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Separation and determination of alditols and sugars by high-pH anion-exchange chromatography with pulsed amperometric detection

Rikke Andersen*, Annemarie Sørensen

Institute of Food Research and Nutrition, Division of Nutrition, Danish Veterinary and Food Administration, 19 Mørkhøj Bygade, 2860 Søborg, Denmark

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Abstract

Carbohydrates such as alditols (polyols or sugar alcohols), monosaccharides and disaccharides are separated as anions by anion-exchange chromatography with a sodium hydroxide eluent, MA1 CarboPac column and pulsed amperometric detection. We report a high-pH anion-exchange chromatographic–pulsed amperometric detection (HPAEC-PAD) method that determines all the polyols used as food additives in food products and the most commonly found mono- and disaccharides on a routine basis. The linearity, repeatability, internal reproducibility and accuracy are described. The applicability of the method has been demonstrated by the analysis of 46 relevant samples and by participation twice in the Food Analysis Performance Assessment Scheme (FAPAS) testing programme for food additives.

Keywords: Food analysis; Alditols; Sugars; Sugar alcohols; Polyols; Monosaccharides; Disaccharides

1. Introduction

The determination of alditols (in food chemistry often called polyols or sugar alcohols) is of great interest as these compounds are often used as food additives, and since polyols can have a laxative effect, they can be a health risk if consumed in large amounts. The osmotic diarrhoea is caused by the slow and uncompleted absorption in the body [1]. Polyols are sweeteners and they also have a bulking effect unlike the intense sweeteners. The sweetening effect of polyols is similar to sucrose or less. Furthermore, polyols have reduced cariogenic properties. Besides the other properties, the low hydroscopicity and high stability of polyols have provoked a growing interest in the food industry.

Polyols are often claimed to be useful sweeteners especially for diabetics. However, this is not true. Polyols affect the blood glucose level (though slower than sucrose). Besides, polyols contain calories; they have energy values of 10 kJ/g on average [2] (compared to 17 kJ/g of sucrose) and therefore must be accounted for in the meal planning of diabetics [1,3,4]. The Danish Diabetic Association does not recommend polyols to diabetics [5].

The polyols are often used in confectionery products such as chewing gum, pastilles, chocolate, candy, sugar free candy, and cakes. Xylitol, sorbitol,

*Corresponding author. Tel.: +45-33-956-000; fax: +45-33-956-001.

E-mail address: ria@fdir.dk (R. Andersen).

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mannitol, isomalt, lactitol, and maltitol are the polyols used in food products.

The determination of carbohydrates by chromatography has been hampered by two factors: the lack of a suitable high-performance separation method and the inability to detect the carbohydrates at low levels [6].

Huges and Johnson introduced pulsed amperometric detection (PAD) and the triple pulsed amperometry in 1981 [7,8]. PAD is nowadays the most commonly used method for detection of sugars and polyols and these non-chromophoric molecules are detected with excellent sensitivity [9–14].

Carbohydrates like mono- and disaccharides and polyols have similar structures, and are difficult to separate by conventional reversed-phased liquid chromatography. The hydroxyl groups of these carbohydrates have pK_a values in the range of 12–14 [15]. The weak acidic properties of carbohydrates allow ionisation and chromatographic separation in alkaline solutions and potential separation by high-pH (or -performance) anion-exchange chromatography (HPAEC) [9,16].

Rocklin and Pohl [6] and Edwards and Haak [17] first applied HPAEC with PAD to the determination of carbohydrates in 1983. Besides providing a more efficient separation and detection than other chromatographic methods, this analytical technique also minimizes the sample preparation [9]. Sensitivity, selectivity, and reliability of this technique are relevant aspects for the analysis of food products, often characterised by complex matrixes [18].

The methods described in the literature so far do not determine all the polyols and sugars relevant in food products, especially confectionery products. Corradini and co-workers [11,19–21] and Cataldi and co-workers [9,18] determined both polyols and sugars, but only a few at the time. Both Corradini's and Cataldi's groups used PA100 or MA1 CarboPac columns. Kerherve and co-workers [22] and Van Riel and Olieman [23] only determined sugars and no polyols using PA1 CarboPac columns.

Since the membership countries of the European Union are obligated to monitor and report the use of food additives it was important to us to develop a method that is able to determine all the polyols used as food additives in food products on a routine basis. Since the intake of sugars, especially sucrose is also of health and nutritional interest, and since the sugars are also detected by PAD, we also determined the most commonly found sugars in confectionery products (glucose, sucrose, lactose, and maltose).

We determined the linearity, repeatability, internal reproducibility, and accuracy of our method, which is able to separate and quantitate six polyols and four sugars in food products using a MA1 CarboPac column. Besides, 6-*O*-α-D-glucopyranosyl-D-mannitol (1,1-GPM) and fructose can be determined and semi quantitated. The applicability of the method has been demonstrated by the analysis of 46 relevant samples and by participation twice in the Food Analysis Performance Assessment Scheme (FAPAS) testing programme for food additives.

