Operating Instruction Manual

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CElixirMS™

Kits for use with CE and Mass Spec

Instructions for use.

1. Intended Use and Background:

This kit consists of a polycation and a polycation solution that can be used for the preparation of volatile buffers fro CE-MS. The polycation and polyanion will dynamically coat the inner surface of the capillary wall and produce a fast and consistent EOF (electro-osmotic flow). Patent 5,611,903

2. Contents:

CElixir-MS Initiator: 1.5ml of polycation in formic acid (100mM) as a ready to use reagent.

CElixir-MS Accelerator: 4ml of concentrated polyanion solution in water.

Not included in this kit: separation buffers, bi-distilled water, NaOH 0.1M, capillary, vials and caps.

3. Operating Instructions:

This Operating Manual is written for the Beckman P/ACE MDQ—other instruments can be programmed differently.

3.1 **Capillary** for use: Simplus™ or other brand, bare fused silica, 75um x 80cm or suitable lengths for CE-MS

3.2 **Instrument**: CE-MS with external adaptor and MS Instrument with ESI Source.

3.3 **Separation Solution**: Prepare a suitable volatile electrolyte or buffer for CE-MS. To 24ml of this buffer, add 1ml of CElixir-MS Accelerator Solution. Also, prepare a suitable make-up solution.

3.4 **Initialize Capillary**: When using a new capillary, initialize it (when connected to the MS with an open ESI interface):

A. Rinse the capillary with NaOH 0.1M for 1 minute at 20psi
B. Rinse the capillary with CElixir-MS Initiator solution for 1 minute at 20 psi.

C. Rinse the capillary with the separation solution for 2 minutes at 20 psi.
D. Rinse the capillary with NaOH for 1 minute at 20 psi.
E. Rinse the capillary with CElixir-MS Initiator solution for 1 minute at 20 psi.

EOF should remain stable for approximately 20 runs.

3.5 Separation:

Close the ESI interface and run the following separation program:

A. Rinse the capillary with the separation solution for 1 minute at 20 psi.
B. Inject the sample, for example; 10 seconds at 0.5psi
C. Separate at 25kV or 30kV with 1 minute ramping time for 15 to 20 minutes.
D. Rinse the capillary with the separation solution for 1 minute at 20 psi.

Note: during injection and current ramping time,

A. Put the ESI needle voltage to 0kV
B. Use a separate vial for rinsing the capillary with the separation solution and f for the separation solution
C. The dynamic coating will remain stable for about 20 runs. Coating can be applied again by re-initializing the capillary: See 3.4

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4. Example E-grams:

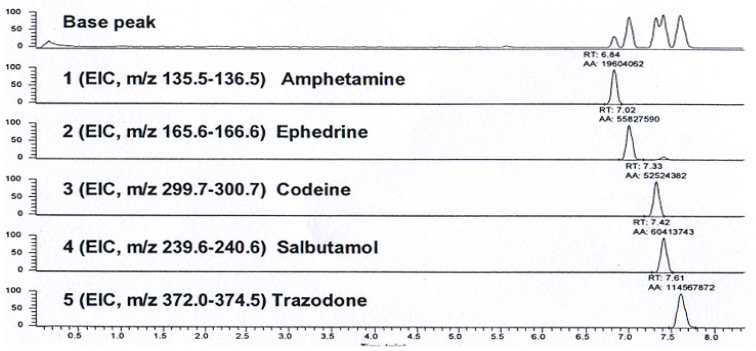

Figure. CEMS analysis of a standard mixture of 5 basic drugs (2ug/ml each) with 100mM formic acid + CElixir-MS accelerator + 1mM TFA solution. Capillary 75um ID x 93.5 cm L tot. CE Current: 25umA, injection 5s at 2 psi spray current: 19uA, N2 gas flow: sheath 20 units/auxiliary 0 units [1].

Instrument and method sued in previous electropherograms:

CE: P/ACE MDQ with EDA accessory to connect to ESI interface (Beckman Coulter, Fullerton CA, USA)
MS: LCQ ion Trap (IT) (Thermo Finnigan, San Jose, CA)
Pre-run rinse: 1 min at 20 psi with buffer
Electrolyte: 100mM formic acid + accelerator + 1mM TFA
Capillary: Simplus™ brand bare fused silica, 75um x 93.5cm
CE Voltage: 30kV (- to +), built up in 1 min ramp
CE Current: 25uA
Make up flow: 2ul/min, methanol/Water (80/20 v/v) with 0.5% formic acid, daily degassed in an ultrasonic bath, provided by a syringe pump installed on the mass spectrometer.
ESI needle voltage: 5 kV (0 kV during injection and voltage build up)
ESI spray current: 19uA
Sheath Gas: 20 units, auxiliary gas: 0 units
Scan Range: 100-400 amu
Position CE-capillary outlet: equal to ESI needle
Test-mix was composed of Amphetamine, Ephedrine, Codeine, Salbutamol and Trazodone each at 2ug/ml.

5. **References**:

[1] G. Vanhoenacker et al. J. Chromatogr. B799 (2004) 323-330
[2] G. Vanhoenacker et al. Journal of Pharmaceutical and Biomedical analysis 34 (2004) 595-606.

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