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Flow injection potentiometric determination of bismuth(III) in anti-acid formulations

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Abstract

A flow injection potentiometric procedure is proposed for determining bismuth(III) in anti-acid formulations. In this work, a tubular electrode coated with an ion-pair formed between $[Bi(EDTA)]^-$ and tricaprylylmethylammonium cation (Aliquat 336) in a poly(vinylchloride) (PVC) was constructed and used in a single channel flow injection system. The effect of membrane composition, pH and flow injection parameter over the Bi(III) tubular electrode response (slope (mV/decade)) was initially evaluated in quintuplicate in 0.5 mol l^{-1} EDTA solution as carrier. The best response (-59.6 ± 0.9 mV/decade) was attained with the 5% m/m ion-pair; 65% m/m *o*-nitrophenyl octyl ether (*o*-NPOE) and 30% m/m PVC in pH 6–9. The electrode showed a linear response to E (mV) versus log $[Bi(EDTA)]^-$ in the bismuth(III) concentration range from 2.0×10^{-5} to 1.0×10^{-2} mol l^{-1} and a useful lifetime of at least 5 months (more than 1000 determinations for each polymeric membrane). The detection limit was 1.2×10^{-5} mol l^{-1} and the R.S.D. was less than 2.0% for a solution containing 5.0×10^{-4} mol l^{-1} bismuth(III) ($n = 10$). Several species such as Cd(II), Mn(II), Ni(II), Zn(II), Co(II), Cu(II), Mg(II), Cr(III) and Al(III) at 1.0×10^{-3} mol l^{-1} concentration in 0.5 mol l^{-1} EDTA solution did not cause any interference. The frequency rate was 90 determinations per hour and the results obtained for bismuth(III) in anti-acid formulations using this flow procedure and those obtained using a spectrophotometric procedure are in agreement at the 95% confidence level. © 2001 Elsevier Science B.V. All rights reserved.

Keywords: Bismuth(III) tubular ion-selective electrode; Ethylenediamintetraacetate; Anti-acid formulations; Flow injection

1. Introduction

Many bismuth compounds have been used orally for a wide range of gastrointestinal tract disorders ranging from constipation to diarrhea, but particularly for peptic gastritis and ulceration (Burtis and Ashwood, 1994).

Official methods commonly used for determining bismuth(III) in various pharmaceutical formulations are based on its titration with iodide and EDTA or spectrophotometric detection of Bi(III) complexes (British Pharmacopoeia, 1980; Melen-

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tyeav and Antonova, 1985; US Pharmacopeia, 1995), but some of which require tedious procedures such as Bi_2O_3(s) precipitation with 6 mol l^{-1} sodium hydroxide for 2 h, exhaustive washing, drying and weighing of this precipitate or are subject to numerous interferences (any cation that precipitates in alkaline medium).

The determination of bismuth(III) in pharmaceutical formulations by spectrophotometric methods has been suggested based on extraction of bismuthate(III) complex anions with protriptylinium (Burns and Dunford, 1996), Cyanex 301 (Argekar and Shetty, 1995), naphthylmethyltriphenylphosphonium (Burns and Chimpalee, 1992), tributylhexadecylphosphonium (Burns and Tungkananuruk, 1987), 2,4,6-triphenylpyrylium (Ortuno et al., 1987), tetrabutylammonium (Hasebe and Taga, 1982), and with cation-exchange resin where Bi(III) was determined spectrophotometrically with thiourea (Desai and Shinde, 1991; Barve et al., 1993) and xylenol orange (Krzek and Al-Mutari, 1980; Agrawal and Bhatt, 1984). In addition, the direct spectrophotometric determination of bismuth has been proposed in the visible region after reaction with porphyrin (Fan and Hu, 1994), 1-[di-(2-pyridyl)methylene]-5-salicylidenethiocarbohydrazide (Rosales et al., 1988; Morales et al., 1990), *o*-hydroxyhydroquinonephthalein in the presence of Brij 58 (Mori et al., 1988), *N*,*N*-bis-(2-hydroxyethyl) glycine (Lopez et al., 1988), 2,6-dihydroxybenzoic acid and crystal violet (Cordoba et al., 1987), galactaric acid (Portal et al., 1986), *N*-(2-acetamido) iminodiacetic acid (Portal et al., 1985), 4,5-diaminopyrimidine-2,6-dithiol (Izquierdo et al., 1984) and iodide (Kar, 1985). Nevertheless, these methods involve a time-consuming extraction step, and require strictly controlled reaction conditions and costly reagents.

