UNIVERSITÉ LAVAL | Faculty of Agriculture and Food Sciences

parera
Food Risk Analysis and Regulatory Excellence Platform

Standardizing Food Allergen Methods –

Addressing the Needs of the Arab Region

Oman 6th International Conference on Food Safety and Quality

Prof. Samuel Godefroy, Ph.D.

Full Professor, Food Risk Analysis and Regulatory Policies

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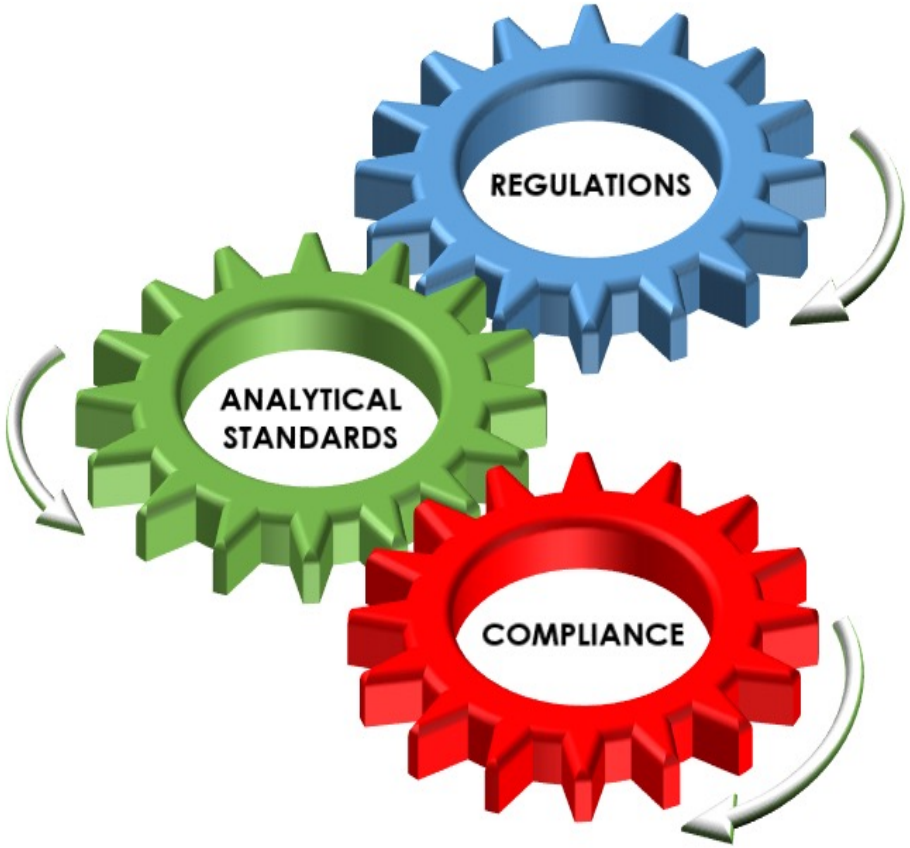
Why Do We Need Analytical Standards?

Analytical standards strengthen food regulatory capacity in its reliance on analytical results to support decision-making



Codex Direction for Analytical Methods …

- Methods are intended to support food regulatory provisions:
  - Support Compliance by Regulated Parties
  - Support Enforcement by Regulators

