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Oxidative and reductive amperometric detection of phenolic and nitroaromatic compounds in packed capillary column supercritical fluid chromatography

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Abstract

Oxidative and reductive amperometric detection of phenols, nitroaromatic compounds and a cyclic ketone is demonstrated in packed capillary column supercritical fluid chromatography (SFC). The detection was performed in combination with isobaric, as well as, pressure programmed SFC separations, using a 25 μm platinum electrode and carbon dioxide modified with a low concentration (1–5%) of methanol as the mobile phase. A relative standard deviation in the peak height of ca. 5% ($n=10$) was found for ng injections of analyte in combination with both oxidative and reductive detection. The detection limit was determined to be 250 pg for oxidative detection of 2,6-dimethylphenol and 100 pg for reductive detection of 1,3-dinitrobenzene. © 1997 Elsevier Science B.V.

Keywords: Detection, SFC; Amperometric detection; Phenolic compounds; Nitroaromatic compounds

1. Introduction

Carbon dioxide is the most frequently employed mobile phase in open tubular column supercritical fluid chromatography (SFC). Separation of polar solutes in combination with the use of packed columns, however, generally requires the addition of a polar modifier to the carbon dioxide to deactivate remaining silanol groups on the packing material [1–3]. The addition of a modifier may also be necessary to increase the solvating power of the mobile phase [4], thus making SFC useful for more hydrophilic solutes also. Methanol is, due to the well-known phase behaviour of carbon dioxide–methanol mixtures [5], a popular mobile phase modifier in SFC. The utilisation of methanol and modifiers other than water [6,7], formic acid [7,8] and formamide [8], however, rules out the use of flame ionisation detection. UV absorbance [9] or light scattering detection [10,11] is therefore generally employed in SFC separations that require modified carbon dioxide as the mobile phase.

Amperometric detection is used frequently for sensitive and selective detection in liquid chromatography and capillary electrophoresis [12–16]. It was recently demonstrated that amperometric detection can be performed at a bare platinum electrode in carbon dioxide modified with only small amounts of water [17], acetonitrile or methanol [18]. Since such detection can in fact provide detection limits in the order of pg when using packed capillary column SFC, electrochemical detection may become a sensitive alternative to, or complement, both UV and light scattering detectors. Electrochemical detection in SFC has previously been based on the use of microelectrode assemblies covered with a conducting

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layer, e.g. a molten salt [19] or a polymer [20–25], or bare microelectrodes in combination with the addition of both mobile phase modifiers and supporting electrolytes [26–28]. While the use of polymer-coated electrodes seems to be incompatible with amperometric detection [25], the use of supporting electrolytes is undesirable, as the addition of salts may limit the stability of the chromatographic system.

In this study, oxidative and reductive amperometric detection at a bare platinum microelectrode is investigated for use in packed capillary column SFC with methanol-modified carbon dioxide as the mobile phase. The versatility of the amperometric detector is demonstrated by the detection of phenol, 2-ethylphenol, 2,6-dimethylphenol, 2-chlorophenol, 4-*tert.*-butylphenol, 1,3-dinitrobenzene, nitrobenzene, 2,6-dinitrotoluene, 2,4-dinitrotoluene and 1,4-naphthoquinone. The detection limits, as well as the reproducibility and linearity of the detector response are discussed.

2. Experimental

2.1. Chemicals

SFC grade carbon dioxide was obtained from Alfax (L'Air Liquide, Paris, France). The mobile phase modifier, methanol (Merck, Darmstadt, Germany), was ultrasonicated for 30 min prior to use. Phenol (Merck), 2-ethylphenol (Aldrich, Steinheim, Germany), 2,6-dimethylphenol (Aldrich), 2-chlorophenol (Fluka, Buchs, Switzerland), 4-*tert.*-butylphenol (Aldrich), 1,3-dinitrobenzene (Riedel de Haën, Hannover, Germany), nitrobenzene (May and Baker, Dagenham, UK), isophorone (Aldrich), 2,6-dinitrotoluene (Aldrich), 1,4-naphthoquinone (Aldrich) and 2,4-dinitrotoluene (Aldrich) were all used as received. A solution of the nitroaromatic compounds and cyclic ketones (EPA standard 8270), at a concentration of 2000 μg ml^{-1}, was obtained from Supelco (Bellefonte, PA, USA). All other chemicals and solvents were of analytical grade.