Burns and co-workers (Burns et al., 1989; Harriott and Burns, 1989) used a tetramethylene bis(triphenylphosphonium) bromide solution (anion-exchange) as carrier in flow injection analysis with spectrophotometric detection for the determination of bismuth(III) after previous liquid–liquid extraction of tetraiodobismuthate(III).

Liquid membrane ion-selective electrodes incorporated into flow injection systems have been widely applied in the analysis of pharmaceutical formulations. The use of these electrode can usually increase the sensitivity, decrease chemical and mechanical interferences and also they have a fast response and good reproducibility (Ruzicka and Hansen, 1975; Pungor et al., 1984; van Standen, 1987). Tubular ion selective electrodes have been shown previously to be optimum detectors in flow injection systems where they propound an ideal geometric design for the hydrodynamic flow conditions (Alegret et al., 1984; Lima and Rocha, 1990; Ferreira and Lima, 1994; Teixeira et al., 1999).

In this work, a flow injection potentiometric procedure is reported for determining bismuth(III) in anti-acid formulations. The construction and analytical evaluation of a graphite-epoxy ion-selective electrode coated with an ion-pair formed between $[Bi(EDTA)]^-$ (EDTA = ethylenediamintetraacetate) and tricaprylylmethylammonium cation (Aliquat 336) in a poly(vinylchloride) (PVC) matrix was recently proposed by us (Teixeira et al., 1997). The same active material was used in a tubular electrode in a flow injection system. The flow injection procedure proposed here for the selective determination of bismuth(III) is a useful alternative to those time-consuming procedures described.

2. Materials and methods

2.1. Apparatus

An eight-channel Ismatec (Zurich, Switzerland) model 7618-40 peristaltic pump supplied with Tygon pump tubing was used for the propulsion of the fluids. The manifold was constructed with polyethylene tubing (0.8 mm i.d.). Sample injection was performed using a laboratory-constructed three-piece manual commutator made of Perspex®, with two fixed sidebars and a sliding central bar (Fatibello-Filho and Vieira, 1997).

The potential differences between the tubular electrode and the Analion (R684 model) Ag/AgCl double junction reference electrode were measured with an Orion model EA 940 pH/ion meter (USA) with ± 0.1 mV precision and the signals were recorded on a Cole Parmer model CR 53125 recorder.

2.2. Reagents and solutions

All solutions were prepared using Millipore Milli-Q water. All chemicals were analytical reagent grade and were used without further purification.

The bismuth(III) and tetraneutralized EDTA stock solutions were prepared as reported previously (Teixeira et al., 1997) and used to prepare bismuth(III) reference solutions in 0.5 mol l^{-1} EDTA medium, at pH 8.

Metallic cation solutions (Cd(II), Mn(II), Ni(II), Zn(II), Co(II), Cu(II), Mg(II), Cr(III) and Al(III)) at 1.0×10^{-3} mol l^{-1} in 0.5 mol l^{-1} EDTA solution were prepared and used in the interference studies.

High-molecular weight poly(vinylchloride) (PVC, pure grade available from Fluka), tetrahydrofurane (reagent grade, Aldrich), and *o*-nitrophenyl octyl ether (Aldrich) were used for the tubular electrode construction.

2.3. Tubular electrode construction

The tubular electrodes were constructed as described previously (Teixeira et al., 1999). The coating solutions were prepared (Teixeira et al., 1997) by dissolving 0.30 g of PVC in 10 ml of tetrahydrofurane (THF) followed by the addition of *o*-nitrophenyl octyl ether (NPOE) and the ion pair [Aliquat 336][Bi(EDTA)] such as: (a) 1% (w/w) ion pair, 69% (w/w) NPOE and 30% (w/w) PVC; (b) 2% (w/w) ion pair, 68% (w/w) NPOE and 30% (w/w) PVC; (c) 5% (w/w) ion pair, 65% (w/w) NPOE and 30% (w/w) PVC; (d) 10% (w/w) ion pair, 60% (w/w) NPOE and 30% (w/w) PVC. These solutions were deposited 3–4 times directly in hole walls using a dropper. The utilization of *o*-nitrophenyl octyl ether as mediator solvent resulted in improved selectivity and fast response times relative to the previously used dibutylphthalate (Montenegro et al., 1993; Teixeira et al., 1997).

