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The determination of sorbic acid and benzoic acid in a variety of beverages and foods by micellar electrokinetic capillary chromatography

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A rapid method for the identification and quantitation of sorbic acid and benzoic acid in a variety of beverages and foods by micellar electrokinetic capillary chromatography (MECC) is described. Dehydroacetic acid was used as the internal standard. The separations were achieved using a 68 cm fused silica capillary column with a buffer comprising 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The amounts of preservatives determined by MECC were in good agreement with those determined by the high-performance liquid chromatographic (HPLC) procedure currently used in the authors' laboratory. The MECC procedure has the same order of repeatability as the HPLC method and is also faster, more efficient and less costly to operate. This procedure can also be used for the screening of sorbic acid, benzoic acid and dehydroacetic acid in beverages using phthalate as the internal standard.

INTRODUCTION

Sorbic acid, benzoic acid and their salts are added as preservatives to a wide variety of foods and beverages available in Australia. Dehydroacetic acid can also serve as a food preservative, but is rarely used and is not listed in the Australian Food Standards Code (Anon., 1994). High-performance liquid chromatography (HPLC) is the most common analytical procedure for detecting and quantifying sorbic, benzoic and dehydroacetic acids in foods and beverages (Ikai *et al.*, 1988). Gas–liquid chromatography (GC) can also be used as the determinative step (Williams, 1984); this is more time-consuming as the workup requires solvent extraction of the sample, followed by derivatisation of the acids to the trimethylsilyl esters before analysis. Sorbic acid, benzoic acid and dehydroacetic acid have recently been separated by capillary isotachophoresis (Karovicova *et al.*, 1991). The related technique of micellar electrokinetic capillary chromatography (MECC) has been used for the separation and quantitation of a wide variety of compounds and is gaining popularity as a viable analytical tool (Li, 1992; Trenerry *et al.*, 1994*a,b*). This paper describes a rapid and sensitive MECC method for the determination of sorbic acid and benzoic acid in a variety of foods. A comparison of the results with the HPLC method currently used in the authors' laboratory is also reported.

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MATERIALS AND METHODS

Materials

Benzoic acid, sorbic acid, dehydroacetic acid and potassium hydrogen phthalate were obtained from BDH Chemicals, Pty Ltd (Kilsyth, Australia). Sodium dodecyl sulphate (SDS) was obtained from E. Merck (Darmstadt, Germany). All other chemicals and solvents were of AR grade or HPLC grade and used without further purification.

Samples

The samples studied included four brands each of 100% orange juice, 100% apple juice, cordial, diet cordial, fruit drink (containing 25% fruit juice), soft drinks and cheese slices. Only two brands of low-alcohol wines, two of low-joule jams and three samples of dips were analysed as these products were not readily available from local suppliers.

Preparation of standards, samples, buffers

Standards

MECC. Stock solutions of benzoic acid, sorbic acid, dehydroacetic acid and potassium hydrogen phthalate were prepared at a concentration of 5 mg/ml by dissolving in methanol. These were then mixed and diluted with deionised water to provide working standards of

different concentrations. Dehydroacetic acid was used as the internal standard at a concentration of 25 μg/ml for the quantitation of sorbic acid and benzoic acid in foods and beverages, while potassium hydrogen phthalate served as internal standard at a concentration of 5 μg/ml for the screening of sorbic, benzoic and dehydroacetic acids in beverages. The standards were stable up to one week. Each standard solution was basified to >pH 9 using 1M NaOH and then filtered through a 0·8 μm cellulose acetate filter unit prior to analysis.

HPLC. Varying concentrations of standard solutions were prepared by mixing and diluting stock solutions of sorbic and benzoic acids with deionised water. These were used without being basified.

Samples

Quantitation

MECC.

