

| Method Title  | Ingredient      | ERP Decision | Reviewers        | Notes to Method Author   | Date Sent                           | Response?  |
|---|-----------------|--------------|------------------|--|-------------------------------------|--|
| "Selected Adulterants in Dietary Ingredients and Dietary Supplements Containing Chondroitin Sulfate"  | chondroitin     | Not Adopted  | Hildreth/Phinney | method author should submit probability of detection (POD) data at the minimum detection level for the compounds listed in Annex I of AOAC SMPR 2014.008.  | Thursday, October 08, 2015 10:47 AM | None   |
| Isotachophoretic Determination of GA and CS in Dietary Supplements  | chondroitin     | Not Adopted  | Reins/Hildreth   | none   | Thursday, October 08, 2015 10:47 AM | Thank you for the report of ERP. Unfortunately the ITP method did not move to the First Action Official Methods status. Would you mind to send me reviewers reports of CHON-002? Could you send me the method for CS determination when it will be proved as official one? |
| Chondroitin by IR and Dimethylmethylene Blue (DMMB) Spectrophotometry   | chondroitin     | Not Adopted  | Solyom/Es Safi   | ask the method author whether they can provide additional data regarding the interfering compounds that are listed on the SMPR008 and if they have additional data for system suitability / all validation data.   | Thursday, October 08, 2015 10:47 AM | None   |
| Determination of Chondroitin Sulfate Content in Raw Materials and Dietary Supplements by High-Performance Liquid Chromatography with Ultraviolet Detection After Enzymatic Hydrolysis | chondroitin     | First Action | Sullivan/Koerner | MOTION to be considered for Final Action Official MethodsSM status, the author(s) should: <ul style="list-style-type: none"> <li>• Optimize and control the moisture in the chondroitin sulfate including appropriate vessels and glassware.</li> <li>• Investigate alternative LC columns.</li> <li>• Optimize the LC conditions.</li> <li>• Look at lessons learned from USP.</li> <li>• Include a potency evaluation of the enzyme use.</li> <li>• Investigate use of the USP standard that is currently available.</li> <li>• Certified reference material recommended.</li> </ul> | Thursday, October 08, 2015 10:47 AM | Moved through Publications Department to First Action  |
| Determination of Anthocyanins in Brazilian and Floridian Açai (Euterpe oleraceae Mart) Using LC-MS/MS   | anthocyanins    | Not Adopted  | Lee/Phillips     | no further action  | Thursday, October 08, 2015 10:47 AM | None   |
| Total Monomeric Anthocyanins By HPLC  | anthocyanins    | Not Adopted  | Solyom/Jennings  | no further action  | Thursday, October 08, 2015 10:47 AM | None   |
| Anthocyanin Profiles by HPLC with DAD and MS Detections   | anthocyanins    | Not Adopted  | Szpylka          | no further action  | Thursday, October 08, 2015 10:47 AM | None   |
| Screening Method for Phosphodiesterase Type 5 (PDE5) Inhibitors in Dietary Ingredients and Supplements using High Resolution Mass Spectrometry  | PDE5 Inhibitors | Not Adopted  | Phillips T.      | would like to see a further submission of data demonstrating applicability towards the SMPR 2014.012.  | Thursday, October 08, 2015 10:47 AM | Telecon held October 8 at 2:00 pm to discuss results. No further action.   |
| Screening Method for Phosphodiesterase Type 5 (PDE5) Inhibitors in Dietary Ingredients and Supplements  | PDE5 Inhibitors | Not Adopted  | Mastovska        | would like to see a further submission of data demonstrating applicability towards the SMPR 2014.012.  | Thursday, October 08, 2015 10:47 AM | None   |
| Adaption of the LC-MS Screen for PDE5 Inhibitors to UHPLC-MS  | PDE5 Inhibitors | Not Adopted  | Koerner/Szpylka  | this method should: <ol style="list-style-type: none"> <li>1. Supply single lab validation data related to LOD.</li> <li>2. Supply LIB data if available, and any other data that exists in support of this method's applicability to the SMPR 2014.012 or 2014.010.</li> </ol>  | Thursday, October 08, 2015 10:47 AM | None from authors; Nickum had another PDE5 Method that did not get reviewed due to administrative error. AOAC has this method on file for a future review if possible.   |
| Testing for Phosphodiesterase Type 5 (PDE5) Inhibitors in Dietary Supplements   | PDE5 Inhibitors | Not Adopted  | Zweigenbaum/Cain | would like to see a further submission of data demonstrating applicability towards the SMPR 2014.012.  | Thursday, October 08, 2015 10:47 AM | None - Author present for ERP but no formal response to ERP Report   |

