

Iron in Soils

Photometric determination subsequent to acid extraction

Introduction

The determination of iron in soil is important as iron content contributes to soil characteristics such as color and composition and is used in the differentiation of soil types. Iron oxides, such as hematite, can significantly influence soil color, particularly in soils with low organic matter content. Iron in soil can exist in different forms, including free and non-free iron, with free iron further classified into crystalline and amorphous forms. These variations influence soil properties and are relevant for soil characterization.¹

A range of analytical methods is available for iron determination, including atomic absorption spectroscopy, ion chromatography, inductively coupled plasma techniques, and ultraviolet-visible spectrophotometry. Among these, spectrophotometric methods are widely used due to their selectivity, sensitivity, simplicity, and cost-effectiveness.²

Experimental

This application note details the photometric determination of iron in soils after its extraction.

Method

In a buffered medium iron(II) ions react with a color reagent (depending on test kit, for **1.14761** and **1.14549** a triazine derivative; for **1.14896** 2,2'-bipyridine; for **1.00796** 1,10-phenanthroline is used) to form a red or red-violet complex that is determined photometrically.

The 1,10-phenanthroline method is analogous to APHA 3500-Fe B and DIN 38406-1.

Measuring Range

Spectroquant® Iron Cell Test (1.14549)	Test kit: 0.05–4.00 mg/L Fe Method: 1.00–80.0 mg/kg Fe
Spectroquant® Iron Cell Test (1.14896)	Test kit: 1.0–50.0 mg/L Fe Method: 20.0–1000 mg/kg Fe
Spectroquant® Iron Test (1.14761)	Test kit: 0.005–5.00 mg/L Fe Method: 0.10–100 mg/kg Fe
Spectroquant® Iron Test (1.00796)	Test kit: 0.010–1.000 mg/L Fe Method: 2.00–20.00 mg/kg Fe

Applicable Sample

Soil samples

Reagents, Instruments and Materials

Test/Reagent Kit(s)

For the measurement one of the following Spectroquant® test kits is necessary:

- Spectroquant® Iron Cell Test (**1.14549**)
- Spectroquant® Iron Cell Test* (**1.14896**)
- Spectroquant® Iron Test (**1.14761**)
- Spectroquant® Iron Test (**1.00796**)

* not compatible with Move 100

Instrument(s) & Devices

For the measurement one of the following Spectroquant® photometers is necessary:

- Spectroquant® VIS Spectrophotometer Prove 100 plus (**1.73026**)
- Spectroquant® UV/VIS Spectrophotometer Prove 300 plus (**1.73027**)
- Spectroquant® UV/VIS Spectrophotometer Prove 600 plus (**1.73028**)
- Spectroquant® Colorimeter Move 100 (**1.73632**)

Note: Also, legacy Spectroquant® instruments are suitable.

Software for Data transfer

- Optional Spectroquant® Prove Connect to LIMS software package (**Y.11086**) to transfer your data into an existing LIMS system.

Instrument Accessories

If a reagent test (not cell test) is used, choose one of the following cells depending on the measuring range.

- Rectangular cells 10 mm (**1.14946**)
- Rectangular cells 20 mm (**1.14947**)
- Rectangular cells 50 mm (**1.14944**)

Other Reagents and Accessories

- Hydrochloric acid 25% for analysis (**1.00316**)
- Hydrochloric acid 1 mol/L for analysis (**1.09970**)
- Sodium hydroxide solution 10% for analysis (**1.05588**)
- MQuant® pH-Indicator strips pH 0–6 (**1.09531**)
- Water for analysis (**1.16754**)
- Analytical balance
- Standard laboratory glassware (e.g., glass beakers) and pipettes
- Shaker or stirring plate
- Spatula
- Sieve
- pH-meter
- Charcoal activated for soil test
- Folded filter

Analytical Procedure

For more information on the measurement see the packaging insert of the test

Sample Preparation

- In a glass beaker place 10 g of air-dried and sieved (mesh 2 mm) sample, with 100 mL hydrochloric acid 1 mol/L and a spatula-tip full of charcoal activated for soil tests and mix well for 1 hour. The pH value should be 1–2 (check with pH-indicator strips). If necessary, adjust with hydrochloric acid 25%.
- Afterwards adjust the pH value of the suspension to 3.0 with sodium hydroxide solution 10%, using a pH meter.
- Then remove the undissolved matter by filtration through a folded filter and wash the filter residues 3 times with water for analysis.
- Collect the filtrate and the washings in a 200-mL volumetric flask, finally fill up to volume with water for analysis and mix well.

Using Cat. No. 1.14549: Procedure and Measurement

For more information on the measurement see the packaging insert for the test.

Procedure

- Pipette 5.0 mL Pretreated sample into a reaction cell and mix.
- Add 1 level blue microspoon (in the cap of the Fe-1K bottle) Reagent Fe-1K, close the cell tightly, and shake vigorously until the reagent is completely dissolved.
- Leave to stand for 3 min (reaction time), then measure the sample in the photometer.

