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Flow-injection potentiometric determination of triiodide by plasticized poly(vinyl chloride) membrane electrodes and its application to the determination of chlorine-containing disinfectants

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Abstract

Plasticized poly(vinyl chloride) (PVC) membranes of different compositions were tested for use in the construction of potentiometric flow detectors for triiodide. A membrane with a 2:1 (w/w) 2-nitrophenyl octyl ether to PVC ratio was selected. The influence of thiosulphate in the carrier solution composition and of the flow-injection variables on the determination of triiodide was studied. In the selected conditions, a linear relationship between peak height and $\log[I_3^-]$ was obtained between 5×10^{-6} and 1×10^{-4} mol l^{-1} triiodide. Peak height relative standard deviations for 2×10^{-5} and 1×10^{-4} mol l^{-1} triiodide were ±0.4 and ±1.8%, respectively, and sampling frequency was 80 samples per hour. The method proposed was applied satisfactorily to the iodometric determination of different chlorine-containing disinfectants, among them trichloroisocyanuric acid and dichloroisocyanurate in several types of commercial sample.

Keywords: Flow-injection potentiometry; Triiodide-selective electrode; Plasticized poly(vinyl chloride) membrane; Chlorine-containing disinfectant

1. Introduction

A selective flow-through potentiometric detector for triiodide may be a useful tool for application in flow-injection analysis because of the number of analytes that can be determined by iodometric methods. Flow-injection iodometric methods for the determination of several oxidants using biamperometric [1], potentiometric [2], photometric [3,4] and fluorimetric [5] detection have been reported. The paper that described potentiometric detection used a platinum electrode to measure the iodine to iodide ratio and an iodide-selective electrode to measure the iodide concentration. The potential difference between both electrodes was related with the iodine concentration.

In the present paper, different plasticized poly(vinyl chloride) (PVC) membranes, blank and doped with lipophilic electrolytes, are studied for use as potentiometric flow detectors of triiodide. The selected membrane was applied to the flow-injection iodometric determination of different chlorine-containing disinfectants. Plasticized polymeric membranes prepared without the addition of electroactive materials (blank membranes) give a potentiometric response to certain ions. Many years ago Higuchi et al. [6] found that simple plastic membrane electrodes were specific for hydrophobic ions. This was corroborated by other authors that used plastic membrane electrode to monitor titrations [7–9]. Vytras et al. [9] studied the role of plasticizer and Martin and Freiser [10] showed the role of solvent extraction in determining the selectivity sequence. More details can be found in an extensive review [11]. To explain the potential response of plasticized poly(vinyl chloride) membranes, an electrical charge conduction mechanism was proposed [12].

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A relationship between the hydrophobicity of cations and the magnitude of the response of plasticized poly(vinyl chloride) membrane electrodes was found [12,13]. Masadome et al. [14–16] used electrodes based on plasticized poly(vinyl chloride) membranes for the determination of surfactants, including their successive potentiometric detection by high performance liquid chromatography. We reported that an electrode containing a poly(vinyl chloride) membrane plasticized with 2-nitrophenyl octyl ether exhibited a sensitive and fast response to triiodide. An explanation to this response was given and the electrode was applied to monitoring iodimetric titrations [17]. Other triiodide-selective electrodes have been reported [18,19]. A platinum electrode was used to monitor ion-pair potentiometric titrations involving triiodide [20–22].

2. Experimental

2.1. Apparatus

Potentials were measured with an Orion 960 Autochemistry System (Boston, MA, USA), whose recorder output was connected to a personal computer via a DGH Corporation 1121 module analog-to-digital converter (Manchester, UK). A Fluka (Buchs, Switzerland) glass ring of 28 mm inner diameter and 30 mm height, a glass plate, vial, punch and electrode body ISE, were used for membrane construction [23] and mounting. An Orion 90-02 double junction silver–silver chloride reference electrode containing 10% potassium nitrate solution in the outer compartment was used. A Gilson (Villiers le Bel, France) Minipuls 3 peristaltic pump, Omnifit (Cambridge, UK) injection valve, connecting tubing of 0.5 mm bore, PTFE tubing and various end fittings and connectors were used to construct the flow-injection system. A flow cell similar to that described previously [24] was used.