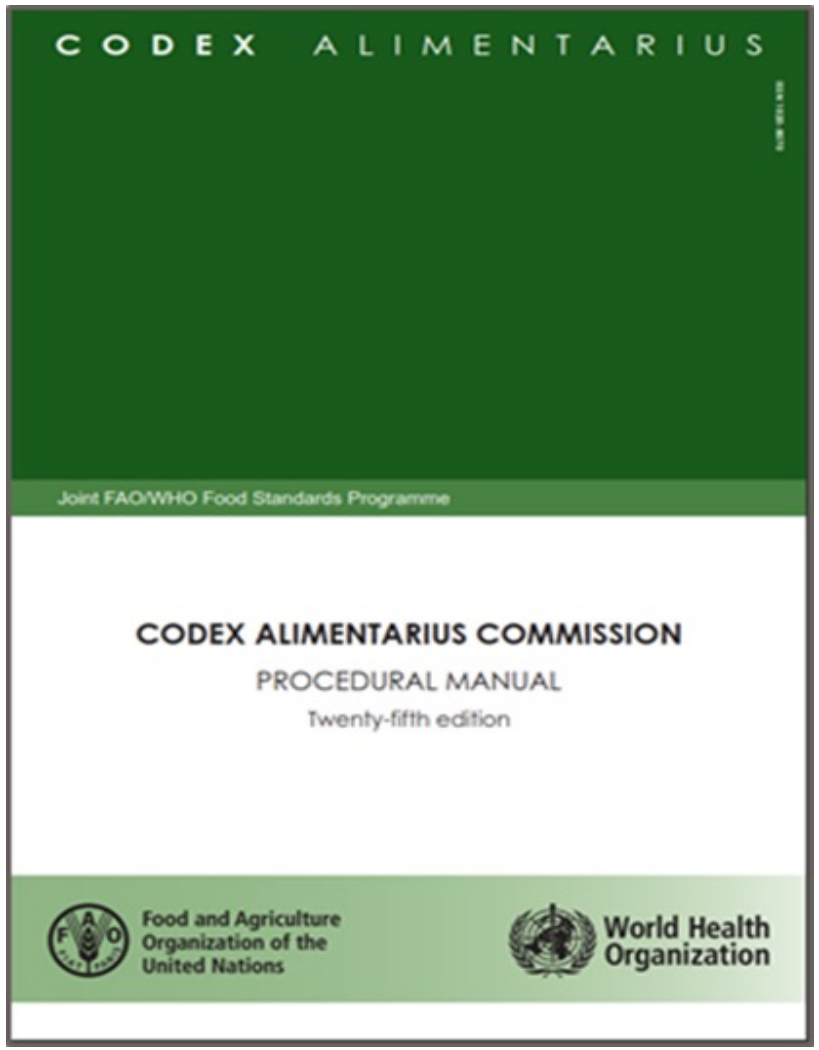
- Methods and their use are therefore an integral part of the implementation of regulatory provisions

Driving for Harmonization Encompasses …

- Ensuring that Methods Used in Support of (International) Standards Satisfy Common Criteria of Performance

- CCMAS Principles:
  - Methods meant to be “international methods are intended for the verification of Codex Standards”.
  - “They should be used for reference in calibration of methods in use or introduced for routine examination and control purposes”.

Codex Procedural Manual



PRINCIPLES FOR THE ESTABLISHMENT OF CODEX METHODS OF ANALYSIS

Purpose of Codex Methods of Analysis

The methods are primarily intended as international methods for the verification of provisions in Codex standards. They should be used for reference, in calibration of methods in use or introduced for routine examination and control purposes.

Methods of Analysis

Definition of types of methods of analysis

(a) **Defining Methods (Type I)**

Definition: A method which determines a value that can only be arrived at in terms of the method per se and serves by definition as the only method for establishing the accepted value of the item measured.

Examples: Howard Mould Count, Reichert-Meissl value, loss on drying, salt in brine by density.

(b) **Reference Methods (Type II)**

Definition: A Type II method is the one designated Reference Method where Type I methods do not apply. It should be selected from Type III methods (as defined below). It should be recommended for use in cases of dispute and for calibration purposes.

Example: Potentiometric method for halides.

(c) **Alternative Approved Methods (Type III)**

Definition: A Type III Method is one which meets the criteria required by the Committee on Methods of Analysis and Sampling for methods that may be used for control, inspection or regulatory purposes.

