

Best Practice to Prevent False Positives & Negatives for Dissolved Metals

Field Filtration as Best Practice

Most environmental practitioners understand that field filtration is considered the best practice when collecting water samples for dissolved metals testing, but many people may be unaware of the causes and potential magnitude of negative consequences associated with delayed filtration.

Dissolved metals in water exist in complex equilibria and can be impacted by many physical and chemical factors, particularly changes to redox conditions, pH or temperature, which can trigger changes to dissolved metals concentrations due to precipitation, co-precipitation, sorption or dissolution of particulate matter. These factors can cause significant positive or negative bias to dissolved metal concentrations, sometimes to an extreme degree. The only way to ensure water samples collected for dissolved metals will accurately represent conditions at the time of sampling is to conduct filtration in the field, preferably immediately after sample collection.

Co-Precipitation of Dissolved Metals with Iron

Perhaps the most common pitfall resulting from delayed filtration of dissolved metals is caused by the precipitation of iron oxides and associated co-precipitation of other metals. This happens because Ferrous Iron (Fe^{2+}) is relatively water soluble (up to $> 100 \text{ mg/L}$), whereas Ferric Iron (Fe^{3+}) is practically insoluble in water under normal environmental conditions (generally $< 10 \text{ }\mu\text{g/L}$). Ferrous Iron exists and is stable in anoxic waters (e.g. many groundwaters), but when an anoxic water sample is brought to the surface, oxidation of Ferrous Iron to Ferric Iron can occur rapidly, sometimes in as little as five or ten minutes (oxidation rate increases substantially with higher pH). When iron precipitation occurs in a sample, other metals can co-precipitate, causing substantial changes to the overall dissolved metal composition of the sample. This is a well-known phenomenon and precipitation of iron under these circumstances is expected, but co-precipitation of other metals is generally less understood.

ALS Co-Precipitation Impact Study

ALS recently studied the impact of iron precipitation on the co-precipitation of other metals with a series of high-iron groundwater samples and found substantial losses of most dissolved metals (in comparison to field filtered metal concentrations), with losses up to 100% in several cases where filtration was delayed (by 6 to 10 days in these somewhat worst-case examples). Arsenic, Lead, and Cadmium were particularly impacted by co-precipitation, in addition to the expected loss of Iron, but most dissolved metals were impacted to some degree.

A summary of the ALS study results for Arsenic, Lead, Cadmium, and Iron are shown in Figure 1. For these 5 samples, Arsenic losses averaged over 80%, Lead losses averaged over 95%, and Cadmium losses varied significantly by sample, ranging from zero to 97% loss. The impact on sample results can clearly be seen, with many of the test samples potentially having key metal concentrations underestimated by a factor of 10 to 100 times if correct field filtration techniques were not employed. This study also highlights the necessity of field filtration for the analysis of dissolved Ferrous Iron.

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ALS Study: Lab Filtered vs Field Filtered Metals Results for 5 High-Iron GW Samples (Worst Affected Metals)

