ELSEVIER

PII: S0308-8146(97)00022-8

Food Chemistry, Vol. 60, No. 4, pp. 667–674, 1997

0308-8146/97 $17.00+0.00
Analytical, Nutritional and Clinical Methods Section

The determination of niacin in cereals, meat and selected foods by capillary electrophoresis and high performance liquid chromatography

Caroline M. Ward[a] & V. Craige Trenerry[b]*

[a]*Australian Government Analytical Laboratories, 338–340 Tapleys Hill Road, Seaton, South Australia, 5023, Australia*
[b]*Australian Government Analytical Laboratories, 51–65 Clarke Street, South Melbourne, Victoria, 3205, Australia*

(Received 6 September 1996; revised version received 23 December 1996; accepted 23 December 1996)

A robust method for the determination of niacin in cereals, meat, fish, yeast, selected nuts, peanut butter, sunflower seeds and other fortified foods using capillary electrophoresis (CE) as the determinative step is described. Niacin is liberated from the food matrix by alkaline digestion with aqueous calcium hydroxide and the extract purified and concentrated using C18 and cation exchange columns. A 75 μm uncoated fused silica capillary column with an effective length of 50 cm and a buffer comprising 15% acetonitrile and 85% of a 1:1 mixture of 0.02 M potassium dihydrogen orthophosphate and 0.02 M disodium hydrogen orthophosphate pH 7 was used for all of the determinations. Saccharin was used as the internal standard. The levels of niacin determined by CE compared favourably with those determined by high performance liquid chromatography. The CE instrument repeatability data for area calculation (CV 1%, $n=7$) and migration time variation (CV 0.7%, $n=20$) for a cereal sample were acceptable. The limit of reporting for the determination is 0.5 mg 100 g^{-1}. © 1997 Elsevier Science Ltd

INTRODUCTION

Niacin activity is possessed by several related compounds found in food. The vitamin occurs naturally both in the free and bound forms (e.g. nicotinic acid and nicotinamide). It is generally considered that only the acid-hydrolysable forms are fully bioavailable for humans; thus, if the nutritive value is to be determined, an acid hydrolysis, is preferable whereas, if the total amount of niacin is to be determined, an alkaline procedure should be chosen. Tryptophan can be converted to niacin in the body, and therefore the total niacin activity of a food can be determined only if the contribution of tryptophan is taken into account (Greenfield & Southgate, 1992). Various methods have been used to determine natural levels of niacin in food. These include the traditional AOAC colorimetric procedure using cyanogen bromide and sulphanilic acid, or the microbiological assay using *Lactobacillus plantarum* (Ball, 1994). The AOAC colorimetric procedure lacks specificity (Vidal-Valverde & Reche, 1991) and is unreliable in certain circumstances (Kwiatkowska *et al.*, (1989)). Chromatographic and electrophoretic procedures add an extra level of confidence to the quality of analytical measurements, especially if a photodiode array detector is used in the determination (Heiger *et al.*, 1994; Vandepeer *et al.*, 1996). High performance liquid chromatographic (HPLC) methods include the determination of niacin in meat (Takatsuki *et al.*, 1987; Tyler & Genzale, 1990; Vidal-Valverde & Reche, 1991; Hamano *et al.*, 1988), cereals (Tyler & Shrago, 1980), coffee (Trugo *et al.*, 1985) as well as other fortified foods (Rees, 1989). The determination of added nicotinamide in meat products by gas chromatography (Tanaka *et al.*, 1989) and an enzymic procedure for the determination of free niacin and nicotinamide in meats has also been reported (Hamano *et al.*, 1995).

We recently reported the determination of total niacin (as nicotinic acid) in concentrated yeast spreads by capillary electrophoresis (CE) using a 75 cm × 75 μm uncoated fused silica capillary column with a buffer comprising a 1:1 mixture of 0.02 M sodium tetraborate and 0.02 M disodium hydrogen orthophosphate pH 9.2 (Ward *et al.*, 1996). The niacin (chemically bound or as added nicotinamide) was liberated from the food by alkaline digestion. A clean-up procedure using commercially available C18 and cation exchange (SCX) columns was used to provide an extract suitable for

*To whom correspondence should be addressed.

analysis by CE. The levels determined using this procedure compared favourably with those determined by the standard AOAC colorimetric procedure (Williams, 1984).

This paper describes the determination of niacin in both fortified and natural foods, e.g. breakfast cereals, grains and grain products, raw, cooked and processed meats, and other selected foods by CE. Two samples of pet food were also included in the study. The majority of the sample extracts were also analysed by HPLC using a procedure based on that described by Tyler and Shrago (1980).

MATERIALS AND METHODS

Reagents

Niacin and sodium saccharin were obtained from Sigma Chemical Co., St Louis, MO, USA. Disodium hydrogen orthophosphate was obtained from BDH Chemicals, Kilsyth, Australia. Oxalic acid and potassium dihydrogen orthophosphate were obtained from Ajax Chemicals, Auburn, Australia. PIC A Reagent and C18 Sep-Pak Vac cartridges (500 mg) were supplied by Waters Corporation, Milford, MA, USA. All other chemicals and solvents were AR grade or HPLC grade and were used without further purification. SCX cation exchange columns (500 mg) were obtained from Varian, CA, USA. AACC Standard Reference Sample VMA 195 was obtained from the American Association of Cereal Chemists, St. Paul, MN, USA. The sample was divided into aliquots of approximately 1 g and stored at −18°C until required.