Example: Volhard Method or Mohr Method for chlorides

(d) **Tentative Method (Type IV)**

Definition: A Type IV Method is a method which has been used traditionally or else has been recently introduced but for which the criteria required for acceptance by the Committee on Methods of Analysis and Sampling have not yet been determined.

Note 1: These criteria are applicable to fully validated methods except for methods such as PCR and ELISA, which require other set of criteria.

Note 2: The approaches described for developing method performance criteria are intended for single-analyte provisions. The approaches described may not be suitable for provisions involving sum of components.

Table 1: Guidelines for establishing numeric values for the criteria:

Applicability:	The method has to be applicable for the specified provision, specified commodity and the specified level(s) (maximum and/or minimum) (ML). The minimum applicable range of the method depends on the specified level (ML) to be assessed, and can either be expressed in terms of the reproducibility standard deviation (s_R) or in terms of LOD and LOQ.
Minimum applicable range:	For ML ≥ 0.1 mg/kg, [ML - 3 s_R, ML + 3 s_R] For ML < 0.1 mg/kg, [ML - 2 s_R, ML + 2 s_R] s_R[14] = standard deviation of reproducibility
Limit of Detection (LOD):	For ML ≥ 0.1 mg/kg, LOD ≤ ML · 1/10 For ML < 0.1 mg/kg, LOD ≤ ML · 1/5
Limit of Quantification (LOQ):	For ML ≥ 0.1 mg/kg, LOQ ≤ ML · 1/5 For ML < 0.1 mg/kg, LOQ ≤ ML · 2/5

Precision:	For ML ≥ 0.1 mg/kg, HorRat value ≤ 2 For ML < 0.1 mg/kg, the RSD_{TR} < 22%. RSD_R[15] = relative standard deviation of reproducibility. $RSD_R \leq 2$. $PRSD_R$			
Recovery (R):	**Concentration**	**Ratio**	**Unit**	**Recovery (%)**
	100	1	100% (100g/100g)	98 – 102
	≥10	10^{-1}	≥ 10% (10g/100g)	98 – 102
	≥1	10^{-2}	≥ 1% (1g/100g)	97 – 103
	≥0.1	10^{-3}	≥ 0.1% (1mg/g)	95 – 105
	0.01	10^{-4}	100 mg/kg	90 – 107
	0.001	10^{-5}	10 mg/kg	80 – 110
	0.0001	10^{-6}	1 mg/kg	80 – 110
	0.00001	10^{-7}	100 µg/kg	80 – 110
	0.000001	10^{-8}	10 µg/kg	60 – 115
	0.0000001	10^{-9}	1 µg/kg	40 – 120
Trueness:	Other guidelines are available for expected recovery ranges in specific areas of analysis. In cases where recoveries have been shown to be a function of the matrix other specified requirements may be applied.			

[14] The s_R should be calculated from the Horwitz/Thompson equation. When the Horwitz/Thompson equation is not applicable (for an analytical purpose or according to a regulation) or when "converting" methods into criteria then it should be based on the s_R from an appropriate method performance study.

[15] The RSD_R should be calculated from the Horwitz/Thompson equation. When the Horwitz/Thompson equation is not applicable (for an analytical purpose or according to a regulation) or when "converting" methods into criteria then it should be based on the RSD_R from an appropriate method performance study.

Codex Committee on Methods of Analysis and Sampling

- Has the mandate to approve reference methods in support of the application of Codex standards

- Codex : is a Risk Management Body and seeks advice to develop and Adopt Standards

- AOAC International Methods is one of the Sources for Codex Standard Methods

CODEX STAN 234-1999

RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

CODEX STAN 234-1999[1]

PART A

METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Infant formula	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Infant formula	Sodium and potassium	ISO 8070 | IDF 119:2007	Flame atomic absorption spectrophotometry	II
Infant formula	Thiamine	AOAC 986.27[12]	Fluorimetry	III
Infant formula	Thiamine	EN 14122:2003 (Measures all vitamin B_1 forms (natural and added free, bound and phosphorylated) following extraction and conversion to thiamine)	HPLC with pre-or post column derivatization to thiochrom	II