- *Juices/fruit drinks:* The juices were mixed thoroughly. A suitable aliquot of the sample (1 ml) was added to 1·25 ml of the internal standard solution (dehydroacetic acid, 500 μg/ml) and diluted with deionised water (10 ml). The solution was basified to pH>9 with 1M NaOH, made to volume (25 ml) with deionised water and then filtered through a 0·8 μm cellulose acetate filter before analysis.
- *Jams/preserves:* Approximately 0·5 g of the sample was weighed accurately, mixed with 1·25 ml of the internal standard solution (dehydroacetic acid, 500 μg/ml) and then diluted with deionised water (10 ml). The solution was basified to pH>9 with 1M NaOH, made to volume (25 ml) with deionised water and then filtered through a 0·8 μm cellulose acetate filter before analysis.
- *Cheese slices/dips:* The acid preservatives were extracted from the sample by steam distillation using a Tecator Kjeltec 1026 distilling Unit (Sweden). The sample was homogenised in a blender and approximately 10 g was weighed accurately into a Kjeltec tube containing 20 g of sodium chloride and 2·5 ml of a 20% tartaric acid solution. The samples were then steam distilled and about 180 ml of the distillate was collected into a 250 ml volumetric flask. The volume was made to the mark with deionised water. Suitable aliquots (1–5 ml) of the solution were diluted with deionised water (10 ml) and the pH adjusted to >9 with 1M NaOH. A 1·25 ml sample of the internal standard solution (dehydroacetic acid, 500 μg/ml) was added and the solution diluted to 25 ml with water, mixed thoroughly and filtered through a 0·8 μm cellulose acetate filter before analysis. The apparatus was thoroughly washed with deionised water between samples.

HPLC. The samples were prepared as described for MECC, except that the solutions were not basified. An internal standard was not used for HPLC analysis.

Screening procedure

MECC and HPLC

(a) This procedure is applicable to grape juice concentrate and white wine. The grape juice concentrate and the wine (10 ml) were diluted with deionised water and the final volume made to 50 ml. The solutions were filtered through a 0·8 μm cellulose acetate filter unit before analysis. For MECC analysis, the solutions were basified to pH>9 with 1M NaOH before making to volume. The limit of reporting for this procedure was 10 mg/litre for MECC and 5 mg/litre for HPLC.

(b) This procedure is applicable to red wine. A 50 ml sample of wine was neutralised with 10% NaOH/10% H_2SO_4. A 5 ml sample of 15% tartaric acid solution and 80 g of sodium chloride were added to the solution and the final volume made to between 150 and 200 ml with deionised water. One drop of anti foaming agent (Dow Corning Antifoam Q2-1614) was added and the solution steam distilled in an all glass apparatus until 500 ml of distillate was collected. The liquid was added to 130 g of sodium chloride and 5 ml of 10% HCl in a 1 litre separating funnel. The mixture was extracted with diethyl ether (3 × 40 ml) and the ethereal solution washed with water and dried (Na_2SO_4). The solvent was removed *in vacuo* with a rotary evaporator and the residue dissolved in acetone (5 ml). An aliquot (2·5 ml) was blown to dryness with a stream of nitrogen and made to 5 ml with deionised water containing 1 drop of 5 M NaOH. The solution was filtered through a 0·8 μm cellulose acetate filter disc and analysed by HPLC. For analysis by MECC, 0·2 ml of an internal standard solution (dehydroacetic acid, 108 μg/ml) was added to 1·8 ml of the above aqueous solution, and the final solution filtered through a 0·8 μm cellulose acetate filter disc and analysed. The limit of reporting for HPLC and MECC was 0·1 mg/litre. An all glass steam distillation apparatus was used in preference to a semi-automatic Tecator Kjeltec 1026 Distilling Unit, as carryover of trace amounts of benzoic acid was observed, even though the unit was washed with deionised water between samples.

Buffer for MECC

SDS (0·05 M) was prepared by dissolving 1·44 g of SDS in 100 ml of 0·02 M disodium hydrogen orthophosphate. The pH of the buffer was 9·2. The buffer was prepared as required and filtered through a 0·45 μm cellulose acetate filter unit prior to use. The buffer solutions in the instrument were changed daily.

Apparatus

MECC

The analyses were performed with a 68 cm × 75 μm i.d. fused silica capillary (Isco Inc., Lincoln, NE, USA) with an effective length of 43 cm to the detector. An Isco Model 3140 Electropherograph (Isco Inc., Lincoln, NE, USA) operating at +25 kV and at 27°C was used for the analyses. The sample was loaded onto the column under vacuum (vacuum level 2, 10 kPa). The compounds were detected at 230 nm at 0·02 AUFS for quantitation and at 0·005 AUFS for screening. Electro-

pherograms were recorded with either the ICE Data Management and Control Software supplied with the Model 3140 Electropherograph or a HP 3350 Laboratory Data System (Hewlett–Packard, Palo Alto, CA, USA).

Procedure for capillary preparation and handling

Prior to each batch of samples, the capillary was filled with 0·1 M sodium hydroxide and allowed to stand for 5 min. The capillary was washed with deionised water (5 min), 0·1 M hydrochloric acid (5 min), followed by deionised water (5 min) before filling with the running buffer. The capillary was also flushed with running buffer for 2 min between samples.