Measurement

- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 3.2–4.5.
- The color of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.

Hints for Measurement

- It is recommended to zero the method each new working day. To do this, open the method, either by manually selecting the method or by inserting a barcoded cell. Tap the <Settings> button and select the <ZERO ADJUSTMENT> menu item. After prompting, insert the 16 mm zero cell through the corresponding opening. The zero adjustment is performed automatically. Confirm the performance of the zero-adjustment procedure by clicking on <OK>.
- After the zero has been performed, insert the barcoded Spectroquant® round cell through the corresponding opening, ensuring that the white position mark on the cell is aligned with the positioning mark on the spectrophotometer. The measurement starts automatically.
- Read off the result in mg/L from the display.

Hint: The above written measurement description is only valid for the Spectroquant® Prove (plus) series photometer. If a different instrument is used, please consult the corresponding instrument manual for more details on how to perform the measurement.

Using Cat. No. 1.14896: Procedure and Measurement

For more information on the measurement see the packaging insert for the test.

Procedure

- Pipette 1.0 mL Pretreated sample into a reaction cell, close the cell, and mix.

- Add 1 dose Reagent Fe-1K, close the cell tightly, and shake vigorously until the reagent is completely dissolved.
- Leave to stand for 5 min (reaction time A), then measure the sample in the photometer

Measurement

- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 5.5–6.5.
- The color of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.

Hints for Measurement

- It is recommended to zero the method each new working day. To do this, open the method, either by manually selecting the method or by inserting a barcoded cell. Tap the <Settings> button and select the <ZERO ADJUSTMENT> menu item. After prompting, insert the 16 mm zero cell through the corresponding opening. The zero adjustment is performed automatically. Confirm the performance of the zero-adjustment procedure by clicking on <OK>.
- After the zero has been performed, insert the barcoded Spectroquant® round cell through the corresponding opening, ensuring that the white position mark on the cell is aligned with the positioning mark on the spectrophotometer. The measurement starts automatically.
- Read off the result in mg/L from the display.

Hint: The above written measurement description is only valid for the Spectroquant® Prove (plus) series photometer. If a different instrument is used, please consult the corresponding instrument manual for more details on how to perform the measurement.

Using Cat. No. 1.14761: Procedure and Measurement

For more information on the measurement see the packaging insert for the test.

Procedure

- Pipette 5.0 mL Pretreated sample into a test tube.
- Add 3 drops Reagent Fe-1 and mix. Hold the bottle vertically while adding the reagent!
- Leave to stand for 3 min (reaction time), then fill the sample into the cell, and measure in the photometer.

Notes:

- For measurement in the 50-mm cell, both the sample volume as well as the quantity of reagent Fe-1 must be doubled. It is recommended to measure against an own prepared blank sample (preparation as per measurement sample, but with distilled water instead of sample) to increase the accuracy. Configure the photometer for blank measurement.

Measurement

- Certain photometers may require a blank (preparation as per measurement sample, but with distilled water instead of sample).
- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 3.2–4.5.
- The color of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.

Hints for Measurement

- It is recommended to zero the method for each new working day. To do this, open the method by inserting the barcode, tap the <Settings> button and select the <ZERO ADJUSTMENT> menu item. Fill the same cell which will be used for the sample measurement with distilled water. After prompting, insert the filled rectangular cell into the cell compartment. The zero adjustment is performed automatically. Confirm the performance of the zero-adjustment procedure by clicking on <OK>.
- After the zero adjustment, fill the measurement sample into the same or a matched rectangular cell and insert the cell into the cell compartment. The measurement starts automatically.
- Read off the result in mg/L from the display.

Hint: The above written measurement description is only valid for the Spectroquant® Prove (plus) series photometer. If a different instrument is used, please consult the corresponding instrument manual for more details on how to perform the measurement.

Using Cat. No. 1.00796: Procedure and Measurement

For more information on the measurement see the packaging insert for the test.

Procedure

- Pipette 8.0 mL Pretreated sample into a test tube.
- Add 1 drop Reagent Fe-1 and mix. Hold the bottle vertically while adding the reagent!
- Add 0.50 mL Reagent Fe-2 with a pipette and mix.
- Add 1 dose Reagent Fe-3 and shake vigorously until the reagent is completely dissolved.
- Leave to stand for 10 min (reaction time), then fill the sample into the cell, and measure in the photometer.

Notes:

- When using the 50-mm cell is recommended to measure against an own prepared blank sample (preparation as per measurement sample, but with distilled water instead of sample) to increase the accuracy.