Preparation of standards, samples and buffer

Standards

A 100 μg ml^{-1} stock standard solution of niacin was prepared by dissolving 20 mg of dry niacin in 200 ml of 75% aqueous ethanol. The solution was stored in the refrigerator. Working standards of between 1 and 50 μg ml^{-1} were prepared by diluting the stock solution with deionised water. Saccharin was added as the internal standard at a final concentration of 40 μg ml^{-1} for CE analysis. No internal standard was used for the HPLC determinations. The detector response for niacin was linear to at least 50 μg ml^{-1} for CE and at least 100 μg ml^{-1} for HPLC.

Samples and sample preparation

The samples were purchased from local outlets and analysed within the recommended 'use by' dates where appropriate. The cereals were ground to a fine powder in a Tecator Cyclotec 1093 sample mill before a suitable aliquot was taken for analysis. The breads were analysed as received or dried in an air oven at 37°C prior to blending in a commercial food processor. Meat and fish samples (except for canned ham, metwurst and pet food) were divided into two approximately equal portions, one for analysis as the raw product and the other for analysis after cooking. The meat and fish samples were baked for approximately 40 min. The raw and cooked meats, nuts and sunflower seeds were homogenised in a commercial food processor. All samples were analysed as soon as possible after preparation.

To approximately 1 g of the food was added 0.75 g of calcium hydroxide and 20 ml of deionised water. For recovery tests, 1 ml of a 100 μg ml^{-1} niacin solution was added.

The mixture was thoroughly mixed and heated in an autoclave for 2 h at 121°C (~104 kPa). The cooled mixture was diluted to 50 ml with deionised water, mixed thoroughly and centrifuged at 2500 rpm for 15 min at 0°C. A 15-ml aliquot of the supernatant was adjusted to pH 7 with aqueous oxalic acid (10% followed by final adjustment with 1%) and made to 25 ml with deionised water. The resultant suspension was centrifuged at 2500 rpm for 10 min at 0°C. A C18 Sep-Pak Vac Cartridge (500 mg) and an SCX column (500 mg) were connected in series and conditioned with 10 ml methanol followed by 10 ml deionised water. A 10-ml aliquot of the supernatant was then loaded onto the C18 column. The columns were washed with 5 ml of water, the C18 Sep-Pak cartridge discarded, and the SCX column washed with 5 ml of methanol. Niacin was removed from the SCX column with 5 ml of freshly prepared 2% solution of concentrated ammonium hydroxide in methanol. The solvent was evaporated to dryness under a stream of nitrogen at room temperature. The residue was dissolved in 1 ml of a 40 μg ml^{-1} aqueous saccharin solution for CE analysis or 1 ml of deionised water for HPLC analysis. The solutions were filtered through 0.8 μm cellulose acetate filter discs before analysis.

Buffer

The CE buffer was prepared by mixing 3.75 ml of acetonitrile and 21.25 ml of a 1:1 mixture of 0.02 M potassium dihydrogen orthophosphate and 0.02 M disodium hydrogen orthophosphate. The buffer was filtered through a 0.45-μm teflon filter disc before use.

Apparatus

CE

The extracts were analysed using a 75 cm × 75 μm uncoated fused silica capillary column with an effective length of 50 cm to the detector (Polymicro Technologies, Arizona, USA), fitted to an Isco Model 3140 Electropherograph (Isco Inc., Lincoln, NE, USA). The separations were performed at +20 kV and at 30°C using a buffer consisting of 15% acetonitrile and 85%

of a 1:1 mixture of 0.02 M potassium dihydrogen orthophosphate and 0.02 M disodium hydrogen orthophosphate, pH 7. The compounds were loaded under vacuum (vac. level 2, 25 kPa sec^{-1}) and were detected at 254 nm at 0.01 AUFS. The capillary was flushed with running buffer for 2 min between runs. Electropherograms were recorded with the ICE Data Management and Control software supplied with the Model 3140 Electropherograph. Peak areas were used in the calculations.

A Hewlett-Packard 3D Capillary Electropherograph fitted with a 75-μm column with an effective length of 66 cm to the detector, and operating under the same conditions as described above, was used to obtain the UV spectra and peak purity data.

HPLC

The analyses were performed with a model 600E HPLC pump, model 712 WISP and a model 490 programmable multiwavelength UV detector using a 4-μm C8 NOVA-PAK Radial-PAK cartridge (8 mm×100 mm) equipped with a C18 pre-column (Waters Corporation, Milford, MA, USA) using a mobile phase consisting of a 15% methanol, 85% deionised water mixture containing 0.005M PIC A Reagent. The eluent flow rate was 1.5 ml min^{-1}. Niacin was detected at 254 nm and the detector output was set at 0.02 AUFS. Peak areas obtained from an HP 3350 laboratory data system (Hewlett-Packard, Palo Alto, CA, USA) were used in the calculations.

RESULTS AND DISCUSSION

Comprehensive reviews by Lindeberg highlight the versatility of CE as an analytical tool for the analysis of foods and beverages (Lindeberg, 1996*a*,*b*). The methods are robust, faster and more cost-effective than methods based on HPLC. CE separations are characterised by their speed and excellent resolution. However, two of the major disadvantages of the technique are the lack of sensitivity compared to HPLC and the potential non-compatibility of the sample solutions with the buffers used for the determinations (Altria *et al.*, 1996). One approach used to overcome these problems has been to isolate the analytes of interest with solid phase extraction (SPE) cartridges and redissolve the purified extract in a solvent that is more compatable with the running buffer (Thompson & Trenerry, 1995).

