Analytical Tools for the Dietary Supplement and Food Laboratory

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Introduction: One of the most significant questions in the dietary supplement analytical laboratory is "how do I know that my method is working"? While linearity and precision tests can provide valuable information about the performance of analytical methods and spike recovery studies may uncover unresolved peaks, it can be very difficult to evaluate how well an analytical method works with a complex sample. Analyte stability, extraction efficiency, and availability of calibration materials and appropriate analytical controls all contribute to the difficulty. The National Institute of Standards and Technology (NIST) in collaboration with the National Institutes of Health (NIH)-Office of Dietary Supplements (ODS) have developed several tools to help laboratories in the dietary supplement and food industries improve measurement capabilities. The tools include calibration solution SRMs, natural matrix SRMs, and a Dietary Supplement Laboratory Quality Assurance Program.

Calibration Solution SRMs: Good quality calibration materials are expensive and difficult to qualify, however, without them, it is impossible to make good measurements. NIST has a number of dietary supplement calibration solutions available or in preparation, including organic acids, catechins, naphthodianthrones, and isoflavones. Prior to the production of the SRM solutions, the purity of the crystalline materials (including residual solvents and moisture analysis) is carefully determined. Next, the solution is prepared gravimetrically, ampouled in amber vials blanketed with inert gas, and stored in conditions set to maximize stability. Finally, the concentration of the analytes in solution is measured relative to independently prepared calibration solutions with either internal or external standard approaches. If the results from the gravimetric solution preparation and instrumental determination match, a statistical analysis is issued, and a certificate of analysis is prepared with certified values and uncertainties. Solution SRMs are intended for use in method development, for quality control checks, and as calibration standards (please note: a solution should not be used as a quality control check and a calibration standard at the same time).

Natural Matrix SRMs: Natural matrix SRMs for dietary supplement analysis have been designed in suites of materials. The SRMs are not meant to represent the "ideal" dietary supplement product, instead, they are meant to encompass the range of analytical challenges. For example, an upcoming soy suite includes soy protein isolate, soy protein concentrate, soy-containing tablets, soy milk, and soy flour. The different processing of each of the materials results in different analyte levels and ratios, as well as different extraction issues and interfering chromatographic peaks. As with solution SRMs, natural matrix SRMs require at least two independent methods for the certification of each analyte. The methods are designed to be as different as possible from the sample extraction/preparation steps through the instrumental method (e.g. changing chromatographic selectivity). The data from the two or more methods including data from collaborating laboratories (when available) are then used to determine the amount of analyte in the matrix and the associated uncertainty. Natural matrix SRMs are intended for use in method development, method validation, and as quality control checks. Typically, they should not be used for calibration as there is usually an extraction step required prior to analysis.

Dietary Supplement Laboratory Quality Assurance Program (DSQAP): In 2007 NIST established the NIST/NIH DSQAP, which is a free, anonymous interlaboratory comparison program. Twice a year, 3 to 6 sets of samples are sent to participants for measurement of analytes such as toxic elements, nutritional elements, fat- and water-soluble vitamins, fatty acids, and botanical compounds. After the data are returned, a report containing consensus and target ranges (where appropriate) as well as coded individual participant data and recommendations for improvement is prepared and distributed to participants. The reporting style is similar to that which is seen in proficiency testing programs; however, pass/fail scores are not issued. In addition to assessing individual laboratory performance, through careful selection of samples problem areas can be identified, such as the difficulty extracting phytosterols from *Serenoa repens* fruit or the importance of selecting high quality niacinamide standards. In addition to providing reports, the DSQAP team holds workshops to help analysts improve their skills and to help the community identify and rectify measurement problems.

Conclusion: Through the use of SRMs and the DSQAP it is possible for dietary supplement and food laboratories to evaluate method performance. Matrix-based reference materials allow for additional method assessment from sample extraction through instrumental analysis. If the certified values from an SRM are obtained using a given method, that method is likely to perform well for similar sample types; if not, there is a problem with some part of the method and it may possible to determine the source of the error. Participation in interlaboratory comparison programs such as the DSQAP can be used to demonstrate the use of "appropriate" analytical methods to investigators and to improve analytical performance. For more information about SRMs visit http://www.nist.gov/srm/index.cfm and to learn more about the DSQAP visit http://www.nist.gov/mml/analytical/dsqaprogram.cfm or send an e-mail to dsqap@nist.gov.