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# Simultaneous gas chromatographic determination of food preservatives following solid-phase extraction

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**Abstract**

A simultaneous method for the solid-phase extraction of five preservatives is presented. Of the different sorbent materials assayed, styrene–divinylbenzene polymers provided the highest adsorption efficiency. Samples are inserted into the flow system at pH 1, which results in increased selectivity. Elution is made with 150 μl of ethyl acetate and 2 μl aliquots of the eluate are manually injected into a gas chromatograph equipped with flame ionization detection. Of the two chromatographic columns of different polarity, that containing the more polar stationary phase assayed provided the better results by virtue of the acid character of the analytes. The limits of detection achieved range from 0.1 to 0.2 mg/l, and the precision is quite high (R.S.D.<4%). The potential of the proposed method was assessed by applying it to the determination of sorbic and benzoic acid in foods. Solid samples require pretreatment involving liquid–liquid extraction, evaporation of the extract and dissolution of the residue in 0.1 *M* $HNO_3$. Recoveries ca. 95% for *p*-hydroxybenzoic acid esters can thus be obtained.
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*Keywords:* Food analysis; Extraction methods; Benzoic acids; Sorbic acid

## 1. Introduction

Antimicrobial food additives are used to prevent the proliferation of bacteria, yeasts and moulds; sorbic acid, benzoic acid and *p*-hydroxybenzoic acid esters are used extensively in this context. Sorbic acid and benzoic acid are widely regarded as the most active against yeasts and moulds, and the least active against bacteria; however, it is difficult to obtain substantial evidence on their relative activity from available studies [1,2]. The alkyl esters (methyl, ethyl and propyl) of *p*-hydroxybenzoic acid are often referred to as ''parabens'' and are widely used for their antifungal properties. The preservative effect of parabens tends to increase with increasing molecular mass; the methyl ester is more effective against moulds and the propyl ester against yeasts, (the latter is favoured for oils and fats for solubility reasons [2]). Because the maximum permitted concentrations of preservatives in each type of food are controlled by legislation [3], their determination is a mandatory step in routine food analyses.

A variety of analytical methods for determining preservatives have been reported to date. Spectroscopic methods are usually employed for the individual determination of these compounds. Thus, sorbic acid in raw beef [4] has been determined with low recoveries (52–84%) by photometry following distillation and extraction. Better recoveries (ca. 100%) and less manipulation are involved the determination of 4-hydroxybenzoic methyl ester in nonalcoholic beverages by the fluorescence technique [5]. However, because the additives can be present in combinations, chromatographic methods

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are often used for their selective individual or joint determination. Sorbic and benzoic acid preservatives in beverages have been determined by thin-layer chromatography with minimal sample manipulation [6,7]. High-performance liquid chromatography (HPLC) is often preferred for the determination of mixtures of additives in foods, which are usually scarcely volatile. The simultaneous determination of sweeteners, preservatives and colorings in soft drinks is usually done by HPLC with UV detection [8–10]. Preservatives in yogurt have also been determined by this technique after laborious manipulation of the sample including digestion, filtration, extraction and evaporation prior to injection into the chromatograph [11]. A strategy for selecting the most suitable separation system for the determination of natural compounds (preservatives included) in beverages by using ion-exchange HPLC has been widely discussed [12]. However, the simultaneous separation of additives by HPLC is hindered by polarity differences. Gas chromatography (GC), with or without derivatization, is also employed for the selective determination of food preservatives. The Association of Official Analytical Chemists (AOAC) official GC method [13] for preservatives in foods involves several extractions, evaporation, derivatization to a trimethylsilyl ester and flame ionization detection (FID). Mass spectrometry (MS) is a sensitive, selective technique and is gradually gaining ground in the determination of these additives by GC. The chemical ionization MS mode [14] is more sensitive than the electron impact ionization mode for this purpose [14,15]. Both allow preservatives to be determined with limits of detection of 100–500 pg but involve sample pretreatments similar to that of the AOAC method, which is time-consuming.