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|---|-----------------|--------------|---------------------------|--|-------------------------------------|--|
| SLV Study of a Method for Screening   | PDE5 Inhibitors | First Action | Cain/Phillips, T.         | the method author(s) should:<br>1. Provide reproducibility data<br>2. Break out AOAC SMPR 2014.011:<br>-Matrix match spike recovery<br>-Recovery options<br>3. Break out AOAC SMPR 2014.012 and AOAC SMPR 2014.010.<br>-Feedback from other users and a mechanism to receive that feedback.  | Thursday, October 08, 2015 10:47 AM | Moved through Publications Department to First Action    |
| Withanolide Glycosides and Aglycones of Ashwagandha (W. somnifera)                        | Ashwagandha     | First Action | Young/ Bzhelyansky/ Ingle | The ERP recommended the method author complete the following actions to be completed prior to Final Action consideration: <ul style="list-style-type: none"> <li>Investigate a better baseline separation for Peak 1</li> <li>pH of phosphate buffer</li> <li>Column temperature</li> <li>Heat stability during the extraction</li> <li>Simplify the preparation of the extract products</li> <li>Limit number of reference standards to one withanolide and one withanoside with use of response factors</li> <li>Provide data on finished products</li> <li>When specifying columns put "or equivalent."</li> <li>Clarify the discard volume in the filtration step</li> <li>Provide data on method LOQ</li> </ul> | Wednesday, January 13, 2016 4:44 PM | Moved through Publications Department to First Action No |
| Folin-Ciocalteu Reagent for Polyphenolic Assay  | Folin C         | Not Adopted  | Finley/ Lee               | the following actions must be completed before this method can be reconsidered for First Action Official Methods status: <ul style="list-style-type: none"> <li>Clarify benefits of single vs. dual; ask which of the two is being submitted</li> <li>Clarification on purpose of the cleanup steps</li> <li>Clarify the major differences in the data between the two methods.</li> <li>Ask if there is data on sample matrices addressed in SMPR</li> <li>Need data on reference materials (available from Kate Rimmer, NIST)</li> <li>Use Gallic Acid for calibrant</li> <li>Provide raw data</li> </ul>  | Wednesday, January 13, 2016 4:44 PM | None   |
| Method for the estimation of total phenolic content using the Folin-C assay               | Folin C         | Not Adopted  | Phillips T./ Solyom       | the following actions must be completed before this method can be reconsidered for First Action Official Methods status: <ul style="list-style-type: none"> <li>Need data on reference materials (available from Catherine Rimmer, NIST)</li> <li>Ask if there is data on sample matrices addressed in SMPR</li> <li>Expand analytical range</li> <li>Data not completely clear</li> <li>More supporting data for recovery and LOQ</li> </ul>  | Wednesday, January 13, 2016 4:44 PM | None   |
| Modified Folin-Ciocalteu Antioxidant Capacity Assay for Measuring Lipophilic Antioxidants | Folin C         | Not Adopted  | Zhou/ Krueger             | the following actions must be completed before this method can be reconsidered for First Action Official Methods status: <ul style="list-style-type: none"> <li>Need more data to meet SMPR</li> <li>Need data on reference materials (available from Catherine Rimmer, NIST)</li> <li>Ask if there is data on sample matrices addressed in SMPR</li> <li>Express as Gallic Acid equivalent</li> <li>Comparative data between traditional folin and modified folin between a range of matrices is required.</li> </ul>   | Wednesday, January 13, 2016 4:44 PM | None   |

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| Quantitative and Qualitative Analysis of Mitragynine in Kratom ( <i>Mitragyna speciosa</i> ) by GC-MS, LC-MS/MS and UPLC-PDA   | Kratom   | Not Adopted  | Wang/ Phillips, T.           | <ul style="list-style-type: none"> <li>Commercially available standards from Chromadex for 7 OH</li> <li>Need to see recovery data</li> <li>Include 7 OH data</li> <li>Range of quantification must cover whole range from SMPR</li> <li>Precision and accuracy data is needed across entire range of quantification</li> <li>Interest from ERP is in the tandem MS method</li> </ul>  | Wednesday, January 13, 2016 4:44 PM | None  |
| Quantification of Mitragynine in Kratom Raw Materials and Finished Products by High-Performance Liquid Chromatography: Singly Laboratory Validation  | Kratom   | Not Adopted  | Es-Safi/ Metcalfe            | <ul style="list-style-type: none"> <li>LOD and LOQ information for 7-OH required.</li> <li>Repeatability must be improved</li> <li>More accuracy and recovery data</li> <li>Peak purity PDA</li> <li>Examine additional wavelength data</li> </ul>   | Wednesday, January 13, 2016 4:44 PM | None (Mudge was present and took notes but not formal response from BCIT on ERP Report) |
