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Measurement of phenols on a loop-supported liquid film by micellar electrokinetic chromatography and direct UV detection

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Abstract

This paper describes a direct measurement of phenolic substances in the gas phase at levels relevant to occupational health by micellar electrokinetic chromatography (MEKC) using direct ultraviolet detection. A small circular loop (2 mm O.D.) is formed with 100 mm Pt-wire at the sampling end of a fused-silica capillary. The capillary tip of the sampling end is present at the center of the loop and in the same plane. A thin film of 0.50 m*M* NaOH is formed on the loop by immersion and withdrawal. The film is in fluid communication with the capillary and acts as an absorber for gaseous phenols. Gas sampling is performed by transferring the film-bearing loop into a chamber through which air is aspirated for a preset time at a preselected flow-rate (typically 1 min at 100 cm^3/min). A part of the film content is then introduced into the capillary by gravity injection and then MEKC is performed. A total of twelve chloro- and nitrophenols were selected for this study. Under the above sampling conditions, limits of detection (LODs) for various phenols range from high-single-digit to low-double-digit ppbv levels. The effect of several critical parameters, such as the composition of the liquid film and the MEKC running electrolyte, sampling period and film evaporation on the collection efficiency, separation efficiency and calibration behaviour are described.

Keywords: Sample preparation; Phenols; Chlorophenols; Nitrophenols

1. Introduction

Gas chromatography (GC) is a commonly used environmental analytical technique for the separation of volatile and semi-volatile analytes [1]. Aside from the United States Environmental Protection Agency (USEPA) methods [2,3], there is a continuing interest in developing improved methods based on GC–mass spectrometry [4,5] and capillary electrophoresis (CE) [5–7].

Substituted phenols such as chlorophenols are of environmental concern because of their toxicity even at low concentrations; several have been designated as priority pollutants by the USEPA [8]. Common sources of some of these phenols are agricultural pesticides, industrial wastes, water supplies and automobile exhausts. Chlorophenols are generally measured by GC [9] and high-performance liquid chromatography (HPLC) [10–12]. Other methods such as liquid–liquid extraction [13], ion-exchange [14], spectrophotometry [15] and colorimetry [16] have also been used for the determination of chlorophenols. These techniques are time consuming and often require derivatization or gradient elution.

CE and MEKC are rapidly growing separation techniques because of their high efficiency and mass sensitivity when compared with GC and HPLC. Both techniques use inexpensive capillaries and produce

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negligible laboratory waste. The separation of chloro- or nitro substituted phenols has been widely studied by several investigators [7], [17–24]. Analysis of phenols has been performed and optimized for CE [17–20] and MEKC conditions [7], [21–24].

Recently, we have reported a technique [25] for the direct measurement of soluble inorganic atmospheric gases such as SO_2 by a suppressed conductometric capillary electrophoresis separation system (SuCCESS) [26,27]. As a sampling interface, a thin film of a liquid was formed on a wire loop around the tip of the analysis capillary for sample collection. The collected sample was injected into the capillary, followed by separation and detection by SuCCESS. In this paper, we show that this principle can be used for direct measurement of organic substances such as gas-phase chloro- and nitrophenols, at levels relevant to occupational health using direct UV-absorption detection.

2. Experimental

2.1. Chemicals and reagents

The substituted phenols (Table 1, includes abbreviations used below) and other chemicals were of analytical grade. 2-NP and 2,6-DNP (Fluka) 2,4-DCP (Eastman) and anhydrous $Na_2B_4O_7$ (Mallinckrodt) were obtained as indicated, all other chemicals were from Aldrich Chemical. Stock solutions (10 000 ppm each) of the phenols were made in methanol. Other solutions are made with distilled deionized water. Stock solutions of NaH_2PO_4, sodium dodecyl sulfate (SDS) (0.5 *M* each) and $Na_2B_4O_7$ (0.1 *M*) were filtered using 0.45-mm pore cellulose–diacetate membrane (Gelman Sciences, Ann Arbor, MI, USA). The running electrolyte was composed of 25, 10 and 10 m*M* borate, phosphate and SDS, respectively, the pH of this solution was measured to be 8.86. A mixture of the borate–phosphate solution was degassed before adding SDS. Liquid-standard analytes were made from their stock solutions by dilution.

