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Capillary electrophoretic analysis of the derivatives and isomers of benzoate and phthalate

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Abstract

A capillary electrophoretic method for the analysis of 12 commonly found derivatives and isomers of benzoate and phthalate, including *p*-toluic acid, *p*-acetamido and *p*-hydroxy derivatives of benzoic acid, salicylic acid and its acetyl ester, 2- and 4-isomers of carboxybenzaldehyde, *meta*-, *para*-, and *ortho*-isomers of phthalic acid, and monomethyl terephthalic acid was developed. Capillary electrophoresis (CE) was performed in the free zone electrophoresis mode. Performing CE in 10 m*M* phosphate buffer, pH 7.0 could separate most of the benzoic acid derivatives except the structural or positional isomers. The positional isomers of phthalic acids could be completely separated with co-addition of α- and β-cyclodextrins. Addition of poly(ethylene glycol) 600 (4%) could further resolve some structural isomers. The CE method developed here is rapid, i.e. complete separation could be achieved in less than 8 min for the nine monoanionic benzoate derivatives and in less than 14 min for the three dianionic phthalate isomers. The new method has good precision and linearity and can be readily applied to real samples for quantitative analysis. It is sensitive and can detect sub-ppm (w/w) level of impurity in real terephthalic samples.

Keywords: Positional isomers; Structural isomers; Benzoic acids; Phthalic acids; Terephthalic acid

1. Introduction

Terephthalic acid (TPA) is the monomer for many important polymers including polyesters, polyamides, polybenzothiazoles, and polybenzoxazoles [1]. Annually 8×10^9 kg of TPA are produced worldwide for use primarily in high-grade polyester fiber production [2]. Feedstocks for polyester production must be extremely pure. If certain impurities are present in TPA, harmful effects can be observed. For example, monofunctional compounds can cap the polyester chain and limit molecular mass build-up. Positional isomers of TPA can lead to undesirable rheological and spinning properties [3]. In order to evaluate the purity of terephthalic acid products and quantify specifically trace amounts of impurities, a sensitive analytical technique needs to be developed and devised to detect traces for chemical quality-control assays.

Of the TPA feedstock used worldwide, around 70% is produced with a soluble cobalt–manganese–bromine catalyst system [4]. This yields nearly

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quantitative oxidation of the *p*-xylene methyl groups in acetic acid with small xylene losses. The liquid-phase oxidation of the *p*-xylene methyl groups occurs in steps, with two intermediates, *p*-toluic acid and 4-carboxybenzaldehyde. The intermediate *p*-toluic acid does not oxidize readily. Its concentration builds up and oxidation tends to stop before reaching high conversions to TPA. There also exists yellow impurities, residual amounts of catalyst metals, and bromine produced from the process. Besides, isophthalic acid and *o*-phthalic acid may produce owing to the oxidation of xylene positional isomers from the impurities of *p*-xylene feedstock. The Amoco purification process removes 4-carboxybenzaldehyde to less than 25 ppm (w/w), and also gives a white powder from the slightly yellow feed; nevertheless, the residues still remain and restrict their usefulness [5].

In the past, the analysis of benzoic acid derivatives and their positional isomers has generally been performed using chromatographic methods [6–8]. However, the separation of structurally closely related compounds has been proved difficult, if not impossible, with the use of liquid chromatographic method. Although the gas chromatographic method is a good alternative in view of its good resolution, it cannot be applied directly to aqueous samples. In addition, considerable care and additional work are required in order to prevent decomposition of the analyte.

Capillary electrophoresis (CE) has been successfully applied to the separation of positional isomers of amino and methyl derivatives of benzoic acid with the aid of organic solvent [9–11]. A reversed electroosmotic flow (EOF) CE method has been developed for the separation of eight aromatic acids [12]. An extensive study has been made on the electrophoretic behavior of 56 aromatic-containing acids using either capillary zone electrophoresis (CZE) or micellar electrokinetic chromatography [13]. Addition of poly(ethylene glycol) (PEG) as a free matrix could enhance the CE separation for some benzoic acid derivatives as well as for mixtures of nucleotides [14]. This interaction is attributable to hydrogen bond formation. Separation mechanism based on the cyclodextrin (CD) complexation is another valuable tool in the CE system for enhancing the separation of enantiomers or positional isomers [15–21]. The extent by which analytes interact with CD depends on their molecular size, polarity, shape, etc. and also the size of the cavity of CD [22–26]. Although a pressing problem in some CE applications is perhaps the lack of sensitivity, nevertheless, for most chemical quality-control assays, the sensitivity of CE is more than adequate [27].