2. Experimental

2.1. Chemicals

All reagents employed were of analytical-grade unless stated differently. Sodium hydroxide, 50% (w/w) (1.54 g/ml) was from J.T. Baker (Deventer, Netherlands). Xylitol, D-sorbitol, D-mannitol, lactitol·H_2O, isomaltitol, maltitol, D-(+)-glucose, D-(−)-fructose, α-lactose·H_2O, sucrose, and maltose·H_2O were all purchased from Sigma (St. Louis, MO, USA). Isomalt (palatinit), which was not of analytical-grade since it only can be purchased as an article of commerce, was from Alsiano (Birkerød, Denmark). The deionized water used in the preparation of standard and sample solutions and eluents was obtained from a Milli-Q water system (Millipore, Bedford, MA, USA).

2.2. Chromatographic system

All chromatography was performed on a Dionex (Sunnyvale, CA, USA) DX-500 ion chromatograph. The system consisted of a quaternary gradient pump (GP40) and an electrochemical detector (ED40) including a detection cell with a gold working electrode and a pH-Ag/AgCl reference electrode. The pump had a standard-bore configuration and polyether ether ketone (PEEK) pump heads and flow paths. The DX-500 system was equipped with a PEEK rotary injection valve and an AS3500 autosampler. A personal computer equipped with the Dionex PeakNet 4.31 Window based software al-

Table 1
Operation parameters for the HPAEC system

Analytical column	CarboPac MA1 (250×4 mm I.D.), Dionex
Guard column	CarboPac MA1 (50×4 mm I.D.), Dionex
Column oven temperature	29°C
Eluent A	100% 1 *M* NaOH, degassed with helium for 30 min
Eluent B	100% water, degassed with helium for 30 min
Gradient[a], %A+%B (time)	30% A+70% B (0 min), 45%A+55%B (40–60 min), 80%A+20%B (80 min), 30%A+70%B (81–90 min)
Eluent flow-rate	0.4 ml/min
Sample loop volume	100 μl
Injection volume	10 μl
Detection	PAD
Detection mode	Integrated amperometry
Detection settings, *E* (time)	0.05 V (0.00 s), 0.05 V (0.20 s), 0.05 V (0.40 s), 0.75 V (0.41 s), 0.75 V (0.60 s), −0.15 V (0.61 s), −0.15 V (1.00 s)

[a] The gradient can vary with the age of the column. This gradient was used on a 1-year-old column set. The last 9 min of the gradient was equilibration before the next injection.

lowed the acquisition and processing of chromatograms and data. Table 1 summarizes the details of the operating parameters.

2.3. Standard solutions

The standard compounds (xylitol, sorbitol, mannitol, lactitol, isomaltitol, glucose, maltitol, fructose, lactose, saccharose, and maltose) were dried in exicator over diphosphorus pentaoxide (P_2O_5) until constant mass, and ca. 10 m*M* solutions were made of each compound. These solutions were kept at −18°C for a maximum of 6 months.

Standard solutions (ca. 0.04 m*M*) were made by mixing 4.00 ml of each standard compound solution in 100 ml and were kept at 5°C for a maximum of 1 week.

2.4. Samples

Forty-six different samples (cake, dessert, candy, wine gum, liquorice, chewing gum, fresh-mouth pastilles, and chocolate) were purchased from local retailers in the area of Copenhagen, Denmark. All samples were analysed in duplicate on two different days and with double injections of each sample extract.

2.5. Sample preparation

The samples were finely ground and homogenized. A food processor (Braun) was used for the cake products. A mini-chopping machine (Osterizer) was used for hard candy products. Softer and stickier products like wine gum, liquorice, and chewing gum were grounded (Ultra Turax) after freezing the products with fluent nitrogen. A suspension of finely ground sample, 2.5 or 5 g in 150 ml of 60°C water, was stirred for 4 h at room temperature using a magnetic bar. The suspension was transferred quantitatively to a 200 ml flask and mixed thoroughly. After centrifugation of the suspension at 2000 *g* it was filtered through a folded filter (S&S, 592.5;, diameter=125 mm). The solution was diluted with water to a concentration best from 0.2 to 0.8 m*M* of the individual carbohydrates. The diluted solution was filtered through a single use filter unit (Minisart, 0.2 μm) directly into the vials, and injected onto the column.

3. Results

3.1. Linearity, detection and determination limits

The linearity on a six-point calibration curve was checked ranging from 0.05 to 0.8 m*M* ($r^2>0.998$) for all the components. Also the linearity of sucrose was checked, ranging from 0.2 to 2.4 m*M* ($r^2>0.998$). The calibration curves were not forced through the origin. The intersections of the *x*-axis were not significantly different from zero.

In order to determine the detection and determi-

nation limits, ten double injections of a 0.01 m*M* standard solution containing xylitol, sorbitol, mannitol, lactitol, glucose, isomaltitol, maltitol, lactose, saccharose and maltose were analysed. This concentration is considered being close to the detection limit. The 0.01 m*M* solution was measured against and made from the dilution of a 0.4 m*M* standard solution. The detection limits were 0.3–1.1 mg/l (calculated at $3\times S$) and the quantitation limits were 1–4 mg/l (calculated at $10\times S$). These values correspond to 0.01–0.04 g/100 g and 0.04–0.16 g/100 g respectively, when a 5 g sample in 200 ml of water is diluted 10 ml in 100 ml.

