Simultaneous spectrophotometric determination of iodate and bromate in water samples by the method of mean centering of ratio kinetic profiles

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Abstract

A new and very simple kinetic-spectrophotometric method was developed for the simultaneous determination of binary mixtures of iodate and bromate in water samples, without prior separation steps. The method is based on the mean centering of ratio kinetic profiles, allows rapid and accurate determination of bromate and iodate. The analytical characteristics of the method such as detection limit, accuracy, precision, relative standard deviation (R.S.D.) and relative standard error (R.S.E.) for the simultaneous determination of binary mixtures of iodate and bromate were calculated. The results show that the method was capable of simultaneous determination of 0.05–$1.50\,\mu\mathrm{g\,mL^{-1}}$ each of iodate and bromate. The results allow simultaneous determination with the ratio 30:1–1:30 for iodate–bromate. The proposed method was successfully applied to the simultaneous determination of iodate and bromate in several water samples.
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1. Introduction

Iodate and bromate have been reported to be toxic ions [1,2]. These ions have been extensively used as ingredients for indicators in catalytic-kinetic analysis involving redox processes. The major route of environmental exposure to these oxihalides is drinking water [3]. Bromate is a by-product of bromide containing water ozonization. Iodide ions may be found in brackish water and to a lesser extent, in freshwater, and may form iodate during ozonization. Therefore, their determinations are required for environmental protection.

Several methods have been reported for the determination of iodate and bromate in mixtures. These include the spectrophotometric determination of periodate-iodate mixtures [4–9], chromatographic [10] and polarographic determination of iodate–bromate and periodate–bromate mixtures [11].

The simultaneous determination of two or more compounds in the same sample without previous chemical separation has been developed from several mathematical procedures.

Derivative of the ratio spectra has been reported for simultaneous determination of binary and ternary mixtures [12–20]. We proposed a new spectrophotometric method for the simultaneous determination of ternary mixtures, without prior separation steps [21]. The method that is called the successive derivative ratio spectra is based on the successive derivatives of ratio spectra in two steps. Unfortunately the advantages of derivative spectra are at least partially offset by degradation in signal-to-noise ratio that accompanies obtaining derivatives.

We also proposed a novel and very simple method based on the mean centering of ratio spectra for the simultaneous determination of binary and ternary mixtures without prior separation steps [22]. This method eliminates derivative steps and therefore, signal-to-noise ratio is enhanced. After modeling procedure, the method has been successfully applied to the simultaneous analysis of binary mixtures of mefnamic acid and paracetamol and ternary mixtures of acetylsalysilic acid, ascorbic acid and paracetamol.

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Recently we proposed a new method based on mean centering of ratio profiles for resolution of binary kinetic profiles and therefore for the simultaneous determination of binary mixtures [23] by kinetic methods. The mathematical explanation of the procedure was illustrated. After modeling procedure, the method has been successfully applied to the simultaneous analysis of binary mixtures of cobalt and nickel based on their complexation reactions with 1-(2′-pyridylazo)-2-naphtol (PAN) in micellar media without any preliminary separation.

In this paper we propose a simple, rapid, precise and accurate method for the simultaneous determination of iodate and bromate based on the difference in the rate of their reaction with iodide ion. In the proposed method we applied mean centering of ratio kinetic profiles for resolution of binary mixtures of iodate and bromate ions.

2. Theoretical background

Consider two analytes A and B that react with a common reagent to give the absorbing species, P_A and P_B, according to the following scheme:

$$A + R \xrightarrow{k_A} P_A \tag{1}$$

$$B + R \xrightarrow{k_B} P_B \tag{2}$$

$$\frac{d[P]}{dt} = k_A C_A + k_B C_A \tag{3}$$

where, k_A, k_B and C_A, C_B are the rate constants and concentration amounts for A and B, respectively.

The product formed, P, can be monitored by recording its absorption spectrum as a function of time or by measuring the absorbance at a fixed wavelength with time.