CODEX ALIMENTARIUS
INTERNATIONAL FOOD STANDARDS

Food and Agriculture Organization of the United Nations
World Health Organization

E-mail: codex@fao.org - www.codexalimentarius.org

Performance Requirements Parameters

Performance Requirements Parameters for chemical quantitative methods:

Analytical Range

Limit of Detection

Limit of Quantitation

Repeatability

Recovery

Reproducibility

4. Method Performance Requirements

Analytical range	0.01–5.0[a]	
Limit of detection (LOD)	≤0.004[a]	
Limit of quantitation (LOQ)	≤0.01[a]	
Repeatability (RSD_r)	0.01[a]	≤15%
	0.2[a]	≤7%
	0.5[a]	
	5.0[a]	
Recovery	0.01[a]	90–110%
	0.2[a]	
	0.5[a]	
	5.0[a]	
Reproducibility (RSD_R)	0.3	≤11%
	0.6	
	1.0	
	2.5	
	5.0	
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.		
[a] µg/100 g expressed as cyanocobalamin in reconstituted final product.		

AOAC Standards Development

Initiating SMPRs to Final Action Consideration of Methods

Application to Allergen Methods

Reliance on non-traditional techniques: Mainly ELISA-based methods

Food Allergen Rules are Multiplying …

- An increased interest in updating food labeling laws or regulations
  - Latin America: Argentina, Columbia, Chile
- An increasing number of ASEAN countries have considered or are undertaking updates to labelling requirements to consider Allergens as a priority
  - Issue moving from import/export concern to domestic issue
  - Increased awareness of burden of incidents on public health systems
- China considering (mandatory) food allergen regulations

AOAC Food Allergen Community Took the Lead ..

AOAC Food Allergen Community

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SPECIAL SECTION ON FOOD ALLERGEN TESTING

Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices

MICHAEL ABBOTT, STEPHEN HAYWARD, WILLIAM ROSS, and SAMUEL BENREJEB GODEFROY
Health Canada, Food Directorate, Health Products and Food Branch, PL 2201C, Ottawa, ON, Canada, K1A 0L2
FRANZ ULBERTH and ARJON J. VAN HENGEL
European Commission, Joint Research Centre, Institute for Reference Materials and Measurements, Retieseweg 111, 2440 Geel, Belgium
JAMES ROBERTS
National Measurement Institute, Department of Innovation, Industry, Science and Research, 1 Suakin St, Pymble NSW 2073, Australia
HIROSHI AKIYAMA
National Institute of Health Sciences, 1-18-1 Kamiyoga, Setagaya-ku, Tokyo 158-8501, Japan
BERT POPPING
Eurofins Scientific, 69a Kilnwick Rd, Pocklington Yorkshire, YO42 2JY, United Kingdom
JUPITER M. YEUNG[1]
Nestlé Nutrition, Product Technology Center–Fremont, 445 State St, Fremont, MI 49413-0001
PAUL WEHLING
General Mills, Inc., 330 University Ave SE, Minneapolis, MN 55414
STEVE L. TAYLOR
Food Allergy Research and Resource Program, University of Nebraska, Lincoln, NE
ROLAND ERNEST POMS
ICC-International Association for Cereal Science and Technology, Vienna, Austria
PHILIPPE DELAHAUT
CER Groupe–Laboratoire d'Hormonologie, Rue du Point du Jour, 8, B-6-900 Marloie, Belgium

This document provides supplemental guidance on specifications for the development and implementation of studies to validate the performance characteristics of quantitative ELISA methods for the determination of food allergens. It is intended as a companion document to other existing publications on method validation. The guidance is divided into two sections: information to be provided by the method developer on various characteristics of the method, and implementation of a multilaboratory validation study. Certain criteria included in the guidance are allergen-specific. Two food allergens, egg and milk, are used to demonstrate the criteria guidance. These recommendations will be the basis of the harmonized validation protocol for any food allergen ELISA method, whether proprietary or nonproprietary, that will be submitted to AOAC and/or regulatory authorities or other bodies for status recognition. Regulatory authorities may have their own particular requirements for data packages in addition to the guidance in this document. Future work planned for the implementation and validation of this guidance will include guidance specific to other priority allergens.