2.2. Reagents and solutions

Poly(vinyl chloride) high molecular mass, 2-nitrophenyl octyl ether (NPOE) and tetrahydrofuran (THF) were Selectophore products from Fluka. Tetrabutylammonium tetraphenylborate (TBA-TPB) and benzalkonium chloride (BZCl) were from Sigma. All other reagents used were of analytical reagent grade and doubly distilled water was used throughout. A concentration of 0.1 mol l^{-1} sodium sulphate or 5×10^{-6} or 5×10^{-5} mol l^{-1} sodium thiosulphate in 0.1 mol l^{-1} sodium sulphate, were used as carrier solutions.

(1) Standard iodate solution $(1/6) \times 10^{-2}$ mol l^{-1}: prepared by accurately weighing 0.3566 g KIO3 dissolved in 1000 ml of water.
(2) Standard thiosulphate solution, 0.1 mol l^{-1}: prepared from Na2S2O3·5H2O and standardised with potassium iodate solution.

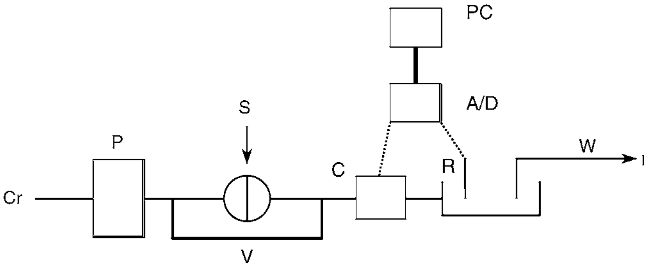

Fig. 1. Flow-injection system: (Cr), carrier solution; (P), pump; (V), injection valve; (S), sample; (C), flow-through cell; (R), reference electrode; (W), waste; (A/D), analog-to-digital converter; (PC), personal computer.

(3) Standard triiodide solution, 5×10^{-2} mol l^{-1}: prepared according to [25] from 40 g of KI and 12.7 g of I2, dissolved in 1000 ml of water and standardised with thiosulphate solution.
(4) Iodide solution, 0.5 mol l^{-1}: prepared daily from potassium iodide.

2.3. Preparation of the membranes

The membranes were prepared by dissolving 200 mg NPOE and 100 mg PVC for membranes A and B, or 100 mg NPOE and 200 mg PVC for membranes C and D, in 3 ml of tetrahydrofuran. For membranes B and D, 3 mg of TBA-TPB were also dissolved. This solution was poured into the glass ring resting on the glass plate and was left overnight to allow the solvent to evaporate slowly. A 7 mm diameter piece was cut out with the punch and incorporated into the electrode body containing 3.8 mol l^{-1} potassium chloride saturated with excess of AgCl as internal filling solution.

2.4. Recommended flow-injection procedure

The flow-injection system used is shown in Fig. 1. The corresponding ion-selective electrode was incorporated in a flow-injection system by means of the flow cell described in [24]. The distance between the injection valve and the cell was 20 cm. A concentration of 5×10^{-6} mol l^{-1} sodium thiosulphate, 0.1 mol l^{-1} sodium sulphate, used as carrier solution, was pumped at a flow rate of 1 ml min^{-1} and 140 µl aliquots of sample solutions were injected. Potentials were monitored with membrane A and peak heights were measured. Working standard solutions, 10^{-6} to 10^{-3} mol l^{-1} I_3^- prepared in 0.1 mol l^{-1} sodium sulphate, 0.01 mol l^{-1} potassium iodide solution were used to obtain the corresponding calibration graphs.

