Arabian Journal of Chemistry (2010) **3**, 33–38

King Saud University

Arabian Journal of Chemistry

www.ksu.edu.sa
www.sciencedirect.com

ORIGINAL ARTICLE

Kinetic spectrophotometric determination of hyoscine butylbromide in pure form and in pharmaceutical formulations

Ayman A. Gouda

Chemistry Department, Faculty of Science, Zagazig University, Zagazig, Egypt

Received 27 June 2009; accepted 21 July 2009
Available online 23 December 2009

KEYWORDS

Kinetic determination;
Hyoscine butylbromide;
Potassium permanganate;
Pharmaceutical analysis

Abstract A simple and sensitive kinetic method was described for the determination of hyoscine butylbromide in pharmaceutical preparations. The method is based upon a kinetic investigation of the oxidation reaction of the drug with alkaline potassium permanganate at room temperature for a fixed time of 15 min. The absorbance of the colored manganate ion was measured at 610 nm. The absorbance–concentration plot was rectilinear over the range of 1.0–10 μg mL^{-1} ($r = 0.9999$) and detection limit of 0.092 μg mL^{-1}. The concentration of hyoscine butylbromide was calculated using the corresponding calibration equation for the fixed-time method. The determination of hyoscine butylbromide by the fixed-concentration and rate constant methods is also feasible with the calibration equations obtained but the fixed-time method has been found to be more applicable. The different experimental parameters affecting the development and stability of the colors were carefully studied and optimized. The proposed method was applied to the determination of hyoscine butylbromide in pharmaceutical formulations. The results obtained were in good agreement with those obtained using the official British Pharmacopeial method (2004).

1. Introduction

Hyoscine butylbromide (HBB) (1S,3s, 5R, 7S,8r)-6,7-epoxy-3-[(S)-(3-hydroxy-2-phenylpropiony)oxy]-8-butyl-8-methyl-8-azoniabicyclol [3.2.1] octane bromide is used as an antispasmodic in treating peptic ulcer, gastritis and various disorders of the gastrointestinal tract which are characterized by spam. It has also found employment for the relief of spasmodic conditions of the bile duct and urinary tract and for the treatment of dysmenorrhoea (Crossland, 1980) (Fig. 1).

Hyoscine butylbromide is a white, crystalline powder or colorless crystals, efflorescent, freely soluble in water, soluble in alcohol. It melts at about 197 °C with decomposition,

E-mail address: aymanchimca@yahoo.com

1878-5352 © 2009 King Saud University. All rights reserved. Peer-review under responsibility of King Saud University.
doi:10.1016/j.arabjc.2009.12.006

Production and hosting by Elsevier

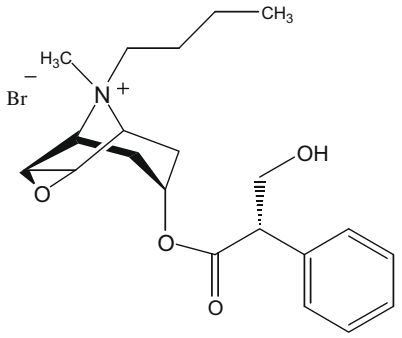

Figure 1 Chemical structure of hyoscine butylbromide (HBB).

determined after drying *in vacuo* for 24 h and then at 100–105 °C for 2 h and it should be protected from light (Neil Maryadele, 2001; British pharmacopoeia, 2004).

Due to the vital importance of HBB determination in pharmaceutical preparations and in biological fluids, many analytical techniques have been reported in the literature. HBB has been determined in pharmaceutical preparations using titrimetric (British pharmacopoeia, 2004; Falco and Vianna, 1979), spectrophotometric (Taha and Gomaa, 1975; Taha et al., 1974; Taha and Ruecker, 1997; Abdelkader et al., 1980; Issa et al., 2005; Thomos et al., 1994; Davidson and Hassan, 1984; Mahrous et al., 1992; Sharaf El-Din, 1994; Erk and Onur, 1996; Erk, 1996; Abramovich et al., 1993; Issopoulos and Pavlouzervou, 1994; Toral et al., 2005), high performance liquid chromatographic (Karali et al., 1998; Huller et al., 1993; Nakagawa et al., 2000; Wang and Zhu, 2000; Mandal et al., 1991; Pohjola and Harpf, 1994; Issopoulos and Pavlouzervou, 1994; Lau and Mok, 1997; Parissipoulou and Panderi, 1999), capillary electrophoresis (Cherkaoui et al., 1999; Chang et al., 2000) and electrochemical methods (Abadia et al., 1982; Ionescu et al., 1983; Cheng and Gao, 1983; Buschmann, 1987; Li et al., 1988; Ganjali et al., 2003, 2004), among other methods.

