

Sample preservation and preparation

Purpose: The purpose of this assignment is to address questions related to sample preservation and preparation for analysis.

Learning Outcomes:

At the end of this assignment you will be able to

1. *Suggest a method to preserve a sample for analysis*
2. *Understand the time frame within which a sample needs to be analyzed*

Assignment:

Once a water sample is collected and possibly filtered if particulate matter is present, it is important to ensure that its chemical composition is not altered prior to analysis. Samples can only be stored for a limited period of time.

In many cases water samples can be stabilized for long term storage by controlling the pH, cooling, limiting light exposure or adding a chemical preservative.

Consult the literature in regards to water sample preservation and maximum holding times (refer to section 7C in Harvey, D. Chapter 7, *Implementing the Sampling Plan*).

The table below provides guidelines on preservation methods and maximum holding times for some analytes in natural waters and wastewaters.

Preservation Methods and Maximum Holding Times for Selected Analytes in Natural Waters and Wastewaters		
Analyte	Preservation Method	Maximum Holding Time
ammonia	cool to 4 °C; add H ₂ SO ₄ to pH < 2	28 days
chloride	none required	28 days
metals—Cr(VI)	cool to 4 °C	24 hours
metals—Hg	HNO ₃ to pH < 2	28 days
metals—all others	HNO ₃ to pH < 2	6 months
nitrate	none required	48 hours
organochlorine pesticides	1mL of 10 mg/mL HgCl ₂ or immediate extraction with a suitable non-aqueous solvent	7 days without extraction 40 days with extraction
pH	none required	analyze immediately

Q1. Under what conditions should the water samples be stored and within what time frame should the samples be analyzed?

Q2. What changes could take place that would cause sample degradation?

Q3. No preservation method is required for nitrates. However, a water sample can only be stored for up to 48 hours. What possible degradation could occur over an extended period of

time? What chemical preservative could be added to prolong the life of the sample? How could you devise an experiment to test whether compositional changes for nitrate occur over time?

Q4. Cr(VI) must be analyzed within 24 hours. What might happen to compromise the analysis of Cr(VI) if the samples were held only longer before completing the analysis?

Q5. Preservation of water samples for metals analysis requires acidification with nitric acid below pH 2. Why is this procedure required for analysis of calcium and magnesium?

Consider the K_{sp} for $\text{Ca}(\text{OH})_2$ and $\text{Mg}(\text{OH})_2$.

$$K_{sp} \text{Ca}(\text{OH})_2 = 6.5 \times 10^{-6}$$

$$K_{sp} \text{Mg}(\text{OH})_2 = 2 \times 10^{-13}$$

Q6. Compare the solubility of $\text{Ca}(\text{OH})_2$ and $\text{Mg}(\text{OH})_2$ at pH 2 and pH 8. What could happen to the Ca^{2+} and Mg^{2+} ions stored over time if the pH was not adjusted to an acidic value?