| Identification and Characterization of Indole and Oxindole Alkaloids from Leaves of <i>Mitragyna speciosa</i> Korth Using Liquid Chromatography-Accurate QToF Mass Spectrometry                      | Kratom   | Not Adopted  | Phillips, T./ Casey/ Szyplka | Need precision and accuracy to demonstrate compliance with the SMPR.   | Wednesday, January 13, 2016 4:44 PM | None  |
| LC/MS Method for the Identification of <i>Mitragyna speciosa</i> (Kratom) and Quantitation of Mitragynine Using Linear Ion Trap Mass Spectrometer  | Kratom   | Not Adopted  | Wang/ Rimmer                 | <ul style="list-style-type: none"> <li>Feedback on recoveries outside the SMPR</li> <li>Demonstration of precision data</li> <li>Additional details around sample preparation</li> <li>Clarification of ability to measure 7-OH</li> <li>Clarify the procedure used to gather accuracy data</li> <li>Calibration curve range needs to be defined.</li> </ul>   | Wednesday, January 13, 2016 4:44 PM | None  |
| Determination of Aloin A and Aloin B in Aloe vera Raw Materials and Finished Products by High-Performance Liquid Chromatography: Single-Laboratory Validation  | Aloin    | Not Adopted  | Lavoie/ ERP                  | <ul style="list-style-type: none"> <li>Is there data on additional matrices?</li> <li>Raw data used for system suitability?</li> <li>Raw data for peak purity data (diode array)</li> <li>Is there data demonstrating limit of quantitation using the target analyte?</li> <li>What was done to HPLC system, was anything done to optimize the system against the column sizes?</li> </ul>   | ERP REPORT NOT YET ISSUED           |   |
| Determination of Aloin A, Aloin B, and Aloe-emodin in Raw Materials and Finished Products by HPLC  | Aloin    | First Action | Johnson/ ERP                 | <ul style="list-style-type: none"> <li>Final Action Requirements</li> <li>demonstrate 5ppb LOQ</li> <li>Correct typo in tables for accuracy and recovery</li> <li>Provide data for other matrices i.e. repeatability</li> <li>Recognition of column temperature control</li> <li>Data clarifying why the lambda max was not used</li> </ul>  | ERP REPORT NOT YET ISSUED           |   |
| Identification of Selected <i>Cinnamomum</i> spp. Bark in Dietary Supplement Raw Materials and/or Finished Products - Gas Chromatography with Flame Ionization Detection After Hydrodistillation     | Cinnamon | Not Adopted  | Phillips, T./ ERP            | <ul style="list-style-type: none"> <li>Provide exclusivity panel data</li> <li>Include the <i>C. tamala</i> data</li> <li>Provide the 33 replicates of each based on SMPR</li> <li>Cross reference species names</li> <li>Further explanation on the software</li> <li>Data on choice of marker compounds for each species (reference)</li> <li>Inclusion of the 7 commercially available compounds</li> <li>Provide representative chromatograms</li> </ul> | ERP REPORT NOT YET ISSUED           |   |
| Analysis of Theanine in Tea ( <i>Camellia sinensis</i> ) Dietary Ingredients and Supplements by High-Performance Liquid Chromatography with Post-Column Derivatization: Single Laboratory Validation | Tea      | First Action | Phillips, M. / ERP           | <ul style="list-style-type: none"> <li>Expanded matrix testing data for chewables and gummies</li> <li>Analyte stability temperature during sample prep</li> <li>Guidance on sample prep - how much sample to use?</li> <li>Check glycine-dimer as possible internal standard</li> <li>Provide calibration curve and the chromatogram of the standard</li> </ul>   | ERP REPORT NOT YET ISSUED           |   |

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| Determination of Catechins and Caffeine in Camillia sinensis Raw Materials, Extracts, and Dietary Supplements by HPLC-UV: Single-Laboratory Validation | Tea | Not Adopted | Solyom/ ERP | First Action Requirements <ul style="list-style-type: none"> <li>• Low level accuracy study and the precision study</li> <li>• Other matrices in SMPR that are not addressed</li> <li>• Add catechin gallate and methyl xanthenes</li> <li>• Address gallic acid interference.</li> <li>• Change blank matrix for recovery study</li> <li>• Confirm sample prep temperature impact</li> <li>• Filtration step validation</li> </ul> | ERP REPORT NOT YET ISSUED |  |
|--|-----|-------------|-------------|---|---------------------------|--|