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Modified carbon paste sensor for the potentiometric determination of neostigmine bromide in pharmaceutical formulations, human plasma and urine

A.F. Khorshid [a],*, Y.M. Issa [b]

[a] Pharmaceutical Analytical Chemistry Department, Faculty of Clinical Pharmacy, Nahda University, NUB, Beni-Sweff, Egypt
[b] Chemistry Department, Faculty of Science, Cairo University, Giza, Egypt

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ABSTRACT

A novel, simple, rapid, selective and sensitive method for the determination of neostigmine (Ns) ion in its bulk powder, different pharmaceutical dosage forms, and biological fluids (plasma and urine) using four modified carbon paste electrodes was developed. Sensor 1 is based on ion-association Ns-TPB, sensor 2 used Ns-PT, sensor 3 comprises a mixture of (Ns-PT+Ns-TPB) and sensor 4 was constructed using (Ns-PT+β-CD). Solvent mediator 2-NPPE exhibited a proper behavior including Nernstian slope ranging from 61.5 ± 0.5 to 64.5 ± 0.5 mV per decade over the pH range of 3.8–10 for the four sensors. Linear responses of Ns within the concentration range 1.0×10^{-7}–1.0×10^{-2} mol/L were obtained. The response time is very short (≤10 s) with a detection limit 6.3×10^{-8} M. In flow injection analysis (FIA), sensor 3 shows a Nernstian slope value 75.5 ± 0.5 mV per decade within the concentration range of 1×10^{-6}–1×10^{-2} mol/L and with a detection limit 7.5×10^{-7} mol/L. The utility of mixed or additives of β-CD had a significant influence on increasing the sensitivity of sensors 3 and 4 compared to sensors 1 and 2. The sensors were applied for the determination of neostigmine (Ns) ion in its bulk powder, different pharmaceutical dosage forms, and biological fluids (plasma and urine). The results obtained were satisfactory with excellent percentage recovery comparable with official method for the assay based on non-aqueous titration using perchloric acid as a titrant.

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1. Introduction

Neostigmine bromide [3-(dimethylcarbamoyloxy)-N,N,N trimethylanilinium bromide (mol. wt. 303.2)] is a quaternary amine that inhibits cholinesterase activity to prolong and intensify the muscarinic and nicotinic effects of acetylcholine. The anticholinesterase actions of neostigmine are reversible. It is used in anesthesia to reverse the neuromuscular blockade produced by competitive neuromuscular blockers. paralytic ileus, postoperative urinary retention and primary open-angle glaucoma (Martindale, 2009).

Several methods (Habib and Gan, 2006) have been reported for the determination of Ns–Br in dosage forms and in biological fluids: potentiometric titration (Diamandis and Christopoulos, 1983), spectrophotometry (Kulikov and Bokovikova, 1985; Sakai et al., 1999), thin-layer chromatography (Stanley et al., 1988), HPLC (Marunouchi et al., 2006; Yeh, 2000; Varin et al., 1999), gas-chromatography (Kokko, 1993), electrophoresis (Mazereeuw and Tjaden, 1994; Chen and Lee, 1998) and voltammetry (Matsue et al., 1987). Two ion-selective electrodes have been reported (Fan et al., 1988; Gupta et al., 2009) using a precipitation-based technique with Na tetraphenylborate as an anionic exchanger without incorporation of ionophores.

Host–guest chemistry, the host component is an organic molecule or ion, whose binding sites converge in the complex and the guest component, is any molecule or ion whose binding sites diverge in the complex. They have been previously applied as sensor ionophores in potentiometric ion-selective electrodes for the determination of drugs like propranolol (Sun et al., 2004; Ganjali et al., 2006), diclofenac (Shamsipur et al., 2005), glutathione (El-Kosasy et al., 2005), and surfactants e.g. sodium dodecylsulfate (SDS) (Fukui et al., 2003), dopamine (Lima and Montenegro, 1999), atropine (Li et al., 2005), zolpidemhemitartrate (Kelani, 2004), tetradecyltrimethylammonium ion and hexadecylpyridinium (Mahajan et al., 2004). The use of some additives in the construction of liquid membrane sensors for the potentiometric detection of different ionic species is described that impart best performance (Sil et al., 2001; Mikhelson et al., 1999; Okada et al., 1987). The reported methods have many draw backs of

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* Corresponding author.
E-mail addresses: amalkorshid@yahoo.com, amal.khorshed@nahdauniversity.edu.eg (A.F. Khorshid), yousrymi@yahoo.com (Y.M. Issa).

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analytical parameters or may need special treatment before analysis, or time consuming, etc.

Chemically modified carbon paste electrodes, CMCPEs, possess important advantages such as ease of preparation, or regeneration, and very stable response in addition to very low Ohmic resistance (Svancara et al., 2009). Therefore, CMCPEs have found direct application in a variety of analytical techniques, such as amperometry (Ozoemena et al., 2004; Malongo et al., 2008), voltammetry (Mashhadizadeh and Akbarian, 2009), and potentiometry (Mostafa and Homoda, 2008). These advantages drew the attention of researchers in recent years (Issa and Khorshid, 2011).

In the present work, four electrodes with different ion-exchangers and modifiers were constructed in an attempt to lower the detection limit and widen the concentration range. Several plasticizers, different ion-associations mixtures and cyclodextrin as additives were tried for the development of the sensors. These sensors were used for the determination of Ns–Br in bulk powder, different pharmaceutical dosage forms, and biological fluids.

2. Experimental

2.1. Chemicals and reagents

Neostigmine bromide, and its pharmaceutical dosage form Amostigmine tablets (15 mg/tablet), and Amostigmine injection (0.5 mg/mL) were provided by Amoun Pharmaceutical Co., Cairo, Egypt).