For the determination of chlorine-containing disinfectants in commercial products, aliquots of 1 ml of household bleach, 5 ml of sterilising solution, one sterilising tablet or 0.5 g of product for the treatment of swimming pools were accurately pipetted or weighed, transferred into a calibrated flask and diluted to 500 ml with water (solutions A–D, respectively). To an aliquot of 1 ml of solutions A or B, or 200 µl of solution C or D, 1 ml of 1 mol l^{-1} acetic acid and 2 ml of 0.5 mol l^{-1} potassium iodide were added and the solution was diluted to 100 ml with 0.1 mol l^{-1} sodium sulphate. Aliquots of this solution were injected into the FI system as described above. Working standard solutions, 5×10^{-6} to 1×10^{-4} mol l^{-1} I_3^- prepared in the same medium than as described for the sample, were used to obtain the calibration graph. The chlorine-containing disinfectant content in the sample was calculated from the corresponding triiodide concentration obtained from the calibration graph. The iodometric titration method with thiosulphate was used as reference method [26].

2.5. Calculation of potentiometric selectivity coefficients

Potentiometric selectivity coefficients were determined by the separate solution method [27] from the potential peak heights (mV), H_j and H_i, obtained by injecting an interfering ion solution (1×10^{-4} M DDS^-, 1×10^{-3} M HTA^+, BZ^+, 1×10^{-2} M I^-, 1×10^{-1} M Cl^-, Br^-, NO_3^-, SO_4^{2-}, $S_2O_3^{2-}$, 1 M $H_2PO_4^-$) and a primary ion solution (1×10^{-4} M I_3^-) in the FI conditions described in Section 2.4. The following expression was used for the calculation

$$K_{i,j} = \left(\frac{C_i}{C_j^{-1/z_j}}\right) \exp\left(\frac{H_j - H_i}{S_{ex}}\right) \quad (1)$$

where S_{ex} represents the experimental slope. The peaks obtained for the cations showed opposite direction to the corresponding for triiodide but the same sign was used for calculating the corresponding selectivity coefficients.

3. Results and discussion

3.1. Influence of membrane composition

Two blank plasticized PVC membranes containing two different NPOE/PVC ratios (2/1 and 1/2 m/m) (membranes A and C, respectively) and another two containing these same ratios and also 1% (m/m) TBA-TPB (membranes B and D, respectively) were tested. Occasionally, salts of a lipophilic cation with a lipophilic anion have been used as membrane additives for neutral carrier-based ion-selective electrodes with the aim of reducing membrane electrical resistance [28,29] and of altering the selectivity by affecting the activity coefficients in the membrane [30]. Fig. 2 shows the corresponding calibration graphs of the four membranes for triiodide, obtained as described in Section 2 using 0.1 mol l^{-1} sodium sulphate as carrier solution. As can be seen, the potential response of the blank (A) and doped (B) membranes containing a 2:1 NPOE/PVC ratio were higher than the corresponding membranes (C and D, respectively) containing a 1:2 ratio. Of the two membranes containing a 2:1 NPOE/PVC ratio, the response of the blank membrane (A) was higher than the membrane (B) doped with the lipophilic electrolyte. Taking these results into account membrane A was selected for further studies.

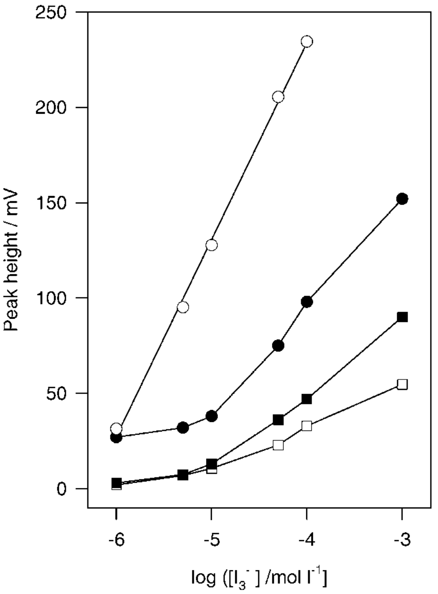

Fig. 2. Calibration graphs for triiodide with membranes A (○); B (●); C (□) and D (■). Carrier solution 0.1 mol l^{-1} sodium sulphate.

