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Capillary electrophoresis of phosphate ester surfactants with adenosine monophosphate and indirect photometric detection

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Abstract

Capillary electrophoresis (CE) with indirect photometric detection (IPD) is being investigated in this laboratory as a complementary technique to reversed-phase ion chromatography (RPIC) for the separation and identification of mono- and diesters of C_1–C_6 phosphates. Separation efficiency, resolution and analysis time were examined by comparing CE with RPIC using suitable indirect photometric reagents such as adenosine monophosphate (AMP) for CE and naphthalene trisulfonate (NTS) for RPIC. A CE optimization study indicated that a baseline separation of ten mono- and diester C_1–C_6 phosphates was possible in 10 min using 5 m*M* AMP–100 m*M* H_3BO_3 (pH 7.2). The separation of the same mixture with isocratic RPIC required two different elution conditions. A mixture of eight mono- and diester C_1–C_4 phosphates required a 5% acetonitrile (ACN)-20 μM NTS mobile phase for separation in 24 min. The remaining two organophosphates, monoethylhexyl and diethylhexyl phosphate, needed 16 min for separation using an 85% ACN–10 μM NTS eluent. The use of diethylenetriamine as an electroosmotic flow suppressor increased the migration time but significantly improved the CE resolution of both aliphatic and aromatic ethoxylated phosphates. The results show that CE is a useful technique for the fingerprint analysis of commercial organophosphate surfactants such as branched- and straight-chain phosphates and polyphosphates.

Keywords: Indirect detection; Phosphate esters; Adenosine monophosphate; Surfactants; Organophosphate surfactants

1. Introduction

Phosphorus-containing surfactants, namely mono- and diesters of aliphatic organophosphates and aliphatic ethoxylated polyphosphates, are water-soluble surface-active materials. These surfactants are used in a wide variety of applications, including detergents [1], medicinal research [2], nuclear and fuel process industries [3] and complexation chemistry involving selective ion-pair extraction of transition metals, uranium and other rare earths [4,5]. The analysis of waste and treated water for phosphates is required especially because environmental legislation in many parts of the world has limited the use of phosphate detergents.

Long-chain phosphate esters are usually derivatized and then determined by HPLC [6], whereas short-chain esters are separated directly by ion chromatography (IC) and detected by

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suppressed conductivity detection [3]. Short-chain alkyl and dialkyl phosphates such as monomethyl, monoethyl, dimethyl and diethyl phosphate (MMP, MEP, DMP and DEP, respectively) are weakly retained on an ion-exchange column. Analysis of real samples using IC for short-chain phosphonates or phosphates requires sample pretreatment [7] owing to the interference of inorganic anions such as chloride and fluoride, which usually elute in the same chromatographic region [8,9]. Capillary electrophoresis (CE) with a positive polarity configuration should eliminate this problem, since high-mobility chloride and fluoride anions either escape detection or migrate very slowly (even after MMP) towards the detector. Velasquez et al. [2] have published a capillary isotachophoresis method for the selective separation of mono- and diethylhexyl phosphate (MEHP and DEHP, respectively). The importance of DEHP relates to its use in hollow-fiber liquid membranes for the removal of histamine from the blood of patients with acute hepatic failure.

Recently, short-chain C_1–C_4 phosphonates have been separated by CE with direct or indirect UV detection. The methodology of direct detection at 214 nm utilizes the in situ complexation ability of phosphonates with borate that acts as both the buffer and the derivatization reagent [10]. CE with indirect photometric detection (IPD) at 200 nm employed phenylphosphonic acid as the electrolyte and detection reagent [11]. The separation of water-soluble myoinositol phosphates with IPD under aqueous CE conditions using a chromate or phthalate electrolyte has been demonstrated [12]. Nevertheless, the CE of mono- and diesters of phosphates has not been investigated.

We recently introduced ribonucleotides as IPD reagents for the CE separation of multi-charged inorganic polyphosphates and organic polyphosphonates [13]. These reagents are found to have a superior separation selectivity to naphthalene di- and trisulfonates commonly used for the CE of small inorganic anions [14]. Our detailed investigations revealed that adenosine monophosphate (AMP), because of its lower mobility, high molar absorptivity and dynamic reserve, is the best among the ribonucleotide class.

The objective of this paper is to report the utility of AMP for CE with IPD of mono- and diesters of aliphatic organophosphates. A comparison to mixed-mode reversed-phase ion chromatography (RPIC) is made in some instances. Application of the developed CE method to the sample analysis of polymeric ethoxylated phosphates and alkyl polyphosphates is also demonstrated. The structures of these classes of organophosphates are shown in Fig. 1.