3.2. Quantification

Quantification of the sugar alcohols and sugars was based on bracket calibrations of external standards. Twelve compounds (seven polyols and five mono- or disaccharides, see Fig. 1) were detected using the detector settings mentioned in Table 1. Ten (xylitol, sorbitol, mannitol, isomaltitol, lactitol, maltitol, glucose, lactose, sucrose and maltose) of these 12 compounds were quantificated easily. 1,1-GPM and fructose were only determined by semi-quantification.

Isomaltitol (6-*O*-α-D-glucopyranosyl-D-sorbitol) is often added to food products as isomalt, which also contains 6-*O*-α-D-glucopyranosyl-D-mannitol dihydrate (1,1-GPM). Isomalt contains about 50% 1,1-GPM and 50% isomaltitol (see Fig. 2). 1,1-GPM can not be purchased as a standard compound. For identification of 1,1-GPM, isomalt purchased as an article of commerce was used. For semi-quantifica-

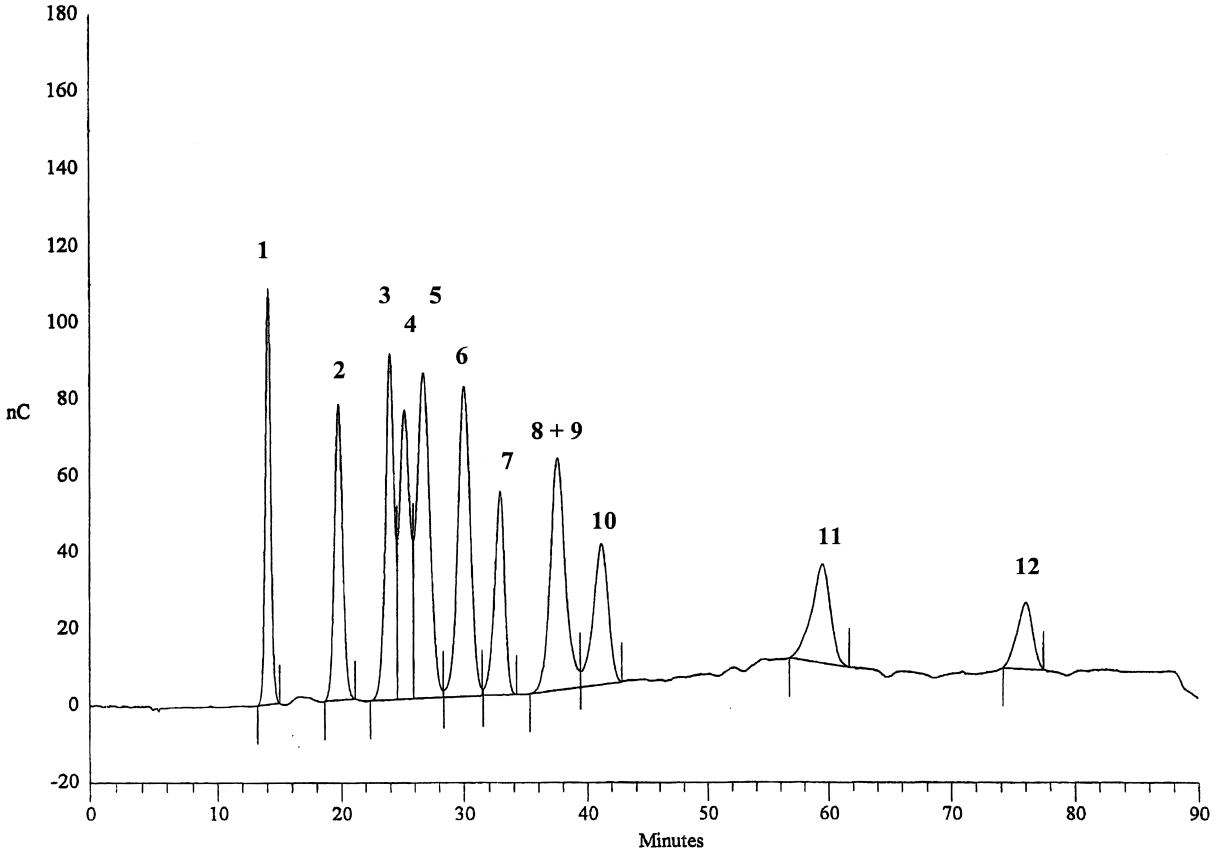

Fig. 1. HPAEC–PAD chromatogram of the 12 standard compounds. For HPAEC–PAD settings see Table 1. Peak assignment: 1=xylitol (14.10 min), 2=sorbitol (19.80 min), 3=mannitol (24.03 min), 4=isomaltitol (25.20 min), 5=lactitol (26.77 min), 6=1,1-GPM (30.07 min), 7=glucose (32.90 min), 8+9=maltitol+fructose (37.57 min), 10=lactose (41.17 min), 11=saccharose (59.47 min), 12=maltose (76.03 min). All 12 compounds were never present in the same sample.

