Report to Community on the
Stakeholder Panel on Dietary Supplements (SPDS),
held on September 16, 2016, Sheraton Dallas Hotel, Dallas, TX

The very production Dietary Supplement Stakeholder Panel considered 4 SPMPRs and launched 4 working groups. A table of all the actions taken thus far was provided by AOAC staff and is attached.

First Action Methods:

- Chondroitin,
- PDES Inhibitor,
- Ashwagandha,
- Aloin
- Tea

SMPRs:

- Authentication of Vaccinium species,
- Determination of total Chondroitin sulfate,
- Adulterants in Chondroitin,
- Identification and determination of Phosphodiesterase type 5 (PDE5),
- Glycosides and Aglycons of Ashwagandha,
- Alkaloids of Kratom,
- Total phenolic content Folin-C Assay,
- ID of Cinnamonum bark,
- Multiple components in Tea supplements
- Determination of Aloin A & B
- Determination of Vitamin D

1. The Vitamin D SMPR was revised to allow multiple methods to be submitted.
   ERP Chair, John Austad reported that no methods had been submitted for both pre D, D2 and D3. This will go back to the ERP for a call for methods again.

2. Aloe Vera Working Group presented an SMPR: (Kan He, Herbalife and John Edwards, Process NMR Associates; Co-Chairs)
   - There was a discussion concerning the possible need for a method for identification (is it authentic aloe) and another for quantitation.
   - There is a need for polysaccharide standards.
   - There is a need to detect and quantitative isocitric acid to identify the source (whole leaf, rind or gel).

- The SMPR proposed to determine total aloe vera but also lists multiple possible adulterants that should not be included.
- It is not clear if the method should identify and quantitate both components and adulterants in the same method.
- The working group will go back to discuss.

3. Protein Working Group presented SMPR: (Spencer Carter, Genesys Labs)
    - A discussion of the need for identification and quantitation in both animal and plant products.
    - The SMPR proposes to detect polypeptides greater than 10,000 daltons.
    - A new definition of protein was proposed by the food industry; "Polymeric chains of amino acid residues connected with peptide bonds".
    - Four SMPRs were approved and sent to ERP: animal and non-animal; identification and quantitation.

4. Vitamin B12 Working Group presented SMPR: (Richard van Breemen, University of Illinois)

    - Must distinguish active vitamin B12 corrinoids (Methylcobalamin, Cyanocobalamin, Adenosylcobalamin, Hydroxocobalamin) from inactive forms at 0.001 ppm to 100% in multiple finished supplements.
    - Stability statement added to SMPR: "Because some vitamin B12 forms are unstable, the stability of vitamin B12 in extracts should be demonstrated during analysis."
    - SMPR approved and sent to ERP

Launch 3 Working Groups

5. *Fit for Purpose* statement: Free Amino Acids, Garrett Zielinski, Covance
    - Many methods are currently available with the pre- and post- column derivitization HPLC seemed most commonly used by stakeholders.
    - There is a problem with supplement homogeneity and should be addressed in the method.
    - Additional compounds to quantitate and many possible interferences were discussed. These will be further clarified by the Working Group

A Fit for Purpose statement was provided and approved for the Working Group.

6. *Fit for Purpose* statement: Ginger, Anton Bzhelyansky, USP
   A very extensive review of the ginger types, constituents and analysis was provided.
    - The method must quantitate the 'pungent principles' derived from the rhizome of ginger, *Zingiber officinale* Roscoe [Fam. Zingiberaceae].
    - The method must quantitate, at a minimum, [6]-, [8]- and [10]-gingerols, and [6]-shogaols.
    - The method should preferably quantitate [8]- and [10]-shogaols, as well as [6]-, [8]- and [10]-paradols, [6]- and [10]-gingerdiols, [6]-, [8]- and [10]-gingerdiones, and zingerone.
    - Individual constituents should be quantifiable within the range of 0.01% - 50% by weight in powdered ginger rhizome, ginger rhizome dry and soft extracts, and

ginger-containing finished products, including capsules and tablets, in the presence of common excipients.
The ability to address softgels and tinctures is advantageous, yet optional.

A detailed Fit for Purpose statement was provided and approved for the Working Group.

7. *Fit for Purpose* statement: Vitamins K1 and K2, Inger Reidun, Kappa Bio

Analytes:
Vitamin K1 (phylloquinone, defined as the sum of *cis* and *trans* isomers)
Vitamin K2 (the menaquinone series, MK4 through MK14). MK4 and MK7 are the most well-studied menaquinones. Defined as all-*trans* K2-MK4 and all-*trans* K2-MK7.

The analytical range of the chosen method must encompass the vitamin K content in dietary supplements and their raw materials

- Dietary supplements 5-200 µg/dose
- Custom premixes
- Raw materials 0.1 -100%

Separate and accurately determine both vitamin K1 (phylloquinone) and K2 (different menaquinones)

- Determination of *trans*-K1 and *cis*-K1 (defined as the sum of *cis* and *trans* isomer of K1)
- Separate and accurately determine three different forms of K2 (MK4, MK6 and MK7)
- Determination of all-*trans*-MK4, all-*trans* MK6 and all-*trans* MK7. Many cis forms maybe
- present.
- Be able to analyze both coated and non-coated formulations
- Determine the above in raw materials used to produce/formulate dietary supplements
- A single, in-house method is desired
- Stability of analyte must be controlled (light, acid, base…)

A Fit for Purpose statement was drafted and approved for the working group.