2.2. Apparatus

The experiments were performed with a 600 SFC system (Dionex, Sunnyvale, CA, USA). Additions of modifier were accomplished by the use of a previously described system [7,18,29], including a pneumatic amplifier (Brigham Young University, Provo, UT, USA) connected to a helium-actuated high pressure prime/purge valve (Valco Instruments, Houston, TX, USA). This valve was controlled using a digital valve sequence programmer (Valco), modified to produce shorter open/close times (i.e. 3.9 to 23.2 ms), and a digital valve interface (Valco). The prime/purge valve was operated using helium at a pressure of 100 p.s.i. A 15-cm piece of 21 μm I.D. (91 μm O.D.) fused-silica tubing (Polymicro Technologies, Phoenix, AZ, USA) was used between the pneumatic amplifier and the prime/purge valve. A mixing device, consisting of a 10-cm stainless steel capillary (1 mm I.D.) filled with glass beads (150 μm diameter), was mounted between the prime/purge valve and the injector, using a 60-cm piece of 50 μm I.D. fused-silica tubing (Polymicro Technologies). The time it took to achieve a stable modifier addition was checked using the on-line UV detector and was typically 15–20 min after the addition had been started. Time split injections were made with a C14W high pressure four-port valve (Valco) equipped with a 0.2-μl sample loop. An injection time of 1 s was employed, resulting in the injection of about 60% of the loop volume. The separations were carried out using 40 cm×200 μm I.D. packed capillary columns, packed with 5 μm Diol S5 particles (YMC, Schermbeck/Weselerwald, Germany). The columns were packed with supercritical carbon dioxide as the packing material carrier [30]. A μPeak monitor (Pharmacia, Uppsala, Sweden) with 400 μm diameter optical fibres (Polymicro Technologies) and a locally built detector cell [31] was used for UV absorbance detection. The detection wavelength was 230 nm.

2.3. Detection system

The two-electrode assembly [17,18,32] was based on a working electrode made from a 25-μm platinum wire coated with a 5-μm layer of polyester (Goodfellow, Cambridge, UK). Epoxy resin (Epoxy Technology, Billerica, MA, USA) was used to seal the platinum wire into a 6-cm, 100 μm I.D. (200 μm O.D.) stainless steel capillary (Goodfellow), which

served as a combined counter and quasireference electrode (QRE). The electrodes were polished on polishing cloths covered with 30, 12 and 3 μm particles (Moyco Industries, Montgomeryville, PA, USA). Fig. 1 shows a scanning electron micrograph of a polished microelectrode assembly. All electrodes were initially cycled in 0.05 *M* H_2SO_4 between −500 and 1700 mV at 50 mV s^{-1}. The shape of the cyclic voltammograms was used to check the function of the electrodes prior to their use in the SFC system. The equilibration time for the microelectrode in the SFC system was found to depend on whether a new or an old electrode was used. A new electrode required up to 1 h to reach a stable background, while an old electrode typically gave a stable background signal within 15 min.

The microelectrode assembly was mounted directly after the UV detector, in a methyl-deactivated piece (250 μm I.D., 400 μm O.D.) of fused-silica (Chrompack, Nacka, Sweden). A 3–4 mm section of the polyimide coating was burned off the fused-silica capillary to provide a window for UV detection. The distance between the column end and the UV detector was 4 cm, while the distance between the point of UV detection and the electrode was ca. 2 cm. Both the UV detector and the electrochemical detector were placed within the chromatographic oven, prior to the 2 m×15 μm I.D. linear fused-silica restrictor (Polymicro Technologies). The restrictor tip was heated to 100°C within a flame ionization detection (FID) heating block. A BAS LC-4B amperometric detector (West Lafayette, IN, USA) was

