DRAFT AOAC SMPR 2016.XXX; Version 5; November 29, 2016

Method Name: Quantitation of proanthocyanidin content in cranberry fruit, juice, beverage, dried cranberry, cranberry sauce, ingredients (concentrates, extracts and powders) and dietary supplement formulations.

Intended Use: Reference method for cGMP compliance.

1. **Purpose:** AOAC 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 the 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 Methods* or AOAC *Official Methods of Analysis*, and can be used as acceptance criteria for verification at user laboratories.

2. **Applicability**:

The method will be able to quantify total proanthocyanidin content as the sum of all extractable oligomers (> DP2) and polymers present in cranberry (*Vaccinium macrocarpon*) fruit, juice, beverage, dried cranberry fruit, cranberry sauce, ingredients (concentrates, extracts, powders, and presscake) or dietary supplements (Table 3).

3. **Analytical Technique**:

Any analytical technique(s) that measures the analytes of interest and meets the following method performance requirements is/are acceptable.

4. **Definitions**:

Cranberry Proanthocyanadins

A mixture of oligomeric and polymeric flavan-3-ols, primarily epicatechin and catechin, of the A and B type.

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.[1]

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.

Limit of Quantitation (LOQ)

[1] Federal Food Drug and Cosmetic Act §201(ff) [U.S.C. 321 (ff)

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

Quantitative method
Method of analysis which response is the amount of the analyte measured either directly (enumeration in a mass or a volume), or indirectly (color, absorbance, impedance, etc.) in a certain amount of sample.

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
The 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
The fraction or percentage of spiked analyte that is recovered when the test sample is analyzed using the entire method.

5. **Method Performance Requirements**:
See table 1 and 2.

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.

7. **Reference Material(s):**

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

8. **Validation Guidance**:

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

Appendix F: Guidelines for Standard Method Performance Requirements; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available at: http://www.eoma.aoac.org/app_f.pdf

Appendix K: Guidelines for Dietary Supplements and Botanicals; 19th Edition of the AOAC INTERNATIONAL Official Methods of Analysis (2012). Available on line at: http://www.eoma.aoac.org/app_k.pdf

9. Maximum Time-To-Result: None

Table 1: Method performance requirements (part 1)

Parameter	**Requirement**
Limit of Quantitation (LOQ) (%)	≤ 0.01
Analytical Range (%)	≤ 0.03 – 55

Table 2: Method performance requirements (part 2)

	Liquids		Solids	
Ranges	0.03 – 15%	> 15% - 55%	0.03 – 15 %	> 15% - 55%
Recovery (%)	97 – 103	97 – 103	90 - 107	97 – 103
% RSD_r	< 10	< 5	< 15	< 10
% RSD_R	< 15	< 8	< 20	< 15

Table 3: Examples of Dietary Supplements

capsules
extracts
liquids
powders
softgel capsules
tablets
tinctures
gummies