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# Simultaneous Determination of Heroin, Amphetamine and their Basic Impurities and Adulterants Using Microemulsion Electrokinetic Chromatography

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**Abstract:** Simultaneous separation of 17 species of heroin, amphetamine and their basic impurities and adulterants was conducted within 10 minutes by using capillary microemulsion electrokinetic chromatography. The influences of pH and 1-butanol cosurfactant on the separation were investigated, and 1-butanol was found to be a principal factor to improve separation efficiency.

**Keywords:** Heroin, amphetamine, microemulsion electrokinetic chromatography, determination.

Heroin and amphetamine are two most abused drugs that can be highly complex, containing various impurities, byproducts, adulterants and diluents due to differences in the agricultural and manufacturing procedures. Therefore, the comprehensive analysis of above illicit drugs seized or purchased undercover by law enforcement authorities is important for legal and intelligence gathering purposes, and clinical and pharmaceutical purposes as well[1]. In last decade, micellar electrokinetic chromatography (MEKC) was introduced to the qualitative and quantitative analysis of the two drugs because it was capable of simultaneously separating different neutral, anionic and cationic substances existed. However, the separation time of MEKC for heroin was usually long[2-3]. Recently, microemulsion electrokinetic chromatography (MEEKC) has gained increasing interest as an interesting alternative to MEKC as well as a new important fast separation technique involving pseudostationary phase[4-5]. To the best of our knowledge, there have been no reports concerning the use of MEEKC for the analysis of heroin and amphetamine preparations. Here we present the rapid simultaneous separation of heroin, amphetamine and their basic impurities/adulterants using MEEKC technique.

## Experimental

Heroin(k), amphetamine(l), caffeine(a), theophylline(b), barbital(c), phentobarbital(d),

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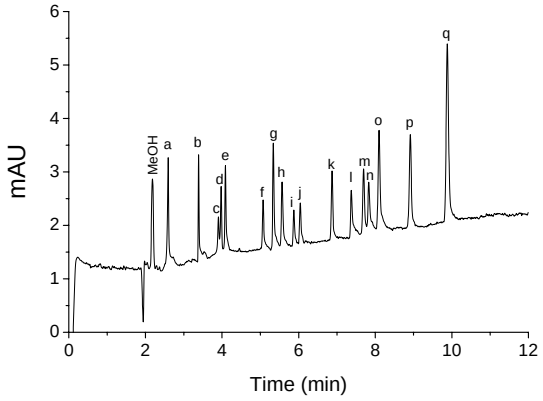
morphine(e), $O^6$-monoacetylmorphine(f), $O^3$-monoacetylmorphine(g), acetylcodeine(m), codeine(h), ephedrine(i), methylephedrine(j), methamphetamine(n), papaverine(p), thebaine(o), and narcotine(q) were provided by Institute of Forensic Science of National Ministry of Public Security. SDS was obtained from Sigma. Sodium tetraborate decahydrate, 1-butanol, octane, sodium hydroxide and phosphoric acid were obtained from Beijing Chemical Reagents Company. Ultrapure water, obtained by a Milli-Q purification unit from Millipore S.A.S, was used for buffer preparation. Test solutes were dissolved in methanol-water 50:50 v/v solution. All microemulsions were prepared on w/w basis.

The experiments were carried out on a Beckman MDQ CE system with DAD, and detection wavelength of 200 nm was used. The separations were performed in a fused-silica capillary, 40 cm × 75 μm ID, 30 cm to the detection window (Yongnian Photoconductive Fiber Factory, Hebei, China). The capillary was thermostated at 25 ℃ and a constant voltage of 20 kV was applied during analysis. Sample injection was achieved using the pressure mode for 5 s at 0.5 psi (3450 Pa).