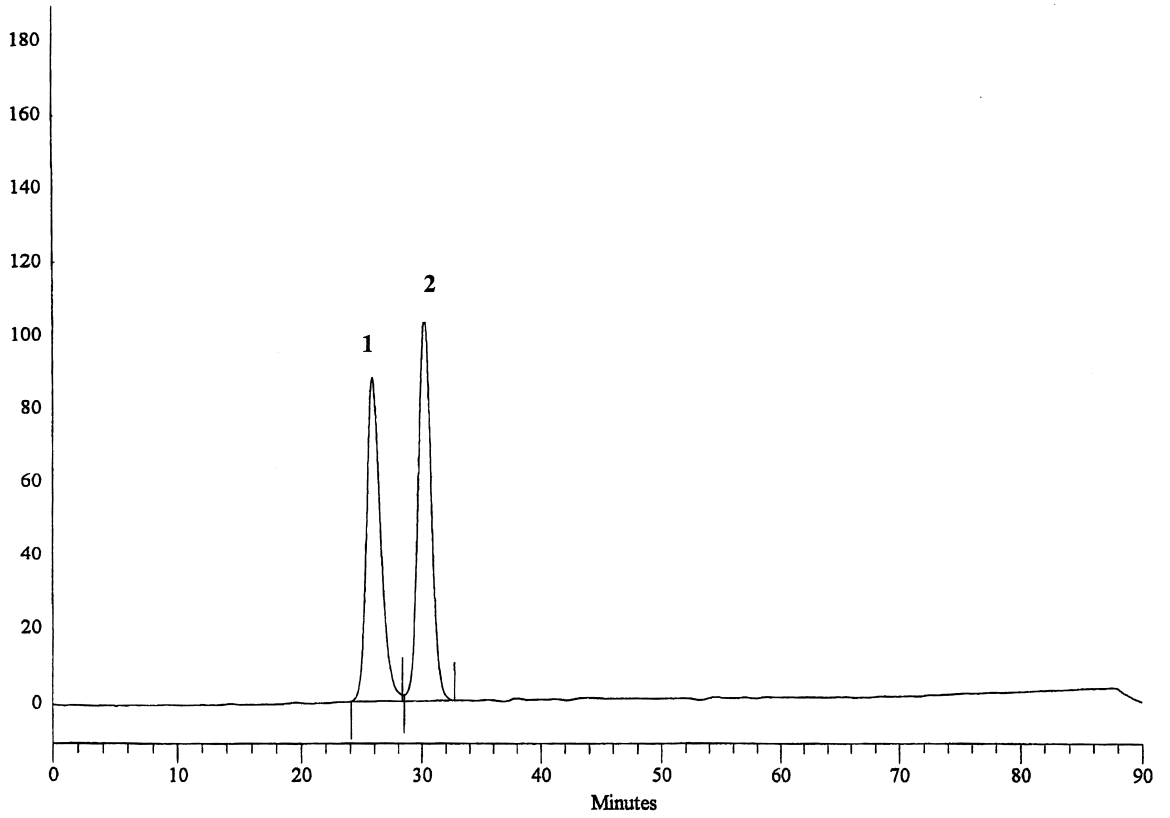

Fig. 2. HPAEC–PAD chromatogram of isomalt (article of commerce). For HPAEC–PAD settings see Table 1. Peak assignment: 1=isomaltitol (6-*O*-α-D-glucopyranosyl-D-sorbitol) (25.83 min), 2=1,1-GPM (6-*O*-α-D-glucopyranosyl-D-mannitol) (30.23 min).

tion of 1,1-GPM, the response factor of isomaltitol was used.

Fructose was also detected. However, fructose decomposes partially during the separation process due to the high pH. Therefore fructose was only measured by semi-quantification.

3.3. Separation and interference

The separation of the compounds depended on the age of the column set. The retention times of the compounds using an 1-year-old column set and the gradient mentioned in Table 1 were: xylitol (14.10 min), sorbitol (19.80 min), mannitol (24.03 min), isomaltitol (25.20 min), lactitol (26.77 min), 1,1-GPM (30.07 min), glucose (32.90 min), maltitol (37.57 min), fructose (37.57 min), lactose (41.17 min), saccharose (59.47 min) and maltose (76.03 min). The resolution between most compounds was >1.5. None of the analysed samples contained all 12 compounds. Two of the analysed samples contained seven compounds, the rest of the samples contained between two and six of the 12 compounds.