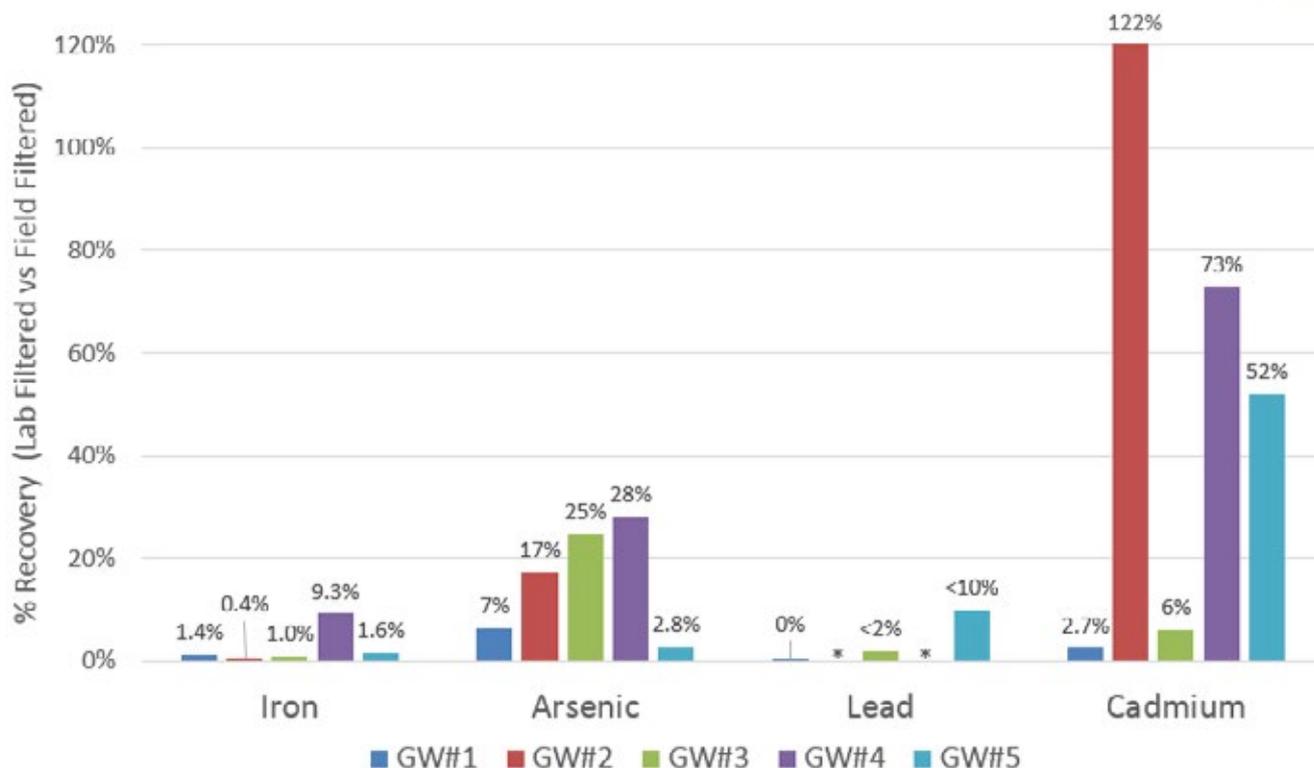


Figure 1- ALS study results for Arsenic, Lead, Cadmium, and Iron

* GW#2 and GW#4 were n.d. for Pb

Recommended Field Filtration Techniques

For small sampling volumes or low flow sampling, ALS recommends the use of syringe filtration for convenient filtration of dissolved metals samples in the field. Disposable syringes and syringe filters that have been proofed for suitability and/or recommended by the laboratory are essential and low cost syringe filters and syringes are available that are also suitable for ultra-trace level metals analysis. Using a 60 mL disposable syringe and syringe filter is very convenient, especially when used with smaller 60 mL sample bottles, which minimizes the amount of sample to be filtered, and also reduces the likelihood of having to use multiple filters for highly turbid samples.

When larger volumes of water are required, inline filtration may be more cost and time effective, but careful attention to recommended rinsing volumes to reduce contamination from the filter itself is essential. ALS studies have shown that pre-rinsing these inline filters with a minimum of 1L greatly reduces the likelihood of contamination from the filter being seen at ultra-trace levels.



Typical Inline Filters

Typical Syringe with disposable Luer-lock style filter.



Field Filtration Blanks are Highly Recommended!

ALS highly recommends the collection of Filtration Blanks whenever field filtration is conducted, as a control of metals background levels that may be contributed by the filtration process. This is the only way to protect and control against contamination, which is a common problem with many types of filtration media. The laboratory can provide de-ionized water to facilitate the collection of filtration blanks.

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