CE and the related technique of micellar electrokinetic capillary chromatography (MECC) have been used to separate and quantify water soluble vitamins, in particular niacin, in vitamin preparations (Fujiwara *et al.*, 1988; Jegle, 1993) and biological samples (Zarzycki *et al.*, 1995). We recently reported a method for the determination of niacin in concentrated yeast spreads using both CE and MECC as the determinative step (Ward *et al.*, 1996). The niacin was liberated from the food using an alkaline digestion with saturated aqueous calcium hydroxide. Removal of the excess calcium by precipitation with oxalic acid followed by clean-up of the extract with C18 and cation exchange cartridges produced an extract suitable for analysis by CE. This procedure also allows for a concentration step to be incorporated into the method, thus permitting much lower levels of niacin to be determined.

The successful use of CE in determining niacin in concentrated yeast spreads prompted us to extend the work to include other foods which are considered to be a good source of niacin in the diet.

Our initial attempt to determine the niacin content of fortified breakfast cereals using the CE conditions described for the concentrated yeast spreads (Ward *et al.*, 1996) was encouraging. The determination of lower levels of niacin was achieved by incorporating a ten-fold concentration step into the clean-up procedure. Niacin was well separated from the majority of the peaks in the electropherogram. However, there were a number of small peaks which had similar migration times to saccharin, the internal standard used in the previous work. The use of internal standards for quantitative CE analyses is recommended as they allow for peculiarities in the injection procedures and for the gradual changes in migration times that are often seen due to buffer degradation (Weinberger, 1993). Attempts to find an alternative internal standard were unsuccessful, so a number of different buffers were examined in order to separate the interfering peaks from saccharin. Changing the buffer from a 1:1 mixture of 0.02 M sodium tetraborate, 0.02 M disodium hydrogen orthophosphate, pH 9.2 to a 1:1 mixture of 0.02 M potassium dihydrogen orthophosphate, 0.02 M disodium hydrogen orthophosphate, pH 7 did not alter the positions of the interfering peaks. Similarly, the addition of 0.05 M sodium dodecylsulphate had no affect. However, the addition of 15% acetonitrile to the phosphate buffer pH 7 gave baseline resolution of all of the peaks of interest.

Using this buffer, the separation of niacin and saccharin in a standard solution was maintained over twenty repetitive injections. To test the suitability of the CE conditions for quantitative purposes, a number of standard solutions of different concentrations of niacin containing the internal standard were analysed seven times. The statistical data for peak area calculation were acceptable in the concentration range 1–50 μg ml^{-1} (1 μg ml^{-1}, CV 4.5%; 2 μg ml^{-1}, CV 3.4%; 5 μg ml^{-1}, CV 2.2%; 10 μg ml^{-1}, CV 0.8%; 15 μg ml^{-1}, CV 1.9%; 25 μg ml^{-1}, CV 0.6%; 50 μg ml^{-1}, CV 1.5%).

A range of fortified cereals, cereal products (e.g. bread) and grains were then analysed by CE. Each different type of food was spiked with niacin to provide recovery data. None of the samples analysed had measurable amounts of naturally occurring compounds that comigrated with the internal standard. The levels of niacin determined by CE compared favourably with the

Table 1. Niacin levels determined by both CE and HPLC for a range of cereals and grains—indicates that the HPLC analyses were not done. Recoveries of niacin added to the samples prior to digesting with aqueous calcium hydroxide are shown

	CE	HPLC	CE Recovery (%)
Breakfast cereals (mg 100 g^{-1})			
Sample 1 (wheat-based)	16.2, 17.0	—	94, 98
Sample 2 (corn-based)	10.2	—	92
Sample 3	9.6	—	104
Sample 4	7.1	6.9	106
Sample 5	3.1	2.5	110
Sample 6	0.9	1.1	102
Sample 7	0.8	—	100
Sample 8	0.7	—	92
Bread (mg 100 g^{-1})			
Sample 1	6.0	5.9	107
Sample 2	5.4, 5.5	—	108
Sample 3 (undried)	3.0	3.4	98
Grains (mg 100 g^{-1})			
Barley	8.5	7.9	101
Rice	4.1	4.2	100
Wheat	3.9	3.7	99
Wheat germ	3.2	2.6	98
Soy bean	1.6	3.0	95
Oats	1.0	1.1	99

levels determined by HPLC (Table 1) and were similar to the manufacturers' labelling claims and to the levels reported by English and Lewis (1992). No direct comparison of the our results with those in the literature could be made as different samples were used to produce the analytical results. The average recovery of niacin added prior to digestion was 100% (Table 1). When possible, the integrity of the peaks corresponding to niacin was confirmed by the UV spectra and peak purity data recorded with the photodiode array detector of the Hewlett-Packard CE. (UV spectra could not be obtained for some samples containing low levels of niacin.) A level of reporting for niacin in cereals of 0.5 mg 100 g^{-1} could be achieved using these conditions. The electropherogram of a cereal is displayed in Fig. 1. Baseline resolution of the niacin peak from other peaks in the electropherograms was evident in all of the samples. The migration time of niacin by CE (8.5 and 12.5 min, respectively, for the Isco and Hewlett-Packard instruments) was similar to the retention time by HPLC (10 min). However, the overall CE run times were much shorter due to the late eluting peaks with HPLC. The CE traces were also much cleaner. Partial chromatograms of a bread sample and a cereal sample are shown in Figs 2a and b.