2. Experimental

2.1. Reversed-phase ion chromatographic (RPIC) system

The RPIC system consisted of a Model 510 HPLC pump, a Model U6K injector with a 20-μl loop and a Model 490 UV–Vis variable-wavelength absorbance detector, all from Waters (Milford, MA, USA). A Model LP-21 Lo-Pulse damper from Scientific Systems (State College, PA, USA) was connected between the pump and the injector. Separations of surfactants were performed on either of two mixed-mode RPIC columns. A RP/Phenyl anion-exchange silica column (250 × 4.6 mm I.D.) from Alltech (Deerfield, IL, USA) was used to separate mono- and diester C_1–C_4 phosphates whereas a polymeric mixed-mode PRP X-100 anion-exchange column (150 × 4.1 mm I.D.) from Hamilton (Reno, NV, USA) was used for separations of MEHP and DEHP. Separations of ethoxylated phosphates and alkyl polyphosphates were also carried out on the PRP column (see Results and Discussion for details).

2.2. Capillary electrophoresis (CE) system

An Applied Biosystems (Foster City, CA, USA) Model 270A CE instrument with a Macintosh SE + computer equipped with a Rainin (Woburn, MA, USA) Mac Integrator data acquisition system was used. Separations were performed at 30°C on a conventional fused-silica capillary column (75 cm × 50 μm I.D. × 320 μm O.D.) from Applied Biosystems, with various effective lengths (L_d) ranging from 45 to 50 cm.

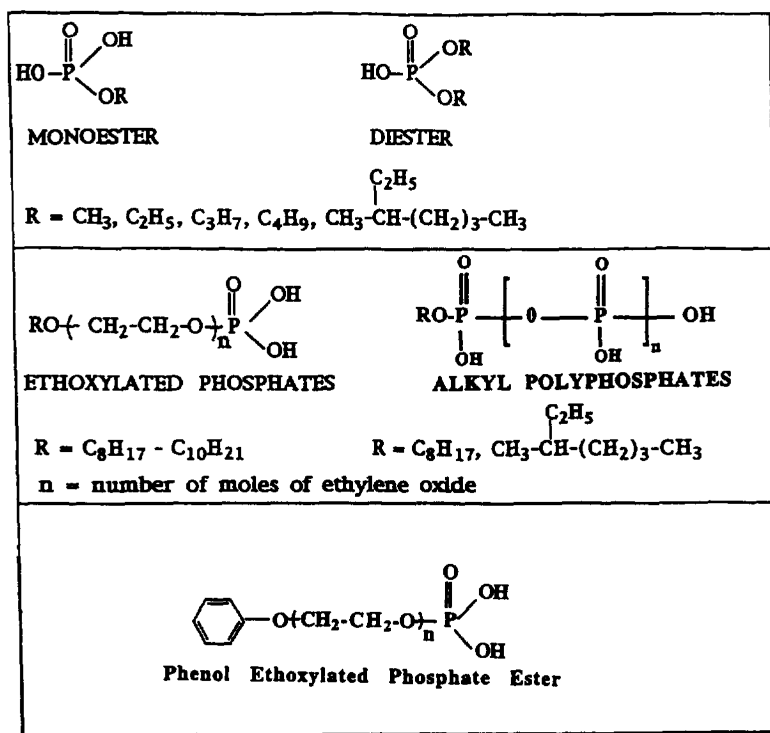

Fig. 1. Structures of various classes of phosphate ester surfactants.

The leads on the Rainin interface module were switched for IPD to obtain positive peaks. Data were collected at 20 points/s. The detector time constant was set to 0.5 s.

2.3. Reagents and samples

The monosodium salt of adenosine monophosphate (AMP) of 99% purity was purchased from Sigma (St. Louis, MO, USA), the monosodium salt of naphthalene monosulfonate (NMS) of 99.5% purity from Eastman Kodak (Rochester, NY, USA), the trisodium salt of naphthalenetrisulfonate (NTS) of 97% purity and the mono- and diesters of phosphates, such as MMP, DMP, MEP, DEP, mono- and diisopropyl phosphate (MIP, DIP), mono- and dibutyl phosphate (MBP, DBP) and mono- and diethylhexyl phosphate (MEHP, DEHP), from American Tokyo Kasei (Portland, OR, USA) and ethylhexyl and capryl polyphosphate and phosphorylated castor oil (formulations unknown) from Chem Service (West Chester, PA, USA). Diethylenetriamine (DETA) of technical grade, boric acid (99.5%) and NaOH were obtained from Fisher Scientific (Fair Lawn, NJ, USA). The ethoxylated (C_8–C_{10}) phosphates and phenol ethoxylated phosphate surfactants were kindly donated by Rhone-Poulenc (Cranbury, NJ, USA).

