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3 **Method Name: Determination of Aloin A and Aloin B in Dietary Supplement**
4 **Products and Raw Materials**

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6 **Intended Use:**

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8 **1. Applicability:**

9 The method must be able to quantitate aloin A and aloin B separately in: raw materials;
10 dietary supplement finished products in liquid, gel, powder, tablet, and softgel matrices; and
11 aloe vera leaf juice and dry juice ingredients.

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14 **2. Analytical Technique:**

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

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18 **3. Definitions:**

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20 **Aloin A**

21 1,8-Dihydroxy-10-(β -D-glucopyranosyl)-3-(hydroxymethyl)-9(10H)-anthracenone. Also
22 known as barbaloin. See figure 1. CAS No.: 1415-73-2.

23
24 **Aloin B**

25 (10R)-1,8-dihydroxy-3-(hydroxymethyl)-10-[(2S,3R,4R,5S,6R)-3,4,5-trihydroxy-6-
26 hydroxymethyl]oxan-2-yl]-10H-anthracen-9-one. Also known as beta-D-isomer barbaloin
27 or isobarbaloin. See figure 2. CAS No.: 28371-16-6.

28
29 **Dietary ingredients.**

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

34
35 **Dietary supplements.—**

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

39
40 **Limit of Quantitation (LOQ)**

41 The minimum concentration or mass of analyte in a given matrix that can be reported as a
42 quantitative result

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44 **Repeatability**

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

51 Reproducibility
 52 The standard deviation or relative standard deviation calculated from among-laboratory
 53 data. Expressed as the reproducibility relative standard deviation (SD_R); or %
 54 reproducibility relative standard deviation (% RSD_R).
 55

56 Recovery
 57 The fraction or percentage of spiked analyte that is recovered when the test sample is
 58 analyzed using the entire method.
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60 **4. Method Performance Requirements:**

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 63 **5. METHOD PERFORMANCE REQUIREMENTS:**

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 65 **Table 1a: Analytical Ranges and LOQ for Aloin A or B**
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	Finished Products	Raw Material
Analytical range (ppm)	0.01 to 100	0.01 to 12,500
LOQ (ppm)	0.005	0.005

67
 68 **Table 1b: Performance Parameters for Aloin A or B**
 69

Parameter	ranges (ppm)					
	finished product and raw ingredients				raw ingredients	
	0.01 – 1	>1 – 10	>10 – 30	>30 – 100	>100 – 1000	>1000- 12,500
% 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

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 74 **6. System suitability tests and/or analytical quality control:**
 75 Suitable methods will include blank check samples, and check standards at the lowest point
 76 and midrange point of the analytical range. A control sample must be included.
 77
 78 **6. Reference Material(s):** Refer to Annex F of Appendix F in OMA. Refer to 19th Edition of the
 79 AOAC INTERNATIONAL Official Methods of Analysis (2012). Annex F: *Development and Use*
 80 *of In-House Reference Materials* in [Appendix F: Guidelines for Standard Method Performance](http://www.eoma.aoc.org/app_f.pdf)
 81 *Requirements*. Available at: http://www.eoma.aoc.org/app_f.pdf
 82
 83 **7. Validation Guidance:**
 84 Recommended level of validation: *Official Methods of Analysis*SM

85 [Appendix D](#): Guidelines for Collaborative Study Procedures To Validate Characteristics of a
86 Method of Analysis; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis
87 (2012). Available at: http://www.eoma.aoac.org/app_d.pdf
88

89 [Appendix N](#): ISPAM Guidelines for Validation of Qualitative Binary Chemistry Methods of the
90 19th edition of the *AOAC INTERNATIONAL Official Methods of Analysis*. Available at
91 http://www.eoma.aoac.org/app_n.pdf
92

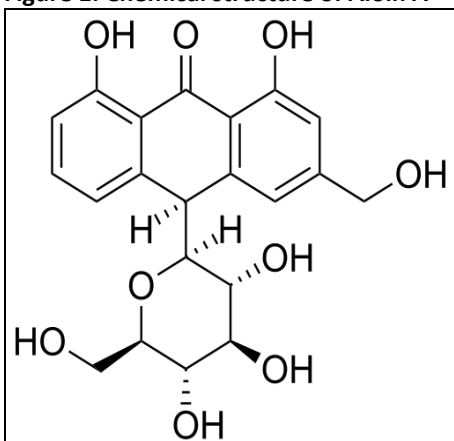
93 [Appendix K](#): Guidelines for Dietary Supplements and Botanicals 19th Edition of the AOAC
94 INTERNATIONAL Official Methods of Analysis (2012). Also at: . AOAC Int. 95, 268(2012); DOI:
95 10.5740/jaoacint.11-447 and available at: http://www.eoma.aoac.org/app_k.pdf
96

97 **8. Maximum Time-To-Signal:** No maximum time.
98

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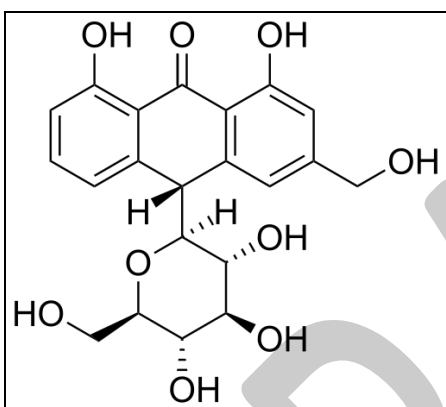
Figure 1: Chemical structure of Aloin A



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Figure 2: Chemical structure of Aloin B



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