

LIQUID CHROMATOGRAPHY/TIME-OF-FLIGHT MASS SPECTROMETRY FOR ROUTINE DRUG SCREENING IN HORSE URINE

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The usefulness of liquid chromatography mass spectrometry mass spectrometry (LC–MS–MS) methods for the unambiguous identification and quantification of drugs in complex matrix samples is well known. Triple quadrupole systems have proven to be useful for this task because of their high specificity in MS–MS mode and their low detection limits. However, working in MS–MS mode makes any MS system blind to other compounds of interest. Therefore, it is difficult to develop methods for simultaneous analysis of high numbers of targets. Thus, other ways of achieving specificity are of interest, such as the high mass accuracy and mass resolution of an electrospray ionization time-of-flight (ESI-TOF) system. It can generate high specificity without limiting the number of simultaneously observed target compounds.

Recently, bench-top Liquid Chromatography/Time-of-Flight Mass Spectrometry (LC/ESI-TOFMS) systems have become available that offer sufficient mass accuracy and resolution to be used in the routine screening of drugs and metabolites in biological samples.

A general drug screening protocol has been developed to detect a wide range of prohibited substances in equine and canine urine using this technique.

Various SPE procedures were used and the extracts combined and evaluated for their suitability for LC/TOFMS analysis. Both positive and negative ionisation modes were employed to give adequate sensitivity for all analytes. A database was established containing the molecular formula and where known, the retention time of all compounds of interest. Data processing software was developed to create and integrate extracted ion chromatograms of the target pseudo-molecular ions in the database within a very narrow mass window (± 3 mDa). Post-processing software evaluates the spectra by using matching criteria based on mass accuracy of the pseudo-molecular ion, SigmaFit™ and retention time. Qualitative findings are then reported automatically in an easily interpretable form.

Positive findings can be investigated and confirmed by conventional LC/MS and GC/MS techniques or by using in-source CID with the ESI-TOFMS.

The screening protocol was shown to meet the required performance specifications for the large majority of the prohibited substances evaluated. LC/ESI-TOFMS analysis can deliver significant savings with respect to sample preparation and analysis time by allowing simplified extraction procedures and analysing groups of compounds simultaneously that are traditionally handled separately.