HPLC

The analyses were performed with a 501 HPLC pump, 710B WISP and a 490 programmable multiwavelength UV detector using a 10 μm Spherisorb ODS2 C18 column (3·9 mm × 300 mm) equipped with a C18 μBondapak pre-column (Waters Chromatography Division of Millipore, Milford, MA, USA). The mobile phase comprised an aqueous solution of potassium dihydrogen orthophosphate and dipotassium hydrogen orthophosphate each at 2·5 g/litre. The flow rate was 1 ml/min. The compounds were detected at 230 nm at 0·1 AUFS. The data were analysed using a HP 3350 Laboratory Data System (Hewlett–Packard, Palo Alto, CA, USA). A second system using an Exmere ODS2-8/5 C18 column (4·6 mm × 250 mm) with a C18 μBondapak pre-column and a mobile phase consisting of 5 g/litre dipotassium hydrogen orthophosphate adjusted to pH 8 with 1 M orthophosphoric acid was also used. This combination is not recommended as the alkaline buffer substantially reduces the operating life of the column.

RESULTS AND DISCUSSION

The MECC procedure was first validated with standard solutions of different concentrations to determine the linearity range and to check the repeatability of the technique. The standard solutions were run seven times to obtain repeatability data. For the quantitative analyses, a variety of actual market samples containing sorbic acid and/or benzoic acid were analysed. One brand of each of the samples was run seven times to obtain statistical data. Then additional brands of each sample were analysed to obtain comparative data using the MECC and HPLC methods.

For the screening procedure, three samples of grape juice concentrate, a sample each of white wine and red wine not containing dehydroacetic acid, sorbic acid or benzoic acid were analysed using the procedure outlined in method (a). The samples were also spiked with low levels of the preservatives and analysed for recovery data. One sample of red wine was also analysed using the procedure outlined in method (b).

The results will be presented in two sections:

(a) Use of the MECC procedure as a method for quantifying acid preservatives.

(b) Use of the MECC procedure as a screening tool for acid preservatives.

(a) Use of the MECC procedure as a method for quantifying acid preservatives

Baseline separation of dehydroacetic, sorbic and benzoic acids was achieved using a fused silica capillary column with the SDS buffer at pH 9·2 with an applied voltage of +25 kV and a temperature of 27°C. With these conditions, dehydroacetic acid migrated first,

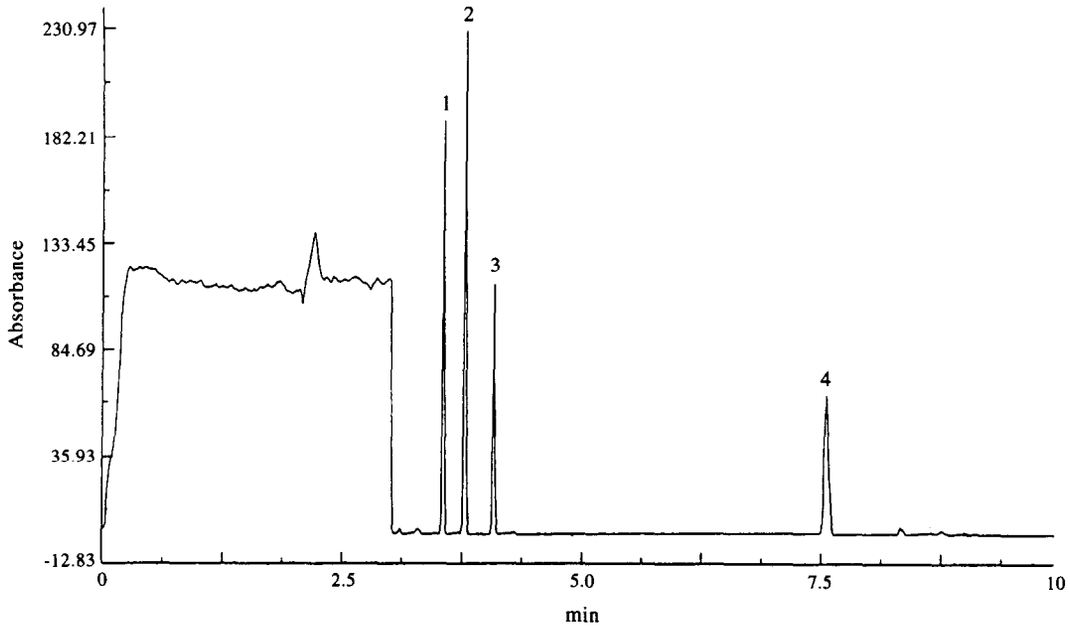

Fig. 1. Separation of (1) dehydroacetate, (2) sorbate, (3) benzoate and (4) phthalate in a standard solution using a buffer consisting of 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The *x*-axis gives the migration times in minutes.

