Analysis of Sumatriptan in Cerebro-spinal Fluid Using Capillary LC/MS/MS

**INTRODUCTION**

Sumatriptan helps to relieve headache pain and associated symptoms of migraine (nausea, vomiting, sensitivity to light and sound). It also helps to constrict dilated blood vessels that may contribute to development of migraines. Most analysis techniques for the separation, detection and quantification of sumatriptan are based on conventional high-performance liquid-chromatography techniques. Here the gain in sensitivity is demonstrated by using capillary LC/MS/MS.

**RESULTS AND DISCUSSION**

The sample preparation steps for sumatriptan in cerebro spinal fluid (CSF) prior to LC/MS analysis is straight forward and demonstrated in the Figure 1. The UltiMate™/FAMOS™ Capillary LC System was equipped with a 300 µm I.D. x 15 cm column packed with C18, 3 µm stationary phase at flow rate of 5 µL/min. Mobile phase A: 0.1% aqueous formic acid, B: acetonitrile. Gradient: 5 to 60% B in 8 min. MS/MS analysis was conducted with a triple quadrupole MS equipped with an electrospray interface.

Figure 2 shows multiple reaction monitoring (MRM) chromatograms of sumatriptan analysis by means of Capillary and conventional HPLC. The upper trace corresponds to a chromatogram taken when 1.25 pg of sumatriptan was injected on column and the lower trace shows the limits of detection achieved with conventional HPLC, which is approximately equal to

![Figure 1. Schematics of sample handling prior to Capillary LC/MS/MS.](image)

![Figure 2. Capillary (top) and conventional (bottom) LC/MRM chromatograms of sumatriptan. Gradient conditions are given in the text.](image)
300 pg. The overall gain in sensitivity is approximately a 700 fold. For spiked CSF samples, the peak area ratios of analyte to internal standard were linear over the calibration range 0.5–15 ng/µL. A typical calibration curve is shown in the Figure 3. The limit of quantitation was found to be equal to 300 pg/µL.

REFERENCES

1. Courtesy Prof. G. Moneti, CIMS, University of Florence, Italy

![Figure 3. Calibration curve for the analysis of sumatriptan in CSF.](image-url)