Electronic Supplemental Information

A Surface Ionization Detector for Capillary Gas Chromatography

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Contents:

S0. Experimental conditions for Fig.2, Table 1 & Fig.3

PerkinElmer Autosystem XL Gas Chromatograph with a PSS injector. Capillary Column: 30 m×0.25 mm id× 0.25 μm HP-1. Carrier: nitrogen. SID temperature: 260 ℃. Makeup gas: air purified by silica gel and active carbon, 120 ml/min. Mo emitter temperature: 370~380 ℃. Bias voltage applied on the Mo emitter: +150 V~+350 V.

S1. Extraction of lidocaine as a free base

The lidocaine hydrochloride (purity >99%) obtained was used without further purifucation. The drug as a salt was first dissolved in purified water (about 1 g in 10mL). The solution was transferred into a separatory funnel. Then, 0.2 mL of 2 M Na_2CO_3 was added into the solution. Instantly, the solution became cloudy with many white fine particles being suspended. To extract the free base of lidocaine, about 5 mL of ethylether was added into a separatory funnel. After shaking the funnel several times, the water layer became clear. The organic layer was transferred into a sample vial and concentrated under nitrogen.

S2. Spiking serum with lidocaine

Serum spiked with 440 μg/L lidocaine was prepared as follows: (1) The stock solution of 110 mg/L lidocaine in ethanol was prepared; (2) Samples of 0.5 mL serum were spiked with 2 μL of the stock solution and were then shaked for 1 min.

S3. Experimental conditions for the analysis of the serum extract

GC-SID conditions:

PerkinElmer Autosystem XL Gas Chromatograph. PSS injector: 330 ℃, splitless mode. Oven temperature programming : 80 ℃ for 2.5 min, programed at 40 ℃/min to 210 ℃, and hold for 15min. Capillary Column: 30 m×0.25 mm id× 0.25 μm HP-1. Carrier: N_2, 0.8 mL/min in the constant flow mode. SID temperature: 280 ℃. Makeup gas of the SID: air purified by silica gel and activitated carbon, 120 mL/min. Mo emitter temperature: 370~380 ℃.

GC-NPD conditions:

Varian CP-3800 Gas Chromatograph. 1177 injector in the splitless mode. NPD temperature: 350 ℃. Bead current: 3.450 A. Gases of the NPD: air purified by silica gel and activitated carbon, 175 mL/min; H_2, 4.0 mL/min, N_2, 30 mL/min. Other conditions were the same as the above GC-SID conditions.

S4. Serum extraction procedures

The serum extraction procedures similar with that described by Fujii[12] were as follows: (1) To 0.5 mL of serum, add 0.4 mL of 0.5 M NaOH. (2) To each add 0.5 mL of isopentanol. (3) Shake 1 min. Centrifuge at 3000 r/min for 8 min. (3) Aspirate the upper organic layer into a vial. (4) Inject 0.1 μL of extracted samples into the GC injector in a splitless mode.

S5. Extraction of tetracaine as a free base

The tetracaine hydrochloride (purity >99%) obtained was used without further purification. The drug as a salt was first dissolved in purified water (about 0.4 g in 20 mL). The solution was transferred into a separatory funnel. Then, 0.2 mL of 2 M Na_2CO_3 was added into the solution. Instantly, the solution became cloudy with many white fine particles being suspended. To extract the free base of tetracaine, about 15 mL of ethylester was added into the funnel. During the extraction, the water layer gradually became clear. The organic layer was transferred into a round bottom flask and concentrated by a rotary evaporator at room temperature.

S6. Spiking urine with tetracaine

Urine spiked with 6 mg/L tetracaine was prepared as follows: (1) The stock solution of 3000 mg/L tetracaine in ethanol was prepared; (2) Samples of 0.5 mL urine were spiked with 1 μL of the stock solution and were then shaked for 1 min.

S7. Experimental conditions for the analysis of the urine extract

The experimental conditions for the analysis of the urine extract were the same as S4 except for the oven temperature programming. Oven: 80 ℃ for 2.5 min, programmed at 40 ℃/min to 250 ℃, and hold for 10min.

S8. Urine extraction procedures and the analysis of the extract

The urine extraction procedures similar with that described by Fujii[12] were as follows: (1) To 0.5 mL of urine, add 0.3 mL of 2 M Na_2CO_3. (2) To each add 0.5 mL of isopentanol (purity >98.5%). (3) Shake 1 min. Centrifuge at 3000 r/min for 8 min. (3) Aspirate the upper organic layer into a vial. (4) Inject 0.1 μL of extracted samples into the GC injector in a splitless mode. The recovery was tested with urine samples containing 6 mg/L tetracaine. The recovery from nine analysis from seven analysis was about 95 % ~125 %. Figure S2 shows SID chromatograms (b) and the NPD chromatogram (c) of extracts of urine containing tetracaine (440 μg/L). Comparing Figure S2 (b) with Figure S2 (a), the trace amounts of tetracaine was detected by the SID with about 300 times peak height of the noise. In Figure S2 (c), the tetracaine was detected by the NPD with about 30 times peak height of the noise. The sensitivity of the SID to tetracaine was more sensitive than that of the NPD we used.