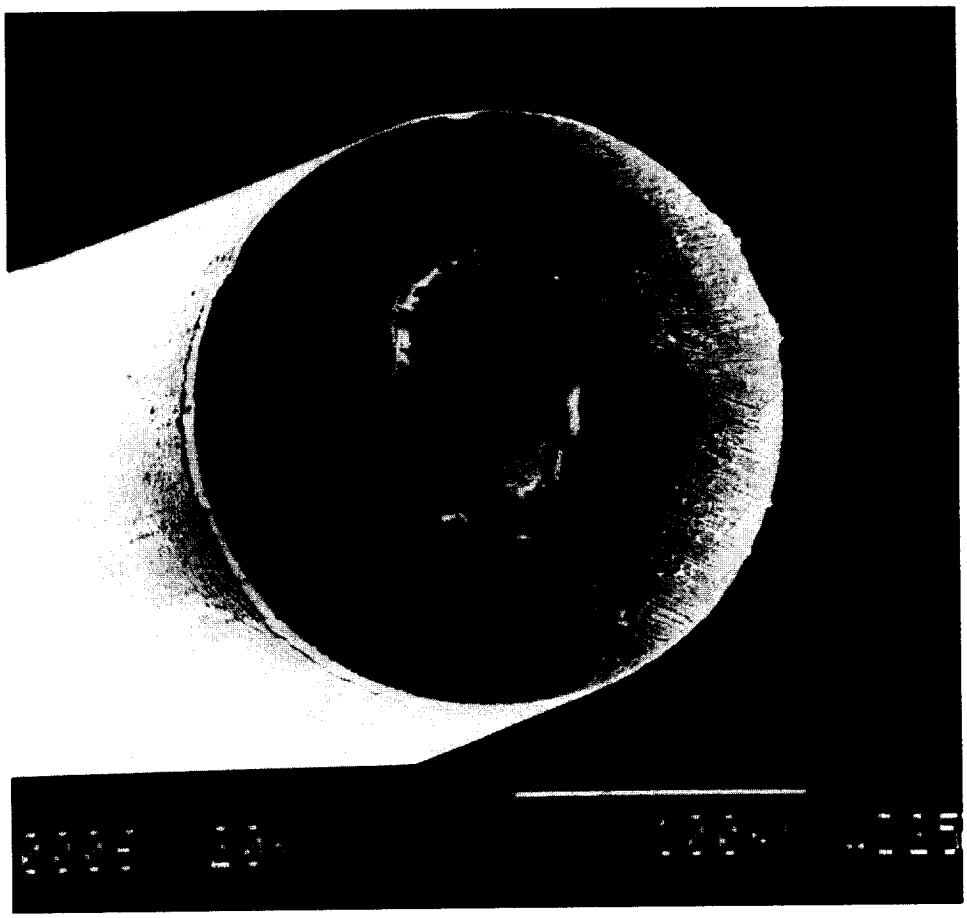

Fig. 1. Scanning electron micrograph of the microelectrode assembly. For details, see the text.

used to control the potential and to measure the currents.

2.4. Data handling

Data from the UV detector was stored on a Victor V286P laptop computer and was then transferred to Microsoft Excel (Microsoft, Redmond, WA, USA). Chromatograms from the electrochemical detector were recorded with a Chrom Jet integrator (Spectra-Physics, Houston, TX, USA). The chromatograms were transferred to a Victor V286P laptop computer and further decoded with a Microsoft Quick BASIC (Microsoft) routine that was written in-house. The peak height measurements were done with Igor (Wave Metrics, Lake Oswego, OR, USA), a program for general graphing and data analysis, using a Macintosh IIci computer.

All injected amounts were calculated from the sample concentration and the loop volume. The amounts were corrected for the fact that only 60% of the loop volume was injected.

To minimise the risk of the formation of a two-phase fluid, a computer program, Physical Property Data Service (PPDS2) (NEL, Glasgow, UK), was used to calculate critical parameters and phase envelopes for different amounts of methanol in carbon dioxide. This program was also used to calculate the density of the mobile phase. In the calculations, which were based on the Peng-Robinson equation [33], the binary interaction parameter for CO_2–MeOH was set to 0.022.

