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Variations in GC–MS response between analytes and deuterated analogs

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Isotopic analogue is commonly used as an appropriate internal standard. It was reported that analytes have usually higher mass responses than their equimolar deuterated analogues (DAs) leading to quantification discrepancy. Standard addition method on dimethyl azelate (DMA) and d6- dimethyl azelate (d6-DMA) was adopted to examine possible reasons for the problem. Cross contribution of mass responses, intermolecular deuterium-hydrogen exchange during chromatographic separation, and deviation in mass ionization response of C-H against C-D bonds were studied as possible reasons for this discrepancy. GC-MS analysis revealed that neither cross contribution of ions nor H2/H exchange were possible reasons behind the difference in responses between DMA and d6-DMA relying on linearity and trans-esterification studies respectively. On the other hand, a study of carbon nucleus relaxation conducted by C13-NMR depicted that energy dissipation through C-D bond is faster than that through the C-H bond; relaxation rate of carbonyl carbon in d6-DMA and DMA were 9 and 3 sec-1 respectively. Accordingly, the energy transfer through the carbon skeleton of analytes and its mass ionization degree are more efficient than those in their DA counterparts. Conclusively, GC-MS analysis of analyte, relying on the assumption of equal response with its DA, generates overestimated analytical results of analytes.

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Simultaneous determination of sofosbuvir, paracetamol and methionine in rat plasma using thin-layer chromatography and its application to pharmacokinetic study

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Sofosbuvir (SOF) is a widely used drug for treatment of chronic hepatitis C while paracetamol (PAR) is the recommended analgesic for patients with hepatitis C because of its effectiveness and safety. Combination of PAR with methionine (MET) is preferred to reduce the severity of liver damage that may be produced from PAR overdose. A sensitive and highly selective TLC-densitometric method was developed for the first time for simultaneous analysis of SOF and the accompanied medications PAR and MET in the presence of the internal standard, naphazoline HCl (NAP) in rat plasma. Complete separation between the studied components peaks and plasma peak was obtained where Rf value of MET=0.18, NAP=0.39, PAR=0.59 and SOF=0.82. FDA recommendations for bio-analytical method validation were obeyed. The linearity of the method was assessed over the concentrations range 160-3000 ng mL-1 for both SOF and PAR and 300-3000 ng mL-1 for MET. Moreover, the accuracy, intra-and inter-day precision of the quality control samples at low, medium and high concentration levels exhibited relative standard deviations (RSD)<10%. Freezing-thawing stability was also tested; additionally pharmacokinetic and pharmacodynamics co-relation of the studied drugs in animal model has been done. The developed method can be easily used during accurate monitoring of the studied drugs.

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