Maltitol and fructose had the same retention times. In order to determine whether a peak at 37.5 min was maltitol, fructose or a mixture of the two the following was done: the sample was analysed again using a column oven temperature of 40°C, instead of 29°C which was normally used, and 0.4 *M* NaOH isocratic elution, instead of a gradient. Fructose was totally decomposed at 40°C and maltitol was not affected (see Fig. 3). Maltitol had the same response at 29 and 40°C. If the peak had the same area in the 40°C as in the 29°C chromatogram it was maltitol. If the peak disappeared in the 40°C chromatogram it was fructose. If the peak was smaller in the 40°C chromatogram it was a mixture of maltitol and fructose. Maltitol could then be quantified from the 40°C chromatogram.

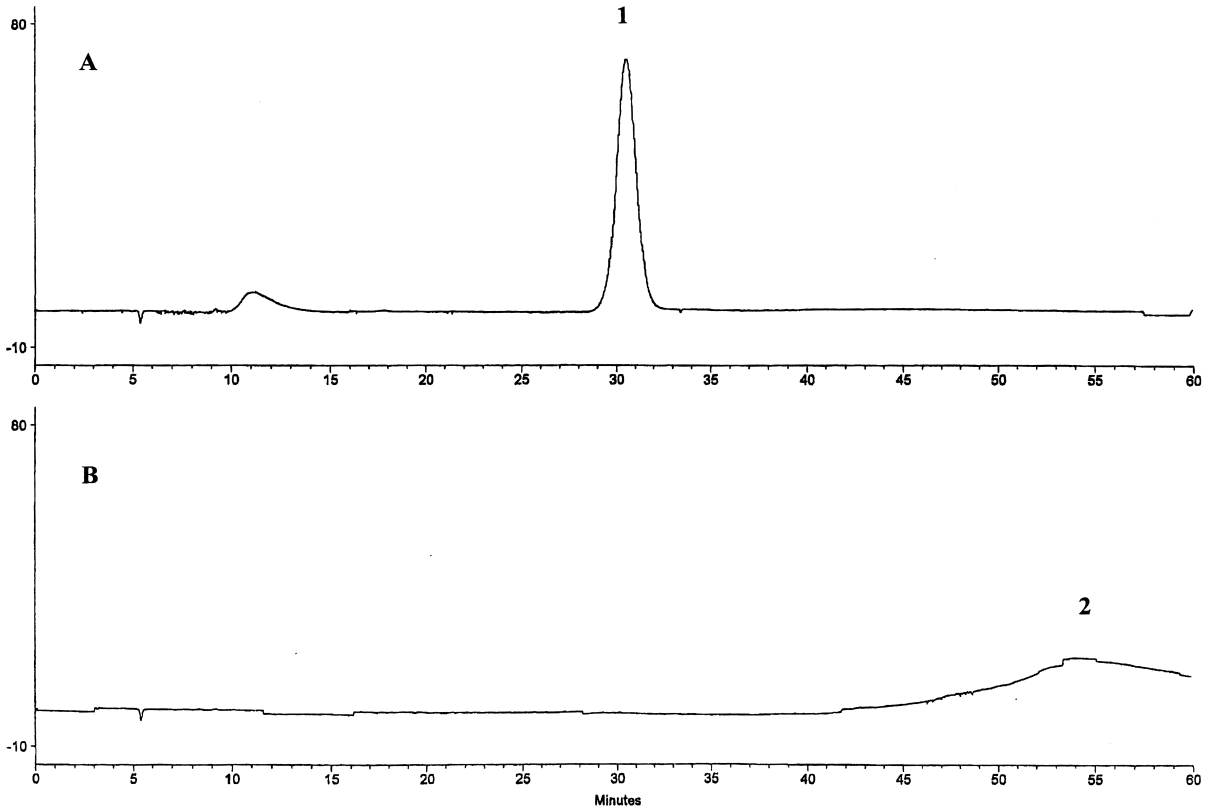

Fig. 3. HPAEC–PAD chromatograms of (A) maltitol and (B) fructose. HPAEC–PAD settings: isocratic eluation=0.4 *M* NaOH, column oven temperature=40°C, for the rest of the operation parameters see Table 1. Peak assignment: 1=maltitol, 2=fructose.

3.4. Repeatability and internal reproducibility

The method was validated with respect to the repeatability (within-day precision) and internal reproducibility (day-to-day precision).

Table 2 shows the repeatability calculated on six repeated injections over 9 h for two different samples (cake and pastille). The two samples are prepared as in-house reference materials and were stored in airtight plastic bags at −24°C. These two sample types are representative for the two major types of confectionery products analysed. The pastille sample has a high content of a few polyols (93.2 g sorbitol/100 g), which is characteristic for samples like chewing gum, fresh-mouth pastilles and sugar free candy. The cake sample has a much lower content of polyols (1.99 g sorbitol/100 g), which are typically used as stabilizer in cake products, and the sweetening effect comes from sucrose (29.3 g/100 g). This more complex content of polyols and sugars is

Table 2
Repeatability: within-day precision (N=6)

	Cake				Pastille sorbitol
	Sorbitol	Glucose	Lactose	Sucrose	
Mean (g/100 g)	1.99	0.63	1.26	29.3	93.2
SD (g/100 g)	0.05	0.02	0.04	0.92	3.0
RSD (%)	2.7	3.3	3.2	3.2	3.2

Table 3
Internal reproducibility: day-to-day precision

		Xylitol	Sorbitol	Mannitol	Isomaltitol	Lactitol	Glucose	Maltitol	Lactose	Sucrose	Maltose
>1 g/100 g	*N*	7	29	7	2	2	16	10	10	26	16
	RSD (%)	2.3	3.5	4.8	–	–	3.7	2	4.9	3	5.2
<1 g/100 g	*N*	1	15	9	0	3	10	6	4	0	4
	SD (g/100 g)	–	0.02	0.01	–	–	0.07	0.04	–	–	–

characteristic for chocolate and candy products as well.