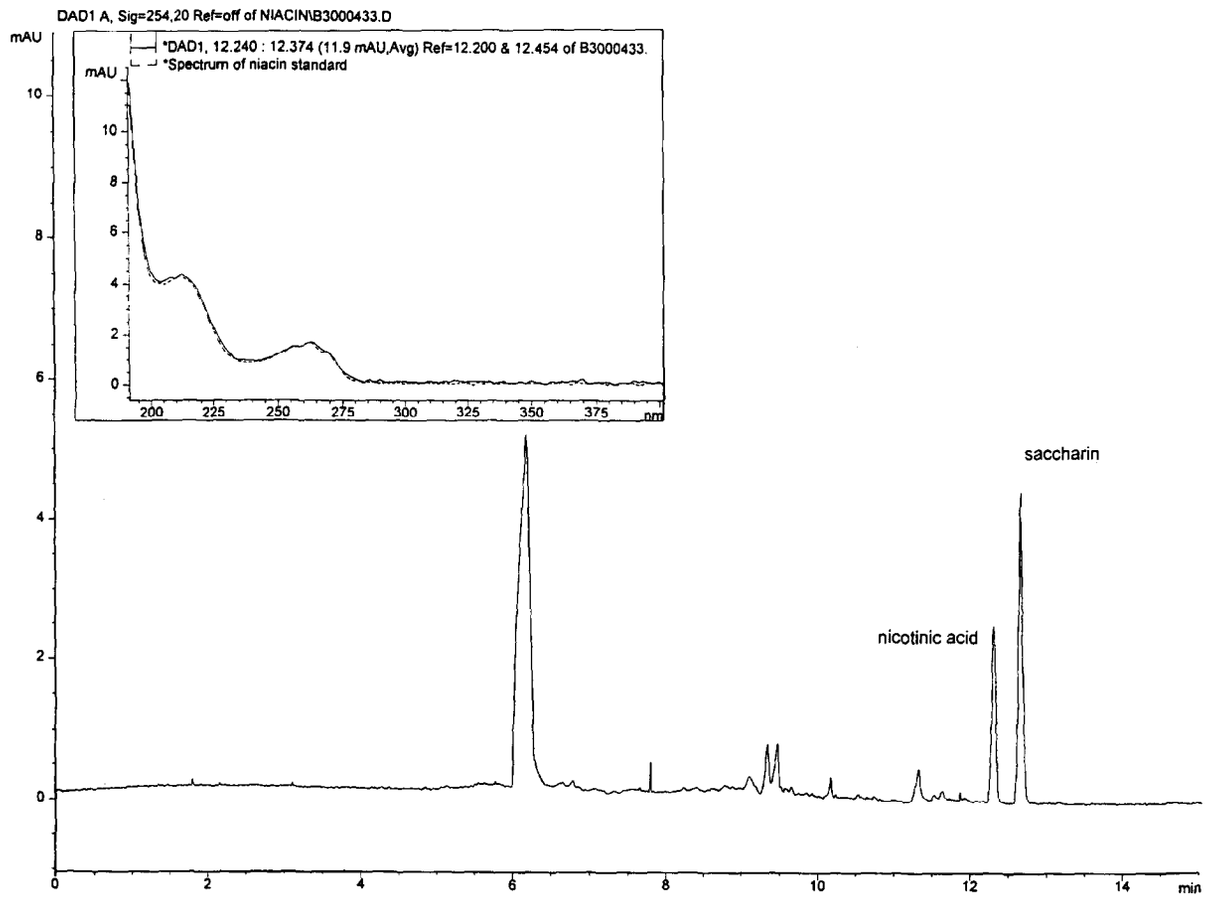

Fig. 1. Electropherogram showing the separation of niacin and saccharin (internal standard) for a cereal sample containing 6.4 mg 100 g^{-1} niacin using the conditions described in the Experimental section, and the UV spectrum of the peak corresponding to niacin compared with the UV spectrum of pure niacin.

