Sivananda Yoga Vedanta Center Chicago

AOAC Official Methods Board
June 22-23, 2017
Remote Meeting & Webconference

AOAC INTERNATIONAL Headquarters
2275 Research Blvd
Suite 300
Rockville, Maryland 20871

Mérieux NutriSciences Headquarters
111 East Wacker Drive
23rd Floor
Chicago, Illinois 60651

WEB & TELE CONFERENCE INFORMATION

Please refer to the meeting appointment information

OFFICIAL METHODS BOARD MEETING

Thursday, June 22, 2017
10:00 AM ET / 9:00 AM CT – 6:00 PM ET / 5:00 PM CT

Friday, June 23, 2017
9:30 AM ET / 8:30 AM ET – 4:00 PM / 3:00 PM ET

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AOAC INTERNATIONAL
OFFICIAL METHODS BOARD
2016 –2017
APPOINTED

Chair	**Erin Sutphin Crowley** Q Laboratories, Inc. ecrowley@qlaboratories.com Term 2+: September 22, 2016 - September 11, 2019	*Member*	**Don Gilliland** Abbott Nutrition don.gilliland@abbott.com Term 1: October 1, 2015 - September 29, 2018
Member	**Doug Abbott** Independent Consultant douglas.abbott@gmail.com Term 2: September 11, 2014 - September 27, 2017	*Member*	**Katerina Mastovska** Covance Laboratories Katerina.Mastovska@covance.com Term 1: October 1, 2015 - September 29, 2018
Member	**Joe Boison** Canadian Food Inspection Agency Joe.Boison@inspection.gc.ca Term 2: September 22, 2016 – September 11, 2019	*Member*	**Wendy McMahon** Mérieux NutriSciences wendy.mcmahon@mxns.com Term 1: September 22, 2016 – September 11, 2019
Member	**Amy Brown** Florida Department of Agriculture and Consumer Services Amy.Brown@freshfromflorida.com Term 1: September 22, 2016 – September 11, 2019	*Member*	**Melissa Phillips** US National Institute of Standards and Technology melissa.phillips@nist.gov Term 1: September 22, 2016 – September 11, 2019
Member	**Esther Campos Gimenez** Nestle Research Centre esther.campos-gimenez@rdls.nestle.com Term 1: September 22, 2016 – September 11, 2019	*Member*	**Yvonne Salfinger,** Independent Consultant ***AOAC Committee on Safety, Chair*** Yhale@aol.com Term 2: September 22, 2016 – September 11, 2019
Member	**Sidney Sudberg,** Alkemist Labs ***AOAC Committee on Statistics, Chair*** Sidney@alkemist.com Term 1: September 22, 2016 – September 11, 2019	*Member*	**Bradley Stawick** Microbac Laboratories, Inc. brad.stawick@microbac.com Term 2: October 1, 2015 - September 29, 2018
Past Chair (Ex-officio Member)	**Shauna Roman** Reckitt Benckiser, Inc. Shauna.Roman@reckittbenckiser.com Term 4: September 22, 2016 – September 11, 2019		

AOAC Staff Liaisons

Deborah McKenzie Sr. Director, AOAC Standards Development Sr. Director, AOAC Research Institute dmckenzie@aoac.org	Delia Boyd Program Manager, AOAC Standards Development dboyd@aoac.org

AOAC INTERNATIONAL
BYLAWS

As Amended September 26, 2010

ARTICLE I
Name

The name by which this Association shall be known is "AOAC INTERNATIONAL" (hereinafter referred to as the "Association").[1]

ARTICLE II
Purpose

The primary purpose of the Association is to promote methods validation and quality measurements in the analytical sciences.

ARTICLE III
Membership

Section 1. Types of Membership

There shall be three (3) types of membership in the Association: Individual Members, Sustaining Member Organizations, and Organizational Affiliates.

A. Individual Members

There shall be four (4) categories of Individual Members in the Association: Members, Retired Members, Student Members, and Honorary Members.

B. Sustaining Member Organizations

There shall be one (1) category of Sustaining Member Organizations.

C. Organizational Affiliate

There shall be one (1) category of Organizational Affiliate.

Section 2. Qualifications for Membership

A. Individual Members

[1] Members

Qualifications for Members shall be a degree in science, or equivalent as approved by the Board of Directors, and interest in supporting and furthering the purpose and goals of the Association. Such scientists shall be eligible for membership provided they are engaged, or have been engaged, directly or indirectly, in a field relevant to the purpose of the Association.

[2] Retired Members

[1] AOAC INTERNATIONAL was incorporated in the District of Columbia on January 20, 1932, as the Association of Official Agricultural Chemists. On November 10, 1965, the name of the corporation was changed to the Association of Official Analytical Chemists, and on September 12, 1991, the current name was adopted.

A current Member who is no longer actively engaged, directly or indirectly, in a field relevant to the purpose of the Association but who has served the Association as a Member for at least ten (10) years shall be eligible for Retired Member status upon written request and payment of the annual Retired Member dues. Any special benefits accorded Retired Members shall be determined by the Executive Director.

[3] Student Members

Any full-time student working toward an undergraduate or graduate degree in the areas of chemistry, microbiology, food science or other related science shall be eligible for Student Membership in AOAC INTERNATIONAL.

[4] Honorary Members

Honorary Members shall be persons recognized for their substantial contribution toward the achievement of the objectives of the Association. They shall be nominated by the Board of Directors and may be elected by a two-thirds vote of the Individual Members voting.

B. Sustaining Member Organizations

A Sustaining Member Organization shall be any agency of a local, state, provincial, national, or international government; a university, college, or academic department; or any firm, business, or organization with an interest in supporting and furthering the purpose of the Association. Every Sustaining Member Organization must have a designated representative(s). All such Sustaining Member Organization representatives must meet the qualifications for Members and become Individual Members with all the rights and privileges thereof.

C. Organizational Affiliate

An Organizational Affiliate Organization shall be any agency of a local, state, provincial, national, or international government; a university, college, or academic department; or any firm, business, or organization with an interest in supporting and furthering the purpose of the Association. Every Organizational Affiliate must have a designated representative(s). All such Organizational Affiliate representatives must meet the qualifications for Members and become Individual Members with all the rights and privileges thereof.

Section 3. Application for Membership

Applications or requests for membership shall be submitted to the Association's headquarters office. Membership shall become effective upon approval of the application or request, payment of any required membership dues, entry on the membership rolls, and assignment of a member number.

Section 4. Expulsion

The Board of Directors, at any duly called meeting of the Board, by a two-thirds vote of those holding office, may terminate the membership of any member who in its judgment has violated the Bylaws or has been guilty of conduct detrimental to the best interests of the Association. Any member convicted of a felony is subject to immediate expulsion from the Association. Expulsion of a member by the Board of Directors shall be final and shall cancel all rights, interest, or privileges of such member in the services or resources of the Association. Any member, for whom expulsion is proposed, for reasons other than conviction of a felony, shall be entitled to not less than 60 days advance notice of the charges, the date upon which a hearing will be scheduled, and the right to present evidence in defense. The date and place of any such hearing, if held other than at the headquarters or annual meeting site of the Association, must be reasonable with respect to the location of any individual so charged.

Section 5. Dues, Membership Year, and Waivers

A. Annual dues for membership in the Association shall be fixed by the Board of Directors, subject to approval by the majority of the Individual Members voting by ballot by any of the following means (whichever is deemed appropriate by the Board at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission.

B. Honorary Members of the Association shall be exempt from payment of dues and annual meeting registration fees.

C. The membership year and the delinquency date shall be determined by the Board of Directors.

D. The authority to grant waivers of membership dues rests with Executive Director.

E. Student Member dues shall be one-third of regular Member dues, rounded up to the nearest $5.00 increment.

Section 6. Members in Good Standing; Rights and Privileges

All Individual Members who maintain their membership by payment of dues as required under these Bylaws and who otherwise qualify shall be considered in good standing and entitled to full privileges of membership.

ARTICLE IV
Officers

Section 1. Elected Officers

The elected officers of the Association shall be Individual Members and shall consist of a President, President-Elect, Secretary, Treasurer, and Immediate Past President.

A. President

The President shall be the principal elected officer of the Association, shall preside at meetings of the Association and of the Board of Directors and of the Executive Committee, and shall be a member ex-officio, with right to vote, of all committees except the Nominating Committee. He or she shall also, at the annual meeting of the Association and at such other times as he or she shall deem proper, communicate to the Association or the Board of Directors such matters and make such suggestions as may in his or her opinion tend to promote the welfare and further the purpose of the Association and shall perform such other

duties as are necessarily incident to the office of President or as may be prescribed by the Board of Directors.

B. President-Elect

In the absence of the President, or in the event of the President's inability or refusal to act, the President-Elect shall perform the duties of the President, and, when so acting, shall have all the powers of and be subject to all the restrictions upon the President. The President-Elect shall perform such other duties as from time to time may be assigned to him or her by the President or by the Board of Directors.

C. Secretary

The Secretary shall give notice of all meetings of the Association, keep a record of all proceedings, attest documents, and, in general, perform such other duties as are usual of the office of Secretary and such other duties as may be assigned by the President or by the Board of Directors.

D. Treasurer

The Treasurer shall be responsible for the funds and securities of the Association; serve as financial officer of the organization and as Chairperson of the Finance Committee; manage the Board of Director's review of and action related to the Board of Director's financial responsibilities; serve as the chief Board liaison in overseeing and reviewing the annual audit, and in general, perform such other duties as are usual of the office of Treasurer and such other duties as may be assigned by the President or by the Board of Directors.

E. Immediate Past President

The Immediate Past President shall serve as advisor to the President and Directors and perform such other duties as may be assigned from time to time by the President or by the Board of Directors.

Section 2. Appointed Officers

The appointed officers shall include the Executive Director and such other appointed officers as may be designated by the Board of Directors from time to time.

A. Executive Director

The day-to-day administration and management of the Association's offices shall be vested in a salaried manager employed or appointed by, and directly responsible to, the Board of Directors. This manager shall have the title of Executive Director with responsibility for the management and direction of all operations, programs, activities, and affairs of the Association, as approved or delegated by the Board of Directors. The Executive Director shall have direct responsibility for employment and termination of employment and the determination of compensation for staff members within the budgetary framework determined by the Board of Directors. The Executive Director functions as the chief operating officer of the Association within the guidelines established by the policies and procedures of the Board of Directors and, as necessary, with the concurrence of the President. The Executive Director shall have such other duties as may be prescribed by the Board.

B. Other Appointed Officers

Other appointed officers shall have such duties as may be prescribed by the Board.

ARTICLE V
Nominations, Elections, Terms, and Appointments to the Board of Directors

Section 1. Nominating Committee

The Nominating Committee shall annually recommend to the Board of Directors a slate of Individual Members as potential nominees for the elected positions where vacancies will occur. The Nominating Committee shall consist of five (5) members who shall be three (3) immediate Past Presidents, as available, and two (2) Individual Members-at-Large of the Association. If three Past Presidents are not available to serve, other Individual Members-at-Large shall be appointed by the President to the extent necessary to form the five (5)-member committee.

Section 2. Elections and Terms of Office

The President-Elect, the Secretary, Treasurer, and the Directors of the Board of Directors shall be elected by a majority of Individual Members voting, from a slate of nominees recommended annually by the Board of Directors.

Terms of office for all Officers and Directors shall begin with the adjournment of the annual meeting following their election and shall end with the adjournment of the annual meeting occurring nearest the expiration of their term. The six (6) Directors shall be elected to staggered three-year terms with two Directors elected to full three-year terms each year, but not to more than two (2), consecutive, three-year terms. Appointment or election to fill an unexpired term shall not affect the eligibility of a person to subsequently be elected to two (2) full terms. The Secretary shall be elected to a one-year term and may be re-elected to successive one-year terms. The Treasurer shall be elected for a one-year term and may be re-elected to successive one-year terms. The President-Elect shall be elected to a one-year term; whereupon the current President-Elect shall become President and the current President shall become the Immediate Past President, each serving a one-year term.

Section 3. Appointments

Directors-at-Large are appointed by the Board in accordance with Article VI, Section 2. Directors-at-Large are appointed for one (1) year terms, renewable at the discretion of the elected Board.

ARTICLE VI
Board of Directors

Section 1. Composition

The Board of Directors shall consist of eleven (11) elected members to include the President, President-Elect, Secretary, Treasurer, Immediate Past President, six (6) Directors, and up to three (3) appointed Directors-at-Large, all of whom shall be Individual Members of the Association. The elected Board shall reflect the makeup of the Association membership and shall not be dominated by any single interest.

Section 2. Powers and Duties

The Board of Directors shall provide supervision, control, and direction of the affairs of the Association, shall determine the Association's policies or changes therein within the limits of the Bylaws, shall actively prosecute

its purpose, and shall have discretion in the disbursement of its funds. It may adopt such rules and procedures for the conduct of its business as shall be deemed advisable, and may, in the execution of the powers granted, appoint such agents as it may consider necessary. The Board of Directors may appoint up to three (3) Directors-at-Large, if, in their opinion, such appointments advance the purpose of the Association. Directors-at-Large shall be accorded the same voting privileges as elected Directors.

Section 3. Meetings

Except that the Board shall have a regular meeting at the time and place of the annual meeting, the Board shall meet, in person or via telephone conference call, upon call of the President at such times and places as he or she may designate within the policies adopted by the Board, and shall be called to meet upon demand of a majority of its members. Notice of all meetings of the Board of Directors shall be sent by any of the following means (whichever is deemed appropriate by the President at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission to each member of the Board at his or her last recorded address or number at least fourteen (14) days in advance of in-person meetings or forty-eight (48) hours in advance of conference call meetings.

Section 4. Quorum

A quorum for any meeting of the Board is six (6) Board members elected in accordance with Article V (1). Any less number may: (1) set a time to adjourn, (2) adjourn, (3) recess, or (4) take measures to obtain a quorum.

Section 5. Absence

Any member of the Board of Directors unable to attend a meeting of the Board shall notify the President and state the reason for his or her absence. If a member of the Board is absent from two (2) consecutive meetings, he or she may be removed by a two-thirds vote of the Board Members then in office.

Section 6. Compensation

Members of the Board of Directors, as such, shall not receive any compensation for their services as Board members, but the Board may, by resolution under policies it may adopt, authorize reimbursement of expenses incurred in the performance of members' duties. Such authorization may prescribe conditions and procedures for approval and payment of such expenses. Nothing herein shall preclude a Board member from serving the Association in any other capacity and receiving compensation for such services, if compensation is customarily paid for such services.

Section 7. Resignation or Removal

Any member of the Board may resign at any time by giving written notice to the President, Secretary, Treasurer, or to the Board of Directors. Such resignation shall take effect at the time specified therein, or, if no time is specified, at the time of acceptance thereof as determined by the President or the Board.

Any member of the Board may be removed by a three-fourths vote of the Board members then in office and present at any regular or special meeting of the Board.

Section 8. Vacancies: Members of the Board

If a vacancy should occur in the membership of the elected Board of Directors, any Past President may be appointed by action of the remaining members of the Board to temporarily fill such vacancy until the next

regularly scheduled election. At the next regularly scheduled election nominations will be presented to fill the vacancy for the unexpired portion of the term remaining.

Section 9. Vacancies: President and Other Officers

If the office of the President shall become vacant, the President-Elect shall thereupon become President of the Association for the unexpired term, followed by his or her duly elected term. In the event the office of President becomes vacant at a time when the office of President-Elect is also vacant, the Presidency shall be filled for the remainder of the term by the action of the Board of Directors. If any other officer position shall become vacant, the office may be filled for the remainder of the term by action of the Board.

ARTICLE VII
Committees

Section 1. Committee Formation

The Board of Directors shall form and adopt terms of reference for such standing or special boards, committees, subcommittees, task forces, or task groups as may be required by these Bylaws or as the Board may determine necessary to carry out the affairs of the Association.

Section 2. Committee Appointments

Subject to the requirements of these Bylaws and the specific terms of reference adopted by the Board, the President shall make the appointments to fill the vacancies occurring in the Association's standing or special boards, committees, subcommittees, task forces, or task groups.

ARTICLE VIII
Official Methods of Analysis

The Board of Directors (BoD) is empowered to develop written policies and procedures for the study, adoption, and change in status of the Official Methods of Analysis of AOAC INTERNATIONAL. Implementation of the policies and procedures shall be delegated to an Official Methods Board (OMB).

Section 1. Composition of the Official Methods Board

The Official Methods Board shall consist of a chair and a vice chair, and members who are recommended by the chair. The chair, vice chair and members are appointed by the President of AOAC INTERNATIONAL. The OMB shall be composed of members representing a balance of government, industry, and academia as appropriate to the scope of the group and shall not be dominated by any single interest.

Section 2. Purpose of the Official Methods Board

The OMB shall serve the Association in a scientific and advisory capacity on methods and the process of their adoption. The OMB shall be responsible for implementation of procedures adopted by the BoD, according to the principles in section 3 below.

Section 3. Principles of the Official Methods Program

A. Adequate records of technical data, discussions, and decisions on the study, adoption, and change of status of Official Methods of Analysis shall be maintained for a reasonable time.

B. Timely notice of proposed method studies, adoption, or change in status shall be published in an Association publication that is circulated to the members.

C. Opportunity shall be provided for materially interested parties to submit input during method study and adoption procedures and to submit comments on the adoption, use of, or change in status of specific methods.

D. Methods submitted to the OMB for inclusion in the OMA shall be thoroughly studied, scientifically reviewed, and available in published form prior to adoption as Final Action by the OMB.

E. The OMB shall adopt methods as Final Action.

ARTICLE IX
Meetings

Section 1. Annual Meeting

The annual business meeting of the Association shall be held at the time and place decided by the Board of Directors. A special meeting of the entire Association may be called by the Board of Directors; announcement thereof shall be made at least thirty (30) days prior to the time of said meeting.

Section 2. Quorum

One hundred Individual Members who are present in person or by proxy and entitled to vote shall constitute a quorum at any meeting of the Association which is duly called pursuant to the provisions of these Bylaws.

ARTICLE X
Voting

Section 1. Voting by Ballot

By direction of the Board of Directors, unless otherwise required by these Bylaws or conducted under alternative procedures established under these Bylaws, voting on any matter, including the election of officers and directors, the election of Honorary Members, amendment of the Bylaws, and the approval of dues, may be conducted by ballot of the voting membership by any of the following means (whichever is deemed appropriate at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission, and the question(s) thus presented shall be determined according to the votes received, provided in each case votes of at least five (5) percent of the voting membership shall be received. Any and all action taken in pursuance of a vote by any of the means indicated above (whichever the Board deemed appropriate at the time)

in each case shall be binding upon the Association in the same manner as would be action taken at a duly called meeting and shall become effective, unless otherwise provided for in these Bylaws or otherwise stated in the ballot, on the day following certification of the vote.

Section 2. Voting by Proxy

At any duly called meeting of Individual Members, a member-of-record, as determined thirty (30) days prior to any meeting and who is entitled to vote, may vote by proxy executed in writing by the Individual Member or his or her duly authorized attorney-in-fact. No proxy shall be valid for more than eleven (11) months after the date of its execution unless otherwise provided in the proxy.

ARTICLE XI
Earnings and Assets

Section 1. Non-Profit Status

A. Regardless of any provision of the Bylaws which may be construed otherwise:

[1] No part of the net earnings of the Association shall under any circumstances inure to the benefit of any member or individual.

[2] The Association shall not be operated for a private profit.

B. On lawful dissolution of the Association and after settlement of all just obligations of the Association, the Board of Directors shall distribute all remaining assets of the Association to one (1) or more organizations selected by the Board of Directors which have been held exempt from Federal Income Tax as organizations described in section 501(c)(3) of the Internal Revenue Code of 1954.

Section 2. Political Activities

A. No substantial part of the Association's activities shall consist of carrying on propaganda or otherwise attempting to influence local, state, or national legislation. All activities of the Association shall be determined by the Board of Directors.

B. The Association shall not participate or intervene in any manner in any campaign on behalf of any candidate for a political office.

ARTICLE XII
Sections

Section 1. Sections

The Board of Directors shall set geographic limits and grant authority to groups of Individual Members of the Association residing or working in the same geographical areas for the establishment of Sections.

Section 2. Purpose of Sections

The purpose of Sections shall be to promote and further the purpose of the Association.

Section 3. Membership in Sections

Individuals interested in the purpose of the Section shall be eligible for Section membership. Only Individual Members of the Association shall be eligible for election to the Executive Committee of the Section.

Section 4. Bylaws of Sections

Subject to approval of the Board of Directors, each Section shall adopt, for its own governance, bylaws not inconsistent with these Bylaws.

Section 5. Dissolution of Sections

When any Section shall cease to function as a Section for a period of more than one year, or if its membership shall be less than ten (10) Individual Members of the Association for a period of one (1) year, the Board of Directors may terminate the existence of such Section.

Section 6. Actions of Sections

No act of a Section or its members shall be considered an act of the Association unless expressly authorized, ratified, or affirmed by the Board of Directors.

ARTICLE XIII
Technical Divisions

Section 1. Purpose

Technical Divisions shall represent communities of interest within the Association which have the purpose of furthering the purpose of the Association through the development of the analytical sciences either in a commodity-based or scientific discipline-based field. Their activities shall not duplicate the organizational structure nor conflict with the policies or procedures for the adoption of official methods of analysis by the Association.