2.2. Apparatus and operational protocol

A Model CES-1 instrument (Dionex, Sunnyvale, CA, USA) was used for carrying out electrophoresis. A 75 mm I.D.×365 μm O.D. 65-cm-long fused-silica capillary (Polymicro Technologies, Phoenix, AZ, USA) was used with an applied voltage of +20.0 kV in the constant voltage mode. A new capillary was rinsed with methanol and treated with 1 *M* NaOH overnight. Then the treated capillary was

Table 1
Chloro- and nitrophenols investigated: relevant physiochemical properties

Compound	Abbreviation	pK_a[a]	Melting point (°C)	Vapor generation temperature (°C)	Migration order
Phenol	Ph	9.99	43	30	1
4-Chlorophenol	4-CP	9.37	43	30	2
3-Chlorophenol	3-CP	9.02	33	30	3
3-Nitrophenol	3-NP	8.4	97	nd[c]	4
3,4-Dichlorophenol	3,4-DCP	8.62[b]	68	55	5
2-Chlorophenol	2-CP	8.48	8	30	6
2,4,6-Trichlorophenol	2,4,6-TCP	6.00	64	30	7
2,4,5-Trichlorophenol	2,4,5-TCP	6.0	70	55	8
2,4-Dichlorophenol	2,4-DCP	7.85	45	nd[c]	9
3,5-Dichlorophenol	3,5-DCP	8.25[b]	69	55	10
2,5-Dichlorophenol	2,5-DCP	7.51[b]	58	30	11
2,6-Dichlorophenol	2,6-DCP	6.78	68	30	12
2,4-Dinitrophenol	2,4-DNP	4.09	115	nd[c]	13
4-Nitrophenol	4-NP	7.15	114	nd[c]	14
2-Nitrophenol	2-NP	7.23	45	30	15
2,6-Dinitrophenol	2,6-DNP	3.71	60	55	16

[a] [28].[b] [19].[c] nd, not determined. Because the volatility was too low, gas generation was not conducted.

flushed with the running electrolyte for 20 min. Unless otherwise indicated, electropherograms were obtained at 205 nm by using the direct UV detection mode. The optical window is made by burning off a 0.8 cm portion (55 cm from the injection end) of the polyimide coating. The data were acquired by a personal computer using Dionex ACI computer interface and Dionex AI-450 software.

The construction of the wire loop and the automated gas sampling scheme has been described previously [25]. Briefly, a small circular (2 mm O.D.) loop was formed with 100 mm Pt-wire at the sampling end of the fused-silica capillary; the sampling end being present at the center of the loop. The rotatable turret (of the CES-1 instrument) contained vials of electrolyte, 0.5 m*M* NaOH, air and electrolyte, in sequence. The standard operating procedure consisted of transporting the capillary-loop assembly and the high voltage (HV) electrode into the first vial, pressurizing to flush the capillary with the running electrolyte, lifting the sampling head and dipping into the film-making liquid (second vial), withdrawing it and introducing it into the gas sampling chamber (GSC), see below. The relative heights assured that there was no significant hydrostatic difference between the film-containing capillary head inside the GSC and the detector end of the capillary (dipped into a small chamber, known as 'destination vial', of the CES-1 containing the running electrolyte and the grounding electrode) during sampling. Air was sampled at a rate of 100 cm^3/min immediately after the head sealed itself on the GSC. Following the sampling period, the head was programmed to pick up the third vial and to lift itself to a height of 10 cm and remain in that position for a fixed period of time (20 s) to introduce an aliquot of the film content into the capillary. The volume of the sample injected from the film during a 20 s period with a 10 cm hydrostatic head is estimated to be 28±3 nl. Then the head returned to the electrolyte (fourth vial) and positive HV was applied to begin electrophoresis.

