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Identification and confirmation of sympathomimetic alkylamines in urine using liquid chromatography-mass spectrometry and gas chromatography-mass spectrometry

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Abstract

The detection of eleven sympathomimetic alkylamines in urine, including seven new molecules not listed by WADA, is proposed using liquid chromatography (LC) tandem mass spectrometry (LC-QqQ) and high resolution mass spectrometry (LC-HRMS), both as potential screening tools after a dilute-and-shoot approach.

The LC analysis encompasses all analytes currently screened by LC for doping control purposes and takes 9.6 min. However, in LC conditions applied, and due to small differences in alkylamines structures, co-elution among them was observed.

Therefore, the chemical conversion of the alkylamines into an appropriate derivative for the confirmation analyses using gas chromatography-mass spectrometry (GC-MS) was performed. Five derivatization approaches were evaluated in an attempt to increase the analytical response and the confidence of the identification. The choice of the appropriated derivative for each alkylamine makes their spectra more easily interpretable, fulfills the WADA's rather strict identification criteria and enables the unequivocal identification of alkylamines in urine.

This work was presented as a contribution to the Manfred Donike Workshop. The full experimental and instrumental conditions and other considerations about alkylamines analysis in urine are described in:

Sardela VF, Sardela P, Deventer K, Araujo A L, Cavalcante KM, Padilha MC, Pereira MG, Van Eenoo P, Aquino Neto FR. (2013) Identification of sympathomimetic alkylamine agents in urine using liquid chromatography-mass spectrometry and comparison of derivatization methods for confirmation analyses by gas chromatography-mass spectrometry. *J Chromatogr A* **12**, 76-85