Solid-phase extraction (SPE) was introduced in the early 1970s to avoid or minimize the shortcomings of liquid–liquid extraction (particularly the large sample/organic solvents volumes required and its slowness) [16]. Since then, this separation technique has superseded other sample preparation alternatives, aided by the variety of polar and nonpolar sorbents, and exchangers, that have been made commercially available. SPE has also been used for determining food additives [9,10]. Preservatives are retained on a quaternary ammonium sorbent cartridge [9] or a Sep-Pak $C_{18}$ cartridge [10,17] in a preparation step of the overall analytical process in HPLC. However, off-line SPE involve conditioning and flushing the cartridge, and eluting the analytes by hand [9,10,17]; on the other hand, it allows the simultaneous use of several cartridges. On-line SPE is also used as a precolumn concentration technique, usually coupled with HPLC but rarely with GC [16]. No reference to the use of continuous SPE for determining preservatives by GC appears to exist.

The aim of this work was to develop a continuous SPE system for the GC determination of food preservatives. Liquid foods, in a nitric acid medium containing an internal standard, are directly loaded, in a volume-based injection mode, onto the sorbent column for simultaneous enrichment and matrix removal. Solid samples required some pretreatment before the residue is dissolved in 0.1 *M* $HNO_3$. The optimum operating conditions for the proposed method are critically discussed with special emphasis on sensitivity, selectivity and simplicity. The method uses straightforward instruments (FID), requires no derivatization, and is simple and fast to implement.

## 2. Experimental section

### *2.1. Standards and reagents*

Preservatives [benzoic acid, sorbic acid, methyl *p*-hydroxybenzoic acid (M-PHBA), ethyl *p*-hydroxybenzoic acid (E-PHBA) and propyl *p*-hydroxybenzoic acid (P-PHBA)] were supplied by Sigma (Madrid, Spain). 2-*tert*.-Butyl-4-methylphenol (internal standard) was purchased from Aldrich (Madrid, Spain). Potassium perchlorate and anhydrous sodium sulphate were obtained from Merck (Darmstadt, Germany). Ethanol, ethyl acetate, acetone, *n*-hexane, ethyl ether and other organic solvents were obtained in HPLC grade from Scharlau (Barcelona, Spain). All sample dilutions were made with deionized water of 18 MΩ/cm resistivity obtained from a Milli-Q water purification system. Polygosyl-bonded silica reversed-phase sorbent with octadecyl functional groups (RP-$C_{18}$), 40–63 μm and 20–60 mesh; XAD-2 and XAD-7 polymeric sorbents; and polymeric styrene–divinylbenzene sorbent (LiChrolut-

EN) were supplied from Sigma and Merck, respectively.

Standard stock solutions containing each preservative and the internal standard, at a 10 mg/ml concentration, in ethanol were prepared and stored in glass-stopped bottles at 4°C. The optimum GC conditions were established by using a mixture of 50 mg/l of each additive and the internal standard in ethanol. Standard working-strength solutions of variable concentration were prepared daily by appropriate dilution of aliquots of the stock in 0.1 *M* $HNO_3$.

### 2.2. GC and flow system components

A Hewlett–Packard 5890 A gas chromatograph equipped with FID was used. Two chromatographic fused-silica columns (15 m×0.53 mm I.D.) of different polarities were used, one packed with 5% diphenyl–95% dimethylsiloxane, 3 μm film thickness (HP-5), and the other with 50% diphenyl–50% dimethylsiloxane, 1 μm film thickness (HP-50). The oven column temperature was raised from 70°C (2 min) to 150°C at 8 C°/min, and then to 160°C at 6 C°/min. The injector and detector temperatures were kept at 250°C. Nitrogen was used as carrier gas, at a flow-rate of 14.7 ml/min. Peak areas were measured by using a Hewlett–Packard 3392 A integrator.

