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Hyphenation of dispersive liquid-liquid microextraction with chromatographic techniques for the detection of drugs of abuse

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Tracing of drugs of abuse at low concentration levels in various matrices of forensic, clinical and biological interest is always been a challenging task for analytical chemists. Traditional sample preparation methods such as solid phase extraction (SPE) and liquid-liquid extraction (LLE) are well known for their disadvantages of being environmentally unfriendly, costly and labor intensive. Dispersive liquid-liquid microextraction (DLLME), introduced in 2006 has gained a large attention from analytical chemists. DLLME is a fast, eco-friendly, cost-effective and easy microextraction technique offering high enrichment factors and extraction efficiencies. Since its introduction, DLLME has been hyphenated with various instrumental techniques like high-performance liquid chromatography (HPLC), gas chromatography-mass spectrometry (GC-MS) and UV-Visible spectrophotometer for the detection of drugs of abuse in various complex matrices such as blood, urine, saliva etc. The capability of coupling of DLLME with injection port derivatization (IPD) offers an attractive alternative for the analysis of polar analytes by GC-MS. This presentation is focused on the discussion of recent advances in coupling of DLLME with chromatographic techniques for the detection of drugs of abuse as well as other analytes of forensic interest in various complex matrices.

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Pharmacokinetic study of oxime prodrug of gliclazide by LC-MS/MS method in rabbit plasma

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Oxime prodrug of gliclazide is a water soluble and biologically inactive derivative of gliclazide, a sulphonyl urea analogue used to treat type II diabetes mellitus. A rapid liquid chromatography tandem mass spectrometry LCMS-MS method has been optimized for analysis of oxime prodrug of gliclazide in rabbit plasma using clopidogrel as internal standard. Following turbo ion spray ionization, the analytes were quantified on a triple–quadrupole mass spectrometer in multiple-reaction-monitoring (MRM) positive ion mode. Sample preparation involved a simple one-step protein precipitation with methanol, followed by centrifugation and evaporation of the organic solvent. The residue was re-dissolved in mobile phase and analyzed by LC–MS/MS. A symmetry C18, 50x4.6, 5µ, a mobile phase composed of Acetonitrile: 25mM Potassium dihydrogen orthophosphate (pH- 6.5) (50:50 v/v), and a flow rate of 0.6 mL/min were employed, and the total run time was 3.0 min. The method was validated for accuracy, precision, linearity, selectivity, lower limit of quantification (LLOQ), recovery and matrix effect. The method was found to be linear in the range of 150 to 6000 ng/mL. LLOQ was found to be 27 ng/mL. All validation parameters met the acceptance criteria according to regulatory guidelines. This method was successfully applied to pharmacokinetic study of the prodrug in rabbit through oral administration.

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