Section 2. Creation, Combination, Discontinuance, or Change

Technical Divisions may be created, existing Technical Divisions may be combined or discontinued, or the name of a Technical Division may be changed under policies and procedures adopted by the Board of Directors. Each Technical Division shall adopt bylaws not inconsistent with these Bylaws. The jurisdiction of each Technical Division shall be described in its bylaws. No act of any Technical Division or its members shall be considered an act of the Association unless expressly authorized, ratified, or affirmed by the Board of Directors.

ARTICLE XIV
Indemnification

The Association shall have the power to pay, by indemnity, reimbursement, or otherwise, to or for the use of any person designated by resolution of the Board of Directors who was or is a party or is threatened to be made a party to any threatened, pending, or completed action, suit, or proceeding, whether civil, criminal, administrative, or investigative (other than an action by or on behalf of the Association), by reason of the fact he or she is or was a director, officer, committee member, employee or agent of the Association, or was serving as such for another at the request of the Association, against expenses (including legal, accounting, witness and other), judgments, fines, and amounts paid in settlement so long as such person was not found by a court of competent jurisdiction to have been willfully negligent of the interests of the Association or such person had reasonable cause to believe that his or her conduct was lawful.

ARTICLE XV
Parliamentary Authority

The rules contained in the current edition of *Robert's Rules of Order Newly Revised* shall govern the Association in all cases in which they are applicable and in which they are not inconsistent with these Bylaws or any special rules of order the Association may adopt.

ARTICLE XVI
Amendments to the Bylaws

These Bylaws may be amended, repealed, or altered, in whole or in part, by a three-fourths vote: (a) of the Individual Members at any annual business or duly called special meeting of the Association, provided notice of any amendment proposed for consideration shall be sent by any of the following means (whichever may be deemed appropriate at the time): mail, telephone call, telegram, cablegram, electronic mail or other means of electronic or telephonic transmission to the last recorded address or number of each Individual Member at least thirty (30) days prior to the date of the meeting; or (b) by approval of the Individual Members through ballot sent by any means indicated above in accordance with the provisions of Article X, Voting.

All proposed amendments of these Bylaws shall be presented in writing to the Board of Directors. The Board shall present the proposals to the Association membership, with recommendations. All amendments to the Bylaws, unless otherwise stated, will become effective at the adjournment of the meeting where action is taken or on the day following the certification of a vote by mail ballot.

AOAC INTERNATIONAL
POLICY ON THE USE OF THE
ASSOCIATION NAME, INITIALS,
IDENTIFYING INSIGNIA, LETTERHEAD, AND BUSINESS CARDS

Introduction

The following policy and guidelines for the use of the name, initials, and other identifying insignia of AOAC INTERNATIONAL have been developed in order to protect the reputation, image, legal integrity and property of the Association.

The name of the Association, as stated in its bylaws, is "AOAC INTERNATIONAL". The Association is also known by its initials, AOAC, and by its logo, illustrated below, which incorporates the Association name and a representation of a microscope, book, and flask. The AOAC logo is owned by the Association and is registered with the U.S. Patent and Trademark Office.

AOAC INTERNATIONAL

The full Association insignia, illustrated below, is comprised of the logo and the tagline, "The Scientific Association Dedicated to Analytical Excellence," shown below. The typeface used is Largo. The AOAC tagline is owned by the Association and is registered with the U.S. Patent and Trademark office.

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

Policy

Policy on the use of the Association's name and logo is established by the AOAC Board of Directors as follows:

> "The Board approves and encourages reference to the Association by name, either as AOAC INTERNATIONAL or as AOAC; or reference to our registered trademark, AOAC®, in appropriate settings to describe our programs, products, etc., in scientific literature and other instances so long as the reference is fair, accurate, complete and truthful and does not indicate or imply unauthorized endorsement of any kind.
>
> The insignia (logo) of AOAC INTERNATIONAL is a registered trade and service mark and shall not be reproduced or used by any person or organization other than the Association, its elected and appointed officers, sections, or committees, without the prior written permission of the Association. Those authorized to use the AOAC INTERNATIONAL insignia shall use it only for the purposes for which permission has been specifically granted.
>
> The name and insignia of the Association shall not be used by any person or organization in any way which indicates, tends to indicate, or implies AOAC official endorsement of any product, service, program, company, organization, event or person, endorsement of which, has not been authorized by the Association, or which suggests that membership in the Association is available to any organization."

The Executive Director, in accordance with the above stated policy, is authorized to process, approve, fix rules, and make available materials containing the Association name and insignia.

It should be noted that neither the Association's name nor its insignia nor part of its insignia may be incorporated into any personal, company, organization, or any other stationery other than that of the Association; nor may any statement be included in the printed portion of such stationery which states or implies that an individual, company, or other organization is a Member of the Association.

Instructions

1. Reproduction or use of the Association name or insignia requires prior approval by the Executive Director or his designate.

2. Association insignia should not be altered in any manner without approval of the Executive Director or his designate, except to be enlarged or reduced in their entirety.

3. Artwork for reproducing the Association name or insignia, including those incorporating approved alterations, will be provided on request to those authorized to use them (make such requests to the AOAC Marketing Department). Examples of the types of alterations that would be approved are inclusion of a section name in or the addition of an officer's name and address to the letterhead insignia.

4. When the Association name is used without other text as a heading, it should, when possible, be set in the Largo typeface.

5. Although other colors may be used, AOAC blue, PMS 287, is the preferred color when printing the AOAC insignia, especially in formal and official documents. It is, of course, often necessary and acceptable to reproduce the insignia in black.

6. Do not print one part of the logo or insignia in one color and other parts in another color.

7. The letterhead of AOAC INTERNATIONAL shall not be used by any person or organization other than the Association, its elected and appointed officers, staff, sections, or committees; except by special permission.

   Correspondence of AOAC official business should be conducted using AOAC letterhead. However, those authorized to use AOAC letterhead shall use it for official AOAC business only.

   Copies of all correspondence using AOAC letterhead or conducting AOAC official business, whether on AOAC letterhead or not, must be sent to the appropriate office at AOAC headquarters.

8. AOAC INTERNATIONAL business cards shall not be used by any person or organization other than the Association, its staff, and elected officials, except by special permission.

   Those authorized to use AOAC business cards shall use them for official AOAC business only and shall not represent themselves as having authority to bind the Association beyond that authorized.

Sanctions

1. Upon learning of any violation of the above policy, the Executive Director or a designate will notify the individual or organization that they are in violation of AOAC policy and will ask them to refrain from further misuse of the AOAC name or insignia.

2. If the misuse is by an Individual Member or Sustaining Member of the Association, and the misuse continues after notification, the Board of Directors will take appropriate action.

3. If continued misuse is by a nonmember of the Association or if a member continues misuse in spite of notification and Board action, ultimately, the Association will take legal action to protect its property, legal integrity, reputation, and image.

* * * * * *

Adopted by the AOAC Board of Directors: September 24, 1989
Revised: June 13, 1991; February 26, 1992; March 21, 1995; October 1996

AOAC INTERNATIONAL
ANTITRUST POLICY
STATEMENT AND GUIDELINES

Introduction

It is the policy of AOAC INTERNATIONAL (AOAC) and its members to comply strictly with all laws applicable to AOAC activities. Because AOAC activities frequently involve cooperative undertakings and meetings where competitors may be present, it is important to emphasize the on_going commitment of our members and the Association to full compliance with national and other antitrust laws. This statement is a reminder of that commitment and should be used as a general guide for AOAC and related individual activities and meetings.

Responsibility for Antitrust Compliance

The Association's structure is fashioned and its programs are carried out in conformance with antitrust standards. However, an equal responsibility for antitrust compliance __ which includes avoidance of even an appearance of improper activity __ belongs to the individual. Even the appearance of improper activity must be avoided because the courts have taken the position that actual proof of misconduct is not required under the law. All that is required is whether misconduct can be inferred from the individual's activities.

Employers and AOAC depend on individual good judgment to avoid all discussions and activities which may involve improper subject matter and improper procedures. AOAC staff members work conscientiously to avoid subject matter or discussion which may have unintended implications, and counsel for the Association can provide guidance with regard to these matters. It is important for the individual to realize, however, that the competitive significance of a particular conduct or communication probably is evident only to the individual who is directly involved in such matters.

Antitrust Guidelines

In general, the U.S. antitrust laws seek to preserve a free, competitive economy and trade in the United States and in commerce with foreign countries. Laws in other countries have similar objectives. Competitors (including individuals) may not restrain competition among themselves with reference to the price, quality, or distribution of their products, and they may not act in concert to restrict the competitive capabilities or opportunities of competitors, suppliers, or customers.

Although the Justice Department and Federal Trade Commission generally enforce the U.S. antitrust laws, private parties can bring their own lawsuits.

Penalties for violating the U.S. and other antitrust laws are severe: corporations are subject to heavy fines and injunctive decrees, and may have to pay substantial damage judgments to injured competitors, suppliers, or customers. Individuals are subject to criminal prosecution, and will be punished by fines and imprisonment.
Under current U.S. federal sentencing guidelines, individuals found guilty of bid rigging, price fixing, or market allocation must be sent to jail for at least 4 to 10 months and must pay substantial minimum fines.

Since the individual has an important responsibility in ensuring antitrust compliance in AOAC activities, everyone should read and heed the following guidelines.

1. Don't make any effort to bring about or prevent the standardization of any method or product for the purpose or intent of preventing the manufacture or sale of any method or product not conforming to a specified standard.

2. Don't discuss with competitors your own or the competitors' prices, or anything that might affect prices such as costs, discounts, terms of sale, distribution, volume of production, profit margins, territories, or customers.

3. Don't make announcements or statements at AOAC functions, outside leased exhibit space, about your own prices or those of competitors.

4. Don't disclose to others at meetings or otherwise any competitively sensitive information.

5. Don't attempt to use the Association to restrict the economic activities of any firm or any individual.

6. Don't stay at a meeting where any such price or anti_competitive talk occurs.

7. Do conduct all AOAC business meetings in accordance with AOAC rules. These rules require that an AOAC staff member be present or available, the meeting be conducted by a knowledgeable chair, the agenda be followed, and minutes be kept.

8. Do confer with counsel before raising any topic or making any statement with competitive ramifications.

9. Do send copies of meeting minutes and all AOAC_related correspondence to the staff member involved in the activity.

10. Do alert the AOAC staff to any inaccuracies in proposed or existing methods and statements issued, or to be issued, by AOAC and to any conduct not in conformance with these guidelines.

Conclusion

Compliance with these guidelines involves not only avoidance of antitrust violations, but avoidance of any behavior which might be so construed. Bear in mind, however, that the above antitrust laws are stated in general terms, and that this statement is not a summary of applicable laws. It is intended only to highlight and emphasize the principal antitrust standards which are relevant to AOAC programs. You must, therefore, seek the guidance of either AOAC counsel or your own counsel if antitrust questions arise.

* * * * *

Adopted by the AOAC Board of Directors: September 24, 1989
Revised: March 11, 1991
Revised October 1996

AOAC INTERNATIONAL

POLICY AND PROCEDURES ON

VOLUNTEER CONFLICT OF INTEREST

Statement of Policy

While it is not the intention of AOAC INTERNATIONAL (AOAC) to restrict the personal, professional, or proprietary activities of AOAC members nor to preclude or restrict participation in Association affairs solely by reason of such activities, it is the sense of AOAC that conflicts of interest or even the appearance of conflicts of interest on the part of AOAC volunteers should be avoided. Where this is not possible or practical under the circumstances, there shall be written disclosure by the volunteers of actual or potential conflicts of interest in order to ensure the credibility and integrity of AOAC. Such written disclosure shall be made to any individual or group within the Association which is reviewing a recommendation which the volunteer had a part in formulating and in which the volunteer has a material interest causing an actual or potential conflict of interest.

AOAC requires disclosure of actual or potential conflicts of interest as a condition of active participation in the business of the Association. The burden of disclosure of conflicts of interest or the appearance of conflicts of interest falls upon the volunteer.

A disclosed conflict of interest will not in itself bar an AOAC member from participation in Association activities, but a three-fourths majority of the AOAC group reviewing the issue presenting the conflict must concur by secret ballot that the volunteer's continued participation is necessary and will not unreasonably jeopardize the integrity of the decision-making process.

Employees of AOAC are governed by the provision of the AOAC policy on conflict of interest by staff. If that policy is in disagreement with or mute on matters covered by this policy, the provisions of this policy shall prevail and apply to staff as well.

Illustrations of Conflicts of Interest

1. A volunteer who is serving as a committee member or referee engaged in the evaluation of a method or device; who is also an employee of or receiving a fee from the firm which is manufacturing or distributing the method or device or is an employee of or receiving a fee from a competing firm.

2. A volunteer who is requested to evaluate a proposed method or a related collaborative study in which data are presented that appear detrimental (or favorable) to a product distributed or a position supported by the volunteer's employer.

3. A referee who is conducting a study and evaluating the results of an instrument, a kit, or a piece of equipment which will be provided gratis by the manufacturer or distributor to one or more of the participating laboratories, including his or her own laboratory, at the conclusion of the study.

4. Sponsorship of a collaborative study by an interest (which may include the referee) which stands to profit from the results; such sponsorship usually involving the privilege granted by the investigator to permit the sponsor to review and comment upon the results prior to AOAC evaluation.

5. A volunteer asked to review a manuscript submitted for publication when the manuscript contains information which is critical of a proprietary or other interest of the reviewer.

The foregoing are intended as illustrative and should not be interpreted to be all-inclusive examples of conflicts of interest AOAC volunteers may find themselves involved in.

Do's and Don'ts

Do avoid the appearance as well as the fact of a conflict of interest.

Do make written disclosure of any material interest which may constitute a conflict of interest or the appearance of a conflict of interest.

Do not accept payment or gifts for services rendered as a volunteer of the Association without disclosing such payment or gifts.

Do not vote on any issue before an AOAC decision-making body where you have the appearance of or an actual conflict of interest regarding the recommendation or decision before that body.

Do not participate in an AOAC decision-making body without written disclosure of actual or potential conflicts of interest in the issues before that body.

Do not accept a position of responsibility as an AOAC volunteer, without disclosure, where the discharge of the accepted responsibility will be or may appear to be influenced by proprietary or other conflicting interests.

Procedures

Each volunteer elected or appointed to an AOAC position of responsibility shall be sent, at the time of election or appointment, a copy of this policy and shall be advised of the requirement to adhere to the provisions herein as a condition for active participation in the business of the Association. Each volunteer, at the time of his or her election or appointment, shall indicate, in writing, on a form provided for this purpose by AOAC, that he or she has read and accepts this policy.

Each year, at the spring meeting of the AOAC Board of Directors, the Executive Director shall submit a report certifying the requirements of this policy have been met; including the names and positions of any elected or appointed volunteers who have not at that time indicated in writing that they have accepted the policy.

Anyone with knowledge of specific instances in which the provisions of this policy have not been complied with shall report these instances to the Board of Directors, via the Office of the Executive Director, as soon as discovered.

* * * * * *

Adopted: March 2, 1989
Revised: March 28, 1990
Revised: October 1996

AOAC INTERNATIONAL

TERMS OF REFERENCE

I. NAME:

OFFICIAL METHODS BOARD (OMB)

II. MISSION:

To serve the Association in a scientific and advisory capacity on standards and methods with ethical, timely, open and independent scientific oversight for the implementation of standards development and conformity assessment policies and procedures of AOAC INTERNATIONAL.

III. RESPONSIBILITIES:

To provide ethical, timely, open and independent scientific oversight for the policies and procedures of AOAC INTERNATIONAL.

To approve "Final Action" status for First Action Methods (new and revised) following a proactive review;

To repeal methods, if necessary, in accordance with established policies and procedures;

To participate in addressing appeals and requests for action or guidance, and in resolving disputes;

To endorse and monitor all voluntary consensus panels for appropriate representation and balance of stakeholders' perspectives;

To endorse and monitor all volunteer subject matter experts for volunteer conformity assessment activities;

To adopt and monitor scientific and technical guidance and references;

To acknowledge outstanding scientific and technical volunteer activity and achievement within AOAC;

To actively participate in AOAC standards development activities and maintain and communicate explicit knowledge of AOAC standards development and conformity assessment;

IV. COMPOSITION AND ORGANIZATION:

The Official Methods Board shall consist of up to 13 voting members including a Chair, a Vice-chair, the Chair of the Committee on Safety and the Chair of the Committee on Statistics. The Committee on Safety and the Committee on Statistics may contain co-chairs. The co-chairs for these committees represent one vote on the OMB. Members of the OMB may serve in multiple volunteer roles for the benefit of the Association. The Chair of the Official Methods Board shall have previously served as a member of the Official Methods Board. The Chair, Vice-chair, and members of the Official Methods Board including the chairs of standing committees shall be appointed for a term of three years. A member of the OMB may be reappointed upon the recommendation of the Chair of the Official Methods Board with a maximum term of service of six (6) years. Exceptions may be made at the discretion of the President. The Chair of the Official Methods Board is eligible to serve an additional post chair term of up to three (3) years as an *ex-officio* member. Members of the Official Methods Board must be members of AOAC.

All members of the Official Methods Board are recommended by the Chair and appointed by the President. All Official Methods Board members serve at the pleasure of the President.

The Official Methods Board represents the membership of AOAC INTERNATIONAL. It shall be composed of members representing a balance of scientific expertise, government, industry, and academia as appropriate to the scope of the Board. Every effort should be made to include international representation on the Board.

Additional working groups, task forces, and other appropriate subgroups shall be appointed as needs arise by the Chair of the Official Methods Board.

V. STAFF LIAISON:

The Executive Director shall assign a member of the staff to serve as staff liaison.

VI. REVIEW SCHEDULE:

Every three years.

VII. DATE ESTABLISHED:

Renamed in 1981

VIII. DATES REVIEWED

01/2008, 7/2016

IX. DATES REVISED:

9/89; 5/90; 1/91; 8/06;
02/07; 07/07; 2/08; 4/13; 8/13

OFFICIAL METHODS BOARD MEETING

Thursday – Friday, June 22-23, 2017
Thursday - 10:00 AM ET / 9:00 AM CT – 6:00 PM ET / 5:00 PM CT
Friday – 9:30 AM ET / 8:30 AM ET – 4:00 PM / 3:00 PM ET

AOAC INTERNATIONAL Headquarters
2275 Research Blvd
Suite 300
Rockville, Maryland 20871

Mérieux NutriSciences Headquarters
111 East Wacker Drive
23rd Floor
Chicago, Illinois 60651

DRAFT MEETING AGENDA

Erin Crowley (Q Laboratories) – Chair and Moderator

I. PRELIMINARY ITEMS

a. Welcome and Introductions *(Goodwin)*
Jonathan Goodwin (Interim Executive Director, AOAC INTERNATIONAL) will greet and welcome OMB members and initiate introductions.

b. Remarks from Mérieux NutriSciences *(Miller)*
Jim Miller (President of Mérieux NutriSciences North America) will welcome and address OMB members.

c. Call to Order and Announcements (*Crowley)*
Erin Crowley (Q Laboratories) will call the meeting to order and share and entertain announcements.

d. Review of AOAC Policy Documents/Terms of Reference *(Crowley)*
Crowley will review AOAC policy documents and the OMB Terms of Reference.

e. Review of Draft Agenda* *(Crowley)*
Crowley will initiate a review of the draft meeting agenda and will facilitate the approval of a final version of the agenda.

f. Review of April 13, 2017 OMB Teleconference Minutes* *(Crowley)*
Crowley will initiate a review of the April 13, 2017 draft OMB Teleconference Meeting Minutes and will facilitate the approval of the final version of the minutes.

g. Executive Office and Board of Directors *(Goodwin/Johnson)*
Goodwin and Johnson will provide an update on Executive Office activities, International activities, AOAC strategic planning, and the June Board of Directors meeting.

