

Iron (total, soluble, ferrous) - Phenanthroline Method

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Applications and Industries

Industrial wastewater influent and effluent, industrial process waters, surface and ground water, seawater, drinking water. Power generation industry.

References

APHA Standard Methods, 23rd ed., Method 3500-Fe B - 1997
ASTM D 1068-77, Iron in Water, Test Method A.
J.A. Tetlow and A.L. Wilson, "The Absorptometric Determination of Iron in Boiler Feed-water", Analyst, Vol. 89, p. 442 (1964).

Chemistry

Ferrous iron reacts with 1,10-phenanthroline to form an orange-colored complex in direct proportion to the ferrous iron concentration.

Soluble iron consists primarily of ferrous iron but can also include a small portion of ferric iron.

The **soluble iron reagent** contains hydroxylamine hydrochloride which converts any dissolved ferric iron to ferrous.

The **ferrous iron reagent** does not contain hydroxylamine hydrochloride and will not measure soluble ferric iron.

Total iron (ferric + ferrous) is determined by adding a solution of thioglycolic acid and ammonia (A-6000 Activator Solution) to the sample. This solution dissolves most forms of particulate iron and converts ferric iron to ferrous. Certain forms of very insoluble iron (magnetite, ferrite, etc.) require a digestion prior to analysis.

Results are expressed as ppm (mg/L) Fe.

Storage Requirements

Products should be stored in the dark and at room temperature.

Sampling and Preservation

For soluble and ferrous iron, analyze sample immediately upon collection. For total iron, analyze sample at the time of collection if possible. Otherwise, adjust the sample pH to less than 2 with nitric or hydrochloric acid. If the pH of the preserved sample is less than 1, adjust to pH 2-3 prior to analysis.

Available Analysis Systems

Visual colorimetric: CHEMets®, HR CHEMets®

Instrumental colorimetric: Vacu-vials®

Interference Information

- While most forms of particulate iron are measured with these tests when the total iron procedure is performed, certain forms of insoluble iron (magnetite, ferrite, etc.) require a digestion. Contact tech@aquaphoenixsci.com for details.
- Iron chelated with EDTA is not measured quantitatively with this test.
- Strong oxidizing agents may interfere. To minimize these interferences, excess hydroxylamine hydrochloride has been added to the soluble iron reagent. The ferrous iron reagent does not contain hydroxylamine hydrochloride.
- Zinc at levels greater than ten times that of iron as well as chromium, cobalt, copper, and nickel have the potential to interfere. Cadmium, silver, mercury, and bismuth may precipitate the reagent. Excess phenanthroline has been added to the reagents to minimize interferences from these metals.
- Cyanide and polyphosphates may interfere.
- Molybdate may precipitate the reagent in the test ampoule, causing low results for soluble and ferrous iron. Upon addition of thioglycolic acid solution (A-6000) during total iron analysis, samples containing molybdate will turn blue.
- Nitrite may cause a false negative result during soluble and ferrous analysis. Nitrite is also a significant interference during total analysis. Samples containing nitrite will turn yellow, orange, or red upon addition of thioglycolic acid solution (A-6000). This sample background color creates a color mismatch between the test ampoule and the comparator color standards. A sample pre-treatment procedure is available that will minimize nitrite interference, refer to the Technical Bulletin called *Total Iron Analysis In the Presence of Nitrite* which can be accessed by clicking on the Technical Information tab on the K-6010/K-6003/K-6210/K-6203.
- Sulfide and ammonia do not interfere.
- If necessary, sample pH should be adjusted to fall within the range of 2 - 7.



- Sample color or turbidity may make a color match difficult during visual colorimetric testing and may cause a false positive result with instrumental colorimetric tests. CHEMetrics' Sample Zeroing Accessory Pack can be used to correct for potential errors during instrumental analysis.

Shelf Life

When stored in the dark and at room temperature:

Visual colorimetric:

CHEMetrics® refills, Activator Solution, color comparators: at least 1 year

Instrumental colorimetric:

Vacu-vials® kits: at least 1 year

Accuracy Statement

Statements of accuracy are based on laboratory tests performed under ideal testing conditions using standards of known concentration prepared in deionized water.

CHEMetrics® kits: ± 1 color standard increment

Vacu-vials® kits K-6003 and K-6023:

- ≤ 0.08 ppm at 0 ppm
- ± 0.09 ppm at 0.30 ppm
- ± 0.30 ppm at 1.50 ppm
- ± 0.45 ppm at 4.50 ppm

Safety Information

Safety Data Sheets (SDS) are available upon request and at www.sdsfetch.com. Read SDS before using these products. Breaking the tip of an ampoule in air rather than water may cause the glass ampoule to shatter. Wear safety glasses and protective gloves.