ENVIRONMENTAL

# Sensitive Determination of Iron in Drinking Water, Mineral Water, Groundwater, and Spring Water Using Rapid Photometric Tests

Katrin Schwind, Application Scientist, Analytical Point-of-Use R&D, **katrin.schwind@merckgroup.com**

Gunter Decker, Senior Global Product Manager, Analytical Point-of-Use Analytics | Photometry, **gunter.decker@merckgroup.com**

The quality of drinking water is regulated by a variety of guidelines, such as the EU Council Directive 98/83[1,2] and WHO guideline.[3] The key principles used to define these limits consider both health hazards and sensory and technical reasons. Iron, for example, does not exhibit a risk for health in concentrations usually found in drinking water.[2,3] However, increased concentrations of iron result in the formation of iron hydroxide products, which can form deposits in water pipe systems and a brown discoloration of the water.[4]

To ensure the supply of clear and colorless water, country-specific limits have been set for drinking water. The limit for iron set by the EU directive is 0.2 mg/L Fe,[2] while the U.S. EPA specifies 0.3 mg/l Fe.[5] To prevent the formation of iron deposits in water pipe systems, a limit of 0.02 mg/L should not be exceeded.[6] To ensure that the specified limits are met, drinking water is, in many cases, subjected to a treatment step in which the iron is precipitated. This method virtually eliminates any iron content, reducing the iron concentration to the lower ppb range.[6]

## Analytical methods

Highly sensitive analytical methods for trace level quantification include flame atomic absorption spectroscopy (flame AAS, F-AAS) and optical emission spectrometry with inductively coupled plasma (ICP-OES). Depending on the dosage volume, the measuring range of the F-AAS method according to DIN EN ISO 38406-32 is 0.002–0.020 mg/L Fe. The limit of quantification (LOQ) for the ICP-OES method according to DIN EN ISO 11885 is 0.002 mg/L Fe.[7,8] In our lab an LOQ of 0.0007 mg/L Fe is achieved by ICP-MS according to the ICH Q2 standard.

### Analysis of iron using analytical test kits (rapid photometric methods)

A practical alternative for swift, sensitive results without investment in expensive instruments are rapid photometric methods. Test kits are generally characterized by their ease of use and speed of the procedure. The choice of the method depends on the application, the measuring range, and the required accuracy. In the case of iron, two sensitive photometric methods can be chosen.

The determination of iron using the 1,10-phenanthroline method according to APHA 3500-Fe B and DIN 38406-1 enables photometric measurement down to a level of 0.01 mg/L, which is entirely sufficient for many samples.[9]

If lower LOQs are required, the triazine method can be chosen. In this method, all iron ions are reduced to iron (II) ions. These react in a thioglycolate-buffered medium containing a triazine derivative to form a red-violet complex, which is subsequently determined photometrically.[10] Using a 100 mm cell and the Prove 600 UV-VIS spectrometer, LOQs for iron as low as 0.0025 mg/L can be achieved. Due to iron removal treatment and the naturally low iron content of most drinking water, preference should be given to the more sensitive triazine method. The Spectroquant® Iron Test (Cat. No. 114761) has an overall measuring range of 0.0025-5.00 mg/L Fe. In the Spectroquant® photometers, the methods are pre-programmed, so no time-consuming calibration curve must be created.

### Sample preparation and performance of the measurement with Spectroquant® Iron Test

Samples must first be acidified with nitric acid to stabilize the iron, while carbonic acid-containing samples must also be degassed in an ultrasonic bath. A detailed description of the measurement procedure is given in the application “Sensitive Measurement of Iron in Water”.[11]

### Method comparison of ICP-MS vs. Spectroquant® Iron Test

The iron content of five different mineral waters was determined by Spectroquant® test kit and ICP-MS. All samples were below the LOQ of the respective method (0.0007 mg/L for ICP-MS, 0.0025 mg/L for Spectroquant® test kit.

The five samples were spiked with iron at three different concentration levels by standard addition, and the respective recovery rates were determined by the photometric method. The results are shown in **Table 1** and **Figure 1**.