The gas generation arrangement, described before [25], is used here with minor modifications. Briefly, the house-purified dry air was passed through two U-tubes (containing analyte source capsules (ASCs), described below) contained in two water baths (maintained at 30 and 55°C). The analyte-bearing air was diluted with dry air at a T-connector. It was then split into two streams, the main stream proceeding through a water-filled bubbler or vented outside the room. The rest, constituting the sampled flow (typically 100 cm^3/cm), proceeded through the GSC.

The ASCs are constructed by encapsulating approximately 50 mg of a solid analyte between cotton plugs inside 3 cm long, 0.48 cm I.D. fluorinated ethylene–propylene (FEP) copolymer tubes (Zeus, Orangeburg, SC, USA). 2-CP and 3-CP are liquid at room temperature; 1 cm long cotton plug was wetted with 100 μl of their stock solutions and encapsulated in a similar manner. The ASCs were put into U-tubes maintained at 30 or 55°C, as indicated in Table 1. The weight of the ASCs were measured periodically to determine the gravimetric loss rate. Necessary care was taken to avoid health hazards.

The 'source vial' of the CES-1, modified previously [25], is used here as the GSC with a slight modification. In this case, a 8.5 mm I.D. glass tube was installed inside the GSC to reduce the effective volume of the source vial. Relative to the use of a polypropylene tube [25], the use of a glass tube also minimizes the carryover between successive samples of the analyte gas; apparently the organic vapours are much more strongly adsorbed on a polypropylene surface.

2.3. Calibration of vapour phase concentrations

The gas-phase concentration of each analyte was measured by two different methods: (a) measuring the weight loss of the ASCs with time (Fig. 1) and dividing this loss rate by the volumetric flow-rate; and (b) measuring of the mass of an analyte collected by a water-filled bubbler after aspirating a known volume of the gaseous standard through it. The analyte concentration in the bubbler solution was measured by MEKC and quantified with the help of aqueous standards. There was no significant difference between these results.

2.4. Collection efficiency of the film

The volume of the liquid film was determined by measuring the mass of water lost from a small tared water-filled vial upon the insertion and withdrawal of an initially empty wire loop, to be 920±50 nl (n=

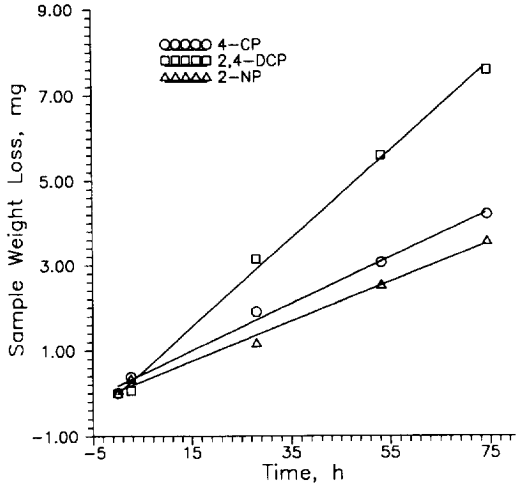

Fig. 1. Weight loss of the analyte source capsules as a function of time. The linear relationship indicates a constant emission rate of the analyte.

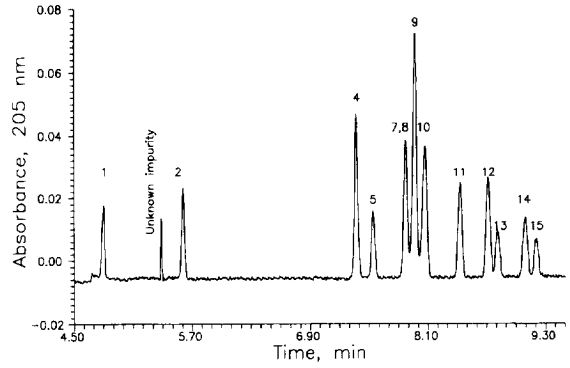

Fig. 2. MEKC separation of 13 phenolic analytes, each at a concentration of 10 mg/l. 20 s injection with a hydrostatic height difference of 10 cm. See text for electrophoretic conditions and Table 1 for peak identities.