2.4. Procedures

Eluents for RPIC were prepared from a 50 m*M* stock solution of NTS and then diluted with the desired volume of acetonitrile (ACN)–water before filtering through 0.2-μm membrane filters purchased from Alltech. A routine column equilibration with the desired mobile phase (1.5 ml/min for at least 1 h) was performed before chromatography was started.

The CE run electrolytes were diluted tenfold from a stock solution of 50 m*M* AMP and 1000 m*M* H_3BO_3. The natural pH of an aqueous solution of 5 m*M* AMP–100 m*M* H_3BO_3 was found to be about 5.4 (before adding methanol). The pH of the electrolyte was varied as specified

in the figure captions, being adjusted with 1 m*M* NaOH. All of the final operating buffers were filtered using 0.2-μm Acrodisc syringe filters from Gelman Science (Ann Arbor, MI, USA) by creating a vacuum inside the syringe. Prior to first use, a new capillary was subjected to a standard wash cycle for 6 h at 60°C with 1 *M* NaOH. As a daily routine procedure, the capillary was flushed with 1 *M* NaOH for 15 min and then equilibrated with water for 5 min and the run electrolyte for 10 min before any sample injections. The separation was initiated by applying a voltage of +30 kV between the two capillary ends immersed in reservoirs containing the run electrolyte. Between injections, the capillary was flushed with triply deionized water, 1 *M* NaOH and triply deionized water again for 2 min each. The capillary was finally filled with the run electrolyte for 3 min. This procedure resulted in improved peak shapes and a good migration time reproducibility range of 1–1.7% (R.S.D.). In IPD, an off-scale peak is usually observed before anion but after cation migration (with a positive polarity CE configuration), due to neutral species or water molecules, and has been used before as a measure of electroosmotic flow (EOF) [15].

3. Results and discussion

3.1. CE separation of mono- and diesters of phosphates

With the CE instrument set to the positive polarity configuration, the capillary injection side is the anode and the detection end is the cathode. Mono- and diesters of phosphates, because of their low mobility (based on charge-to-size ratio), would be expected to reach the detection window by the EOF, which exceeds the electrophoretic migration (e_p) of the individual anions. Fig. 2 shows that the pH of the AMP–

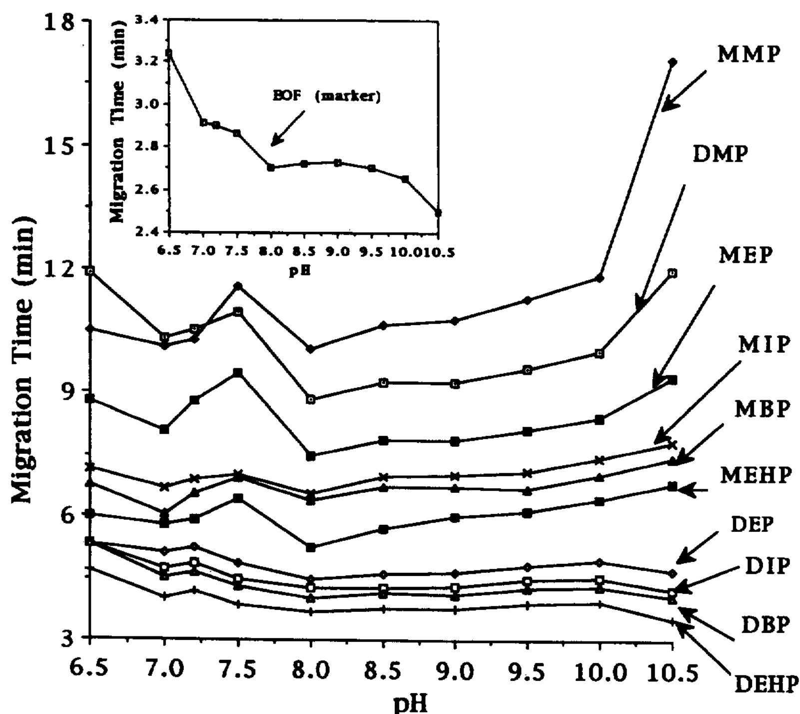

Fig. 2. Effect of electrolyte pH on the separation of ten mono- and diester phosphates. The inset shows a plot of the migration time for the neutral marker as a function of pH. Electrolyte, 5 m*M* AMP–100 m*M* H_3BO_3. Vacuum injection for 2 s (100 mg/l) for all analyte anions. Separation voltage, +30 kV; current, 8–70 μA; IPD at 259 nm.

