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Storage and Preservation of Water Samples for Chemical Analysis

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تخزين وحفظ عينات المياه للتحليل الكيميائي

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Abstract:
Sample storage and preservation are important steps in water chemical analysis. There are many changes may occur during the sample storage step. To minimize changes between sample collection and the analysis some probable problems have been discussed and some recommendations for sample preservation e.g. chemical additions, temperature control, choice of sampling container and holding time have been given.

Keywords: Sample storage, Sample preservation, water analysis.

الملخص
يُعدّ تخزين العينات وحفظها من الخطوات المهمة في التحليل الكيميائي للمياه. وقد تطرأ العديد من التغييرات على العينة خلال هذه المرحلة. ولتقليل هذه التغييرات بين جمع العينة وتحليلها، نناقش بعض المشكلات المحتملة ونقدم بعض التوصيات لحفظ العينات، مثل إضافة مواد كيميائية، والتحكم في درجة الحرارة، واختيار وعاء أخذ العينات، ومدة الحفظ.

الكلمات المفتاحية: تخزين العينات، حفظ العينات، تحليل المياه.

Introduction

For many practical reasons it is usually difficult to start the analysis of water immediately after sample collection and as most of the trace analytes cannot be determined on-site due to technical limitations, it is a general practice to collect a representative sample and preserve it until analyses in a laboratory. Therefore, it is essential to assure the water samples integrity from collection to data reporting, but in the chain of current procedures complete preservation of each constituent in the samples can never be achieved. Many chemical, physical and biological processes may occur in water between collection and analysis. Hence, preserving samples is as important as the analysis itself. The preservation steps aim to maintain the original concentration of the analytes and their chemical nature.[1,2]

Data from the analysis of waters are no better than the techniques by which such samples are gathered, prepared, and stored pending analysis. There have been numerous recent developments of techniques for the collection, processing, and storage of water samples that minimize contamination and maximize the integrity of the initial dissolved analyte loading.[3],[4],[5]

Sample storage is a necessary and critical step in chemical analysis. During the storage, many changes in the sample may occur, namely chemical and physical reactions, microbiological degradation and the nature of sample container may produce analyte losses.[1]

No storage protocol is sufficiently effective to preserve the sample integrity for ever. Sample deterioration starts at the point of collection, all we can hope for it, is to slow it down.

The selection of a preservation method is dependent upon: (1) the analyte(s), (2) the analyte concentration level(s), (3) the sample matrix, and (4) the sample preparation method.

Sampling

Sampling could be defined as a process of selecting a portion of material small enough in volume to be transported conveniently and handled in the laboratory, while still accurately representing the part of the environment sampled. The main difficulties in sampling are representativeness and integrity. Sampling is so important that, in some cases, it represents the main contribution to the error of the whole analytical process, especially when trace contamination is being measured.[6]

Contamination and Losses

The major problem in sample preparation for trace analysis is the contamination of the sample. Contamination is associated with several probable causes, i.e. the type and the grade of the used reagents, sample container, digestion or dilution process of the sample, human intervention, and many others.

Losses are also a significant problem in trace analysis. Container surfaces, for example, may adsorb the analyte on large area of contact. At higher levels such a small absolute loss would have little effect on the concentration but at trace levels a large proportion of the analyte may be stripped from the solution.[7,8,9]

Factors influencing sample stability

Many factors influence the stability of a sample and the analyte, but certain guidelines are useful in the sample storing. From an understanding of the mechanism which governs the stability of the material in storage step it is possible to predict the conditions for keeping the sample the sample unaltered.

The main factors affecting the stability of analytes in water samples include hydrolysis, photolysis, physical adsorption or release to or from suspended particulates, chemical degradation by oxidative free radicals present in natural waters, or biological degradation.[10]

Chemical Factors

Surface activity of sample container

Sample containers are a possible source of contamination of the sample, therefore the nature of sample container material is of high importance. A sample component in solution is normally only one side of an equilibrium between the free form of the analyte and a proportion of it which is attached to the container wall as a precipitate, by adsorption or by ion exchange.

Glass is suitable for most samples, but the concentration of trace analytes can be affected by reactive sites on the glass surface. The glass surface consists of silicate lattice bounded by a surface of SiOH groups. These are readily ionized, producing an effective ion-exchange surface, therefore the acidification of the sample has an important stabilization effect, since it protonates the glass surface and releasing the metal back into solution.

There are different ways to achieve a similar effect, e.g. adding a chelating agent like EDTA, and for the amphoteric metals, stabilization can be achieved under alkaline conditions.[5]

Generally, glass containers are preferred for the collection when dealing with organic compounds. The composition of the container caps in contact with the sample or with the extraction solvent can be a problem.[1]

But this is not the case in metals preservation where the recommended sample container is a plastic bottle that is typically polyethylene, polypropylene or polyvinyl chloride.[11,12,13] It should be first shown that the container does not contribute volatiles or plasticizers to the sample.