10). The efficiency of the film for collecting various analytes was measured at a constant sampling rate. The mass of different analytes collected on the film was computed by comparison with calibration data obtained using aqueous standards. The mass of an analyte passed through the sampling chamber during the sampling period was already known from vapour-phase calibration described above, permitting the evaluation of collection efficiency.

3. Results and discussions

3.1. MEKC separation of phenols and test gases

Separation of this type of compounds by MEKC is not difficult, a typical illustrative separation of aqueous standards is shown in Fig. 2. Generation of measurable concentrations for test purposes in the gas phase, however, requires perceptible volatility. Twelve compounds were chosen on this basis and the test vapours were generated using a source temperature of either 30 or 55°C as indicated in Table 1. A typical MEKC electropherogram of 12 such analyte gases, collected into the film, is shown in Fig. 3. The wavelength for maximum absorption for the various phenolic analytes are obviously not the same. As a compromise, to monitor a large number of compounds with reasonable sensitivity, we chose a monitoring wavelength of 205 nm. It should be noted, however, that with the specific detector we used, in the range of 200–300 nm the baseline noise was the highest at around 205 nm. Therefore, if one is interested in a specific compound, a monitoring wavelength other than 205 nm might provide the best LOD.

Better separation of the analytes was observed under MEKC conditions relative to the use of a borate–phosphate electrolyte without SDS. Addition of 25 m*M* β-cyclodextrin to the running electrolyte provided a different elution order, however, no

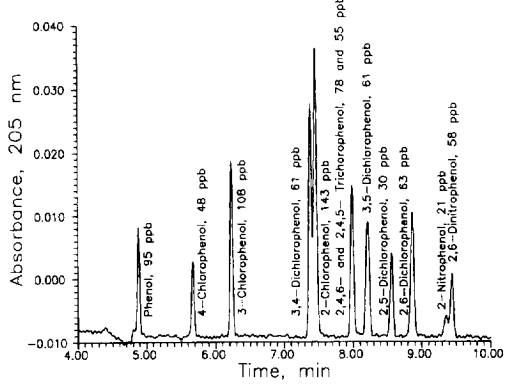

Fig. 3. A typical electropherogram showing collection of 12 analyte gases on an NaOH film and direct MEKC separation.

improvement in separation was observed. Separation of most of the analyte species investigated could be accomplished with the running electrolyte used in this study. Increasing concentrations of the buffer constituents provided better baseline separation but at the cost of longer migration time and larger electrophoretic currents, resulting in deteriorated migration time reproducibility.

3.2. Choice of the liquid-absorber film

Presently, the use of diffusion denuders represents the method of choice in macroscale atmospheric trace gas determinations [29]. This method eliminates the interference from aerosol phase components. This laboratory has reported automated and continuous collection/analysis systems with diffusion denuders for soluble gases [30,31]. Using the same principle, Jiang et al. [32] has monitored trace levels of 2,4,5-TCP. Although, excellent sample collection efficiency was observed, such techniques are time consuming and generally require preconcentration of the collected sample before analysis.

Fig. 4 shows the electropherograms obtained from (a) gases sampled with the MEKC carrier-electrolyte solution as the film; (b) gases sampled with H_2O as the film; and (c) gases sampled with a 0.5 m*M* NaOH film. All the analytes are freely soluble in alcohol and generally less soluble in water. However, a pure alcohol (methanol or ethanol) was not as good an absorber film. Because of their high volatility, the alcohol films evaporated and hence disintegrated only after a few seconds of sampling. A film containing up to 10% methanol did not break for a sampling duration of at least 60 s. However, in this case, the collection efficiency was worse than that with pure water as absorber. The greater evaporative flux of methanol molecules from the film surface (cf., Stefan Flow [33]) is likely responsible for this. Addition of a relatively non-volatile organic modifier, e.g., N,N-dimethylformamide, may have been advantageous but was not investigated.