A Gilson Minipuls-2 peristaltic pump (France) and two Rheodyne 5041 six-way injection valves were used throughout. Poly(vinyl chloride) and Solvaflex pumping tubes for aqueous and organic solutions, respectively, and poly(tetrafluoroethylene) (PTFE) tubing of 0.5 mm I.D. for coils, were also used. Laboratory-made adsorption columns packed with different sorbents were constructed from PTFE capillaries of 3 mm I.D. and their ends capped by fitting 30 mm×0.5 mm I.D. PTFE tubing into a 10 mm×1 mm I.D. PTFE tube, which facilitated insertion into the continuous system. Columns were hand-packed with different amounts of sorbent and sealed at both ends with small plugs of cotton wool to prevent material losses. Initially, the columns were conditioned with 2 ml 0.1 *M* $HNO_3$ at a flow-rate of 2 ml/min. To avoid memory effects, the columns were flushed with acetone between samples.

### 2.3. Sample pretreatment

Soft drinks were degassed in an ultrasonic bath. An accurately weighed amount of ca. 1 g was spiked with 0.25 mg of internal standard (2-*tert*.-butyl-4-methylphenol) and diluted in 25 ml of 0.1 *M* $HNO_3$. A 5 ml aliquot of the diluted sample was directly inserted into the continuous-flow system.

The other samples (skim yogurts, jams and sauces) were homogenized by magnetic stirring. Portions of 0.5 to 1 g, and 0.25 mg of the internal standard, were placed in a separatory funnel and mixed with 25 ml of $10^{-3}$ *M* HCl and 2 ml of saturated potassium perchlorate. The aqueous phase was extracted twice with 10 ml of ethyl ether, for 5 min, the efficiency of the first and second extraction being 80 and 20%, respectively [14]. The collected ethyl ether phase was pooled (20 ml) and evaporated to dryness; the residue was dissolved in 25 ml of 0.1 *M* $HNO_3$ and a 5 ml aliquot was introduced into the SPE system as described below.

### 2.4. Procedure

The continuous-flow system used to isolate and preconcentrate the food preservatives is depicted in Fig. 1. In the preconcentration step, 5 ml of the treated sample or 5 ml of a standard solution containing between 0.2 and 25 μg/ml of additives plus 10 μg/ml of internal standard, at pH 1, was continuously pumped into the system at 2 ml/min. The analytes were adsorbed on the XAD-2 column placed in the loop of the injection valve ($IV_1$) and the effluent was sent to waste. Then, the column was flushed with 150 μl of *n*-hexane (from $IV_2$), carried out by $N_2$ in order to remove residual aqueous phase from the column and connections. Simultaneously, the loop of $IV_2$ was filled with eluent (ethyl acetate) by aspiration from a displacement bottle (DB). As $IV_2$ was switched, 150 μl of ethyl acetate was injected into the $N_2$ stream at 1.5 ml/min and passed through the column to elute preservatives and the internal standard. The eluate was collected in glass vials containing anhydrous sodium sulphate, and a 2 μl aliquot was manually injected into the chromatograph. Between samples, the column was flushed with acetone (three times, with 150 μl volumes from

