



ORIGINAL ARTICLE

Selective spectrophotometric determination of some primary amines using 2,4-dinitrofluorobenzene reagent



Theia'a N. Al-Sabha *, Intisar A. Hamody

Chemistry Department, College of Education, Mosul University, Mosul, Iraq

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Abstract A spectrophotometric method is developed for the quantitative determination of some primary aliphatic and aromatic amines, i.e., allyl amine, 1,5-diaminopentane, 1,6-diaminohexane, cyclohexylamine, *m*-aminophenol, benzidine and *p*-phenylenediamine. The method is based on the interaction between these amines and 2,4-dinitrofluorobenzene (DNFB) reagent. The spectra of the products show maximum absorption that ranged from 355–357 nm and 366–377 nm with molar absorptivities that ranged from 1.086×10^4 – 6.398×10^4 and 7.566×10^3 – 1.581×10^4 l/mol cm for aliphatic and aromatic primary amines, respectively. Beer's law is obeyed in the concentration range of 0.25–8.0, 1.0–10, 0.25–2.50, 1.0–8.0, 2.0–20, 1.0–12.0 and 1.0–10.0 µg/ml for the above mentioned amines, respectively, and the mean percent recoveries ranged between 97.8% and 103.3% with precision (RSD) better than 5.5% for all the amines under study. In addition, the stability constant has been determined and the mechanism is proposed for the DNFB-amine products.

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1. Introduction

A great number of aromatic amines are of considerable importance in industrial, toxicological and pharmaceutical aspects (Kirk-Othmer, 1979). Short-chain aliphatic amines are presented widely in the aquatic environment due to their wide spread use in several industrial, chemical and manufacturing

applications (Lloret et al., 2004; Eller et al., 2007). Also; these amines are common components of biological systems as degradation products of organic materials, such as amino acids and proteins. In addition to hygienic problems due to stinky smell, these compounds may be hazardous to human health as they are sensitizers and irritants to skin, eyes, mucus membranes and respiratory tract. In addition; they can react with certain nitrogen-containing compounds to form nitrosamines, which are potentially carcinogenic substances (Namiesnik et al., 2003).

Several spectrophotometric methods using various reagents have been described for the determination of aliphatic and aromatic amines. The interaction of *n*-alkylamine with chloranil has been investigated (Dwivedi and Banga, 1981), which in-

* Corresponding author. Tel.: +964 7701647599.
 E-mail address: dr_theiaa@yahoo.co.uk (T.N. Al-Sabha).
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volves the participation of electron donor–acceptor complex between the primary aliphatic amines and chloranil resulting in mono and di-substituted products. Primary, secondary and tertiary amines, both aliphatic and aromatic amines, are shown to react with *p*-chloranil and form blue to purple colour in dioxane/2-propanol (1:4 v/v) (Smith and Davis, 1984) or used as a selective reagent for the determination of primary amines in aqueous solution (Al-Sabha, 1984). Also, 2,3-dichloro-5,6-dicyano-1,4-benzoquinone (DDQ) (Al-Sabha, 1997) and *p*-fluoranil (Hanna, 1986) have been used as π -acceptors for the determination of primary aliphatic and aromatic amines as *n*-donors in aqueous medium. Some new 9-substituted 10-methyl acridinium trifluoromethanesulfonate salts have been synthesized and shown to react in methanol with aniline and *n*-butylamine to form derivatives which are absorbed at 445 and 439 nm, respectively (Dunning and Stewart, 1991). Sodium 1,2-naphthoquinone-4-sulphonate in the presence of a non-ionic surfactant and carbonate buffer solution is used for the determination of primary and secondary amines at 45 °C (Cabeza et al., 1994), also it was used for the determination of some drugs containing the primary amino group (Iskender and Sagirli, 2000). Terephthaloylacetylene forms an yellow or orange coloured product with primary and secondary amines and may be used for their detection (Tiwari et al., 2002). Quinoline-2-(carboxaldehyde) has been used as a reagent for the spectrophotometric detection of primary amines (You-Zung et al., 1990). *N*-Hydroxysuccinimidyl-naphthapyrone acetate has been utilized for the determination of primary and secondary amines at pH 8.5 which give the corresponding derivatives within 5 and 15 min (Liu et al., 2001). 9-Chloroacridine reagent (Steward et al., 1969; Steward and Lotti, 1970) has also been used for the determination of primary aromatic amines. This method needs heating the analytical solution and requires an excess of acridine reagent in order to achieve the reproducible results. *m*-Dinitrobenzene as π -acceptor has been used for the determination of several aliphatic amines as *n*-donors in acetone medium and the formation of charge transfer complex has been proposed to explain the spectroscopic behaviour of the mixtures (Siddiqi and Pathania, 2003). Most of the above methods suffer from several disadvantages that (a) they cannot be applied for all types of primary aliphatic and aromatic amines, (b) there is a lack of sensitivity and selectivity and (c) the estimation of these compounds is applied in the organic medium.