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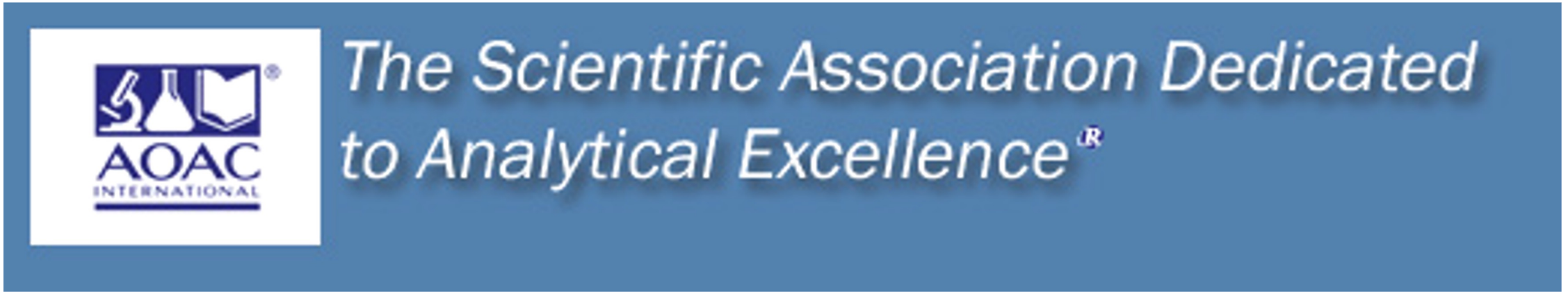
Corresponding author's e-mail: Michael.Abbott@hc-sc.gc.ca
[1] Part of this work was done while author Yeung was employed by the Grocery Manufacturers Association.

