

# SIMULTANEOUS SPECTROPHOTOMETRIC DETERMINATION OF ALUMINIUM AND IRON USING MICELLAR AIDED MIXED CHROMOGENIC REAGENTS

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## Abstract

Simple and novel spectrophotometric method is described for simultaneous determination of aluminium and iron. The method is based on the metal ion - complexes formed by aluminium and iron with mixed chromogenic reagents of orthophenanthroline and 8-hydroxyquinoline (oxine). The derivatives have absorption maxima at 400 nm and 510 nm respectively in the presence of cetyltrimethylammonium bromide (CTAB) as micellar media (surfactant) maintained at pH 5. Reaction conditions were optimized and the linear dynamic ranges for determination of aluminum and iron were found to be 0.8 - 12 µg/mL and 0.6 - 8 µg/mL respectively. The standard deviation (S.D) and coefficient of variation (CV) for the simultaneous determination was 0.018 and 0.45% for aluminium and 0.03 and 1.5% for iron (II). The recoveries were between 102 % and 106 %, 101.2 % and 104 % for aluminium and iron respectively at 95 % confidence level. The proposed method was successfully applied to the assay of aluminium and iron in rock minerals, lubricating oil and water samples. The results and paired t-test for the analyzed samples were found to be in satisfactory agreement (shows no significant difference) with those acquired by the flame atomic absorption spectrophotometric (FAAS) technique.

**Keywords:** Mixed chromogenic reagent, micellar media, Aluminum, Iron.

## Introduction

The determination of trace amounts of aluminium and iron is important in industry, food products, potable water, metallurgy and environmental analytical chemistry. There is a growing need for procedures with high sensitivity and selectivity for the determination of aluminium and iron, because they have important roles in biological sciences and industry.<sup>[1]</sup>

The role of trace and toxic levels of elements in human health has become an important area of scientific research and hence much time and resources have been put into the development of analytical techniques for single or simultaneous multicomponent metals quantitation.<sup>[2]</sup>

These two elements, iron and aluminium are usually present together in real and synthetic samples. Therefore, it is important to measure them in the presence of each other. Among the most widely used analytical methods are those based on the UV-visible spectrophotometric techniques, due to both the simplicity and rapidness of the method.<sup>[3-5]</sup>

Most techniques available for simultaneous assaying of aluminium and iron usually involve complex instrumentation, long analysis time and huge capital outlay. For these reasons, there is a growing need for simple, rapid, low cost and sensitive techniques for the simultaneous quantitation of aluminium and iron. Spectrophotometric methods offer these advantages.

This research work was aimed at developing a spectrophotometric technique capable of simultaneous determination of aluminium and iron. Since these metals occur together in environmental samples, biological and industrial wastes, it is important to measure them in the presence of each other. This work explores the possibility of the method herein being used and well suited for the routine analytical simultaneous determination of these metals.

## Materials and Method

UV-Visible absorbance spectra were collected on a UV-VIS Spectronic D20+ spectrophotometer, using a 5 cm quartz cell. Measurements of pH were made with a Metrohm hand held pH-meter.

Flame atomic absorption spectrophotometry was carried out using Perkin-Elmer AA Analyst with deuterium arc background correction. All solutions were prepared with doubly distilled water. Chemicals used were of analytical grade.

Prepared samples solution containing different concentrations of Al and Fe were prepared by taking aliquots of the working standard solutions of the metals and mixed thoroughly.

Under optimized conditions and at the respective wavelengths of maximum absorption of the derivatives, 2 mL of prepared mixtures of iron and aluminium was measured into 25 mL volumetric flasks and then treated with 2 mL of acetate buffer solution, 1mL ascorbic acid, 1mL of micellar solution and 2 mL of the mixed chromogenic reagent. The absorbance values were taken against reagent blank. Fe was taken at 510nm after 5 minutes and Al was taken at 400nm after 15 minutes. The concentrations of the metals in the prepared samples were evaluated from standard calibration curves of Al and Fe standards respectively. The method of standard addition was also used to determine the metals concentrations for samples with complex matrix.

## Results

### Optimization of Analytical Variables

Various factors and conditions were optimized using the method of one at a time and the results are shown in Table 1. These conditions were found and were used to validate the applicability of the proposed method in the determination of iron and aluminium in real and complex environmental samples as shown in Table 2, 3 and 4.

Table 1. Shows selected analytical parameters obtained from optimization.