## Results and Discussion

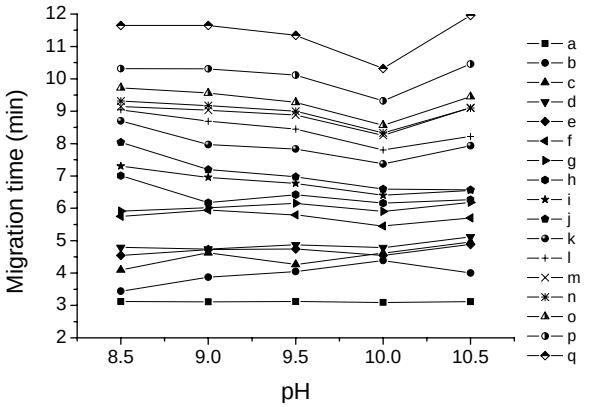
At present, among the different CE modes, MEKC is regarded as the only one to be able to resolve the 17 acidic, basic and neutral drugs tested, but the separation time is usually over 45 min[2-3]. Compared to MEKC, with the introduction of organic phase and cosurfactant, MEEKC delivers much higher selectivity. A typical MEEKC separation result of different 17 drugs is shown in **Figure 1**. It can be seen that the 17 tested drugs had been successfully separated within 10 min. Accordant with the results of MEKC separation results reported, acidic barbitals, theophylline and caffeine eluted first, showing a retarding effects by the SDS micelles. Other basic analytes eluted latterly, mainly as congeners according to their increasing degree of hydrophobicity.

**Figure 1** Typical example of MEEKC separation of the drug test mixture.




Buffer: 3.31% SDS / 6.72% 1-butanol / 0.90% octane / 89.07% 5 mmol/L sodium tetraborate solution (pH 9.25).

**Figure 2** The influence of pH on the migration time for 17 drugs tested.

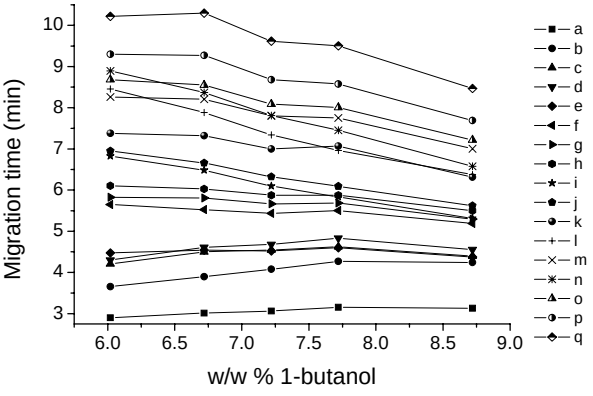



Buffer: 3.31% SDS / 6.72% 1-butanol / 0.90% octane / 89.07% 5 mmol/L sodium tetraborate solution.

Among the different controlling factors for MEEKC technique, pH is an important one to the separation[5]. The influences of pH on the separation were investigated in normal alkali range for MEEKC. As the results shown in **Figure 2**, pH variations did not change the elution sequence of 17 drugs tested. Negligible influences on the separation efficiency were shown in lower alkali pH range, and the separation resolution could remain relatively stable. For high pH range, poor separation efficiency was shown. It was also interesting that the separation window remained about the same in the most pH range.

The cosurfactant is another important controlling factor for MEEKC separation[6-7]. 1-Butanol was used as the cosurfactant in our separation and its impact on separation was shown in **Figure 3**. As illustrated in **Figure 3**, the whole elution window time was compressed with the increase of the amount of 1-butanol, indicating the analytes could

**Figure 3** The influence of 1-butanol concentration on the migration time for 17 drugs tested.




Buffer: 3.31% SDS / 0.90% octane / 5 mmol/L sodium tetraborate solution (pH 9.50).

more freely penetrate in and out the microemulsion drops as 1-butanol increased. 1-Butanol mainly existed inside the Stern layer or the core of microemulsion[7], so the microemulsion drops became more swollen and flexible with the addition of 1-butanol, thus resulting in faster exchange speed of analytes between buffer solution and micoremulsion. As the results shown in **Figure 3**, the influences of 1-butanol on congeners were relatively similar and the elution sequence of congeners remained the same. The elution time of amphetamines decreased more rapidly than other drugs tested with the addition of 1-butanol, so their elution positions could be adjusted by the concentration of 1-butanol cosurfactant.

In conclusion, all 17 species of heroin, amphetamine and their basic impurities and adulterants had been simultaneously separated within 10 minutes by MEEKC. 1-Butanol was the principal component controlling the separation selectivity of the MEEKC system. The most suitable conditions for heroin by MEEKC determination were: 1-butanol 6.0% - 7.3% and pH 8.5-10.0.

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