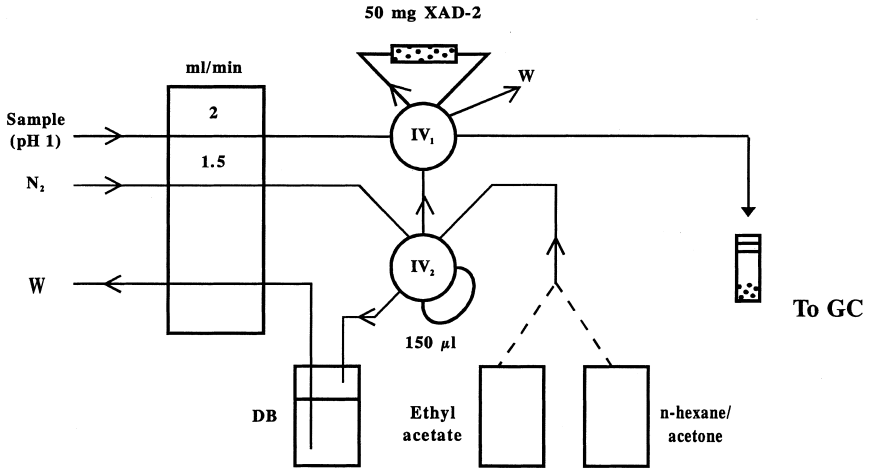


Fig. 1. Flow system for the preconcentration/isolation of preservatives from foods. IV=injection valve; W=waste; DB=displacement bottle; GC=gas chromatograph with FID.

$IV_2$). Finally, the column was conditioned with 2 ml of 0.1 *M* $HNO_3$.

## 3. Results and discussion

### *3.1. Development of the SPE system*

An SPE system was assembled in order to obtain highly sensitive, accurate and reproducible results, with minimal sample handling and maximal throughput. Commonly used sorbents differ in their capacity to adsorb polar or nonpolar organic compounds. Thus, Amberlite XAD resin is typical material for polar aromatic compounds, while $C_{18}$ bonded silica is more suitable for nonpolar ones. In addition to the sorbent properties, the SPE of preservatives is critically influenced by pH in the adsorption step. Because the proposed method was intended for use with food samples (with high moisture contents), standards were dissolved in water. The sample pH should be such that it ensures maximal retention of the analytes and minimal retention of interferents on the column. The effect of this variable was studied by using four columns packed with 50 mg of RP-$C_{18}$, XAD-2, XAD-7, or LiChrolut-EN at pH 2.5, 4 and 7. A manifold similar to that depicted in Fig. 1 was used; the eluent was 5 ml of ethanol and 2 µl aliquots were injected into the chromatograph. Volumes of 5 ml of standard solutions containing 25 mg/l of the five additives were passed through each sorbent column. The results are listed in Table 1. If ethanol is assumed to effect quantitative elution, the conclusions can be drawn: first, pH has more marked effects on sorbic acid and benzoic acid than on the esters (the highest adsorption efficiency is obtained

Table 1
Percent adsorption of preservatives on various sorbent columns (50 mg) at pH 2.5, 4 and 7

| Preservative | RP-$C_{18}$ | | | XAD-2 | | | XAD-7[a] | | | LiChrolut-EN | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 2.5 | 4 | 7 | 2.5 | 4 | 7 | 2.5 | 4 | 7 | 2.5 | 4 | 7 |
| Sorbic acid | <10 | <10 | <10 | 90 | 90 | 70 | 20 | 20 | 15 | 85 | 85 | 20 |
| Benzoic acid | <10 | <10 | <10 | 90 | 85 | 55 | 20 | 20 | 10 | 75 | 70 | 35 |
| M-PHBA | <10 | <10 | <10 | 70 | 65 | 50 | 30 | 25 | 20 | 70 | 70 | 65 |
| E-PHBA | 25 | 25 | 25 | 70 | 70 | 60 | 30 | 30 | 30 | 60 | 60 | 60 |
| P-PHBA | 50 | 50 | 50 | 75 | 75 | 70 | 30 | 30 | 30 | 50 | 50 | 50 |

[a] XAD-7 was previously dried at 80°C.

