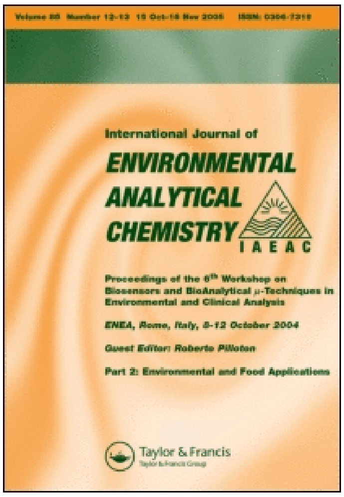

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Spectrophotometric Determination of 8-Hydroxyquinoline (Oxine) in Aqueous Solution by Azo Dye Formation with Diazotized *p*-Aminoacetophenone

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Spectrophotometric Determination of 8-Hydroxyquinoline (Oxine) in Aqueous Solution by Azo Dye Formation with Diazotized *p*-Aminoacetophenone

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A sensitive spectrophotometric method for the determination of oxine in aqueous solution is investigated. It is based on the reaction of the analyte with diazotized *p*-aminoacetophenone to form, in the presence of sodium hydroxide, an intense cherry-red water-soluble and stable azo dye which shows maximum absorption at 520 nm. The linear absorbance plot with the concentration indicates that Beer's law is adhered over the range 10–100 μg of oxine in a final volume of 25 ml, i.e., 0.4–4 ppm, with a molar absorptivity of $3.37 \times 10^4 \mathrm{l\,mol^{-1}\,cm^{-1}}$, a relative error of +0.44 to −1.12%, and a relative standard deviation of 0.54–2.9%, depending on the level of analyte concentration. The optimum conditions affecting the color reaction and interference due to foreign organic compounds have been examined.

KEY WORDS: Oxine determination, aqueous solution, azo dye formation, diazotized *p*-aminoacetophenone reagent, spectrophotometry.

INTRODUCTION

The determination of oxine is important from the point of view of pollution.[1] For the determination of oxine at the microlevel, spectrophotometric methods are the most suitable and yet the reported procedures[1-4] seem to be too elaborate for routine use. Therefore, a method for the spectrophotometric trace characterization of oxine seems desirable. The present investigation is devoted to such an approach.

EXPERIMENTAL

Reagents

Stock oxine solution, 10 mg ml^{-1}. 1.0000 g of recrystallized oxine is dissolved in absolute ethanol and the volume is adjusted to 100 ml, in a volumetric flask, with the same solvent. A 50 μg ml^{-1} oxine solution is prepared by dilution with distilled water.

Diazotized *p*-aminoacetophenone reagent solution, 5 mM. This solution is prepared as described before.[5]

Sodium hydroxide solution, 1 N. This solution is prepared by dilution of the concentrated BDH solution.

Apparatus

The same as those used before.[5]

Procedure

To a series of 25-ml volumetric flasks, transfer an aliquot of the aqueous sample solution containing 10–100 μg of oxine. Add about 10 ml of distilled water, 0.5 ml of diazotized *p*-aminoacetophenone reagent solution, 1.5 ml of sodium hydroxide solution, and dilute to the mark with distilled water. Mix and allow the reaction mixture to stand for 10 minutes. Measure the absorbances against the reagent blank solution at 520 nm using 1-cm cells. A straight-line calibration curve passing through the origin is obtained, with a molar absorptivity,[6] of 3.37×10^4 l mol^{-1} cm^{-1}, and an average sensitivity index of 0.0043 μg cm^{-2}.

RESULTS AND DISCUSSION

Absorption spectra

The absorption spectrum obtained on treating oxine according to the recommended procedure is shown in Figure 1. The maximum absorption at 520 nm, characteristic of the cherry-red azo dye, is adapted in all subsequent investigations.