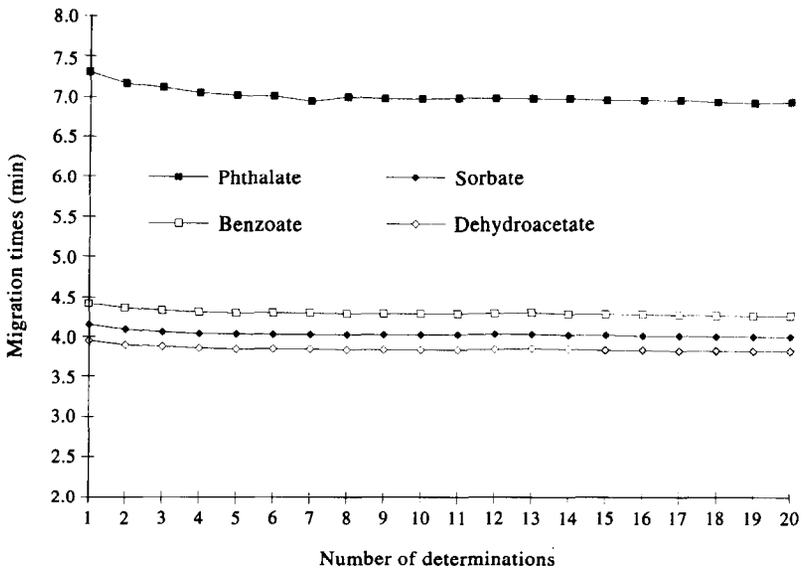

Fig. 2. Migration times for phthalate, benzoate, sorbate and dehydroacetate for 20 replicate determinations using a buffer consisting of 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The *x*-axis gives the number of determinations.

followed by sorbic and lastly benzoic acid. Phthalate migrated much later than the three acids and was therefore suitable as the internal standard for the analysis (Fig. 1).

The standard solution was run 20 times to study the drift in the migration times of the compounds. There were no significant shifts in the migration times of any of the compounds over this period (Fig. 2).

The MECC procedure was then trialled on actual samples. Initially, one sample each of orange and apple juice were prepared and run by MECC. Unfortunately, a naturally occurring compound co-migrated with phthalate in both samples, precluding its use as an internal standard. Dehydroacetic acid was not listed as a preservative in the Australian Food Standards Code (Anon, 1994). It was therefore assumed that it would not be found in any of the Australian foods and so be suitable for use as the internal standard. This had the added advantage of shortening the run time from 8 min to 5 min. It was also assumed that if any samples that were analysed did contain dehydroacetic acid, then either sorbic acid or benzoic acid could be used as internal standard.

Standards were then prepared with dehydroacetic acid as internal standard at a concentration of 25 μg/ml and they were run for linearity and repeatability data at 0·02 AUFS. The statistical data are presented in Table 1. The detector response was linear to 50 μg/ml for sorbic acid and benzoic acid.

When the orange juice and apple juice samples were run with dehydroacetic acid as internal standard and the values compared with those obtained by HPLC, the results were not satisfactory. Re analysing the standard and sample solutions after basifying to pH > 9 with 1M sodium hydroxide resulted in a much better correlation between the two methods. This suggested that the acidic nature of the sample solution, when loaded onto the capillary column, interfered with the electrophoretic behaviour of the acids in the alkaline buffer. Changing the pH of the sample solution from acidic to alkaline would not only make the sample solution more compatible with the alkaline buffer, but would also convert the acids to their corresponding carboxylate anions, and so affect their electrophoretic mobility. It was therefore decided to basify all the standard and sample solutions to pH > 9 by the addition of 1M NaOH prior to analysis by MECC.

Table 1. Linearity and repeatability data showing the area ratios and %CV data for standard solutions of sorbic acid and benzoic acid analysed by MECC using dehydroacetic acid as the internal standard[a]

Standard (μg/ml)	Sorbic acid		Benzoic acid	
	Area ratio	%CV	Area ratio	%CV
10·0	0·50	2·2	0·28	1·3
25·0	1·25	1·3	0·68	1·0
50·0	2·4	1·5	1·31	1·3
100·0	4·6	1·4	2·6	1·8

[a]Results for seven replicate determinations.