Figure S1. Retention time difference between TEA and cyclohexane under various oven temperatures.

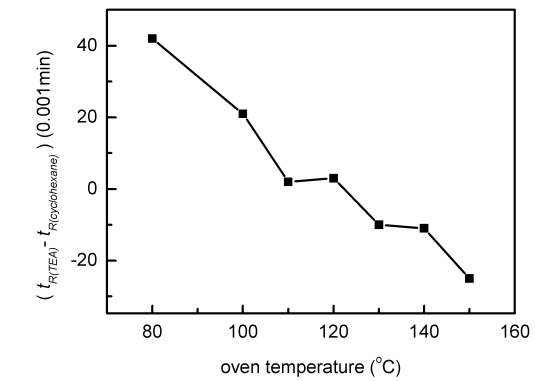

Figure S1. Retention time difference between TEA and cyclohexane under various oven temperatures.

Figure S2. Comparison of GC-SID with GC-NPD for the analysis of extract of the urine spiked with 6 mg/L tetracaine.

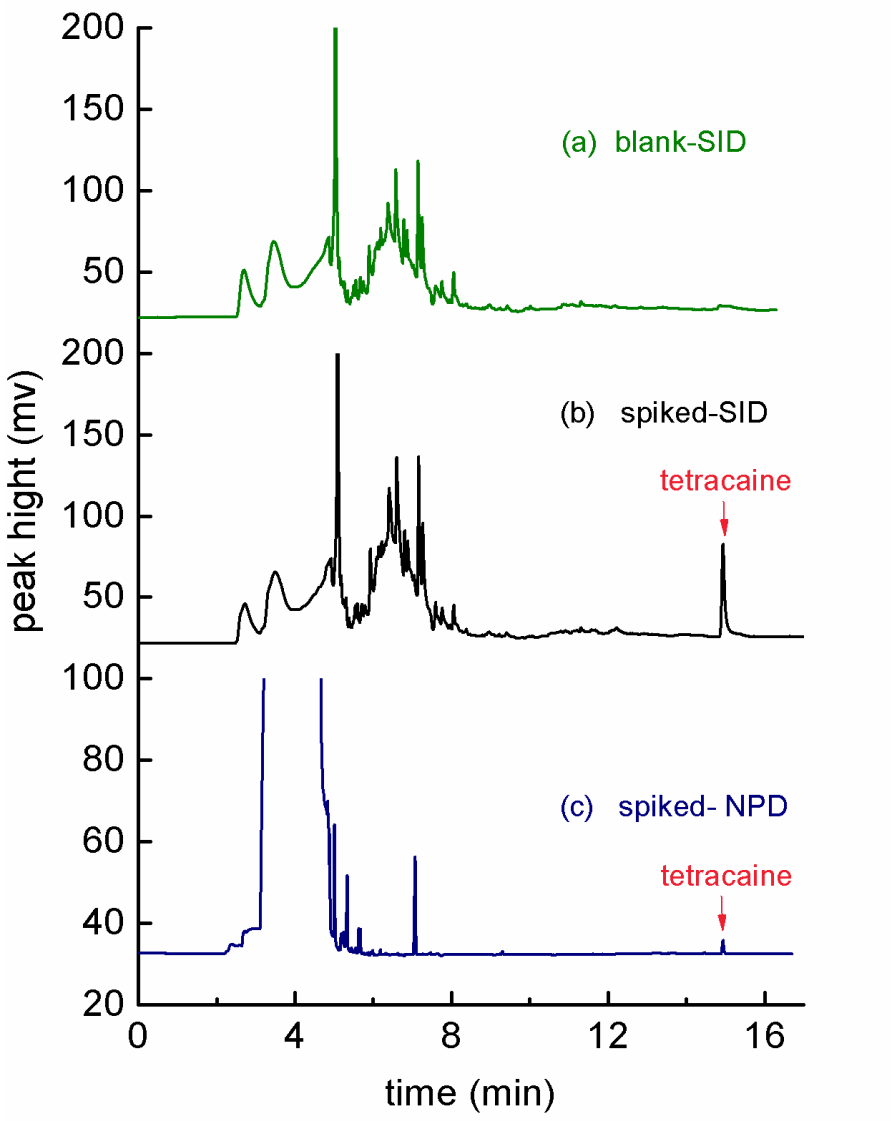

Figure S2. Comparison of GC-SID with GC-NPD for the extract of the urine spiked with 6 mg/L tetracaine. (a) GC-SID chromatogram of the blank urine extract; (b) GC-SID chromatogram of the spiked urine extract; (c) GC-NPD chromatogram of the spiked urine extract.