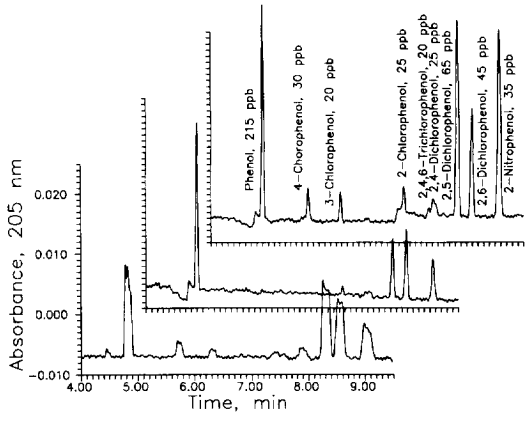

Fig. 4. Dependence of the results upon film composition. Bottom: MEKC carrier electrolyte solution as film; middle: pure water as film; top: 0.5 m*M* NaOH as film.

Phenols are acidic. An alkaline medium, such as the MEKC carrier electrolyte solution serves as an effective and stable absorber film for sampling gaseous phenols. However, due to the high ionic conductance, electrostacking [34] of sample zones cannot occur, resulting in significantly broader peaks. Pure water is a reasonable choice as an absorbing medium. Plate counts ($n=5.54(t_m/w_{1/2})^2$, where n is the plate count of an analyte eluting at time t_m with a peak half width of $w_{1/2}$) were highest with pure water because of optimum electrostacking. With respect to collection efficiency, our experiments indicated that a dilute basic solution, e.g., 0.5 m*M* NaOH, is the best choice as stable absorbing-film medium (Table 2). Because of ionization of the phenols, a basic solution serves as an effective sink. Sufficient electrostacking should also occur as long as the ionic strength of the film solution is substantially less than that of the MEKC electrolyte. Under the experimental conditions, the percent collection efficiency with this absorber is significantly higher (phenol 1.11%, 2,5-DCP 1.35%, 2,6-DCP 0.54% and 2-NP 6.86%) relative to those observed with pure water (0.80, 0.31, 0.27 and 1.37%, respectively, for the four compounds above). Compared to the collection efficiency obtained with diffusion denuders [30–32], the collection efficiency is much smaller, presumably due to the significantly smaller absorber surface area. Extant theory [29] predicts that by reducing the effective diameter of the GSC, collection efficiencies can be further improved. The parameters that affect the collection efficiency, most notably the temperature (which affects the diffusion coefficient of the gas sample), should be controlled. Based on the overall performance, we decided to use the 0.5 m*M* NaOH solution as the film solution for

Table 2
Effect of NaOH concentrations, as film-solution, on relative sensitivity and separation efficiency

	Relative response (absolute plate numbers in thousands)			
	Water	0.25 m*M* NaOH	0.50 m*M* NaOH	1.0 m*M* NaOH
Phenol	77.7±6.4 (300±20)	93.7±6.9 (236±28)	100±6.2 (214±16)	94.7±4.6 (222±13)
2,6-DCP	52.3±3.3 (166±21)	88.6±4.0 (114±4)	96.0±6.2 (142±8)	100±5.7 (113±6)
2-NP	31.7±2.1 (197±1)	92.2±3.7 (102±10)	95.4±5.0 (131±6)	100±5.9 (119±9)

the entire study. The observed plate numbers, however, are slightly less than those observed from a water absorber (Table 2). Besides lower electrostacking efficiency, higher sample loading is partly responsible for the reduced plate counts.

3.3. *Effect of the sampling period.*

Fig. 5 shows the dependence of the signal on the sampling period for dry air (ca. 10% RH) containing phenol, 2,5-DCP, 2,6-DCP and 2-NP at the respective concentrations of 216, 65, 43 and 34 ppbv at 5 different sampling periods ranging from 20–100 s at a constant sampling rate of 100 cm^3/min. Longer sampling periods were impractical with the loop size presently used. Again, the use of a less volatile liquid in the film may be beneficial. The response behaviour in Fig. 5 indicates an initial linear dependence on the sampling period with an upward curvature at longer sampling periods. This is due to the evaporation of the film solution, resulting in increased effective sample concentration before injection. The post-sampling transport of the capillary to the sample introduction position also requires 12–13 s; during this time, further evaporation occurs. Under these sampling and injection conditions, we have calculated and experimentally confirmed that the sample becomes adequately mixed in the film before injection [25]. It has also been previously demonstrated that an internal standard can be incorporated in the film to compensate for effects of film evaporation.