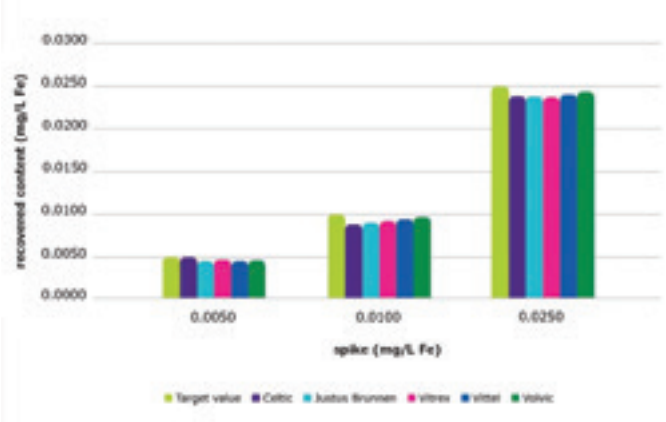
The added concentrations of iron were accurately recovered. The recovery rates in the spiked samples ranged between 89% and 99% over all experiments, with an average recovery rate of 95%.

(continued on next page)

## Table 1. Iron content recovered after standard addition

| Mineral water | Addition [mg/L Fe] | Recovered concentration [mg/L Fe] | Recovery rate |
|---|---|---|---|
| **Celtic** natural | 0.0050 | 0.0050 | 99% |
| | 0.0100 | 0.0089 | 89% |
| | 0.0250 | 0.0239 | 96% |
| **Justus Brunnen** medium | 0.0050 | 0.0046 | 91% |
| | 0.0100 | 0.0091 | 91% |
| | 0.0250 | 0.0239 | 96% |
| **Vitrex** natural | 0.0050 | 0.0048 | 95% |
| | 0.0100 | 0.0093 | 93% |
| | 0.0250 | 0.0238 | 95% |
| **Vittel** natural | 0.0050 | 0.0046 | 91% |
| | 0.0100 | 0.0095 | 95% |
| | 0.0250 | 0.0241 | 97% |
| **Volvic** natural | 0.0050 | 0.0047 | 93% |
| | 0.0100 | 0.0098 | 98% |
| | 0.0250 | 0.0244 | 98% |

**Figure 1**: Results of the standard addition

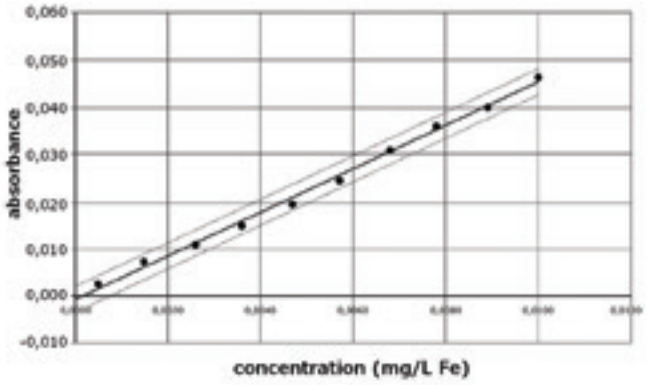



An even higher accuracy can be achieved by a custom calibration curve. **Table 2** shows the performance characteristics of the pre-programmed method for Cat. No. 114761 determined according to DIN 38402 A51 and ISO 8466-1 compared with a manually made calibration curve for the measurement range 0.0005 – 0.0100 mg/l Fe using the photometric test kit. The calibration curve is shown in **Figure 2**.

At 4.35%, the coefficient of variation of the custom calibration curve is 3.3 times higher than that of the pre-programmed method. This is due to the fact that at these lower concentrations, the deviations have a stronger relative effect in the custom calibration. Seen in absolute terms, the custom calibration procedure provides considerably lower method errors, as shown by the values of the method standard deviation and the method confidence interval for P=95%, which are 13 to 14 times lower than those of the pre-programmed method.

In the case of the standard additions, the use of such a custom calibration resulted in a further enhancement of the recovery rate, which now achieved a mean value of 101%. The individual values are between 95% and 106% (see **Table 3**).

