

Sampling design and preparation

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Choosing a laboratory and analytical method, holding times and preservation

1 Purpose and scope

This document outlines the requirements involved in selecting a laboratory and appropriate analytical methods for a sampling program and associated holding times and preservation of samples.

2 Associated documents

Sampling design and preparation:

- *Preparation for sampling*
- *Sampling scope and design*

3 Selection of laboratory

Select laboratories that demonstrate Good Laboratory Practice (GLP) and perform according to the standard AS ISO/IEC 17025-2005 (R2016): *General requirements for the competence of testing and calibration laboratories*. This is usually demonstrated by laboratories holding accreditation with the National Association of Testing Authorities (NATA). Although NATA accredited laboratories are recommended, on occasion exceptional circumstances arise where specialist analysis and interpretation may be required. If the analysis is not available through using a NATA accredited laboratory, select another laboratory that holds the required expertise. It is important to validate the procedure including its detection or reporting limits and accuracy.

Analysis of samples taken for statutory purposes under the *Environmental Protection Act 1994* and its subordinate legislation, including the *Environmental Protection Regulation 2008* and the *Environmental Protection (Water) Policy 2009* should be undertaken by NATA accredited laboratories.

4 Selection of analytical methods

The laboratory should be consulted prior to sampling in regards to the appropriate analytical method that will provide the required information to meet the purpose of the sampling program. The analytical methods selected may well dictate the sampling procedures required.

The method should be:

- appropriate for the type of sample and for the expected concentration range of the analyte to be measured
- verified or validated (e.g. by proficiency tests, using certified reference materials or standard addition techniques) (see NATA Technical Note No. 17; NATA 2013)
- accredited by NATA, or at least equivalent to (e.g. at least national accreditation if samples sent overseas).

Note: Laboratories receive NATA accreditation to perform specified analytical methods. Always check the laboratory has accreditation or validation for the required methods.

If the laboratory is not NATA accredited, samples should be analysed in accordance with a method specified in one of the following reference texts:

- *APHA AWWA Standard Methods for the Examination of Water and Wastewater (current version)*

- USEPA Clean Water Act Analytical Methods (current version)
- ASTM, *Annual Book of ASTM Standards Volume 11.01 Water (I), 11.02 Water (II)* (current version)
- relevant Australian Standards (current version)
- relevant ISO Standard (current version).

Alternative methods to those described in the reference texts can be used, provided the laboratory has validated the alternative method and proven that the results obtained are equivalent to those that would be obtained using the prescribed method. The laboratory also needs to show that the results are within the uncertainty stated for the prescribed method.

5 Limits of detection and reporting

The limit of reporting (LOR) for an analytical method should be lower than the benchmark (such as water quality objective, water quality guidelines or trigger values, Environmental Authority (EA) limits or other standards) to which the results will be compared. The LOR is the lowest concentration of an analytical parameter that can be detected by a particular method that has acceptable precision and accuracy. If the LOR is higher than the benchmark, alternative methods of sampling and/or analysis should be investigated (e.g. passive sampling).

6 Holding times and preservation

Holding times (how long a sample can be kept between collection and analysis), and preservation of samples is critically important for the integrity of a sampling program, as the constituents of samples may change between collection of samples and analysis (as a result of chemical, biological or physical reactions). (AS/NZS 5667.1:1998). Guidance for different holding times and preservation methods is provided in:

- APHA AWWA *Standard Methods for the Examination of Water and Wastewater* (current version)
- USEPA *Clean Water Act Analytical Methods* (current version)
- *Australian and New Zealand Standard: AS/NZS 5667.1:1998.*

Ideally the holding times and preservation methods specified in the aforementioned documents should be adhered to. However alternative methods may be determined for samples collected as part of an on-going monitoring program (e.g. Oudyn et al. 2012) or say if a preservative is considered hazardous. Alternative methods can be used in such cases, if the alternative method has been validated and the laboratory can demonstrate the results obtained are equivalent to the results that would be obtained using the relevant prescribed holding times/preservation method.

The analytical laboratory should be contacted for advice on holding times and preservation before any samples are collected.

Some holding times are extremely short (for example pH and chlorine) and the use of *in situ* tests and test kits may be more appropriate than laboratory analysis.

7 References and additional reading

APHA AWWA (current version) *Standard Methods for the Examination of Water and Wastewater*, American Public Health Association (APHA), the American Water Works Association (AWWA) and the Water Environment Federation (WEF).

ASTM (current version), *Annual Book of ASTM Standards: Volume 11.01 Water (I), 11.02 Water (II)*.

Australian and New Zealand Standard (AS/NZS) 5667.1:1998, *Water quality—Sampling Part 1: Guidance on the design of sampling programs, sampling techniques and the preservation and handling of samples.*

National Association of Testing Authorities, Australia 2014, *Guidelines for the validation and verification of quantitative and qualitative test methods Technical Note 17—October 2013*, viewed 8 December 2016, http://www.nata.com.au/nata/phocadownload/publications/Guidance_information/tech-notes-information-

[papers/technical_note_17.pdf](#).

Oudyn, F, Lyons, DJ, Pringle, MJ 2012, 'Appropriate maximum holding times for analysis of total suspended solids concentration in water samples taken from open-channel waterways', *Water Science and Technology*, 66, 1310-1316.

USEPA (current version), *Clean Water Act Analytical Methods*, US Environmental Protection Agency, Available from: <https://www.epa.gov/cwa-methods>.