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ORIGINAL ARTICLE

Flow injection potentiometric sensor for determination of phenylpropanolamine hydrochloride

Y.M. Issa [a], **M.M. Khalil** [b], **S.I.M. Zayed** [c,*], **Ahmed Hussein** [b]

[a] *Faculty of Science, Cairo University, Giza, Egypt*
[b] *Faculty of Science, Beni Suef University, Beni Suef, Egypt*
[c] *Faculty of Industrial Education, Beni Suef University, Beni Suef, Egypt*

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Abstract A new polymeric membrane electrode has been constructed for the determination of phenylpropanolamine hydrochloride. The electrode was prepared by solubilizing the phenylpropanolamine-phosphomolybdate ion associate into a polyvinyl chloride matrix plasticized by dibutylphthalate as a solvent mediator. The electrode showed near-Nernstian response over the concentration range of 1×10^{-5}–1×10^{-2} M with low detection limit of 6.3×10^{-6} M. The electrode displays a good selectivity for phenylpropanolamine with respect to a number of common inorganic and organic species. The electrode was successfully applied to the potentiometric determination of phenylpropanolamine ion in its pure state and its pharmaceutical preparation in batch and flow injection conditions.

1. Introduction

Phenylpropanolamine hydrochloride (PPACl), Benzenemethanol, α-(1-aminoethyl) hydrochloride, (R^*, S^*) (±) [154-41-6], belongs to the sympathomimetic amine class of drugs and is structurally related to ephedrine hydrochloride (USP Dictionary of USAN and International Drug Names, 1996) (Scheme 1).

A number of analytical methods have been reported for the determination of PPACl. Among these are HPLC (Dowse et al., 2006; Kaddoumi et al., 2004; Nakashima et al., 2002; Rind et al., 2001; Zaater et al., 1999; Yamashita et al., 1990), gas chromatographic (Harsono et al., 2005; Van-Eenoo et al., 2001), capillary electrophoresis (Mateus-Avois et al., 2003; Suntornsuk, 2001; Wang et al., 2000), conductimetric (Issa et al., 2005) and spectrophotometric methods (Khuhawar et al., 2005; Ferreyra and Ortiz, 2002; Goicoechea and Olivieri, 1999; Le-Hazif et al., 1996; Ma et al., 1991; Onur and Acar, 1990). Potentiometric ion-selective electrodes based on phenylpropanolamine–tetraphenylborate or phenylpropanolamine–phosphotungstate have been reported (Badawy et al., 2004). Ion-selective membrane electrodes play an increasing role in pharmaceutical analysis with further use in FI (Vytras, 1989;

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[*] Corresponding author.
E-mail address: Yousrimi@yahoo.com (S.I.M. Zayed).

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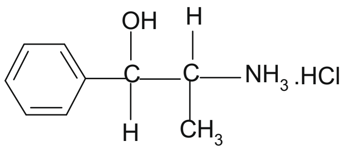

Scheme 1

Vire and Kauffmann, 1994; Cosofret, 1991) offering advantages of simplicity, rapidity and accuracy. Liquid membrane electrodes using phosphotungstic and phosphomolybdic acids were previously described (Issa and Zayed, 2006).

The present work describes the construction and potentiometric characterization of new potentiometric sensor for PPA. The electrode is based on the incorporation of phenylpropanolamine-phosphomolybdate $(PPA)_3$-PM ion associate in a polyvinyl chloride (PVC) membrane plasticized with dibutylphthalate (DBP). Applications of the electrode for the determination of PPACl in pharmaceutical preparation for batch and FI analysis system were also described.

2. Experimental

2.1. Reagents and materials

All chemicals were of analytical grade. Double distilled water was used throughout all experiments. Pure grade phenylpropanolamine hydrochloride (PPACl) and the pharmaceutical preparation Contac 12 capsules were provided by Kahira pharmaceutical and Chemical Industries Co., Egypt, and Egyptian International Pharmaceutical Industries Co., (EIPICO), respectively. Phosphomolybdic acid (PMA), dioctyl sebacate (DOS), and tricresyl phosphate (TCP) were from Fluka, dibutyl phthalate (DBP), and dioctyl phthalate (DOP) from Merck. PVC of relatively high molecular weight was from Aldrich.