at a low pH); second, RP-$C_{18}$ is the poorest adsorbent, particularly for more polar compounds; and third, the adsorption efficiency is similar for LiChrolut-EN and XAD-2, but slightly better for the latter. XAD-2 and LiChrolut-EN, two hydrophobic styrene–divinylbenzene polymers, exhibited the highest efficiency for the preservatives, so both were initially selected for use. A more exhaustive study of the sample pH was made over the range 0.5–10 by adjusting the standard solution with diluted $HNO_3$ or NaOH. After elution, the column was conditioned with 2 ml of aqueous solution at the same sample pH. The results obtained with the two columns are shown in Fig. 2. The adsorption efficiency of the XAD-2 column for sorbic acid and benzoic acid remained constant over the pH range 1 to 5, which was wider for *p*-hydroxybenzoic acid esters by virtue of their lower polarity. The adsorption efficiency of the LiChrolut-EN column was lower for all the preservatives, particularly esters. In order to remove aromatic amines and related compounds, which are ionized and hence not retained on the resin at a low pH, a sample pH of 1 was chosen that was also adopted for the conditioning cycle. In subsequent experiments, standard solutions and samples were prepared in 0.1 *M* $HNO_3$. The optimum amount of XAD-2 sorbent was determined; adsorption peaked above 45 mg, so 50 mg selected as optimal. The influence of the sample flow-rate was studied by inserting a standard solution containing 25 mg/l of each antimicrobial additive in 0.1 *M* $HNO_3$. Retained analytes were eluted with 5 ml of ethanol after the column was dried with a $N_2$ stream. The flow-rate of the sample (5 ml) was varied between 1 and 4 ml/min; changes over the range 1–2.5 ml/min were very small; higher flow-rates decreased the analytical signal through decreased residence times in the sorbent column. A flow-rate of 2 ml/min was selected as optimal to retain the preservatives.

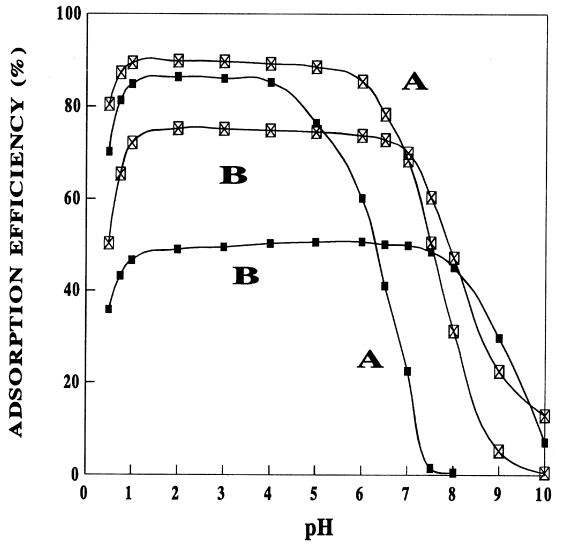


Fig. 2. Effect of pH on the adsorption of two representative preservatives on XAD-2 (☒) and LiChrolut-EN (■) sorbent columns. (A) Sorbic acid and (B) propyl *p*-hydroxybenzoic acid (P-PHBA).

Because the sample was introduced in aqueous medium, the column and flow connectors should be dried in order to remove residual water before elution. Initially, the column was dried with $N_2$ for 10 min, but some aqueous phase was found to still remain in the system. We thus assayed alternative solvents (*n*-hexane, cyclohexane and acetonitrile) for this purpose. *n*-Hexane was chosen because although it is immiscible with water, it helps remove it while leaving retained analytes untouched. Therefore 150 μl of *n*-hexane, carried by a $N_2$ stream, was used to flush and dry the column.

Several eluents of variable polarity were tested, namely: ethyl acetate, ethanol, methanol, 2-propanol and petroleum ether. Elution was optimized by using a standard solution containing 25 mg/l in 0.1 *M* $HNO_3$ that was passed through the column at 2 ml/min for 2.5 min (sample volume, 5 ml); retained preservatives were eluted with 5 ml of each solvent and then analysed in the chromatograph. Ethyl acetate was found to be the best eluent; the analytical signals were 1.2- or 1.7-times higher than those obtained with ethanol and 2-propanol, or methanol, respectively. The elution efficiency of petroleum ether was lower than 3%. The effect of the eluent volume was studied between 100 and 300 μl by using loops of variable length in the injection valve ($IV_2$ in Fig. 1). Obviously, as the eluent volume was increased, desorption was more efficient (but analytes were also more dilute). Because of these two opposing effects, the only way to correctly determine the most appropriate eluent volume was to dilute extracts to a constant volume with the same solvent. Thus, the column effluent (between 100 and 300 μl

