Combating Food Fraud

How Consensus Standards and Official Methods of Analysis Overcome the Challenges Facing Food Authenticity

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Food Labs Conference
June 5, 2020

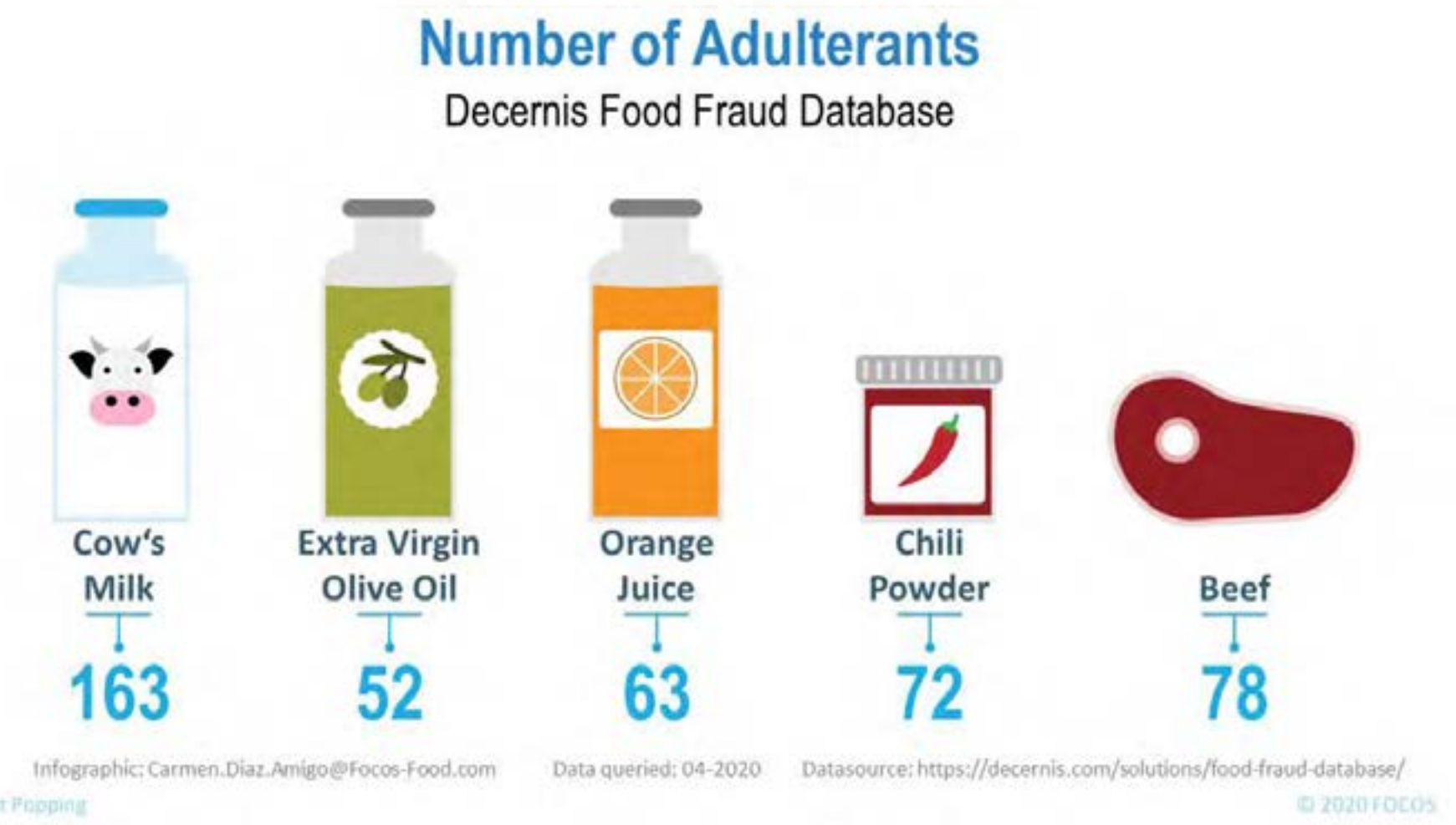
Food Authenticity

- Assurance that raw ingredients purchased by the food manufacturer are accurately documented;
- Assurance that products purchased by consumers are safe and reflect the stated quality.

Economically Motivated Adulteration

- A wide range of deliberate acts designed to misrepresent the authenticity and value of a food product without the purchaser's knowledge for the economic gain of the seller;
- Alteration of a commodity through:
  - Addition of nonauthentic substances, or,
  - Removal or replacement of authentic substances

Breadth of Food Adulteration



†*To learn more about AOAC's Food Authenticity Methods Program go to www.aoac.org/scientific-solutions/food-authenticity-fraud/*