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Complex cleaning protocols for water sample bottles have been suggested and adopted by many environmental agencies throughout the world. These cleaning procedures are often so elaborate and time consuming that they can hardly be used for routine water sampling programs.[14],[15]

Based on their results from the study of bottle type and acid washing influence on trace element analyses by ICP-MS in water samples Reimann et al.[16] suggested that:

– Adding acid to the containers prior to sampling is an unacceptable procedure, it may damage the plastic and result in contamination of the sample with a variety of elements.
– Acid washing will not necessarily result in cleaner plastic vessels, it appears that some procedures may damage the bottle walls, resulting in an unpredictable release of elements to a water sample.
– The most expensive vessels are not in general superior to the cheapest bottles tested.

Oxidation

Oxidation or more rarely reduction. Atmospheric oxygen is the most common oxidant to affect the stability of water samples. Its solubility in water is very high. e.g., sulphide, sulphite, ferrous iron, iodide, and cyanide may be lost through oxidation and hexavalent chromium may be reduced to trivalent chromium. Cations may precipitate depending on the redox potential of the sample.[7,17]

Atmospheric Carbon dioxide

The dissolution of atmospheric Carbon dioxide results in a significant lowering of pH and as a consequence water is normally acidic. Carbon dioxide will also react with alkali to give an insoluble carbonate and precipitate other in soluble metal carbonate species.[5,13]

Photochemical changes.

Some components can undergo changes associated with light catalysed reactions; however, they can be minimized by the collection of samples in opaque (e.g. brown) containers.[13] Brown glass is not generally recommended in trace analyses because of the impurities the glass may introduce into the sample. It is better to keep the sample in a dark place.7,[18]

Physical factors

Adsorption

There are two possible types of adsorption. Components can adsorb sometimes irreversibly onto the walls of the sample containers. For instance, metals can be adsorbed onto glass surfaces. Therefore the usual sampling approach for metal determinations is to collect the samples in plastic containers and thus eliminate the glass contact.[19]
Appreciable losses may occur for chlorinated insecticides, polychlorinated biphenyls and polynuclear aromatic hydrocarbons6 by adsorption on the walls of the plastic container.[20] The second type is the adsorption on particulate matter present in the sample. The effect of clay on adsorption of some organic compounds is well known.[21]

Volatilisation

In this process volatile species can be lost to the atmosphere, depending on the vapour pressure of the compound to be measured, and the temperature of the sample. The loss of volatile materials can be avoided by collecting the sample in a completely filled container, keeping the samples as cool as possible without freezing or changing the pH value of the samples so that the component is converted to a more stable form.[22,23,24,1]

Biological Factors

The biological aspect of sample stability can arise as a result of live organisms, and through the presence of inactive cellular components. Active biological processes result in the turnover of sample material as it is used as an energy and nutrient source. The metabolic activity of micro-organisms in a sample can affect a large number of constituents, changing their oxidation state. It can be responsible for changes in nitrate, nitrite and ammonia content, phosphorus cycle and reduction of sulphate to sulphide. Soluble constituents may also be converted to organically bound materials in cell structures. The addition of of poisons such as azides, mercury salts, carbon tetrachloride and antibiotics may in some cases be appropriate. Acidification of the sample or freezing will often achieve the same goal. Algal and bacterial activity can often be sufficiently reduced by simply filtering (e. g. at 0.45 µm mesh) the sample during or immediately after collection.[1,7,13]

Storage time

On this subject all the authors agree that the shorter time between the sample collection and its analysis makes the analytical results more reliable. It is difficult to state exactly how much time may be allowed between sample collection and analysis once it depends on the character of the sample, the analysis to be made and the conditions of storage. The maximum holding time is dependent of the matrix and the analytes.

Recommendations

As mentioned before, there is no absolute guideline for sample storage and preservation. During sample transport it is necessary to ensure that some precautions are undertaken. For example, samples should not be exposed unnecessarily to any source of light, facilities for refrigeration of samples should be arranged during transportation, and sample containers should be cleaned and packed so that contamination is avoided.

The samples to be analysed for organics are generally preserved by cooling them to 4°C.[2] Samples that require cooling (4ºC) should be stored under ice in transit and then refrigerated after arriving at the laboratory.

Acidification of water samples ($pH < 2$) preserves most trace metals and reduces precipitation, microbial activity and sorption losses to container walls. For ground waters and dissolved metals in water samples, acidification should only be carried out on filtered samples.

For metals determined by hydride generation-atomic absorption spectrometric method (e.g. arsenic and selenium), acidification should be done with hydrochloric acid.[25]

Freezing of water samples in polyethylene containers is a very popular method, as it holds all of the desirable qualities. Sufficient air gaps in containers should be provided to allow for expansion during freezing.[26,27]

Some papers have demonstrated that solid-phase extraction (SPE) is a good alternative for the storage of pesticides pre-concentrated from water samples.[28,29] Storage of different pesticide classes on the C18 silica-gel surface of solid-phase cartridges for 30 days at 4°C or -18°C is effective.[30,31,32]

Conclusion

A successful preservation technique must maintain the original concentration of an analyte during storage to within the level of accuracy required by the particular program. The optimal method will preserve all of the constituents of interest in a single sample without requiring different procedures for different chemical species. Proper containers, cleaning procedure and storage conditions are essential for high quality data. This maximum holding time is dependent of the matrix and the analytes. No storage protocol is sufficiently effective to preserve the sample integrity for ever.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare that they have no conflict of interest.

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