II. AOAC AND CODEX ENGAGEMENT *(Sullivan/Szyplka)*
- **a. Overview of CODEX Method Process**
- **b. Review of CODEX Standard 234**

III. OMB AWARDS FOR 2017 (*Crowley/McKenzie)*
- **a. Award in Recognition for Technical and Scientific Excellence (Team award) ***
- **b. Expert Review Panel of the Year (Team award) ***
- **c. Technical Service Award (Individual award) ***
  - i. Nominations

**** Items that require or may require a vote***

ii. AOAC Experts (formerly known as General Referees)

d. Method of the Year (Individual award) *

IV. AOAC ERP RECOMMENDATIONS

a. ERP for SPIFAN Nutrient Methods* *(Sullivan/Crowley)*

i. Biotin

ii. Vitamin D

b. ERP for PAH Methods* *(OMB Liaison/Crowley)*

i. AOAC 2014.08

c. ERP for Pesticide Residue Methods* *(Boison/Crowley)*

i. AOAC 2014.09

V. AOAC OMB COMMITTEES AND WORKING GROUPS

a. Committee on Safety *(Salfinger)**

i. Committee's Revisions to Terms of Reference

b. Committee on Statistics *(Sudberg)**

i. Intermediate Precision *(Coates/Sudberg)*

ii. Committee's Revisions to Terms of Reference *(Sudberg/McKenzie/Boyd)*

c. OMB New Member Selection Committee (*Crowley/McKenzie)*

d. OMB Selection of Vice-Chair *(Crowley/McKenzie)*

VI. AOAC CONFORMITY ASSESSMENT ACTIVITIES *(McKenzie)*

a. Training and Education

b. Volunteer Confidentiality and Nondisclosure

c. Sole Source Modifications – Round Two

i. ERP for Solids in Syrups

VII. AOAC STANDARDS DEVELOPMENT ACTIVITIES *(Crowley/McKenzie)*

a. Status of Projects in AOAC Stakeholder Panels

b. OMB Liaisons

c. Review and approval of Stakeholder Panel Representative Voting Members*

d. Training and Education

VIII. AOAC ANNUAL MEETING ACTIVITIES *(McKenzie)*

a. Standards Activities and ERP Meetings

b. OMB Meeting

c. New AOAC Member Engagement

IX. OMB MEMBER UPDATES *(Crowley)*

X. ADJOURNMENT

**** Items that require or may require a vote***

AOAC OFFICIAL METHODS BOARD TELECONFERENCE

April 13, 2017

1:00pm – 2:30pm ET

DRAFT MEETING MINUTES

OMB MEMBERS *(present during all or part of the meeting)*

Erin Crowley	Q Laboratories	Chair
Douglas Abbott	Independent Consultant	Member
Joe Boison	Canadian Food Inspection Agency	Member
Amy Brown	Florida Dept. of Agriculture and Consumer Services	Member
Esther Campos Giménez	Nestlé Research Centre	Member
Don Gilliland	Abbott Nutrition	Member
Katerina Mastovska	Covance	Member
Wendy McMahon	Mérieux NutriSciences	Member
Melissa Phillips *(and with proxy)*	US NIST	Member
Yvonne Salfinger *(proxy)*	Independent Consultant	Member
Brad Stawick	Microbac	Member
Sidney Sudberg	Alkemist	Member

OMB MEMBERS ABSENT *(without proxy)*

Shauna Roman	Reckitt Benckiser	Past Chair-Ex-Officio

AOAC STAFF *(present during all or part of the meeting)*

Scott Coates
Deborah McKenzie

I. PRELIMINARY ITEMS

a. Call to Order/Introductions/Announcements
Crowley called the meeting to order at 1:03pm ET.

b. Crowley called OMB's attention to the AOAC policy documents and reminded all attendees to review the documents and that the meeting will be held in accordance to these policies.

c. Review and Approval of Draft Meeting Agenda
MOTION: For OMB to approve the agenda as presented.
Boison moved and Gilliland seconded. Consensus: Unanimous.

d. Review and Approval of Draft OMB meeting minutes.
MOTION: For OMB to approve the March 2, 2017 OMB meeting minutes as presented.
Brown moved and Sudberg seconded. Consensus: Unanimous.

II. OMB SUMMER MEETING

a. Crowley and McKenzie reviewed the survey results and confirmed the OMB summer meeting dates and locations with OMB. The meeting is being held on June 22-23, 2017. The meeting will be a webconference hosted out of both AOAC HQ and Mérieux NutriSciences in Chicago.

b. **ACTION ITEMS:** Crowley and McKenzie to follow up with John Szpylka of Mérieux NutriSciences.

III. AOAC STANDARDS DEVELOPMENT & CONFORMITY ASSESSMENT ACTIVITIES

a. OMB liaisons and staff provided a briefing to the OMB of all Mid-Year meeting events.
ACTION ITEMS: Staff to add specific discussion points regarding refinement of procedures for voting panel changes, quorum, and sole source modification for OMB on the draft agenda for the OMB summer meeting.

IV. ADJOURNMENT

a. Meeting adjourned on Thursday, April 13, 2017 at 2:34 pm ET with unanimous consensus on the following motion.
MOTION: To adjourn the meeting.
Boison moved; Sudberg seconded. Consensus: Unanimous – Meeting adjourned.

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item 1g – Executive Office and Board of Directors Update**

This will be a brief presentation with a verbal update on the AOAC strategic planning and the June 14, 2017 Board of Directors Meeting by Jonathan Goodwin and Ron Johnson.

2

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item 2 – AOAC and Codex Engagement**

Darryl Sullivan and John Szpylka will share outcomes of the Codex Committee on Methods of Analysis and Sampling (CCMAS) meeting in May 2017 and an AOAC opportunity and effort resulting from the meeting.

Enclosures:

1. Presentation by Sullivan and Szpylka
2. Codex Standard 234
3. Report of the CCMAS Meeting

AOAC and CODEX Engagement

June 2017

Darryl Sullivan, Sect., AOAC Board of Directors
John Szpylka, Mérieux NutriSciences
,

AOAC INTERNATIONAL HEADQUARTERS
2275 Research Boulevard, Suite 300
Rockville, Maryland 20840, USA

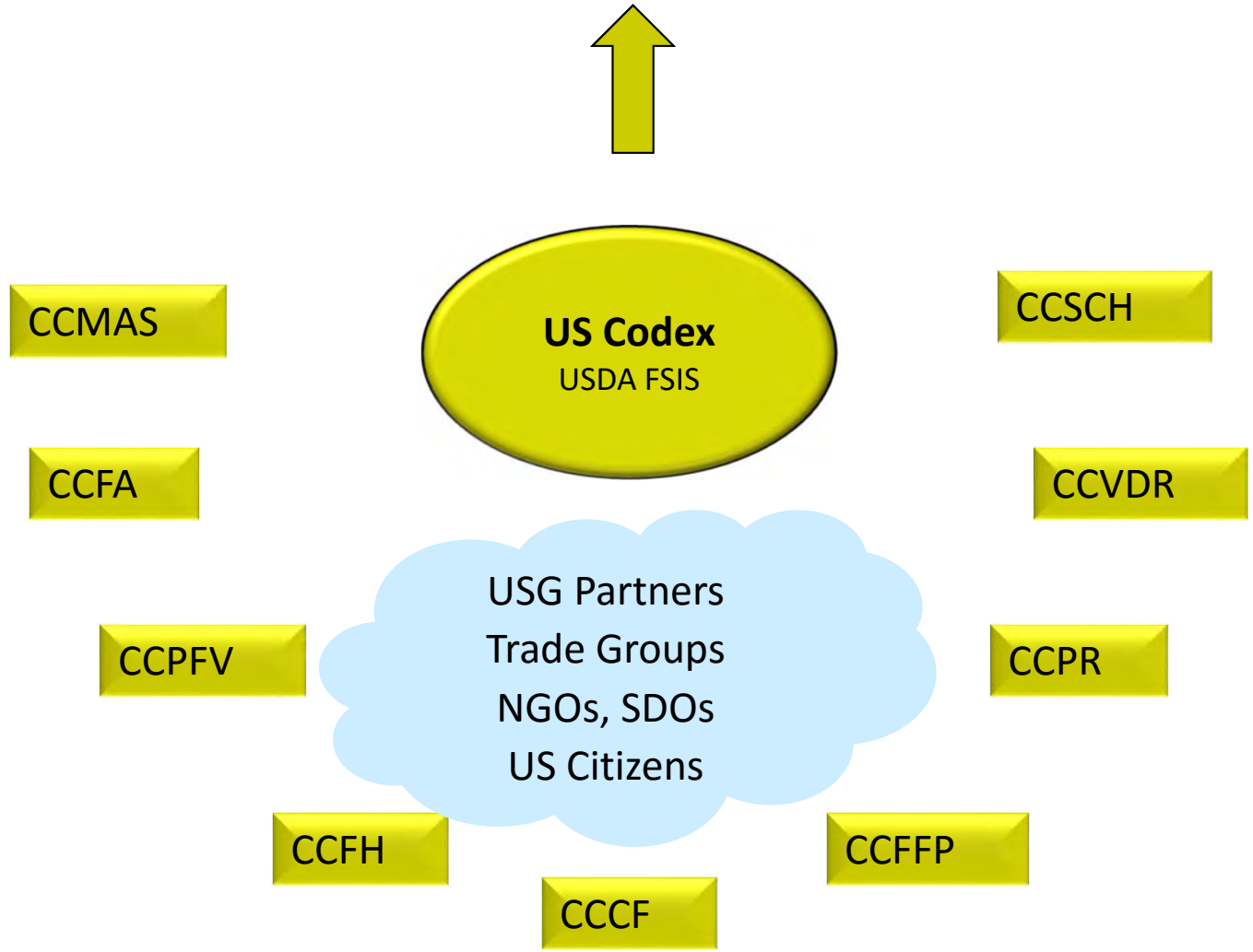
- Updates on CCMAS Meeting
- Codex Adoption of Methods of Analysis
- Maintenance of Codex STAN 234-1999
- AOAC Methods in Codex

CCMAS 2017

- Meeting held on May 8-12, 2017
- Endorsed the following methods to go to CAC
  - Vitamin B12 (AOAC 2011.10 | ISO 20634)
  - Vitamin C (AOAC 2012.22 | ISO DIS 20635)
  - Vitamin E (AOAC 2012.10 | ISO 20633)
  - Chromium/Molybdenum/Selenium (AOAC 2011.19 | ISO 20649 | IDF 235)
  - Myo-inositol (AOAC 2011.18 | ISO 20637)
  - Total Fatty Acid Profile (AOAC 2012.13 | ISO 16958 | IDF 231)

CODEX ALIMENTARIUS
International Food Standards

World Health Organization
Food and Agriculture Organization of the United Nations



AOAC INTERNATIONAL

Role of SDOs in Codex

- Codex membership are countries
- SDOs are observers
  - For CCMAS
    - Interagency Meeting
      - Consists of SDOs
      - Meet as the Interagency Meeting (IAM) in advance of CCMAS
      - IAM meeting summary of incorporated into CCMAS meeting report

Codex Committee on Methods of Analysis and Sampling (CCMAS)

CCMAS Endorses Methods, Establishes Numeric Criteria and Establishes Procedures and Guidance for Evaluating Methods

~~Microbiological, Food Additives, Pesticides, Veterinary Drugs~~

STAN 234-1991 Recommended Methods of Analysis

GL 50-2004 General Guidelines on Sampling

GL 54-2004 Guidelines on Measurement Uncertainty

Codex Committee on Methods of Analysis and Sampling (CCMAS)

- CCMAS does not develop methods, but endorses methods developed by others
- New methods are proposed by the Commodity Committee
- CCMAS then reviews method performance and determines if it should be endorsed
- At endorsement the method is Typed (I-IV) by CCMAS
- Once endorsed Method must be Adopted by CAC

Codex Committee on Methods of Analysis and Sampling (CCMAS)

- CCMAS does not develop methods, but endorses **methods developed by others**
- **New methods are proposed by the Commodity or General Subject Committee**
- CCMAS then **reviews method performance and determines if it should be endorsed**
- At endorsement the **method is Typed (I-IV) by CCMAS**
- Once endorsed Method must be Adopted by CAC

CCMAS Method Typing

Defining Methods (Type I)

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.

- If a Type I Method is listed in STAN 234 for a particular Commodity and Provision, no other method will be listed.
- The Criteria Approach is not applicable to Type I Methods

CCMAS Method Typing

Reference Methods (Type II)

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.

Alternative Approved Methods (Type III)

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.

Tentative Method (Type IV)

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.

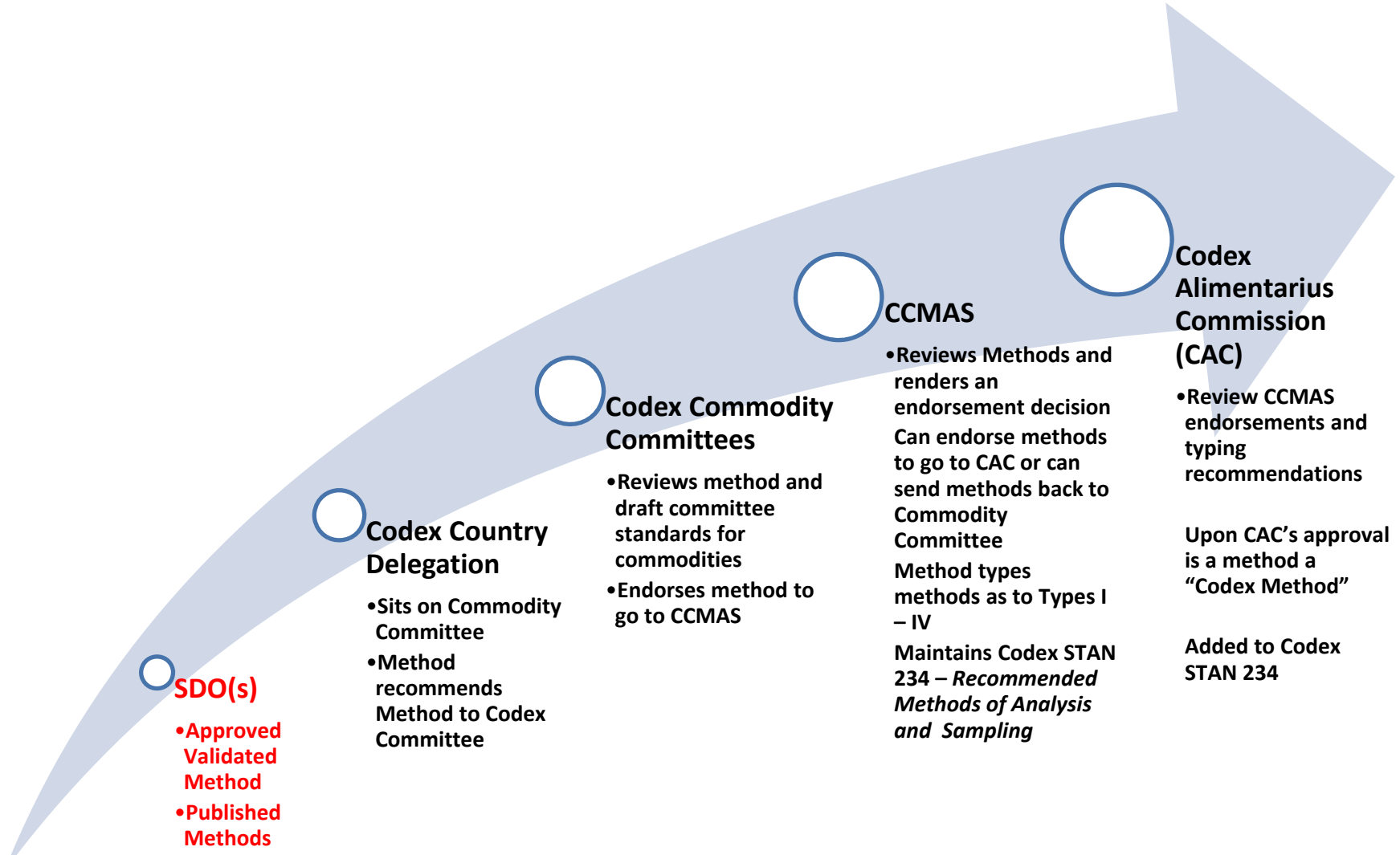
Method Criteria

"Any Codex Committee may…and/or develop a set of criteria to which a method used for the determination must comply."

Procedural Manual 24th Edition

- Creating Numeric Criteria
  - Determined from a specification, or "Convert" a Specific Method
- Establishing Numeric Criteria can be Performed by CCMAS
  - Codex Committee must approve of the approach.
- No "dispute resolution" method identified.

Step to a Codex Method



Next Steps for CCMAS Endorsed Methods

- CCMAS endorsed the following methods to go to CAC as Type II methods (used for trade dispute resolution)
  - Vitamin B12 (AOAC 2011.10 | ISO 20634)
  - Vitamin C (AOAC 2012.22 | ISO DIS 20635)
  - Vitamin E (AOAC 2012.10 | ISO 20633)
  - Chromium/Molybdenum/Selenium (AOAC 2011.19 | ISO 20649 | IDF 235)
  - Myo-inositol (AOAC 2011.18 | ISO 20637)
  - Total Fatty Acid Profile (AOAC 2012.13 | ISO 16958 | IDF 231)
- Methods will be on the CAC agenda
  - July 22, 2017 in Rome, Italy

CCMAS Maintenance of STAN 234

- CODEX STAN 234-1999
  - Last adopted in 1999, but modified in 2016
- Methods of analysis are incorporated by reference from SDOs and others
- Parts A and B
  - Part A - Methods of analysis listed by commodity categories
  - Part B - Methods of sampling listed by commodity categories

Codex STAN 234-1999

Dilemma

- The standard has a review schedule of every 5 years or so.
- The last adoption of the standard was in 1999
- Some of the methods are outdated, modified, etc…
- Codex process does not allow for review the standard as methods are incorporated by reference
- AOAC has the most number of methods in the standard
- AOAC methods were candidates for removal in a number of areas about seven years ago

Recommended Solution

- Each SDO to review their own methods in Codex STAN 234-1999
- Approximate five-year plan for review
- Make recommendations back to Codex regarding the methods

Discussion and Questions?

CODEX ALIMENTARIUS
INTERNATIONAL FOOD STANDARDS

Food and Agriculture Organization of the United Nations
World Health Organization

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

RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

CODEX STAN 234-1999[1]

Adopted in 1999

[1] The most updated version of the method should be used, in application of ISO/IEC 17025. The present list of methods reflects the amendments adopted by the 39th Session of the Codex Alimentarius Commission in 2016.

Table of Contents

PART A – METHODS OF ANALYSIS BY COMMODITY CATEGORIES AND NAMES

Commodity	*Provision*	*Method*	*Principle*	*Type*
All Foods				
All foods	Acesulfame K, Aspartame	EN 12856	High performance liquid chromatography	II
All foods	Cyclamate	EN 12857	High performance liquid chromatography	II
All foods	Cyclamate	NMKL 123	Spectrophotometry	III
All foods	Saccharin	EN 12856	High performance liquid chromatography	III
All Foods (see also meat products)	Nitrates and/or Nitrites	EN 12014-1	Part 1- General considerations	N/A
Individual Foods[2]	Sulphites	EN 1988-1 AOAC 990.28	Part 1: Optimized Monier-Williams method	III
Individual Foods[3]	Sulphites	EN 1988-2 NMKL 135	Part 2: Enzymatic method	III
Cereals, Pulses and Legumes and Derived Products				
Certain pulses	Moisture	ISO 665	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Ash	AOAC 923.03 ISO 2171 ICC Method No 104/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Degermed maize (corn) meal and maize (corn) grits	Moisture	ISO 712 ICC Method No 110/1	Gravimetry	I
Degermed maize (corn) meal and maize (corn) grits	Particle size (granularity)	AOAC 965.22	Sieving	I

[2] Hominy, fruit juice, sea food
[3] Wine, dried apples, lemon juice, potato flakes, sultanas, beer

Cereals, Pulses and Legumes and Derived Products				
Degermed maize (corn) meal and maize (corn) grits	Protein	ICC Method No 105/1	Titrimetry, Kjeldahl digestion	I
Durum wheat semolina and durum wheat flour	Ash (semolina)	AOAC 923.03 ISO 2171	Gravimetry	I
Durum wheat semolina and durum wheat flour	Moisture	ISO 712 ICC 110/1	Gravimetry	I
Durum wheat semolina and durum wheat flour	Protein (N x 5.7)	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Instant Noodles	Extraction of oil from instant noodles	described in the standard	Gravimetry	I
Instant Noodles	Acid Value	described in the standard	Titrimetry	I
Instant Noodles	Moisture	described in the standard	Gravimetry	I
Maize (corn)	Moisture	ISO 6540	Gravimetry	I
Peanuts (raw)	Aflatoxins, total	AOAC 991.31	Immunoaffinity column (Aflatest)	II
Peanuts (raw)	Aflatoxins, total	AOAC 993.17	Thin layer chromatography	III
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 975.36	Romer minicolmn	III
Peanuts (Cereals, shell-fruits and derived products (including peanuts))	Sum of aflatoxins B_1, B_2, G_1 and G_2	EN 12955 ISO 16050	HPLC with post column derivatization and immunoaffinity column clean up	III
Peanuts (intended for further processing)	Aflatoxins, total	AOAC 979.18	Holaday-Velasco minicolumn	III
Pearl millet flour	Ash	AOAC 923.03	Gravimetry	I
Pearl millet flour	Colour	*Modern Cereal Chemistry*, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Pearl millet flour	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Pearl millet flour	Fibre, crude	ISO 5498: (B.5 Separation)	Gravimetry	I
Pearl millet flour	Moisture	ISO 712: ICC 110/1	Gravimetry	I

