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Analytical, Nutritional and Clinical Methods Section

Addendum to "Capillary electrophoresis in food analysis"

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The number of published applications of CE to food analysis is rapidly increasing. A great number of relevant reports will have appeared in the interval between the submission of the preceding review (Lindeberg, 1995) and its eventual publication. This Addendum presents recent articles and applications that were not available to the author at the time of submission, thus updating the review to include reports published in the early part of 1995. Additional applications of CE to quantitative food analysis are listed in Table 1.

CE was presented as a new tool in food analysis in recent reviews including short sections on principles and equipment and discussions on the advantages and disadvantages of the technique (Cancalon, 1995*a*; Cancalon, 1995*b*). Approaches to optimise precision in order to improve quantitative CE were suggested in a thorough study (Altria & Fabre, 1995).

Anions were determined in vegetables, and a thoroughly validated method for analysing nitrate and nitrite was presented (Jimidar *et al.*, 1995). Indirect detection in the visible range was used to achieve very low detection limits for inorganic cations and anions in drinking water (Malá *et al.*, 1994). Inorganic cations were analysed in Chinese tea infusions (sodium, potassium, calcium, magnesium and manganese; Yang *et al.*, 1994) and in a standard reference material (milk powder; sodium, potassium, calcium, magnesium; Pretswell *et al.*, 1995). Cations (ammonium, potassium, calcium and sodium) were separated in a cola drink by CE with indirect fluorescence detection (Bächmann *et al.*, 1992). The possibility of using CE for the analysis of low-molecular-weight ions in wine (mineral acids, organic acids and inorganic cations) was demonstrated (Dedieu *et al.*, 1994). Selenium and arsenic compounds were determined in spiked tap water and mineral water (Li & Li, 1995).

MEKC was used to determine total ascorbic acid in fruits and vegetables (Thompson & Trenerry, 1995*a*), and in beers, wines and fruit drinks (Marshall *et al.*, 1995). Sorbate and benzoate were determined in beverages and foods (Pant & Trenerry, 1995). Cyclamate and sorbate were determined and other artificial sweeteners were separated (Thompson *et al.*, 1995*a*). Artificial sweeteners, sorbate, benzoate and caffeine were quantified in low-Joule soft drinks and other foods (Thompson *et al.*, 1995*b*). Aspartame was determined in diet soft drinks (Ye & Baldwin, 1994*a*). Sucrose and fructose were detected and glucose and glucitol were quantified in apple juice (Ye & Baldwin, 1994*b*). Ethanol was determined in whisky (Altria & Howells, 1995). Confectionery and cordials were analysed for the presence of synthetic colours (Thompson & Trenerry, 1995*b*).

Sulfonylurea herbicides were determined in grains at higher resolution and better sensitivity than with existing HPLC methods (Krynitsky & Swineford, 1995). Formaldehyde and acetaldehyde were quantified (Takeda *et al.*, 1994) and terbuthylazine was detected (Dinelli *et al.*, 1995) in spiked tap water samples. Plant growth regulators (gibberellic acid, α-naphtaleneacetic acid, 2,4-dichlorophenoxyacetic acid and *N*-(2-chloro-6-fluorobenzyl)-*N*-ethyl-α,α,α-trifluoro-2,6-dinitro-*p*-toluidine) were analysed in citrus crops (Lancas & Galhiane, 1995). Chloramphenicol was determined in milk with an immunofluorescence CE technique (Blais *et al.*, 1994).

Flavonoids were identified in food pools typical of the mediterranean diet (soup, legumes/vegetables, salad and fruit) (Pietta *et al.*, 1995). CE was used together with SFC and MS to identify 2-thiothiazolidine-4-carboxylic acid in crude cabbage and in urine after ingestion of crude cabbage (Simon *et al.*, 1994). Grass pea (*Lathyrus sativus*) was analysed for the neurotoxic amino acid 3-(*N*-oxalyl)-L-2,3-diaminopropanoic acid and its α-isomer (Arentoft & Greirson, 1995). A number of phenolic compounds were analysed in red wines (Cartoni *et al.*, 1995; Gil *et al.*, 1995). FSCE was used to distinguish Class III caramel colour from Classes I and IV (Coffey & Castle, 1994). CE was used with indirect UV detection to analyse fatty acids in butter (Erim *et al.*, 1995) and coco oil (Roldan-Assad & Gareil, 1995). Seleno amino acids were identified and quantified in human milk (Michalke, 1995). FSCE was used to demonstrate changes in peptide patterns due to proteolysis during dry-curing of ham (Rodríguez-Nuñez *et al.*, 1995).