In this paper, we attempt to optimize capillary electrophoretic separation of various benzoic acid derivatives by utilizing the effects of CDs and PEG. The effects exerted by pH and various additives on the selectivity and resolution of CE were evaluated. A complete baseline separation for the positional isomers of phthalic acid could be achieved by adding mixtures of α-CD, β-CD, and PEG. The new cyclodextrin-mediated CZE method developed here is sensitive, quantitative, and can be readily applied to the quality-control assay of terephthalic acid in industrial areas.

2. Experimental

2.1. Chemicals

All chemicals were of analytical or reagent grade, or the highest purity available from several suppliers and were used as received. Monomethyl terephthalate, isophthalic acid (*m*-phthalic acid), *o*-phthalic acid, terephthalic acid (*p*-phthalic acid, TPA), 4-carboxybenzaldehyde, and *p*-toluic acid were obtained from Merck (Darmstadt, Germany). Benzoic acid, salicylic acid, aspirin (acetylsalicylic acid), *p*-hydroxybenzoic acid, 2-carboxybenzaldehyde, *p*-acetamidobenzoic acid, meistyl oxide, α-CD, β-CD, and γ-CD were purchased from Sigma (St. Louis, MO, USA). Sodium phosphate and PEG 600 (poly(ethylene glycol), M_r 600) were from Riedel-de Haën (Seelze, Germany). Doubly deionized water prepared with a Milli-Q system (Millipore, Bedford, MA, USA) or doubly deionized-distilled water was used exclusively for all solutions (≥18.2 MΩ cm resistivity).

2.2. Samples, buffers and pH adjustment

The standard solutions of benzoic acid and derivatives were prepared, each in 10 m*M*. These solutions

were mixed and diluted to 0.1 m*M* prior to injection into the CE system. Solution pH was measured with a Sundex Model SP-2200 pH meter (Taipei, Taiwan) and combination glass electrode (Mettler Toledo Inlab 439/120). The pH of the running phosphate buffer was adjusted by adding aliquots of 1 or 0.1 *M* NaOH to the desired pH as specified in the figures. The pH of the buffer was checked periodically and readjusted when necessary. For the CD experiments, 10–40 m*M* solutions were made and then diluted to the desired concentration. Real samples of phthalic acid were obtained from the four local plants of chemicals manufactures in Taiwan. The powder samples (~1 g) were dissolved in 2 ml methanol and sonicated for 5 min. Most of the TPA solids were undissolved but all the impurities were completely dissolved according to the solubility data. The saturated solution was equilibrated for 5 min and let settle. Then ~1 ml of clear solution was carefully withdrawn with a pipette and filtered through a 0.2-μm membrane. The filtered solution was directly injected into the capillary. All buffer solutions were also filtered through 0.2-μm membranes. For the determination of the molar absorptivities for the various organic acids, the UV–Vis absorption spectra of organic acids (10^{-5} to 10^{-4} *M* in 10 m*M* phosphate buffer, pH 7.0 or 11.0) were measured in 1.0-cm quartz cuvettes (190–400 nm) by using a double-beam scanning spectrophotometer (Shimadzu UV-1601, Tokyo, Japan).