The Challenge

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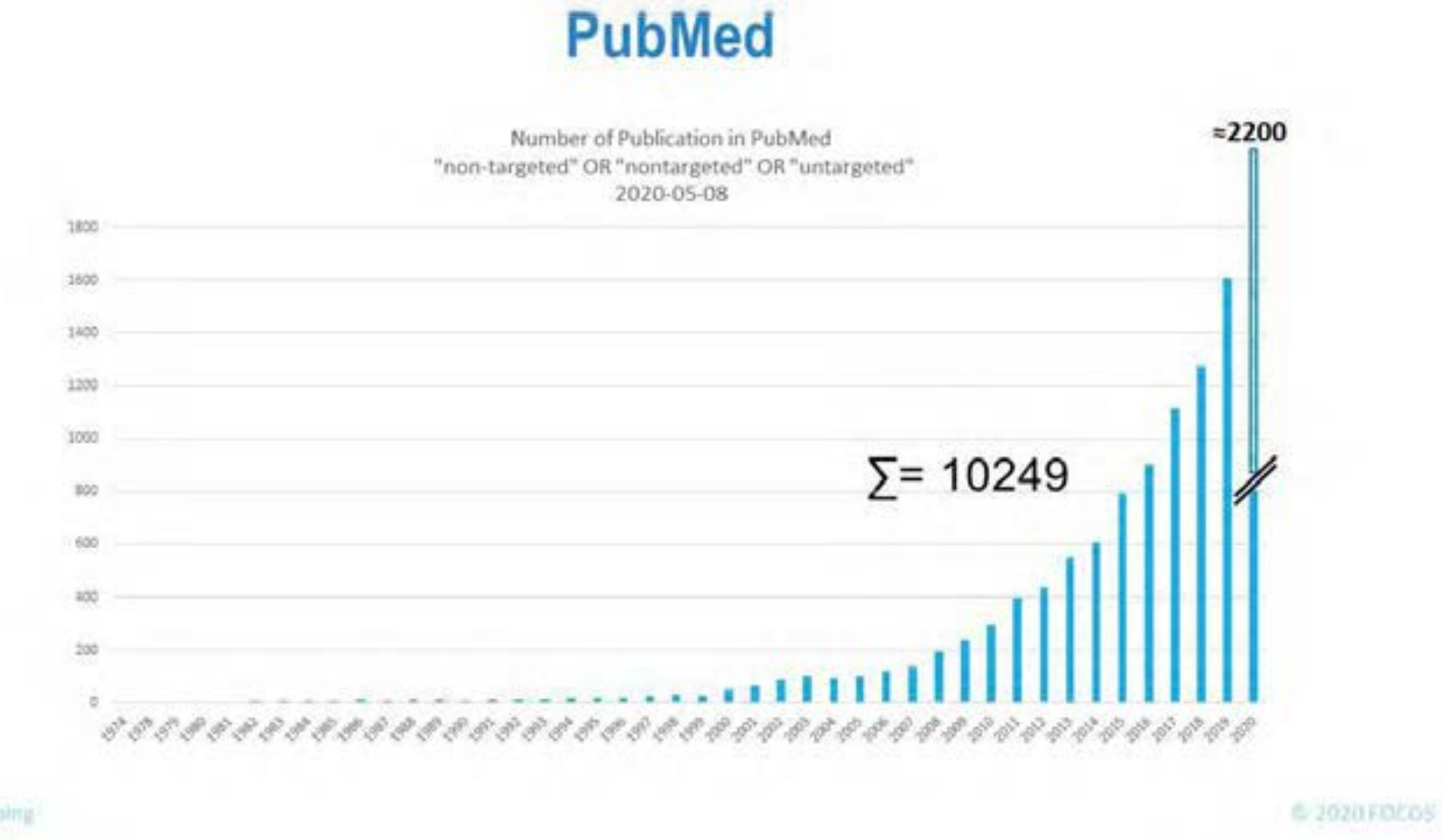
A Snapshot of the Analytical Landscape



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Analytical Challenges For Food Producers, Regulators and Analysts

- What method to choice,
- Breadth of applicability,
- Technology,
- Disparate performance requirements,
- Disparate regulatory requirements,
- Changing environment, rapidly expanding scope.

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Consensus Analytical Standard

- Documents the need for an analytical method,
- Provides a detailed description of how that method must perform,
- Includes method acceptance criteria,
- Agreement among stakeholders and subject matter experts.

Consensus

con·sen·sus | \ kən-ˈsen(t)-səs \

1a : General agreement

b : The judgment arrived at by most of those

2 : Group solidarity in sentiment and belief

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Standards Development: Principles

- **Transparency:** Adequate notification of standards activities
- **Openness:** All stakeholders with a material interest can participate
- **Balance of Interests:** All relevant perspectives are encouraged to participate in standards activities
- **Lack of Dominance:** All relevant perspectives are eligible to participate; the process shall not be weighted to one entity
- **Due Process:** Opportunities for stakeholders to provide input without undue barriers to participation
- **Consensus:** A mechanism to render a decision on a standard
- **Appeals:** A process for stakeholders to object the consensus decision and/or a mechanism for addressing the objections.

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Standards Development: Balancing of Perspectives †

- Varies depending on the project/program.
- Requires either mapping of stakeholders

Broad Perspectives	Specific Perspectives	Regional Perspective
Academia	CRO	Africa
Government	Food & Beverage	Asia
Industry	Product Standards	Europe
Nongovernment Organization	Reference Materials	North America
	Regulator	Oceania
	Research	South America
	Technology Provider	

†*Examples of perspectives of many of AOAC program stakeholders*

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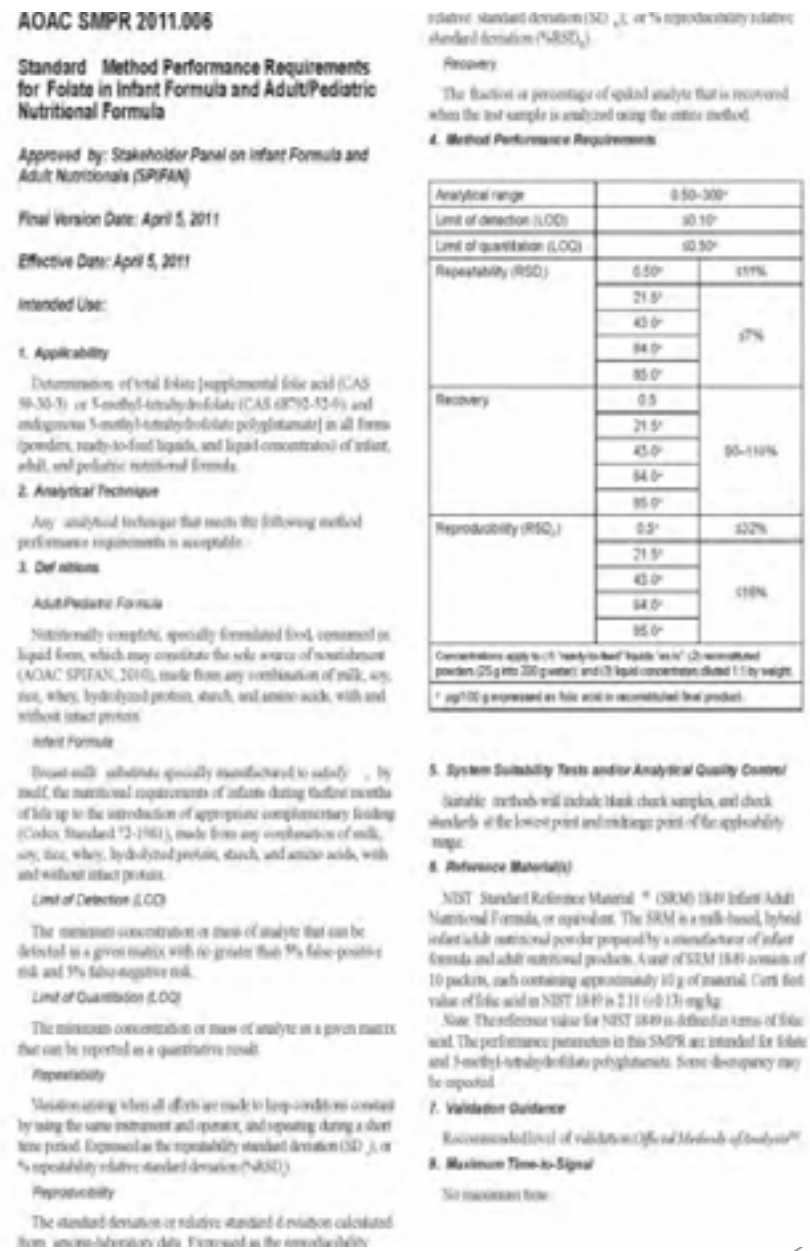