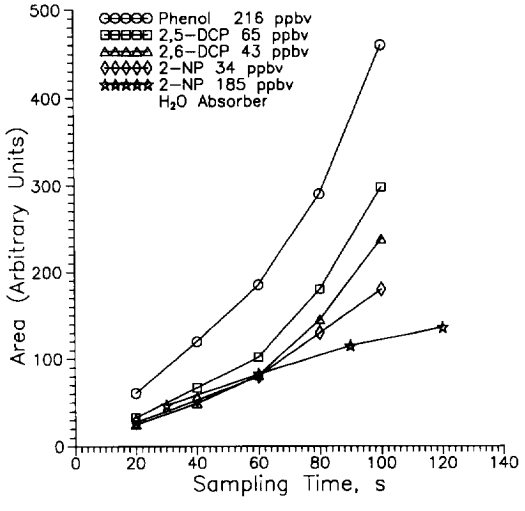

Fig. 5. Influence of sampling-time on the system response using a constant sampling rate of 100 cm^3/min.

The dependence of the response on the sampling duration is also affected by the choice of the absorber. For 2-NP, the effect of the sampling period on the observed signal was measured with both pure water and NaOH as the film. Unlike in the case of the NaOH absorber, with a water film, the response slope decreased with increasing sampling time, notwithstanding any effect of evaporation. This indicates a situation where the film surface becomes saturated with the analyte in little more than 60 s. Although the tested 2-NP vapour concentration in this case is approximately 5.5-times higher than that used with the NaOH absorber, the analytical signals obtained after 60 s sampling are identical in the two cases, suggesting that the collection efficiency for this analyte with the NaOH absorber is much higher.

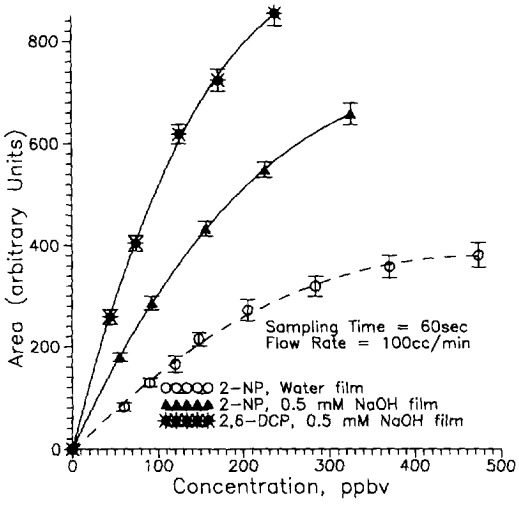

Fig. 6. Calibration curves for gas phase 2-NP using water; and for gas phases 2-NP and 2,6-DCP using 0.5 m*M* NaOH as the absorber film.

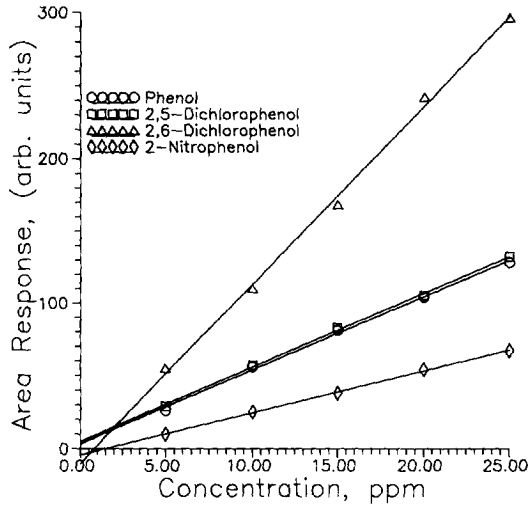

Fig. 7. Liquid-phase calibration curve obtained by injecting standard solutions directly from the loop film.