2.3. Electrophoretic procedures

CE experiments were carried out in an automated PrinCE-C455 system (Prince Technologies, Emmen, The Netherlands), equipped with a Varian ProStar 340 UV–Vis detector (Palo Alto, CA, USA). The real samples of phthalic acid were also analyzed by using a Beckman P/ACE 5000 system (Fullerton, CA, USA). The two instruments gave essentially identical results. The analytes were detected at two fixed wavelengths 215 and 240 nm, depending on the experiments as specified in the figures. The data were collected and processed by a DaX data system (Prince Technologies). The separation capillaries (bare fused-silica), 68 cm (61 cm to the detector)× 75 μm I.D.×365 μm O.D., were purchased from Polymicro Technologies (Phoenix, AZ, USA). Procedures for capillary pretreatment, pre- and post-washing were similar to those reported previously [28]. Samples were introduced using the controlled-pressure system (50 mbar for 0.05 min). The instrument injection performance was typically 0.5% RSD. The separation run was carried out at +25 kV constant voltage at 25 °C constant temperature and with a current of ~40 μA. Peak identification for each analyte was carried out by spiking with the known standard and the peak with increased height was identified. Mesityl oxide was added to the samples as a neutral marker for the electrophoretic mobility determination. The electroosmotic mobility and the electrophoretic mobility of the analytes were calculated as described previously [28]. When comparing the electropherograms at different pHs, or for different concentrations of CD, the relative migration time was calculated with respect to the migration time of the neutral marker, mesityl oxide.

3. Results and discussion

3.1. CE separation of benzoic acid and its derivatives

Displayed in Fig. 1 is the electropherogram of a mixture of benzoic acid, phthalic acid, and their derivatives and isomers run in 10 m*M* phosphate buffer at pH 7.0. A positive voltage is applied to the CE system. Although all the anions have negative mobilities, they migrate toward the cathode due to the strong EOF. The migration times of the analytes increase in the following order: *p*-acetamidobenzoic acid (1), monomethyl terephthalate (2) and aspirin (3) (not separable), *p*-hydroxybenzoic acid (4), *p*-toluic acid (5), 2-carboxybenzaldehyde (6) and 4-carboxybenzaldehyde (7) (not separable), benzoic acid (8), salicylic acid (9), *o*-phthalic acid (10), and terephthalic acid (TPA) (11) and isophthalic acid (12) (not separable). Of these 12 analytes, the first nine are monocarboxylic acid or monoester of phthalic acid, whereas the last three are dicarboxylic acids. Of the first nine analytes, monomethyl terephthalate and aspirin (peaks 2 and 3) were not separable under this condition. Also 2- and 4-carboxybenzaldehydes (peaks 6 and 7) were not separable. Of the three position isomers of phthalic acid, only