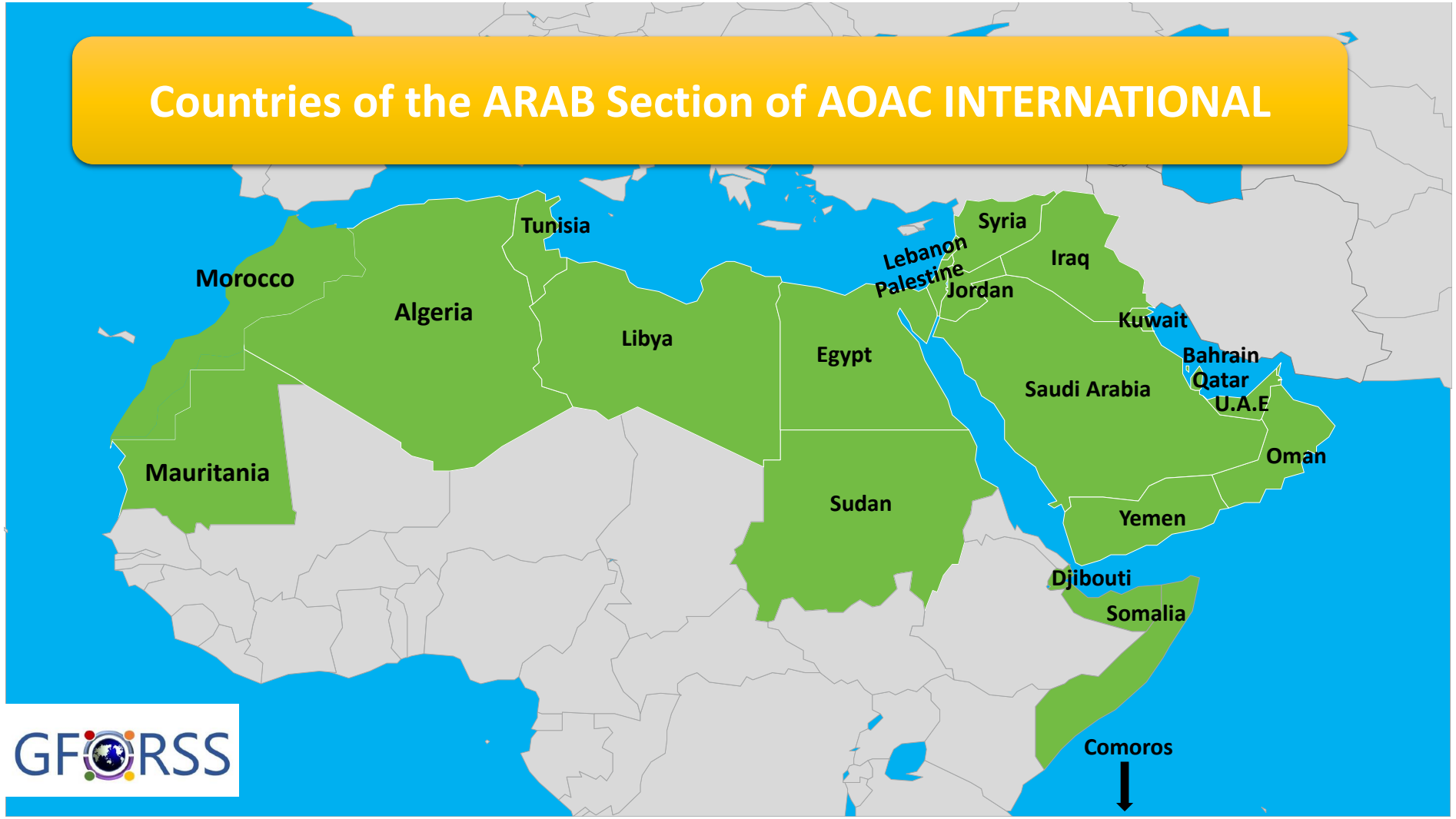
Although there are a number of documents published on method validation (1, 2) which target analytical methods in general, and there are numerous publications on validation of ELISA methods for pesticides, these documents do not address specific areas of concern for food allergen analysis, such as reference materials, spiking methods, or choice of matrixes. In the absence of a universally recognized reference standard for food allergen ELISAs, many organizations and end-users use different validation protocols and different analytical standards. Such inconsistency and duplication inevitably has a negative economic impact on the food allergen community. This document is designed to accompany the AOAC Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis (1), and to provide guidance specific to the validation of quantitative ELISA-based methods for food allergens. This protocol was designed to meet or exceed the minimum requirements set forth in Appendix D of the AOAC Guidelines; it was developed with input from a wide range of experts in the area

Establishing a Section of AOAC INTERNATIONAL in the Arab Region

AOAC International



Introduction: Arab Region



What Can be Achieved ….Standardized Food Allergen Methods for the Arab Region

- A consensus validation protocol for ELISA-based allergen determination methods based on AOAC Protocols

  - Recognize AOAC Processes and leverage achievements
  - Enabler to Undertake Inter-laboratory validation studies for ELISA-based test kits (targeting food allergens) for food matrices of interest to the **Arab Region**
  - Enabler to foster data gathering on allergen methods to be submitted for evaluation under the auspices of AOAC International

Creating Momentum for

- Developing Common Validation Protocols
- **Food Allergen Methods as a Pilot for the Standard on Arab Food Analytical Methods**


thank you
danke
merci
gracias
obrigado
dziękuję
bedankt
go raibh maith agat
teşekkür ederim
ngiyabonga
謝謝
спасибо
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