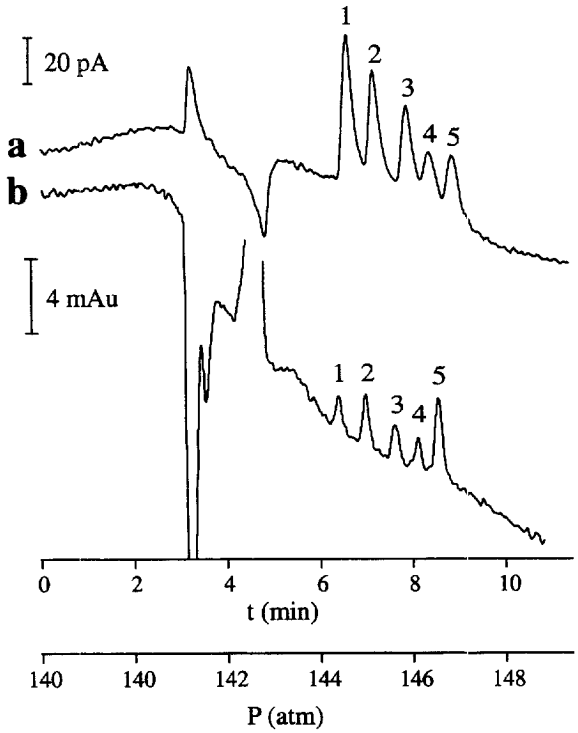

Fig. 2. Pressure programmed SFC separations with (a) amperometric detection and (b) UV absorbance detection. Peak identity, 1=2,6-dimethylphenol, 2=2-chlorophenol, 3=2-ethylphenol, 4=phenol and 5=4-tertbutylphenol. Conditions: Injected amounts in (a), 1=5 ng, 2=12 ng, 3=6 ng, 4=5 ng and 5=6 ng and in (b), 1=25 ng, 2=60 ng, 3=30 ng, 4=27 ng and 5=32 ng. Detection potential, +0.85 V vs. QRE; detection wavelength, 230 nm; mobile phase modifier, 4.6% methanol; temperature, 80°C; pressure program, 140 atm for 2 min, followed by 2 atm min^{-1}.

3. Results and discussion

3.1. Oxidative amperometric detection of phenols

Packed column SFC with modified carbon dioxide as the mobile phase, enables rapid and efficient separations of phenols [34]. There is thus a need for sensitive detection methods that are compatible with modified carbon dioxide [34]. Fig. 2 shows pressure programmed separations of five phenols with amperometric and UV absorbance detection, respectively. It is clearly seen that amperometric detection may be an alternative to UV absorbance detection in this case, especially as the UV chromatogram in Fig. 2b was recorded for a five times higher concentration than that used together with the amperometric detection. Despite the higher concentration employed for UV detection, the signal-to-noise ratios were found to be between 20 and 40 for the amperometric detector compared to between 4 and 10 for the UV detector. The positive and negative peaks seen at the beginning of all chromatograms are system peaks due to the hexane used as the injection solvent. The drift in the background signal, observed with the UV detector (see Fig. 2b), reflects the variations in the refractive index of the supercritical mobile phase [9] during the pressure program. Due to these changes in the refractive index, the use of UV detection in combination with pressure programmed separations

generally requires either a background subtraction or detection at temperatures that ensure the presence of a liquid phase [9]. Cooling of the detector cell can, however, cause phase separations and/or precipitation of analytes [5]. As seen in Fig. 2a, the drift in the baseline is significantly less pronounced if electrochemical detection is employed. A further advantage with electrochemical detection is that such detectors, unlike e.g. UV detectors, whose sensitivity is dependent on the length of the light path, generally can be miniaturized with preserved performance. The larger peak tailing observed in the amperometric detector was most likely due to adsorption on the electrode surface [18]. In accordance with previous results [18], the peak asymmetry was, however, found to be smaller when less than one ng of the analytes was injected. Directed studies towards continuous removal of the reaction product would thus be of interest.

3.2. Reductive detection of nitroaromatic compounds and cyclic ketones

To extend the applicability of amperometric detection in SFC, it is important that both oxidative and reductive detection can be utilised. We have previously shown that reductive detection of azobenzene and 4-nitrodiphenylamine is possible using a bare platinum electrode in carbon dioxide that has been modified with methanol [18]. To our knowledge, reductive amperometric detection has, however, never been used in combination with pressure programmed separations. Fig. 3a shows that reductive detection under such conditions is indeed possible. The simultaneously recorded chromatogram in the UV detector is shown in Fig. 3b. The isophorone peak was not observed in the chromatogram recorded with the amperometric detector. This compound most likely requires the use of a more negative detection potential than the −2 V allowed by the present instrumental set-up. This conclusion is also supported by available literature data [35,36], where the half-wave potentials for isophorone, nitrobenzene and 1,3-dinitrobenzene in acetonitrile and methanol have been reported to be −1.65, −1.15 and −0.91 V vs. SCE, respectively [35,36]. These data clearly show that the reduction of isophorone requires a more negative potential than that required for nitrobenzene and 1,3-dinitrobenzene, and that nitrobenzene should require a more negative detection potential than 1,3-dinitrobenzene. The latter is in good agreement with the experimental findings, as nitrobenzene required a detection potential of ca. −1.2 V vs. the QRE while 1,3-dinitrobenzene could be detected at a potential of −0.85 V. These results also demonstrate that the selectivity of an amperometric detector can be tuned by changing the applied potential.

