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Optimization of Liquid Chromatography-Tandem Mass Spectrometry for Paraquat Detection in Biological Tissue Samples

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Abstract : A simple liquid chromatography-tandem mass spectrometry (LC-MS/MS) method for quantifying paraquat(PQ) level was described. Ethyl paraquat (EPQ) was used as an internal standard. The effects of varying the mobile phase composition, flow rate and splitter ratio were investigated. Analytes detection was conducted using a tandem mass spectrometer in multiple reactions monitoring (MRM) modes with the electrospray ionization (ESI) source operated in a positive ion mode. To optimize the MS-MS condition, the effects of varying instrumental parameters such as capillary voltage, collision energy, drying gas and spray chamber temperature as well as nebulizing gas pressure were investigated. Initial tissue sample preparation involved a simple one-step protein precipitation using acetonitrile. Chromatographic separations of PQ and EPQ were successfully performed on an HILIC column (3 µm; 150 × 4.6 mm) with mobile phase 250 mM ammonium formate/ acetonitrile (6:4 v/v). The flow rate was 1 ml/min and reduced to 0.1 ml/min for MS detection using splitter. The MRM transitions (precursor ion/product ion) for quantitation were m/z 186/171 for PQ and m/z 107/185 for EPQ. The optimized condition for MS-MS detection include: drying gas, 375 °C, 15 psi; nebulizing gas pressure, 50 psi.Overall, the method provides a simple and direct analysis for detection of PQ with a total run time of less than 10 minutes and is applicable for quantification of PQ in biological tissue sample with good recovery and precision achieved.

Keywords : paraquat, ethyl paraquat, LC-MS/MS, HILIC.

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