AOAC SMPR 2011.006

Standard Method Performance Requirements for Folate in Infant Formula and Adult/Pediatric Nutritional Formula

Approved by: Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN)

Final Version Date: April 5, 2011

Effective Date: April 5, 2011

Intended Use:

1. Applicability

Determination of total folate [supplemental folic acid (CAS 59-30-3) or 5-methyl-tetrahydrofolate (CAS 68792-52-6) and endogenous 5-methyl-tetrahydrofolate polyglutamate] in all forms (powders, ready-to-feed liquids, and liquid concentrates) of infant, adult, and pediatric nutritional formula.

2. Analytical Technique

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

3. Definitions

Adult/Pediatric Formula

Nutritionally complete, specially formulated food, consumed as liquid form, which may constitute the sole source of nourishment (AOAC SPIFAN, 2010), made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

Infant Formula

Breast milk substitute specially manufactured to satisfy , by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding (Codex Standard 72-1981), made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

Limit of Detection (LOD)

The minimum concentration or mass of analyte that can be detected in a given matrix with no greater than 5% false-positive risk and 5% false-negative risk.

Limit of Quantitation (LOQ)

The 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

The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility

relative 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.

4. Method Performance Requirements

Analytical range	0.50–300*	
Limit of detection (LOD)	≤0.10*	
Limit of quantitation (LOQ)	≤0.50*	
Repeatability (RSD$_r$)	0.50*	≤11%
	21.5*	≤7%
	43.0*	
	84.0*	
	85.0*	
Recovery	0.5	90–110%
	21.5*	
	43.0*	
	84.0*	
	85.0*	
Reproducibility (RSD$_R$)	0.5*	≤22%
	21.5*	≤16%
	43.0*	
	84.0*	
	85.0*	

Concentrations apply to (1) "ready-to-feed" liquids "as is" (2) reconstituted powders (25 g into 200 g water); and (3) liquid concentrates diluted 1:1 by weight.

* µg/100 g expressed as folic acid in reconstituted final product.

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 applicability range.

6. Reference Material(s)

NIST Standard Reference Material ® (SRM) 1849 Infant/Adult Nutritional Formula, or equivalent. The SRM is a milk-based, hybrid infant/adult nutritional powder prepared by a manufacturer of infant formula and adult nutritional products. A unit of SRM 1849 consists of 10 packets, each containing approximately 10 g of material. Cert. first value of folic acid in NIST 1849 is 2.11 (±0.13) mg/kg.

Note: The reference value for NIST 1849 is defined in terms of folic acid. The performance parameters in this SMPR are intended for folate and 5-methyl-tetrahydrofolate polyglutamates. Some discrepancy may be expected.

7. Validation Guidance

Recommended level of validation in *Official Methods of Analysis*℠

8. Maximum Time-to-Signal

No maximum time.

AOAC INTERNATIONAL's Standard Method Performance Requirements (SMPR™)

- Used to adopt AOAC Official Methods by Expert Review Panels.
- Published as a standard in the OMA and status changes in the AOAC *Inside Laboratory Management* (ILM)

†*To learn more about AOAC's Food Authenticity Methods Program go to www.aoac.org/scientific-solutions/food-authenticity-fraud/*

