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Simultaneous determination of artificial sweeteners, preservatives, caffeine, theobromine and theophylline in food and pharmaceutical preparations by ion chromatography

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Abstract

A novel ion chromatographic method was proposed for the simultaneous determination of artificial sweeteners (sodium saccharin, aspartame, acesulfame-K), preservatives (benzoic acid, sorbic acid), caffeine, theobromine and theophylline. The separation was performed on an anion-exchange analytical column operated at 40°C within 45 min by an isocratic elution with 5 m*M* aqueous NaH_2PO_4 (pH 8.20) solution containing 4% (v/v) acetonitrile as eluent, and the determination by wavelength-switching ultraviolet absorbance detection. The detection limits (signal-to-noise ratio 3:1) for all analytes were below the sub-μg/ml level. Under the experimental conditions, several organic acids, including citric acid, malic acid, tartaric acid and ascorbic acid, did not interfere with the determination. The method has been successfully applied to the analysis of various food and pharmaceutical preparations, and the average recoveries for real samples ranged from 85 to 104%. The levels of all analytes determined by this method were in good agreement with those obtained by the high-performance liquid chromatographic procedure. The results also indicated that ion chromatography would be possibly a beneficial alternative to conventional high-performance liquid chromatography for the separation and determination of these compounds.

Keywords: Food analysis; Sodium saccharin; Aspartame; Acesulfame-K; Benzoic acid; Sorbic acid; Caffeine; Theobromine; Theophylline

1. Introduction

Food additives play a vital role in the modern food industry, and are generally used for maintaining food quality and characteristics as well as promoting food safety. Among them, artificial sweeteners, preservatives and caffeine (CA) are widely used throughout the world. In recent years, concern about their safety in foods has been growing.

The most common artificial sweeteners include sodium saccharin (SA), aspartame (ASP) and acesulfame-K (AK), which are all permitted for use in about 90 countries. They provide benefits to consumers by improving food choice, particularly for those demanding such products in order to reduce energy or sugar intakes for health reasons, and to the food industry by increasing shelf life of some products compared with the sugar sweetened ones. SA has been the center of controversy during the last few decades due to its possible carcinogenic effects [1,2]. The products containing it must carry a warning

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label in the United States [1,2]. Because a small group of people who have the hereditary disease phenylketonuria are sensitive to phenylalanine, one of the metabolites of ASP, all products containing ASP must be labeled for phenylalanine in many countries [1–3]. The acceptable daily intake (ADI) values, determined by the Joint FAO/WHO Expert Committee on Food Additives, are 0–5, 0–40, and 0–15 mg/kg body mass for SA, ASP, and AK, respectively [4].

As widely used food preservatives, benzoic acid (BA) and sorbic acid (SOR) exhibit inhibitory activity against a wide variety of fungi, yeasts, molds, and bacteria, including most foodborne pathogens [5]. A broader spectrum of microbicidal activity is often achieved by using a combination of them, which inhibit several bacterial strains better than either of them alone [5]. The ADI values for BA and SOR are 0–5 and 0–25 mg/kg body mass, respectively [4].

CA is added to soft drinks as a flavor. It also naturally occurs in tea, coffee, cocoa beans, accompanied by its analogues theobromine (TB) and theophylline (TP). These three alkaloids can produce extensive bioactivity. However, excessive intake of them will cause many undesirable side effects [6].

In many instances, more than one kind of these compounds is added to foods to realize different functions. Accordingly, it is very necessary to establish an accurate and reliable method for the simultaneous determination of these eight compounds for assurance of food safety. Although a great variety of methods have been applied to the analysis of these compounds in foods, only a few [7–21] are suitable for the simultaneous determination of more than three kinds of them. High-performance liquid chromatography (HPLC) is the most frequently used method nowadays. Most HPLC procedures were based on isocratic or gradient reversed-phase (RP) chromatographic separation with acetonitrile, methanol or isopropanol in phosphate buffer or acetic acid solution as mobile phase, and ultraviolet (UV) absorbance detection [7–15]. RP ion-pair chromatography [16] was also used for the simultaneous determination of SA, ASP, BA and CA. In the past few years, micellar electrokinetic chromatography has been applied to the simultaneous determination of several kinds of these compounds in foods [17–21] due to its high separation efficiency and its feature of separation of neutral molecules as well as ions. In addition, ion chromatography (IC) offered an attractive alternative to traditional HPLC methods. Chen et al. proposed two isocratic IC assays for the simultaneous determination of CA, TB and TP in various foods and pharmaceutical preparations [6]. SA, ASP and AK in foods could also be separated on an anion-exchange separation column by using gradient [22] or isocratic elution [23]. To the best of our knowledge, no IC assay has been reported for the simultaneous determination of all these eight compounds so far. The aim of the present study is to develop an IC method for the separation and determination of all these compounds in one chromatographic run.