2.2. Apparatus

Potentiometric and pH measurements were carried out using a Seibold G-103 digital pH/mV meter (Vienna, Austria). A techne circulator thermostat Model C-100 was used to control the temperature of the test solutions. A saturated calomel electrode (SCE) was used as the external reference, while an Ag/AgCl wire as an internal electrode.

The flow injection setup as previously reported (Issa and Zayed, 2006). Fig. 1 represents the schematic diagram of the flow injection system used in the measurements.

2.3. Preparation of the ion associate

The ion associate $(PPA)_3$-PM, was prepared by mixing 150 ml of 10^{-2} M PPACl solution with 50 ml of 10^{-2} M phosphomolybdic acid. The formed precipitate was filtered, washed thoroughly with bidistilled water until chloride free and dried at room temperature. The composition of the ion-associate was found to be 3:1 as confirmed by elemental analysis data done at microanalytical research laboratory in National Research Centre (Dokki, Cairo, Egypt). The percentages values found are 14.20, 1.91 and 1.82 and the calculated values are 14.24, 1.73 and 1.84 for C, H and N, respectively.

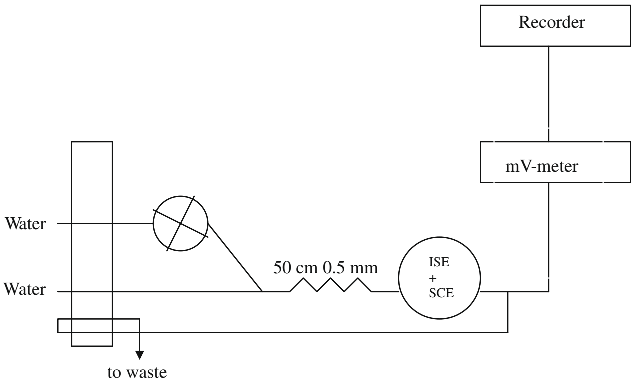

Figure 1 Schematic diagram of the flow injection system used in the measurements.

2.4. Electrode preparation

The electrode was constructed as previously described (Issa and Zayed, 2006). The membranes were prepared by dissolving the required amount of the ion associate, PVC and DBP, in about 10 ml of THF. The solution mixture was poured into a 6.0 cm Petri dish and left to dry in air. To obtain a uniform membrane thickness, the amount of THF was kept constant, and its evaporation was fixed for 24 h. The thickness of the membrane was about 0.2 mm.

A 12 mm diameter disk was cut out from the prepared membrane and glued using PVC–THF paste to the polished end of a plastic cap attached to a glass tube. The electrode body was filled with a solution of 1×10^{-1} M NaCl and 1×10^{-2} M PPACl. The electrode was pre-conditioned before use by soaking in a 1×10^{-3} M PPACl solution.

2.5. Potentiometric determination of PPACl

The standard addition method (Baumann, 1986) was applied, in which small increments of the standard solution (10^{-1} M) of PPACl were added to 50 ml aliquot samples of various concentrations from pure drug or pharmaceutical preparations. The change in millivolt reading was recorded for each increment and used to calculate the concentration of PPACl sample solution using the following equation:

$$C_x = C_s\left(\frac{V_s}{V_x + V_s}\right)\left(10^{n(\Delta E/S)} - \frac{V_x}{V_x + Vs}\right)^{-1},$$

where C_x and V_x are the concentration and the volume of the unknown, respectively, C_s and V_s the concentration and the volume of the standard solution, respectively, s the slope of the calibration graph and ΔE is the change in millivolt due to the addition of the standard solution.

2.6. Determination of phenylpropanolamine hydrochloride in Contac 12 capsules

Twenty capsules were accurately weighed and powdered in a mortar, the required amount from the capsules powder was dissolved in chloroform to separate phenylpropanolamine hydrochloride from the capsules matrix (chloroform dissolves isopropamide iodide only).

The separated phenylpropanolamine hydrochloride was dried and then dissolved in about 30 ml bidistilled water and