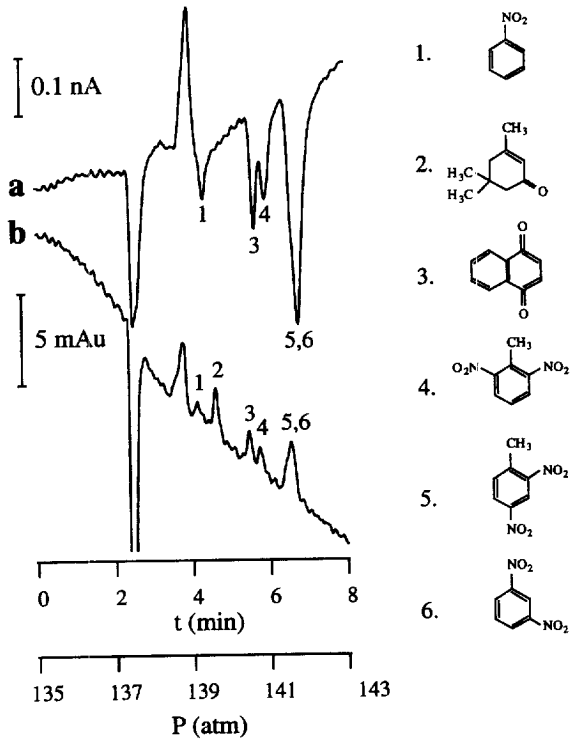

Fig. 3. Pressure programmed SFC separation of a 100-times diluted EPA 8270 standard mixture with (a) amperometric and (b) UV detection. Peak identity, 1=nitrobenzene, 2=isophorone, 3=1,4-naphthoquinone, 4=2,6-dinitrotoluene, 5=2,4-dinitrotoluene and 6=1,3-dinitrobenzene. Conditions: Injected amounts, 2.4 ng of the respective compound; detection potential, −1.85 V vs. QRE; detection wavelength, 230 nm; mobile phase modifier, 2.5% methanol; temperature, 75°C; pressure program, 135 atm and then 2 atm min^{-1}.

The effects of altering the modifier concentration and the mobile phase densities are shown in Fig. 4. As can be seen in this figure, the peak currents for the reduction of 1,3-dinitrobenzene increase with increasing modifier concentration and decreasing

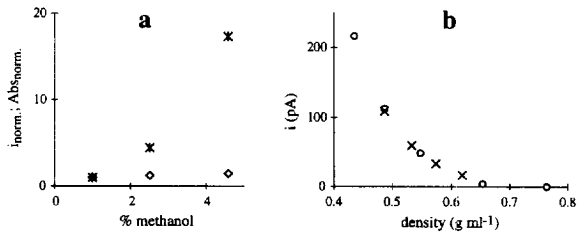

Fig. 4. The influence of (a) the modifier concentration and (b) the mobile phase density on the peak current for the reduction of 3.1 ng of 1,3-dinitrobenzene. The peak heights in the amperometric (*) and the UV detector (◇) in (a) were normalised with respect to the response obtained with 1% methanol as the modifier. Conditions (a): Temperature, 80°C; pressure, 170 atm; detection potential, −1.3 V vs. QRE. Conditions (b): Mobile phase modifier, 2.5% methanol; detection potential, −1.5 V vs. QRE; (○) 180 atm, 50 to 100°C and (×) 80°C, 160 to 210 atm.

mobile phase density. The peak heights in Fig. 4a have been normalised with respect to the peak height obtained with 1% methanol in the mobile phase. The large dependence of the peak current on the modifier concentration, which was also seen for the oxidative detection of phenols, is consistent with that for oxidative detection of ferrocenes [18]. The phenomenon is most likely coupled to an increased solubility of the reaction product in the mobile phase with increasing concentrations of the modifier. The dependence of mobile phase density on the peak currents seen in Fig. 4b, is, in contrast, not yet fully understood. A possible explanation could be an increase in the diffusion coefficients of the analytes with decreasing mobile phase densities [18,37].