3.2. Influence of carrier composition

A 0.1 mol l^{-1} sodium sulphate concentration was used as carrier solution and as diluting solution in the preparation of the working standards and of the sample solution before injecting them in order and to keep the ionic strength constant. However, when this carrier solution was used, and high triiodide concentrations were injected a long time was needed to return to the baseline. Peak tails of this type are commonly observed in flow-injection potentiometry and are due to a slower electrode response in the case of decreasing than in the case of increasing ion concentrations. This is a limitation for practical analytical measurements and it is commonly avoided by introducing a small concentration of the analyte ion into the carrier solution, at the expense of a slightly worse detection limit [31]. In our case, as suggested in a previous paper [17], in which a plasticized PVC membrane electrode was proposed for monitoring titrations of triiodide, this ion may penetrate the membrane via an anion-exchange or salt-extraction process, although the excess of titrant (thiosulphate) removed the triiodide from the plasticized membrane and left it ready for the next titration. Accordingly, the addition of thiosulphate to the carrier composition was tested in the present work as a way of avoiding long tailing. It was thought that the thiosulphate would react with the triiodide present in the extremes of the sample plug and with the triiodide that had penetrated into the membrane during exposure to the sample plug. The thiosulphate concentration must be kept very low and a FI system of low dispersion such as that proposed should be used in order to keep unreacted most triiodide present in the

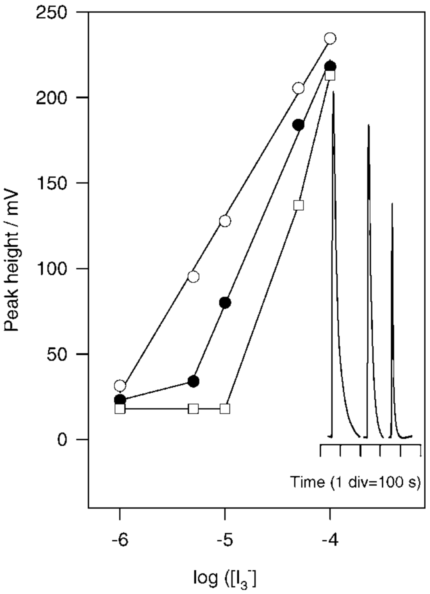

Fig. 3. Calibration graphs for triiodide with membrane A using the following carrier solutions: (○) 0.1 mol l^{-1} sodium sulphate; (●) 0.1 mol l^{-1} sodium sulphate, 5×10^{-6} mol l^{-1} sodium thiosulphate; (□) 0.1 mol l^{-1} sodium sulphate, 5×10^{-5} mol l^{-1} sodium thiosulphate. The transient signals (from left to right) correspond to a concentration 5×10^{-5} mol l^{-1} triiodide using the three carrier solutions, respectively.

sample plug. Two different sodium thiosulphate concentrations, 5×10^{-6} or 5×10^{-5} mol l^{-1} in 0.1 mol l^{-1} sodium sulphate were tested as carrier solutions. The corresponding calibrations are shown in Fig. 3 together with that obtained in the absence of thiosulphate in the carrier solution. The transient signals obtained for 5×10^{-5} mol l^{-1} triiodide are also included. As can be seen, the potential response and the linear response triiodide concentration range decreased as the thiosulphate concentration in the carrier increased. On the other hand, the time to return to the baseline decreased, thus improving the sampling frequency. As a compromise between sensitivity and sampling frequency a 5×10^{-6} mol l^{-1} thiosulphate concentration in the carrier solution composition was selected.

3.3. *Influence of pH*

The influence of pH on the response of the plasticized PVC membrane towards triiodide was studied by injecting 140 μl of 5×10^{-5} mol l^{-1} triiodide standard solution at different pH values adjusted with sulphuric acid, acetic/acetate buffer or ammonium acetate, on the selected carrier described above pumped at 1 ml min^{-1}. Constant peak height was obtained in the pH range 0.8 and 6.0.