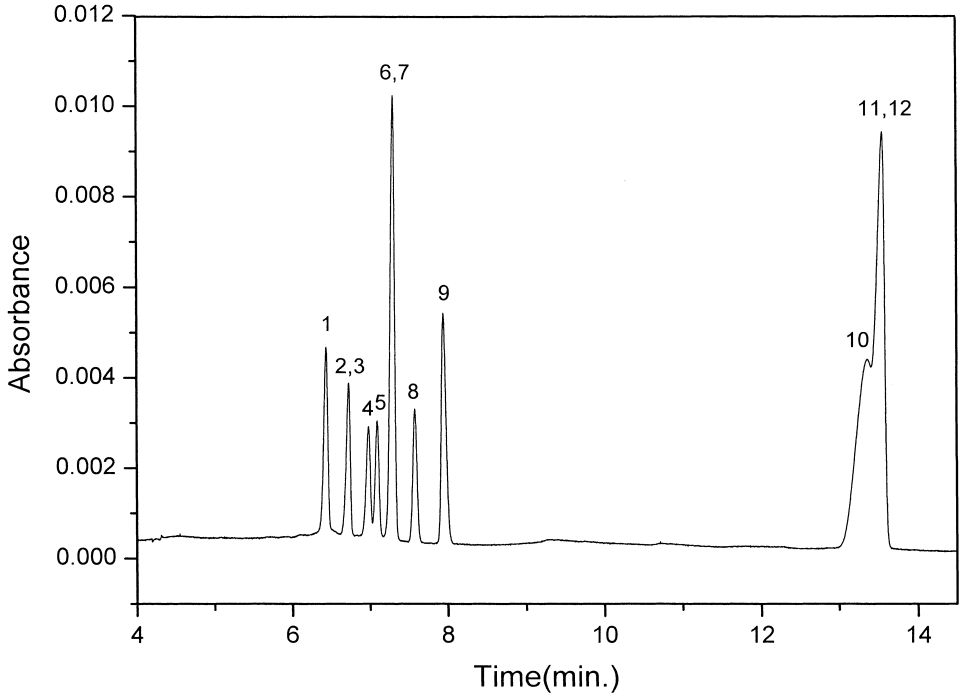

Fig. 1. Electropherogram of benzoic acid and 11 of its derivatives (0.1 m*M* each) run in 10 m*M* phosphate buffer (pH 7.0). Peaks: 1=*p*-acetamidobenzoic acid; 2=monomethyl terephthalate; 3=aspirin; 4=*p*-hydroxybenzoic acid; 5=*p*-toluic acid; 6=2-carboxybenzaldehyde; 7=4-carboxybenzaldehyde; 8=benzoic acid; 9=salicylic acid; 10=*o*-phthalic acid; 11=TPA; 12=isophthalic acid. Absorbance measured at 215 nm. See the Experimental section for other CE conditions.

o-phthalic acid which migrated ahead of TPA and isophthalic acid was partially separated from the other two; the latter two were not resolved. The three position isomers of phthalic acid, which carried two negative charges, were the fastest migrating toward the anode and were the last to migrate passing by the detector (at the cathode). Similarly, we have also shown that when a positive voltage is applied, trimellitate (tricarboxylic) and pyromellitic acid (tetracarboxylic) migrate even slower (toward the detector) than phthalic acids [28].

3.2. Effect of pH

Since the electrophoretic mobility of ion is strongly dependent on its degree of ionization, a careful manipulation of pH can sometimes assist in resolving peaks that are difficult to separate. The influence of pH on CE separation was investigated. As listed in Table 1, the first pK_a values of these analytes are from 2.95 to 4.58. The second pK_a of the three phthalic acids are from 4.50 to 5.41 whereas the pK_a for the hydroxyl groups of *p*-hydroxybenzoic acid and salicylic acid are 9.46 and 13.70, respectively.

Table 1
Molecular masses (M_r) and pK_a values of the various aromatic organic acids

Analyte	M_r	pK_{a1} [a]	pK_{a2} [a]
p-Acetamidobenzoic acid	179.2	3.62[b]	
Monomethyl terephthalate	180.2	3.67[c]	
Aspirin	180.2	3.38[b]	
p-Hydroxybenzoic acid	138.1	4.58	9.46
p-Toluic acid	136.2	4.34	
2-Carboxybenzaldehyde	150.1	4.36[c]	
4-Carboxybenzaldehyde	150.1	3.66[c]	
Benzoic acid	122.1	4.20	
Salicylic acid	138.1	2.97	13.70
o-Phthalic acid	166.1	2.95	5.41
TPA	166.1	3.51[b]	4.82[b]
Isophthalic acid	166.1	3.50	4.50

[a] From Ref. [31] except where indicated.
[b] From Ref. [32].
[c] Determined in the present study by the pH titration method.

Therefore in the pH range from 6.0 to 8.0, the effect of pH will exert most significantly on the EOF and will not affect the migration order of these analytes as shown in Fig. 2. Only *p*-hydroxybenzoic acid changed its migration order at pH above its second pK_a (~9.4) where it carries a second negative charge and migrated faster toward the anode. To resolve peaks 2 and 3, it was necessary to lower pH below 3.4. In this acidic pH range, these peaks still did not resolved well. Furthermore, at low pH value under isocratic conditions, the migration times for all analytes became exceedingly long thus rendering the CE analysis impractical. In order to shorten the migration times of samples, conditions at alkaline pH (11.0) were considered and are discussed in the following section.