Cereals, Pulses and Legumes and Derived Products				
Pearl millet flour	Protein	AOAC 920.87	Titrimetry, Kjeldahl digestion	I
Sorghum flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum flour	Colour	*Modern Cereal Chemistry*, 6th Ed., D.W. Kent-Jones and A.J. Amos (Ed.), pp. 605-612, Food Trade Press Ltd, London, 1969.	Colorimetry using specific colour grader	IV
Sorghum flour	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Sorghum flour	Fibre, crude	ICC 113 ISO 6541	Gravimetry	I
Sorghum flour	Moisture	ISO 712 ICC 110/1	Gravimetry	I
Sorghum flour	Particle size (granularity)	AOAC 965.22	Sieving	I
Sorghum flour	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Sorghum flour	Tannins	ISO 9648	Spectrophotometry	I
Sorghum grains	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Sorghum grains	Fat, crude	AOAC 945.38F, 920.39C	Gravimetry (ether extraction)	I
Sorghum grains	Moisture	ISO 6540	Gravimetry	I
Sorghum grains	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Sorghum grains	Tannins	ISO 9648	Spectrophotometry	I
Soy protein products	Ash	AOAC 923.03 ISO 2171: (Method B)	Gravimetry	I
Soy protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Soy protein products	Fibre, crude	ISO 5498	Gravimetry	I
Soy protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven)	I

Cereals, Pulses and Legumes and Derived Products				
Soy protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry , Kjeldahl digestion	II
Vegetable protein products	Ash	AOAC 923.03 ISO 2171 (Method B)	Gravimetry, Direct	I
Vegetable protein products	Fat	CAC/RM 55 - Method 1	Gravimetry (extraction)	I
Vegetable protein products	Fibre, crude	AACC 32-17	Ceramic fiber filteration	I
Vegetable protein products	Moisture	AOAC 925.09	Gravimetry (vacuum oven)	I
Vegetable protein products	Protein	AOAC 955.04D (using factor 6.25)	Titrimetry, Kjeldahl digestion	II
Wheat flour	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Wheat flour	Fat acidity	AOAC 939.05	Titrimetry	I
Wheat flour	Moisture	ISO 712: ICC 110/1	Gravimetry	I
Wheat flour	Particle size (granularity)	AOAC 965.22	Sieving	I
Wheat flour	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Wheat protein products including wheat gluten	Protein	Vital wheat gluten and devitalized wheat gluten AOAC 979.09 (wheat protein in grain N x 5.7)	Kjeldahl	I
		Solubilized wheat protein AOAC 920.87 (wheat protein in flour N x 5.7)	Kjeldahl	I
Wheat protein products including Wheat gluten	Fibre, crude	AOAC 962.09	Ceramic fiber filteration	I
Wheat protein products including Wheat gluten	Ash	AOAC 923.03 ISO 2171: method B	Gravimetry	I

Cereals, Pulses and Legumes and Derived Products				
Whole and decorticated pearl millet grains	Ash	AOAC 923.03	Gravimetry	I
Whole and decorticated pearl millet grains	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Whole and decorticated pearl millet grains	Fibre, crude	ISO 5498 (B.5 Separation)	Gravimetry	I
Whole and decorticated pearl millet grains	Moisture	ISO 712 ICC 110/1	Gravimetry	I
Whole and decorticated pearl millet grains	Protein	AOAC 920.87	Titrimetry, Kjeldahl digestion	I
Whole maize (corn) meal	Ash	AOAC 923.03 ISO 2171 ICC 104/1	Gravimetry	I
Whole maize (corn) meal	Fat, crude	AOAC 945.38F; 920.39C	Gravimetry (ether extraction)	I
Whole maize (corn) meal	Moisture	ISO 712 ICC 110/1	Gravimetry	I
Whole maize (corn) meal	Particle size (granularity)	AOAC 965.22	Sieving	I
Whole maize (corn) meal	Protein	ICC 105/1	Titrimetry, Kjeldahl digestion	I
Cocoa Products and Chocolate				
Chocolate and chocolate products	Cocoa butter	AOAC 963.15 IOCCC 14	Gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Fat-free cocoa solids	AOAC 931.05	Oven evaporation and factor	I
Chocolate and chocolate products	Fat-free milk solids	IOCCC 17 or AOAC 939.02	Titrimetry, Kjeldahl digestion; after extraction of milk proteins	II
Chocolate and chocolate products	Fat, total	AOAC 963.15	Gravimetry (Soxhlet extraction)	I
Chocolate and chocolate products	Milkfat	IOCCC 5 AOAC 945.34; 925.41B; 920.80	Titrimetry/Distillation	I

Cocoa Products and Chocolate				
Chocolate and chocolate products	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method); or AOAC 931.04 or IOCCC 1	Gravimetry	I
Chocolate and chocolate products	Non-cocoa butter vegetable fat	AOCS Ce 10/02 and described in the Standard	Described in the Standard	I
Cocoa (Cacao) Mass or Cocoa/ Chocolate Liquor, and Cocoa Cake	Cocoa shell	AOAC 968.10 and 970.23	Spiral vessel count, Stone cell count	I
Cocoa (Cacao) Mass or Cocoa/ Chocolate Liquor, and Cocoa Cake	Fat	AOAC 963.15 or IOCCC 14	Gravimetry (Soxhlet extraction)	I
Cocoa butter	Free fatty acids	ISO 660 or AOCS Cd 3d-63	Titrimetry	I
Cocoa butter	Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	I
Cocoa powders (cocoa) and dry cocoa-sugar mixtures	Moisture	IOCCC 26 or AOAC 977.10 (Karl Fischer method)	Gravimetry	I
Fats and Oils and Related Products				
Fats and Oils (all)	Arsenic	AOAC 952.13 (Codex general method)	Colorimetry (diethyldithiocarbamate)	II
Fats and Oils (all)	Arsenic	AOAC 942.17 (Codex general method)	Colorimetry (molybdenum blue)	III
Fats and Oils (all)	Arsenic	AOAC 986.15 (Codex general method)	Atomic absorption spectrophotometry	III
Fats and oils	Butylhydroxyanisole, butylhydroxytoluene, tert-butylhydroquinone, & propyl gallate	AOAC 983.15; or AOCS Ce-6-86	Liquid chromatography	II
Fats and Oils (all)	Insoluble impurities	ISO 663	Gravimetry	I
Fats and Oils (all)	Lead	AOAC 994.02 ISO 12193 (Codex general method) or AOCS Ca 18c-91	Atomic absorption spectrophotometry (direct graphite furnace)	II
Fats and Oils (all)	Matter volatile at 105°C	ISO 662	Gravimetry (open-drying)	I

Fats and Oils and Related Products				
Fats and Oils (all)	Soap content	BS 684 Section 2.5; or AOCS Cc 17-95	Gravimetry	I
Fats and oils not covered by individual standards	Acid Value	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Fats and oils not covered by individual standards	Copper and Iron	AOAC 990.05 ISO 8294 or AOCS Ca 18b-91 (Codex general method)	Atomic absorption Spectrophotometry (direct graphite furnace)	II
Fats and oils not covered by individual standards	Peroxide value	AOCS Cd 8b-90 ISO 3960	Titrimetry using *iso*-octane	I
Fat spreads and blended spreads	Fat content	ISO 17189 | IDF 194	Gravimetry	I
Fish oils	Fatty acid composition	ISO 5508	Gas chromatography	III
Fish oils	Fatty acid composition	ISO 12966-2	Gas chromatography	III
Fish oils	Fatty acid composition	AOCS Ce 1b-89	GLC	III
Fish oils	Fatty acid composition	AOCS Ce 1-07	Capillary GLC	III
Fish oils	Fatty acid composition	AOCS Ce 2b-11	Alkali hydrolysis	III
Fish oils	Fatty acid composition	AOCS Ce 1a-13	Capillary GLC	III
Fish oils	Fatty acid composition	AOCS Ce 2-66	Preparation of methyl esters by fatty acids	III
Fish oils	Acid value	AOCS Ca 5a-40 AOCS CD 3D-63 ISO 3960 NMKL 38	Titration	I
Fish oils	Peroxide value	AOCS Cd 8b-90 ISO 3960 NMKL 158	Titration	I
Fish oils	Peroxide value	European Pharmacopeia 2.5.5 (Part B Iso-octane as solvent)	Titration	I
Fish oils	P-Anisidine value	Aocs Cd 18-90	Spectrometry	I

Fats and Oils and Related Products				
Fish oils	Vitamin A	European Parharmcopeia Monograph on Cod Liver Oil (Type A), monograph 01/2005:1192, with LC end-point 2.2.29	LC	III
Fish oils	Vitamin A	EN 12823-1 (Determination of vitamin A by high performance liquid chromatograph – Part 1: Measurement of all-E-retinol and 13-Z-retinol	LC	III
Fish oils	Vitamin D	EN 12821 (Determination of vitamin D by high performance liquid chromatography – Measurement of cholecalciferol (D3) or ergocalciferol (D2))	LC	III
Fish oils	Vitamin D	NMKL 167 (Cholecalciferol (vitamin D3) and Ergocalciferol (vitamin D2). Determination by HPLC in foodstuffs	LC	III
Named Animal Fats	Acidity	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Named Animal Fats	Copper and Iron	AOAC 990.05 ISO 8294; or AOCS Ca 18b-91 (Codex general method)	Atomic absorption Spectrophotometry (direct graphite furnace)	II
Named Animal Fats	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2 or AOCS Ce 2-66 and Ce 1e-91 or Ce 1f-96	Gas chromatography of methyl esters	II
Named Animal Fats	Iodine value (IV)	ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92	Wijs-Titrimetry	I
Named Animal Fats	Peroxide value	AOCS Cd 8b-90 ISO 3960	Titrimetry using *iso*-octane	I
Named Animal Fats	Relative density	ISO/AOCS method for apparent density to be inserted	Pycnometry	II
Named Animal Fats	Refractive index	ISO 6320; or AOCS Cc 7-25	Refractometry	II
Named Animal Fats	Saponification value	ISO 3657; or AOCS Cd 3-25	Titrimetry	I
Named Animal Fats	Unsaponifiable matter	ISO 3596 or ISO 18609; or AOCS Ca 6b-53	Titrimetry after extraction with diethyl ether	I
Named Animal Fats	Titre	ISO 935; or AOCS Cc 12-59	Thermometry	I

Fats and Oils and Related Products				
Named Vegetable Oils	Acidity	ISO 660; or AOCS Cd 3d-63	Titrimetry	I
Named Vegetable Oils	Apparent density	ISO 6883, with the appropriate conversion factor; or AOCS Cc 10c-95	Pycnometry	I
Named Vegetable Oils	Baudouin test (modified Villavecchia or sesameseed oil test)	AOCS Cb 2-40	Colour reaction	I
Named Vegetable Oils	Carotenoids, total	BS 684 Section 2.20	Spectrophotometry	II
Named Vegetable Oils	Copper and iron	ISO 8294; or AOAC 990.05; or AOCS Ca 18b-91	AAS	II
Named Vegetable Oils	Crismer value	AOCS Cb 4-35 and AOCS Ca 5a-40	Turbidity	I
Named Vegetable Oils	GLC ranges of fatty acid composition	ISO 5508 and ISO 12966-2; or AOCS Ce 2-66 and Ce 1--62 or Ce 1h-05	Gas chromatography of methyl esters	II
Named Vegetable Oils	Halphen test	AOCS Cb 1-25	Colorimetry	I
Named Vegetable Oils	Insoluble impurities	ISO 663	Gravimetry	I
Named Vegetable Oils	Iodine value (IV)	Wijs - ISO 3961; or AOAC 993.20; or AOCS Cd 1d-92; or NMKL 39	Wijs-Titrimetry [4]	I
Named Vegetable Oils	Lead	AOAC 994.02; or ISO 12193; or AOCS Ca 18c-91	Atomic Absorption	II
Named Vegetable Oils	Moisture & volatile matter at 105°C	ISO 662	Gravimetry	I
Named Vegetable Oils	Peroxide value (PV)	AOCS Cd 8b-90 or ISO 3960	Titrimetry	I
Named Vegetable Oils	Refractive index	ISO 6320 or AOCS Cc 7-25	Refractometry	II
Named Vegetable Oils	Reichert value and Polenske value	AOCS Cd 5-40	Titrimetry	I
Named Vegetable Oils	Relative density	IUPAC 2.101 with the appropriate conversion factor See comment above (Named Animal Fats) [5]	Pycnometry	I

[4] It is possible to calculate the Iodine Value from fatty acid composition data obtained by gas chromatography e.g. using AOCS Cd 1b-87
[5] The method is no longer available.

Fats and Oils and Related Products				
Named Vegetable Oils	Saponification value (SV)	ISO 3657 or AOCS Cd 3-25	Titrimetry	I
Named Vegetable Oils	Slip point	ISO 6321 for all oils; AOCS Cc 3b-92 for all oils except palm oils; AOCS Cc 3-25 for palm oils only	Open ended capillary tube	I
Named Vegetable Oils	Soap content	BS 684 Section 2.5; or AOCS Cc 17-95	Gravimetry	I
Named Vegetable Oils	Sterol content	ISO 12228; or AOCS Ch 6-91	Gas chromatography	II
Named Vegetable Oils	Tocopherol content	ISO 9936 or AOCS Ce 8-89	HPLC	II
Named Vegetable Oils	Unsaponifiable matter	ISO 3596; or ISO 18609; or AOCS Ca 6b-53	Gravimetry	I
Olive Oils and Olive Pomace Oils	Absorbency in ultra-violet	COI/T.20/Doc. No. 19 or ISO 3656 or AOCS Ch 5-91	Absorption in ultra violet	II
Olive Oils and Olive Pomace Oils	Acidity, free (acid value)	ISO 660 or AOCS Cd 3d-63	Titrimetry	I
Olive Oils and Olive Pomace Oils	Alpha-tocopherol	ISO 9936	HPLC	II
Olive Oils and Olive Pomace Oils	Difference between the actual and theoretical ECN 42 triglyceride content	COI/T.20/Doc. no. 20 or AOCS Ce 5b-89	Analysis of triglycerides of HPLC and calculation	I
Olive Oils and Olive Pomace Oils	Erythrodiol + uvaol	COI/T.20/Doc.no. 30	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Halogenated solvents, traces	COI/T.20/Doc. no. 8	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Insoluble impurities in light petroleum	ISO 663	Gravimetry	I
Olive Oils and Olive Pomace Oils	Iodine value	ISO 3961 or AOAC 993.20 or AOCS Cd 1d-92 or NMKL 39	Wijs-Titrimetry	I
Olive Oils and Olive Pomace Oils	Iron and copper	ISO 8294 or AOAC 990.05	AAS	II
Olive Oils and Olive Pomace Oils	Lead	AOAC 994.02 or ISO 12193 or AOCS Ca 18c-91	AAS	II
Olive Oils and Olive Pomace Oils	Moisture and volatile matter	ISO 662	Gravimetry	I
Olive Oils and Olive Pomace Oils	Organoleptic characteristics	COI/T.20/Doc. no. 15	Panel test	I
Olive Oils and Olive Pomace Oils	Peroxide value	ISO 3960 or AOCS Cd 8b-90	Titrimetry	I
Olive Oils and Olive Pomace Oils	Relative density	IUPAC 2.101, with the appropriate conversion factor. See comment above	Pycnometry	I

Fats and Oils and Related Products				
Olive Oils and Olive Pomace Oils	Refractive index	ISO 6320 or AOCS Cc 7-25	Refractometry	II
Olive Oils and Olive Pomace Oils	Saponification value	ISO 3657 or AOCS Cd 3-25	Titrimetry	I
Olive Oils and Olive Pomace Oils	Sterol composition and total sterols	COI/T.20/Doc. no. 30 | ISO 12228-2 or AOCS Ch 6-91	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Stigmastadienes	COI/T.20/Doc. no. 11 or ISO 15788-1 or AOCS Cd 26-96	Gas chromatography	II
Olive Oils and Olive Pomace Oils	Stigmastadienes	ISO 15788-2	HPLC	III
Olive Oils and Olive Pomace Oils	*Trans* fatty acids content	COI/T.20/Doc no. 17 or ISO 15304 or AOCS Ch 2a-94	Gas chromatography of methyl esters	II
Olive Oils and Olive Pomace Oils	Unsaponifiable matter	ISO 3596 or ISO 18609 or AOCS Ca 6b-53	Gravimetry	I
Olive Oils and Olive Pomace Oils	Wax content	COI/T.20/Doc. no. 18 or AOCS Ch 8-02	Gas chromatography	II
Fish and Fishery Products				
Fish and fishery products	Histamine	AOAC 977.13	Fluorimetry	II
Fish and fishery products	Mercury	AOAC 977.15	Flameless atomic absorption spectrophotometry	III
Fish and fishery products: canned products	Drained weight	Described in the Standard	Weighing	I
Fish and fishery products: canned products	Net weight	Described in the Standard	Weighing	I
Boiled Dried Salted Anchovies	Sodium Chloride (chloride expressed as sodium chloride)	AOAC 937.09	Titrimetry	II
Canned shrimps or prawns	Size, determination of	Described in the Standard	Number per 100 g	I
Fish Sauce	total nitrogen	AOAC 940.25	digestion	I
Fish Sauce	amino acid nitrogen	AOAC 920.04 and AOAC 920.03	determining formaldehyde titration method subtracting by ammoniacal nitrogen (magnesium oxide method)	I

Fish and Fishery Products				
Fish Sauce	pH	AOAC 981.12 The pH shall be measured in a sample of fish sauce diluted with water to 1:10 using a pH meter. The dilution of fish sauce is necessary because of the high ionic strength in the undiluted sauce.	electrometry	III
Fish Sauce	sodium chloride	AOAC 976.18	potentiometry	II
Fish Sauce	sodium chloride	AOAC 937.09	titrimetry	IV
Fish Sauce	histamine	AOAC 977.13	Fluorimetry	II
Frozen abalone (covered by glaze)	Net weight	AOAC 963.18	Gravimetry	I
Frozen fish and fishery products	Thawing and cooking procedures	Described in the Standards	Thawing and heating	I
Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Proportion of fish fillet and minced fish	AOAC 988.09	Physical separation	I
Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Net content of frozen fish blocks covered by glaze	Described in the Standard	Gravimetry	I
Quick frozen blocks of fish fillet, minced fish flesh and mixtures of fillets and minced fish flesh	Sodium chloride	AOAC 971.21 (Codex general method)	Potentiometry	II
Quick frozen fish fillets	Net weight of products covered by glaze	Described in the Standard	Water spraying and sieving	I
Quick Frozen Fish sticks (fish fingers) and fish portions - breaded or in batter	Fish content (declaration)	AOAC 996.15 and calculation (described in the standard)	Gravimetry	I
Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Net weight	Described in the Standard	Weighing	I
Quick Frozen Fish Sticks (fish fingers) and Fish Portions-Breaded and in Batter (except for certain fish species with soft flesh)	Proportion of fish fillet and minced fish	WEFTA Method (described in the Stnadard)	Gravimetry	I

Fish and Fishery Products				
Quick frozen fish sticks (fish fingers) and fish portions - breaded or in batter	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Salted Atlantic Herring and Salted Sprat	Water content	AOAC 950.46B	air drying	I
Salted Fish of the *Gadidae* Family	Salt	Described in CODEX STAN 167-1989	Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride	I
Salted Fish and Dried Salted Fish of the *Gadidae* Family of Fishes	Salt Content Water content	Sampling and method described in the Standard	Gravimetry	I
Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish	Water phase salt	AOAC 952.08 AOAC 937.09 Described in standard[6]	Calculation	I
Smoked Fish, Smoke-Flavoured fish and Smoke-dried fish	Water activity	NMKL 168 | ISO 21807	Electrometry	III
Sturgeon Caviar	Salt content	Described in CODEX STAN 167-1989	Titrimetry (Mohr) Salt determined as chloride expressed as sodium chloride	I
Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 959.08	Mouse bioassay	IV
Live and raw bivalve molluscs	Paralytic shellfish toxicity	AOAC 2011.27	Receptor binding assay	IV

Method Performance Criteria for histamine for fish and fishery products

Provision	ML (mg/100 g)	Minimum applicable range (mg/100 g)	LOD (mg/100 g)	LOQ (mg/100 g)	RSD_R(%)	Recovery	Applicable methods that meet the criteria	Principle
Histamine	10 (average)	8 – 12	1	2	16.0	90 – 107	AOAC 977.13 | NMKL 99, NMKL 196,	Fluorometric HPLC
Histamine	20 (each unit)	16 – 24	2	4	14.4	90 – 107	AOAC 977.13 | NMKL 99, NMKL 196,	Fluorometric HPLC

Determination of Biotoxins in live and raw bivalve molluscs

The method selected should be chosen on the basis of practicability and preference should be given to methods which have applicability for routine use.

[6] % salt × 100 / (%water + %salt)

Criteria for determination of Toxin Analogues by chemical methods

Methods shall meet the numerical criteria listed in Table 1 and may either meet the minimum applicable range, or LOD and LOQ criteria listed.