Parameter	Studied range	Selected value
Wavelength (nm)	300-700	400 (Al) and 510 (Fe)
pH	3-8	5
Time/h	0-72	5 mins (Fe) and 15 (Al)
Mixed reagent (mL)	1-5	2
Micellar solution (CTAB)	0.1-2.0/1-3	1/2
Concn/vol (%/mL)		
Linear range (µg/mL)	0.1-12	0.6-8 (Fe) and 0.8-12 (Al)
Mean recovery (%)	-	100.03 (Fe) and 96.5(Al)
Standard deviation	-	0.030 (Fe) and 0.018 (Al)
Coefficient of variation	-	1.5 (Fe) and 0.45 (Al)
Regression coefficient (R <sup>2</sup> )	-	0.9972 (Fe) and 0.998(Al)
Interferences		Cu (II) and Fe (III)

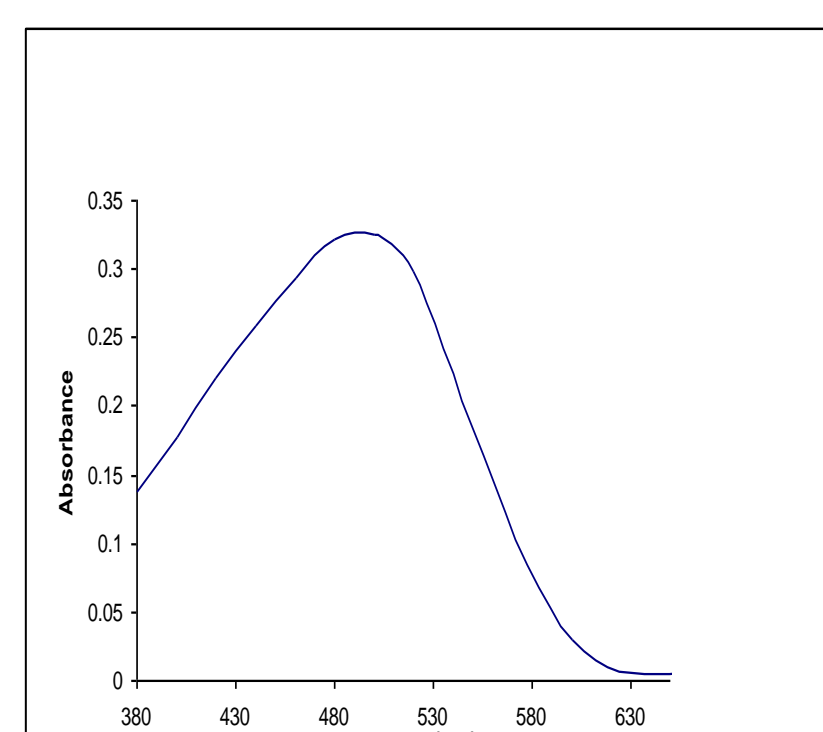


Fig. 1 Absorption spectra of Iron-Phen complex at pH 5 in micellar media

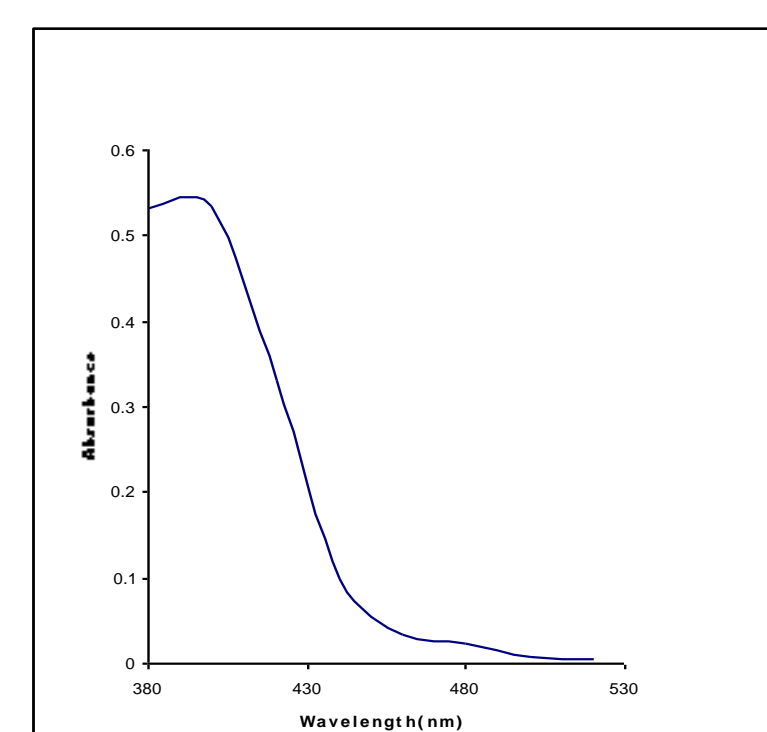


Fig. 2 Absorption spectra of Aluminium-Oxine complex at pH 5 in micellar media

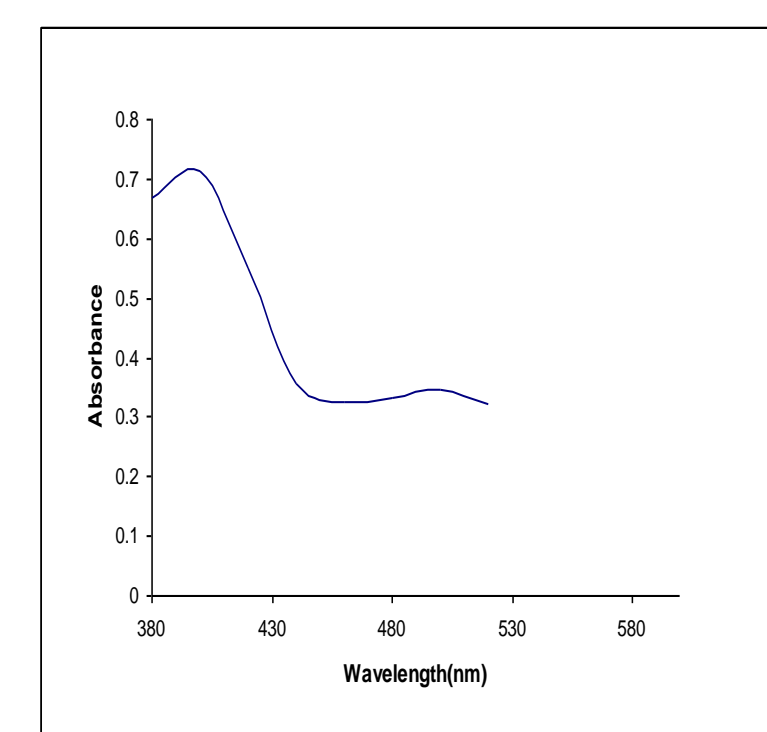


Fig. 3 Absorption spectra of Aluminium and Iron mixture complexes at pH 5 in micellar media

Table 2. Determination of Aluminium and Iron in real samples by the proposed method and comparison with other method.

Sample	No		Found (µg/mL)			
			Proposed Method		AAS	
	Al	Fe	Al	Fe	Al	Fe
1*	1.06 ± 0.013	3.42 ± 0.052	0.996	3.592		
2**	ND	0.1 ± 0.013	ND	0.166		
3***	3.26 <sup>#</sup> ± 0.028	7.22 <sup>#</sup> ± 0.182	3.15	7.522		

\*\*\* Rock Sample

\*\* Lube Oil Sample

\* Lagoon Water Sample

<sup>#</sup> Mean value of three replicate determinations.

<sup>#</sup> Measured in µg/g.

Table 3. Reproducibility of the proposed method.

Present (µg/mL)		Found (µg/mL)	
Al <sup>3+</sup>	Fe <sup>2+</sup>	Al <sup>3+</sup>	Fe <sup>2+</sup>
4.00	2.00	4.01	1.98
4.00	2.00	3.98	2.00
4.00	2.00	4.02	1.97
4.00	2.00	3.99	2.02
4.00	2.00	4.02	2.01
Mean (Found)		4.004	1.996
Standard Deviation		0.018	0.030
Coefficient of Variation		0.45	1.50

Table 4. Recovery Studies of the proposed method

Added amount(µg/mL)		Found(µg/mL)		Recovery (%)	
Al	Fe	Al	Fe	Al	Fe
5.00	3.00	4.88	2.85	97.6	95.0
1.00	1.00	0.98	1.05	98.0	105.0
2.50	6.00	2.47	6.01	98.8	100.1
Mean Recovery				96.5	100.03