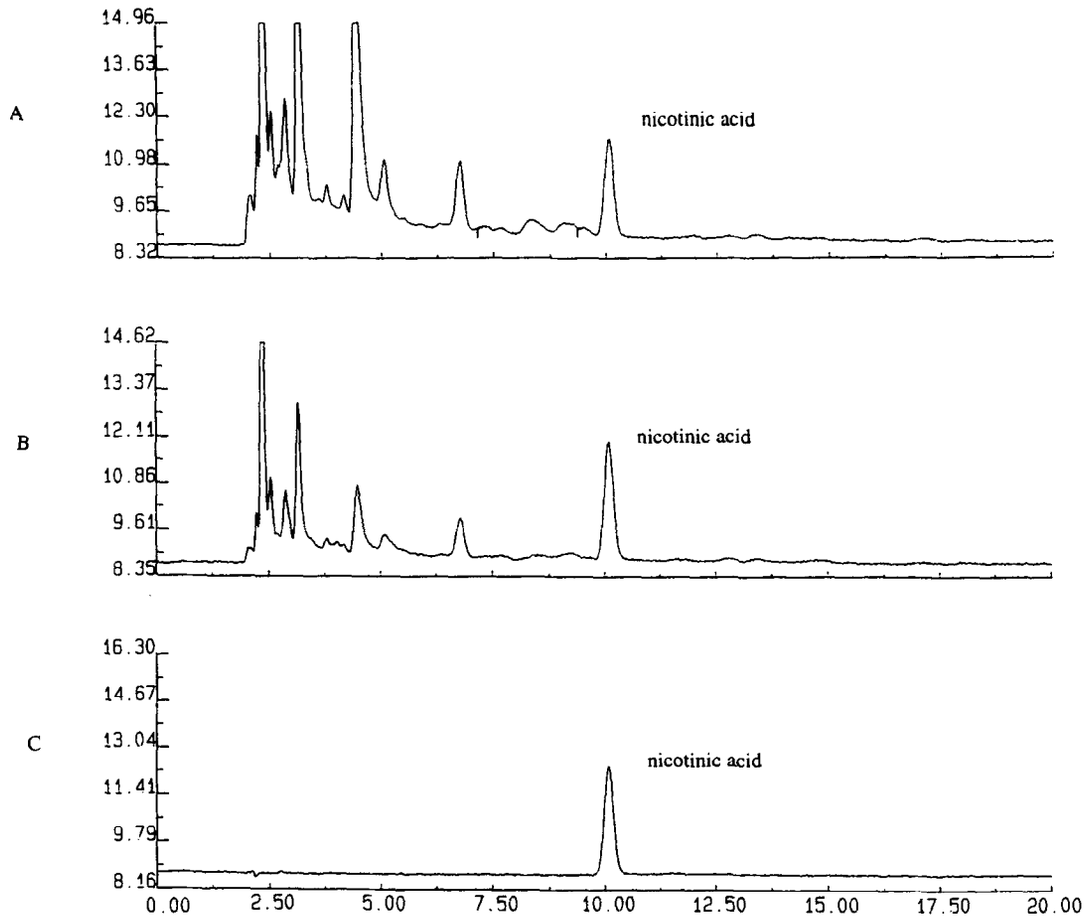

Fig. 2. Partial chromatograms of A) bread sample, B) cereal sample and C) standard solution using the conditions described in the Experimental section.

Seven portions of the VMA 195 reference sample were analysed in one batch to check the accuracy/repeatability of the method. The average level of niacin (18.6 mg 100 g^{-1}) compared well with the data supplied with the sample (mean result of 18.0 mg 100 g^{-1}, range 13.9–20.1 mg 100 g^{-1}, $n=13$). The instrument repeatability data for area calculation for seven repeat injections of the cereal extract (CV of 1%) compared favourably with the HPLC data (CV 0.5%). Also, CE instrument repeatability data for the variation in migration time of niacin was good (CV 0.7%, $n=20$). The CV improved to 0.1% when the ratio of the migration time of niacin to the migration time of the internal standard was used in the calculation. An overall CV of 2.6% for the procedure indicated that the proposed method was robust.

The method was also suitable for determining the levels of niacin in a variety of other foods considered to be a good source of niacin (e.g. meat, fish, chicken, nuts, yeast and sunflower seeds). Two samples of pet food were also analysed. Niacin and saccharin were well separated in all of the electropherograms and the levels determined by CE were similar to the levels determined by HPLC (Table 2). As was noted for the cereals, no samples had any naturally occurring compounds that comigrated with saccharin. Small differences in the CE and HPLC values may be attributable to the two analyses being carried out on different extracts. The food digest was subjected to two SPE extractions to obtain solutions for the comparative analyses. One extract was dissolved in deionised water containing the internal standard (for CE) and the other in deionised water without an internal standard (for HPLC). The CE solution could not be used for HPLC analysis as saccharin had a much longer retention time than niacin and interfered with the following chromatogram. Recoveries of niacin added prior to digestion were also excellent (Table 2). The levels of niacin in the foods were also similar to the values reported by English and Lewis (1992) and by Greenfield (1987). As with the cereals, no direct comparison of the results can be made as different samples were used to produce the analytical results. The UV spectra and the peak purity data of the peaks corresponding to niacin in the electropherograms confirmed the integrity of the peaks. Electropherograms of canned ham and yeast are displayed in Figs 3 and 4. Partial chromatograms of a sample of canned ham and a yeast sample are shown in Figs 5a and b.

Seven samples of canned ham were also analysed in one batch to check the accuracy/repeatability of the

Table 2. Niacin levels determined by both CE and HPLC for a range of meats and other foods—indicates that the HPLC analyses were not done. Recoveries of niacin added to the samples prior to digesting with aqueous calcium hydroxide are shown

		CE	HPLC	CE Recovery(%)
Meats (mg 100 g^{-1})				
Beef	raw	3.6	3.2	101
	cooked	7.5	7.4	95
Lamb	raw	4.8	4.6	98
	cooked	7.5	7.2	95
Kangaroo	raw	6.4	5.3	98
	cooked	8.7	8.7	100
Pork	raw	6.2	5.1	103
	cooked	9.9	9.6	82
Chicken	raw	10.2	9.7	101
	cooked	14.0	14.6	99
Mullet	raw	6.0	6.3	98
	cooked	7.5	7.3	115
Snook	raw	1.2	1.1	90
	cooked	1.8	1.9	110
Metwurst		4.0	4.1	94
Canned ham		2.7	2.7	109
Other food (mg 100 g^{-1})				
Beef and tortellini		13.5	12.1	106
Beef and sweet/sour sauce		14.2	13.3	106
Baked beans		12.5	10.9	110
Peanut butter		14.9	13.1	116
Almonds		3.4	2.9	103
Sunflower seeds		11.5	10.5	123
Cashew nuts		1.3	1.2	105
Yeast		19.2	18.5	112
Petfood (mg 100 g^{-1})				
Sample 1 (meat-based pet food)		2.3	2.3	92
Sample 2 (dried pet food)		4.1	—	104

