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Comparison Of Different Mass Spectrometry Ionization Techniques to Analyze Desogestrel in Plasma Samples

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Introduction: The development of HPLC-MS/MS has opened completely new horizons in analytical chemistry: polar and unstable compounds can be now easily analyzed with adequate sensitivity. Apolar compounds, once considered as best target for mass spectrometry, are now a critical class of compounds for HPLC-MS. In the present work desogestrel, a progestogen often used in therapy, has been selected as a model apolar compound to compare GC/MS and HPLC-MS/MS, with and without derivatization, in order to develop a highly sensitive quantitative method.

Materials and methods: In case of HPLC-MS/MS electrospray, atmospheric pressure chemical ionization, photoionization and experimental electron capture sources were tested. In case of GC/MS both electron impact as well as chemical ionization (positive and negative) were employed. The samples were analyzed as such or after derivatization with trimethylsilylimidazole (GC only), heptafluorobutyrylimidazole or pentafluoropropionylimidazole. The same SLE method was used for desogestrel isolation from plasma samples (0.6 ml aliquots) both for GC-MS and HPLC-MS/MS analyses.

Results and conclusions: In HPLC-MS/MS the best results (LLOQ 200 pg/ml desogestrel in plasma), without derivatization, were obtained with the photoionization source using a reversed phase separation. The derivatization with perfluorinated imidazoles gave adequate derivatives for electron capture ionization; critical technical aspects are still under evaluation due to the experimental source employed. With GC-MS a better sensitivity was achieved in case of the derivatives (LLOQ 50 pg/ml), using negative chemical ionization; a long analytical separation, with a 60 m DB-5 column, was however mandatory in order to eliminate the interference from endogenous compounds.