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Implementation of HPLC/Orbitrap mass spectrometry as screening method for doping control [1]

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Summary

A new doping control screening method for the analysis of doping agents in human urine using HPLC/Orbitrap with in-source collision-induced dissociation and atmospheric pressure chemical ionization has been developed. The developed method allows the detection of 29 compounds, including agents with antiestrogenic activity, β_2 agonists, exogenous anabolic steroids, and other anabolic agents. The mass accuracy of this method was better than 2 ppm using an external reference. The detection limit for all compounds tested was better than 100 pg/mL. The recoveries of most analytes were above 70%. The measured median repeatability values for doping agents included in the method at concentrations of 1 ng/mL and 10 ng/mL, were 21% and 17%, respectively. The relative standard of the intraday precision (n = 6) ranged from RSD = 16%–22%, whereas the interday precision (n = 18), ranged from RSD = 17%–26%, depending on the solute concentration investigated.

Although the sensitivity is similar for the HPLC/MS/MS and HPLC/HRMS techniques, the latter technique has the advantage of acquiring full scan MS spectra. The proposed method also matches the basic requirements of all methods used to analyze drugs or metabolites in an antidoping laboratory, i.e., the sensitivity, selectivity, specificity, and speed of analysis. In conclusion, this system is sufficiently robust to carry out 450 analyses without routine maintenance.

Reference

1. Virus E. D., Sobolevsky T. G., Rodchenkov G. M. (2008) Introduction of HPLC/orbitrap mass spectrometry as screening method for doping control. *J Mass Spectrom.* **43**, 949-957.