3.4. Calibration behaviour

Calibration plots for 56–325 and 45–237 ppbv of 2-NP and 2,6-DCP, respectively, are shown in Fig. 6 for 0.5 m*M* NaOH as the absorber. A calibration plot for 60–473 ppbv 2-NP is also shown for pure water as the absorber. Comparison of the two sets of 2-NP data again demonstrates that the alkaline absorber permits a more sensitive measurement. While the initial response is linear under these conditions (60 s sampling at 100 cm^3/min), negative curvature from linearity is clearly observed at higher concentrations. This behaviour suggests that the collection efficiency eventually decreases as the sample concentration is increased, regardless of the nature of absorber solution composition. Clearly, at what sampling time the onset of surface saturation is first noticeable will depend both on the absorber composition and the sample concentration. In any case, if the intended measurement range is known, the upper range of linearity can easily be manipulated by controlling the amount of analyte collected, most easily by changing the sampling time.

We have implicitly assumed in the above discussion that the aqueous-phase calibration shows linear response behaviour. Calibration plots were generated for 5–25 ppm standard solutions of phenol, 2,5-DCP, 2,6-DCP and 2-NP. Linear behaviour is observed, as shown in Fig. 7. The linear r^2 values for the concentration–peak area relationships are 0.9972, 0.9986, 0.9973 and 0.9991, respectively.

3.5. Reproducibility and limits of detection.

With the NaOH absorber, the overall reproducibility of the liquid film–gas sampling MEKC system (expressed as % R.S.D.) were as follows (analyte concentration in parentheses): 2-NP: 4.5% (56 ppb), 4.8% (92 ppb), 3.4% (156 ppb), 2.7% (225 ppb) and 3.2% (325 ppb); 2,6-DCP: 5.6% (45 ppb), 3.8% (74 ppb), 3.1% (125 ppb), 3.0% (170 ppb) and 2.8% (240 ppb). Considering the uncertainties in generation/transmission of source gases, dilution gas flow, sample collection and injection variability, etc., these results are quite reasonable. The precision was worse when pure water was used as the absorber, ranging from 6.2–9.8% in R.S.D. over a concentration range of 60–470 ppb 2-NP.

The electropherogram resulting from sampling eight analytes at concentrations near the LOD is shown in Fig. 8. Under these conditions, LODs of single-digit to low-double-digit ppbv levels of the various phenols can be conservatively estimated.

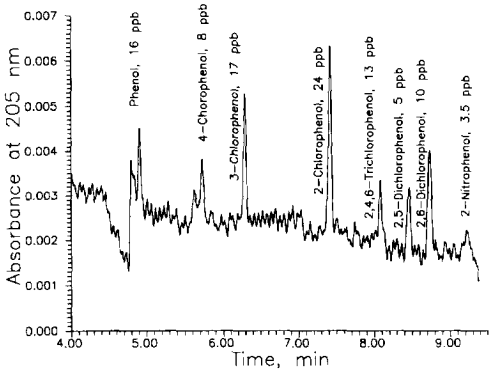

Fig. 8. Performance near the LOD. 60 s sample obtained at 100 cm^3/min.

However, the LOD is obviously dependent on the sampling time and the sampling rate [25]. Larger sampling periods will be possible if the sample air is sufficiently humid or if a low volatility liquid is used as the film. Electromigration injection can potentially improve the LOD as well [34,35].

4. Conclusion

We have demonstrated a simple and direct determination of gas- phase UV absorbing analytes by MEKC. The gases are collected for a fixed period of time on a suitable liquid-absorber film that is in direct contact with the separation capillary inlet. The analytes are then directly injected into the capillary and separation is performed. In future, we plan to utilize this concept of sample collection and measurement in the analysis of polynuclear aromatic hydrocarbons.

Acknowledgments

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