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Application of UHPLC-orbitrap based mass spectrometry with scan-to-scan polarity switching for the direct detection of doping agents in urine.

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Abstract

Introduction: Stimulants and diuretics are included in the prohibited list published by the World Anti Doping Agency (WADA). Application of full scan high resolution mass spectrometry (HRMS) for screening allows not only to detect target compounds but allows to evaluate retrospectively the previously obtained data for the presence of emerging drugs. Therefore, a procedure to detect stimulants and diuretics in urine by LC-HRMS was developed.

Methods. Sample preparation was limited to a 10-fold dilution of the urine-samples. Chromatography was performed on a rapid resolution column with gradient elution. The detection was performed on an Exactive® orbitrap HRMS operated in full scan MS with scan-to-scan polarity switching. Analytical parameters of the method in terms of sensitivity and mass accuracy were investigated and the method was validated for doping control purposes.

Results. Due to the acidic nature of diuretics, the highest sensitivity was observed in negative mode. Most of the stimulants showed maximum sensitivity in the positive ionization mode. Validation of the method yielded detection limits for diuretics between 25 and 250 ng/mL and for stimulants between 5 and 500 ng/mL. The method also showed to be very selective since no interferences were found when other common doping agents and blank urines were analysed.

Conclusions. A comprehensive open screening UHPLC-HRMS method has been developed and validated for the analysis of 122 diuretics and stimulants in human urine. All detection limits were compliant with the minimum required reporting levels (MRPL) imposed by WADA.

References

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