2. Experimental

2.1. Apparatus

A Waters high-performance liquid chromatograph (Milford, MA, USA) equipped with a 600 gradient pump was employed together with a Waters Millennium 2010 chromatography manager workstation (version 2.10) for instrument control as well as data acquisition and processing. The separation was achieved by a Shimadzu Shim-pack IC-GA3 guard column (Kyoto, Japan) and a Shimadzu Shim-pack IC-A3 analytical column (150×4.6 mm, 5 μm) with 5 m*M* aqueous NaH_2PO_4 (adjusted to pH 8.20 with 0.5 mol/l NaOH) solution containing 4% (v/v) acetonitrile as eluent (1.00 ml/min), and the determination by a Waters 486 tunable absorbance detector which was controlled by a wavelength-switching program. The detection wavelength was set at 205.0 nm initially, and switched to 227.0 nm at 12.70 min of run time, and then back to 205.0 nm at 28.00 min. When the detection wavelength was switched from one wavelength to another, the detector would automatically perform the ''auto zero'' function [24] to maintain a flat and stable baseline without a drift. The column temperature was maintained at 40°C by using a Waters temperature control module, and the injection volume of standard and sample solutions at 50 μl by a Waters 717plus autosampler. In this study, the peak area measurements for all calculations were adopted.

The preliminary spectrometric experiments were carried out using a Hitachi U-3000 spectrophotometer (Tokyo, Japan) with 1-cm quartz cells at room temperature.

2.2. Reagents

The water for the preparation of all solutions was made by a Millipore Milli-Q RG ultra-pure water system (Bedford, MA, USA), and HPLC-grade acetonitrile was obtained from Fisher Scientific (Fair Lawn, NJ, USA). All chemicals were of analytical grade or higher purity unless otherwise stated.

CA and TP were obtained from the National Institute for the Control of Pharmaceutical and Biological Products (Beijing, China), ASP and TB from Sigma (St. Louis, MO, USA), and AK from Supelco (Bellefonte, PA, USA). The stock solutions (0.25 mg/ml for TB, 0.5 mg/ml for CA and TP, and 1.00 mg/ml for ASP as well as AK) were prepared separately by dissolving appropriate amounts of these compounds in water. The stock solutions (1.00 mg/ml) of SA, BA and SOR were obtained from the National Research Center for Certified Reference Materials (Beijing, China). The working solutions were prepared by serial dilution of the stock solutions with water.

2.3. Preparation of real samples

All samples, including two carbonated cola drinks (samples A and B), one fruit juice drink (sample C), one fermented milk drink (sample D), one preserved fruit (sample E) and one pharmaceutical preparation (tablet of CA and sodium benzoate, sample F), were purchased from local market. They were pretreated by the relevant procedures as follows, and the final solutions of all samples were filtered through 0.45-μm filters before the chromatographic analysis.

2.3.1. Cola drinks

An amount of ca. 2 g of cola drink was accurately weighed into a 50-ml volumetric flask, and then degassed for 3 min in an ultrasonic bath followed by the dilution to volume with water.

2.3.2. Fruit juice drink

An accurately weighed amount of ca. 10 g of fruit juice was diluted to 25 ml with water. A Millipore Sep-Pak C_{18} cartridge (Milford, MA, USA) was conditioned by successive elution of 5 ml of acetonitrile and 5 ml of water prior to use. An aliquot of 5 ml of diluted sample solution was applied to this cartridge at a flow-rate of ca. 2 ml/min, and then the analytes were eluted with 5 ml of 10 m*M* NaH_2PO_4–acetonitrile (3:1, v/v). All filtrates were combined and diluted to 25 ml with water.