3.3. Effect of CD

Besides studying the effects of pH in an attempt to further resolve the phthalate isomers, BSA, a protein that has been shown to improve chiral separation, has also been investigated but neither could it bring a complete separation of phthalate isomers. CDs were then investigated for their influence on CE resolution. The effects of α- and β-CD on the mobilities of the three phthalate isomers are shown in Fig. 3. The absolute electrophoretic mobilities all decrease when increasing CD concentration. The top curve shown indicates that the mobility of TPA was most affected by the concentration of β-CD. The bottom curve indicates that the mobility of isophthalate was least influenced by α-CD. By comparing the effects of CDs on the mobility, the faster mobilities imply that phthalate isomers interact more closely with β-CD than α-CD. One factor may be the size of the CD cavity, which governs to some extent the ability of the phthalate isomers to form inclusion complexes with CDs. CDs are toroid-shaped molecules with cavities that are hydrophobic inside. The cavity of α-CD (~5.2 Å) favors small linear molecules such as sorbic acid, whereas larger molecules such as salicylic acid would fit into the cavity of β-CD (~6.4 Å) [29]. The cavity of α-CD is probably too small for the benzene ring to fit in, so phthalate isomers show

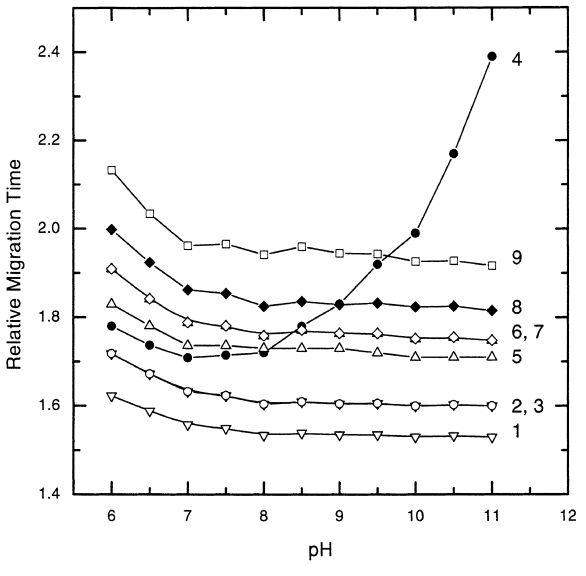

Fig. 2. Effects of pH on the relative migration times of benzoic acid and its derivatives. Except the pH, other CE conditions are the same as in Fig. 1. Data for the three position isomers of phthalic acid are not shown, since pH has no effect on their migration order. Relative migration times were calculated with respect to that of the neutral marker.

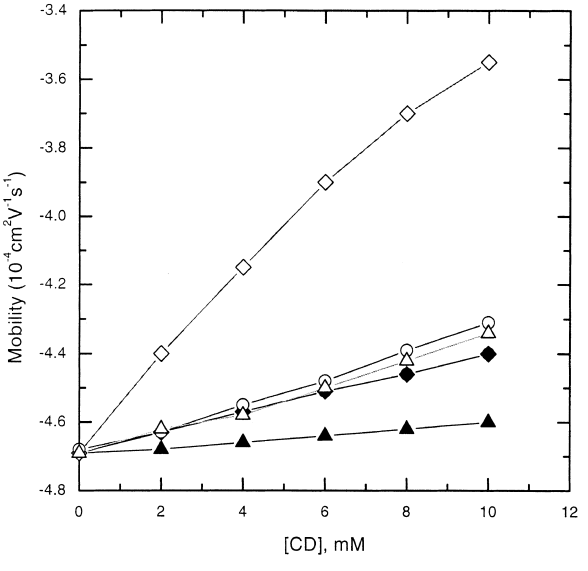

Fig. 3. Effect of the concentrations of α-CD in (●) *o*-phthalic acid, (♦) TPA and (▲) isophthalic acid and β-CD in (○) *o*-phthalic acid, (◇) TPA and (△) isophthalic acid on the electrophoretic mobilities of the three phthalate isomers run in 10 m*M* phosphate buffer, pH 11.0. See the Experimental section for other CE conditions. Note: TPA and OPA are not resolved with concentrations of α-CD in the range 0–10 m*M*.

interactions in preference to β-CD. The other factor may be the position of dicarboxyl groups on the benzene ring [19]. The strongest interaction between TPA and β-CD probably occurs because the dicarboxyl groups on the benzene rings are in the para position and may form hydrogen bonds with the hydroxyl groups outside CDs [26]. Their molecular sizes and the positions of dicarboxyl groups result in their interactions with α-CD being much weaker than with β-CD.