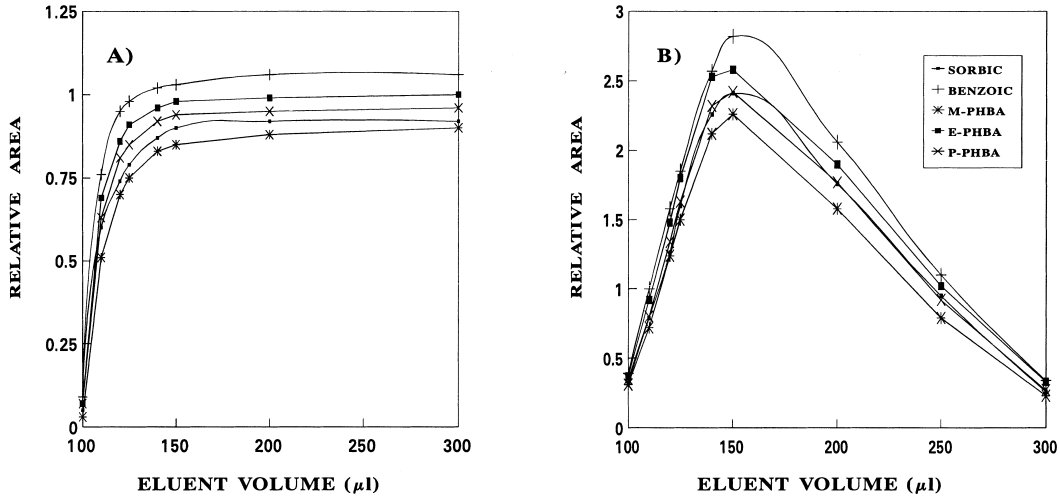


Fig. 3. Variation of the relative (analyte/internal standard) area with the eluent volume. (A) Dilution of extracts to a final ethyl acetate volume of 500 μl. (B) Undiluted extracts. Sample: a standard solution containing 5 mg/l of each additive in 0.1 *M* $HNO_3$.

of ethyl acetate) was always diluted to 500 μl with ethyl acetate (Fig. 3A). The desorption efficiency increased with increasing injected volume up to 150 μl and remained constant above this value. Unless the final extract was made to a constant final volume, the desorption efficiency decreased above 150 μl through increased dilution of desorbed analytes (see Fig. 3B). An injected volume of 150 μl of ethyl acetate was selected as optimal. A second injection with the same eluent volume revealed the absence of carry-over; thus, complete elution of analytes was obtained with one injection of 150 μl of ethyl acetate. The influence of the flow-rate of the $N_2$ stream (the carrier of the eluent volume) was examined between 0.5 and 2 ml/min. No effect on preservative recoveries was observed, so 1.5 ml/min was selected for further experiments.

Several solvents were assayed to flush the sorbent column following elution and acetone was selected. It was introduced via the loop of $IV_2$, carried by $N_2$; three sequential injections of 150 μl sufficed to remove unwanted substances and/or avoid memory effects.

### *3.2. GC behaviour*

Preservatives are normally determined by GC after derivatization to trimethylsilyl esters [13,15], which entails heating at 60–80°C for 15–30 min. In this work, we assayed direct injection into a GC of 50 mg/l of benzoic acid, sorbic acid, M-PHBA, E-PHBA and P-PHBA in ethanol, and found no tailing in the chromatographic peaks obtained. Therefore, for simplicity, the analytes were determined underivatized. Benzoic acid and sorbic acid are weakly acidic compounds of higher polarity than *p*-hydroxybenzoic acid esters. Therefore, their retention on the chromatographic column can be affected by the composition and polarity of the stationary phase. Two chromatographic columns of different polarity were assayed in order to select the better chromatogram, where peaks could be sensitively and selectively, detected without no interference.