Table 3 shows the internal reproducibility during analyses of different food products. The samples were analysed in duplicate on 2 different days. The internal reproducibility was calculated as RSD of the number (N) of the individual double determinations for samples with a content of the components ≥ 1 g/100 g and as the absolute SD for samples with content of the components <1 g/100 g. The standard deviations were not calculated for $N \leq 4$.

3.5. Accuracy

To determine the accuracy, recovery was measured in seven samples (five different matrices) by spiked analysis by adding the same amount as found in the samples. For contents <1 g/100 g the recoveries were found from 85.8 to 105%. For contents ≥ 1 g/100 g the recoveries were from 91.4 to 107%. Furthermore, the analysis was evaluated for sorbitol by participation twice in the FAPAS testing programme for food additives (Series XX, Round 2 and Round 5). The compatibilities with the mean values were 99.4 and 96.4%, respectively.

Since the extraction of the polyols and sugars are very simple in this method, the recovery was also measured in four ''home-made'' samples with high fat contents. Three cakes with 1, 3 and 6% sorbitol and sucrose were baked and analysed. The recoveries were from 95.8 to 102%. Finally, one chocolate sample containing maltitol from a pilot study at Toms Confectionery Group, Denmark was analysed with a recovery of 98.2%.

Table 4 shows the results in detail of the accuracy testing.

Table 4
Accuracy – recovery

Sample	Recovery (%)							
	Xylitol	Sorbitol	Mannitol	Glucose	Maltitol	Lactose	Sucrose	Maltose
Chewing gum 1[a]	99.1	95.4	99.8	–	–	–	–	–
Chewing gum 3[a]	91.4	102	–	–	–	–	–	–
Chewing gum 4	103	99.5	102	–	97.2	–	–	–
Candy 6[a]	–	85.8[b]	–	103	–	98.3	102	100
Candy 8	–	103	–	103	–	–	107	–
Candy 14[a]	–	88.7[b]	–	107	–	–	103	95.3
Pastille 4	100[b]	98	105[b]	–	–	–	–	–
Home-made cake 1%	–	96.7	–	–	–	–	100[c]	–
Home-made cake 3%	–	102	–	–	–	–	95.8[c]	–
Home-made cake 6%	–	98.5	–	–	–	–	98.3[c]	–
Chocolate from Tom	–	–	–	–	98.2	–	–	–
FAPAS, Round 2	–	99.4[d]	–	–	–	–	–	–
FAPAS, Round 5	–	96.4[d]	–	–	–	–	–	–

[a] Single determination.
[b] Content <1 g/100 g.
[c] White sugar.
[d] Compatibility with mean value.