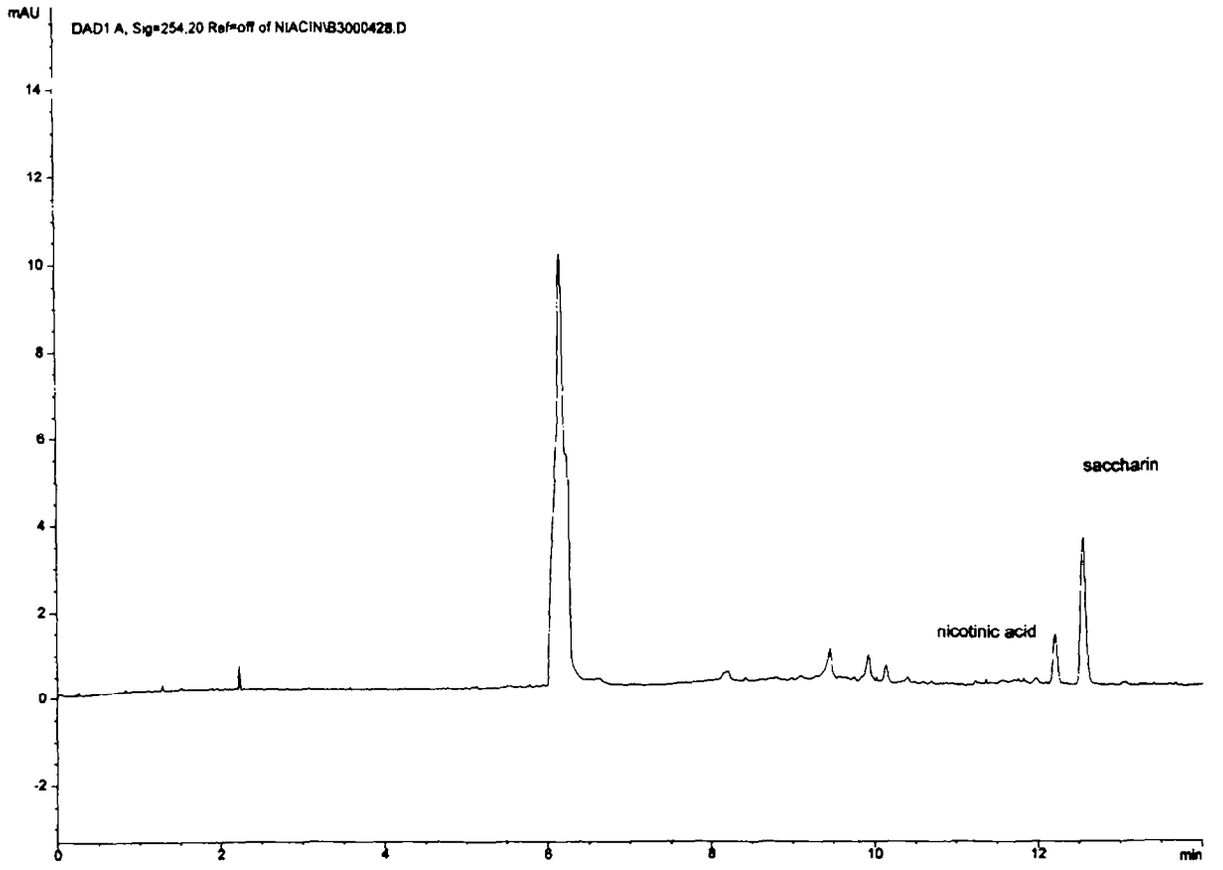

Fig. 3. Electropherogram showing the separation of niacin and saccharin (internal standard) for a sample of canned ham using the conditions described in the Experimental section.