A sample volume of 5 ml at pH 1 and the SPE manifold of Fig. 1 were adopted. 2-*tert*.-Butyl-4-methylphenol was used as internal standard because it was found to be compatible with the flow system (ca. 60% adsorption and 100% elution efficiency) and the chromatographic behaviour of the analytes. The sensitivity (slope of the calibration graphs) and the linear ranges achieved with both chromatographic columns are shown in Table 2. Correlation coefficients ranged from 0.996 to 0.999. Limits of detection, defined as the minimum concentrations providing a chromatographic signal three times higher than background noise (at the prevailing

Table 2
Analytical figures of merit of the proposed SPE method

| Preservative | Sensitivity[c] | Linear range (mg/l) | Detection limit (mg/l) | R.S.D. (%) |
|---|---|---|---|---|
| Sorbic acid[a] | $8.4\cdot10^{-2}$ | 0.3–25 | 0.10 | 3.8 |
| Sorbic acid[b] | $7.1\cdot10^{-2}$ | 0.5–25 | 0.20 | 4.0 |
| Benzoic acid[a] | $9.4\cdot10^{-2}$ | 0.2–25 | 0.07 | 2.9 |
| Benzoic acid[b] | $7.5\cdot10^{-2}$ | 0.4–25 | 0.15 | 3.0 |
| M-PHBA[a] | $7.1\cdot10^{-2}$ | 0.5–25 | 0.20 | 2.8 |
| M-PHBA[b] | $6.7\cdot10^{-2}$ | 0.5–25 | 0.20 | 2.7 |
| E-PHBA[a] | $7.6\cdot10^{-2}$ | 0.4–25 | 0.15 | 2.7 |
| E-PHBA[b] | $7.3\cdot10^{-2}$ | 0.4–25 | 0.15 | 2.6 |
| P-PHBA[a] | $7.5\cdot10^{-2}$ | 0.4–25 | 0.15 | 2.5 |
| P-PHBA[b] | $7.1\cdot10^{-2}$ | 0.5–25 | 0.20 | 2.5 |

[a] With an HP-50; [b] with an HP-5 chromatographic column; [c] (analyte/internal standard peak area ratio)/(mg/l).

retention times), are also listed in Table 2. Within-day precision (repeatability), expressed as relative standard deviation, was calculated for 11 standard solutions containing 10 mg/l of each additive and the internal standard, and found to range from 2.5 to 4%. A comparison of the results provided by the two columns (Table 2) allows one to conclude the following: (a) the sensitivity is higher for the HP-50 column (the more polar) than for the HP-5 one; (b) the detection limit is lowest for sorbic acid and benzoic acid with the HP-50 column; and (c) the precision is similar in all instances. Finally, as can be shown in Fig. 4, similar retention times were obtained for both columns, and no tailing peaks. From the foregoing it follows that the HP-50 column is the better choice.

### *3.3. Determination of sorbic and benzoic acid, and p-hydroxybenzoic acid esters in foods*

The sample pretreatment prior to GC analysis in the determination of preservatives in foods involves liquid–liquid extraction, anion-exchange clean-up, precipitation of proteins etc. The AOAC method [13] is laborious and time-consuming, so it was discarded. We chose the most simple reported sample pretreatment for complex matrices [14], albeit slightly

