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Preventive doping control screening analysis of prohibited substances in human urine using rapid-resolution liquid chromatography/high-resolution time-of-flight mass spectrometry.

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Unification of the screening protocols for a wide range of doping agents has become an important issue for doping control laboratories. This study presents the development and validation of a generic liquid chromatography/time-of-flight mass spectrometry (LC/TOFMS) screening method of 241 small molecule analytes from various categories of prohibited substances (stimulants, narcotics, diuretics, beta(2)-agonists, beta-blockers, hormone antagonists and modulators, glucocorticosteroids and anabolic agents). It is based on a single-step liquid-liquid extraction of hydrolyzed urine and the use of a rapid-resolution liquid chromatography/high-resolution time-of-flight mass spectrometric system acquiring continuous full scan data. Electrospray ionization in the positive mode was used. Validation parameters consisted of identification capability, limit of detection, specificity, ion suppression, extraction recovery, repeatability and mass accuracy. Detection criteria were established on the basis of retention time reproducibility and mass accuracy. The suitability of the methodology for doping control was demonstrated with positive urine samples. The preventive role of the method was proved by the case where full scan acquisition with accurate mass measurement allowed the retrospective reprocessing of acquired data from past doping control samples for the detection of a designer drug, the stimulant 4-methyl-2-hexanamine, which resulted in re-reporting a number of stored samples as positives for this particular substance, when, initially, they had been reported as negatives.

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