

Simultaneous Determination of p-Phenylenediamine, N-Acetyl-p-phenylenediamine and N,N-Diacetyl-p-phenylenediamine in Human Urine by LC-MS/MS

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Abstract : Background: P-Phenylenediamine (PPD) is used in the manufacture of hair dyes and skin decoration. In some developing countries, suicidal, homicidal and accidental cases by PPD were recorded. In this work, a sensitive LC-MS/MS method for determination of PPD and its metabolites N-acetyl-p-phenylenediamine (MAPPD) and N,N-diacetyl-p-phenylenediamine (DAPPD) in human urine has been developed and validated. Methods: PPD, MAPPD and DAPPD were extracted from urine by methylene chloride at alkaline pH. Acetanilide was used as internal standard (IS). The analytes and IS were separated on an Eclipse XDB- C18 column (150 X 4.6 mm, 5 μ m) using a mobile phase of acetonitrile-1% formic acid in gradient elution. Detection was performed by LC-MS/MS using electrospray positive ionization under multiple reaction-monitoring mode. The transition ions m/z 109 \rightarrow 92, m/z 151 \rightarrow 92, m/z 193 \rightarrow 92, and m/z 136 \rightarrow 77 were selected for the quantification of PPD, MAPPD, DAPPD, and IS, respectively. Results: Calibration curves were linear in the range 10-2000 ng/mL for all analytes. The mean recoveries for PPD, MAPPD and DAPPD were 57.62, 74.19 and 50.99%, respectively. Intra-assay and inter-assay imprecisions were within 1.58-9.52% and 5.43-9.45% respectively for PPD, MAPPD and DAPPD. Inter-assay accuracies were within -7.43 and 7.36 for all compounds. PPD, MAPPD and DAPPD were stable in urine at -20 degrees for 24 hours. Conclusions: The method was successfully applied to the analysis of PPD, MAPPD and DAPPD in urine samples collected from suicidal cases.

Keywords : p-Phenylenediamine, metabolites, urine, LC-MS/MS, validation

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