Table 1. Criteria for determination of Toxin Analogues by Chemical Methods

Toxin Group	Toxin	Minimum applicable range (mg/kg)	LOD (mg/kg)	LOQ (mg/kg)	Precision (RSD_R) (%) No more than	Recovery percent	Applicable methods that meet the criteria
STX Group	Saxitoxin (STX)	0.05 – 0.2	0.01	0.02	44%	50 – 130	AOAC 2005.06 NMKL 182, EN 14526 AOAC 2011.02 NMKL 197
	NEO	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	dcSTX	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	GTX1	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	GTX2	0.1 – 0.5	0.03	0.06	38%	50– 130	
	GTX3	0.1 – 0.5	0.03	0.06	38%	50– 130	
	GTX4	0.05 – 0.2	0.01	0.02	44%	50 – 130	
	GTX5	0.1 – 0.5	0.03	0.06	38%	50– 130	
	GTX6	0.1 – 0.5	0.03	0.06	38%	50– 130	
	dcGTX2	0.1 – 0.5	0.03	0.06	38%	50– 130	
	dcGTX3	0.1 – 0.5	0.03	0.06	38%	50– 130	
	C1	0.1 – 0.5	0.03	0.06	38%	50– 130	
	C2	0.1 – 0.5	0.03	0.06	38%	50– 130	
	C3	0.5 – 1.5	0.1	0.2	32%	50– 130	
	C4	0.5 – 1.5	0.1	0.2	32%	50– 130	
OA Group	OA	0.03 – 0.2	0.01	0.02	44%	60-115	See reference below
	DTX1	0.03 – 0.2	0.01	0.02	44%	60-115	
	DTX2	0.1 – 0.5	0.03	0.06	38%	60-115	
Domoic Acid	DA	14 – 26	2	4	20%	80-110	
AZA Group	AZA1	0.03 – 0.2	0.01	0.02	44%	40 - 120	See reference below
	AZA2	0.03 – 0.2	0.01	0.02	44%	40 - 120	
	AZA3	0.03 – 0.2	0.01	0.02	44%	40 - 120	

Reference: http://aesan.msssi.gob.es/en/CRLMB/web/procedimientos_crlmb/crlmb_standard_operating_procedures.shtml Harmonised-SOP-LCMS-OA-Version4.pdf

Total toxicity is estimated as the sum of the molar concentrations of detected analogs multiplied by the relevant specific toxicity equivalency factors (TEFs). Internationally scientifically validated TEFs must be used. The science behind TEFs is developing. Current internationally validated TEF's will be found on the FAO website. Information on TEFs could be incorporated in this standard at a future date.

Methods should be validated and used for the relevant toxin analogues that may contribute to total toxicity. Currently known toxin analogues to consider are listed in Table 1.

Where toxin analogues that are not listed in Table 1 are determined the competent authority must assess the contribution of these analogs to total toxicity whilst conducting further investigations.

Foods for Special Dietary Uses				
Special foods	Ash	AOAC 942.05	Gravimetry	I
Special foods	Calcium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Calories by calculation	Method described in CAC/VOL IX-Ed.1, Part III	Calculation method	III
Special foods	Carbohydrates	Method described in CAC/VOL IX-Ed.1, Part III	Calculation	III
Special foods	Chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Special foods	Dietary fibre, total	AOAC 985.29	Gravimetry (enzymatic digestion)	I
Special foods	Fat	CAC/RM 55	Gravimetry (extraction)	I
Special foods	Fat in foods not containing starch, meat or vegetable products	CAC/RM 1, B-2	Gravimetry	I
Special foods	Fill of containers	CAC/RM 46	Weighing	I
Special foods	Folic acid	AOAC 944.12	Microbioassay	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 969.33; 963.22	Acid hydrolysis, preparation of methyl esters and gas chromatography	II
Special foods	Linoleate (in the form of glycerides)	AOAC 922.06; 979.19	Acid hydrolysis and spectrophotometry	III
Special foods	Loss on drying (milk based)	AOAC 925.23 -ISO 6731|IDF 21	Gravimetry	I
Special foods	Nicotinamide for foods not based on milk	AOAC 961.14	Colorimetry	II
Special foods	Nicotinamide for milk-based foods	AOAC 944.13	Microbioassay	II
Special foods	Pantothenic acid/enriched foods	AOAC 945.74	Microbioassay	II
Special foods	Pantothenic acid/non-enriched foods	*The Analyst* 89 (1964):1, 3-6, ibid. 232 US Dept Agr., *Agr. Handbook* 97 (1965)	Microbioassay	IV
Special foods	Phosphorous	AOAC 986.24	Colorimetry (molybdovanadate)	II
Special foods	Protein efficiency ratio (PER)	AOAC 960.48	Rat bioassay	I

Foods for Special Dietary Uses				
Special foods	Protein, crude	Method described in CAC/VOL IX-Ed. 1,Part III	Titrimetry, Kjeldahl digestion	I
Special foods	Riboflavin	AOAC 970.65	Fluorometry	II
Special foods	Sodium and Potassium	ISO 8070|IDF 119	Flame atomic absorption spectrometry	II
Special foods	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Special foods	Vitamin A	AOAC 974.29	Colorimetry	IV
Special foods	Vitamin A in foods in which carotenes have been added as a source of vitamin A	AOAC 941.15	Spectrophotometry	III
Special foods	Vitamin B_{12}	AOAC 952.20	Microbioassay	II
Special foods	Vitamin B_6	AOAC 961.15	Microbioassay	II
Special foods	Vitamin C	AOAC 967.22	Microfluorometry	II
Special foods	Vitamin C	AOAC 967.21	Colorimetry (dichloroindophenol)	III
Special foods	Vitamin D	AOAC 936.14	Rat bioassay	IV
Special foods	Vitamin D (D_3, milk based infant formula)	AOAC 992.26	Liquid chromatography	II
Special foods	Vitamin E	AOAC 971.30	Colorimetry	IV
Special foods	Vitamin E (milk based infant formula)	AOAC 992.03	Liquid chromatography	II
Special foods	Sodium and Potassium	ISO 8070|IDF 119	Flame atomic absorption spectrometry	II
Follow-up formula	Dietary fibre, total	AOAC 991.43	Gravimetry (enzymatic digestion)	I
Follow-up formula	Iodine (milk based formula)	AOAC 992.24	Ion-selective potentiometry	II
Follow-up formula	Pantothenic acid	AOAC 992.07 Measures total pantothenate (free pantothenic acid + CoA- + ACP-bound) and measured as D-pantothenic acid (or calcium D-pantothenate)	Microbioassay	II
Follow-up formula	Vitamin A	AOAC 974.29	Colorimetry	IV
Follow-up formula	Vitamin A (retinol isomers)	AOAC 992.04	HPLC	II

Foods for Special Dietary Uses				
Follow-up formula	Vitamin A (retinol) (above 500 IU/l milk after reconstitution)	AOAC 992.06	HPLC	III
Follow-up formula	Vitamin K	AOAC 999.15 EN 14148 (vitamin K_1) (Measures either aggregated cis + trans K_1 or can measure individual cis and trans forms depending on LC column.)	HPLC with C30 column to separate the cis- and the trans- K vitamins	II
Foods with low-sodium content (including salt substitutes)	Iodine	AOAC 925.56	Titrimetry	II
Foods with low-sodium content (including salt substitutes)	Silica (colloidal, calcium silicate)	AOAC 950.85N	Gravimetry	IV
Gluten-free foods	Gluten	Enzyme-Linked Immunoassay R5 Mendez (ELISA) Method *Eur J Gastroenterol Hepatol* 2003; 15: 465-474	Immunoassay	I
Infant formula	Biotin	EN 15607 (d-biotin) (Measures total D-biotin (free + D-biocytin)	HPLC	II
Infant formula	Calories (by calculation)	Method described in CAC/Vol IX-Ed.1, Part III[7]	Calculation	I
Infant formula	Calcium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	II

[7] Section 9. Calories by calculation – Section 9.2 Conversion Factors

(a) protein 4 kcal per g

(b) carbohydrate 4 kcal per g

(c) fat 9 kcal per g

(d) monosaccharides 3.75 kcal per g

(e) specific food ingredients See “Energy and Protein Requirements”(FAO Nutrition Meeting Report Series No. 52 or WHO Technical Report Series No. 522)

(f) other specific calorie conversion factors maybe used where the formulation of the food and the nutrient content are known and where such specific conversion factors are physiologically more meaningful than the factors listed above

Foods for Special Dietary Uses				
Infant formula	Calcium	AOAC 985.35	Flame atomic absorption spectroscopy	III
Infant formula	Calcium	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Chloride	AOAC 986.26	Potentiometry	III
Infant formula	Choline	AOAC 999.14	Enzymatic Colorimetric Method with limitations on applicability due to choline and ascorbate concentration.	II
Infant formula	Copper	AOAC 985.35	Flame atomic absorption spectroscopy	II
Infant formula	Copper	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	EN 14082	Graphite furnace atomic absorption after dry ashing	II
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	III
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	III
Infant formula	Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 |IDF 235	ICP-MS	III
Infant formula	Crude protein[8]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Infant formula	Fatty acids (including trans fatty acid)	AOAC 996.06	Gas chromatography	III
Infant formula	Fatty acids (including trans fatty acid)	AOCS Ce 1i-07	Gas chromatography	III
				II

[8] **Determination of Crude Protein**

The calculation of the protein content of infant formulas prepared ready for consumption may be based on N x 6.25, unless a scientific justification is provided for the use of a different conversion factor for a particular product. The value of 6.38 is generally established as a specific factor appropriate for conversion of nitrogen to protein in other milk products, and the value of 5.71 as a specific factor for conversion of nitrogen to protein in other soy products

Foods for Special Dietary Uses				
Infant formula	Folic acid	AOAC 992.05 (Measures free folic acid + free, unbound natural folates, aggregated and measured as folic acid) EN 14131 (Total folate (free + bound), aggregated and measured as folic acid)	Microbioassay	II
Infant formula	Folic acid	J AOAC Int. 2000:83; 1141-1148 (Measures free folic acid + proportion of free, natural folate)	Optical Biosensor Immunoassay	IV
Infant formula	Folic acid	J Chromatogr. A., 928, 77-90, 2001 (Measures total folates after conversion to, and measurement as 5-Me-H4PteGlu)	HPLC, incorporating immunoaffinity clean-up and conversion to 5-methyltetrahydrofolate	IV
Infant formula	Iodine (for milk-based formula)	AOAC 2012.15 | ISO 20647 | IDF 234	ICP-MS	II
Infant formula	Iron[9]	AOAC 985.35	Flame atomic absorption spectrophotometry	III
Infant formula	Iron	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Iron	AOAC 999.11 | NMKL139	AAS after dry ashing	II
Infant formula	Magnesium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	II
Infant formula	Magnesium	AOAC 985.35	Flame atomic absorption spectroscopy	III
Infant formula	Magnesium	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Manganese	AOAC 985.35	Flame atomic absorption spectrophotometry	II
Infant formula	Manganese	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Melamine	ISO/TS 15495 | IDF/RM 230	LC-MS/MS	IV

[9] General Codex methods are also available

Foods for Special Dietary Uses				
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	EN 14083	Graphite furnace AAS after pressure digestion	II
Infant formula	Molybdenum (Section B of CODEX STAN 72-1981 only)	AOAC 2006.03	ICP emission spectroscopy	III
	Molybdenum (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	III
				II
Infant formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay and turbidimetry	III
Infant formula	Niacin	EN 15652 (Free and bound and phosphorylated forms measured either as aggregate of nicotinic acid + nicotinamide, or as individual forms)	HPLC	II[10]
Infant formula	Pantothenic acid	AOAC 2012.16 ISO 20639	UHPLC-MS/MS	II
Infant formula	Phosphorus	AOAC 986.24	Spectrophotometry (molybdovanadate)	II
Infant formula	Phosphorus	AOAC 984.27	ICP emission spectroscopy	III
Infant formula	Riboflavin	AOAC 985.31[11]	Fluorimetry	III
Infant formula	Riboflavin	EN 14152 (Measures natural and supplemental forms, free, bound and phosphorylated (FMN and FAD) aggregated and measured as riboflavin.)	HPLC	II
Infant formula	Selenium	AOAC 996.16 or AOAC 996.17	Continuous hydride generation Flame atomic absorption spectrometry (HGAAS)	III
Infant formula	Selenium	EN 14627	Hydride generation atomic absorption spectrometry (HGAAS)	II

[10] When published as EN method
[11] Care should be taken in the application of the method due to spectral interference

Foods for Special Dietary Uses				
Infant formula	Selenium	AOAC 2006.03	ICP emission spectroscopy	III
	Selenium	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	III
Infant formula	Sodium and potassium	AOAC 984.27	ICP emission spectrometry	III
Infant formula	Sodium and potassium	ISO 8070 | IDF 119	Flame atomic absorption spectrophotometry	II
Infant formula	Thiamine	AOAC 986.27[12]	Fluorimetry	III
Infant formula	Thiamine	EN 14122 (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
Infant formula	Total carbohydrates	AOAC 986.25 AOAC 990.19 or	Determination by difference	I
	Moisture/Total Solids	AOAC 990.20 ISO 6731|IDF 21	Gravimetry	
	Ash	AOAC 942.05	Gravimetry	
Infant formula	Total fat	AOAC 989.05 ISO 8381|IDF 123	Gravimetry (Röse-Gottlieb)	I
Infant formula	Total fat for milk-based infant formula (Products not completely soluble in ammonia)	ISO 8262-1 |IDF 124-1	Gravimetry (Weibull-Berntrop)	I
Infant formula	Total nucleotides	AOAC 2011.20 ISO 20638	LC	II
Infant formula	Total phospholipids	AOCS Ja7b-91	Gas chromatography with suitable extraction and preparation procedures	III

[12] Care should be taken in the application of the method due to spectral interference

Foods for Special Dietary Uses				
Infant formula	Vitamin A	EN 12823-1 (all-trans-retinol and 13-cis-retinol) Vitamin A (both natural + supplemental ester forms) aggregated and quantified as individual retinol isomers (13 - cis and all-trans)	HPLC	III
Infant formula	Vitamin A Palmitate (Retinyl Palmitate), Vitamin A Acetate (Retinyl Acetate)	AOAC 2012.10 ISO 20633	HPLC	II
Infant formula	Vitamin D	AOAC 992.26 D_3 measured	HPLC	III
Infant formula	Vitamin D	EN 12821 (D2 and/or D3 measured as single components. Hydroxylated forms not measured.) NMKL 167	HPLC	II
Infant formula	Vitamin D	AOAC 995.05 D2 and D3 measured	HPLC	III
Infant formula	Vitamin E	AOAC 992.03 Measures all rac-vitamin E (both natural + supplemental ester forms) aggregated and quantified as α-congeners	HPLC	III
Infant formula	Vitamin E	EN 12822 (Measures Vitamin E (both natural + supplemental ester forms) aggregated and quantified as individual tocopherol congeners (α, β, γ, δ).	HPLC	II
Infant formula	Vitamin B_6	AOAC 985.32	Microbioassay	III
Infant formula	Vitamin B_6	EN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxamine and measures as pyridoxine)	Microbioassay	III

Foods for Special Dietary Uses				
Infant formula	Vitamin B_6	AOAC 2004.07 EN 14164 (Free and bound phosphorylated forms (pyridoxal, pyridoxine and pyridoxamine) converted and measured as pyridoxine)	HPLC	II
Infant formula	Vitamin B_6	EN 14663 (includes glycosylated forms) (Free and bound phosphorylated and glycosylated forms measured as the individual forms pyridoxal, pyridoxine and pyridoxamine)	HPLC	III
Infant formula	Vitamin B_{12}	AOAC 986.23 (Measures total vitamin B_{12} as cyanocobalamin)	Turbidimetric Method	II
Infant formula	Zinc	AOAC 985.35	Flame atomic absorption spectroscopy	II
Infant formula	Zinc	AOAC 984.27	ICP emission spectroscopy	III

Methods of analysis for dietary fibre: Guidelines for Use of Nutrition and Health Claims: Table of Conditions for Claims

Standard	Provisions	Method	Principle	Type
General methods that do not measure the lower molecular weight fraction (i.e. monomeric units<=9)[(2)]				
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. (4)	AOAC 985.29 AACC Intl 32-05.01	Enzymatic gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction and also includes determination for soluble and insoluble dietary fibres (4)	AOAC 991.43 AACC Intl 32-07.01 NMKL 129	Enzymatic gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction, in foods and food products containing more than 10% dietary fibres and less than 2% starch (e.g. fruits) (4)	AOAC 993.21	gravimetry	Type I
All foods (1)	Method applicable for determining dietary fibres that do not include the lower molecular weight fraction. Provides sugar residue composition of dietary fibre polysaccharides, as well as content of Klason lignin (4).	AOAC 994.13 AACC Intl 32- 25.01 NMKL 162	Enzymatic GC/ colorimetry gravimetry	Type I
All foods (1)	Insoluble dietary fibres in food and food products (4)	AOAC 991.42 (Specific for insoluble fibre) AACC Intl 32-20.01	Enzymatic gravimetry	Type I
All foods (1)	Soluble dietary fibres in food and food products (4)	AOAC 993.19 (Specific for soluble fibre)	Enzymatic gravimetry	Type I
General methods that measure both the higher (monomeric units > 9) and the lower molecular weight fraction (monomeric units <=9) [(2)]				
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight, in food where resistant starches are not present	AOAC 2001.03 AACC Intl 32-41.01	Enzymatic gravimetry and Liquid chromatography	Type I
All foods (1)	Method applicable for determining the content of dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches.	AOAC 2009.01 AACC Intl 32-45.01	Enzymatic-Gravimetry High Pressure Liquid Chromatography	Type I
All foods (1)	Method applicable for determining the content of insoluble and soluble dietary fibres of higher and lower molecular weight. The method is applicable in food that may, or may not, contain resistant starches	AACC Intl 32-50.01 AOAC 2011.25	Enzymatic-Gravimetry High Pressure Liquid Chromatography	Type I

Standard	Provisions	Method	Principle	Type
Methods that measure individual specific components (monomeric units: the whole range for each type of components is covered)[2]				
All foods (1)	(1→3)(1→4) *Beta*-D-Glucans	AOAC 995.16 AACC Intl 32-23.01	Enzymatic	Type II
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (applicable to added fructans)	AOAC 997.08 AACC Intl 32-31.01	Enzymatic & HPAEC-PAD	Type II
All foods (1)	Fructans (oligofructoses, inulin, hydrolyzed inulin, polyfructoses, fructooligosaccharides) (not applicable highly depolymerised fructans)	AOAC 999.03 AACC Intl 32-32.01	Enzymatic & colorimetric	Type III
All foods (1)	Polydextrose	AOAC 2000.11 AACC Intl 32-28.01	HPAEC-PAD	Type II
All foods (1)	Trans-galacto-oligo saccharides	AOAC 2001.02 AACC Intl 32-33.01	HPAEC-PAD	Type II
All foods (1)	Resistant starch (Recommended for RS3)	AOAC 2002.02 AACC Intl 32-40.01	Enzymatic	Type II

Other methods[2] that have not been subjected to interlaboratory evaluation under AOAC international guidelines				
Yeast cell wall	Insoluble glucans and mannans of yeast cell wall (for yeast cell wall only)	Eurasyp (European association for specialty yeast product) – LM Bonanno. Biospringer- 2004 – online version : http://www.eurasyp.org/public.technique.home.screen.	Chemical & HPAEC-PAD	Type IV
All foods	Fructo-oligosaccharides (monomeric units<5)	Ouarné et al. 1999 in *Complex Carbohydrates in Foods*. Edited by S. Sungsoo, L. Prosky & M. Dreher. Marcel Dekker Inc, New York	HPAEC-PAD	Type IV
All foods	Non-starch polysaccharides (NSP) (3)	Englyst H.N, Quigley M.E., Hudson G. (1994) Determination of dietary fibre as non-starch polysaccharides with gas-liquid chromatographic high performance liquid chromatographic or spectrophotometric measurement of constituent sugars – Analyst 119, 1497-1509	Gas-Liquid Chromatography	Type IV

(1) Users should consult the description of each method for the food matrices that were the subject of interlaboratory study in the Official methods of Analysis of AOAC International.

(2) Two issues are left for national authorities: to include monomeric units 3-9 and which isolated or synthetic compounds have physiological benefit. (Refer to the Guidelines for Nutrition Labelling (CAC/GL 2-1985).

(3) Quantitation lost for resistant starch. Refer to specific methods.

(4) Quantitation lost for inulin, resistant starch, polydextrose and resistant maltodextrins. Refer to specific methods.