2.3.3. Fermented milk drink

A quantity of ca. 2.5 g of homogenized milk drink was accurately weighed into a 25-ml volumetric flask, and 10 ml of water was added. The solution was shaken vigorously, extracted by ultrasonication for 10 min, and then added with 2 ml of 0.085 mol/l $K_4[Fe(CN)_6]$ and 2 ml of 0.25 mol/l $ZnSO_4$ solutions after cooling to room temperature. The resulting solution was vigorously shaken for 2 min and diluted to volume with water. This solution was completely transferred to a centrifuge tube, and centrifuged at 1600 *g* for 10 min. A 10-ml volume of supernatant was applied to a conditioned Sep-Pak C_{18} cartridge. The subsequent solid-phase extraction procedure was the same as mentioned above.

2.3.4. Preserved fruit

An amount of ca. 2 g of finely ground and homogenized preserved fruit was accurately weighed into a 50-ml volumetric flask, and 30 ml of water was added. This solution was extracted ultrasonically for 30 min, and diluted to volume with water after cooling to room temperature. A 1-ml volume of supernatant was applied to a conditioned Sep-Pak C_{18} cartridge. The subsequent solid-phase extraction was conducted as mentioned above.

2.3.5. Tablet

An accurately weighed amount of ca. 0.1 g of powdered and homogenized tablet was dissolved and diluted to 100 ml with water, and 1 ml of this solution was diluted to volume with water in a 100-ml volumetric flask.

3. Results and discussion

3.1. Choice of separation system

Because the pK_a values of BA, SOR and TP are 4.19, 4.76 and 8.8, respectively [25], and the pK_1 and pK_2 values of ASP, a dipeptide consisting of aspartic acid and phenylalanine, are 2.96 and 7.37, respectively [26], these compounds exist completely or partially as anions in basic solution. AK and SA exist as anions even in acidic solution [15,27]. In principle, apart from pure ion-exchange processes, hydrophobic interactions between ion-exchange resin and organic compounds contribute to the retention [28]. So, CA and TB, which exist as molecules in weak basic solutions (the pK_a values of CA and TB are 14.0 and 10.0, respectively [25]), are also possibly retained on an anion-exchange column. Therefore, it is possible to separate all these eight compounds by anion-exchange chromatography with basic eluent. Our previous studies [22,23] showed that three sweeteners could be separated on an ethylenevinylbenzene–divinylbenzene based anion-exchange column. TP was strongly retained while TB and CA were hardly retained on the same column [6]. On a more hydrophobic anion-exchange column, the retentions of CA and TB were enhanced, but their peaks still appeared just after the void time [6]. Considering both columns were designed for use in suppressed IC, in this study, a Shim-pack IC-A3 anion-exchange column packed with polyacrylate resin with quaternary amine groups, designed for use in nonsuppressed IC, was employed for varying the selectivity and thus improving the separation performance.

Because the usable pH of eluent is in the range of 3.0–8.5 for this column, the typical eluents used in nonsuppressed IC, such as phthalic acid and 4-hydroxybenzoic acid solutions, were firstly employed. Unfortunately, when using conductivity detection, the sensitivities were all poor, and three alkaloids even had no response. UV absorbance detection was not applicable either because of the strong background absorbance of eluent. Finally, phosphate buffer solution with low UV absorbance was selected because its pH value could be easily controlled in a wide range. In order to improve selectivity and prevent the peak broadening, acetonitrile was added into eluent due to its advantages for low-wavelength UV detection. The retention behavior of each analyte was evaluated based on the retention factor (k), and 50 μl of water was injected in each chromatographic system to determine the system void time [29].

