

WORK INSTRUCTIONS

Distribution 2023/I.

General instructions

Samples in the package and respective determinants are as follows (*please note that your package contains only the samples you applied for*):

- SW-M-1 and SW-M-2: real surface water samples in 250 cm³ plastic bottles for the analysis of Cd, Ni and Pb;
- SW-N-1 and SW-N-2: real surface water samples in 500 cm³ plastic bottles for the analysis of NH₄⁺-N, NO₃⁻-N, organic N, total N, PO₄³⁻-P, total P (TP);
- SW-N/M-1 and SW-N/M-2: spike solutions (synthetic concentrates) in 20 cm³ plastic containers containing NO₂⁻-N and Hg;
- WATER FOR DILUTION - NO₂⁻-N: simulated surface water in 500 cm³ plastic bottles to be used for the dilution of NO₂⁻-N spike solutions SW-N/M-1 and SW-N/M-2;
- WATER FOR DILUTION - Hg: simulated surface water in 500 cm³ plastic bottles to be used for the dilution of Hg spike solutions SW-N/M-1 and SW-N/M-2;
- FISH-M-1 and FISH-M-2: fish muscle samples in 30 cm³ amber glass bottles for the analysis of Hg.

Samples should be stored unopened at 4 ± 2°C in the dark until analysis. Appropriate measures should be taken to prevent cross-contamination. In water samples, all constituents are present in soluble form, samples should not be filtered.

Samples should be analysed as soon as possible and immediately after opening sample containers. Samples should be analysed by the method routinely used in the laboratory.

Please follow the safety instructions for laboratory chemicals when handling samples.

Sample preparation and analysis

- SW-M-1 and SW-M-2: Samples are prepared from real surface water, pretreated and preserved according to the requirements of ISO 5667-3, i.e. by addition of nitric acid. Samples should be analysed directly.
- SW-N-1 and SW-N-2: Samples are prepared from real surface water, pretreated and preserved according to the requirements of ISO 5667-3, i.e. by addition of sulphuric acid. Samples should be analysed directly. Please remember to adjust pH before analysis.
- SW-N/M-1 and SW-N/M-2: Spike solutions are preserved by addition of Hg(II)Cl₂. Proficiency testing samples should be prepared in situ by adding spike solutions to water issued for dilution, which is simulated surface water, pretreated by bringing to boiling point.

- Preparation instructions for NO₂⁻-N: dilute an aliquot of spike solution by a factor of 50 (e.g. 1,0 cm³ to 50 cm³) using “WATER FOR DILUTION - NO₂⁻-N”. Measurement results should be reported for the samples prepared as above.
- Preparation instructions for Hg: dilute an aliquot of spike solution by a factor of 10000 (e.g 0,5 cm³ to 50 cm³ in two consecutive steps) using “WATER FOR DILUTION - Hg”. Measurement results should be reported for the samples prepared as above.
- FISH-M-1 and FISH-M-2: each bottle contains approximately 6 g of freeze-dried fish muscle sample with a particle size of < 0,5 mm. Samples are prepared from freshwater fish muscle and spiked with inorganic mercury. Samples should be analysed directly. Suggested sample intake for analysis: ~0,5 g. Results should be reported to the material provided “as is”, i.e. no separate determination of and correction with dry matter content is required. Expected concentration range: 1,0 – 6,0 mg/kg.

Reporting of results

Participants are asked to report their expanded measurement uncertainty together with the measurement results using a coverage factor of $k = 2$, so that E_n numbers can be calculated during evaluation. E_n numbers help participants to assess the validity of their uncertainty estimation. Please make sure that expanded measurement uncertainties are reported in the **same unit of measurement** as the measurement results.

Results should be reported **electronically via our webpage** (www.qualcodanube.eu) using your personal customer ID which has been communicated earlier by e-mail. If you have problems with online reporting, it is also possible to report results by e-mail (info@qualcodanube.eu) using the Data report form available from the project website.

Reporting deadline: 31st October, 2023

Please make the appropriate rounding and report in the unit of measurement stated on data report forms. Results reported after the above deadline or as „< LOD/LOQ” will be excluded from evaluation, results reported in inappropriate units of measurement will not be converted. (Reference: ISO 13528:2022. Statistical methods for use in proficiency testing by interlaboratory comparisons.)

Please note that in case zero (“0”) is reported as measurement result, it is regarded as a valid result and is evaluated accordingly.

Budapest, 01st September 2023

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