

In the Name of Allah

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**INVESTIGATION ON KINETIC METHODS FOR
DETERMINATION OF SINGLE AND
SIMULTANEOUS MULTICOMPONENT SPECIES**

To:
My parents, my wife
and my daughters

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Abstracts

The determination of iron (III) based on its highly catalytic effect on the oxidation of indigo carmine with periodate in the presence of 1,10-phenanthroline as an activator is described. The reaction was followed spectrophotometrically by measuring the rate of change in absorbance of indigo carmine at 612 nm, pH 2 and 25 °C. The acquired data based on the initial rate and fixed time are processed. Iron (III) can be determined in the range 5-200 ng.mL⁻¹. The limit of detection and relative standard deviation are 2 ng.mL⁻¹ and 1.5% respectively. The effect of foreign ions on the determination of iron (III) is also discussed. The proposed method is highly sensitive and was applied to the determination of iron (III) in blood serum samples.

A sensitive kinetic method for determination of vanadium is reported at pH 2 and 25 °C. The catalytic effect of vanadium on the oxidation of indigo carmine by bromate goes through an induction period and then decreases in absorbance, at 612 nm, which is the maximum absorption wavelength of indigo carmine. Both the induction period and slope methods were applied for determination of vanadium. Concentration ranges of 0.3-30 (µg.mL⁻¹) of vanadium were determined. The limit of detection was found to be 0.27 (µg.mL⁻¹). Six replicate analysis of a sample solution containing 10 (µg.mL⁻¹) vanadium gave a relative standard deviation of 1.3%. The interference effects of various cations and anions on vanadium determination is reported, and the interference of ions are eliminated by a

simple procedure. Application of the method to real samples such as crude petroleum are performed which gave acceptable results.

Vanadium (V) and iron (III) can be determined simultaneously at pH 2 and 25 °C by a single experiment using their kinetic effect on the oxidation of indigo carmine by bromate which goes through an induction period and then decreases in absorbance, at λ_{max} , 612 nm. The rate of the color-fading of indigo carmine is proportional to the concentration of vanadium and is independent of the concentration of iron. The length of the induction period of the reaction is related to the concentration of iron and is independent of the concentration of vanadium. Concentrations of 0.3-2 ($\mu\text{g.mL}^{-1}$) vanadium (V) and 6-12 ($\mu\text{g.mL}^{-1}$) iron (III) were determined with mean relative errors of 2.7 and 1.6%, respectively. The interference effects of various cations and anions on determination of mixtures of vanadium and iron is reported. Application of the method to real samples and several mixtures of standard solutions are performed which gave acceptable results.

A kinetic-spectrofluorimetric technique for the determination of the cationic surfactant cetylpyridinium chloride by variable time method is proposed. The method is based on the retarding effect of surfactant cetylpyridinium chloride on the oxidation reaction of Rhodamine 6G by periodate in the presence of Mn^{2+} as catalyst at pH 6 and 40 °C. Cetylpyridinium chloride is determined in the range 1-15 $\mu\text{g mL}^{-1}$ and detection limit 0.4 $\mu\text{g mL}^{-1}$. Relative standard deviation is 1.5% for 10 $\mu\text{g mL}^{-1}$ of surfactant. The proposed method was applied to the direct

determination of the surfactant cetylpyridinium chloride in synthetic and real samples which gave acceptable results.

Finally the complex formation reaction of iodine with hexamethylenetetramine (HMTA) is studied spectrophotometrically in different solvents and varying temperatures. Based on the observed spectral behavior the resulting 2:2 molecular complex between the reactants was formulated as $[(\text{HMTA})_2 \dots \text{I}^+]\text{I}_3^-$. The formation constant in different solvents of the resulting molecular complex is calculated from the computer fitting by Microsoft EXCEL SOLVER using absorbance-mole ratio data. The enthalpy and entropy of complexation reaction with HMTA is determined from the temperature dependence of the formation constant. It is found that the complexation reaction are enthalpy stabilized, but entropy destabilized.

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