In addition to supply elution ions, phosphate solution regulated the ionic charge of analytes and the fraction of analytes in anionic forms by regulating the pH value. Because the pK_{a1}, pK_{a2} and pK_{a3} values of phosphoric acid are 2.16, 7.21 and 12.32, respectively [30], in the pH range we studied (pH 5.4–8.2), the predominant elution ions are $H_2PO_4^-$ and HPO_4^{2-}, and the elution power of HPO_4^{2-} is stronger. In the case of the constant total concentration of phosphates, the higher the pH value, the more fraction of HPO_4^{2-} thus the stronger the eluent strength. The pH value of phosphate solution was adjusted by adding 0.5 mol/l NaOH into NaH_2PO_4 solution rather than adding H_3PO_4 into Na_2HPO_4 solution because the total concentration of phosphates could not maintain constant under the different pH conditions by the latter method. By increasing the pH value, the retention factors of ASP and TP increased, those of TB and CA basically kept constant, while those of the other analytes decreased (Fig. 1). In the pH range of 5.4–8.2, CA and TB existed in the molecular forms and were retained by hydrophobic interactions. On this account, the varying of pH had little effect on their retention. SOR, BA, AK and SA existed as anions and were retained by ion-exchange in combination with hydrophobic interactions. So, their retention decreased with an increase in elution strength of eluent caused by the increased pH. When the acidity of eluent was strong enough, TP existed as molecules and retained by hydrophobic interactions. When the pH value was as low as 5.8, TP was eluted before CA. By increasing the pH value, TP could be converted to anions in part and retained by both ion-exchange and hydrophobic interactions. Although the eluent strength increased, the retention factor of TP increased significantly, demonstrating the ionization of TP was the more important factor for determining its retention under this condition. For ASP, which existed as zwitterion or/and anion in the same pH range, both ion-exchange and hydrophobic interactions contributed to its retention. As the pH value of eluent was increased, the positive charge of the protonated

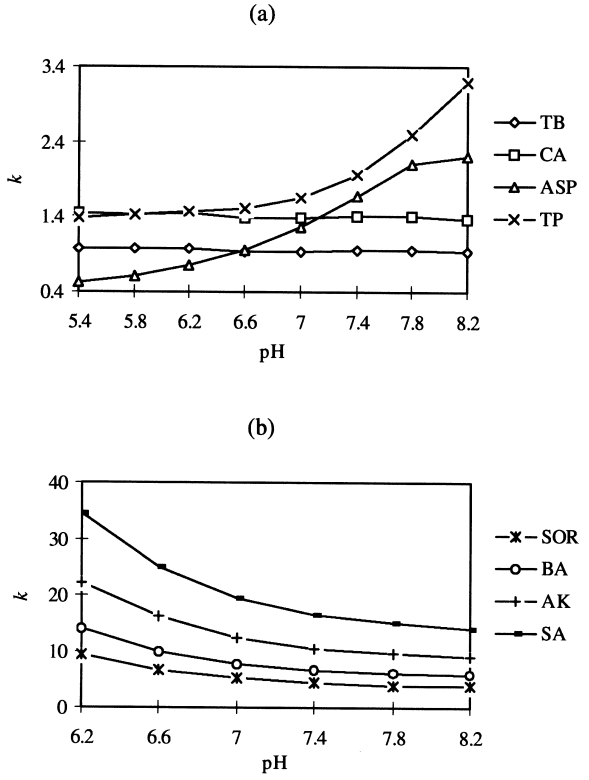

Fig. 1. The effect of pH of phosphate buffer solution on the retention of analytes. Eluent: 6 m*M* aqueous NaH_2PO_4 solution containing 4% (v/v) acetonitrile. Column temperature: 40°C.

amino group in zwitterion decreased, and the fraction of ASP in anionic form increased, resulting in the increased ion-exchange interactions and the increased retention factor. Because the peaks of ASP and TP overlapped partially when pH value was 7.40 or 7.80, taking all factors into consideration, the pH value of phosphate solution was set at 8.20.

As the increase of phosphate concentration, the elution strength for anions increased and thus the retention factors of ASP, TP, SOR, BA, AK and SA decreased. Changes in phosphates concentration had little effect on the retention factors of TB and CA. Finally, phosphates concentration was selected as 5.0 m*M*.