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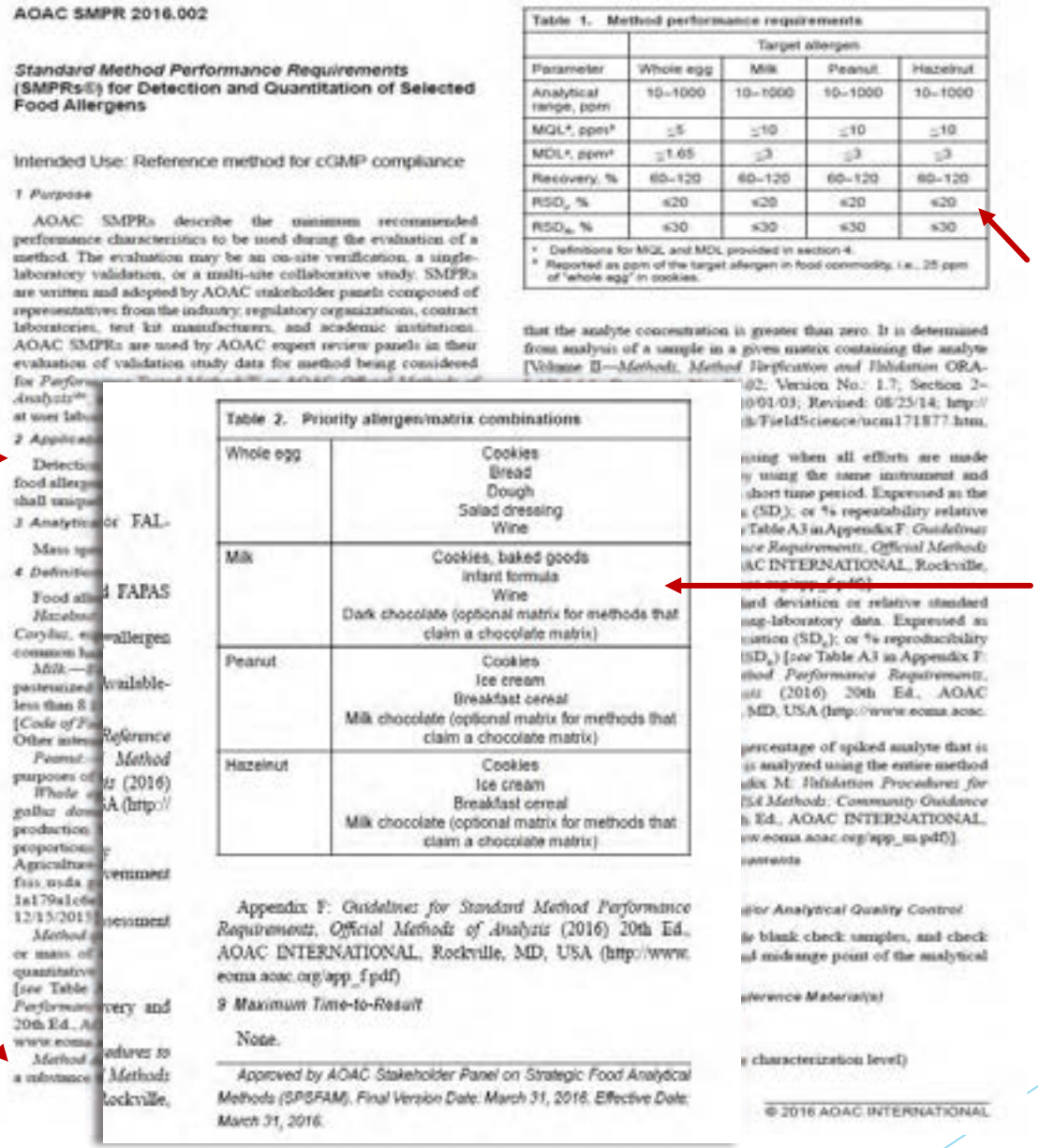
The SMPR™

Specified intended method use

How the method is to be applied

Definitions used for this SMPR as it relates to the method



AOAC SMPR 2016.002

***Standard Method Performance Requirements* (SMPRs®) for Detection and Quantitation of Selected Food Allergens**

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 *Performa*

Table 1. Method performance requirements

	Target allergen			
Parameter	Whole egg	Milk	Peanut	Hazelnut
Analytical range, ppm	10–1000	10–1000	10–1000	10–1000
MQL*, ppm†	≤5	≤10	≤10	≤10
MDL*, ppm†	≤1.65	≤3	≤3	≤3
Recovery, %	60–120	60–120	60–120	60–120
RSD_r, %	≤20	≤20	≤20	≤20
RSD_{wR}, %	≤30	≤30	≤30	≤30

* Definitions for MQL and MDL provided in section 4.
† Reported as ppm of the target allergen in food commodity, i.e., 25 ppm of "whole egg" in cookies.

Table 2. Priority allergen/matrix combinations

Whole egg	Cookies Bread Dough Salad dressing Wine
Milk	Cookies, baked goods Infant formula Wine Dark chocolate (optional matrix for methods that claim a chocolate matrix)
Peanut	Cookies Ice cream Breakfast cereal Milk chocolate (optional matrix for methods that claim a chocolate matrix)
Hazelnut	Cookies Ice cream Breakfast cereal Milk chocolate (optional matrix for methods that claim a chocolate matrix)

Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_f.pdf)

9 Maximum Time-to-Result

None.

Approved by AOAC Stakeholder Panel on Strategic Food Analytical Methods (SPSFAM). Final Version Date: March 31, 2016. Effective Date: March 31, 2016.

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Performance parameters and targets that method must meet