Table 5
Content of polyols and sugars in 46 samples

Sample type	No.	Content (g/100 g)												Sum polyols	Sum sugars
		Xylitol	Sorbitol	Mannitol	Lactitol	Glucose	Isomaltitol	1,1-GMP[a]	Maltitol	Fructose[a]	Lactose	Sucrose	Maltose		
Cake	1	–	3.87	0.13	–	13.40	–	–	–	15.40	–	21.60	–	4.00	50.4
	2	–	3.71	0.12	–	0.67	–	–	–	2.67	2.08	32.40	0.38	3.83	38.2
	3	–	2.16	–	–	0.39	–	–	–	–	0.74	23.20	1.18	2.16	25.5
	4	–	0.46	–	–	6.45	–	–	0.49	–	1.03	24.00	5.51	0.95	37.0
	5	–	0.69	–	–	4.96	–	–	0.26	–	0.85	22.90	2.65	0.95	31.3
	6	–	0.62	–	–	–	–	–	–	–	–	38.40	–	0.62	38.4
	7	–	0.57	–	–	0.74	–	–	–	–	–	33.90	–	0.57	34.7
	8	–	0.47	–	–	1.05	–	–	–	–	–	35.40	–	0.47	36.4
	9	–	0.22	–	–	7.99	–	–	–	6.14	–	35.40	1.14	0.22	50.7
Chocolate	1	–	0.29	–	12.2	0.56	–	–	–	–	–	–	–	12.5	0.56
	2	–	0.39	–	–	0.85	–	–	10.90	–	10.20	–	–	11.3	11.0
	3	–	0.39	–	5.35	0.74	–	–	4.65	–	9.32	3.45	–	10.4	13.5
	4	–	1.95	–	–	8.12	–	–	–	8.37	0.80	25.60	–	1.95	42.9
	5	–	–	–	–	0.58	–	–	–	–	1.77	38.00	–	0	40.3
Chewing gum	1	29.0	33.4	4.75	–	–	–	–	1.63	–	1.63	–	–	67.2	1.63
	2	7.75	52.1	0.24	–	–	–	–	1.27	–	–	–	–	61.4	0
	3	6.34	59.3	0.24	–	–	–	–	0.71	–	–	–	–	66.6	0
	4	20.20	32.8	1.03	–	–	–	–	1.54	–	–	–	–	55.6	0
Pastilles	1	1.94	94.4	0.41	–	–	–	–	–	–	–	–	–	96.8	0
	2	–	91.9	0.59	–	–	–	–	–	–	–	–	–	92.4	0
	3	–	2.20	–	–	–	–	–	71.8	–	–	–	2.13	74.0	21.3
	4	0.96	38.6	0.85	0.70	–	–	–	6.34	–	–	–	1.29	47.5	1.29
	5	10.30	1.20	–	–	–	–	–	21.8	–	–	–	13.4	33.3	13.4
	6	–	0.51	–	–	0.44	–	–	29.2	–	–	–	10.5	29.7	10.9
Sugar free candy	1	–	–	–	–	–	48.7	64.4	–	–	–	–	–	113[b]	0
	2	–	88.3	4.13	–	–	–	–	–	–	–	–	–	92.4	0
	3	–	86.4	4.02	–	–	–	–	–	–	–	–	–	90.4	0
	4	–	85.7	4.09	–	–	–	–	–	–	–	–	–	89.8	0
	5	–	84.8	4.04	–	–	–	–	–	–	–	–	–	88.9	0
	6	–	34.2	2.30	–	–	–	–	–	–	–	–	–	36.5	0
Candy	1	–	–	–	–	–	16.5	21.4	36.0	–	–	–	8.36	73.8	8.36
	2	–	10.1	–	–	–	–	–	–	–	–	80.4	–	10.1	80.4
	3	–	6.62	–	–	5.44	–	–	–	–	–	48.2	7.44	6.62	61.1
	4	–	4.21	–	–	6.20	–	–	–	–	–	49.3	7.91	4.21	63.4
	5	–	2.86	0.14	0.27	11.40	–	–	–	10.2	–	15.1	4.93	3.27	41.7
	6	–	3.07	–	–	0.84	–	–	–	–	6.33	38.1	3.23	3.07	48.5
	7	–	2.49	0.17	–	4.29	–	–	–	–	–	56.8	3.51	2.66	64.6
	8	–	1.83	–	0.09	4.04	–	–	–	4.44	0.80	39.7	0.47	1.92	49.5
	9	–	1.82	–	–	4.27	–	–	–	–	5.18	29.8	3.11	1.82	42.4
	10	–	1.33	–	–	2.18	–	–	–	–	2.57	48.9	9.21	1.33	62.8
	11	–	0.51	–	–	16.3	–	–	0.5	–	–	27.4	14.2	1.01	57.9
	12	–	0.15	–	–	17.7	–	–	0.61	–	–	22.1	17.1	0.76	56.8
	13	–	–	–	–	4.83	–	–	–	–	–	56.3	3.52	0	64.6
Various	1	–	3.39	–	–	0.84	–	–	0.51	–	–	39.0	0.58	3.9	40.4
	2	–	0.81	–	–	–	–	–	–	–	–	4.31	–	0.81	4.31
	3	–	5.18	–	–	10.9	–	–	–	12.4	–	38.7	0.37	5.18	62.3
All samples	*N*	7	42	16	5	27	2	2	15	7	13	27	23	44	35
	Min.	0.96	0.15	0.12	0.09	0.39	16.50	21.4	0.26	2.67	0.74	3.45	0.38	0.09	0.38
	Max.	29.0	94.4	4.75	12.20	17.70	48.70	64.4	71.80	15.40	10.20	80.40	21.3	94.4	80.4

[a] Semi quantitative determination.
[b] Sum of polyols >100 properly due to semi-quantiative determination of 1,1-GMP.

