Avoid Mycotoxin Quantitation Errors When Using Stable Isotope Dilution Assay (SIDA)

There are significant benefits to using the stable isotope dilution assay (SIDA) calibration approach for mycotoxin analyses [1], and one of the biggest is being able to use a single calibration curve for multiple matrix types. Compared to making individual matrix-matched calibration standards for each sample type, the time savings offered by the SIDA technique can make up for the relatively high price of isotopically labeled internal standards (IS), especially for high-throughput food safety laboratories that frequently deal with a wide variety of samples. However, labeled standards are not always commercially available for every mycotoxin of interest. Due to these cost and availability issues, labs may be interested in using a practice that is common among many other types of methods that use internal standards: using the same internal standard to calibrate a group of analytes. That approach may seem logical, but in the case of mycotoxin SIDA methods, it can lead to significant errors.

Table I compares the observed concentrations of four different mycotoxins present in two maize flour reference materials to the concentrations reported by the reference material supplier. For the three mycotoxins that were quantified using their corresponding isotopically labeled internal standards, the agreement between the two values is excellent. However, in the case of the mycotoxin zearalenone, we did not have its analogous labeled internal standard, so we quantified it using 13C17-aflatoxin G1 because it elutes nearby. As the data show, the agreement between the observed results and those reported by the reference material supplier is very poor, which illustrates that similar chromatographic retention alone is not enough to predict the effects of sample preparation and/or matrix-related changes in ionization efficiency. A more detailed description of Restek's research on the LC-MS/MS analysis of mycotoxins in various foods comparing SIDA to matrix-matched calibration is published in a peer-reviewed study [2].

In light of these results, it is strongly recommended that only matching isotopically labeled internal standards be used for quantification in mycotoxin SIDA methods. If matching internal standards are not available, other calibration approaches, such as matrix-matched calibration, should be used.

Table I: Mycotoxin SIDA calibration should only be used for matching analyte and IS pairs.

Reference Material	Analyte	IS	Measured Concentration (ng/g), n=3	Assigned Concentration (ng/g)	Percent Accuracy (RSD, %)
TET017RM	Deoxynivalenol	$^{13}C_{15}$-Deoxynivalenol	1867.9 ± 37.36	1971 ± 195	94.8 (2)
TET017RM	Aflatoxin B1	$^{13}C_{17}$-Aflatoxin B1	8.68 ± 0.434	9.49 ± 0.85	91.4 (5)
TET017RM	Ochratoxin A	$^{13}C_{20}$-Ochratoxin A	4.48 ± 0.134	4.81 ± 0.75	93.2 (3)
TET017RM	Zearalenone	$^{13}C_{17}$-Aflatoxin G1	31.26 ± 2.19[a]	231 ± 25	13.5 (7)[a]
T04301Q	Deoxynivalenol	$^{13}C_{15}$-Deoxynivalenol	639.7 ± 19.19	649 ± 222	98.6 (3)
T04301Q	Aflatoxin B1	$^{13}C_{17}$-Aflatoxin B1	8.69 ± 0.348	9.21 ± 4.05	94.4 (4)
T04301Q	Ochratoxin A	$^{13}C_{20}$-Ochratoxin A	2.81 ± 0.197	3.03 ± 1.33	92.6 (7)
T04301Q	Zearalenone	$^{13}C_{17}$-Aflatoxin G1	16.2 ± 0.810[a]	138.5 ± 59.6	11.7 (5)[a]

* Results quantified using a nonmatched labeled IS.

References

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2. D. Li, J.A. Steimling, J.D. Konschnik, S. Grossman, T. Kahler, Quantitation of mycotoxins in four food matrices comparing stable isotope dilution assay (SIDA) with matrix-matched calibration methods by LC–MS/MS, J. AOAC Int. (2019) DOI: 10.5740/jaoacint.19-0028 https://doi.org/10.5740/jaoacint.19-0028

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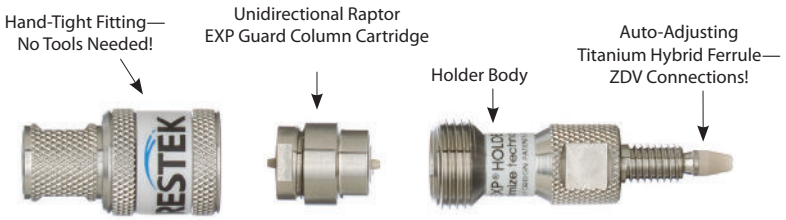

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