

UV/Vis Spectroscopy

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Water Analysis Using LAMBDA UV/Visible Spectrophotometers: Iron Determination

Introduction

Iron is rarely found in its elemental form in nature due to the high tendency

of its ions, Fe(II) and Fe(III), to form oxygen and sulphur containing compounds. Concentrations of iron found in surface waters are typically no greater than 1 mg/L, unless contaminated by industrial effluents, whilst much higher concentrations are found in ground waters. The World Health Organization guideline for iron in drinking water is 0.3 mg/L as undesirable bacteria growth in water systems occurs above this concentration.¹

In this application, the quantitative analysis of iron was performed using the LAMBDA 265™ UV/Vis spectrophotometer and CHEMetrics iron cell test kit.

Principle

In aqueous solution, Fe(II) (ferrous iron) reacts with 1,10-Phenanthroline to form an orange-red complex (tri-o-phenanthroline iron (II) ion) which can be detected spectrophotometrically at 505 nm. Thioglycolic acid solution is added to reduce Fe(III) (ferric ions) to their ferrous state in order to determine total iron concentration and interferences from other metals are minimised with the cell reagent formulation. This method is suitable for the concentration range of 0 – 6.00 mg/L iron in water allowing its concentration to be determined without the use of a calibration curve by incorporating the measured absorbance at 505 nm into a known equation.

Reagents and Apparatus

1. CHEMetrics iron Vacu-vials® kit (K-6203) – containing 30 vials, reference sample, sample cup and A-6000 activator solution
2. PerkinElmer LAMBDA 265 PDA UV/Visible spectrophotometer
3. UV Lab™ software
4. Iron standard solution (10 +/- 0.1 mg/L)
5. Deionised (DI) water
6. Volumetric flasks (100 ml)
7. Micropipettes

Method

A stock solution of iron (10.00 mg/L) in water was used to prepare a 3.00 mg/L iron solution in a 100 ml volumetric flask by dilution with DI water.

Following preparation of solutions, the sample cup was filled with the 3.00 mg/L iron solution up to the 25 mL mark. The tip of the Vacu-vial ampule was placed in the sample cup, snapped, and the ampule then inverted several times to promote mixing. The ampule was dried and left to stand for one minute and the absorbance measured in the spectrophotometer. This technique was also carried out for the reference sample supplied in the test kit. In order to calculate the total iron concentration in a real sample, a further step would involve adding five drops of A-6000 activator solution and waiting four minutes before snapping the ampule tip.

Using the UV Lab software, the LAMBDA 265 instrument parameters were set, as shown in Figure 1, to measure the absorbance at 505 nm. An equation was set up to calculate the ferrous iron concentration as shown in Equation 1, which is also applicable for calculating total iron concentration. Following measurement of the blank, the absorbance of the known iron solution in the Vacu-vial was recorded.

Equation 1.

$$\text{Ferrous iron concentration (mg/L)} = 6.35(A_{505}) - 0.03$$

Results

Figure 2 shows spectra from 5 repeat runs of the 3.00 mg/L iron sample, with the results shown in Table 1. The mean absorbance at 505 nm was determined to be 0.496, which corresponded to a calculated concentration of 3.12 mg/L ferrous iron. The results obtained had a high level of accuracy and repeatability with a relative standard deviation of 0.21%.

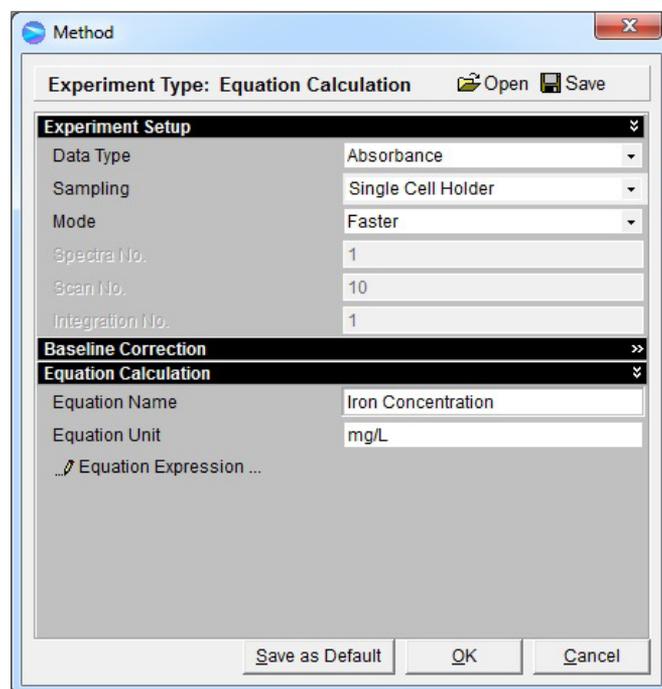


Figure 1. Instrument parameters and method setup.

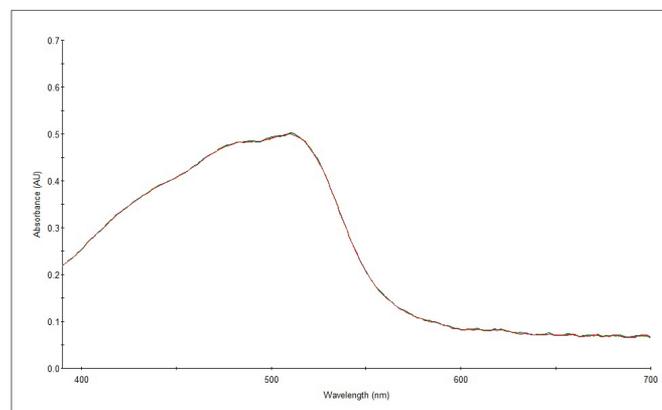


Figure 2. Overlaid UV/Vis spectra of repeat measurements of iron solution.

Table 1. Results for repeat measurements.

Iron Solution	Absorbance at 505 nm	Ferrous Iron Concentration (mg/L)
Repeat 1	0.496	3.12
Repeat 2	0.495	3.11
Repeat 3	0.497	3.13
Repeat 4	0.496	3.12
Repeat 5	0.497	3.13

Conclusion

Quantitative analysis of ferrous iron in water was achieved rapidly, with minimal sample preparation and no calibration standards, using the CHEMetrics iron test kit. Simple determination of total iron concentration can be achieved by implementing an extra step in the method. Results obtained showed a high level of repeatability and accuracy using the LAMBDA 265 UV/Vis spectrophotometer and UV Lab software, whilst providing immediate results.

References

1. http://www.who.int/water_sanitation_health/dwq/chemicals/iron.pdf Date accessed: 09/11/2015