**Figure 2:** Calibration curve for the measuring range 0.0005–0.0100 mg/L Fe




## Table 2: Comparison of performance characteristics

| | Pre-programmed method 0.0025 – 0.5000 mg/L Fe | Custom calibration 0.0005 – 0.0100 mg/L Fe |
|---|---|---|
| Method standard deviation [mg/L] | ± 0.00328 | ± 0.00023 |
| Method coefficient variation [%] | ± 1.31 | ± 4.35 |
| Confidence interval (P=95 %) [mg/L] | ± 0.0079 | ± 0.0006 |

## Table 3: Iron content recovered after standard addition with custom calibration

| Mineral water | Addition [mg/L Fe] | Recovered concentration [mg/L Fe] | Recovery rate |
|---|---|---|---|
| **Celtic** natural | 0.0050 | 0.0053 | 106% |
| | 0.0100 | 0.0095 | 95% |
| | 0.0250 | 0.0255 | 102% |
| **Justus Brunnen** medium | 0.0050 | 0.0049 | 97% |
| | 0.0100 | 0.0097 | 97% |
| | 0.0250 | 0.0255 | 102% |
| **Vitrex** natural | 0.0050 | 0.0051 | 102% |
| | 0.0100 | 0.0099 | 99% |
| | 0.0250 | 0.0254 | 102% |
| **Vittel** natural | 0.0050 | 0.0049 | 97% |
| | 0.0100 | 0.0102 | 102% |
| | 0.0250 | 0.0257 | 103% |
| **Volvic** natural | 0.0050 | 0.0050 | 99% |
| | 0.0100 | 0.0105 | 105% |
| | 0.0250 | 0.0261 | 104% |

Since mineral waters have only low iron content, the experiments were also carried out using samples of groundwater and spring water, whose iron concentrations are naturally higher due to the lack of any water treatment. The measurement was carried out using the pre-programmed method. Here again the measurement results were verified by reference analysis using the ICP-MS method. **Table 4** shows a comparison of the results obtained with the two methods.

**Table 4: Iron content of groundwater and spring water – comparison of ICP-MS and Spectroquant® Iron Test 114761**

| | Concentration [mg/L Fe] | |
|---|---|---|
| Groundwater and spring water | ICP-MS | Spectroquant® Iron Test 114761 |
| Spring water Bad König | 0.0047 | 0.0041 |
| Spring water Höchst Himmelsleiter | 0.0043 | 0.0051 |
| Spring water Breitenbrunn | 0.0022 | < 0.0025 |
| Spring water Vielbrunn | 0.0017 | < 0.0025 |
| Spring water Rai-Breitenbach | 0.0059 | 0.0051 |
| Groundwater Bensheim | 2.70 | 2.71 |

The results yielded by the Spectroquant® Iron Test are in agreement with those obtained using the ICP-MS method. Due to the very high iron content of the Bensheim groundwater sample of 2.7 mg/L Fe, in deviation from the defined procedure, a 10 mm cell was used. The recovery rate here was 100%. These results show that even very high concentrations of iron can be precisely determined by means of the iron test.

In the case of the low iron concentrated spring water samples, the measurement results differed only by a maximum value of 0.0008 mg/L. Even those iron concentrations that are below the LOQ of the photometric method were confirmed by the ICP-MS measurements.

## Summary

The Spectroquant® Iron Test offers a good alternative to ICP or AAS when it comes to determining the iron content in drinking water, mineral water, groundwater, and spring water. The method yields results comparable to those obtained by the ICP-MS method and is easy to perform. For all laboratories for which the purchase of an ICP-OES or ICP-MS system is inexpedient for economic reasons, the Spectroquant® Iron Test Cat. No. 114761 offers a swift, sensitive, and precise alternative for the determination of the iron content of drinking water, mineral water, groundwater, and spring water.

### Chemicals, samples, and instruments used:

All measurements were conducted using a Prove 600 photospectrometer. The reference system was a Thermo Fisher Scientific HR-ICP mass spectrometer (method on the Element 2 device).

### Featured products

| Description | Cat. No. |
|---|---|
| Spectroquant® Prove 600 UV/VIS spectrophotometer 1,8 nm spectral bandwidth | 173018 |
| Spectroquant® Iron Test 0.0025-5.00 mg/L Fe | 114761 |
| Iron Standard Solution CertiPUR® 1000 mg/l in 0.5 mol/l $HNO_3$ | 119781 |
| Nitric acid 65% for analysis EMSURE® ISO | 100456 |
| Water Ultrapur | 101262 |

To read more about the Spectroquant® line for Spectrophotometric Analysis visit us at **SigmaAldrich.com/spectroquant**

#### References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