The addition of α-CD was beneficial mainly for separating isophthalic acid (▲ labels) from TPA (✦ labels) or *o*-phthalic acid (● labels). On the other hand, β-CD was primarily responsible for separating TPA (◇ labels) from the other two isomers. The effect of γ-CD was similar to that of β-CD (data not shown), but it required a higher concentration and the resolution was less effective. Not a single kind of CD alone could resolve all three isomers; a mixture of α- and β-CD was, therefore, utilized in the running buffer in an attempt to improve separation efficiency. Still, the addition of CDs affected adversely the CE resolution of some of the monoanionic benzoate derivatives (data not shown). The CE separation conditions were still unsatisfactory.

3.4. Effect of PEG

Fig. 4 shows the separation electropherogram of 12 benzoate and phthalate derivatives and isomers under the optimized conditions. Improved separation was achieved by addition of PEG. It was reported that addition of PEG was found to enhance resolution for the benzoic acid derivatives that contain hydroxyl, amide, or amine groups [14]. The hydrogen bond is observed in the interaction between polyethers and some hydrogen donors. An example is the interaction between Lasalocid A (a non-cyclic polyether ionophore) and amine complexes such as $[Co(NH_3)_6]^{3+}$, which leads to corresponding adduct formation involving hydrogen bonding in hydrophobic media [30]. Although the hydrogen bond is relatively weak in strength, it is frequently encountered with organic compounds and may be utilized for separation in CE. The attractive interaction of an analyte anion with PEG should decrease the relative electrophoretic velocity in the positive potential direction and accelerate the relative mobility of the analyte in the negative potential direction because PEG migrates in the negative potential direction at the EOF rate. In comparison to previous work, the

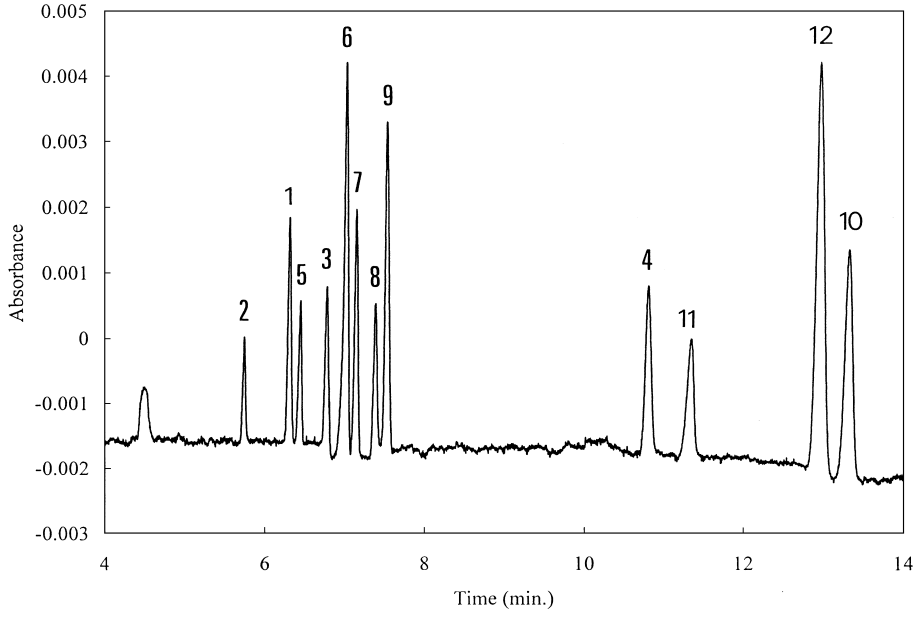

Fig. 4. Electropherogram of mixtures of standards (0.1 m*M* each) run in 10 m*M* phosphate buffer with 4 m*M* α-CD, 8 m*M* β-CD, and 4% PEG 600 at pH 11.0. Other conditions as in the Experimental section.

