FAST SIMULTANEOUS DETERMINATION OF SEVEN OPIATES BY GRADIENT LIQUID CHROMATOGRAPHY/TANDEM MASS SPECTROMETRY USING A MONOLITHIC SILICA COLUMN

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Monolithic silica columns have proven to be an excellent tool in the development of high-throughput applications, without any loss in chromatographic performance compared to traditional packing materials. Combined with liquid chromatography-mass spectrometry, an opportunity is given to develop rapid, selective and specific methods. We present a fast LC-ESI-MS/MS method, with post column split, for the simultaneous determination of the seven prime opium alkaloids in illicit heroin samples, namely acetylcodeine, 6-monoacetylmorphine, codeine, heroin, morphine, noscapine, and papaverine. The effect of organic solvents, volatile acids, and buffer systems, present in the mobile phase, on the ionization efficiency of the target compounds will be described. Final chromatographic separation was performed on a monolithic silica column (Chromolith™ Performance, 100 x 4.0 mm) eluted with water/acetonitrile (90:10, v/v) (A) and acetonitrile (B) in a gradient system. The flow was optimized at 4.5 mL/min, resulting in a total analysis time of only 7 minutes. Limits of detection (LOD) and quantitation (LOQ) were always lower than respectively 1 and 2 ng injected on column for all compounds. Furthermore, complete validation of the method was performed. Linearity, using 8 calibrator samples, proved to fulfill analytical standard criteria. Finally, the method was applied to real-time seized heroin street samples in an effort to test the applicability of the analysis to heroin impurity profiling. No interference showed up and the analyses could be performed without technical and analytical problems, demonstrating the robustness of the method.

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