2.4. Flow injection system

The tubular electrode was inserted into a flow injection system schematically shown in Fig. 1. The 0.5 mol l^{-1} EDTA solution was used as the carrier (C) at a flow rate of 4.4 ml min^{-1}. In order to stabilize the baseline, a concentration of 10^{-7} mol l^{-1} bismuth(III) was added to carrier solution. A bismuth(III) sample or reference in 0.5 mol l^{-1} EDTA solution contained in the sample loop (L, 250 μl) was injected and transported by the carrier stream after the baseline had reached a steady-state value. The analytical path was 40 cm and the entire flow injection system was maintained at 25°C, and the potential differences between the tubular and reference electrodes were measured in the pH/ion meter.

2.5. Preparation and analysis of pharmaceutical samples

For potentiometric determination of bismuth(III) in three anti-acids powder samples (Brazilian formulas), accurately weighed amounts between 3 and 5 g of solid samples were initially dissolved in 1 ml aqua-regia (conc. HCl/conc. HNO_3 3:1 v/v) and heated to dryness. After that, the samples were dissolved in 0.5 mol l^{-1} EDTA solution and were transferred to a 100-ml volumetric flask and this volume completed with the same solution. The percentage content of bismuth

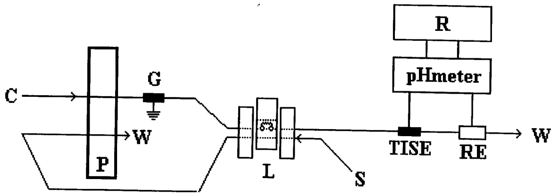

Fig. 1. Schematic diagram of the flow injection system used for bismuth(III) determination. The central bar of the manual injector–commutator shows the injection position after commutation. P, peristaltic pump; S, sample or reference solutions; L, sample loop (250 μl); C, carrier solution, 0.5 mol l^{-1} EDTA (pH 8), flowing at 4.4 ml min^{-1}; G, earthed stainless-steel tube; TISE, tubular ion-selective electrode; RE, Ag/AgCl reference electrode; R, record and W, waste.

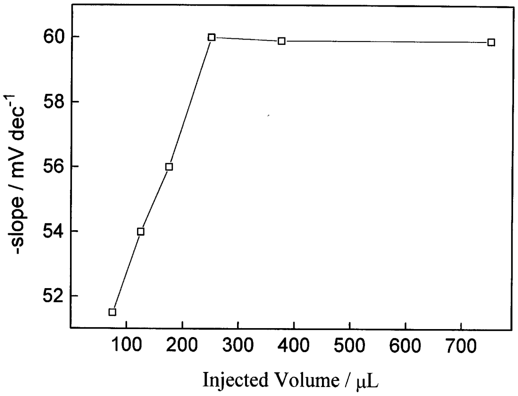

Fig. 2. Influence of injected volume on the potentiometric response (mV/decade) for Bi(EDTA)$^-$ solutions ranging from 1.0×10^{-5} to 1.0×10^{-2} mol l^{-1}, at 25°C.

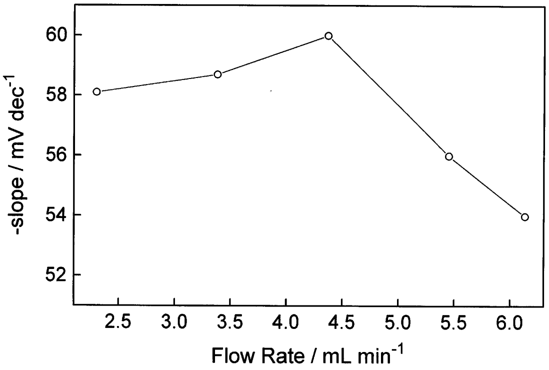

Fig. 3. Influence of flow rate on the potentiometric response (mV/decade) for Bi(EDTA)$^-$ solutions ranging from 1.0×10^{-5} to 1.0×10^{-2} mol l^{-1}, at 25°C.

in these samples was determined by the standard addition method and by visible spectrophotometric detection at a wavelength of 540 nm with xylenol orange (Marczenko, 1980).

3. Results and discussion

3.1. Effect of membrane composition and pH

The effect of membrane composition upon the bismuth(III) tubular electrode response (slope (mV/decade)) was initially evaluated in quintuplicate in 0.5 mol l^{-1} EDTA solution at pH 8. The best response (signal/noise) was reached for the 5% (w/w) ion pair; 65% (w/w) NPOE and 30% (w/w) PVC, indicating that the tubular electrode preferentially responds to the anionic [Bi(EDTA)]$^-$ species, in pH 8. The effect of pH in the range 3–11 on the potentiometric response of a 1.0×10^{-3} mol l^{-1} bismuth(III) solution under the conditions specified in the legend of Fig. 1 was investigated. The pH of these solutions was adjusted by the addition of 2 mol l^{-1} HCl or 2 mol l^{-1} NaOH solutions. The results obtained show that there are no significant variations in the potential in the pH ranging from 3 to 11.