3.3. Reproducibility, linearity and detection limit

The reproducibility of the response of the amperometric detector was studied for oxidative and reductive detection in a mobile phase consisting of carbon dioxide modified with methanol. The results of these experiments, which are summarised in Table 1, show that the relative standard deviations (R.S.D.s) in the peak height for ng injections of analyte were about 5% in both the oxidative and reductive mode. These values are in good agreement with those previously reported [18] for oxidative detection of ferrocene in a mobile phase consisting of carbon dioxide modified with acetonitrile. In the latter case [18], the repeatability was, however, most likely limited by factors other than the detection, since similar R.S.D. values were obtained with on-line UV detection. As seen in Table 1 and Fig. 5, showing the first and tenth chromatogram for the oxidative detection of ng amounts of 2,6-dimethylphenol, 2-ethylphenol and phenol, amperometric detection at a bare platinum microelectrode provides reliable results for both oxidative and reductive detection in SFC, with methanol-modified carbon dioxide as the mobile phase. Dressman et al. [25], on the other hand, recently reported the rapid loss of sensitivity when employing amperometric detection in SFC using an electrode assembly covered with a poly(ethylene oxide)-based film.

The linear range and detection limit for the amperometric detector were investigated for both oxidative and reductive detection. The linear range was studied by injecting either 0.3 to 15 ng of

Table 1
Reproducibility of the response of the amperometric detector

	Retention time		Peak height	
	Mean[a] (min)	R.S.D.[a] (%)	Mean[a] (pA)	R.S.D.[a] (%)
Phenol[b]	6.4	0.3	10.2	4.1
2,6-Dimethylphenol[b]	4.8	0.2	24.0	3.1
2-Ethylphenol[b]	5.9	0.3	12.1	4.9
1,3-Dinitrobenzene[c]	4.2	0.2	30.6	5.3
2,4-Dinitrotoluene[d]	6.2	0.3	39.7	4.1

[a] $n=10$.

Conditions: [b] 80°C, 160 atm., 4.6% methanol; detection potential, +0.85 V vs. QRE; injected amount, 6 ng. [c] 80°C, 170 atm, 2.5% methanol; detection potential, −1.0 V vs. QRE; injected amount, 3 ng. [d] 80°C, 50 atm, 2.5% methanol; detection potential, −1.0 V vs. QRE; injected amount 4.6 ng.

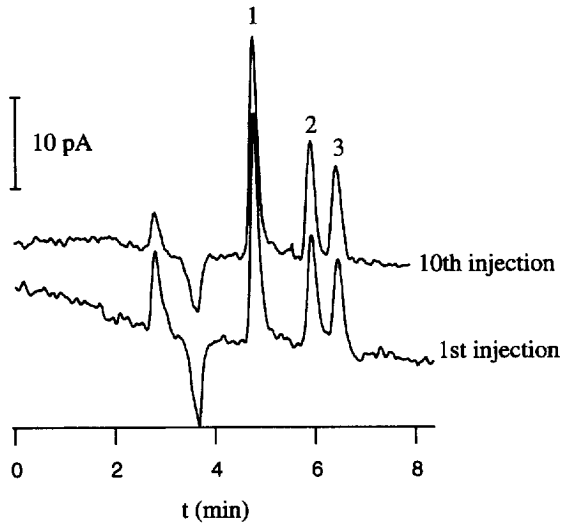

Fig. 5. SFC chromatograms obtained after the first and the tenth injection of 6 ng of (1) 2,6-dimethylphenol, (2) 2-ethylphenol and (3) phenol, respectively. Conditions: Mobile phase modifier, 4.6% methanol; temperature, 80°C; pressure, 160 atm; detection potential, +0.85 V vs. QRE.