Fruit Juices and Nectars

Commodity	Provisions	Method	Principle	Type
Fruit Juices and Nectars	Ascorbic acid-L (additives)	IFUMA 17A	HPLC	II
Fruit Juices and Nectars	Ascorbic acid-L (additives)	ISO 6557-1	Fluorescence spectrometry	IV
Fruit Juices and Nectars	Ascorbic acid-L (additives)	AOAC 967.21 IFUMA 17 ISO 6557-2	Indophenol method	III
Fruit Juices and Nectars	Carbon dioxide (additives and processing aids)	IFUMA 42	Titrimetry (back-titration after precipitation)	IV
Fruit Juices and Nectars	Cellobiose	IFUMA 4	Capillary gas chromatography	IV
Fruit Juices and Nectars	Citric acid [13] (additives)	AOAC 986.13	HPLC	II
Fruit Juices and Nectars	Citric acid [5] (additives)	EN 1137 IFUMA 22	Enzymatic determination	III
Fruit Juices and Nectars	Glucose and fructose (permitted ingredients)	EN 12630 IFUMA 67 NMKL 148	HPLC	III
Fruit Juices and Nectars	Glucose-D and fructose-D (permitted ingredients)	EN 1140 IFUMA 55	Enzymatic determination	II
Fruit Juices and Nectars	HFCS & HIS in apple juice (permitted ingredients)	Determination of HFCS & HIS by Capillary GC method JAOAC 84, 486 (2001)	CAP GC Method	IV
Fruit Juices and Nectars	Malic acid (additives)	AOAC 993.05	Enzymatic determination and HPLC	III
Fruit Juices and Nectars	Malic acid-D	EN 12138 IFUMA 64	Enzymatic determination	II
Fruit Juices and Nectars	Malic acid-D in apple juice	AOAC 995.06	HPLC	II
Fruit Juices and Nectars	Malic acid-L	EN 1138 IFUMA 21	Enzymatic determination	II
Fruit Juices and Nectars	Pectin (additives)	IFUMA 26	Precipitation/photometry	I
Fruit Juices and Nectars	Benzoic acid and its salts; sorbic acid and its salts	IFUMA 63 NMKL 124	HPLC	II
Fruit Juices and Nectars	Benzoic acid and its salts	ISO 5518, ISO 6560	Spectrometry	III
Fruit Juices and Nectars	Preservatives in fruit juices (sorbic acid and its salts)	ISO 5519	Spectrometry	III

[13] All juices except citrus based juices

Fruit Juices and Nectars				
Fruit Juices and Nectars	Quinic, malic & citric acid in cranberry juice cocktail and apple juice (permitted ingredients and additives)	Determination of quinic, malic and citric acid in cranberry juice cocktail and apple juice AOAC 986.13	HPLC	III
Fruit Juices and Nectars	Saccharin	NMKL 122	Liquid chromatography	II
Fruit Juices and Nectars	Soluble solids	AOAC 983.17 EN 12143 IFUMA 8 ISO 2173	Indirect by refractometry	I
Fruit Juices and Nectars	Sucrose (permitted ingredients)	EN 12146 IFUMA 56	Enzymatic determination	III
Fruit Juices and Nectars	Sucrose (permitted ingredients)	EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit Juices and Nectars	Sulphur dioxide (additives)	Optimized Monier Williams AOAC 990.28 IFUMA 7A NMKL 132	Titrimetry after distillation	II
Fruit Juices and Nectars	Sulphur dioxide (additives)	NMKL 135	Enzymatic determination	III
Fruit Juices and Nectars	Sulphur dioxide (additives)	ISO 5522, ISO 5523	Titrimetry after distillation	III
Fruit Juices and Nectars	Tartaric acid in grape juice (additives)	EN 12137 IFUMA 65	HPLC	II
Fruit Juices and Nectars	Total nitrogen	EN 12135 IFUMA 28	Digestion/titration	I
Fruit Juices and Nectars	Sections 3.2 Quality Criteria and 3.3 Authenticity[14]	Determination of acetic acid EN 12632; IFUMA 66	Enzymatic determination	II
Fruit Juices and Nectars		Determination of alcohol (ethanol) IFUMA 52	Enzymatic determination	II
Fruit Juices and Nectars		Detection of anthocyanins IFUMA 71	HPLC	I
Fruit Juices and Nectars		Determination of ash in fruit products AOAC 940.26; EN 1135; IFUMA 9	Gravimetry	I
Fruit Juices and Nectars		Detection of beet sugar in fruit juices AOAC 995.17	Deuterium NMR	II

[14] **3.4 Verification of Composition, Quality and Authenticity**

Fruit juices and nectars should be subject to testing for authenticity, composition, and quality where applicable and where required. The analytical methods used should be those found in Section 9, Methods of Analysis and Sampling.

The verification of a sample's authenticity / quality can be assessed by comparison of data for the sample, generated using appropriate methods included in the standard, with that produced for fruit of the same type and from the same region, allowing for natural variations, seasonal changes and for variations occurring due to processing.

Fruit Juices and Nectars			
Fruit Juices and Nectars	Determination of benzoic acid as a marker in orange juice AOAC 994.11	HPLC	III
Fruit Juices and Nectars	Determination of C^{13}/C^{12} ratio of ethanol derived from fruit juices JAOAC 79, No. 1, 1996, 62-72	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carbon stable isotope ratio of apple juice AOAC 981.09 - JAOAC 64, 85 (1981)	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carbon stable isotope ratio of orange juice AOAC 982.21	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of carotenoid, total/individual groups EN 12136; IFUMA 59	Spectrophotometry	I
Fruit Juices and Nectars	Determination of centrifugable pulp EN 12134; IFUMA 60	Centrifugation/% value	I
Fruit Juices and Nectars	Determination of chloride (expressed as sodium chloride) EN 12133 IFUMA 37	Electrochemical titrimetry	III
Fruit Juices and Nectars	Determination of chloride in vegetable juice AOAC 971.27 (Codex general method) ISO 3634	Titration	II
Fruit Juices and Nectars	Determination of essential oils (Scott titration AOAC 968.20 - IFUMA 45*	(Scott) distillation, titration	I
Fruit Juices and Nectars	Determination of essential oils (in citrus fruit) (volume determination)* ISO 1955	Distillation and direct reading of the volume determination	I
Fruit Juices and Nectars	Determination of fermentability IFUMA 18	Microbiological method	I
Fruit Juices and Nectars	Determination of formol number EN 1133 IFUMA 30	Potentiometric titration	I
Fruit Juices and Nectars	Determination of free amino acids EN 12742 IFUMA 57	Liquid Chromatography	II
Fruit Juices and Nectars	Determination of fumaric acid IFUMA 72	HPLC	II

Fruit Juices and Nectars			
Fruit Juices and Nectars	Determination of glucose fructose and saccharose EN 12630 IFUMA 67 NMKL 148	HPLC	II
Fruit Juices and Nectars	Determination of gluconic acid IFUMA 76	Enzymatic determination	II
Fruit Juices and Nectars	Determination of glycerol IFUMA 77	Enzymatic determination	II
Fruit Juices and Nectars	Determination of hesperidin and naringin EN 12148 IFUMA 58	HPLC	II
Fruit Juices and Nectars	Determination of hydroxymethylfurfural IFUMA 69	HPLC	II
Fruit Juices and Nectars	Determination of hydroxymethylfurfural ISO 7466	Spectrometry	III
Fruit Juices and Nectars	Determination of isocitric acid-D IFUMA 54	Enzymatic determination	II
Fruit Juices and Nectars	Determination of Lactic acid- D and L EN 12631 IFUMA 53	Enzymatic determination	II
Fruit Juices and Nectars	Determination of L-malic/total malic acid ratio in apple juice AOAC 993.05	Enzymatic determination and HPLC	II
Fruit Juices and Nectars	Determination of naringin and neohesperidin in orange juice AOAC 999.05	HPLC	III
Fruit Juices and Nectars	Determination of pH-value NMKL 179 EN 1132 IFUMA 11 ISO 1842	Potentiometry	II IV
Fruit Juices and Nectars	Determination of phosphorus/phosphate EN 1136 IFUMA No 50	Photometric determination	II
Fruit Juices and Nectars	Determination of proline by photometry – non-specific determination EN 1141 IFUMA 49	Photometry	I
Fruit Juices and Nectars	Determination of relative density EN 1131 (1993); IFUMA 01 & IFU Method No General sheet (1971)	Pycnometry	II
Fruit Juices and Nectars	Determination of Relative density IFUMA 01A	Densitometry	III

Fruit Juices and Nectars			
Fruit Juices and Nectars	Determination of sodium, potassium, calcium, magnesium in fruit juices EN 1134 IFUMA 33	Atomic Absorption Spectroscopy	II
Fruit Juices and Nectars	Determination of sorbitol-D IFUMA62	Enzymatic determination	II
Fruit Juices and Nectars	Determination of stable carbon isotope ratio in the pulp of fruit juices ENV 13070 Analytica Chimica Acta 340 (1997)	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of stable carbon isotope ratio of sugars from fruit juices ENV 12140 Analytica Chimica Acta.271 (1993)	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of stable hydrogen isotope ratio of water from fruit juices ENV 12142	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Determination of stable oxygen isotope ratio in fruit juice water ENV 12141	Stable isotope mass spectrometry	II
Fruit Juices and Nectars	Detection of starch AOAC 925.38 IFUMA 73	Colorimetric	I
Fruit Juices and Nectars	Determination of sugar beet derived syrups in frozen concentrated orange juice $\delta^{18}O$ Measurements in Water AOAC 992.09	Oxygen isotope ratio analysis	I
Fruit Juices and Nectars	Determination of titrable acids, total EN 12147 IFUMA 03 ISO 750	Titrimetry	I
Fruit Juices and Nectars	Determination of total dry matter (vacuum-oven drying at 70°C)* EN 12145 IFUMA 61	Gravimetric determination	I
Fruit Juices and Nectars	Determination of total solids (Microwave oven drying)* AOAC 985.26	Gravimetric determination	I
Fruit Juices and Nectars	Determination of Vitamin C (dehydro-ascorbic acid and ascorbic acid) AOAC 967.22	Microfluorometry	III

* *Because there is no numerical value in the Standard duplicate Type I methods have been included which may lead to different results.*

Milk and Milk Products				
Milk products	Iron	NMKL 139 AOAC 999.11 (Codex general method)	Atomic absorption spectrophotometry	II
Milk products	Iron	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	III
Milk products	Iron	AOAC 984.27	Inductively Coupled Plasma optical emission spectrophotometry	III
Milk products	Iron	ISO 6732 | IDF 103	Photometry (bathophenanthroline)	IV
Milk and Milk Products	Melamine	ISO/TS 15495 | IDF/RM 230	LC-MS/MS	IV
Milk products (products not completely soluble in ammonia)	Milk fat	ISO 8262-3 | IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat[15] (MSNF)	ISO 6731 | IDF 21 and ISO 1737| IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[15]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	IV
Blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[15]	AOAC 991/20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Total fat	ISO 1737| IDF 13	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)	ISO 6731 | IDF 21 and ISO 1737 | IDF 13	Calculation from total solids content and fat content Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[15]	ISO 8968-1 | IDF 20-1/	Titrimetry (Kjeldahl)	IV
Reduced fat blend of evaporated skimmed milk and vegetable fat	Milk protein in MSNF[15]	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Total fat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I

[15] Milk total solids and Milk solids-not-fat (MSNF) content include water of crystallization of lactose

Milk and Milk Products				
Blend of skimmed milk and vegetable fat in powdered form	Water[16]	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[15]	ISO 8968-1 | IDF 20-1/	Titrimetry (Kjeldahl)	IV
Blend of skimmed milk and vegetable fat in powdered form	Milk protein in MSNF[15]			
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Total fat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Water[16]	ISO 5537 | IDF 26	Gravimetry, drying at 87 °C	I
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[15]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of skimmed milk powder and vegetable fat in powdered form	Milk protein in MSNF[15]	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Blend of sweetened condensed skimmed milk and vegetable fat	Sucrose	ISO 2911 | IDF 35	Polarimetry	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)	ISO 6734 | IDF 15	Calculation from total solids content, fat content and sugar content	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF[15]	ISO 8968-1|IDF 20-1/	Titrimetry (Kjeldahl)	IV
Blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF[15]	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Total fat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk solids-not-fat (MSNF)	ISO 6734 | IDF 15	Calculation from total solids content and sugar content	IV

[16] Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)

Milk and Milk Products				
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein in MSNF[15]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	IV
Reduced fat blend of sweetened condensed skimmed milk and vegetable fat	Milk protein MSNF[15]	AOAC 991.20	Titrimetry (Kjeldahl)	IV
Butter	Copper	ISO 5738 | IDF 76AOAC 960.40	Photometry, diethyldithiocarbamate	II
Butter	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Butter	Milk solids-not-fat (MSNF)	ISO 3727-2 | IDF 80-2	Gravimetry	I
Butter	Milkfat	ISO 17189 | IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
Butter	Milk fat purity	ISO 17678 | IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Butter	Salt	ISO 1738 | IDF 12/ AOAC 960.29	Titrimetry (Mohr: determination of chloride, expressed as sodium chloride)	III
Butter	Salt	ISO 15648 | IDF 179	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Butter	Vegetable fat (sterols)	ISO 12078 | IDF 159	Gas chromatography	II
Butter	Vegetable fat (sterols)	ISO 18252 | IDF 200	Gas chromatography	III
Butter	Water[16]	ISO 37271 | IDF 80	Gravimetry	I
Cheese	Citric acid	ISO/TS 2963 | IDF/RM 34	Enzymatic method	IV
Cheese	Citric acid	AOAC 976.15	Photometry	II
Cheese	Milkfat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Cheese	Moisture	ISO 5534 | IDF 4	Gravimetry, drying at 102 °C	I

Milk and Milk Products				
Cheese (and cheese rind)	Natamycin	ISO 9233-1 | IDF 140-1	Molecular absorption spectrophotometry	III
		ISO 9233-2 | IDF 140-2	HPLC	II
Cheese	Sodium chloride	ISO 5943 | IDF 88	Potentiometry (determination of chloride, expressed as sodium chloride)	II
Cheeses, individual	Dry matter (Total solids)	ISO 5534 | IDF 4	Gravimetry, drying at 102°C	I
Cheeses, individual	Milk fat in dry matter	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cheeses, individual	Dry matter (Total solids)	ISO 5534 | IDF 4	Gravimetry, drying at 102°C	I
Cheeses in brine	Milk fat in dry matter (FDM)	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Cottage cheese	Fat-free dry matter		Calculation from dry matter content and fat content	I
		ISO 5534 | IDF 4 and ISO 1735 | IDF 5	Gravimetry, drying at 102 °C Gravimetry (Schmid-Bondzynski-Ratzlaff)	
Cottage cheese	Milk fat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff) (for samples containing lactose up to 5%)	I
		ISO 8262-3 | IDF 124-3	Gravimetry (Weibull-Berntrop) (for samples containing lactose over 5%)	I
Cottage cheese	Milk fat in dry matter	ISO 8262-3 | IDF 124-3	Gravimetry (Weibull-Berntrop)	I
Cheese, Unripened Including Fresh Cheese	Milk Protein	ISO 8968-1 | IDF 20-1	Titrimetry, Kjeldahl	I
Cream and Prepared Creams	Milk protein	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Cream	Milkfat	ISO 2450 | IDF 16	Gravimetry (Röse-Gottlieb)	I
Cream	Solids	ISO 6731 | IDF 21	Gravimetry (drying at 102°C)	I
Creams Lowered in Milkfat Content	Milkfat	ISO 2450 | IDF 16 / AOAC 995.19	Gravimetry (Röse-Gottlieb)	I
Creams, Whipped Creams and Fermented Creams	Milk solids-not-fat (MSNF)[15]	ISO 3727-2 | IDF 80-2 AOAC 920.116	Gravimetry	I

Milk and Milk Products				
Cream cheese	Dry matter	ISO 5534 | IDF 4	Gravimetry drying at 102 °C (forced air oven)	I
Cream cheese	Moisture on fat free basis		Calculation from fat content and moisture content	I
		ISO 5534 | IDF 4	Gravimetry drying at 102°C (forced air oven)	
		ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	
Dairy fat spreads	Milk fat purity	ISO 17678 | IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Dairy fat spreads	Total fat	ISO 17189 | IDF 194	Gravimetry Direct determination of fat using solvent extraction	I
Dairy fat spreads	Vegetable fat (sterols)	ISO 12078 | IDF 159	Gas chromatography	II
Dairy fat spreads	Vegetable fat (sterols)	ISO 18252 | IDF 200	Gas chromatography	III
Edible casein products	Acids, free	ISO 5547 | IDF 91	Titrimetry (aqueous extract)	IV
Edible casein products	Ash (including P_2O_5)	ISO 5545 | IDF 90 or[17] ISO 5544 | IDF 89	Gravimetry (ashing at 825 °C)	I
Edible casein products	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II
Edible casein products	Copper	ISO 5738 | IDF 76	Colorimetry (diethyldiethiocarbamate)	III
Edible casein products	Lactose	ISO 5548 | IDF 106	Photometry (phenol and H_2SO_4)	IV
Edible casein products	Lead	NMKL 139 (Codex general method) AOAC 999.11	Atomic absorption spectrophotometry	II
Edible casein products	Lead	NMKL 161 / AOAC 999.10	Atomic absorption spectrophotometry	III
Edible casein products	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	III
Edible casein products	Lead	AOAC 982.23 (Codex general method)	Anodic stripping voltanmetry	III

[17] Refer to scope of methods

Milk and Milk Products				
Edible casein products	Lead	ISO/TS 6733 | IDF/RM 133	Spectrophotometry (1,5-diphenylthiocarbazone)	IV
Edible casein products	Milkfat	ISO 5543 | IDF 127	Gravimetry (Schmid-Bondzynski-Ratslaff)	I
Edible casein products	pH	ISO 5546 | IDF 115	Electrometry	IV
Edible casein products	Milk Protein (total N x 6.38 in dry matter)	ISO 8968-1 | IDF 20-1	Titrimetry, Kjeldahl	I
Edible casein products	Sediment (scorched particles)	ISO 5739 | IDF 107	Visual comparison with standard disks, after filtration	IV
Edible casein products	Water[16]	ISO 5550 | IDF 78	Gravimetry (drying at 102 °C)	I
Emmental	Calcium >= 800mg/100g	ISO 8070 | IDF 119	Flame atomic absorption	IV
Evaporated milks	Milk fat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Evaporated milks	Milk Protein in MSNF[15]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Evaporated milks	Solids, total	ISO 6731 | IDF 21	Gravimetry (drying at 102°C)	I
Fermented milks	Colony-forming units of yeasts and/or moulds	ISO 6611 | IDF 94	Colony-count at 25 °C	IV
Fermented milks	Dry matter (total solids)	ISO 13580 | IDF 151	Gravimetry (drying at 102 °C)	I
Fermented milks	total acidity expressed as percentage of lactic acid	ISO/TS 11869 | IDF/RM 150	Potentiometry, titration to pH 8.30	I
Fermented milks	*Lactobacillus acidophilus*	ISO 20128 | IDF 192	Colony count at 37 °C	I
Fermented milks - Yoghurt and yoghurt products	*Lactobacillus delbrueckii* subsp *bulgaricus* & *Streptococcus thermophilus*	ISO 7889 | IDF 117	Colony count at 37°C	I
Fermented milks - Yoghurt and yoghurt products	*Lactobacillus delbrueckii* subsp *bulgaricus* & *Streptococcus thermophilus*	ISO 9232 | IDF 146	Test for strain identification	I

Milk and Milk Products				
Fermented milks	Microorganisms constituting the starter culture	ISO 27205 | IDF 149(Annex A)	Colony count at 25 °C, 30 °C, 37 °C and 45 °C according to the starter organism in question	IV
Fermented milks	Milk fat	ISO 1211 | IDF 1 / AOAC 989.05	Gravimetry (Röse-Gottlieb)	I
Fermented milks	Milk Protein	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Milk powders and cream powders	Acidity, titratable	ISO 6091 | IDF 86	Titrimetry, titration to pH 8.4	I
Milk powders and cream powders	Milk fat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Milk powders and cream powders	Milk Protein	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Milk powders and cream powders	Scorched particles	ISO 5739 | IDF 107	Visual comparison with standard disks, after filtration	IV
Milk powders and cream powders	Solubility Index	ISO 8156 | IDF 129	Centrifugation	I
Milk powders and cream powders	Water[16]	ISO 5537 | IDF 26[18]	Gravimetry (drying at 87°C)	I
Milk fat Products	Copper	ISO 5738 | IDF 76 AOAC 960.40	Photometry, diethyldithiocarbamate	II
Milk fat products	Fatty acids, free (expressed as oleic acid)	ISO 1740 | IDF 6	Titrimetry	I
Milk fat products	Milk fat purity	ISO 17678 | IDF 202	Calculation from determination of triglycerides by gas chromatography	I
Milk fat Products	Peroxide value (expressed as meq. of oxygen/kg fat)	ISO 3976 | IDF 74	Photometry	I
Milkfat products (anhydrous milkfat)	Peroxide value	AOAC 965.33	Titrimetry	I
Milk fat products	Vegetable fat (sterols)	ISO 12078 | IDF 159	Gas chromatography	II
		ISO 18252 | IDF 200	Gas chromatography	III
ilk fat products	Water	ISO 5536 | IDF 23	Titrimetry (Karl Fischer)	II
Milk fat products (anhydrous milk fat)	Peroxide value	ISO 3976 | IDF 74	Photometry	I
Milkfat products (anhydrous milkfat)	Peroxide value	AOAC 965.33	Titrimetry	I
Mozzarella	Milkfat in dry matter – with high moisture	ISO 1735 | IDF 5	Gravimetry after solvent extraction	I
Mozzarella	Milkfat in dry matter – with low moisture	ISO 1735 | IDF 5	Gravimetry after solvent extraction	I
Sweetened condensed milk	Milkfat	ISO 1737 | IDF 13	Gravimetry (Röse-Gottlieb)	I
Sweetened Condensed Milks	Milk Protein in MNSF[15]	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I

[18] Water content excluding the crystallized water bound to lactose (generally known as “moisture content”).