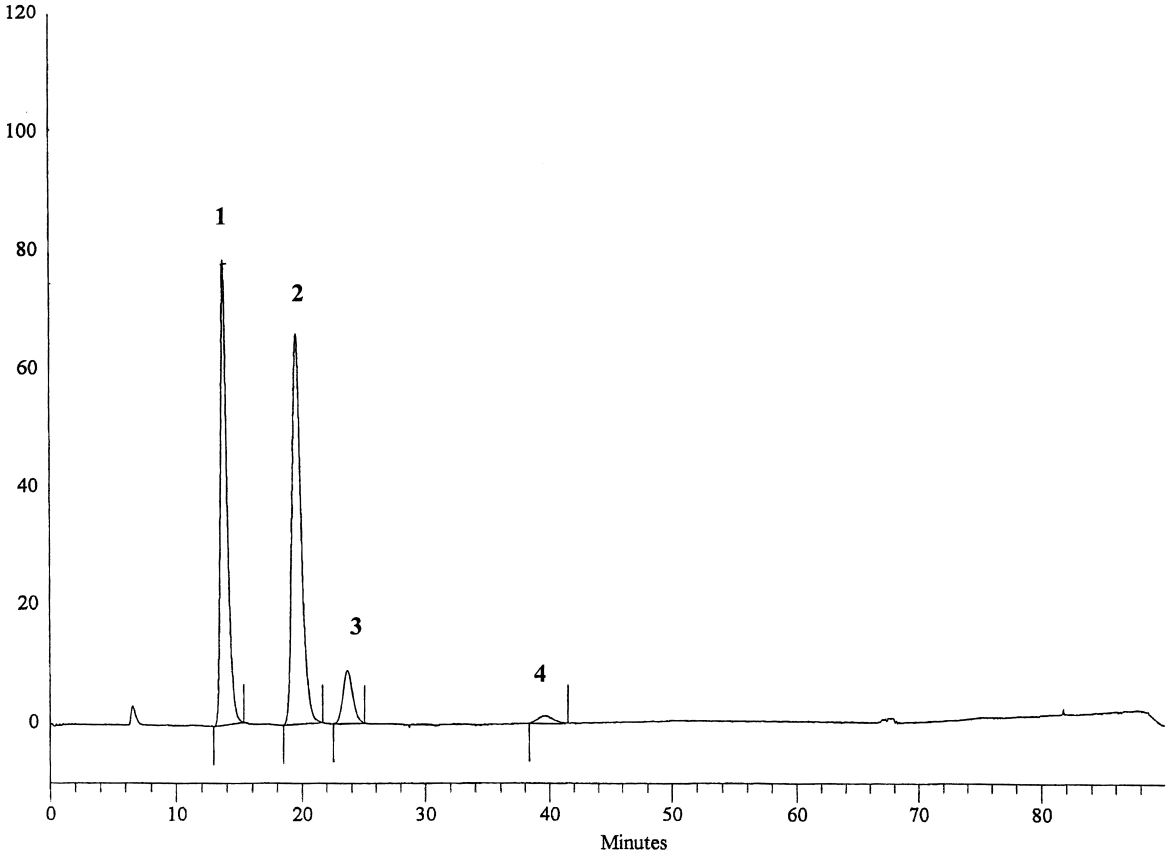

Fig. 4. HPAEC–PAD chromatogram of a chewing gum sample (no. 1). For HPAEC–PAD settings see Table 1. Peak assignment: 1=xylitol (13.77 min), 2=sorbitol (19.57 min), 3=mannitol (23.67 min), 4=maltitol (39.63 min).

3.6. Content of polyols and sugars in 46 samples

The method was tested on 46 different relevant samples. Table 5 shows the results. Forty-four of the samples contained polyols and 35 samples contained mono- or disaccharides. For polyols the level ranged from 0.09 g/100 g to 94.4 g/100 g. The sugars ranged from 0.38 g/100 g to 80.4 g/100 g. Most of the samples (42) contained sorbitol. Glucose and sucrose were found in 27 of the samples. An example of a sample chromatogram (chewing gum no. 1) is shown in Fig. 4.

4. Discussion and conclusion

The HPAEC of carbohydrates with alkaline eluent systems provides a powerful tool in the analysis of carbohydrates. The combination of this chromatographic system with PAD provides a highly selective and sensitive method well suited to complex samples like food products. The CarboPac MA1 column permits appropriate separation of polyols and several mono- and disaccharides.

The proposed method allows the determination of all the polyols used as food additives in food products on a routine basis. The linearity, repeatability, internal reproducibility and accuracy have been determined with satisfactory results. Also, the applicability of the method has been demonstrated by the analysis of 46 relevant samples and by the participation twice in the FAPAS testing programme for food additives.

However, the following limitations of our method

must be mentioned: (1) Our method is not optimal for samples containing only mono- and disaccharides and no polyols, since fructose decomposes on the column due to the high NaOH concentration used. In that case CarboPac PA1, PA10 or PA100 columns can be recommended, since lower NaOH concentrations are used. (2) The poor separation between fructose and maltitol is a limitation of the method. However, to our knowledge there does not seem to be other methods in the literature separating these two compounds on the CarboPac MA1 column. At least we suggest how to distinguish between fructose and maltose. Cataldi and co-workers [24–26] suggest the use of Barium ions in the mobile phase in order to improve the separation in general. However, Cataldi and co-workers do not attempt to separate fructose and maltitol. (3) We determined only mono- and disaccharides. The samples could also contain some trisaccharides, other oligosaccharides and polysaccharides, since maltitol syrup and sorbitol syrup, which are used by the food industry, also contain some hydrogenated oligo- and polysaccharides. (4) The response was often declining during analysing a series. We used bracketing calibration in order to compensate for this decline. The deviation between the standards in a bracket was less than 5%; otherwise the series was rejected.

In the future more foods and food products will be analysed in order to monitor the use of these food additives and in order to prepare dietary intake surveys of polyols and sugars.

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