3.4. *Influence of FI-variables*

The influence of the injected sample volume on the determination of triiodide was studied by injecting different volumes ranging from 100 to 700 μl of 1×10^{-5} mol l^{-1} triiodide standard solution at a flow rate of 1 ml min^{-1}. An increase in the sample volume produced an increase in the peak height to reach an almost constant value around 700 μl. However, the time needed to return to the baseline also increased considerably. Thus, from 100 to 500 μl the peak height only increased one-third, while the peak width increased by a factor of three, thus substantially decreasing the sampling frequency. A value of 140 μl was selected for further studies. The influence of the flow rate was studied in the range 0.5–1.25 ml min^{-1}. By increasing the flow rate between 0.5 and 0.75 ml min^{-1} slight decrease in peak height was observed. Over this range almost constant values were obtained. When higher flow rates were tested a lost of reproducibility was observed. A flow rate of 1 ml min^{-1} was selected. The selected sample volume and flow rate values together with the distance between the injection valve and the cell used (see Section 2.4) provide a low dispersion system such as recommended in Section 3.2.

3.5. *Features of the method*

In the selected conditions, the calibration graph (potential versus log concentration) showed a linear relationship in the range 5×10^{-6} to 1×10^{-4} mol l^{-1} triiodide, with a slope of 152 mV/dec. This response is highly hyper-Nernstian. This type of deviation from the theoretical values observed in FIP with ion-selective electrodes has been explained by the lower response rate of the corresponding electrodes at lower concentration [31]. Also, in our case, there are two other reasons that may contribute to the observed hyper-Nernstian response: the super-Nernstian steady-state response of the membrane itself and the partial reaction between triiodide of the sample plug and thiosulphate of the carrier solution. In the first case, a concentration region with enhanced steady-state potential response has been theoretically predicted and experimentally observed for ion-exchange membrane electrodes exposed to ions that are preferred to that initially present in the membrane [32,33]. A similar effect was observed in the calibration graph for triiodide obtained in batch condition of a plasticized PVC membrane similar to that used in the present work [17]. As regards the other reason that may contribute to the observed hyper-Nernstian response, the partial reaction between triiodide and thiosulphate, would give rise to a relatively higher decrease in the concentration of the less concentrated triiodide samples.

The repeatability of the method was studied by carrying out five consecutive injections of 2×10^{-5} and 1×10^{-4} mol l^{-1} triiodide working standard solutions. The relative standard deviations of the peak height were ±0.4 and ±1.8%, respectively. The between-day reproducibility was studied by carrying out the same described procedure on five consecutive days with a 2×10^{-5} mol l^{-1} triiodide working standard solution. The peak height standard deviation obtained from the corresponding means was ±1.0%. The sampling frequency of the method obtained at 2×10^{-5} mol l^{-1} triiodide was 80 samples per hour.

Table 1
Potentiometric selectivity coefficients

Ion	$\log K_{I_3^-,j}$
$H_2PO_4^-$	−5.6
Cl^-	−4.5
$S_2O_3^{2-}$	−4.7
SO_4^{2-}	−4.7
NO_3^-	−4.3
Br^-	−4.0
I^-	−3.0
DDS^-	−0.5
HTA^+	−2.7
BZ^+	−2.3

3.6. Selectivity

The apparent potentiometric selectivity coefficients $K_{I_3^-,j}$ of the developed triiodide-selective electrode in the selected FI system for several inorganic anions, including those present in the carrier composition and for some common anionic and cationic surfactants, were obtained as described in Section 2. The results are shown in Table 1. The values obtained with respect to Cl^-, NO_3^- and SO_4^{2-} are better than those reported in [18] for the corresponding triiodide-selective electrode. The value obtained for benzalkonium, a cationic surfactant commonly used in swimming pools as algicide, is very low, which means that it should not produce any error in the iodometric determination of chlorine-containing compounds using the proposed method.