Milk and Milk Products				
Sweetened Condensed Milks	Solids	ISO 6734 | IDF 15	Gravimetry, drying at 102 °C	I
Whey cheeses by coagulation	Milk fat	ISO 1735 | IDF 5	Gravimetry (Schmid-Bondzynski-Ratzlaff)	I
Whey cheeses by coagulation	Milk fat in dry matter	ISO 1735 | IDF 5and ISO 5534 | IDF 4	Calculation from fat content and dry matter content Gravimetry (Schmid-Bondzynski-Ratzlaff) Gravimetry, drying at 102°C	I
Whey cheeses by concentration	Milk fat	ISO 1854 | IDF 59	Gravimetry (Röse Gottlieb)	I
Whey cheeses by concentration	Milk fat in dry matter	ISO 1854 | IDF 59and ISO 2920 | IDF 58	Calculation from fat content and dry matter content Gravimetry (Röse Gottlieb) Gravimetry, drying at 88 C	I
Whey powders	Ash	ISO 5545 | IDF 90	Gravimetry (ashing at 825°C)	IV
Whey powders	Copper	AOAC 985.35	Atomic absorption spectrophotometry	II
Whey powders	Copper	ISO 5738 | IDF 76	Photometry (diethyldithiocarbamate)	III
Whey Powders	Lactose	ISO 5765-1/2 | IDF 79-1/2	Enzymatic method: Part 1 - Glucose moiety or Part 2 - Galactose moiety	II
Whey powders	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Whey powders	Milkfat	ISO 1736 | IDF 9	Gravimetry (Röse-Gottlieb)	I
Whey powders	Milk protein (total N x 6.38)	ISO 8968-1 | IDF 20-1	Titrimetry (Kjeldahl)	I
Whey powders	Moisture, "Free"	ISO 2920 | IDF 58	Gravimetry (drying at 88°C ±2°C)	IV
Whey powders	Water[19]	ISO 5537 | IDF 26	Gravimetry (drying at 87°C)	I

[19] Water content excluding the crystallized water bound to lactose (generally known as “moisture content”)

Natural Mineral Waters				
Natural mineral waters	Calcium	ISO 7980	Atomic absorption spectrophotometry	III
Natural mineral waters	Chloride	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol. 2, pp. 205-208		II
Natural mineral waters	Chloride	AOAC 973.51	Titrimetry (Mercuric nitrate)	III
Natural mineral waters	Chloride	ISO 9297	Titrimetry	III
Natural mineral waters	Iron, dissolved	ISO 6332	Spectrophotometry	II
Natural mineral waters	Magnesium	ISO 6059	Titrimetry	II
Natural mineral waters	Magnesium	ISO 7980	Atomic absorption spectrophotometry	III
Natural mineral waters	Phenols	ISO 6439	Spectrophotometry	I
Natural mineral waters	Potassium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol.2, pp. 142-145		II
Natural mineral waters	Sodium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol.2 pp. 148-151		II
Natural mineral waters	Sodium	*Examination of Water Pollution Control.* WHO Pergamon Press (1982) Vol.2, pp. 151-152		III
Natural mineral waters	Sulphates	ISO 9280	Gravimetry	III
Natural mineral waters	Sulphide	*Handb. Spurenanal.* 1974		IV

Criteria applicable to health-related substances in the *Standard for Natural Mineral Waters* (CODEX STAN 108-1981)

Provision	ML (mg/L)	Min. applicable range (mg/L)	LOD (mg/L)	LOQ (mg/L)	Precision RSDR (%) No more than	Recovery (%)	Suggested methods meeting the criteria	Principle
Antimony	0.005	0.0028	0.001	0.002	44	80-110	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Arsenic	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 ISO 11969 EPA 200.8	ICP-MS GF-AAS AAS (Hydride) ICP-MS
Barium	0.7	0.35	0.07	0.14	34	95-105	ISO 11885 ISO 17294-2 EPA 200.8	ICP-OES ICP-MS ICP-MS
Borate	5	3.1	0.5	1	25	97-103	ISO 9390 ISO 11885 ISO 17294-2	Spectrophotometry ICP-OES[20] ICP-MS[20]
Cadmium	0.003	0.0017	0.0006	0.0012	44	80-110	ISO 11885 ISO 17294-2 ISO 15586 ISO 5961 (Section 3) EPA 200.8	ICP-OES ICP-MS GF-AAS AAS ICP-MS
Chromium	0.05	0.028	0.01	0.02	44	90-107	ISO 11885 ISO 17294-2 ISO 15586ISO 18412 (Cr VI) ISO 23913 (Cr VI) ISO 9174 (Section 4) EPA 200.8	ICP-OES ICP-MS GF-AAS Photometric CIA, spectrophotometry AAS ICP-MS
Copper	1	0.52	0.1	0.2	32	97-103	ISO 11885 ISO 17294-2 ISO 15586 ISO 8288 EPA 200.8	ICP-OES ICP-MS GF-AAS Flame-AAS ICP-MS
Cyanide	0.07	0.039	0.014	0.028	44	90-107	ISO 14403ISO 6703-1	CFA Photometric, trimetric
Fluoride	1.0	0.52	0.1	0.2	32	97-103	ISO 10304-1 ISO 10359-1 (dissolved fluoride) ISO 10359-2 (inorganic bound)	LC of ions Electrochemical probe Digestion, distillation

[20] Total Boron is determined

Provision	ML (mg/L)	Min. applicable range (mg/L)	LOD (mg/L)	LOQ (mg/L)	Precision RSDR (%) No more than	Recovery (%)	Suggested methods meeting the criteria	Principle
Lead	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Manganese	0.4	0.18	0.04	0.08	37	95-105	ISO 11885I SO 17294-2 ISO 15586 EPA 200.8	ICP-OES ICP-MS GF-AAS ICP-MS
Mercury	0.001	0.00056	0.0002	0.0004	44	80-110	EN 1483 ISO 17852 ISO 5666 ISO 16590 EPA 200.8	AAS Enrichment by amalgamation (III) AFS AAS after tin(II) chloride reduction Enrichment by amalgamation (III) ICP-MS
Nickel	0.02	0.011	0.004	0.008	44	90-107	ISO 17294-2 ISO 15586 EPA 200.8	ICP-MS GF-AAS ICP-MS
Nitrate	50	37	5	10	18	98-102	ISO 10304-1 ISO 13395 ISO 7890-3	LC of ions CFA, FIA, Spectrophotometry Spectrophotometry
Nitrite	0.1	0.03	0.01	0.02	44	95-105	ISO 10304-1 ISO 13395 ISO 6777	LC of ions UV CFA, FIA, Spectrophotometry Spectrophotometry
Selenium	0.01	0.0056	0.002	0.004	44	90-107	ISO 17294-2 ISO 15586 ISO 9965 EPA 200.8	ICP-MS GF-AAS AAS (Hydride) ICP-MS

Performance characteristics of suggested methods

Provision	ML	Applicable range-from:	LOD	RSDR (%)	Recovery (%)	Suggested methods	Principle
Surface active agents	-	0.05 – 5.0 mg/L	0.05 mg/l	< 44	70-100	ISO 16265	CFA
Mineral oil (hydrocarbon index)	-	>0.1 mg/L		< 41	71-102	ISO 9377-2	GC
PCB	-	>15 ng/L		<20	70-130	AOAC 990.06	GC ECD
Pesticide (organochlorine)	-	> 15 ng/ L		<20	70-130	AOAC 990.06	GC ECD
PAH	-	0.005 µg/L 0.04 µg/L 0.005 µg/L		<10 <18 <19	80-110 80-110 80-100	ISO 17993 ISO 7981-1 ISO 7981-2	HPLC FD TLC HPLC

Processed Fruits and Vegetables

Commodity	*Provision*	*Method*	*Principle*	*Type*
Processed fruits and vegetables	Benzoic acid	NMKL 124	Liquid Chromatography	II
Processed fruits and vegetables	Benzoic acid	NMKL 103; or AOAC 983.16	Gas Chromatography	III
Processed fruits and vegetables	Calcium	AOAC 968.31	Complexometry/ Titrimetry	II
Processed fruits and vegetables	Drained Weight	AOAC 968.30 (Codex General Method)	Sieving Gravimetry	I
Processed fruits and vegetables	Fill of containers	CAC/RM 46 (reference to “metal containers” deleted and refer to ISO 90-1 for determination of water capacity in metal containers)	Weighing	I
Processed fruits and vegetables	Lead	AOAC 972.25 (Codex general method)	AAS (Flame absorption)	III
Processed fruits and vegetables	Packing medium Canned berry fruits (raspberry, strawberry)	AOAC 932.12 ISO 2173	Refractometry	I
Processed fruits and Vegetables (except canned bamboo shoots, pH determined by AOAC 981.12)	pH	ISO 1842	Potentiometry	IV

Processed Fruits and Vegetables

Commodity	*Provision*	*Method*	*Principle*	*Type*
Processed fruits and vegetables	pH	AOAC 981.12	Potentiometry	III
Processed fruits and vegetables	pH	NMKL 179	Potentiometry	II
Processed fruits and vegetables	Soluble solids	ISO 2173 AOAC 932.12	Refractometry	I
Processed fruits and vegetables	Sorbates	NMKL 103 / AOAC 983.16	Gas Chromatography	III
Processed fruits and vegetables	Sorbates	NMKL 124	Liquid Chromatography	II
Processed fruits and vegetables	Tin	AOAC 980.19 (Codex general method)	AAS	II
Processed fruits and vegetables	Total solids	AOAC 920.151	Gravimetry	I
Aqueous Coconut Products	Total Fats	ISO 1211 | IDF 1	Gravimetry (Röse-Gottlieb)	I
Aqueous Coconut Products	Total solids	ISO 6731 | IDF 21	Gravimetry	I
Aqueous Coconut Products	Non-fat solids	ISO 1211 | IDF 1ISO 6731 | IDF 21	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I
Aqueous Coconut Products	Moisture	ISO 6731 | IDF 21	Calculation: Gravimetry	I
Canned Apple Sauce	Fill of containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	I
Canned Apple Sauce	Soluble solids	AOAC 932.12 ISO 2173 (Codex general method for processed fruits and vegetables)	Refractometry	I
Canned green beans and wax beans	Tough strings	CAC/RM 39	Stretching	I

Processed Fruits and Vegetables

Commodity	*Provision*	*Method*	*Principle*	*Type*
Canned green peas	Proper fill (in lieu of drained weight)	CAC/RM 45	Pouring and measuring	I
Canned green peas	Types of peas, distinguishing	CAC/RM 48	Visual inspection	I
Canned mangoes	Syrup	AOAC 932.14C	Brix spindle method	I
Canned mushrooms	Washed drained weight	CAC/RM 44	Sieving	I
Canned palmito	Mineral impurities	ISO 762	Gravimetry	I
Canned Stone Fruits	Drained weight	AOAC 968.30 ISO:2173	Gravimetry	I
Canned Stone Fruits	Soluble solids	AOAC 932.14C	Refractometry	I
Canned strawberries	Calcium	AOAC 968.31	Complexometric titrimetry	II
Canned strawberries	Mineral impurities	AOAC 971.33	Gravimetry	I
Certain canned citrus fruits	Calcium	NMKL 153	Atomic Absorption Spectrophotometry	II
Certain canned citrus fruits	Calcium	AOAC 968.31	Complexometry Titrimetry	III
Certain Canned Vegetables (palmito)	Mineral impurities (sand)	AOAC 971.33 ISO 762	Gravimetry	I
Citrus marmalade	Calcium	AOAC 968.31	Complexometric titrimetry	II
Dates	Identification of defects	Described in the Standard	Visual inspection	I
Dates	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Desiccated coconut	Total acidity of the extracted oil	ISO 660 or AOCS Cd 3d-63	Titrimetry	I
Desiccated coconut	Ash	AOAC 950.49	Gravimetry	I
Desiccated coconut	Extraneous vegetable matter	Described in the Standard	Counting extraneous material with the naked eye	IV
Desiccated coconut	Moisture	AOAC 925.40	Gravimery (loss on drying)	I

Processed Fruits and Vegetables

Commodity	*Provision*	*Method*	*Principle*	*Type*
Desiccated coconut	Oil content	AOAC 948.22	Gravimetry	I
Dried apricots	Identification of defects	Described in the Standard	Visual inspection (weighing)	I
Dried apricots	Moisture	AOAC 934.06	Gravimetry (vacuum oven)	I
Dried apricots	Sulphur dioxide	AOAC 963.20	Colorimetry	II
Jams (fruit preserves) and jellies	Fill of Containers	CAC/RM 46	Weighing	I
Jams (fruit preserves) and jellies	Soluble solids	ISO 2173 AOAC 932.12	Refractometry	I
Mango chutney	Ash insoluble in HCl	ISO 763	Gravimetry	I
Pickled cucumbers	Acidity, total	AOAC 942.15	Titrimetry	I
Pickled cucumbers	Drained weight	AOAC 968.30	Gravimetry	I
Pickled cucumbers	Mineral impurities	AOAC 971.33	Gravimetry	I
Pickled cucumbers	Salt in brine	AOAC 971.27 (Codex general method)	Potentiometry	II
Pickled cucumbers	Volume fill by displacement	Described in the Standard	Displacement	I
Preserved tomatoes	Calcium	AOAC 968.31	Complexometric titrimetry	III
Preserved tomatoes	Calcium	NMKL 153	Atomic Absorption Spectrophotometry	II
Preserved tomatoes	Minimum Drained Weight	AOAC 968.30	Gravimetry (sieving) note: Use a No. 14 screen instead of ‘7/16’ or No. 8	I
Preserved tomatoes	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Lactic acid	EN 2631	Enzymatic determination	II
Processed tomato concentrates	Mineral impurities (sand)	AOAC 971.33	Gravimetry	IV
Processed tomato concentrates	Mould count	AOAC 965.41	Howard mould count	I
Processed tomato concentrates	Natural tomato soluble solids	AOAC 970.59	Refractometry	I

Processed Fruits and Vegetables

Commodity	*Provision*	*Method*	*Principle*	*Type*
Processed tomato concentrates	Sodium chloride	AOAC 971.27 (Codex general method)	Potentiometry	II
Processed tomato concentrates	Tomato soluble solids	AOAC 970.59	Refractometry	I
Raisins	Mineral impurities	CAC/RM 51	Ashing	I
Raisins	Mineral oil	CAC/RM 52	Extraction and separation on alumina	II
Raisins	Moisture	AOAC 972.20	Electrical conductance	I
Raisins	Sorbitol	AOAC 973.28	Gas chromatography	II
Raisins	Sulphur dioxide	AOAC 963.20	Colorimetry	II
Table olives	Drained weight	AOAC 968.30 (Codex general method for processed fruits and vegetables)	Sieving Gravimetry	I
Table olives	Fill of containers	CAC/RM 46* (for glass containers) (Codex general method for processed fruits and vegetables) and ISO 90-1 (for metal containers) (Codex general method for processed fruits and vegetables)	Weighing	I
Table olives	pH of brine	NMKL 179 (Codex general method for processed fruits and vegetables)	Potentiometry	II
		AOAC 981.12 (Codex general method for processed fruits and vegetables)		III
		ISO 1842		IV
Table olives	Salt in brine	AOAC 971.27 | NMKL 178 (Codex general method)	Potentiometry	II
Table olives	Lead	AOAC 999.11 | NMKL 139 (Codex general method)	AAS (Flame absorption)	II
Table olives	Tin	NMKL 190 | EN 15764	AAS	II

*** DETERMINATION OF WATER CAPACITY OF CONTAINERS (CAC/RM 46)**

1. **SCOPE**

This method applies to glass containers.

2. **DEFINITION**

The water capacity of a container is the volume of distilled water at 20°C which the sealed container will hold when completely filled.

3. **PROCEDURE**

3.1 Select a container which is undamaged in all respects.

3.2 Wash, dry and weigh the empty container.

3.3 Fill the container with distilled water at 20°C to the level of the top thereof, and weigh the container thus filled.

4. **CALCULATION AND EXPRESSION OF RESULTS**

Subtract the weight found in 3.2 from the weight found in 3.3. The difference shall be considered to be the weight of water required to fill the container. Results are expressed as mL of water.

Products	Provisions	Method	Principle	Type
Aqueous coconut products	Total Fats	ISO 1211 | IDF 1	Gravimetry (Röse-Gottlieb)	I
Aqueous coconut products	Totals Solids	ISO 6731 | IDF 21	Gravimetry	I
Aqueous coconut products	Non-fat solids	ISO 1211 | IDF 1 ISO 6731 | IDF 21	Calculation: Gravimetry (Röse-Gottlieb) Gravimetry	I
Aqueous coconut products	Moisture	ISO 6731 | IDF 21	Gravimetry	I

Quick Frozen Fruits and Vegetables				
Quick frozen fruits and vegetables	Net weight	CAC/RM 34	Weighing	I
Quick frozen fruits and vegetables	Thawing procedure	CAC/RM 32	Thawing	I
Quick frozen fruits and vegetables: Berries, leek and carrot	Mineral impurities	CAC/RM 54	Flotation and sedimentation	I
Quick frozen fruits and vegetables: Berries, Whole kernel corn and Corn-on-the-cob	Soluble solids, total	CAC/RM 43	Refractometry	I
Quick frozen fruits and vegetables: Peaches and berries	Drained fruit/drained berries	Described in the Standards	Draining	I
Quick frozen fruits and vegetables: Vegetables	Cooking procedure	CAC/RM 33	Cooking	I
Quick frozen French fried potatoes	Moisture	AOAC 984.25	Gravimetry (convection oven)	I
Quick frozen green and wax beans	Tough strings	CAC/RM 39	Stretching	I
Quick frozen peas	Solids, alcohol insoluble	CAC/RM 35	Gravimetry	I
Quick frozen spinach	Dry matter, Salt-free	Described in the Standard	Weighing	I
Processed Meat and Poultry Products and Soups and Broths				
Meat Products	Nitrates and/or Nitrites	EN 12014-3	Spectrometric determination of nitrate and nitrite content of meat products after enzymatic reduction of nitrate to nitrite	III
Meat Products	Nitrates and/or Nitrites	EN 12014-4 NMKL 165	Ion-exchange chromatographic method	III
Processed meat and poultry products	Fat	ISO 1443	Gravimetry	I
Processed meat and poultry products	Lead	AOAC 934.07	Colorimetry (dithizone)	II
Processed meat and poultry products	Nitrates	ISO 3091	Colorimetry (cadmium reduction)	II
Processed meat and poultry products	Nitrites	ISO 2918	Colorimetry	IV

Processed Meat and Poultry Products and Soups and Broths				
Processed meat and poultry products	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Processed meat and poultry products	Nitrogen/protein	ISO 937	Titrimetry	II
Bouillons and Consommés (soups and broths)	Amino nitrogen	AIIBP Method No 2/7	Volumetry (modified Van Slyke)	II
Bouillons and Consommés (soups and broths	Creatinine	AIIBP Method No 2/5	HPLC	II
Bouillons and Consommés (soups and broths	Nitrogen, total	AOAC 928.08	Kjeldahl	II
Bouillons and Consommés (soups and broths)	Sodium chloride	AIIBP Method No 2/4	Potentiometric titration (chloride expressed as sodium chloride)	II
Canned corned beef	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Canned corned beef	Nitrites, potassium and/or sodium salt	AOAC 973.31 (Codex general method)	Colorimetry	II
Canned corned beef	Nitrites, potassium and/or sodium salt	ISO 2918	Colorimetry	IV
Canned corned beef	Tin (Products in tinplate and other containers)	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured chopped meat	Fat	ISO 1443	Gravimetry (extraction)	I
Cooked cured chopped meat	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured chopped meat	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured chopped meat	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured chopped meat	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured ham	Fat	ISO 1443	Gravimetry (extraction)	I

Processed Meat and Poultry Products and Soups and Broths				
Cooked cured ham	Gelatin, added	Described in the Standard	Calculation	I
Cooked cured ham	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured ham	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured ham	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured ham	Protein (conversion factor 6.25)	ISO 937	Titrimetry, Kjeldahl digestion	II
Cooked cured ham	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured pork shoulder	Fat	ISO 1443	Gravimetry (extraction)	I
Cooked cured pork shoulder	Gelatin, added	Described in the Standard	Calculation	I
Cooked cured pork shoulder	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Cooked cured pork shoulder	Nitrites	AOAC 973.31 (Codex general method)	Colorimetry	II
Cooked cured pork shoulder	Nitrites	ISO 2918	Colorimetry	IV
Cooked cured pork shoulder	Protein	ISO 937	Titrimetry, Kjeldahl digestion	II
Cooked cured pork shoulder	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II
Luncheon meat	Fat	ISO 1443	Gravimetry (extraction)	I
Luncheon meat	Lead	AOAC 972.25 (Codex general method)	Atomic absorption spectrophotometry	II
Luncheon meat	Nitrites, potassium and/or sodium salt	AOAC 973.31 (Codex general method)	Colorimetry	II
Luncheon meat	Nitrites, potassium and/or sodium salt	ISO 2918	Colorimetry	IV
Luncheon meat	Tin	AOAC 985.16 (Codex general method)	Atomic absorption spectrophotometry	II