Commodities from which the method should be able to determine allergenic targets

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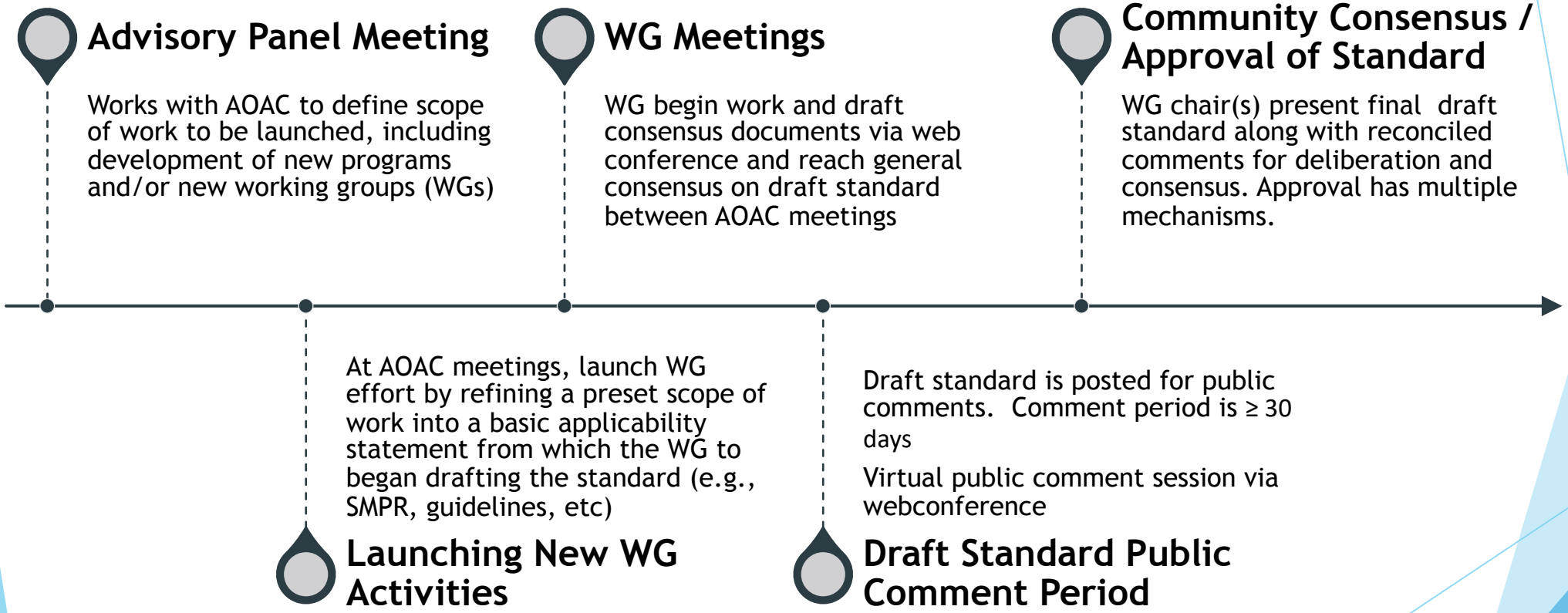
AOAC Standards Development Framework



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Basic AOAC *Official Methods*^{SM} *(OMA)* Program Framework

Call for Methods or Experts

AOAC issues call for methods and call for experts

Method author orientation incl SMPR overview is done

Submission of Methods

Method authors may submit methods that may meet the approved standard

Method authors will want to attend the SMPR or standard orientation for method authors.

ERP Assembly & Review of Methods

ERPs are vetted, approved, and appointed. Methods are reviewed and ERPs meet to review and reach consensus on methods

First Action Method Publication & Tracking

Approved methods are published and are tracked by ERP in partnership with the method author. Methods that satisfy ERP requirements and demonstrate reproducibility are recommended for Final Action

Final Action Review Status

Final versions of methods along with supporting documentation are submitted to OMB for their decision on the recommendation

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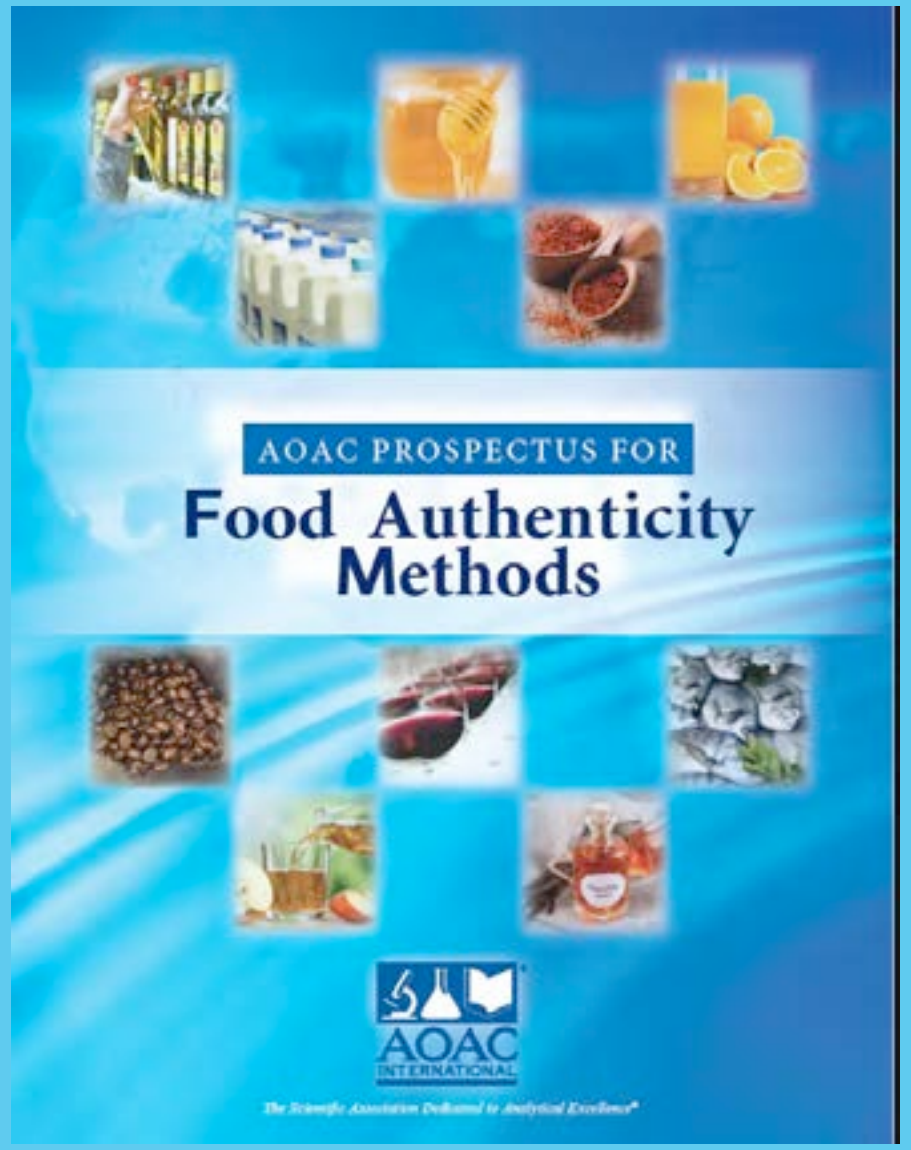
Food Authenticity Methods Program

OBJECTIVES

- To address the analytical needs for combatting intentional and economically motivated food adulteration;
- SMPR™ development for Targeted Testing (TT) and Non-targeted Testing (NTT);
- Develop rapid response guidance for method development in the event of an emergency.