It has been assumed that the reactions involved both processes to follow a first or pseudo-first order kinetic with respect to the analyte concentrations. If the absorbance is assumed to be proportional to the amount of product formed, then, in the absence of interactions between the kinetics of both analytes, it will be given by

$$A_T = A_{P_A} + A_{P_B} \tag{4}$$

where A_{P_A} and A_{P_B} are the absorbencies for the reaction product of analyte A and B, respectively. Eq. (4) can be rewritten as:

$$A_{T(t)} = \sum_i E_{(i,t)} C_i^0 \tag{5}$$

where, C^0 is the initial concentration of the specie i to be quantified, for first-order reactions and $E_{(i,t)} = \varepsilon_\lambda [1 - \exp(-kt)]$.

By analogy between $E_{(i,t)}$ and molar absorptivity in the Beer–Lambert law in spectrophotometric determinations, the variation of the absorbance as a function of time at a given wavelength can be used to construct a 'kinetic profile', A_{t1}, A_{t2}, ..., A_{tn} at times t_1, t_2, ..., t_n.

For a binary mixture of A and B if Eq. (5) is divided by $E_{(B,t)}$ corresponding to the kinetic profile of a standard solution of B in binary mixture, the ratio profile is obtained in the form of Eq. (6) (for possibility of dividing operation, the zero values of $E_{(B,t)}$ should not be used in the divisor):

$$D = \frac{A_{T(t)}}{E_{B(t)}} = \frac{E_{A(t)} C_A}{E_{B(t)}} + C_B \tag{6}$$

where $E_{A(t)}$ is the kinetic profile of A and $E_{B(t)}$ is the kinetic profile of B. If the Eq. (6) is mean centered (MC), since the mean center of a constant (C_B) is zero, Eq. (7) would be obtained:

$$MC(D) = MC\left[\frac{E_{A(t)} C_A}{E_{B(t)}} \right] \tag{7}$$

Eq. (7) is the mathematical foundation of multi-component analysis that permits the determination of concentration of each of the active compounds in the solution (A in this equation) without interfering from the other compound of the binary system (B in these equations). As Eq. (7) shows there is a linear relation between the amount of MC(D) and the concentration of A in the solution.

A calibration curve could be constructed by plotting MC(D) against concentration of A in the standard solutions of A or in the standard binary mixtures. For more sensitivity the amount of MC(D) corresponding to maximum or minimum time could be measured.

Calibration graphs for B could also be constructed as described for A.

3. Experimental

3.1. Apparatus

A Perkin Elmer Lambda 45 UV/Vis spectrometer was used for recording and storage of UV–vis absorbance spectra and kinetic curves using quartz cells and slit width of 0.5 cm. A Metrohm model 713 pH-meter with a combined glass electrode was used for pH measurements. All calculations in the computing process were done in Matlab6.5 and Microsoft Excell for windows. A simple program was written for this purpose in Matlab6.5.

3.2. Reagents

Triply distilled water and analytical-reagent grade chemicals were used. Standard solutions of iodate and bromate were prepared by dissolving appropriate amounts of sodium iodate and potassium bromate (all from Merck) in water. A $0.1\,\text{mol}\,\text{L}^{-1}$ iodide solution was prepared by dissolving 3.75 g sodium iodide (Merck) in water and diluting to the mark in a 250 mL volumetric flask. A $0.5\,\text{mol}\,\text{L}^{-1}$ sulfuric acid solution was prepared with diluting concentrated sulfuric acid (Merck).

3.3. Procedure

Appropriate volumes of iodate–bromate standard solutions and 1 mL of 0.5 mol L^{-1} sulfuric acid were added into a 10 mL volumetric flask. The solution was diluted to ca. 9 mL with water and 1 mL of 0.1 mol L^{-1} iodide solution was added to initiate the reaction. A stopwatch was started just after the addition of the iodide solution. The solution was diluted to the mark and a portion of it was transferred into a 1-cm glass cell to measure the absorbance change with time at 352 nm.