3.7. Response mechanism

In a the previous paper [17], we discussed the different mechanisms proposed to explain the potentiometric response of blank plasticized PVC membranes to hydrophobic ions and explained the high response obtained to triiodide. Since then, experimental results obtained with neutral carrier-based ion-selective electrodes based on membranes without added ionic sites and with membranes free of intrinsic ionic sites (prepared by the previous purification of solvent) have been reported by others authors [34,35] and by our group [36]. Transient potentials and optical second harmonic generation signals, which have been attributed to the extraction of primary ion salts, were obtained. The combined study using both potential and optical second harmonic generation signals allowed the authors to confirm the occurrence of time-dependent charge separation across the phase boundary. The transient potential signals showed a maximum or minimum for cationic or anionic primary ions, respectively. The duration of the whole transient signal was about 40 min for a liquid membrane without PVC. For plasticized PVC membranes the maximum and minimum had the form of transient potential plateau several minutes long. This duration permits stable potential measurement to be carried out within that zone, which can be used for calibration purposes in a similar way to equilibrium potentials.

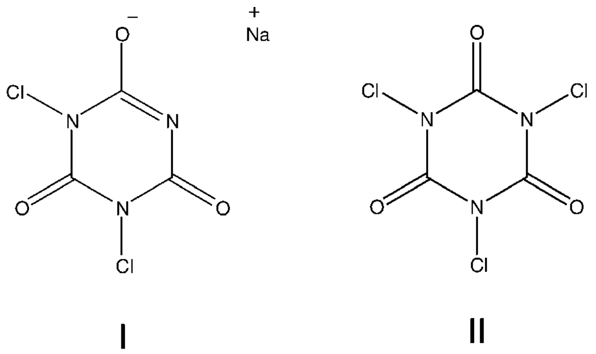

Fig. 4. Formulas of (I) sodium dichloroisocyanurate and (II) trichloroisocyanuric acid.

3.8. Applications

The FI potentiometric method proposed for the determination of triiodide using a plasticized PVC membrane-based detector was applied to the determination of different chlorine-containing disinfectants in several commercial products. The method relies on their reaction with excess iodide to produce triiodide. It was applied to the determination of active chlorine in household bleaches and sterilising tablets, and of sodium hypochlorite in sterilising solutions and of chlorine-release products for the treatment of swimming pools (Fig. 4). The results obtained were compared with those obtained by the corresponding reference method (Table 2) by applying the paired *t*- and *F*-test at the 95% confidence level. No significant difference in accuracy or precision was found between the methods.

Table 2
Determination of chlorine-containing disinfectants in commercial products

Sample	Labeled	Present method[a]	Comparative method[a]
Bleach	Active chlorine ($g\,l^{-1}$)		
1	54	53.5 ± 0.6	53.5 ± 0.4
2	50	50.0 ± 0.8	50.6 ± 0.2
3	40	39.6 ± 0.5	39.2 ± 0.2
4	–	54.3 ± 0.5	55.6 ± 0.2
5	–	51.2 ± 0.8	51.5 ± 0.1
Sterilizing tablets	Active chlorine ($g\,tablet^{-1}$)		
1	–	0.231 ± 0.001	0.246 ± 0.002
2	–	0.513 ± 0.003	0.526 ± 0.002
Sterilizing solution	Sodium hypochlorite (%)		
1	2	1.83 ± 0.03	1.86 ± 0.03
Swimming pool product	Polychloro triazinetrione (%)		
1	99[b]	98.9 ± 0.3	98.1 ± 0.3
2	99[c]	100 ± 2	100 ± 1
3	–[c]	94.1 ± 0.8	93.7 ± 0.2

[a] Mean ± standard deviation (*n* = 3).
[b] 1,3-Dichloro-s-2,4,6-triazinetrione sodium derivative dihydrate.
[c] 1,3,5-Trichloro-s-2,4,6-triazinetrione.

4. Conclusion

A plasticized PVC membrane can be used as a selective potentiometric detector of low triiodide concentrations in flow-injection systems. Good sampling throughput can be achieved by introducing a low concentration of thiosulphate in the carrier solution. The detector is useful for the iodometric determination of chlorine-containing disinfectants in commercial samples and can probably be applied to the determination of other oxidants.

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