Advantages of using CE as a method for wheat varietal identification were discussed (Bietz & Schmalzried, 1995). Closely related wheat cultivars were differentiated by FSCE analysis of gliadin extracts (Lookhart & Bean, 1995*a*). In a similar way, oat and rice cultivars were differentiated by analysis of endosperm storage proteins (Lookhart & Bean, 1995*b*). Capillary electrophoresis in

Table 1. CE applied to the quantitative analysis of food samples - additional applications

Analyte	Sample matrix	Typical concentration	CE mode	Validation^{a-c}	Internal standard	References
Inorganic compounds						
Sodium, potassium, calcium, magnesium	Milk powder	1–20 mg/g	FSCE, indirect UV (creatinine, 220 nm), creatinine/HIBA (pH 3.5)	Linearity; detection limit (0.1–0.5 μg/g); comparison with AAS; standard reference material		Pretswell *et al.* (1995)
Nitrate, nitrite	Vegetables	0.03–4 mg/g (nitrate)	FSCE, indirect UV (chromate, 254 nm), CTAB and acetonitrile in chromate (pH 11.5)	Linearity; detection limit (0.3 μg/ml); quantification limit (1 μg/ml); RSD_r (3–5%); RSD_R (4–7%); Accuracy; comparison with spectrophotometry		Jimidar *et al.* (1995)
Nitrate, nitrite	Vegetables		FSCE, indirect UV (chromate, 254 nm), CTAB and acetonitrile in chromate (pH 11.5)	Recovery (94–99%); linearity; detection limit (0.04 μg/ml); quantification limit (0.1 μg/ml); RSD_r (2–8%); RSD_R (5–14%); Accuracy	Thiosulfate	Jimidar *et al.* (1995)
Chloride, bromide, sulfate, phosphate	Vegetables	0.1–2 mg/g, 0.001–0.8 mg/g (bromide), 0.6–10 mg/g (phosphate)	FSCE, indirect UV (chromate, 254 nm), CTAB and acetonitrile in chromate (pH 11.5)			Jimidar *et al.* (1995)
Arsenite, dimethylarsenite, selenite, arsenate	Spiked tap water, spiked mineral water	100 ng/ml	FSCE, UV (195 nm), borate/phosphate (pH 7.65)	Recovery (88–116%); detection limit (12–25 ng/ml, 800 ng/ml (arsenite)); RSD_r (4–16%)		Li & Li (1995)
Organic acids						
Formate, citrate, oxalate	Vegetables	0.2 mg/g (formate), 1–20 mg/g (citrate), 0.002–9 mg/g (oxalate)	FSCE, indirect UV (chromate, 254 nm), CTAB and acetonitrile in chromate (pH 11.5)			Jimidar *et al.* (1995)

Table 1. — *continued*

Analyte	Sample matrix	Typical concentration	CE mode	Validation^{a-c}	Internal standard	References
Total ascorbic acid	Beers, wines, fruit drinks	1–100 μg/ml, 0.2–1 mg/ml (fruit drinks)	MEKC, UV (254 nm), SDS in phosphate (pH 9.2), deoxycholate or CTAB in phosphate/borate (pH 8.6)	Linearity; RSD_r (0.5–4%); comparison with HPLC	Isoascorbic acid or nicotinic acid	Marshall *et al.* (1995)
Total ascorbic acid	Fruits, vegetables	0.02–2 mg/g	MEKC, UV (254 nm), sodium deoxycholate in borate/phosphate (pH 8.6)	Linearity; quantification limit (0.01 mg/g); RSD_r (0.5–3%); comparison with HPLC	Isoascorbic acid	Thompson & Trenerry (1995*a*)
Sorbate, benzoate	Fruit juices/drinks, jam, cheese slices, dips	0.07–2 mg/g	MEKC, UV (230 nm), SDS in phosphate (pH 9.2)	Recovery (73–108%); linearity; RSD_r (0.7–2%); quantification limit (10 μg/ml, 0.1 μg/ml (wine, benzoic acid); comparison with HPLC	Dehydroacetic acid	Pant & Trenerry (1995)
Other compounds						
Aspartame	Diet soft drinks	2 mg/ml	FSCE, EC (+0.69 V *vs* Ag/AgCl), sodium hydroxide	RSD_r (3–4%); detection limit (0.03 mg/ml)		Ye & Baldwin (1994*a*)
Aspartame, saccharin, acesulfame-K, benzoic acid, sorbic acid, caffeine	Diet soft drinks, cordials	0.04–0.5 mg/g	MEKC, UV (220 nm), deoxycholate in borate/phosphate (pH 8.6)	Recovery (100–112%); linearity; RSD_r (0.5–2%); comparison with HPLC	Dehydroacetic acid	Thompson *et al.* (1995*b*)
Aspartame, saccharin	Tomato sauce, marmalade	0.3 mg/g	MEKC, UV (220 nm), deoxycholate in borate/phosphate (pH 8.6)	Linearity; comparison with HPLC	Dehydroacetic acid	Thompson *et al.* (1995*b*)

