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SAMPLING PROCEDURE	EXP 62

The sampling procedure is of the utmost importance. It is too often neglected when the laboratory people in charge of the analysis do not sample water by themselves; its bad execution may cause all results to be false; as a matter of fact, one must be sure of the reliability of the water sample taken (according to the case, we try to emphasise either the average quality, or the extreme features or both) and the preservation of all characteristics during the sampling process and possibly during transportation (avoiding gaseous emissions, air oxidation, reactions with pipes or collecting vessel walls, accidental contaminations, etc.).

Sampling vessels

Preferably choose clear glass bottles (except for highly alkaline waters), plugged with emery or polyethylene corks; these bottles shall be new or otherwise abundantly rinsed after treatment either with a sulpho-chromic mixture (except if chromium traces are searched for in water) or with acid (preferably nitric, or even hydrochloric or sulphuric); rinsing shall be first performed with ordinary water (at least three times, fully filling the bottle at each time), then several times with distilled water. During sampling, the bottle shall be rinsed several times with the water under analysis.

If no such special bottle is available (bottles may be ordered in advance from the laboratory in charge of the analysis), table water bottles with plastic plugs having served only once may be used (do not use bottles having contained a very saline mineral water) (if only a cork plug is available, previously dip it in boiling water for some time). Before sampling, wash and rinse the bottles as described before. Clear glass table water bottles are always preferred, but ordinary plastic bottles may be tolerated, unless otherwise stipulated by the analyst (for instance, they must be proscribed when performing organic micro-contaminants searches).

Other types of vessels may be accepted, plastic material cans (or "jerrycans") for instance when the investigation in progress calls for a large volume of water (provided, of course, that they are new or have only contained water, they have been abundantly rinsed and there is no contra-indication in relation with the scheduled analyses).

On the other hand, one should proscribe standard tinted glasses, various alcohol bottles, metal vessels, and generally any container or former contents likely to impair the analysis result.

Some types of analyses require specific sampling procedures, using different bottles from those provided for the general analysis:

- *for the bacteriological analysis*, sterile bottles shall be compulsorily used, to be provided by the laboratory in charge of the analysis;

- *for determining certain minerals*, sample the water in clear glass or polyethylene bottles in which some hydrochloric acid (when searching for Fe, Al, Cr, Cu, Pb) or nitric acid (if in addition to the previously mentioned elements, other metals, in particular Mn, are searched for) will have been previously poured; of course, these acids should no longer contain the searched metals ("pure for analysis quality"); the quantity to be used is of about 2 - 5 ml of pure acid per litre of water;

- *for determining the silicon contents*, use polyethylene bottles if silicon traces appear in water; when large amounts are searched for, glass bottles may be used, provided they have been used for a long time (as opposed to what is recommended for the other determinations) and water is not very alkaline; the same remarks apply to sodium and boron measurement;

- *for phytoplankton assessment (algae)*, also perform a special sampling into a bottle containing 3 to 5 ml of 40% trade formol solution in 100 ml of water;

- *for cyanide dosing*, take a clear glass bottle and introduce soda (about 1 to 2 ml of concentrated lye per litre of water) to alkalinise the sample to a pH of up to 11 - 12. This will stabilise the cyanides;

- *for other determinations*, it is also necessary to stabilise the samples if the analysis is not readily performed: 40 mg/l $HgCl_2$ for the various nitrogen forms (preservation: 7 days), 2 ml/l H_2SO_4 for organic contamination criteria (DCO, COT, oils, greases, ...).

Sampling mode

It is necessary to select a sampling mode suited to water origin.

Distribution network or industrial system

Let water flow out of tap long enough to make sure that a representative sample is produced; this period of time will depend on site layout and tap flow-rate: it may range from 30 seconds (boilers) to 10 minutes (especially if the tap has not been used for some time).

In addition, the tap should deliver a constant flow-rate to avoid any velocity change likely to detach deposit elements (dirt, corrosion, etc.) from pipe walls. For a bacteriological sampling, remove the jet-breaker and char tap outlet (with a welding torch for instance) before opening it and collecting the sample.