Table 2. Linearity and repeatability data showing the area ratios and %CV data for standard solutions of sorbic acid and benzoic acid analysed by MECC using dehydroacetic acid as the internal standard and %CV for the solutions analysed by HPLC. The standard solutions were basified to pH>9 for MECC[a]

Standard (μg/ml)	Sorbic acid			Benzoic acid		
	Area ratio	%CV		Area ratio	%CV	
	MECC	MECC	HPLC	MECC	MECC	HPLC
10·0	0·48	1·1	0·4	0·29	0·8	1·3
25·0	1·22	1·1	0·6	0·71	0·6	0·5
50·0	2·38	1·1	0·3	1·36	1·4	0·4
100·0	4·52	1·2	0·4	2·76	1·9	0·2

[a]Results for seven replicate determinations.

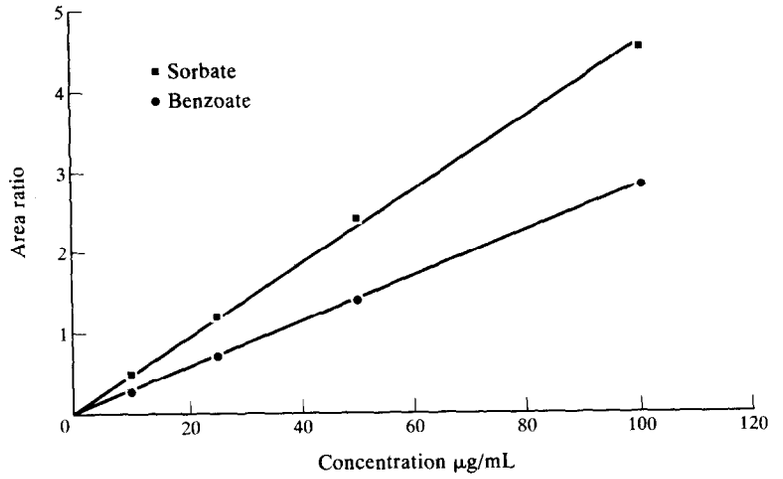

Fig. 3. Linearity plots for sorbate and benzoate in alkaline solution with dehydroacetate as the internal standard. Data bars indicate the coefficient of variation for seven replicate determinations.

All standards were basified and run again for linearity and repeatability data. The values obtained for the statistical data and the comparison with the HPLC data are presented in Table 2. The %CV was slightly better with basified standards. The values obtained by MECC were slightly higher than those obtained by HPLC but were found to be satisfactory. The detector response was linear up to 100 μg/ml for sorbic acid and benzoic acid in the basified solutions (Fig. 3).

A variety of foods and beverages of different brands were selected and prepared as outlined in the experimental section. These were analysed by MECC and HPLC. The content of sorbic and/or benzoic acid was found to be comparable by both procedures in most of the samples analysed (Table 3). The electropherogram of a fruit drink containing both sorbic acid and benzoic acid is displayed in Fig. 4. The analysis time for MECC (7 min, including a column wash) was shorter than for HPLC (10 min). The frugal use of reagents and the low cost of the silica capillary column compared with the HPLC column, coupled with the shorter analysis time, make MECC a viable alternative to HPLC for this analysis.

An additional compound was observed in both the electropherograms and the chromatograms for the diet/low-joule samples. The labels on most of these samples indicated that they contained saccharin or cyclamate as artificial sweetening agents. To identify the extra peak, sodium cyclamate and sodium saccharin were run by both procedures. Based on the migration time it was concluded that the peak which appeared immediately after the benzoic acid peak in MECC was saccharin. The electropherogram for a diet cordial containing benzoic acid and saccharin is shown in Fig. 5. Saccharin eluted much later in the HPLC run. Cyclamate was not seen as it does not absorb at the detector wavelength used in these analyses (230 nm).