A calibration graph for iodate is obtained by recording and storing the kinetic profiles of standard solutions containing different concentrations of iodate and bromate. The stored kinetic profiles of the solutions of iodate are divided by standard kinetic profile of bromate according to Eq. (6). Then mean centering (MC) of these vectors with respect to time are obtained according to Eq. (7). The minimum or maximum of the mean centering of ratio profiles with respect to time is used for the construction of calibration graph for iodate. For the prediction of concentration of iodate in synthetic binary mixtures and real samples the same procedure was used except that the kinetic profiles of the mixtures were used instead of the kinetic profiles of standard solution of iodate. The construction of calibration curves for bromate and also its prediction step was performed as described for iodate.

Simultaneous determination could be performed with the ratio 30:1–1:30 for iodate–bromate. Iodate and bromate can be determined simultaneously in the range 0.05–1.50 µg mL^{-1}.

4. Results and discussion

Iodate and bromate react with iodide in acidic media to produce triiodide according to the following reactions:

$$IO_3^- + 8I^- + 6H^+ \rightleftarrows 3I_3^- + 3H_2O \tag{8}$$

$$BrO_3^- + 9I^- + 6H^+ \rightleftarrows 3I_3^- + Br^- + 3H_2O \tag{9}$$
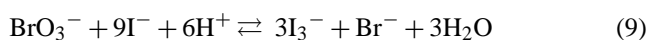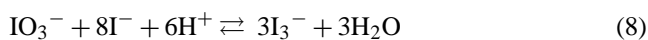
The reactions could be monitored spectrophotometrically by measuring the absorbance of the solutions at 352 nm with time, which is proportional to the produced I$_3^-$ concentration.

It was observed that the reaction of iodate with iodide in acidic media was completed immediately after mixing; while, the reaction of bromate with iodide in the same conditions was relatively slow (Fig. 1). Therefore, mean centering of ratio profiles can be used for binary kinetic profiles resolution of iodate and bromate.

4.1. Preliminary study of system

We have previously reported the optimum conditions such as sulfuric acid concentration, iodide concentration and temperature for the reactions of iodate and bromate with iodide
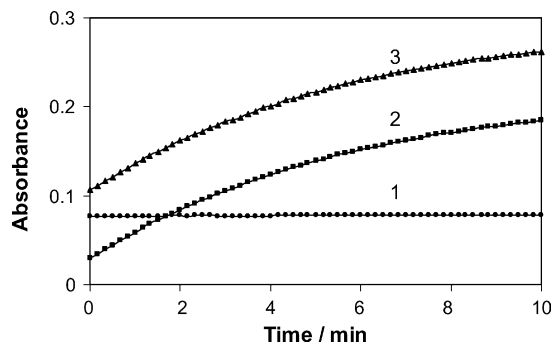

Fig. 1. Absorption kinetic profiles for the reaction of 0.50 µg mL^{-1} iodate (1), 0.50 µg mL^{-1} bromate (2) and their mixture (0.50 µg mL^{-1} each of them) (3) with 0.01 mol L^{-1} iodide in 0.05 mol L^{-1} sulfuric acid solution and 25 °C at 352 nm.

[9]. Therefore, optimum conditions for simultaneous determination of iodate and bromate were chosen as follows: the sulfuric acid concentration is 0.05 mol L^{-1}, iodide concentration is 0.01 mol L^{-1} and temperature is 25 °C.

4.2. Proposed method

The absorption kinetic profiles of the standard solutions of iodate with different concentrations were recorded at 352 nm in the time range 0–10 min with 10 s intervals (Fig. 2a) and divided by the normalized kinetic profile of the bromate and the ratio profiles were obtained (Fig. 2b). Mean centering (MC) of the ratio profiles were obtained in the time range of 1.5–10 min (Fig. 2c). The concentration of iodate was determined by measuring the amplitude at 1.5 min corresponding to a maximum time shown in Fig. 2c. For the prediction of concentration of iodate in synthetic binary mixtures and real samples the same procedure was used except that the kinetic profiles of the mixture were used instead of the kinetic profiles of standard solution of iodate.