3.2. Flow injection parameters and tubular electrode characteristics

Preliminary studies were carried out to establish the best flow injection parameters and tubular electrode characteristics.

The effect of varying sample loop length from 15 to 150 cm (75–750 µl) for [Bi(EDTA)]$^-$ solution ranging from 1.0×10^{-5} to 1.0×10^{-2} mol l^{-1} on the potentiometric response (slope in mV/decade) at pH 8 was initially evaluated. The potentiometric response increased with the increase of sample volumes from 75 to 250 µl and was maintained constant in sample volume higher than 250 µl (Fig. 2). Therefore, a sample volume of 250 µl was selected for further experiments. The effect of flow rate from 2.3 to 6.1 ml min^{-1} for [Bi(EDTA)]$^-$ solutions ranging from 1.0×10^{-5} to 1.0×10^{-2} mol l^{-1} over the potentiometric response (slope in mV/decade) of the bismuth(III) tubular electrode is presented in Fig. 3. The optimal flow rate was 4.4 ml min^{-1}. In flow rates lower than 4.4 ml min^{-1}, the tubular electrode showed slight memory effects and long washing times, decreasing the analytical frequency. At flow rates higher than 4.4 ml min^{-1} the electrode slope decreases strongly. The dynamic response of the tubular electrode was studied by injecting 1.0×10^{-4} mol l^{-1} bismuth(III) solution. A time of 20 s was obtained when a volume of 250 µl was injected. All subsequent experiments were carried out in these experimental conditions.

At pH 8, the flow injection system shows a linear response to E (mV) versus log [Bi(EDTA)]$^-$

in the bismuth(III) concentration range from 2.0×10^{-5} to 1.0×10^{-2} mol l^{-1} (analytical curve: $E = (-364 \pm 3) - (59.6 \pm 0.9)C$; $r = 0.9996$, where E is the potential of the tubular electrode in mV, C the concentration of bismuth(III) in mol l^{-1}) and 59.6 the slope (in mV/decade) of the analytical curve. The detection limit (threefold blank standard deviation/slope) was 1.2×10^{-5} mol l^{-1} and the relative standard deviation (R.S.D) was less than 2% for a solution containing 5.0×10^{-4} mol l^{-1} bismuth(III) ($n = 10$). The tubular electrode useful lifetime was at least 5 months (more than 1000 determinations/polymeric membrane) and several species such as Cd(II), Mn(II), Ni(II), Zn(II), Co(II), Cu(II), Mg(II), Cr(III), Fe(III) and Al(III) at 1.0×10^{-3} mol l^{-1} in 0.5 mol l^{-1} EDTA solution (pH 8) did not cause any interference. In this study, 1.0 ×