Measurement

- Configure the photometer for blank measurement.
- Certain photometers may require a blank (preparation as per measurement sample, but with distilled water instead of sample).
- For photometric measurement the cells must be clean. Wipe, if necessary, with a clean dry cloth.
- Measurement of turbid solutions yields false-high readings.
- The pH of the measurement solution must be within the range 3.5–4.0.
- The color of the measurement solution remains stable for at least 60 min after the end of the reaction time stated above.
- In the event of iron concentrations exceeding 50 mg/L, other reaction products are formed and false-low readings are yielded. In such cases it is advisable to conduct a plausibility check of the measurement results by diluting the sample (1:10, 1:100).

Hints for Measurement

- It is recommended to zero the method for each new working day. To do this, open the method by inserting the barcode, tap the <Settings> button and select the <ZERO ADJUSTMENT> menu item. Fill the same cell which will be used for the sample measurement with distilled water. After prompting, insert the filled

rectangular cell into the cell compartment. The zero adjustment is performed automatically. Confirm the performance of the zero-adjustment procedure by clicking on <OK>.

- After the zero adjustment, fill the measurement sample into the same or a matched rectangular cell and insert the cell into the cell compartment. The measurement starts automatically.
- Read off the result in mg/L from the display.

Hint: The above written measurement description is only valid for the Spectroquant® Prove (plus) series photometer. If a different instrument is used, please consult the corresponding instrument manual for more details on how to perform the measurement.

Analytical Quality Assurance

Analytical quality assurance (AQA) is recommended before each measurement series.

To check the photometric measurement system (test reagents, measurement device, handling) and the mode of working, the iron standard solutions (see section 5 of the respective packaging insert) or a diluted iron standard solution containing 25.0 mg/L Fe³⁺ or Spectroquant® CombiCheck 90 can be used. Besides a standard solution with 1.00 mg/l Fe, this article also contains an addition solution for determining sample-dependent interferences (matrix effects).

Sample-dependent interferences (matrix effects) can be determined by means of standard addition or dilution.

To view additional notes, visit [SigmaAldrich.com/qa-test-kits](https://www.sigmaaldrich.com/qa-test-kits).

Calculation

Iron content in mg/kg Fe = analysis value in mg/L Fe x 20

References

1. Hu, C.; Guo, H.; Mei, H.; Zhu, J. Prediction of Iron Content in Soil Based on Microspectrophotometry Analysis. *Forensic Sci. Int.* **2021**, *318*, 110600. DOI: [10.1016/j.forsciint.2020.110600](https://doi.org/10.1016/j.forsciint.2020.110600).
2. Nurchi, V. M.; Cappai, R.; Spano, N.; Sanna, G. A Friendly Complexing Agent for Spectrophotometric Determination of Total Iron. *Molecules* **2021**, *26* (11), 3071. DOI: [10.3390/molecules26113071](https://doi.org/10.3390/molecules26113071).

Featured Products

Description	Cat. No.
Test Kits	
Spectroquant® Iron Cell Test	1.14549
Spectroquant® Iron Cell Test	1.14896
Spectroquant® Iron Test	1.14761
Spectroquant® Iron Test	1.00796
Instruments & Accessories	
Spectroquant® VIS Spectrophotometer Prove 100 plus	1.73026
Rectangular cells 10 mm	1.14946
Rectangular cells 20 mm	1.14947
Rectangular cells 50 mm	1.14944
Reagents & Test Strips	
Hydrochloric acid 1 mol/L for analysis	1.09970
Hydrochloric acid 25% for analysis	1.00316
Sodium hydroxide solution 10% for analysis	1.05588
MQuant® pH-Indicator strips pH 0–6	1.09531
Water for analysis	1.16754

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Description	Cat. No.
Instruments & Software	
Spectroquant® UV/VIS Spectrophotometer Prove 300 plus	1.73027
Spectroquant® UV/VIS Spectrophotometer Prove 600 plus	1.73028
Spectroquant® Colorimeter Move 100	1.73632
Spectroquant® Prove Connect to LIMS software package	Y.11086
Reference Materials	
Spectroquant® CombiCheck 90	1.18700
Iron standard solution, 0.0500 mg/L Fe ³⁺	1.33014
Iron standard solution, 0.1000 mg/L Fe	1.33018
Iron standard solution, 0.300 mg/L Fe	1.33019
Iron standard solution, 1.00 mg/L Fe	1.33020
Iron standard solution Certipur®, 1000 mg/L Fe ³⁺	1.19781
Reagents, Test Strips & Accessories	
Nitric acid 65% for analysis EMSURE®	1.00456
Spectroquant® Crack Set 10C	1.14688
Spectroquant® Crack Set 10	1.14687
Empty cells 16 mm with screw caps (25 pcs)	1.14724
MQuant® Iron Test, measuring range 3–500 mg/L Fe ²⁺	1.10004
MQuant® Universal indicator strips pH 0–14	1.09535
Nitric acid Titrisol® for 1 mol/L	1.09966
Sodium hydroxide solution 1 mol/L Titripur®	1.09137
Hydrochloric acid 1 mol/L Titripur®	1.09057
Thiourea GR for analysis	1.07979

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