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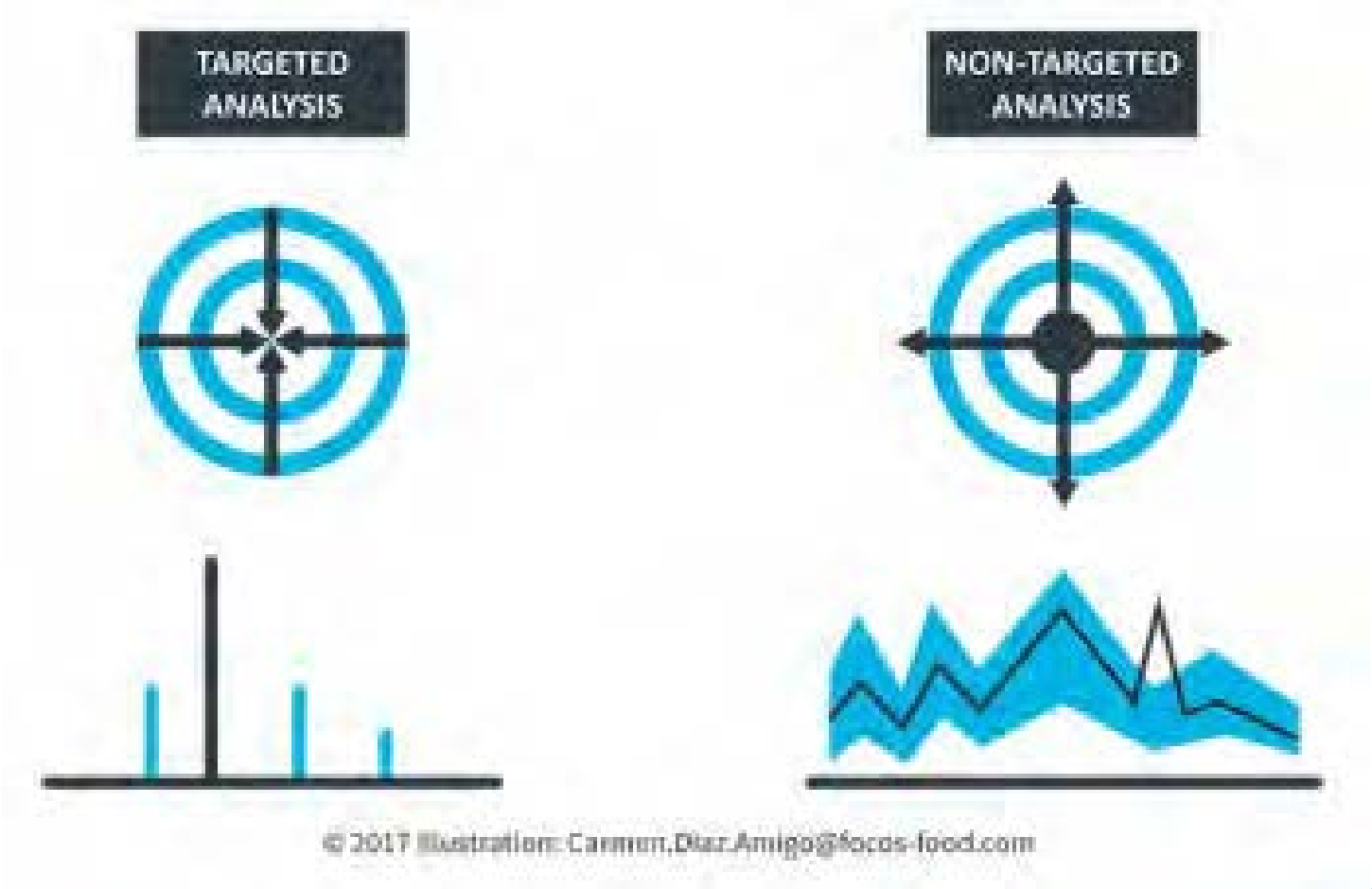
Distinct but Complementary Approaches for Food Authenticity



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SMPR™ Framework for Targeted and Non-Targeted Testing

SMPR Section	Targeted Testing	Non-Targeted Testing
Applicability	ID & Measure specific analyte	Assess if something is "different"
Definitions	Defines targeted analyte. Includes reference standards	Define "authentic" List some adulterations
Method Performance Requirements	Analytical range Accuracy Repeatability Reproducibility	1) Can determine if food has known adulterants 2) Performance on food with unknown adulterant(s)
System Suitability / Quality Control	CRMs in each batch	Adulterated samples in batch; but unknown?
Reference Materials	CRM / SRM	????
Validation Guidance	Established	Newer
Maximum Time to Results	Variable	Variable

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Targeted Testing

- Determination of ***known*** molecules (the adulterant),
- Requires their prior ***identification as an economically motivated adulterant (EMA).***

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Non-targeted Testing

The Concept

- Create a standardized fingerprint for an ingredient.
- Compare new lots of the test ingredient to the fingerprint.
- Quantify “degree of difference”
  - Small amount of difference is a yellow flag
  - Large difference is a red flag

The Process

- Creation of a standardized baseline *i.e.* “authentic profile” to assess the degree of “difference” among authentic foods or food lots,
- Establishes the true breadth of “authenticity” for the ingredient or food,
- Requires a 2-step method development process,
- Highlights the importance of reference materials in establishing the standard profile.

