Standard Method Performance Requirements for Determination of Aloin A and Aloin B in Dietary Supplement Products and Ingredients

1 Applicability

The method must be able to quantitate aloin A and aloin B separately in aloe vera leaf juice and dry juice ingredients, and dietary supplement finished products.

2 Analytical Technique

Any analytical technique that meets the following method performance requirements is acceptable.

3 Definitions

Aloin A.—1,8-Dihydroxy-10-(β-D-glucopyranosyl)-3-(hydroxymethyl)-9(10*H*)-anthracenone. Also known as barbaloin. *See* Figure 1. CAS No. 1415-73-2.

Aloin B.—(10*R*)-1,8-dihydroxy-3-(hydroxymethyl)-10-[(2*S*,3*R*,4*R*,5*S*,6*R*)-3,4,5-trihydroxy-6-hydroxymethyl)oxan-2-yl]-10*H*-anthracen-9-one. Also known as beta-D-isomer barbaloin or isobarbaloin. *See* Figure 2. CAS No. 28371-16-6.

Dietary ingredients.—Vitamin, mineral, 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)]}

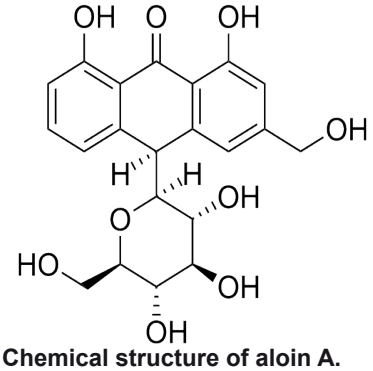

Figure 1. Chemical structure of aloin A.

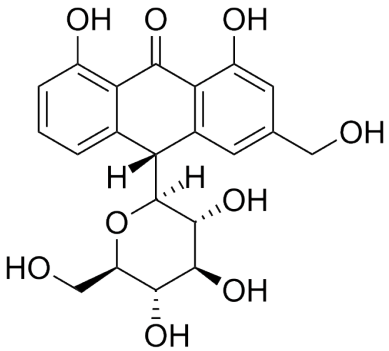

Figure 2. Chemical structure of aloin B.

Table 1. Analytical ranges and LOQ for aloin A or B

	Finished products	Raw material
Analytical range, ppm	0.01–100	0.01–12500
LOQ, ppm	0.005	0.005

Dietary supplements.—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, gels, softgels, gelcaps, liquids, or powders.

Limit of quantitation (LOQ).—Minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result.

Repeatability.—Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation (%RSD_r).

Reproducibility.—Standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation (% RSD_R).

Recovery.—Fraction or percentage of spiked analyte that is recovered when the test sample is analyzed using the entire method.

4 Method Performance Requirements

See Tables 1 and 2.

5 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. A control sample must be included.

6 Reference Material(s)

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements, Official Methods of Analysis* (current edition), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_f.pdf)

7 Validation Guidance

Recommended level of validation: *Official Methods of Analysis*^{SM}

Appendix D: *Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis, Official Methods of Analysis* (current edition), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_d.pdf)

Appendix N: *ISPAM Guidelines for Validation of Qualitative Binary Chemistry Methods, Official Methods of Analysis* (current edition), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_n.pdf)

Appendix K: *Guidelines for Dietary Supplements and Botanicals, Official Methods of Analysis* (current edition), AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_k.pdf). Also at: *J. AOAC Int*. **95**, 268(2012); DOI: 10.5740/jaoacint.11-447

8 Maximum Time-to-Signal

No maximum time.

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Table 2. Performance parameters for aloin A or B						
	Range, ppm					
	Finished product and ingredient				Ingredient	
Parameter	0.01–1	>1–10	>10–30	>30–100	>100–1000	>1000–12500
Repeatability (RSD_r), %	≤21	≤11	≤7	≤6	≤5	≤3
Recovery, %	60–115	80–110			90–107	95–105
Reproducibility (RSD_R), %	≤32	≤16	≤11	≤9	≤7	≤4