present study shows that addition of PEG did help in resolving structural or position isomers that are otherwise difficult to separate. The addition of PEG 600 enables the 2- and 4-carboxybenzaldehyde pair (peaks 6 and 7) to be baseline resolved. PEG also further separated the peaks of *p*-hydroxybenzoic acid (peak 4) and TPA (peak 11), the major component in real samples. Notice that the migration order for isophthalic acid (peak 12) and *o*-phthalic acid (peak 10) is reversed in the presence of PEG. It is possible that the formation of intramolecular hydrogen bonding of *o*-phthalic acid reduces the interaction between *o*-phthalic acid and PEG. The addition of PEG decreased the EOF due to the elevated viscosity. Varying the concentration of PEG from 1 to 5% had no noticeable benefit for enhancing resolution. The optimal concentration of PEG was ~4%.

3.5. Precision and linearity

The precision (expressed in terms of relative standard deviation, % RSD) of the present method for the six selected analytes which were present in phthalate samples is summarized in Table 2. The RSDs for the migration times are typically less than 2%, except for aspirin (2.1%). The RSDs for the peak areas typically are less than 5%, except for *p*-acetamidobenzoic acid (5.2%) and 4-carboxybenzaldehyde (5.7%). The RSDs for the peak heights are a little better for some analytes (less than 4%), but mostly comparable to those for the peak areas. The linearity of the present method was investigated by examining the standard solutions which contain a mixture of the 12 analytes with known concentrations ranging from $2\cdot10^{-5}$ to $4\cdot10^{-4}$ *M*. The data points from calibration curves were subjected to least-squares regression analysis and slope a, intercept b, and correlation coefficient R^2 for the various analyte are listed in Table 2. The linearity of the present method for most analytes is good as suggested by the square of correlation coefficients being better than 0.991.

3.6. Analysis of terephthalic acid samples

The purity of TPA is very important for polyester production. Depending on the manufacturing process, the finished product (after purification) contains various amounts of isomers, side products, and starting materials. Here, representative samples from four local chemical manufacturers were analyzed to demonstrate the practical applicability of the present CE method. Approximately 1 g of phthalic acid powders were weighed, dissolved, treated, and analyzed by the CE method according to the procedure and the method described. Depicted in Fig. 5 are

Table 2
Precision and linearity for the various analytes[a]

Analyte	RSD (%)[b]			Linearity[c]		
	t_m	Peak area	Peak height	$a\times10^3$	$b\times10^3$	R^2
p-Acetamidobenzoic acid	1.4	5.2	1.3	3.2	0.1	0.994
Monomethyl terephthalate	0.9	3.7	1.1	2.1	0.03	0.999
Aspirin	2.1	3.6	3.4	2.1	0.05	0.997
p-Hydroxybenzoic acid	0.2	4.0	4.7	3.2	0.004	0.996
p-Toluic acid	0.3	1.7	1.4	0.4	0.06	0.997
2-Carboxybenzaldehyde	0.5	4.3	5.0	3.4	0.02	0.998
4-Carboxybenzaldehyde	1.1	5.7	3.8	0.4	0.02	0.991
Benzoic acid	0.6	3.0	1.2	2.0	0.05	0.998
Salicylic acid	1.3	3.3	6.8	0.7	0.1	0.995
o-Phthalic acid	0.9	2.8	2.7	5.5	0.2	0.992
TPA	1.0	3.3	1.6	1.2	0.07	0.995
Isophthalic acid	1.0	2.7	4.5	8.4	0.5	0.992

[a] The concentrations of analytes are in the range of 20–400 μ*M* in a set of 12 standard solution for each analyte.
[b] Relative standard deviation (%), $n=5$.
[c] *a*, *b*, and *R* are the slope, intercept, and correlation coefficient of the least-squares linear regression line using peak area data, respectively.