†To learn more about AOAC’s Food Authenticity Methods Program go to www.aoac.org/scientific-solutions/food-authenticity-fraud/

Starting Priorities

- Formaldehyde/Formalin
- Starch
- Soy Protein

- Barley & Malt Extract
- Beet Sugar Syrup
- Corn & Cane Sugar Syrup
- C-4 Plant Sugars
- High Fructose Corn Sugar

- Low Quality Olive Oils
- Other Vegetable Oils
  - Sunflower oil
  - Soybean oil
  - Hazelnut oil
  - Corn and seed oils
  - Waste cooking oil

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Targeted Testing SMPR™ for Olive Oil

Analytical Parameter	Acceptance Criteria for Other Vegetable Oils	Acceptance Criteria for Low Level Olive Oils
Analytical Range (%)	10 – 50 % (w/w) of EVOO	10 – 50 % (w/w) of EVOO
LOQ	≥ 10%	≥ 10%
Recovery	80 -120 %	80 -120 %
Accuracy	± 20%	± 20%
RSD_r	14	14
RSD_R	19	19

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Non-targeted Testing SMPR™ for Olive Oil

Test	Adulterant	% Adulterant in Test Materials	Number of Samples to be Tested[1]	Number of Test Results Qualified as Adulterated
Baseline	None (Authentic EVOO)	0%	Establish Baseline Fingerprint[2]	
Validation using Authentic Samples[3]	None	0%	30	0
Validation[4]	Sunflower Oil	5%	30	30
Validation	Validation[4]	5%	30	30
Validation[4]	Corn Oil	5%	30	30
Validation[4]	Hazelnut Oil	5%	30	30
Validation[4]	Canola Oil	5%	30	30
Validation[4]	Safflower Oil	5%	30	30
Validation[4]	Non-EVOO	5%	30	30
Validation[4]	False Origin	5%	30	30

1. Multiple samples from the same batch of adulterated material can be used for method evaluation.
2. Full details on protocol used to establish an authentic fingerprint must be supplied.
3. Samples used for this step must be independent than those used to create the baseline and must cover the entire scope of the method.
4. Method validation using adulterated samples shall cover the entire scope used in creating the baseline fingerprint.

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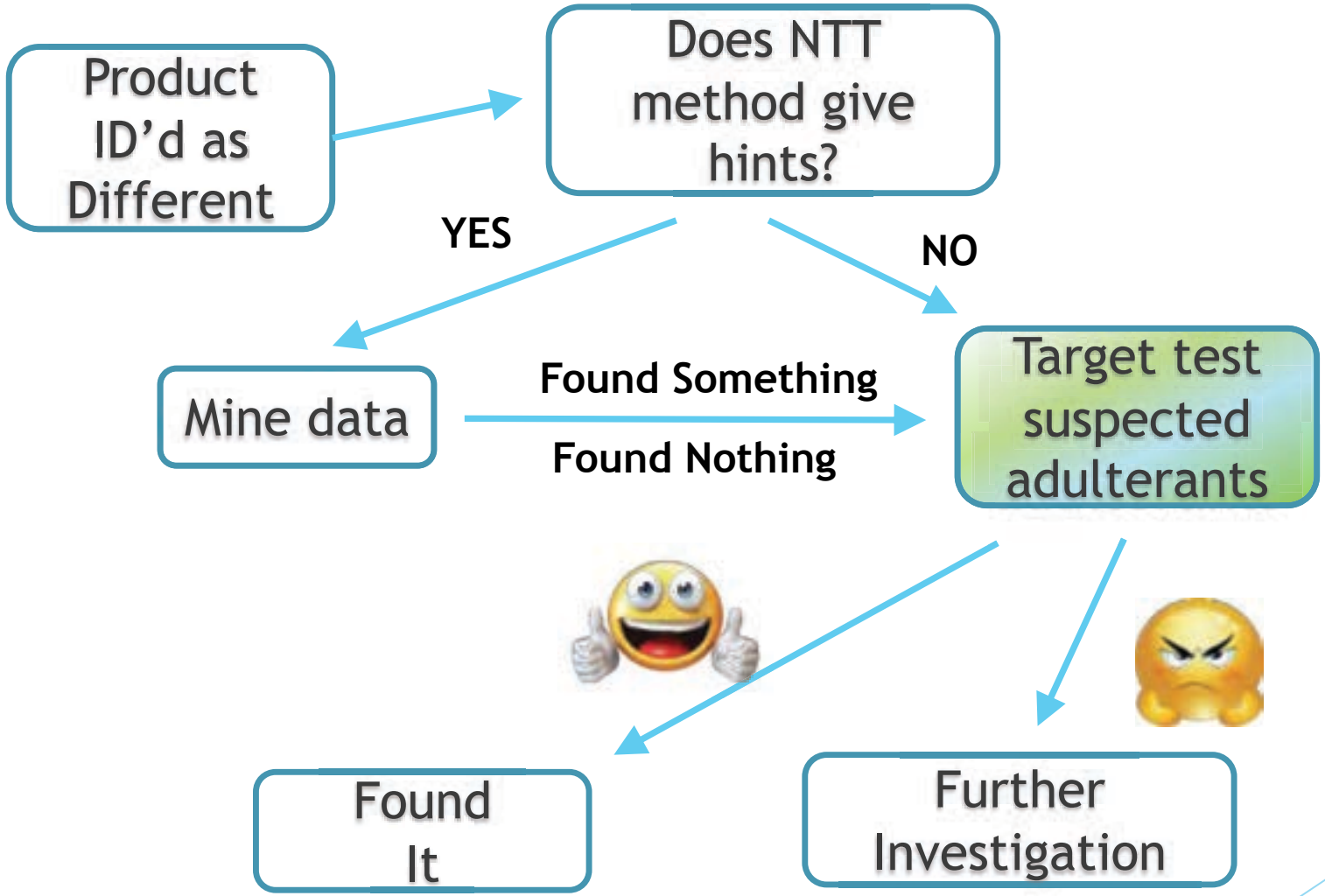
TT & NTT Complementarity