The absorption kinetic profiles of the standard solutions of bromate with different concentrations were recorded at 352 nm in the time range 0–10 min with 10 s intervals (Fig. 3a) and divided by the normalized kinetic profile of the iodate and the ratio profiles were obtained (Fig. 3b). Mean centering (MC) of the ratio profiles were obtained in the time range of 1.5–10 min (Fig. 3c). The concentration of bromate was determined by measuring the amplitude at 1.5 min corresponding to a minimum time shown in Fig. 3c. For the prediction of concentration of bromate in synthetic binary mixtures and real samples the same procedure was used except that the kinetic profiles of the mixtures were used instead of the kinetic profiles of standard solution of bromate.

4.3. Analytical characteristics

In the proposed method, Beer's law was obeyed in the concentration range 0.05–1.50 µg mL^{-1} for both iodate and bromate. Table 1 shows the linear regression parameters for
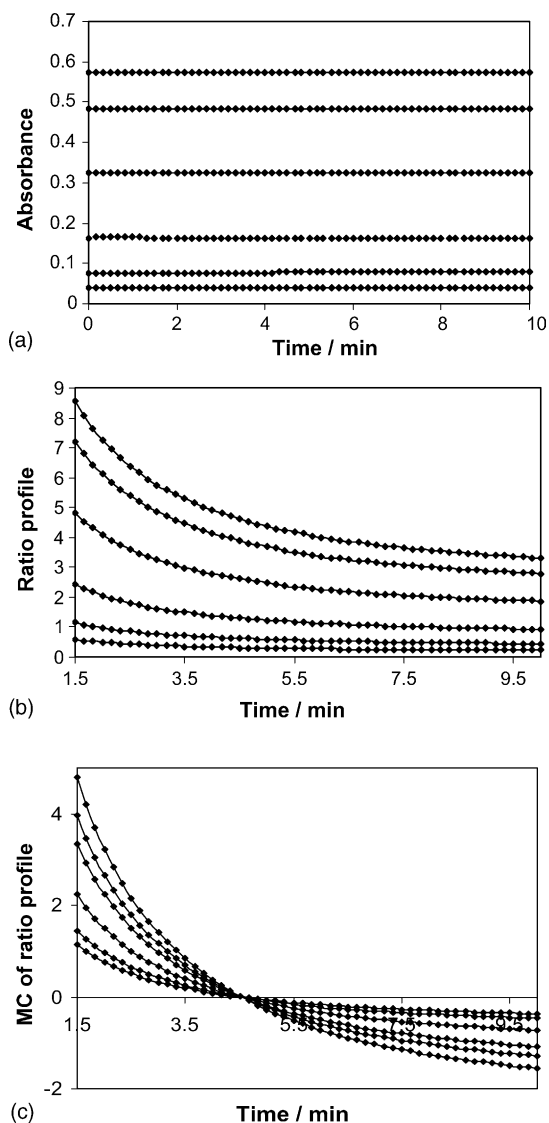
Fig. 2. The absorption kinetic profiles for the standard solutions of the iodate with different concentrations (0.1, 0.2, 0.5, 1 and 1.50 μg mL^{-1}) at 352 nm (a), the ratio profiles obtained by dividing the normalized kinetic profile of the bromate (b) and the mean centering of ratio profiles (c). Conditions: iodide, 0.01 mol L^{-1}; sulfuric acid, 0.05 mol L^{-1}; temperature, 25 °C.
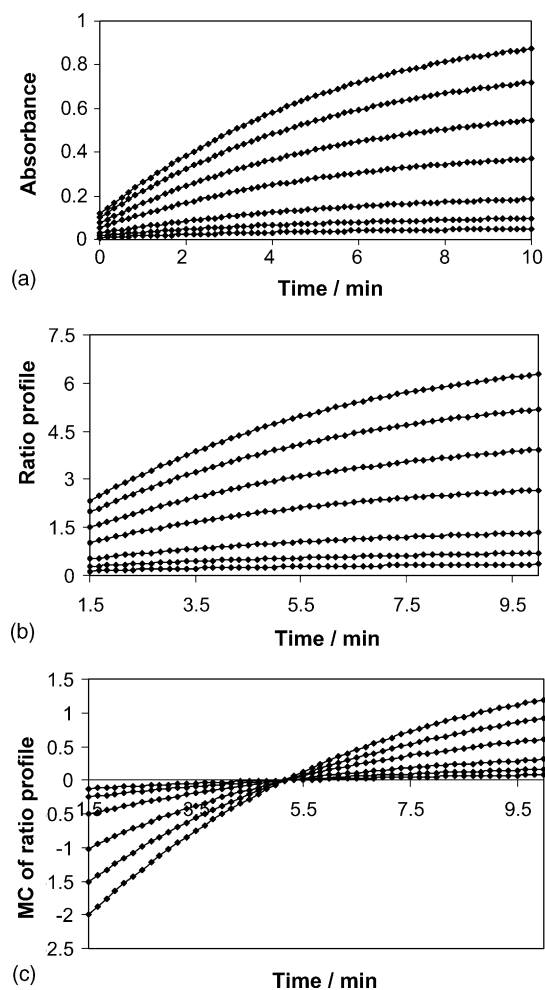