By increasing the acetonitrile concentration, the retention factors of all analytes decreased. When acetonitrile concentration was too low, the separation time was unnecessarily prolonged and the peaks of strongly retained compounds, such as BA, AK, and SA, were seriously tailing. When acetonitrile concentration was too high, the baseline separation could not be achieved for TB and CA. Finally, acetonitrile concentration was chosen at 4% as a compromise.

3.2. Choice of column temperature

By increasing the column temperature in the range of 30–50°C, the retention factors of all analytes except ASP decreased, and the resolutions between TB and CA, as well as TP and SOR were improved. The increase of retention factor of ASP (Fig. 2) possibly resulted from the increased ionization of ASP at higher temperatures, and consequently the increased fraction of the anionic form in eluent, and the stronger ion-exchange interactions with stationary phase. Even so, the resolution between ASP and TP was not damaged because both peaks became sharper due to the improved column efficiency. The different effects of column temperature on retention of analytes in IC will likely provide a means for improving the separation selectivity. Since the highest operating temperature was 50°C for this column, finally, the column temperature was chosen as 40°C.

3.3. Choice of detection wavelength

Since all these analytes have UV chromophores, UV detection has reasonably been the first choice in liquid chromatographic and capillary electrophoretic analysis of these compounds [7–23]. Because their maximum absorption wavelengths are significantly different, a wavelength-switching technique should

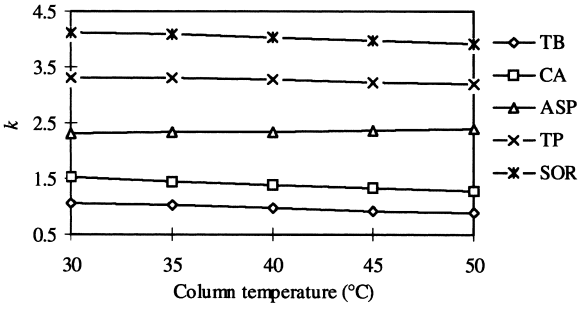

Fig. 2. The effect of column temperature on the retention of some analytes. Eluent: 5 m*M* aqueous NaH_2PO_4 (pH 8.20) solution containing 4% (v/v) acetonitrile.

be a better solution for improving the detection limits of all analytes. Because the maximum absorption wavelengths obtained from a spectrophotometer (eluent as blank) for TB, CA, ASP, TP and SOR were 203.0, 205.5, 192.5, 202.5 and 254.0 nm, respectively, and their peaks were very close, for detection of these five analytes, 205.0 nm was selected as a compromise where the background absorbance of eluent was low. The maximum absorbance wavelength of BA, 196.5 nm, was not adopted because the background absorbance of eluent was also high. Because its secondary maximum absorption wavelength, 224.0 nm, was very near to the maximum absorption wavelength of AK, 227.0 nm, in order to simplify detection, the latter was selected for detection of these two analytes. For detection of SA, which maximum absorption wavelength is 201.5 nm, 205.0 nm was chosen where could give satisfactory detection sensitivity while maintaining a low background absorbance of eluent.

3.4. Interference study

In general, some organic acids are naturally present or intentionally added in soft drinks, which can provide sharpness, bite and brightness. Under the specified experimental conditions, the commonly used organic acids including citric acid, malic acid, tartaric acid and ascorbic acid, which were eluted near the void time or have very low UV responses, did not interfere with the detection of eight target analytes. Besides, phosphoric acid, normally used in cola-based drinks, was eluted within the void volume.

3.5. Analytical characteristics

Under the optimum experimental conditions, all analytes showed good linearity between the concentrations and peak area responses. The detection limits (signal-to-noise ratio 3:1) were also calculated (Table 1). The precisions were evaluated by performing eight replicate analyses of a standard solution where the concentration of SA was 10 μg/ml, the concentrations of CA, TB and TP were 5 μg/ml, and the concentrations of the other analytes were 20 μg/ml. The relative standard deviations (RSDs) were 1.0, 1.2, 1.7, 0.8, 0.4, 1.0, 1.9 and 1.5% for TB, CA, ASP, TP, SOR, BA, AK and SA, respectively.

