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A comprehensive and sensitive screening of doping agents on UPLC-MS/MS: An approach towards fulfillment of WADA TD2013 MRPL

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Abstract

The screening of wide variety of banned substances adhering to latest WADA (World Anti-Doping Agency) guidelines in a time bound manner is a challenging task for doping control laboratories. The revised WADA Minimum Required Performance Limit (MRPL) criterion applicable from January 2013 has further necessitated the need to review and revise testing procedures in the anti-doping laboratories. The aim of present study was to develop a fast, sensitive and comprehensive screening method for the detection of maximum drugs based on single solid phase extraction (SPE) procedure and ultra performance liquid chromatography tandem mass spectrometry (UPLC-MS/MS). To cover most of the compound groups in the prohibited list of WADA, a method was developed by optimization of various analytical steps: 1) hydrolysis for deconjugation of phase-II metabolite/s 2) single SPE using mixed-mode ion cartridges for extraction of acidic, basic and neutral compounds and 3) rapid and sensitive detection on UPLC-MS/MS-5500 Q Trap using polarity switching.

A fast, sensitive and selective method has been developed and validated to detect approximately 165 drugs and/or their metabolites from the categories of S1, S3, S4, S5, S6, S7, S8, S9, P2 and M1 sections of WADA prohibited list. The runtime of 8 minutes allowed testing of approximately 180 samples in 24 hours at the limit of detection (LOD) of 50% or below of minimum required performance limit (Table 1). The recovery for all compounds ranged from 30 % (ritalinic acid) to 115 % (canrenone). The method was also validated by successfully reanalyzing thirty urine samples reported positive using earlier method. The specificity of the method was determined by analyzing hundred urine samples which were already reported negative by previous method.

The complete study is being published elsewhere.