Fig. 3. The absorption kinetic profiles of the standard solutions of the bromate with different concentrations (0.1, 0.2, 0.5, 1 and 1.50 μg mL^{-1}) at 352 nm (a), the ratio profiles obtained by dividing the normalized kinetic profile of the iodate (b) and the mean centering of ratio profiles (c). Conditions: iodide, 0.01 mol L^{-1}; sulfuric acid, 0.05 mol L^{-1}; temperature, 25 °C.

calibration data for simultaneous determination of iodate and bromate in their binary mixtures. Limit of detection of the method for determination of iodate and bromate (defined as the concentration equivalent to three times the standard deviation of five replicate measurements of the blank) are also shown in Table 1.

To check the reproducibility of the method five replicate resolving of iodate and bromate mixtures were performed. The relative standard deviation (R.S.D.) for five replicate determinations of 0.5 μg mL^{-1} each of iodate and bromate was 3.90 and 1.75%, respectively. The mean recoveries for simultaneous determination of these species in binary mixtures were 100 and 102% for iodate and bromate, respectively.

The effect of divisor concentration on the analytical parameters such as detection limit, slope, intercept and

Table 1
Analytical characteristics for analysis of iodate and bromate in binary mixtures by the proposed method

Analyte	Time (min)	Calibration equation[a]	R^{2}[b]	Linear range (μg mL^{-1})	LOD (μg mL^{-1})[c]
Iodate	1.5	$y = 2.8203x - 0.0156$	0.9997	0.05–1.50	0.02
Bromate	1.5	$y = -1.2612x - 0.0067$	0.9971	0.05–1.50	0.01

[a] C is the concentration of metal ion in μg mL^{-1}.

[b] Squared correlation coefficient.

[c] Limit of detection.

Table 2
Results for several experiments for analysis of iodate and bromate in binary mixtures in different concentration ratios by proposed method

Taken (μg mL^{-1})		Found (μg mL^{-1})		Recovery (%)	
Iodate	Bromate	Iodate	Bromate	Iodate	Bromate
0.5	0.2	0.52	0.20	104	100
0.2	1.0	0.22	0.96	110	96
1.0	0.1	0.98	0.10	98	100
1.0	0.5	0.99	0.49	99	98
0.8	0.8	0.80	0.77	100	96
0.05	1.5	0.06	1.54	120	103
1.5	0.3	1.45	0.27	97	90
0.2	0.5	0.21	0.50	105	100
0.1	1.5	0.11	1.47	110	98
0.4	1.0	0.42	0.96	105	96
0.6	1.0	0.60	0.95	100	95
1.5	0.05	1.45	0.05	97	100
Mean recovery				104	98
R.S.E. (%) single[a]				2.96	3.40
R.S.E.$_t$ (%) total[b]					3.19

[a] Calculated from Eq. (8).
[b] Calculated from Eq. (9).

correlation coefficient of the calibration graphs was also tested. It was observed that changing the concentration of divisors in their linear calibration range had no significant effect on the analytical parameters. Therefore, a normalized kinetic profile of each of the iodate and bromate was used as divisor profile in the proposed method.