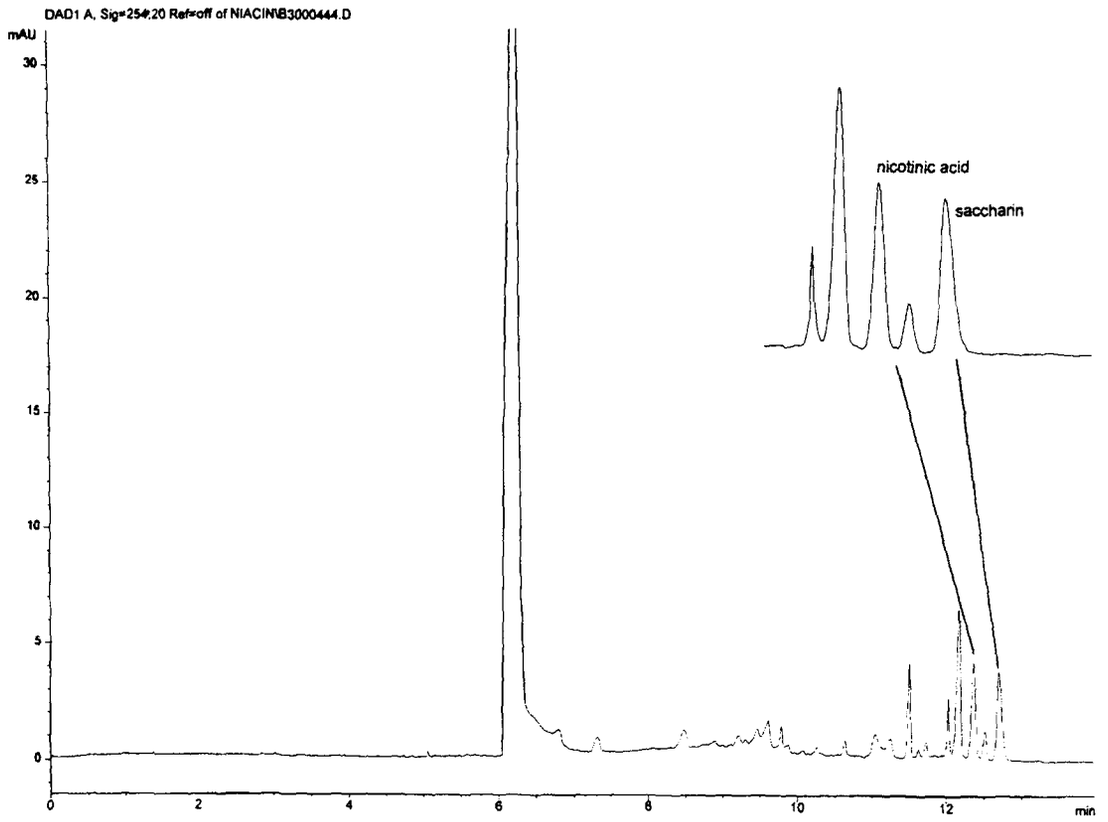

Fig. 4. Electropherogram showing the separation of niacin and saccharin (internal standard) for a yeast sample using the conditions described in the Experimental section.

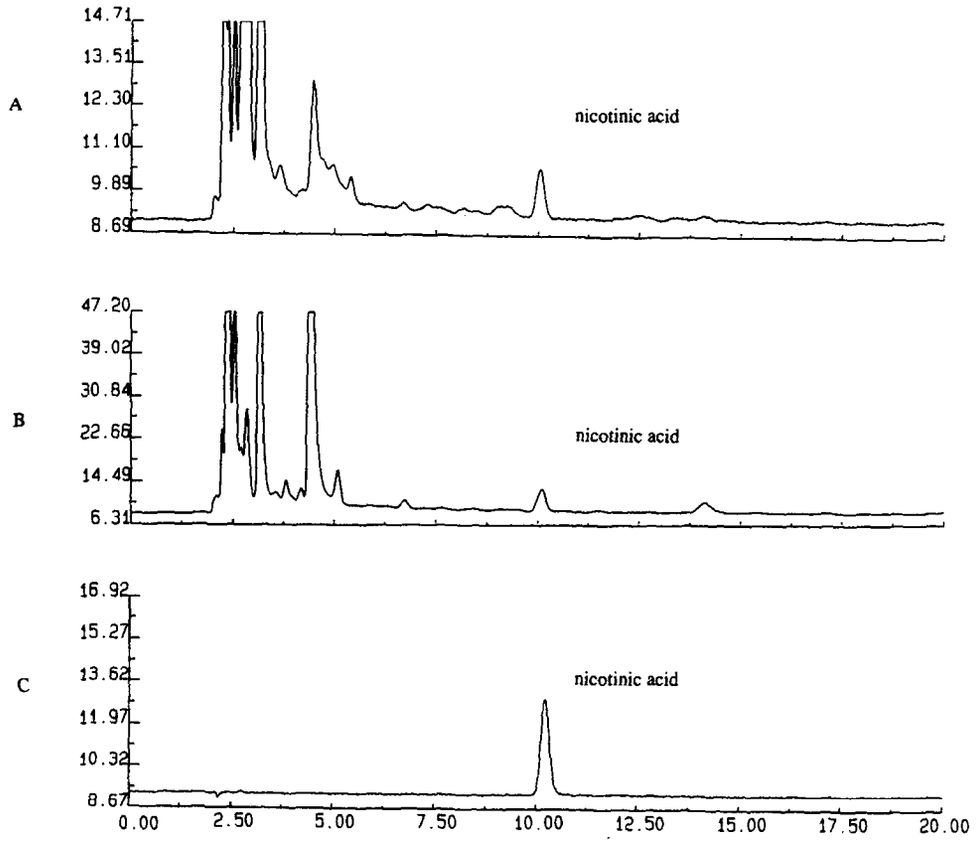

Fig. 5. Partial chromatograms of A) canned ham sample, B) yeast sample and C) standard solution using the conditions described in the Experimental section.

method for meat samples. The average of the seven results was 2.7 mg 100 g^{-1}. The CE instrument repeatability data for area calculation matched the HPLC data (CV 0.7%, $n=7$). Also, CE instrument repeatability data for the variation in migration time of niacin was good (CV 0.3%, $n=20$). The CV improved to 0.2% when the ratio of the migration time of niacin to the migration time of the internal standard was used in the calculation. An overall CV for the procedure of 0.9% indicated that the method was robust.