The literatures are still poor in analytical procedures based on kinetics especially for pharmaceutical or biological fluids. Furthermore, some specific advantages in the application of kinetic methods can be expected (Espinosa-Mansilla et al., 1998):

1. Selectivity due to the measurement of the evolution of the absorbance with the time of reaction instead of the measure of a concrete absorbance value.
2. Possibility of no interference of the colored and: or turbidity background of the samples.
3. Possibility of no interference of other active compounds present in the commercial product if they are resisting the chemical reaction conditions established for the proposed kinetic method.

In the present work, kinetically based method was proposed for the determination of HBB by measuring the absorbance at 610 nm after oxidation reaction with alkaline $KMnO_4$. Although, the poor selectivity of the proposed oxidation method with $KMnO_4$, it is simpler than the time consuming HPLC methods and is more sensitive than the other spectrophotometric methods.

The aim of the present work was to study the reaction between HBB and potassium permanganate kinetically in an attempt to evaluate the drug in dosage forms. The results obtained were promising. The proposed method was simple and did not need sophisticated instrument or special skill.

2. Experimental

2.1. Apparatus

All absorption spectra were made using Kontron 930 (UV–Visible) spectrophotometer (German) with a scanning speed of 200 nm/min and a band width of 2.0 nm, equipped with 10 mm matched quartz cells.

2.2. Materials and reagents

All chemicals and materials were of analytical grade and all solutions were freshly prepared in bidistilled water.

Pure grade hyoscine butylbromide (HBB) was kindly supplied by Amriya Pharmaceutical Industries, Alexandria, Egypt. Its purity was found to be 100.28 ± 0.77 ($n = 5$) according to BP method (British pharmacopoeia, 2004).

The following commercial formulations were subjected to the analytical procedures: Nu-Spasm tablets (Egyptian International Pharmaceutical Industries Company (EIPICO), Tenth of Ramadan City, Egypt) labeled to contain 10 mg HBB/tablet. Farcorelaxin ampoules (Pharco Pharmaceuticals Company, Al Amriya, Alexandria, Egypt) labeled to contain 20 mg HBB/mL. Spasmocin ampoules (Memphis Company for Pharmaceutics and Chemistry, Cairo, Egypt) labeled to contain 20 mg HBB/mL.

A stock solution (100 μg mL^{-1}) was prepared by dissolving 10 mg of HBB in 100 mL of distilled water and further diluted with the same solvent as appropriate.

Potassium permanganate (Merck, Darmstadt, Germany): 5×10^{-3} M aqueous solutions, freshly prepared and standardized. Sodium hydroxide (BDH, UK): 0.5 M aqueous solution.

2.3. Recommended general procedures

Transfer aliquots (0.1–1.0 mL) of 100 μg mL^{-1} standard solution accurately measured, into series of 10 mL volumetric flasks; add 1.0 mL of 0.5 M NaOH followed by 2.0 mL of 5×10^{-3} M $KMnO_4$ to each flask and shake the mixture well. Allow the reaction mixture to stand for 15 min. Make up to the volume with bidistilled water. The HBB concentration was determined by measuring the rate of manganate formation at 610 nm as the tangent of the kinetic curve during the first 3.0 min of reaction and using the appropriate graphs. Log reaction rate versus log concentration of HBB was plotted to get order of the reaction. To get the standard calibration graph, the above procedure was carried out and the reaction mixture was allowed to stand for 15 min, where the absorbance of the reacting solution was measured at 610 nm against a blank solution prepared simultaneously. Plot the values of the absorbance against the final concentration in μg mL^{-1} to get the calibration graph. Alternatively, derive the regression equation.