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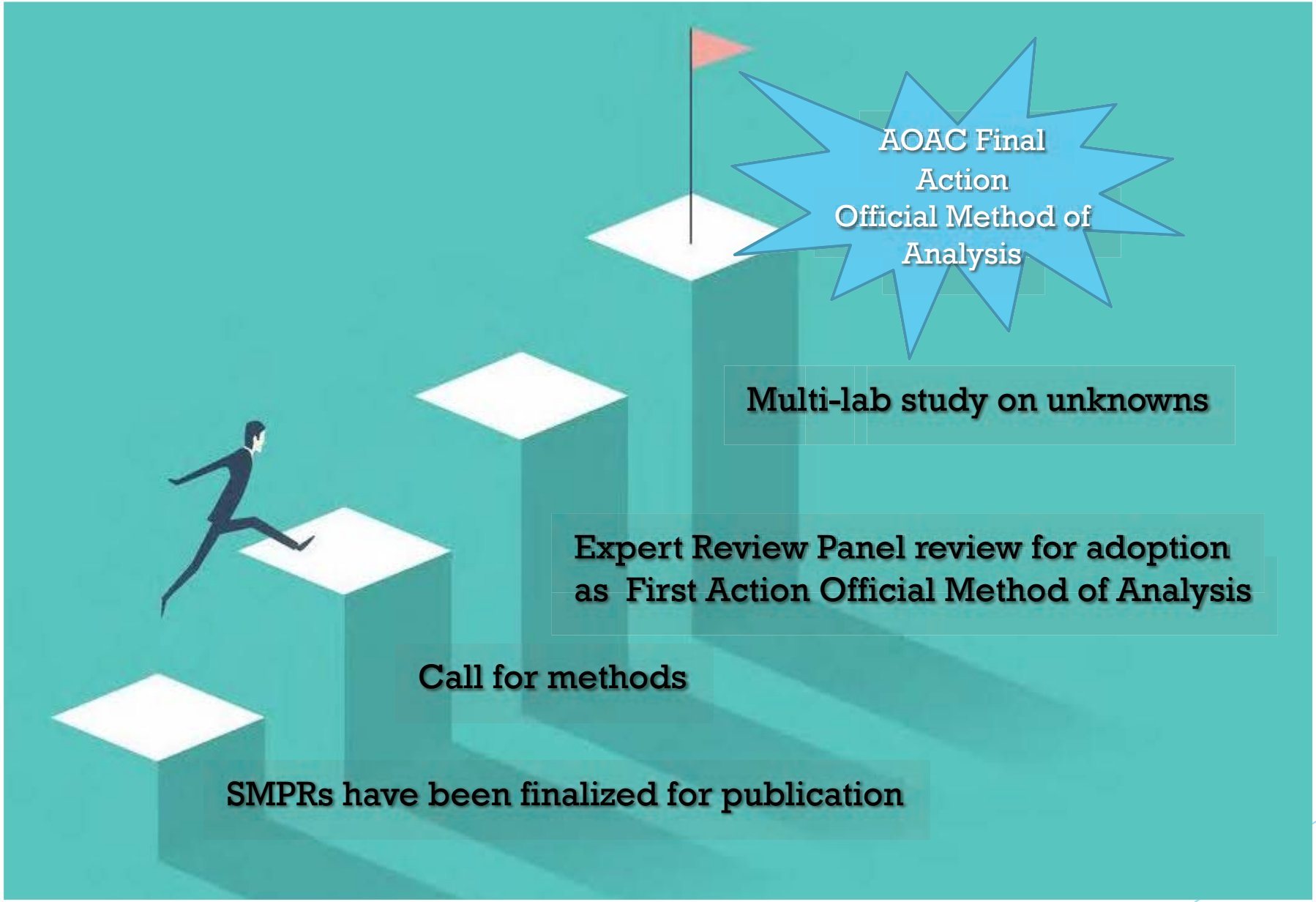

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Strategic Priorities and Goals
2020

- Use of the foundational concepts established using the NTT and TT approaches to create standards and methods for ***additional high priority raw materials and finished products*** involving:
  - Botanicals
  - Spices
- A new working group to create standards and methods for ***molecular and genomic applications***;
- Development of a ***decision tree*** to combine non-targeted screening with targeted confirmatory methods for specific adulterants;
- Develop an ***emergency response*** guidance document for rapid method development and validation review, in the context of a major international food fraud incident, requiring mobilization of resources including those of AOAC INTERNATIONAL;
- Guidance for ***reference and testing material*** development and selection (commodity and adulterant).

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The 2020 FAM Advisory Panel

- The Advisory Panel is comprised of funding organizations,
- Open enrollment format for new organizations interested in participating,
- Establishes the overall program direction; rank priorities for the funding year,
- Reviews progress on a quarterly basis,
- Participating Organizations:

Abbott Nutrition
BioRad
The Coca-Cola Company
Eurofins Scientific
Herbalife
Mars
SGS-North America
Tentamus Group
Thermo Fisher Scientific

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Summary: *Benefits to Standards Development Program*

Method Developers

- Consensus Standard Method Performance Requirements, SMPRs℠;
- AOAC *Official Methods of Analysis*℠: the benchmark for trade resolutions, to instill consumer confidence, and contribute to consumer safety.

Food manufacturers or food distributors:

- Address analytical challenges through AOAC INTERNATIONAL's recognized standards development process,
- *Official Methods of Analysis*, the highest level of analytical ***confidence*** for authenticity claims and detect fraudulent adulteration in priority commodities
- ***Validated*** analytical methods to meet regional and internationally adopted regulatory requirements,
- ***Protect*** producers and consumers alike, maintain the ***reputation*** of products and ultimately improve the ***quality*** and ***safety*** of the food supply

For all:

- Consensus standards & reference methods for commodities that do not exist;
- Provide data for compliance-driven quality control of food materials and products;
- Standards leading to Codex Type II methods for dispute resolution in international trade.

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Learn more about the AOAC INTERNATIONAL Food Authenticity Methods Program at…

- *www.aoac.org/scientific-solutions/food-authenticity-fraud/*
- Or contact us:

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Special Recognition

AOAC FAM Program
Volunteer Science Advisor

Bert Pöpping

AOAC FAM Program Working Group Chairs

JOE BOISON

TARGETED ANALYSIS

JOHN SZPYLKA

NON-TARGETED ANALYSIS

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