ALS Laboratory Group
ANALYTICAL CHEMISTRY & TESTING SERVICES

Environmental Division

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Ferrous Iron – extended holding times plus sampling protocols to avoid false positives.

Following a review of internationally recognised standards plus internal validation, ALS is pleased to confirm extended holding times for ferrous iron determination in waters effective immediately. This will result in the holding time moving from 24 hours to seven days.

Background

The Environmental industry regularly requests analyses for Ferrous Iron in ground waters. This data is useful in evaluating the status and change with respect to biological or chemical oxidation of organic analytes from TPH and BTEX to chlorinated solvents. Ferrous Iron is, in particular, often analysed as part of a suite of tests commonly referred to as "Natural Attenuation" or "Monitored Natural Attenuation Parameters".

Field Preparation

Historically, ALS has requested clients to field filter samples for Ferrous Iron, into small plastic bottles with minimal headspace and that these be preserved with Hydrochloric Acid. Based upon APHA holding times, which ALS has followed, these samples have then been rushed to the laboratory for immediate analysis, something that puts pressure on consultants and the laboratory and makes holding time compliance difficult for remote sites. This past practice has also added complexity from a sampling and logistics perspective and has contributed to less efficient sampling and submission to laboratories.

New Preparation Procedures and Holding Times

Under the new standard being followed by ALS, samples will be similarly prepared in that they **must** be field filtered into the existing ALS containers. They should also be presented with zero headspace just as for VOCs as an added precaution. The reference international document used to support this change is ISO 5667-3, *"Water Quality-Sampling-Part 3: Guidance on the preservation and handling of water samples".* Samples collected and preserved under this new protocol will see the holding time recognised as **seven days** rather than 24 hours. This change is also based upon internal verification / validation test work performed by ALS which supported these extended holding times. The key here is that field filtration is mandatory and ZHE is beneficial – refer to the following page on false positives

Right solutions….
….Right partner

Avoiding False Positives in Ferrous Iron results

When determining Ferrous Iron, field filtration and preservation are both critical for different reasons as outlined below.

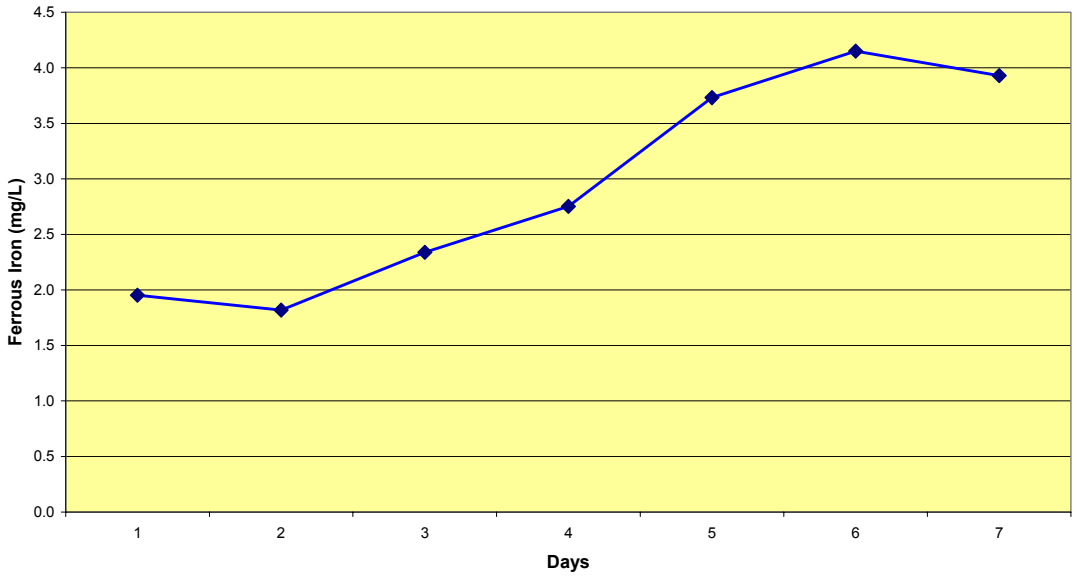
Preservation: The preservation with HCl (and Zero headspace) is designed to keep the iron in solution and avoid precipitation in the presence of any oxygen. If Iron does oxidise and precipitate in the container, even at low levels, this can prove difficult to re-dissolve. The result in this case can be false negative results. It is also interesting to note that this same precipitation can also cause false negatives if other metals are not field filtered e.g. Lead, Arsenic etc.

Filtration: The filtration is also critical but for a different reason. If a groundwater sample containing even traces of sediment is placed in an acidified sample bottle the acid preservative can start to dissolve the iron from any suspended sediment. Given that most sediment contains Iron, the net effect is an increasing Iron result over time and this can lead to FALSE positive results for ferrous Iron. This incorrect protocol often shows up with ferrous Iron results being higher than total Iron results on a report. This usually occurs when the dissolved metals have been field filtered into a nitric acid preserved bottle (correct protocol) for dissolved metals, but the ferrous Iron sample is unfiltered.

How much can field procedures influence my results?

The key point to note when determining Ferrous Iron is that all samples behave differently depending upon their redox potential and the individual components. The following data shows the effect of not field filtering a sample for Ferrous Iron Determinations. Even more important in this case, was the fact that when filtered immediately (day 1) into the correct preservative no ferrous Iron was found in this sample at all. This sample saw slight increases over time. ALS has observed many cases where Ferrous Iron results are significantly higher than Dissolved Iron results simply due to field preparation.

Ferrous Iron over time on an Unfiltered Ground Water sample.



For further information please contact your local ALS team.