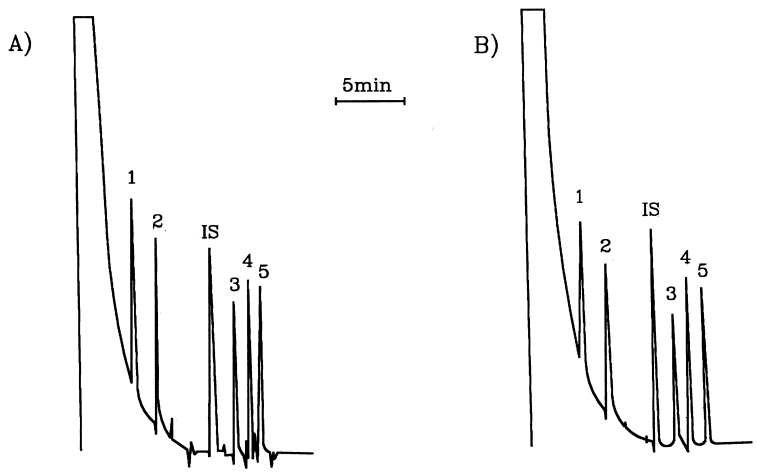


Fig. 4. Gas chromatograms for preservatives (10 mg/l) after on-line enrichment of a 5 ml sample volume, using an HP-50 (A) and an HP-5 (B) chromatographic column. Peaks: 1=sorbic acid, 2=benzoic acid, 3=M-PHBA, 4=E-PHBA, 5=P-PHBA, I.S.=internal standard.

modified as described in Section 2.3. Soft drinks only required degassing of the sample.

The proposed method was applied to the determination of preservatives in soft drinks, jams, and low-fat yogurts and sauces (to avoid the need to previously remove the fat). Following pretreatment (see Section 2.3), the aqueous solution was introduced into the SPE system and analysed by GC–FID. The results are given in Table 3; the average concentrations were calculated from five individual amounts of each sample and determinations were done in triplicate in the SPE manifold ($n=15$). The preservatives contained in each food (sorbic and benzoic acid) were stated on the labels, but their amounts were not. Only plum jam and the ketchups were found to contain both additives. All the results were below allowed levels in the European Union (EU), both for individual and for mixed additives [3]. EU regulations prohibit the use of *p*-hydroxybenzoic acid esters in the foods studied, so they could obviously not be detected.

Because only two additives were detected, the potential of the proposed method for determining all five studied was assessed by spiking the foods before treatment with 200 mg/kg of the three *p*-hydroxybenzoic acid esters studied and analysing them in quintuplicate. The recoveries thus obtained ranged from 92 to 103%.

Table 3
Determination of sorbic and benzoic acid in foods (mg/kg food)

| Food | Sorbic acid | Benzoic acid |
|---|---|---|
| Soft drinks | | |
| Cola | – | 130±5 |
| Orange 1 | – | 120±4 |
| Orange 2 | 170±7 | – |
| Lemon 1 | – | 140±5 |
| Lemon 2 | 190±8 | – |
| Apple | – | 145±5 |
| Skim yogurts | | |
| Strawberry | 280±10 | – |
| Apple–plum–cereals | 265±10 | – |
| Lime–lemon | 250±10 | – |
| Tropical fruits | 175±8 | – |
| Forest fruits | 270±10 | – |
| Ketchups | | |
| Hot | 280±15 | 255±20 |
| Plain | 170±7 | 485±20 |
| Jams | | |
| Strawberry | – | 215±10 |
| Peach | 255±15 | – |
| Plum | 225±10 | 190±8 |

## 4. Conclusions

The proposed flow injection on-line SPE method affords high sensitivity and selectivity in preservative determinations and is free of interferences from other concomitants or the solvent peak. It delivers results within 5 min after sample preconcentration/elution, so throughput is limited only by the chromatographic conditions (sample preparation time excluded). The limits of detection achieved are similar to or better than those of the existing standard method, even with liquid extraction. Also, the use of large amounts of expensive and environmentally hazardous organic solvents and derivatizing reagents is avoided. Complex matrices (dairy products, sauces etc) entail converting the sample into an aqueous solution at pH 1 (the lowest of all reported so far). The proposed method can be used by control laboratories to identify and quantify the five preservatives studied in a wide variety of foods.

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