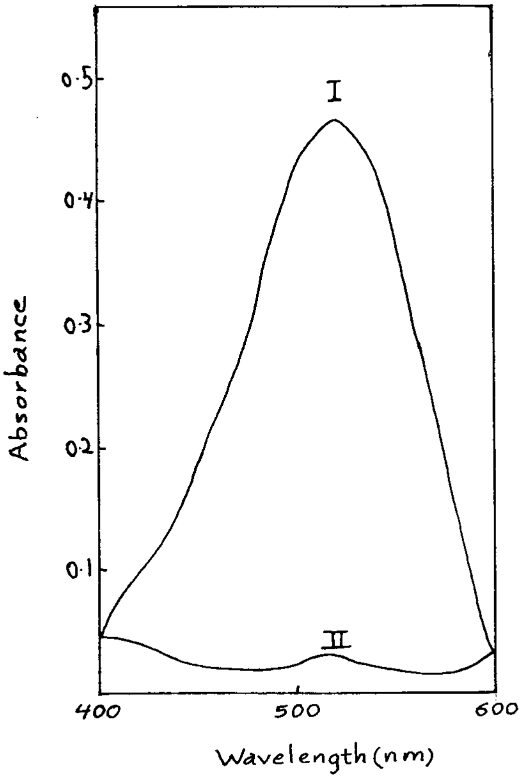


FIGURE 1 Absorption spectra of (I) 50 μg of oxine, treated as in the procedure, measured against reagent blank, and (II) reagent blank measured against distilled water.

Effect of diazotized reagent

Of the several diazotized reagents examined, diazotized *p*-aminoacetophenone is found to be the most useful. The reaction is observed to be complete on using 0.5–3 ml of 5 mM diazotized reagent solution. 0.5 ml of the prescribed reagent concentration is selected for the recommended procedure.

Effect of alkali

The presence of alkali is essential for developing the colored azo dye. Of the many alkalis tested, sodium hydroxide is the alkali giving the best results, and with 1.5 ml of 1 N solution the color formed retains maximum intensity and stability.

Order of addition of reagents

For optimum results, the order cited in the procedure has to be followed, otherwise the sensitivity decreases.

Rate of reaction

In spite of the rapid color formation, the reaction mixture has to stand for 10 minutes to attain full color formation, after which the absorbance remains stable for about 1 hr.

Accuracy and precision of the method

Under the above conditions, the accuracy and precision of the method are checked. The results (5 determinations) are compiled in Table I.

TABLE I

Accuracy and precision of the method.

Oxine taken/μg	Relative error %	Relative standard deviation % ($n=5$)
10	+1.96	±2.9
75	−1.12	±1.4
100	+0.44	±0.54

Interferences

In order to realize the analytical applications of the method, the effect of foreign compounds is examined. The results are shown in

TABLE II

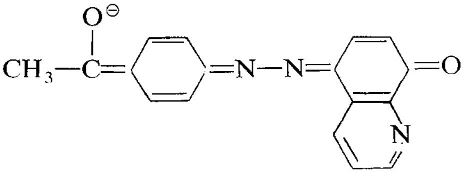
Effective of diverse compounds on oxide (50 μg) determination.

Interferent	Amount added/μg	Interference %
m-Aminobenzoic acid	100	+0.4
p-Aminobenzoic acid	100	+0.9
4-Aminopyridine	100	−1.4
4-Aminosalicylic acid (sodium salt monohydrate)	100	+2.1
Benzoic acid	100	−1.3
EDTA ($Na_2H_2Y \cdot 2H_2O$)	100	−1.1
Hippuric acid	100	−2.2
1-Hydroxy-2-naphthoic acid	100	+3.6
1-Naphthylamine-4-sulfonic acid (sodium salt)	100	+2.1
p-Nitroaniline	100	−0.7
Picolinic acid	100	−1.9
Salicyclic acid	100	+1.8

Table II. Compounds tested at the 100 μg-level and found to interfere seriously are: 1-naphthol, 2-naphthol, aminophenols, resorcinol, and 1-naphthylamine.

Nature of the dye

Oxine (as phenol) couples with diazonlum salts. The two possible positions of attack are the 5- and 7-positions. However, because of steric consideration, the 5-position may be more favorable. Job's method of continuous variation shows that the dye has a composition of 1:1 (oxine to diazotized *p*-aminoacetophenone), ondicating that a mono azo dye is formed, for which structure suggested may be as follows:



The formation constant of the dye in aqueous solution, under the conditions of determination, is found to be 13×10^5. The dye is extracted into amylalcohol and isoamyl alcohol. After acidification, the dye is extracted into benzene, carbon tetrachloride, and chloroform where it shows a yellow color.

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