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Ultra-reliable LC-MS methods: Moving mass spectrometry to the core laboratory

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Many clinical laboratory assays are performed every day at high volumes with only limited numbers of calibration standards. This has lead us to ask whether mass spectrometry based assays are stable enough to perform routine patient sample testing without the need for analyzing multiple calibration standards in a batch-wise fashion. To examine overall assay stability, we categorize sources of variability into ten separate categories (e.g., pipetting and integration errors) and then examine options for reducing overall variability. For example, by making use of a weighted calibration update strategy we have shown that, over a seven month period, we are able to reduce the number of calibration standards analyzed by 80%, over a conventional approach, yet with improved precision for three androgen analytes. The weighting strategy effectively reduces the variance around the calibration curve slope used for quantitation. A second source of variability examined is that resulting from the mass spectrometric determination of analytes to internal standard ratio. In general, we expect this ratio to remain constant during the analytical process but recent studies have shown that partial chromatographic separation of deuterated internal standards can result in differential suppression effects and nonlinear curves. In addition, data presented here shows that the mass spectrometer itself may not always yield the constant area ratios particularly when deuterated internal standards are used. In comparison, we found that 13C labeled internal standards behave better, not only chromatographically, but in terms of MS measurement as well.

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Differential systemic exposure to galangin after oral and intravenous administration to rats

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Galangin (3,5,7-trihydroxyflavone) is present in high concentrations in Alpinia officinarum Hance and it shows multifaceted *in vitro* and *in vivo* biological activities. The number and position of hydroxyl groups in this molecule play an important role in these biological activities. In order to systemic clarify the exposure of galangin after oral and intravenous administration to rats, two LC-MS/MS methods were developed and validated and successfully applied to analyze the parent drug molecules and aglycones liberated from plasma samples via β-glucuronidase hydrolysis. Our major findings were as follows: (1) The routes of administration showed significant influences on the systemic exposure of galangin and its metabolites; (2) Galangin was preferentially glucuronidated after p.o. dosing but sulfated after i.v. medication; (3) Kaempferol conjugates were detected demonstrating that oxidation reaction occurred; however, both glucuronidation and sulfation were more efficient; and (4) Oral bioavailability of free parent galangin was very low. The results showed that systemic exposure to galangin and its metabolites was different in rat plasma between oral and intravenous administration. Further research is needed to characterize the structures of galangin conjugates and to evaluate the biological activities of these metabolites.

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