In order to obtain the accuracy and precision of the method, several synthetic mixtures with different concentration ratios of iodate and bromate were analyzed using the proposed method. The results are given in Table 2. The prediction error of a single component in the mixtures was calculated as the relative standard error (R.S.E.) of the prediction concentration [23]:

$$\text{R.S.E. (\%)} = \left(\frac{\Sigma_{j=1}^{N}(\hat{C}_j - C_j)^2}{\Sigma_{J=1}^{N}(C_j)^2} \right)^{1/2} \times 100 \qquad (8)$$

where N is the number of samples, C_j the concentration of the component in the jth mixture and $\hat{C}_j$ the estimated concentration. The total prediction error of N samples is calculated as follows:

$$\text{R.S.E.}_t \text{ (\%)} = \left(\frac{\Sigma_{i=1}^{M} \Sigma_{j=1}^{N}(\hat{C}_{ij} - C_{ij})^2}{\Sigma_{i=1}^{M} \Sigma_{J=1}^{N}(C_{ij})^2} \right)^{1/2} \times 100 \qquad (9)$$

where C_{ij} is the concentration of the component in the jth samples and $\hat{C}_{ij}$ is its estimation. Table 2 also shows the reasonable single and total relative errors for such system.

4.4. Interference study

To study the selectivity of the proposed method, the effect of various ions on the determination of a mixture of 0.5 μg mL^{-1} of iodate and 0.5 μg mL^{-1} of bromate was tested under the optimum conditions. The tolerance limit was defined as the concentration of added ion causing less than a ±5% relative error. The results show that the ions CO_3^{2-}, ClO_4^-, ClO_3^-, SO_4^{2-}, citrate, tartarate, NO_3^-, acetate, NH_4^+, Cl^- and Br^- did not interfere, even when present in 2000-fold excess over iodate and bromate. Sulfide and sulfite ions interfered by competing with I^- in the reduction of

Table 3
Determination of iodate and bromate mixtures in water samples by the proposed method

Sample	Spiked (μg mL^{-1})		Found (μg mL^{-1})[a]		Recovery (%)	
	IO_3^-	BrO_3^-	IO_3^-	BrO_3^-	IO_3^-	BrO_3^-
River water	0.50	0.50	0.50 ± 0.025	0.51 ± 0.012	100	102
	0.70	0.70	0.68 ± 0.0173	0.69 ± 0.010	97	98
Spring water	0.50	0.50	0.52 ± 0.015	0.50 ± 0.015	104	100
	0.70	0.70	0.71 ± 0.0278	0.72 ± 0.0208	101	103
Well water	0.50	0.50	0.51 ± 0.021	0.49 ± 0.010	102	98
	0.70	0.70	0.70 ± 0.0306	0.68 ± 0.0252	100	97

[a] Mean ± S.D. ($n = 3$).

iodate and bromate, and therefore caused negative interferences. MoO_4^{2-}, WO_4^{2-} and NO_2^- caused negative interference because of their reaction with iodide ion.

4.5. Application

The proposed method was successfully applied to the determination of mixtures of iodate–bromate after addition to water samples. The results are given in Table 3. As it can be seen the results are all satisfactory.

5. Conclusion

The proposed method for the resolving of binary mixtures of iodate–bromate is simple, rapid, accurate, very sensitive and easy to understand and apply. The proposed method was compared with the existing methods reported for the simultaneous determination of iodate and bromate [9,24–26]. The results show that the proposed method is simpler [24,25], more accurate [9,26] and more rapid [9,24–26] than the existing methods and provided a wider dynamic range.

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