Moreover, if the sampling procedure applies to an industrial system where high temperatures and pressures are prevailing (boilers in particular), all useful precautions must be taken to avoid partial evaporation, dilution with drinkable or condensation water, etc.; the sampling tap should normally be fitted with a coil dipped in cold running water before relief; for lower pressures (less than 10 bar), it may be sufficient to collect water in a vessel dipped in cold running water.

Reservoir or cistern

In a reservoir or cistern, proceed as described below for lakes, dams or springs.

Natural environment

- *Surface waters*

Sampling should be performed at the location selected for the future water tapping.

In a river, avoid dead zones along the banks; to take the water in full stream and fairly far from the bottom, put on boots or even thigh-boots to walk in water (while monitoring the deposits possibly returned to suspension), or throw a clean bucket fastened to a rope from and downstream of a bridge.

In the case of a lake, a pond, a dam or a deep river, carry out samplings at various levels; the sampler shall stand (as applicable) in a boat, on a gangway, a bridge or a water sampling tower; he shall use either a hand-pump or a mechanical pump, or a special deep water sampling device (various mobile clamp, valve-operated models, etc. are available on the market), which should not contact the air previously contained in the vessel.

Such a gear may also be used for sampling a spring water, but in such a case, it is also possible to directly immerse the vessel (to be as clean inside as outside, if possible with a broad neck) in water; for a bacteriological sampling, this method implies previous charring of bottle outside, which will be additionally held by small clips, to be also charred: spring water may also be sampled from a weir.

Surface water analyses must take into account variations as a function of time (seasonal cycle) and space (e.g., changes of characteristics as a function of depth in a lake or a dam).

- *Underground waters*

Since an unused well water is absolutely not representative and its analysis cannot provide any usable information, the well must be compulsorily equipped with a pump which will be operated as in normal operation several days prior to water sampling, if possible.

Swimming-pools

Immerse the vessel in water, taking the same precautions as for springs; also take water from sampling taps when these are fitted to the regeneration system. In addition, it may be useful to know the back-up water quality.

Common sampling rules

- Hands must be very clean.

- Rinse the bottle several times with the water under analysis, unless the bottle has been previously prepared (bacteriological sampling, heavy metals, cyanides, etc.).

- Request information about the volume required by the scheduled determinations (generally from 2 to 5 litres).

- Clearly label the bottles to avoid any mistake. An indelible ink marker may also be used.

- Fill bottles up to overflow so that they can be plugged without air bubbles (if not possible, only allow a bubble of 1 cm^3 under the cork). It is not recommended to seal corks.

- When this is absolutely required by the survey, determine those characteristics that are likely to change during transportation: temperature, resistivity (or conductivity), pH, dissolved gasses (O_2, CO_2, H_2S, etc.), nitrogen compounds (more especially NH_4^+ et NO_2^-), residual disinfectants (chlorine, ozone, chloramines, ClO_2), etc.

- Take special precautions for measuring the pH and dissolved gasses: bottle to be filled without contacting the air, using a hose the end of which reaches the bottom, and renewing bottle contents several times by overflow out of the upper section. If an intermediate container is used (deep sampler, bucket very slowly dipped in horizontal position, etc.), the bottle shall be filled by siphoning.

- For those determinations to be carried out in laboratory, water samples shall be carried as rapidly as possible, more especially if bacteriological analyses have been ordered (4 hours maximum in this case; a one-day delay may be granted if bottles are maintained in ice). In any case, refrigeration at about 4°C is highly desirable.

List of standardised analysis methods

The French Standards Association (1) publishes the "Water Tests" analysis methods listed hereafter.