H_3BO_3 electrolyte affected the migration time of mono- and dialkyl phosphates significantly. The bulk EOF and the e_p of phosphate esters depend on the electrolyte pH, which influences the charge on the mono- and dialkyl-phosphate species. As expected, the order of migration within a class is generally inversely related to the number of CH_2 groups. DMP elutes later than the other diesters owing to its small size and greater tendency to move towards the anode. Diesters (DMP, DEP, DIP, DBP and DEHP) with pK_a values ranging from 1.5 to 2.5 exist predominantly as monoanions in solution at the experimental pH values of 6.5–10.5 studied. The pK_{a_2} values of the monoesters (MMP, MEP, MIP, MBP and MEHP) varied from 6.5 to 7.5 and therefore these compounds should exist as dianions only at high pH values [16]. Initially, as the pH is increased from 6.5 to 7.0 the migration times decrease because of the increase in EOF. However, between pH 7.0 and 7.5, encompassing the pK_{a_2} values of the monoesters, the mobility of the dianion form of the monoalkyl phosphates increases towards the anode. This results in an increase in their migration time towards the cathode. At pH 7.5 MMP, because of its increased charge, is retained longer than DMP. A sudden drop in migration time between pH 7.5 and 8.0 for all phosphorylated esters is probably due to an increase in EOF (i.e., a sudden decrease in migration time of the EOF marker peak) which might be larger than the e_p velocities. Between pH 8.0 and 10.0, the monoesters show a general trend of increasing migration time (due to increased ionization). The diesters show only a slight variation in migration time, which is consistent with the migration time of this EOF marker, which remains steady in this pH range (see Fig. 2, inset). Above pH 10.0, the predominance of doubly charged monoester species and the increase in EOF explain the increase and decrease in the migration times of mono- and diesters, respectively. The only unexpected migration time increase can be seen with DMP. It can be noted that the diesters (DEP, DIP and DBP) are better separated at pH 7.2 than at pH 6.5. Further, at lower pH values (≤ 6.5), MEHP overlaps with an AMP system peak (electropherogram not shown).

Fig. 3a shows the separation in 10 min of a standard mixture of mono- and diesters using AMP at pH 7.2. The peaks are narrow and well shaped, indicating a good mobility match [15] between AMP and the analytes. Encouraged by

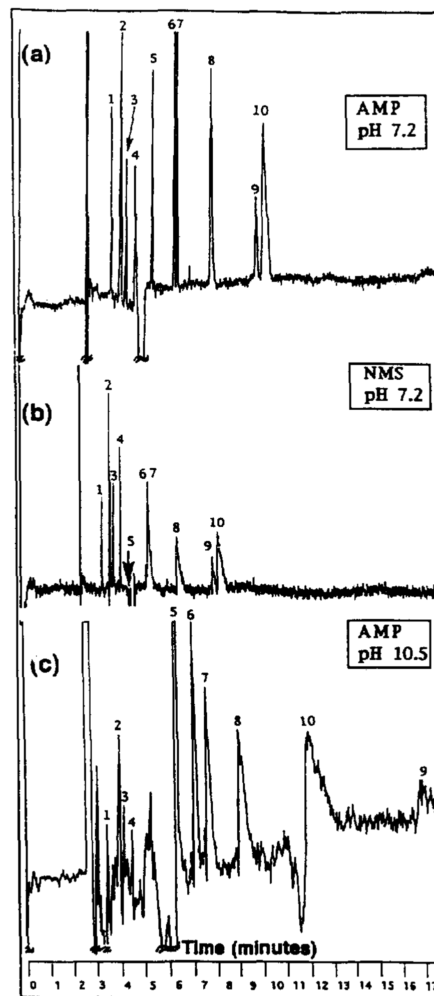

Fig. 3. Comparison of (a) AMP (pH 7.2), (b) NMS (pH 7.2) and (c) AMP (pH 10.5) electrolytes for the separation of a standard mixture of ten mono- and diester phosphates. All electrolytes contained 100 mM H_3BO_3. Peaks: 100 mg/l each of 1 = DEHP; 2 = DBP; 3 = DIP; 4 = DEP; 5 = MEHP; 6 = MBP; 7 = MIP; 8 = MEP; 9 = MMP; 10 = DMP. Vacuum injection for 2 s, +30 kV applied for separation, 8 and 70 μA at pH 7.2 and 10.5 with AMP and 9 μA at pH 7.2 with NMS. IPD at 259 nm with AMP and 274 nm with NMS.