The present work describes a simple and sensitive spectrophotometric method for the analyses of some primary aliphatic and aromatic amines. The method is based on the formation of DNFB-amine derivative from the amine and DNFB.

2. Experimental

2.1. Apparatus

All absorption measurements were made on a Shimadzu UV-210A double-beam spectrophotometer supplied with a digital printer DP80Z and matched 1-cm optical silica cells. Heating of solutions was carried out on a water bath of frost instruments, LTD. The reading of pH was made on a PW 9420 pH meter supplied with an electrode type CE 10–12 pH. Weighing was carried out on a balance type of Mettler H 54 AR.

2.2. Reagents

Analytical grade chemicals, acetonitrile and distilled water were used. Standard solutions of primary aliphatic amines (250 μ g/ml) in acetonitrile were prepared and aromatic amines (100 μ g/ml) were dissolved in a small amount of absolute ethanol and diluted with distilled water. These amines are supplied by FLUKA & BDH companies. 0.01 M of DNFB (Sigma Co.) was prepared in acetonitrile for the determination of aliphatic amines or by dissolving in a small amount of absolute ethanol and diluting with distilled water for the determina-

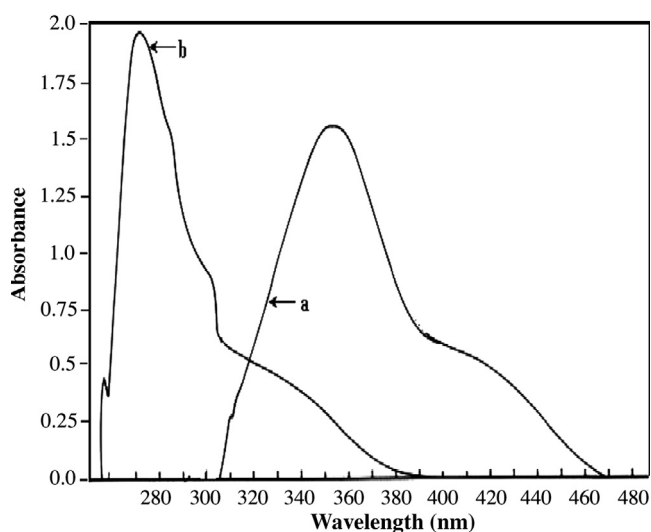


Figure 1 Absorption spectra of 15 μ g/5 ml 1,6-diaminohexane with DNFB against reagent blank (a) and blank against acetonitrile (b) plotted under the general procedure conditions.

Table 1 Wavelengths, bases and the optimum amounts of base and DNFB for the studied amines.

Compounds	λ_{\max} (nm)	Base	Base amount 10^{-2} M (ml)	DNFB 10^{-2} M (ml)
1,5-Diaminopentane	355	–	–	0.2
Allylamine	353	–	–	0.2
1,6-Diaminohexane	353	NaOH	0.5	0.6
Cyclohexylamine	357	–	–	0.4
Benzidine	377	NaHCO ₃	0.4	3.0
<i>m</i> -Aminophenol	366	CH ₃ COONa	1.4	1.5
<i>p</i> -Phenylenediamine	375	NaHCO ₃	1.0	1.0

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