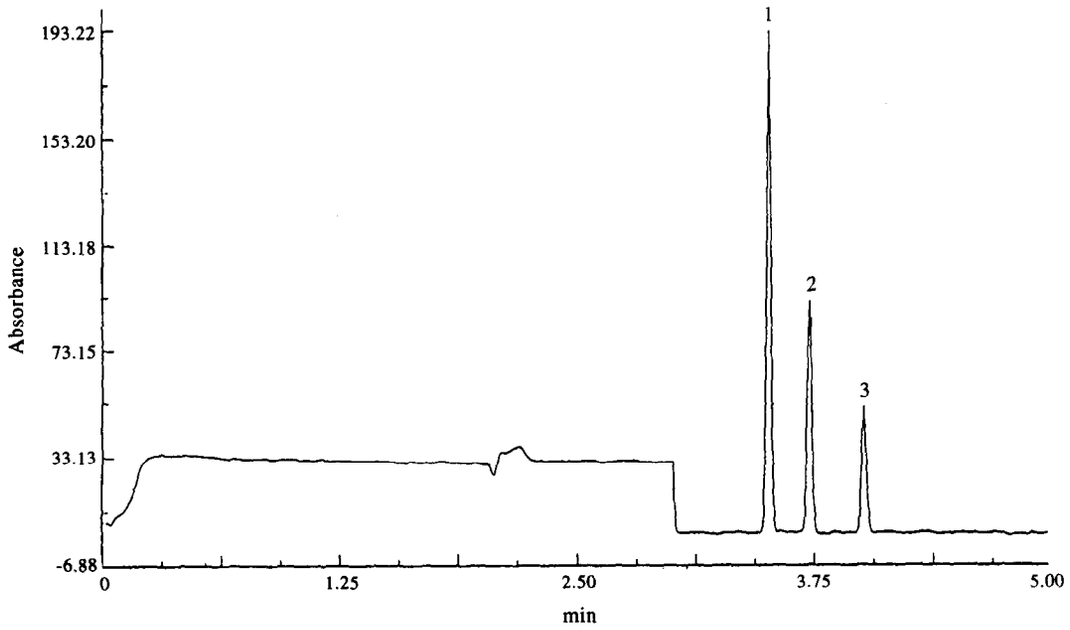

Fig. 4. Separation of (1) dehydroacetate (internal standard), (2) sorbate and (3) benzoate in a fruit drink using a buffer consisting of 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The *x*-axis gives the migration times in minutes.

Table 3. Comparison of the amounts of sorbic acid and benzoic acid present in a variety of beverages and foods determined by MECC and HPLC[a]

Sample (mg/litre)	Sorbic acid		Benzoic acid		%CV	
	MECC	HPLC	MECC	HPLC	MECC	HPLC
Orange juice						
Brand 1	370	380	—	—	1·40	0·12
Brand 2	150	150	—	—		
Brand 3	320	340	—	—		
Brand 4	360	420	—	—		
Apple juice						
Brand 1	350	370	—	—	1·74	0·61
Brand 2	170	180	160	190		
Brand 3	210	200	230	240		
Brand 4	280	270	—	—		
Cordials						
Brand 1	—	—	390	350	0·73	0·40
Brand 2	—	—	440	400		
Brand 3	—	—	350	350		
Brand 4	—	—	390	400		
Diet cordial						
Brand 1	—	—	440	450	0·92	1·47
Brand 2	70	60	—	—		
Brand 3	—	—	100	90		
Brand 4	—	—	370	340		
Fruit drink (25% juice)						
Brand 1	270	280	170	160	1·25*	0·44*
Brand 2	430	410	—	—		
Brand 3	240	240	120	120		
Brand 4	250	250	120	120		
(* %CV for sorbic acid)						
Soft drinks						
Brand 1	—	—	220	260	1·28	1·29
Brand 2	—	—	170	170		
Brand 3	—	—	130	140		
Brand 4	—	—	150	140		
Low-alcohol wine						
Brand 1	170	220	—	—	1·06	0·50
Brand 2	—	—	240	280		
Cheese slices (mg/kg)						
Brand 1	960	830	—	—	1·8	1·52
Brand 2	1680	1830	—	—		
Brand 3	790	810	—	—		
Brand 4	1140	1240	—	—		
Dips (mg/kg)						
Brand 1	1270	1250	—	—	1·05	1·91
Brand 2	460	450	—	—		
Brand 3	810	800	—	—		
Low-joule jam (mg/kg)						
Brand 1	960	930	—	—	1·68	1·17
Brand 2	—	—	400	400		

[a]Repeatability data, expressed as %CV, from seven replicate determinations of one sample of each type of product is also shown.

(b) Use of the MECC procedure as a screening tool for acid preservatives

To detect lower concentrations for screening purposes, the AUFS setting on the detector was changed to 0·005. Standard solutions of 1 and 5 μg/ml with phthalate as the internal standard at 5 μg/ml were basified and analysed for statistical data and compared with the data obtained by HPLC. The results are presented in Table 4.

Table 4. Repeatability data (%CV) for standard solutions of dehydroacetic acid, sorbic acid and benzoic acid analysed by MECC and HPLC. Phthalate was used as the internal standard for MECC[a]

Standard (μg/ml)	%CV					
	Dehydroacetic acid		Sorbic acid		Benzoic acid	
	MECC	HPLC	MECC	HPLC	MECC	HPLC
1	3·7	—	6·5	1·3	7·8	1·5
5	3·8	—	4·4	0·6	6·0	1·2

[a]Results for seven replicate determinations.