NF T 90 000	Guide for writing analysis bulletins	NF T 90 016	Gravimetric calcium contents measurement
NF T 90 001	Sampling procedure for boiler waters and pressurised system waters	NF T 90 017	Colorimetric iron contents measurement
NF T 90 003	Measuring hardness to complexing reactants	NF T 90 018	Measuring the oxygen released by the potassium permanganate
NF T 90 004	Fluorine spectrophotometric measurement	NF T 90 019	Sodium ions contents measurement
NF T 90 005	Magnesium contents measurement	NF T 90 020	Gravimetric potassium ion contents measurement
NF T 90 006	pH colorimetric measurement	NF T 90 021	Photometric iodine contents measurement
NF T 90 007	Silicon contents measurement	NF T 90 022	Copper spectrophotometric measurement
NF T 90 008	pH electrometric measurement using a glass electrode	NF T 90 022 1	Low copper contents spectrophotometric measurement (Amendment 1 to NF T 90 022, November 1962)
NF T 90 009	Sulfate ions measurement	NF T 90 023	Orthophosphates and polyphosphates spectrophotometric measurement
NF T 90 010	Colorimetric measurement of low free oxygen contents (Orthotolidine method)	NF T 90 024	Manganese spectrophotometric measurement
NF T 90 011	Carbon dioxide measurement	NF T 90 025	Selenium spectrophotometric measurement
NF T 90 012	Nitrate contents measurement	NF T 90 026	Arsenic contents measurement
NF T 90 013	Nitrite contents measurement	NF T 90 027	Overall search for heavy metals
NF T 90 014	Chlorine ions measurement	NF T 90 028	Colorimetric lead contents measurement
NF T 90 015	Ammonia nitrogen measurement	NF T 90 029	Determining dry residues, burnt residue and sulphated residue

NF T 90 030	Determining the clogging power	NF T 90 111	Assessing the dissolved salts contents from determination of theoretical electrical conductivity
NF T 90 031	Determining the resistivity (or electrical conductivity)	NF T 90 112	Measuring ten metallic elements (Cr, Mn, Fe, Co, Ni, Cu, Zn, Ag, Cd, Pb) by atomic absorption spectrometry
NF T 90 032	Solubility table of oxygen in water	NF T 90 113	Measuring the mercury contents by atomic absorption spectrometry
NF T 90 033	Measuring the scattering index or turbidity measurement	NF T 90 114	Total hydrocarbons contents measurement (infrared spectrophotometry method)
NF T 90 034	Measuring the colour versus the Hazen scale	NF T 90 201	Oil refinery water effluents - Sampling
NF T 90 035	Assessing the taste	NF T 90 202	Oil refinery water effluents - Measuring water suspended organic materials extractible with hexane
NF T 90 036	Determining alkalinity (TA and TAC)	NF T 90 203	Oil refinery water effluents - Total hydrocarbons contents measurement
NF T 90 100	Sampling: precautions to be taken to perform, preserve and process samples	NF T 90 204	Oil refinery water effluents - Phenols contents measurement
NF T 90 101	Determining the oxygen chemical demand (DCO) (potassium dichromate method)	NF T 90 301	Determining mobility inhibition of daphnia magna strauss (cladocere crustacea)
NF T 90 103	Determining the biochemical oxygen demand (DBO)	NF T 90 302	Assessment method in aqueous environment of the so-called total biodegradability of organic products
NF T 90 104	Putrescibility tests	NF T 90 303	Determining the sharp toxicity of a substance versus brachydanio rerio (static test)
NF T 90 105	Determining suspended materials	NF T 90 401	Micro-organisms counting procedure: counting colonies at 37 °C (agar incorporation method)
NF T 90 106	Dissolved oxygen contents measurement	NF T 90 402	Micro-organisms counting procedure: counting colonies at 20 °C (agar incorporation method)
NF T 90 107	Total cyanides contents measurement	NF T 90 413	Assumed coliforms and faecal coliforms counting procedure after seeding in liquid medium (calculation of the most likely number)
NF T 90 108	Free cyanides contents measurement	NF T 90 414	Assumed coliforms and faecal coliforms counting procedure (diaphragm filtering method)
NF T 90 109	Determining the phenol index		

(1) AFNOR, Association Française de NORmalisation, Tour Europe, Cedex 7, 92080 Paris La Défense