our previous study [14] with NMS for the separation and indirect detection of alkyl sulfates and alkanesulfonate surfactants, we decided to compare the performance of NMS for mono- and diesters of phosphates (Fig. 3b). Even though a slightly shorter migration time can be obtained with NMS, peak 5 is lost in the system peak and peaks 6 and 7 are not resolved. The *S/N* and the resolution are improved using the AMP electrolyte. A comparison of Fig. 3a and c shows that although the resolution of some of the esters, such as the MBP–MIP peak pair, is improved at a higher pH value of 10.5, the separation time increases and the *S/N* ratio decreases significantly under such conditions. Therefore, the separation of mono- and diesters is recommended at a lower pH in the neutral range.

3.2. RPIC separation of mono- and diesters of phosphates

The mono- and diesters of phosphate are also separated by mixed-mode RPIC using NTS as the indirect photometric reagent. However, the separation requires two different mobile phase conditions. Fig. 4a represents an optimized chromatogram of a standard mixture of eight mono- and diester (C_1–C_4) phosphates, which required 5% ACN–20 μM NTS with a separation time of 24 min. In general, the order of peak elution is opposite to that found for CE. Two compounds, MIP and DMP, co-eluted. The remaining two esters (MEHP, DEHP), being more lipophilic, needed a different optimized mobile phase composition of 85% ACN–10 μM NTS to be separated in 16 min (Fig. 4b).

3.3. Comparison of CE and RPIC

In Fig. 3a and b, CE shows a higher peak capacity and a shorter analysis time than RPIC (Fig. 4). The most interesting difference between the techniques is selectivity. Using CE, the double-chain diester phosphates are swept rapidly by the EOF and all except DMP are detected first followed by the single-chain monoesters. As expected for RPIC, the phosphorylated esters are eluted with increasing hydrophobicity; all

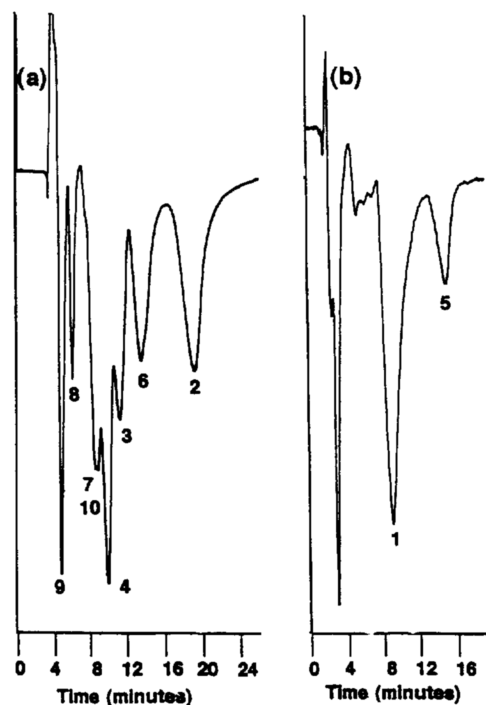

Fig. 4. RPIC for the separation of (a) a mixture of eight mono- and diester C_1–C_4 phosphates and (b) a binary mixture of mono- and diester C_6 phosphates with IPD at 284 nm (0.05 AUFS). Mobile phase: (a) 20 μM NTS–5% ACN with RP/Phenyl anion-exchange column for the separation of C_1–C_4 phosphate esters; (b) 10 μM NTS–85% ACN with a PRP-X100 anion-exchange column for the separation of C_6 phosphate esters. Peaks: (a) 100 mg/l each of 9 = MMP; 8 = MEP; 7 = MIP; 10 = DMP; 4 = DEP; 3 = DIP; 6 = MBP; 2 = DBP; and (b) 200 mg/l each of 1 = DEHP; 5 = MEHP.

C_1–C_4 monoesters eluted before their corresponding diesters and the most hydrophobic pair (DEHP–MEHP) eluted last. It was found that the relative migration times (ratio of migration time to the neutral marker) obtained by CE for most of the compounds are in the range 1–4, similar to the relative retention times (ratio of retention time to the injection peak) obtained by RPIC. Exceptions noted were DEHP, MEHP and DBP, which had retention time ratios of about 5–7 for RPIC but only 1–2 for CE. The separation efficiency as measured by the column plate count ranged from 50 to 500 times better for CE than for RPIC for all the mono- and dialkyl phosphates. For example, the plate count

difference is 50× for MMP, 230× for DMP and 520× for MBP. The resolution values between MBP and DBP and between MEHP and DEHP using CE are about fourteen and seven times higher, respectively, than with the RPIC method. An improved resolution by CE for other mono- and dialkyl phosphates pairs was also observed, with only one exception (MMP–DMP).