The %CV data for MECC was higher than for HPLC but nevertheless acceptable.

One sample each of white wine and grape juice concentrate were basified with 1M NaOH and analysed. The samples were reported not to contain either dehydroacetic, sorbic or benzoic acid; none of the compounds were detected by the above MECC procedure. To confirm these results, the samples were spiked with a solution containing the three acids at 10 mg/litre, basified to pH > 9 and re-analysed. The electropherograms for the white wine and the spiked white wine are displayed in Fig. 6. The basified spiked samples were then run seven times each for repeatability data. The mean recoveries and %CV data are presented in Table 5. The mean recoveries for each of the preservatives were acceptable, as were the %CV data, even though the %CV values were much higher for dehydroacetic acid and sorbic acid in the white wine than the grape juice concentrate. There was no obvious explanation for these results.

The above procedure was then applied to a sample of red wine suspected to contain benzoic acid. When the wine was basified and analysed, benzoic acid was not detected. However, the electropherogram showed a great degree of baseline drift and there was also a naturally occurring compound that had the same migration time as phthalate. This precluded the use of potassium hydrogen phthalate as the internal standard. There were no peaks corresponding to dehydroacetic acid or sorbic acid in the wine, and so these two compounds

Table 5. Recovery and repeatability data (%CV) for samples of white wine and grape juice concentrate containing dehydroacetic acid, sorbic acid and benzoic acid added at a level of 10 mg/litre. Phthalate was used as the internal standard[a]

Sample	Dehydroacetic acid		Sorbic acid		Benzoic acid	
	% Rec	%CV	%Rec	%CV	%Rec	%CV
White wine	104	16	102	14	105	7
Grape juice concentrate	112	4	87	6	106	6

[a]Results for seven replicate determinations.

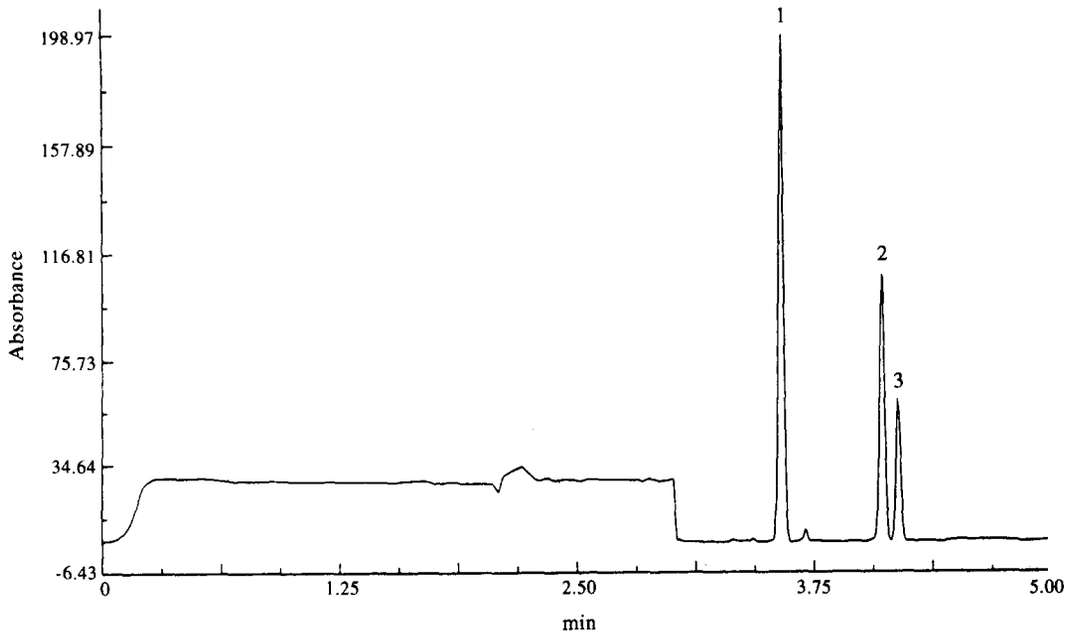

Fig. 5. Separation of (1) dehydroacetate (internal standard), (2) benzoate, and (3) saccharin in a diet cordial using a buffer consisting of 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The *x*-axis gives the migration times in minutes.

were added as internal standards. The wine sample was spiked with benzoic acid at 10, 20 and 50 mg/litre, respectively, while dehydroacetic acid and sorbic acid were each added at 5 μg/ml. The samples were basified and analysed seven times for repeatability data. Results are presented in Table 6.