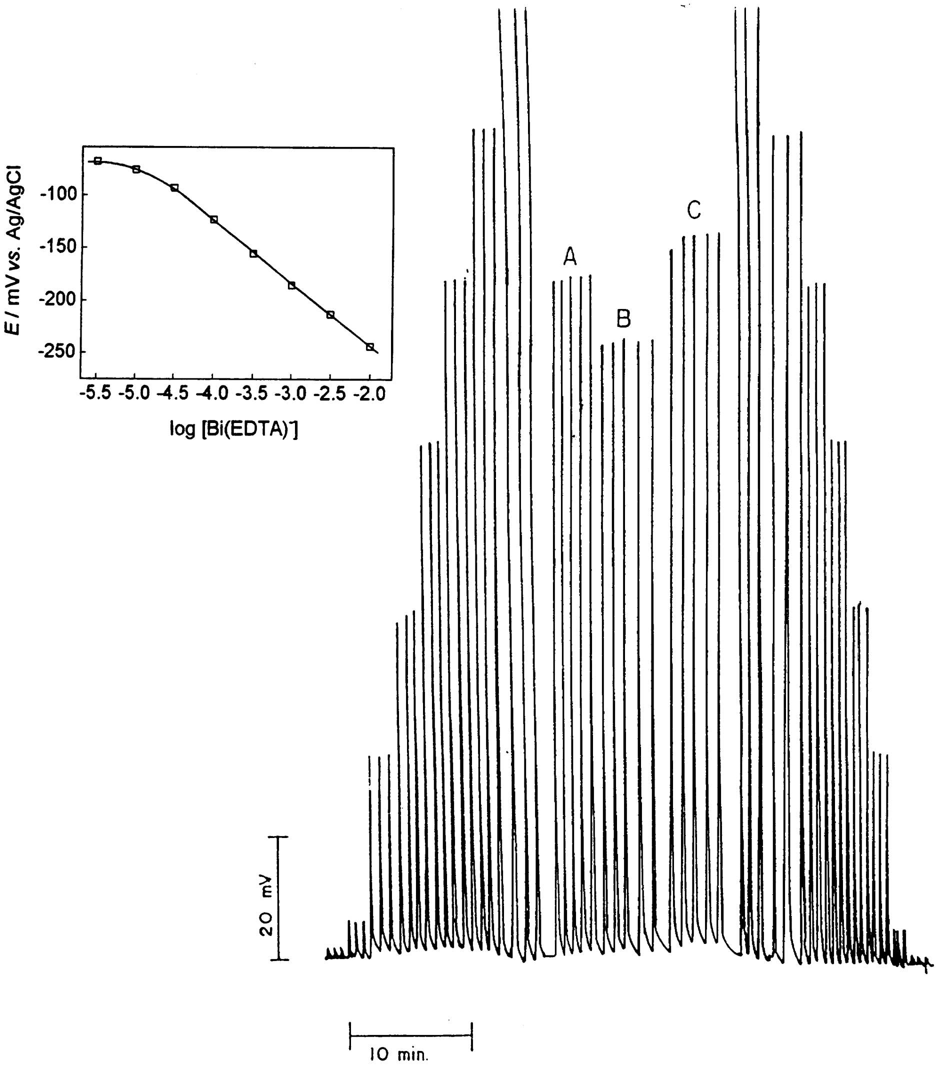

Fig. 4. Transient potentiometric signals obtained in triplicate for eight reference solutions (3.2×10^{-6}; 1.0×10^{-5}; 3.2×10^{-5}; 1.0×10^{-4}; 3.2×10^{-4}; 1.0×10^{-3}; 3.2×10^{-3} mol l^{-1} and 1.0×10^{-2} mol l^{-1}), three samples (A, Bisuisan: B, Magnésia Bisurada and C, Salicilato de Bismuto) and reference solutions again. Conditions: carrier solution, 0.5 mol l^{-1} EDTA; flow rate, 4.4 ml min^{-1}; injection volume, 250 µl; analytical path, 40 cm, at 25°C. Inset: Calibration graph obtained.

Table 1
Determination of bismuth(III) in anti-acid formulations using a bismuth(III) tubular ion selective electrode and visible spectrophotometric methods (Marczenko, 1980)

Sample	mg of Bi/g of sample[a]		Relative error (%)
	Visible spectrophotometry	Potentiometry–FIA	
Bisuisan	8.4 ± 0.2	8.6 ± 0.4	2.4
Magnésia Bisurada	5.0 ± 0.2	5.1 ± 0.2	+2.0
Salicilato de Bismuto	12.6 ± 0.5	12.1 ± 0.4	−4.0

[a] Five replicates ($n = 5$).

10^{-4} mol l^{-1} bismuth(III) solution mixed with each of these species above mentioned in 0.5 mol l^{-1} EDTA was injected separately.

Recoveries of 98.0–102.1% of bismuth(III) from three pharmaceutical formulations ($n = 5$) were obtained using the flow-injection potentiometric procedure. In this study, 1.0×10^{-4}; 5.0×10^{-4}; 1.0×10^{-3}, 5.0×10^{-3} mol l^{-1} of bismuth(III) were added to each product. The recovery results obtained suggest an absence of the matrix effect in the analysis procedure.

3.3. Determination of bismuth(III) in anti-acid formulations

Fig. 4 shows triplicate transient signals for eight reference bismuth(III) solutions (3.2×10^{-6}; 1.0×10^{-5}; 3.2×10^{-5}; 1.0×10^{-4}; 3.2×10^{-4}; 1.0×10^{-3}; 3.2×10^{-3} and 1.0×10^{-2} mol l^{-1}) and quintuplicate transient signals for three pharmaceutical formulations. As can be seen from the transient signals presented in this figure, the procedure demonstrated good precision and baseline stability with an analytical frequency of 90 determinations per h. Table 1 presents the results obtained using a visible spectrophotometric procedure (Marczenko, 1980) and the flow injection method proposed. Applying paired t-tests to the results obtained by either procedure, it was found that all results are in agreement at the 95% confidence level and within an acceptable range of error, confirming the accuracy of the FI potentiometric method.

4. Conclusions

The bismuth(III) tubular electrode which has been developed is easy to make, has a long lifetime, good selectivity and its larger sampling rate permits the determination of the bismuth(III) in anti-acid formulations with good accuracy and precision.

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