2,6-dimethylphenol in a mobile phase containing carbon dioxide modified with 4.6% methanol, or 0.2 to 4.2 ng of 1,3-dinitrobenzene in carbon dioxide modified with 2.5% methanol. The peak height for the oxidation of 2,6-dimethylphenol was found to depend linearly on the injected amount in the range between 0.3 and 3 ng (slope, 7.0±0.5 pA ng^{-1}; intercept, 2.2±3.7 pA; r, 0.990) based on two repeated injections at six concentrations. The corresponding range for the reduction peak for 1,3-dinitrobenzene was between 0.2 and 3.2 ng (slope, 12.1±1.0 pA ng^{-1}; intercept, 5.5±1.9 pA; r, 0.996) for two repeated injections at eight concentrations. The slopes and intercepts within parentheses are given as 95% confidence limits. When more than ca. 4 ng of both compounds were injected, the currents obtained in the oxidative as well as in the reductive mode were too small, indicating a saturation effect, in agreement with previous findings [18].

The detection limit, based on a signal-to-noise ratio of two, was approximately 250 pg for the oxidation of 2,6-dimethylphenol and 100 pg for the reduction of 1,3-dinitrobenzene. These values should be compared with the detection limit of 2 ng previously reported for 2,4-dimethylphenol in packed column SFC with electrochemical detection at a poly-(ethylene oxide)-coated electrode [25]. As can be seen in Fig. 6, which shows two consecutive injections of 730 pg of 2,6-dimethylphenol and 210 pg of 1,3-dinitrobenzene, respectively, good repeatability in the peak current was obtained, even for

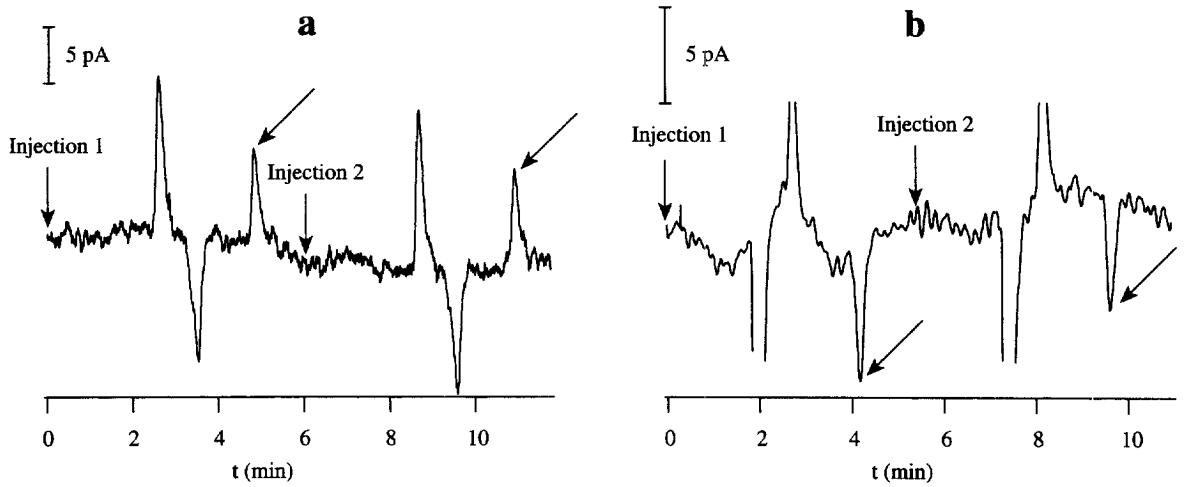

Fig. 6. SFC chromatograms showing two consecutive injections of (a) 700 pg of 2,6-dimethylphenol and (b) 200 pg of 1,3-dinitrobenzene. Conditions: (a): Mobile phase modifier, 4.6% methanol; temperature, 85°C; pressure, 160 atm; detection potential, +0.85 V vs. QRE. Conditions (b): Mobile phase modifier, 2.5% methanol; temperature, 80°C; pressure, 170 atm; detection potential, −1.0 V vs. QRE.

amounts close to the detection limit. The background noise in Fig. 6a–b was approximately 1.3 pA, while the average peak currents were 8.2 and 6.5 pA, respectively.

In conclusion, these results clearly show that amperometric detection at bare microelectrodes provides sensitive and reproducible detection for a variety of compounds separated by SFC using methanol-modified carbon dioxide as the mobile phase.

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