CONCLUSION

A robust method for the determination of niacin in cereals, meat and other foods using solid phase extraction to isolate and concentrate the niacin and CE as the determinative step has been developed. The sample extracts can also be analysed by HPLC; however, the CE produces cleaner traces, is faster and more cost-effective.

ACKNOWLEDGEMENTS

The authors wish to thank Ms Julie Pleydell for her technical assistance and the Australian Government Analyst, Dr J. Smith, for his permission to publish.

REFERENCES

Altria, K., Kelly, T. & Clark, B. (1996). CE methods and buffer preparation. *LC–GC*, **14**, 398–404.

Ball, G. F. M. (1994). *Water-soluble Vitamin Assays in Human nutrition*. Chapman & Hall, London, UK.

English, R. & Lewis, J. (1992). *Nutritional Values of Australian Foods*. Australian Government Publishing Service, Canberra, Australia.

Fujiwara, S., Iwase, S. & Honda, S. (1988). Analysis of water-soluble vitamins by micellar electrokinetic capillary chromatography. *J. Chromatogr.*, **447**, 133–140.

Greenfield, H. (Ed.) (1987). The nutrient composition of Australian meats and poultry. *Food Technol. Aust.*, **39**, 181–240.

Greenfield, H. & Southgate, D. A. T. (1992). *Food Composition Data: Production, Management and Use*. Elsevier Applied Science, London, UK.

Hamano, T., Mitsuhashi, Y., Aoki, N., Yamamoto, S. & Oji, Y. (1988). Simultaneous determination of niacin and niacinamide in meats by high performance liquid chromatography. *J. Chromatogr.*, **457**, 403–408.

Hamano, T, Mitsuhashi, Y, Kojima, N, Aoki, N, Semma, M, Ito, Y & Oji, Y. (1995). Enzymatic method for the amperometric determination of niacin in meat products. *Analyst*, **120**, 135–138.

Heiger, D. N., Kaltenbach, P. & Seivert, P. H-J. (1994). Diode array detection in capillary electrophoresis. *Electrophoresis*, **15**, 1234–1247.

Jegle, U. (1993). Separation of water soluble vitamins via high performance capillary electrophoresis. *J. Chromatogr.*, **652**, 495–501.

Kwiatkowska, C. A., Finglas, P. M. & Faulks, R. M. (1989). The vitamin content of retail vegetables in the UK. *J. Human Nut. and Diet.*, **2**, 159–172.

Lindeberg, J. (1996*a*). Capillary electrophoresis in food analysis. *Food Chem.*, **55**, 73–94.

Lindeberg, J. (1996*b*). Addendum to Capillary electrophoresis in food analysis. *Food Chem.*, **55**, 95–101.

Rees, D. I. (1989). Determination of nicotinamide and pyridoxine in fortified food products by HPLC. *J. Micronutr. Anal.*, **5**, 53–61.

Takatsuki, K., Suzuki, S., Sato, M., Sakai, K. & Ushizawa, I. (1987). Liquid chromatographic determination of free and added niacin and niacinamide in beef and pork. *J. AOAC.*, **70**, 698–702.

Tanaka, A., Iijima, M., Kikuchi, Y., Hoshino, Y. & Nose, N. (1989). Gas chromatographic determination of nicotinamide in meats and meat products as 3-cyanopyridine. *J. Chromatogr.*, **466**, 307–317.

Thompson, C. O. & Trenerry, V. C. (1995). Determination of synthetic colours in confectionery and cordials by micellar electrokinetic capillary chromatography. *J. Chromatogr.*, **704**, 195–201.

Trugo, L. C., Macrae, R. & Trugo, N. M. F. (1985). Determination of niacin in instant coffee using high performance liquid chromatography. *J. Micronutr. Anal.*, **1**, 55–63.

Tyler, T. A. & Genzale, J. A. (1990). Liquid chromatographic determination of total niacin in beef, semolina and cottage cheese. *Journal of AOAC.*, **73**, 467–469.

Tyler, T. A. & Shrago, R. R. (1980). Determination of niacin in cereal samples by HPLC. *J. Liq. Chromatogr.*, **3**, 269–277.

Vandepeer, J., Trenerry, V. C. & Keogh, G. (1996). Some examples of the analysis of complex samples by micellar electrokinetic capillary chromatography with photodiode array detection. *Electrophoresis*, **17**, 1361–1366.

Vidal-Valverde, C. & Reche, A. (1991). Determination of available niacin in legumes and meat by high performance liquid chromatography. *J. Agric. Food Chem.*, **39**, 116–121.

Ward, C., Trenerry, V. C. & Pant, I. (1997). The application of capillary electrophoresis to the determination of total niacin in concentrated yeast spreads. *Food Chem.*, **58**, 185–192.

Weinberger, R. (1993). *Practical Capillary Electrophoresis*. Academic Press Inc., San Diego, CA, USA.

Williams, S. (1984). *Official Methods of the AOAC (14th edn)*. Association of Official Analytical Chemists, Inc., Arlington, VA, USA. Method 43.051., 842–843.

Zarzycki, P. K., Kowalski, J., Nowakowska, J. & Lamparczyk, H. (1995). High performance liquid chromatographic and capillary electrophoretic determination of free niacin in human plasma and separation of its metabolites by capillary electrophoresis. *J. Chromatogr.*, **709**, 203–208.