Estimation of sampling uncertainty in lake-water monitoring in a collaborative field trial

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A collaborative sampling trial was performed by AQUAREF (French national reference laboratory for water and the aquatic environment) at Paladru Lake, Isere, France. This trial was the first national test aiming at improving knowledge of the effect, in lake water, of sampling on global uncertainty.

Ten sampling teams regularly involved in Water Framework Directive monitoring of lake water were asked to participate, in agreement with the Regional Water Agencies. All the samples were collected and analyzed by INERIS in order to minimize the analytical impact on global uncertainty. The data treatment was performed according to standard NF ISO 5725-5.

Overall results for a few metals (cobalt and nickel) showed predominance (>90%) of sampling uncertainty compared to analytical uncertainty.

For other metals, the initial blank tests showed possible contamination of sampling systems, and really pointed to the importance of blank sampling.

Finally, this trial highlighted the need for general guidelines for sampling that could minimize the risk of misclassification of lake-water status.

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1. Introduction

Water-quality data are collected on a large scale as part of the French national permanent monitoring program for the Water Framework Directive (WFD), which surveys the current status of surface waters and groundwater.

Concerning the surface-water compartment, lake-water quality is part of this monitoring program. These data are generated using different analytical and methods. Particularly sampling sampling, a lack of national or European detailed standards is relevant. In France. this led to different sampling protocols, depending on the area monitored [1]. As a result of this regional non-homogeneity, it is difficult to compare data on hazardoussubstance concentration. In order to arrive at a common view on the necessary monitoring for the WFD, a process of analytical chain improvement was first embarked upon. Quality assurance and quality control (QA/QC) procedures for analytical laboratories were studied and then applied. This process, supported by regulation and accreditation, was completed at the beginning of 2000 [2].

Since the first attempt of Bowen, in the 1960s, to improve the comparability of outcomes of various scientific teams through the use of a common certified reference material, the analytical community has constantly expressed interest in metrological aspects of measurements. The uncertainty associated with the result of an analytical measurement performed for water control monitoring is quite known and in few cases agreed. However, the variability introduced by sampling in an aquatic matrix is more difficult to evaluate. The uncertainty and the variability can relate to the sampling methods or the materials. This means that there is a risk of potential errors in sampling chain prior to the samples reaching the laboratory. For example, sampling materials can be polluted before sampling operations, frequently related to an

*Corresponding author. E-mail: fabrizio.botta@ineris.fr inadequate storage or even cross-contamination during transportation.

Consciousness of sampling uncertainty since 2000 has basically arisen from other fields. The first discussions on sampling uncertainty had a focus on soils. Soil-sampling-intercomparison exercises aimed at assessing uncertainties associated with soil sampling in agricultural, semi-natural, urban and industrial environments [3]. Inter-organizational sampling trials were also performed at a reclaimed landfill site in England to assess the uncertainty in measured concentrations of landfill gas [4].

An increase in consciousness of water-metrology uncertainty has registered at the European level since 2007, when the JRC (European Commission Joint Research Centre Institute for Environment and Sustainability) introduced for the first time a proficiency-testing service (PTS) [5,6]. In this campaign, nine European institutional laboratories were invited to sample surface water of the Po River (Pontelacoscuro, near Ferrara, Italy), and to transport the field samples to their own analytical facilities. The second campaign took part on the Danube River (Hungary) two years later. Other water compartments were also investigated in other Member States. However, with these types of exercise, the results could not identify which part of the result variability was introduced by analyses and which part was allocated to sampling. A sampling collaborative trial in the Baltic Sea (German marine monitoring program) was carried out in 2007, with a very similar principle to our trial [7], but on other variables (nutrient parameters). In this case, all chemical analyses were performed within one laboratory in order to eliminate the effect of analytical bias between participants for coastal water.

In France, the lack of standards in sampling activities led AQUAREF (French national reference laboratory for

water and the aquatic environment) to develop national working groups. Consequently, since 2009, a few sampling standards were published by AFNOR [8] and sampling teams can be accredited even if they do not carry out analytical activities [9] (ISO 17025, 2005).

In 2007, AQUAREF started a series of collaborative field trials on different water categories {e.g., surface water [10] or groundwater [11]}. These two trials were the first national intercomparison field tests, bringing together private sampling teams, sub-contracting with French authorities in water monitoring under the WFD [12].

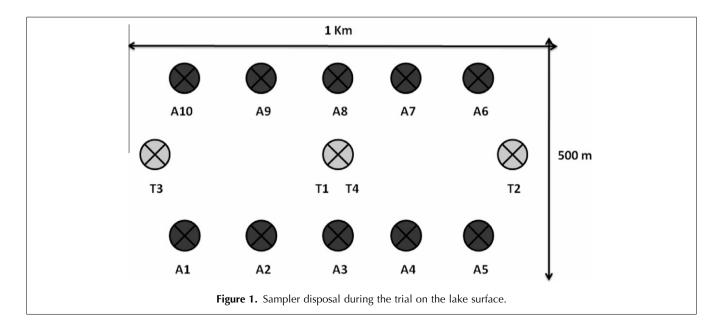
The 2010 trial campaign was organized as a continuation of these previous actions. Some 10 French sampling teams, working for chemical-monitoring activity, were selected by water agencies. The trial campaign was expected to clarify few questions:

- (I) knowledge of sampling procedures and materials used for monitoring lake water under the WFD;
- (II) understanding of sampling procedures and their accordance to the requirements of the WFD; and,
- (III) evaluation and assessment of the impact of sampling on the final uncertainty.

We would like to state that it was not a proficiency test of the capability of the sampling team. All those trials will contribute to complete the national and European guidance on chemical monitoring of surface water under the WFD.

2. Material and methods

This trial was organized by INERIS in partnership with LNE and INRA. According to expert assessment and field availabilities, two sites were pre-selected, Paladru Lake and Bourget Lake. Preliminary tests started in 2009 to choose the best site. Water analyses were carried out to



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