The recoveries were more consistent when sorbic acid was used as the internal standard rather than dehydroacetic acid, suggesting that sorbic acid be the preferred internal standard for routine work. There was no obvious explanation for the recoveries being more consistent when sorbic acid was used as the internal standard. The level of reporting was 10 mg/litre and it

Table 6. Recovery and repeatability data (%CV) for benzoic acid added to red wine at a level of 10, 20 and 50 mg/litre using both dehydroacetic acid and sorbic acid as internal standards[a]

Level of spiking (mg/litre)	Internal standard			
	Dehydroacetic acid		Sorbic acid	
	%Rec	%CV	%Rec	%CV
10	108	11·6	88	13·6
20	89	15·4	98	2·7
50	73	2·5	95	8·6

[a]Results for seven replicate determinations.

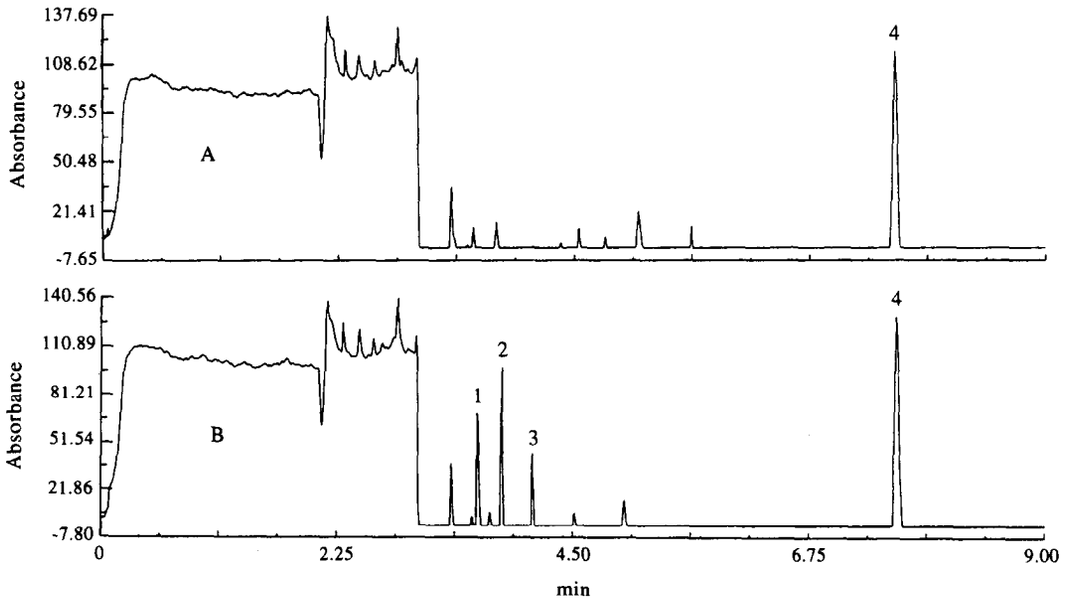

Fig. 6. Electropherograms of (A) wine, and (B) wine spiked at 10 mg/litre showing the separation of (1) dehydroacetate, (2) sorbate, (3) benzoate and (4) phthalate (internal standard) using a buffer consisting of 0·05 M sodium dodecylsulphate and 0·02 M disodium hydrogen phosphate, pH 9·2. The *x*-axis gives the migration times in minutes.

was concluded that the sample had no detectable level of benzoic acid.

A sample of the wine was subjected to the more time consuming, yet more sensitive, steam distillation/solvent extraction procedure as outlined in method (b). The level of reporting for both HPLC and MECC was 0·1 mg/litre. Benzoic acid was not detected by either MECC or HPLC. A sample spiked with benzoic acid at a level of 4 mg/litre was also analysed using this procedure and the recoveries of 84% for MECC and 93% for HPLC were recorded.

CONCLUSION

The MECC procedure outlined was found suitable for quantifying the content of sorbic acid and benzoic acid in a variety of food samples. The amounts of preservatives determined by MECC were in good agreement with those determined by the HPLC procedure currently used in the authors' laboratory. The MECC procedure has the same order of repeatability as the HPLC method and is also faster, more efficient and less costly to operate. This procedure can also be used for the screening of sorbic acid, benzoic acid and dehydroacetic acid in beverages using phthalate as the internal standard.

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