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Workflow improvements for the Thermo Scientific™ Orbitrap Exploris™ Isotope Solutions

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Orbitrap-based Isotope Ratio MS is becoming increasingly visible in the community as a unique and complimentary approach to classical IRMS techniques for measuring relative abundances of isotopically substituted species. Electrospray ionization (ESI) offers the specific advantage of performing “soft” ionization, which produces intact molecular ions and provides unique insight into the molecular anatomy of polar compounds in aqueous solutions.

Drift associated with ESI ionization can be a major factor in data quality including the ultimate precision and accuracy achieved. Correcting for drift while measuring isotope ratios by ESI is done by sample-standard bracketing. This is currently achieved by one of two approaches. The dual syringe inlet approach uses a diverter valve switching between two syringes filled with sample or standard solution, enabling time efficient analysis. This method requires as little as 20 seconds of unusable time between samples and standards, allowing for close temporal measurement of a reference material. The major drawback of this approach is the necessity for the operator to be present to load the syringes. The second approach automizes the alternating injection of sample and standard solution using HPLC autosamplers. This approach enables fully automated analyses while lacking the temporal proximity of sample and standard seen with the dual syringe inlet. Cleaning and refilling of the autosampler loop causes a major gap (ca. minutes) of unused time between injections.

Here we will showcase a new workflow for isotope ratio standardization combining the best features from dual syringe inlet and autosampler injections. This method utilizes two flows of liquid controlled by a switching valve: one flow from an HPLC autosampler for sample and standard introduction, and a second flow is for a reference material. This setup allows for the injection of a reference material during the washout period of the HPLC, which reduces wasted time while simultaneously improving the quality of the measurement. This improved sample introduction technique enables efficient and fully automated sample analysis. Precision and accuracy are verified by drift correction and 1- or 2-point calibration. The novel approach will be demonstrated for inorganic (sulfate) and organic (vanillin) samples.