Standard Method Performance Requirements for Screening Method for Selected Adulterants in Dietary Ingredients and Supplements Containing Chondroitin Sulfate

Intended Use: Routine Surveillance of Dietary Ingredients and Products by a Trained Technician

1 Purpose

AOAC Standard Method Performance RequirementsSM (SMPRs) describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, a single-laboratory validation, or a multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholder panels composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels in their evaluation of validation study data for method being considered for Performance Tested MethodsSM or AOAC Official Methods of AnalysisSM, and can be used as acceptance criteria for verification at user laboratories. [Refer to Appendix F: Guidelines for Standard Method Performance Requirements, Official Methods of Analysis of AOAC INTERNATIONAL (2012) 19th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, USA.]

2 Applicability

Screening method for selected adulterants (as identified in Annex I: Target Compounds) in dietary ingredients and supplements claiming to contain chondroitin sulfate.

3 Analytical Technique

Any analytical technique(s) that detects the analytes of interest and meets the following method performance requirements is/are acceptable. It is acceptable to have a different analytical method for each class of analytes.

4 Definitions

Adulterant.—Any poisonous or deleterious substance which may render a product injurious to users under the conditions of use prescribed in the labeling thereof; or any valuable constituent that has been substituted wholly or in part; or a substance that has been added so as to increase the bulk or weight, or reduce the quality or strength, or make a product appear better or of greater value than it is. (United States Code of Federal Regulations Title 21 §402)

Chondroitin sulfate (CS).—CS salts consist mostly of the salts of the sulfate ester of N-acetylgalactosamine (2-acetamido-2-deoxy-d-galactopyranose usually abbreviated as GalNAc) and d-glucuronic acid copolymer. These hexoses are alternately linked -1,4 and -1,3 in the polymer. It is closely related to other glycosaminoglycans (GAGs), such as dermatan sulfate, hyaluronic acid, heparin, heparan sulfate, and keratan sulfate, which contain other hexosamines and/or glycuronic acid residues. Either of the residues can be sulfated at different positions. (See Figure 1.)

Dietary ingredients.—A vitamin; a mineral; an herb or other botanical; an amino acid; a dietary substance for use by man to supplement the diet by increasing total dietary intake; or a concentrate, metabolite, constituent, extract, or combination of any of the above dietary ingredients. [United States Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)]]

Dietary supplements.—A product intended for ingestion that contains a “dietary ingredient” intended to add further nutritional value to (supplement) the diet. Dietary supplements may be found in many forms such as tablets, capsules, softgels, gelcaps, liquids, or powders.

Laboratory probability of detection (LPOD).—The POD value obtained from combining all valid collaborator data sets for a method for a given matrix at a given analyte level or concentration. [Appendix H: Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods, Official Methods of Analysis of AOAC INTERNATIONAL (2012) 19th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, USA]

Probability of detection (POD).—(Ibid) The proportion of positive analytical outcomes for a qualitative method for a given matrix at a given analyte level or concentration.

5 Method Performance Requirements

See Table 1.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blank check samples, and check standards at the lowest point and midrange point of the analytical range, and a protocol to demonstrate suitability.

7 Reference Material(s)


ISO Guide 34:2009 General requirements for the competence of reference material producers

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**Table 1. Method performance requirements**

<table>
<thead>
<tr>
<th>Type of study</th>
<th>Parameter</th>
<th>Parameter requirements</th>
<th>Target test concn, % (w/w)</th>
<th>Minimum acceptable results</th>
</tr>
</thead>
<tbody>
<tr>
<td>Single-laboratory</td>
<td>Matrix</td>
<td>Minimum of 33 replicates representing ideally all target compounds in Annex I and all matrix types listed in Annex II, spiked at or below the designated low-level target test concentration</td>
<td>≤5</td>
<td>90% POD(^a) of the pooled data for all target compounds and matrices</td>
</tr>
<tr>
<td>validation</td>
<td>studies</td>
<td>High concentration. Minimum of five replicates per matrix type spiked at the designated high-level target test concentration.</td>
<td>ca 20</td>
<td>100% correct analyses are expected per matrix type(^c)</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Zero concentration. Minimum of five replicates per matrix type that have tested negative with a second method and have not been spiked.</td>
<td>0</td>
<td></td>
</tr>
<tr>
<td>Multilaboratory study</td>
<td>LPOD(^b)</td>
<td>Use Appendix N: ISPAM Guidelines for Validation of Qualitative Binary Chemistry Methods</td>
<td>≤5</td>
<td>≥0.85</td>
</tr>
<tr>
<td></td>
<td>LPOD(_{0})</td>
<td></td>
<td>ca 20</td>
<td>≥0.95</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>0</td>
<td>≤0.05</td>
</tr>
</tbody>
</table>

\(^a\) 95% confidence interval.

\(^b\) 100% correct analyses are expected. Some aberrations may be acceptable if the aberrations are investigated, and acceptable explanations can be determined and communicated to method users.

\(^c\) LPOD and LPOD\(_{0}\) are not required for First Action Official Methods of Analysis approval.

**8 Validation Guidance**

Ideally all target compounds in Annex I and all matrices in Annex II shall be evaluated.


**9 Maximum Time-to-Result**

No maximum time to result.

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