A summary of concentration and mass limits of detection is presented in Table 1. The mass limit of detection (MLOD) range with CE for organophosphates (0.07–0.21 pmol) obtained in this work is comparable to the previous data published for C_1–C_4 phosphonates (0.2 pmol) [11]. This MLOD range for CE is at least three orders of magnitude lower than that of RPIC (72–955 pmol) for five of the compounds and at least two orders of magnitude better for the other five. This is because the calculated injection volume for CE (48 nl) is much smaller than the injection volume for RPIC (20 μl). However, it was interesting that the concentration limits of detection (CLOD) of the two separation methods are comparable or for some analytes even lower with CE. For instance, the CLOD of relatively longer chain phosphorylated surfactants such as MEHP, DEHP, DIP and DBP by CE (0.5–1.0 mg/l) are about 5–10 times lower than those for RPIC (2.5–10 mg/l). A possible explanation is that the strong hydrophobic/electrostatic attraction of longer chain phosphate esters in particular with the RPIC stationary phase resulted in broad peaks and in turn generated a higher CLOD. This hypothesis is consistent with our previous RPIC comparison of organosulfate and -sulfonate surfactants, which showed better detection limits (as low as 0.25 mg/l) than those for phosphorylated surfactants [17]. Although the detection limits of 0.3–0.9 nmol obtained by RPIC for MEHP and DEHP are higher than those for any other analytes studied in this work, this detection limit range is still very comparable to the 3 nmol obtained by capillary isotachophoresis [2].

3.4. Application to ethoxylated phosphates and alkyl polyphosphates

The RPIC and CE methods were further examined regarding their feasilibty and limitations by application to some common water-soluble ethoxylated phosphates and alkyl polyphosphates (see Fig. 1 for structures). An RPIC separation of 0.2% (w/v) of Rhodofac RA-600 polymer of average molecular mass 500 was performed on the PRP-X100 anion-exchange column using 10 μM NTS with 85% acetonitrile in about 10 min. This sample has been formu-

Table 1
Summary of aliphatic organophosphates detection limits using IPD for both CE and RPIC

Analyte	Concentration LOD (mg/l)[a]		Mass LOD (pmol)[a]	
	CE[b]	RPIC[c]	CE[b]	RPIC[c]
MEHP	0.5	10	0.12	311
DEHP	0.5	5.0	0.07	955
MBP	1.0	2.5	0.19	324
DBP	0.5	2.5	0.11	238
MIP	1.0	0.5	0.21	72
DIP	0.5	2.5	0.13	274
MEP	0.5	0.5	0.19	79
DEP	0.5	1.0	0.16	130
MMP	0.5	0.5	0.20	89
DMP	0.5	0.5	0.19	79

[a] Limits of detection (LOD) expressed as $S/N = 3$.
[b] CE conditions: 5 mM AMP–100 mM H_3BO_3 (pH 7.10); vacuum injection for 16 s for all mono- and diesters except for MBP and MIP, for which vacuum injection for 10 s was used to keep these two peaks resolved.
[c] RPIC conditions: see Fig. 4, 20-μl injection, 2 ml/min.