## Discussion

The linear ranges were determined which was 0.6 - 8 µg/mL for iron (II), and 0.8 - 12 µg/mL for aluminium (III). The wavelengths of maximum absorption for the respective metal ion - complexes were found to be 400 nm and 510 nm for Al and Fe (II) complexes in the presence of micellar solution of cetyltrimethyl ammonium bromide (CTAB). The relative standard deviation and coefficient of variation show that the method has good accuracy and precision. A paired t-test was done for replicate determinations by the proposed method and the established flame atomic absorption spectrophotometric method (FAAS) and the result did not vary significantly. Good recoveries were achieved for both metal ions; Fe was between 102 % and 106 %, while Al was between 101.2 % and 104 %.

In order to check the accuracy of the method, several mixtures prepared using different concentration ratios of Al (III) and Fe (II) were analyzed using the proposed method. The metal ions were determined in five replicate mixtures, the standard deviation (SD) and coefficient of variation (CV) were found to be 0.018 and 0.45% for Al (III) and 0.03 and 1.5% for Fe (II) as shown in Table 3. The good agreements between amount present and amount found for different compositions of the binary mixtures as shown in Table 4 are indicative of satisfactory accuracy and the precision of the proposed method. Interference studies carried out show no significant interferences from anions of sulphate, phosphates and chlorides. Amongst metals tested, only Cu(II) and Fe(III) show little interferences.

## Conclusion

The determination of aluminium and iron in rock samples, lubricating oil sample and water samples and the close agreements between the established method and the atomic absorption spectrophotometric method may indicate the wide applicability and reliability of the method. This shows that the method can be well adapted for analyses of complex environmental and biological samples. The high degree of reproducibility, valuable dynamic range, simplicity and relatively short experimental period of the proposed method permit the handling of a large number of samples. This research reports the simultaneous spectrophotometric determination of total iron and aluminium with the use of mixed chromogenic reagents, thereby increasing the array of methods that are available for the simultaneous determination of aluminium and iron. The proposed method has low cost, simplicity and green process as added advantages.

## References

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