3.6. Analysis of real samples

All samples were analyzed according to the experimental conditions, and ASP, TB as well as TP were not detected. The results are summarized in Table 2, and the chromatograms of standard solution and sample A shown in Fig. 3. The accuracy of the proposed method was evaluated by comparing the results with those obtained by an RP-HPLC procedure [15]. The results demonstrated in Table 2 reveal an excellent agreement between the results determined by these two methods, which indicates that IC would be a beneficial alternative to HPLC for the separation and determination of these compounds. Additionally, the spike recovery studies were conducted by using samples A, D and E, and the average recoveries ranged from 85 to 104%, with RSDs of 1–5% ($n=4$).

Table 1
Linearity and detection limits for analytes

Analyte	Concentration range (μg/ml)	Regression equation[a]	Correlation coefficient ($n=5$)	Detection limit (ng/ml)
TB	0.25–10	$A=(3.78c+0.30)\cdot10^5$	0.9999	4
CA	0.25–10	$A=(4.36c+0.43)\cdot10^5$	0.9999	4
ASP	1–40	$A=(1.02c+0.50)\cdot10^5$	0.9999	30
TP	0.25–10	$A=(4.78c+0.16)\cdot10^5$	0.9982	6
SOR	1–40	$A=(1.39c+0.46)\cdot10^5$	0.9999	20
BA	1–40	$A=(1.74c+0.64)\cdot10^5$	0.9997	10
AK	1–40	$A=(1.55c-0.02)\cdot10^5$	0.9999	20
SA	0.5–20	$A=(3.43c-0.12)\cdot10^5$	0.9993	20

[a] A=Peak area response (arbitrary unit), c=concentration of analytes (μg/ml).

Table 2
Analysis of real samples

Sample	Content[a] (mg/kg or mg/g)									
	IC					HPLC				
	CA	SOR	BA	AK	SA	CA	SOR	BA	AK	SA
A	109±1		145±4		133±6	114±1		149±3		137±2
B	91±1		120±1			90±4		120±4		
C				82±3					81±1	
D		529±11					545±9			
E					4720±140					4810±80
F	402±4		432±6			409±2		445±4		

[a] Average of four determinations±standard deviation, mg/g for sample F and mg/kg for the other samples.

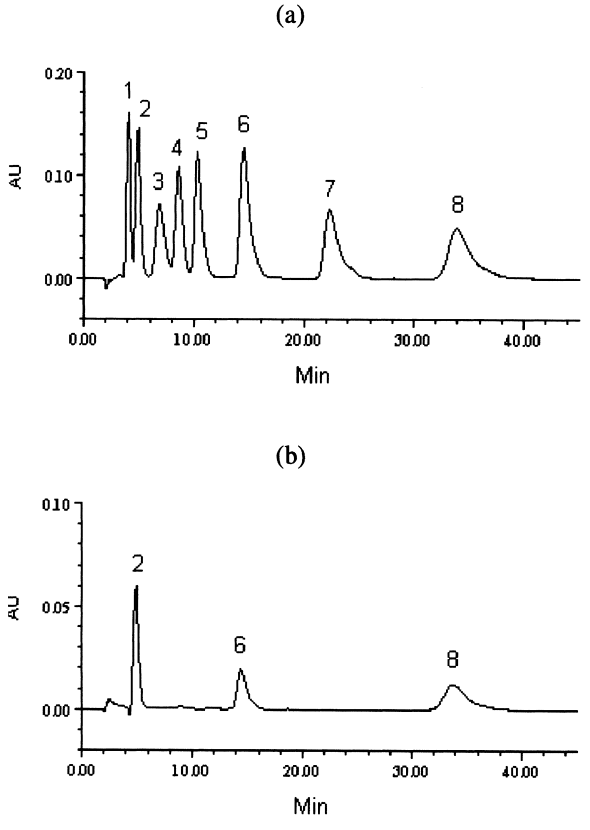

Fig. 3. Chromatograms of (a) standard solution where the concentration of SA was 20 μg/ml, the concentrations of CA, TB and TP were 10 μg/ml, and the concentrations of the other analytes were all 40 μg/ml, and (b) the final solution of sample A. Peaks: 1=TB, 2=CA, 3=ASP, 4=TP, 5=SOR, 6=BA, 7=AK, 8=SA. Detailed experimental conditions as in the text.

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