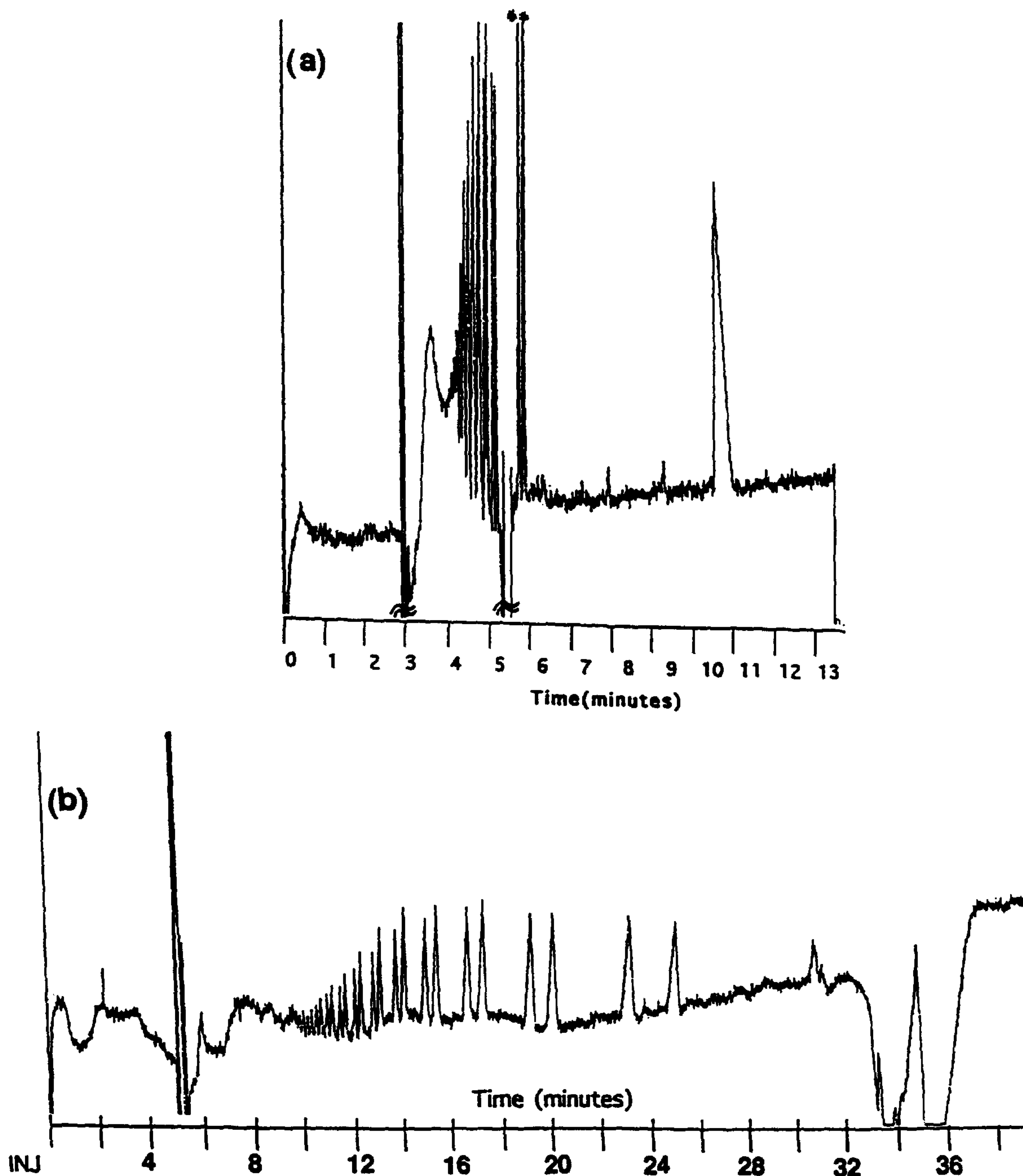

Fig. 5. Comparison of CE in the (a) absence and (b) presence of DETA in the electrolyte for the separation of C_8–C_{10} ethoxylated phosphates. Electrolytes: (a) 5 m*M* AMP–100 m*M* H_3BO_3, adjusted to pH 7.2, and (b) 5 m*M* AMP–100 m*M* H_3BO_3–1.0 m*M* DETA, adjusted to pH 7.2. Sample, 0.2 g per 100 ml of Rhodfac RA-600. Vacuum injection for 1.0 s. IPD at 259 nm. The electropherogram was actually taken for 75 min (see discussion in the text).

lated by Rhone-Poulenc to contain a 5% (w/w) aqueous solution of C_8–C_{10} alcohol ethoxylated phosphates. Only three peaks, probably representing the homologues, were evident by RPIC. It is clear that CE gave a partial separation of individual ethoxylated oligomers, indicating a fairly complicated mixture (Fig. 5a). In an attempt to improve the resolution of the oligomers further, the buffer modifier diethylenetriamine (DETA) was added. DETA has been successfully used previously in our laboratory as an EOF suppressor in a negative polarity CE configuration for the separation of inorganic anions, organic acids and inorganic polyphosphates [13,14]. Fig. 5b illustrates that the addition of 1 m*M* DETA improves the resolution and selectivity at the expense of a longer migration time. However, only for analytes of lower mobility [marked with asterisks in (a)] is the migration time excessive at about 60–70 min (peaks not shown). The peak at 11 min in Fig. 5a would elute at too long a time to be seen.