All chemicals and reagents used were of analytical reagent grade, and water was bi-distilled. Graphite powder, 2-nitrophenyl phenyl ether (2-NPPE), dioctyl phthalate (DOP), dibutyl phthalate (DBP), and tricresyl phthalate (TCP) were purchased from Aldrich. Phosphotungstic acid (PTA), sodium tetraphenylborate (NaTPB) and β-cyclodextrin were obtained from Sigma. The following compounds were used in the investigation of the selectivity of the electrode; chlorides of Na^+, K^+, Ca^{2+}, Mg^{2+}, $NH_4{}^+$, Ba^{2+}, Mn^{2+}, Zn^{2+}, Fe^{2+}, Co^{2+}, Ni^{2+}, Cu^{2+}, Cd^{2+}, Sr^{2+}, Cr^{3+}, Al^{3+}; and nitrate of Pb^{2+}; glucose, fructose, maltose, lactose, urea, Vit. C, thiamine HCl, pyridoxine HCl, physostigmine salicylate, pyridostigmine bromide, distigmine bromide, rivastigmine hydrogen tartrate, L-cysteine, alanine and glycine.

The performance of the electrodes was investigated by measuring the emfs of Ns–Br solutions in the concentration range of 10^{-7}–10^{-1} mol/L by serial dilution.

In FIA measurements, the carrier and reagent solutions were degassed by means of vacuum-suction to remove dissolved oxygen which may affect the potential response of the electrode or the flow of the carrier solution. Sample solutions used for injections were freshly prepared prior to measurements. The samples were initially diluted with distilled water. The pH and ionic strength were adjusted in the interior of the manifold.

In the analysis of biological fluids, human urine and plasma were used; plasma was obtained from VESRA, Cairo, Egypt.

2.2. Apparatus

The potentiometric measurements in batch were carried out with a Jenway 3515 digital pH/mV meter. A WTW-packed saturated calomel electrode (SCE) was used as an external reference electrode.

A single-stream FIA system was used. It is composed of a four-channel peristaltic pump (Ismatec, ISM 827) (Zurich, Switzerland) and an injection valve model 5020 with an exchangeable sample loop from Rheodyne (Cotati CA, USA). The electrodes were connected to a WTW micro-processor pH/ion-meter pMx 2000 (Weilheim, Germany) and interfaced to a strip chart recorder model BD 111 from Kipp and Zonne (Delft, Netherlands). A wall-jet, thin-layer and flow-through cell can be applied to this system.

2.3. Preparation of ion-pair

An ion-pair of Ns–Br and PTA, or NaTPB were prepared by addition of 100 mL of 10^{-2} mol/L Ns–Br solution to 100 mL of 10^{-2} mol/L of NaTPB or 100 mL of 3.3×10^{-3} mol/L PTA. The chemical compositions of the precipitates were confirmed by the elemental analysis using automatic CHN analyzer (Perkin-Elmer model 2400) in the Micro Analytical Center, Faculty of Science, Cairo University. The C, H and N percentages of Ns-TPB and Ns-PT are 12.27%, 2.32% and 1.61% and 79.66%, 5.17% and 7.20% compared with the corresponding calculated values of 12.17%, 2.36% and 1.60% and 79.66%, 5.16% and 7.19% respectively, thus confirming the purity of such ion-pairs. These precipitated ion-pairs were washed, dried and used as the active substances for preparing sensors of neostigmine bromide.

2.4. Construction of the sensors

A Teflon holder (12 cm length) with a hole at one end (7 mm diameter, 3.5 mm deep) for the carbon paste filling served as the electrode body. Electrical contact was made with a stainless steel rod through the center of the holder. This rod moved up and down by screw movement to press the paste down when renewal of the electrode surface was needed. The modified paste of each electrode was prepared by mixing the appropriate weight(s) of ion-exchanger(s) and high purity graphite with acetone. The mixture was homogenized, left at room temperature to evaporate acetone, and then the impregnated carbon powder was added to a weighed amount of plasticizer. Very intimate homogenization is then achieved by careful mixing with glass rod in an agate mortar and afterwards, rubbed by intensive pressing with a pestle. The ready-prepared paste was then packed into the hole of the electrode body. The carbon paste was smoothed onto paper until it had a shiny appearance and was used directly for potentiometric measurements without preconditioning requirements. The electrochemical system is represented as follows: CMCPE/test solution//SCE.

A fresh surface of the paste was obtained when required by driving more paste out. The surplus paste was wiped out and the freshly exposed surface was polished on a weighing paper until the surface showed shiny appearance.

2.5. Calibration of the sensors

For batch measurements, suitable increments of standard Ns solution were added to 50 mL doubly distilled water so as to cover the concentration range $1.0\times10^{-7}-1.0\times10^{-1}$ M. The sensor and the reference electrodes were immersed in the solution and the emf value was recorded at 25 ± 1 °C, after each addition. The values were plotted versus the negative logarithmic value of the drug concentration (pNs).

For FIA measurements, a series of freshly prepared solutions of the drug covering the range 1.0×10^{-7}–1.0×10^{-2} mol/L was injected to the flow stream and the corresponding peaks heights were recorded and used to draw the calibration graphs.

2.6. Effect of pH

In batch measurements the effect of pH of the test solution on the potential values of the electrode system in solutions of different concentrations (1.0×10^{-3}, 1.0×10^{-4} and 1.0×10^{-5} mol/L) of the pharmaceutical compounds, was studied. Aliquots of the drug solution (50 mL) were transferred to 100 mL titration cell and the

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