Table 1. — *continued*

Analyte	Sample matrix	Typical concentration	CE mode	Validation^{a-c}	Internal standard	References
Aspartame, saccharin	Artificial sweetener	20–30 mg/g	MEKC, UV (220 nm), deoxycholate in borate/phosphate (pH 8.6)	Linearity; comparison with HPLC	Dehydroacetic acid	Thompson *et al.* (1995*b*)
Cyclamate, sorbate	Diet foods	3 mg/ml, 80 μg/ml (sorbate)	FSCE, UV/indirect UV (benzoate, 254 nm), benzoate in hexadecyltrimethylammonium hydroxide	Linearity; Repeatability (1%); comparison with HPLC	HIBA	Thompson *et al.* (1995*a*)
Glucose, glucitol	Apple juice	2–10 mg/ml	FSCE, EC (+0.60 V *vs* Ag/AgCl), sodium hydroxide	RSD_r(3–5%)		Ye & Baldwin (1994*b*)
Ethanol	Whisky	41% (v/v)	MEKC, indirect UV (diethylbarbituric acid, 230 mn), SDS in 5,5-diethylbarbituric acid (pH 9.5)		2-propanol	Altria & Howells (1995)
Synthetic colours	Confectionery cordials		MEKC, UV (214 nm) acetonitrile and deoxycholate in borate/phosphate (pH 8.6)	Reporting level (5 μg/g): comparison with HPLC	Azorubine or sunset yellow	Thompson & Trenerry (1995*b*)
Herbicides (metsulfuron methyl, thifensulfuron methyl, chlorsulfuron, rimsulfuron, tribenuron methyl)	Grains (wheat, barley, corn)	0.05 μg/g	MEKC, UV (234 nm), SDS in phosphate (pH 6.15)	Recovery (71–118%); linearity; detection limit (0.02–0.04 μg/g)		Krynitsky & Swineford (1995)
Formaldehyde, acetaldehyde	Spiked tap water	1 μg/ml	MEKC, UV (360 nm), SDS in borate/phosphate (pH 9)	Recovery (97–100%); linearity; detection limit (0.05–0.08 μm/ml); RSD_r (2–3%)		Takeda *et al.* (1994)
Terbuthylazine	Spiked tap water	1 ng/ml	MEKC, UV (214 nm), SDS and acetonitrile in borate (pH 8.0)	Recovery (99%); linearity; RSD_r (<99%); comparison with HPLC and immunoassay		Dinelli *et al.* (1995)
Chloramphenicol	Milk		FSCE, laser-induced fluorescence (exc. 488 nm, em. 560 nm), borate (pH 7.5)	Recovery (16–33%); detection limit (<0.1 ng/ml)		Blais *et al.* (1994)

Table 1. — ***continued***

Analyte	Sample matrix	Typical concentration	CE mode	Validation[a-c]	Internal standard	References
3-(*N*-oxalyl)-L-2,3-diaminopropanoic acid, 2-(*N*-oxalyl)- L-2,3-diaminopropanoic acid	Grass pea (*Lathyrus sativus*)	5 mg/g	FSCE, UV (195 nm), phosphate (pH 7.8)	Linearity; detection limit (0.1 mg/g), RSD_r (3.2%); comparison with colorimetry	Hippuric acid	Arentoft & Greirson (1995)
Phenolic compounds	Red wine	2–100 μg/ml	FSCE, UV (280 nm), borate (pH 9.5)	RSD_r (0–20%)		Gil *et al.* (1995)
Free fatty acids	Coco oil	50–400 μg/g	FSCE, indirect UV (*p*-anisate, 270 nm), trimethyl-β-cyclodextrin and methanol in TRIS/p-anisate (pH 8.2)	Linearity; detection limit (0.2–0.5 μg/g); comparison with gas chromatography	Undecanoic acid	Roldan-Assad & Gareil (1995)
Seleno amino acids (glutathione, seleno-cystamine, seleno-cystine, seleno-methionine)	Human milk	1–4 μg/ml	FSCE, UV (200 nm), phosphate (pH 2.5) or acetic acid (pH 1.9), glutathione: borate (pH 8.5) or phosphate (pH 6.0), Se-cystamine: borate (pH 8.5) or acetate (pH 5.5)	RSD_r (10–20%)		(Michalke, 1995)

[a] Repeatability (r) and reproducibility (R) are expressed as relative standard deviations (RSD_r and RSD_R) and relate to area unless otherwise indicated. Unless further specified, reproducibility data are taken as within-day variation (r).

[b] Values are for analytes in unaugmented samples unless otherwise indicated.

[c] Accuracy is comparison between standard addition and external calibration curve.

the presence of SDS was used to perform Ferguson plot analysis of high-molecular-weight glutenin subunits from wheat endosperm (Werner, 1995). Separation of fish muscle sarcoplasmic proteins by FSCE was reported to indicate both species differences and frozen storage changes (LeBlanc *et al.*, 1994). It was also used to differentiate and identify flatfish species (Gallardo *et al.*, 1995).

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