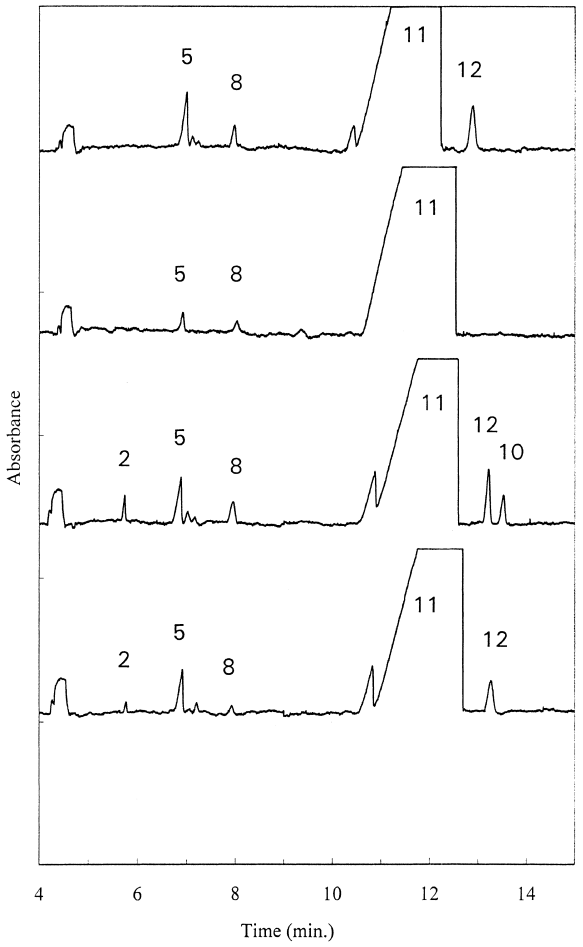

Fig. 5. Electropherograms of four real samples of phthalic acids containing various impurities of aromatic organic acids. From top to bottom: Sample 1 to Sample 4. Other conditions as in Fig. 4.

representative electropherograms of four phthalic acid samples. The samples contain at least 99.9% TPA, thus analyzing its impurity is a challenging problem. Although the three positional isomers could be electrophoretically separated by CE with the aid of co-addition of 4 m*M* α-CD, 8 m*M* β-CD, and 4% PEG 600, the very large TPA peak still obscured the smaller peaks of *o*-phthalic acid and isophthalic acid. To avoid this potential problem, methanol was used to dissolve the TPA samples. The solubility of TPA in methanol is far less than that of *o*-phthalic acid or isophthalic acid [1]. The TPA peak migrated far ahead of the *o*-phthalic acid and isophthalic acid peaks in the presence of β-CD. The contents of the various impurities in the four samples vary, as shown in Table 3. Benzoic acid and *p*-toluic acid are the only two impurities that are present in all four samples. In contrast, it is worth noting that 4-carboxybenzaldehyde, the intermediate occurring from the liquid-phase oxidation of *p*-xylene, was not detected. While sample 2 contained only two impurities, sample 3 had all five impurities present. The concentrations of the impurities present in sample 3 were higher compared to the other samples. Nevertheless, the total amounts of all impurities present were minute, constituting less than 0.002% (w/w). Thus, the present method provides a very sensitive means for checking the purity of real phthalic acid samples.

4. Conclusions

Analysis of 12 benzoate and phthalate derivatives

Table 3
Contents of the aromatic organic acids in real samples[a]

Compound	Sample 1	Sample 2	Sample 3	Sample 4
TPA	0.99923[b]	0.99096[b]	0.99516[b]	0.98557[b]
Monomethyl terephthalate	ND[c]	ND[c]	1.1	0.3
p-Toluic acid	12.4	0.6	11.9	8.4
Benzoic acid	2.4	0.3	5.3	0.2
o-Phthalic acid	ND[c]	ND[c]	1.2	ND[c]
Isophthalic acid	1.6	ND[c]	2.0	1.2
Total contents of impurities	16.4	0.9	21.5	10.1

[a] Concentration in ppm (w/w) except TPA. Calculated by the standard addition method.
[b] Sample weight in g.
[c] ND, not detectable.

and isomers could be accomplished by using a CZE method in the presence of 4 m*M* α-CD, 8 m*M* β-CD, and 4% PEG 600 with 10 m*M* phosphate buffer at pH 11.0. The new method developed has good precision and linearity. It is fast, allowing 12 analytes to be separated in less than 14 min. The sensitivity for detecting impurity in terephthalic acid is in the sub-ppm to ppm range.

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