It was found that with a positive polarity CE configuration, the EOF decreases from $7.6 \cdot 10^{-4}$ to $3.8 \cdot 10^{-4}$ $cm^2/V \cdot s$ on addition of 1 m*M* DETA. DETA concentrations of 0.1 m*M* (EOF = $6.6 \cdot 10^{-4}$ $cm^2/V \cdot s$) and 0.5 m*M* ($4.6 \cdot 10^{-4}$ $cm^2/V \cdot s$) were found to be useful for some subsequent CE separations. It is conceivable that this dynamic coating of DETA also reduces any hydrogen bonding interaction of polymeric phosphates with the surface of bare silica capillary. However, this approach was not found to be useful for the RPIC work. Even though some CE peak broadening and decrease in sensitivity are observed with the addition of DETA, this decrease in detectability can be compensated by increasing the injection size (by at least twofold) without overloading the CE column. In Fig. 6, the electropherograms for phosphorylated castor oil, ethylhexyl polyphosphate and capryl polyphosphate when DETA was added to the running electrolyte are compared. All the major peaks but not the minor peaks could be resolved by CE in the absence of DETA in about half the time (data not shown). Again, RPIC of the same type of surfactants gave only a single peak or two partially resolved peaks for each sample.

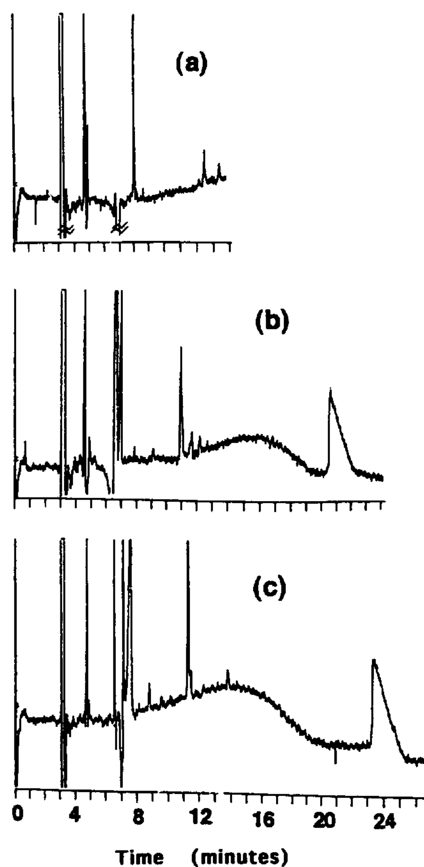

Fig. 6. Electropherograms of (a) phosphorylated castor oil, (b) ethylhexyl polyphosphate and (c) capryl polyphosphate. All samples 100 mg/l. Vacuum injection for 7, 12 and 7 s, respectively. Electrolyte: 5 m*M* AMP–100 m*M* H_3BO_3–0.1 mM DETA, adjusted to pH 7.2. IPD at 259 nm. The off-scale peak at 3–4 min is a water peak and can be considered as the EOF marker.

Fig. 7 shows an electropherogram with direct photometric detection at 220 nm of a phenol ethoxylated phosphate ester of average molecular mass 600. Without DETA, all the sample components eluted quickly in 5 min with only two major peaks clearly resolved and several shoulder peaks around the second peak (electropherogram not shown). The resolution of these surfactants was markedly improved by the addition of DETA to the electrolyte, although the separation time was about six times longer. The cluster of peaks appearing at about 6 min can be resolved more clearly by computer expan-

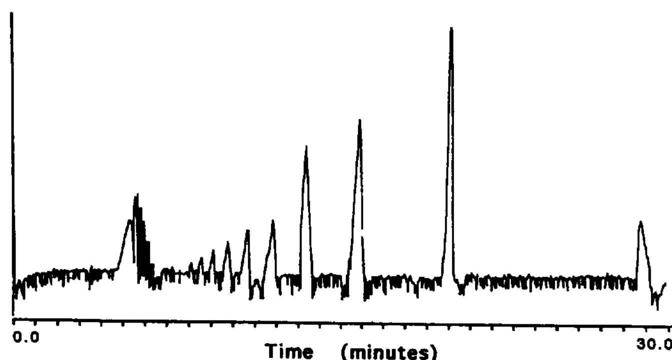

Fig. 7. Electropherogram of a phenol ethoxylated (*n* in Fig. 1 unknown) phosphate (0.1 g per 100 ml). Electrolyte conditions as in Fig. 6 except AMP was omitted and 0.5 m*M* DETA used. Vacuum injection for 1.0 s. Direct UV detection at 220 nm.

sion of the time axis in that region. All of these electropherograms suggest that CE is a useful technique for fingerprinting and molecular mass distribution of various commercial organophosphate samples.

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