>2</sup> Swiss Centre for Applied Ecotoxicology Eawag-EPFL, Überlandstrasse 133 8600 Dübendorf, Switzerland

**Introduction:** Since 2015, the Swiss Centre for Applied Ecotoxicology (EC) and the Federal Office for the Environment (FOEN) are working on the harmonization of sediment quality assessment strategies at the national level. According to a survey performed to all Cantonal Environmental Agencies (CEAs) in Switzerland [1], important discrepancies in sampling and pretreatment methodologies exist that prevent building up a common approach. A harmonization process for sediment sampling and pretreatment was developed and implemented over the last two years. This process included a first draft of harmonized protocol including sieving at 2 mm and extraction with *Aqua regia* for metal analysis, and a two-step validation exercise of this protocol for evaluating its suitability for implementation at the national level.

**Methods:** The first step included a method comparison exercise that involved eight different CEAs and 14 sites. The EC and each CEA sampled sediments at one or two sites using the proposed protocol and their in-house protocol under repeatability conditions. The exercise was completed in triplicate to assess intra and inter-operator variability. To study the influence of the grain size of the sediment sample in which the analyses are performed, sediments collected using the proposed sampling protocol (2 mm) were also sieved in the laboratory to obtain a subsample of the 63 µm fraction. The influence of four different extraction methods in use by the CEAs was tested on a subset of 20 samples. Statistical analyses were performed with linear mixed effect model using R software [2] to discern significant differences between methods. The second step of the validation exercise included an estimation of the uncertainty of the proposed method and involved eight different operators taking sediment samples simultaneously at one site under repeatability conditions. All samples were analyzed for trace elements by the same laboratory to minimize this source of variability. The repeatability coefficient and estimated uncertainty of the method were calculated using R software [2].

**Results and discussion:** The comparison of the different methods showed that the harmonized

protocol provides significantly different results from the sampling protocol used by five of the eight CEAs that participated in the exercise. The type of samples collected (composite or single), the grain size analyzed (2mm or 63µm) and the type of sieving (dry or wet) had a significant influence on measured metal concentrations. However, the harmonized protocol showed a high potential for easy and quick implementation, with not significantly different results between trained and non-trained operators. Metal concentrations showed a high variability when the 2 mm fraction is targeted in the analyses at sites with approx. less than 20% of fine sediment. In such case, the protocol of targeting the fine fraction (< 63 µm) may be more appropriate for specific study objectives (e.g. trend monitoring). Global uncertainty, expressed as the relative expanded uncertainty, related to the sampling step according to our second field trial is 18-67% (0.95 probability). Uncertainty including sampling, extraction by *Aqua regia* in the microwave and analysis by ICP-MS is 39-103%. The resulting sampling uncertainty from our exercise was higher compared to that obtained for surface water sampling [3]. The outcomes of our validation exercise will be discussed, together with recommendations for future harmonization of sediment quality assessment strategies.

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**References:** [1] Wildi et al. (2018). *Ecotox Centre report*. [2] R Development Core Team (2008). [3] Rode and Suhr, *Hydrolog. Earth Syst. Sci.*, 11, 863- 74, (2007).