Sugars and Honey				
Honey	Acidity	MAFF Validated Method V19 *J. Assoc. Public Analysts* (1992) 28 (4) 171-175	Titrimetry	I
Honey	diastase activity	IHC Method for Determination of Diastase activity with Phadebas, 2009 except that the incubation time should be increased from 15 to 30 minutes.		IV
Honey	Moisture	AOAC 969.38B or MAFF Validated Method V21	Refractometry	I
Honey	Sample preparation	AOAC 920.180	-	-
Honey	Solids, water-insoluble	MAFF Validated Method V22 *J. Assoc. Public Analysts* (1992) 28(4) 189-193	Gravimetry	I
Honey	Sugars added (for sugar profile)	AOAC 998.18	Carbon isotope ratio mass spectrometry	I
Honey	Sugars added: detection of corn and cane sugar products	AOAC 978.17	Carbon isotope ratio mass spectrometry	I
Sugars (dextrose anhydrous and dextrose monohydrate)	D-Glucose	ISO 5377	Titrimetry	I
Sugars (dextrose anhydrous and dextrose monohydrate)	Solids, total	ISO 1741	Gravimetry (vacuum oven)	I
Sugars (dextrose anhydrous and dextrose monohydrate, dried glucose syrup, glucose syrup, powdered dextrose, lactose)	Sulphated ash	ISO 5809	Single sulphonation	I
Sugars (dextrose anhydrous and dextrose monohydrate)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (fructose)	pH	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (fructose)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I

Sugars and Honey				
Sugars (fructose)	D-Fructose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	D-Glucose	ISO 10504	Liquid chromatography (refractive index detection)	II
Sugars (fructose)	Loss on drying	ISO 1742	Gravimetry	I
Sugars (fructose)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (glucose syrup and dried glucose syrup)	Reducing sugar	ISO 5377	Titrimetry	I
Sugars (glucose syrup and dried glucose syrup)	Solids, total	ISO 1742	Gravimetry (vacuum oven)	I
Sugars (glucose syrup and dried glucose syrup)	Sulphur dioxide	ISO 5379	Acidimetry and nephelometry	IV
Sugars (lactose)	Lactose, anhydrous	ICUMSA GS 4/3-3	Titrimetry	II
Sugars (lactose)	Loss on drying	USP General Chapter 731	Gravimetry (Drying at 120°C for 16 h)	I
Sugars (lactose)	pH	ICUMSA GS 1/2/3/4/7/8-23	Potentiometry	I
Sugars (plantation and mill white sugar)	Colour	ICUMSA GS9/1/2/3-8	Photometry	I
Sugars (plantation or mill white sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I
Sugars (plantation or mill white sugar)	Invert sugar	ICUMSA GS 1/3/7-3	Titrimetry (Lane & Eynon)	I
Sugars (plantation or mill white sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (plantation or mill white sugar)	Polarization	ICUMSA GS 1/2/3-1	Polarimetry	II
Sugars (plantation or mill white sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (powdered sugar and powdered dextrose)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II

Sugars and Honey				
Sugars (powdered sugar)	Colour	ICUMSA GS 2/3-9	Photometry	I
Sugars (powdered sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (powdered sugar)	Invert sugar	ICUMSA GS 2/3-5 after filtration if necessary to remove any anticaking agents	Titrimetry	I
Sugars (powdered sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (powdered sugar)	Polarization	ICUMSA GS 2/3-1 after filtration if necessary to remove any anticaking agents	Polarimetry	II
Sugars (raw cane sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (soft white sugar and soft brown sugar)	Conductivity ash	ICUMSA GS 1/3/4/7/8-13	Conductimetry	I
Sugars (soft white sugar and soft brown sugar)	Invert sugar	ICUMSA GS 4/3-3 (applicable at levels >10% m/m)	Titrimetry (Lane & Eynon)	I
Sugars (soft white sugar and soft brown sugar)	Invert sugar	ICUMSA GS 1/3/7-3 (applicable at levels <10% m/m)	Titrimetry (Lane & Eynon)	I
Sugars (soft white sugar and soft brown sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I
Sugars (soft white sugar and soft brown sugar)	Sucrose plus invert sugar	ICUMSA GS 4/3-7	Titrimetry	I
Sugars (soft brown sugar)	Sulphated ash	ICUMSA GS 1/3/4/7/8-11	Gravimetry	I
Sugars (soft white sugar and soft brown sugar)	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Sugars (soft white sugar)	Colour	ICUMSA GS 2/3-9	Photometry	I
Sugars (white sugar)	Conductivity ash	ICUMSA GS 2/3-17	Conductimetry	I
Sugars (white sugar)	Invert sugar	ICUMSA GS 2/3-5	Titrimetry	I
Sugars (white sugar)	Loss on drying	ICUMSA GS 2/1/3-15	Gravimetry	I

Sugars and Honey				
Sugars (white sugar)	Polarization	ICUMSA GS 2/3-1	Polarimetry	II
Sugars (white sugar	Sulphur dioxide	ICUMSA GS 2/3-35 NMKL 135 EN 1988-2	Enzymatic method	II
Miscellaneous Products				
Chili sauce	pH	NMKL 179 (Codex general method)	Potentiometry	II
Chili sauce	pH	AOAC 981.12 (Codex general method)	Potentiometry	III
Chili sauce	Fill of containers	CAC/RM 46 (Codex general method)	Weighing	I
Date Paste	Moisture	AOAC 934.06	Gravimetry	I
Date Paste	Mineral impurities	ISO 762	Gravimetry	I
Date Paste	Ash	AOAC 940.26	Gravimetry	I
Date Paste	Acid Soluble Ash	AOAC 900.02D	Gravimetry, Calculation	I
Edible cassava flour	Fibre, crude	ISO 5498 (B.5 separation)	Gravimetry	I
Edible cassava flour	Granularity	ISO 2591-1	Sieving	I
Edible cassava flour	Moisture	ISO 712	Gravimetry	I
Fermented Soybean Paste	Total Nitrogen	AOAC 984.13	Kjeldahl	I
Fermented Soybean Paste	Amino Nitrogen	AOAC 920.154 on the conditions specified in the standard [21]	Volumetry	I
Fermented Soybean Paste	Moisture	AOAC 934.01 (≤70°C, ≤ 50 mm Hg)	Gravimetry	I

[21] **Section 9.2 Determination of Amino Nitrogen**

Preparation of test samples: Weigh 2 g of sample into a 250 ml beaker and mix the sample with 100 ml of cold (15°C) NH_3-free H_2O and then stir the mixture for 60 min. Next, decant the mixture through a quantitative filter and collect the filtrate in a 100 ml volumetric flask.

Endpoint - A pH meter shall be used to determine the endpoint instead of optical verification of colours

Miscellaneous Products				
Food grade salt	Arsenic	EuSalt/AS 015	ICP-OES	IV
Food grade salt	Cadmium	EuSalt/AS 015	ICP-OES	III
Food grade salt	Cadmium	EuSalt/AS 014	Atomic absorption spectrophotometry	IV
Food grade salt	Calcium and magnesium	ISO 2482	Complexometric titrimetry	II
Food grade salt	Calcium and magnesium	EuSalt/AS 009	Flame atomic absorption spectrometry	III
Food grade salt	Calcium and magnesium	EuSalt/AS 015	ICP-OES	III
Food grade salt	Copper	EuSalt/AS 015	ICP-OES	III
Food grade salt	Insoluble matter	ISO 2479	Gravimetry	II
Food grade salt	Iodine	EuSalt/AS 002	Titrimetry using sodium thiosulphate	II
Food grade salt	Iodine	EuSalt/AS 019	ICP-OES	III
Food grade salt	Iodine	WHO/UNICEF/ICCIDD method[22] Only applicable to a product which has been fortified with iodate	Titrimetry using sodium thiosulphate	IV
Food grade salt	Lead	EuSalt/AS 015	ICP-OES	III
Food grade salt	Lead	EuSalt/AS 013	Atomic absorption spectrophotometry	IV
Food grade salt	Loss on drying	ISO 2483	Gravimetry (drying at 110°C)	I
Food grade salt	Mercury	EuSalt/AS 012	Cold vapour atomic absorption spectrophotometry	IV
Food grade salt	Potassium	EuSalt/AS 008	Flame atomic absorption spectrophotometry	II
Food grade salt	Potassium	EuSalt/AS 015	ICP-OES	III

[22] Assessment of iodine deficiency disorders and monitoring their elimination. A guide for programme managers. Third edition, Annex 1: Titration method for determining salt iodate and salt iodine content. World Health Organization, Geneva, 2007. The report is available from http://www.who.int/nutrition/publications/micronutrients/iodine_deficiency/WHO_NHD_01.1/en/index.html

Miscellaneous Products				
Food grade salt	Sodium chloride	Described in the Standard	Calculation	I
Food grade salt	Sulphate	ISO 2480	Gravimetry	II
Food grade salt	Sulphate	EuSalt/AS 015	ICP-OES	III
Food grade salt	Sulphate	EuSalt/AS 018	Ion chromatography	III
Foul medames	Sample Preparation	AOAC 945.68		_
Foul medames	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II
Foul medames	Drained weight	AOAC 968.30	Sieving	I
Gari	Ash	ISO 2171	Gravimetry	I
Gari	Fibre, crude	ISO 5498 (B.5 separation)	Gravimetry	I
Gari	Granularity	ISO 2591-1	Sieving	I
Gari	Moisture	ICC 109/1 ISO 712	Gravimetry	I
Ginseng Products	Moisture	AOAC 925.45 B (Dried ginseng) Quantity of sample: 2 g	Gravimetry	I
Ginseng Products	Moisture	AOAC 925.45 D (Ginseng extract) Quantity of sample: 1.5 g (mixing with 20 g of sea sand)	Gravimetry	I
Ginseng Products	Solids	AOAC 925.45 B (Dried ginseng) calculated by subtracting the content of water from 100% Quantity of sample: 2 g	Calculation	I
Ginseng Products	Ash	AOAC 923.03 AACC Intl 08-01.01	Gravimetry	I
Ginseng Products	Water-insoluble Solids	described in the Standard (Annex I)	Gravimetry	I

Miscellaneous Products				
Ginseng Products	Water-saturated n-butanol extracts	described in the Standard (Annex II)	Gravimetry	I
Ginseng Products	Identification of ginsenosides Rb1 and Rf	described in the Standard (Annex III)	TLC or HPLC	IV
Gochujang	Capsaicin	AOAC 995.03	HPLC	II
Gochujang		described in the Standard (Annex D)	Gas chromatography	IV
Gochujang	Crude protein	AOAC 984.13 (Nitrogen conversion factor: 6.25)	Kjeldahl	I
Gochujang	Moisture	AOAC 934.01 (≤ 70°C, ≤ 50 mm Hg)	Gravimetry	I
Guideline level for acrylonitrile	Acrylonitrile	AOAC 985.13	Gas chromatography	II
Guideline levels for mercury in fish	Methyl mercury	AOAC 988.11	Atomic absorption spectrophotometry	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	ISO 6401	Gas chromatography	II
Guideline levels for vinyl chloride monomer	Vinyl chloride monomer	Commission Directive 81/432/EEC O.J. No. L.167, p. 6, 24.6.81	Gas chromatography ("head-space")	III
Guidelines for nutrition labelling	Polyunsaturated fatty acids	AOCS Ce 1h-05[23]	Gas liquid chromatography	II
Guidelines for nutrition labelling	Saturated fat	AOAC 996.06; or AOCS Ce 1h-05	Gas liquid chromatography	II
Guidelines for nutrition labelling	Saturated fatty acids	AOCS Ce 1h-05	Gas liquid chromatography	II
Harissa	Acidity	ISO 750	titrimetry	I
Harissa	Acid insoluble ash	ISO 763	gravimetry	I

[23] Can also be used to measure *trans* unsaturated fatty acids

Miscellaneous Products				
Harissa	Dry extract – soluble solids	ISO 2173	refractometry	I
Halwa Tehenia	Acidity	AOAC 924.53, AOAC 942.15	Titrimetry	IV
Halwa tehenia	Ash	AOAC 900.02 | AACC Intl 8.14.01	gravimetry	I
Halwa tehenia	Fat	AOAC 963.15	gravimetry	I
Halwa tehenia	Moisture	AOAC 925.45 | AACC Intl 44.60.01	gravimetry	I
Halwa Tehenia	Sugars	ISI 28-1e [24]	Titrimetry	IV
Humus with tehena	Salt content	AOAC 971.27 NMKL 178	Potentiometry	II
Humus with tehena	Total acidity	AOAC 925.53	Titrimetry	I
Non-fermented soybean products	Moisture content	AOAC 925.09 | AACCI 44-40.01	Gravimetry (vacuum oven)	I
Non-fermented soybean products	Protein content	NMKL 6 or AACCI 46-16.01 or AOAC 988.05 or AOCS Bc 4-91 or AOCS Ba 4d-90 (Nitrogen factor 5.71)	Titrimetry, Kjeldahl digestion	I
Sago Flour	Moisture Content	ISO 712	Gravimetry	I
Sago Flour	Ash (inorganic extraneous matter)	ISO 2171	Gravimetry	I
Sago Flour	Acidity	AOAC 939.05	Titrimetry	I
Sago Flour	Crude Fibre	ISO 6541	Gravimetry	I

[24] http://www.starch.dk/isi/methods/28luff.htm

Miscellaneous Products				
Sago Flour	Starch	AOAC 920.44	Gravimetry	I
Tehena	Moisture Content	ISO 934	Gravimetry	I
Tehena	Protein content	ISO 1871	Titrimetry, Kjeldahl	I
Tehena	Total Ash	ISO 6884	Gravimetry	I
Tehena	Acid Insoluble Ash	ISO 735	Gravimetry	I
Tehena	Total Acidity	ISO 729	Titrimetry	I
Tehena	Sesame oil	AOCS Cb 2-40 (Baudouin Test)	Colour reaction	I
Tempe	Moisture content	AOAC 925.09 | AACCI 44-40.01	Gravimetry (vacuum oven)	I
Tempe	Protein content	NMKL 6 or AOAC 988.05 or AACCI 46-16.01 (Nitrogen factor 5.71)	Titrimetry, Kjeldahl digestion	I
Tempe	Lipid Content	AOAC 983.23	Gravimetry	I
Tempe	Crude fibre	ISO 5498 or AOAC 962.09 or AACCI 32-10.01	Gravimetry	I

PART B – METHODS OF SAMPLING BY COMMODITY CATEGORIES AND NAMES

Commodity Categories	Method of Sampling	Notes
Cereals, Pulses and Legumes and Derived Products		
Wheat protein products including wheat gluten	ISO 13690	
Fats and Oils		
Olive Oils and Olive-Pomace Oils	ISO 661 and ISO 5555.	
Fish oils	ISO 5555	
Milk and Milk Products		
Milk products	ISO 707 | IDF 50	General instructions for obtaining a sample from a bulk
Milk products	ISO 5538 | IDF 113	Inspection by attributes
Milk products	ISO 3951-1	Inspection by variables
Processed Fruits and Vegetables		
Desiccated coconut	Described in the Standard	
Certain canned vegetables, jams and jellies	Described in the Standard	
Chili sauce	Described in the Standard	
Table Olives	Described in the Standard	

CODEX ALIMENTARIUS COMMISSION

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World Health Organization

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REP17/MAS

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX ALIMENTARIUS COMMISSION

40th Session

Geneva, Switzerland, 17 – 22 July 2017

REPORT OF THE 38th SESSION OF THE CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING

Budapest, Hungary

8 – 12 May 2017

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SUMMARY AND STATUS OF WORK

Responsible Party	Purpose	Text / Topic	Code / Reference	Step	Para(s)
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Members CCEXEC73 CAC40	Adoption	Amendment to the Procedural Manual	Procedural Manual	-	36
CAC40	Revocation	Codex recommended methods in Codex standards	CODEX STAN 234-1999	-	28
CCFO	Information	Conversion factor phosphorous to phospholipids (draft Standard for fish oils)	-	-	9
CCPFV	Action / Information	Method for free fatty acids (annex on quick frozen French fries – Standard for quick frozen vegetables) Sampling plans (standards for ginseng and for quick frozen vegetables)	-	-	13, 14
CCASIA	Information / action	Method for acid value of laver proudct (and extraction method) Sampling plans (Standard for laver)	-	-	16, 17 and 18
CCNFSDU CCMAS39	Information / action	Methods for chromium, molybdenum and selenium, and trans fatty acids of infant formula And alternative methods to biological methods	-	-	19, 21, 28 and 40
CCAFRICA	Information	Methods for arsenic, lead and iron (Standard for unrefined shea butter)	-	-	22
CCSCH	Information / action	Sampling plans in the standards for cumin and thyme	-	-	24
All committees developing commodity standards	Information	Presentation of methods of analysis for endorsement	-	-	27
Secretariat / Committees	Publication Information / Use	Information documents: criteria for methods which use a “sum of components” and Practical examples for the selection of appropriate sampling plans	-	-	36 and 62
EWG (Chile and Mexico) CCMAS39	Development	Biological methods criteria	-	-	46

Responsible Party	Purpose	Text / Topic	Code / Reference	Step	Para(s)
CCEXEC73 CAC40 EWG (Brazil and Uruguay) CCMAS39	New work / review and update	Revision of CODEX STAN 234 / review and update of CODEX STAN 234	CODEX STAN 234-1999	1/2/3	25, 61
IDF/ISO/AOAC CCMAS39	Update	Review and update of methods of analysis for dairy products	CODEX STAN 234-1999	-	61
EWG (Germany) CCMAS39	Discussion	Revision of the *Guidelines on measurement uncertainty*	CAC/GL 54-2004	-	69
EWG (New Zealand) CCMAS39	Discussion	Revision of the *Guidelines on Sampling*	CAC/GL 50-2004	-	77
PWG (USA / Australia) CCMAS39	Endorsement	Methods of analysis and sampling for endorsement	CODEX STAN 234-1999	-	30, 40, 61

LIST OF ABBREVIATIONS

AOAC	AOAC International (formerly known as Association of Official Agricultural Chemists)
AOCS	American Oil Chemists' Society
CAC	Codex Alimentarius Commission
CCAFRICA	FAO/WHO Coordinating Committee for Africa
CCASIA	FAO/WHO Coordinating Committee for Asia
CCFO	Committee on Fats and Oils
CCMAS	Committee on Methods of Analysis and Sampling
CCNFSDU	Committee on Nutrition and Foods for Special Dietary Uses
CCPFV	Committee on Processed Fruits and Vegetables
CCSCH	Committee on Spices and Culinary Herbs
CRD	Conference room document
EU	European Union
EWG	Electronic working group
FDA	Food and Drug Administration (of the United States Department of Health and Human Services)
HPLC	High performance liquid chromatography
IAM	Interagency Meeting
ISO	International Organization for Standardization
NFCSO	National Food Chain Safety Office (Hungary)
MU	Measurement uncertainty
PWG	Physical working group
SDO	Standards development organisations

INTRODUCTION

1. The Codex Committee on Methods of Analysis and Sampling (CCMAS) held its 38th Session in Budapest, Hungary, from 8 to 12 May 2017, at the kind invitation of the Government of Hungary. The Session was chaired by Dr. Marót Hibbey, Veterinary officer, Ministry of Agriculture. Dr Ákos Jóźwiak, Vice director, National Food Chain Safety Office (NFCSO) and Dr Andrea Zentai, Food Safety Analyst (NFCSO), acted as the Vice-Chairpersons.

2. The Session was attended by 47 Member countries, 1 Member organization and 11 observer organizations. A list of participants is given in Appendix I.

OPENING OF THE SESSION

3. The Session was opened by Dr Lajos Bognár, Chief Veterinary Officer of Hungary and Deputy State Secretary of the Ministry of Agriculture who welcomed delegates to Hungary. Dr Márton Oravecz, President of the NFCSO also attended at the opening ceremony. Dr Bognár reminded the delegates of the importance of Codex in protecting public health and promoting fairness in trade. He highlighted the inter-dependency of Codex work and importance of food chain safety and wished the Committee successful deliberations.

Division of Competence[1]

4. The Committee noted the division of competence between the European Union and its Member States, according to paragraph 5, Rule II of the Rules of Procedure of the Codex Alimentarius Commission.

ADOPTION OF THE AGENDA (Agenda item 1)[2]

5. The Committee adopted the Provisional Agenda as its Agenda for the Session.

MATTERS REFERRED TO THE COMMITTEE BY THE CODEX ALIMENTARIUS COMMISSION AND OTHER SUBSIDIARY BODIES (Agenda item 2)[3]

6. The Committee noted (i) the matters of interest arising from the Codex Alimentarius Commission and its subsidiary bodies; and (ii) several matters for action had been considered by the physical Working Group (PWG) on endorsement and would be considered under Agenda item 3.

7. In addition the Committee took the following decision.

Committee on Fats and Oils

Conversion factor for phosphorous to phospholipids

8. The Observer of AOCS informed the Committee that while it would be possible to establish a theoretical conversion factor, establishment of a practical single conversion factor was not possible.

9. The Committee agreed to inform CCFO that CCMAS was not in a position to recommend a single conversion factor.

ENDORSEMENT OF METHODS OF ANALYSIS PROVISIONS AND SAMPLING PLANS IN CODEX STANDARDS (Agenda item 3)[4]

10. The Committee considered the recommendations on methods of analysis and sampling plans proposed for endorsement and other related matters as presented in CRD2. The Committee agreed with some of the recommendations of the WG and made the following amendments or recommendations. All decisions are presented in Appendix II.

Committee on Processed Fruits and Vegetables

Methods for quick frozen vegetables – RM methods

11. In view of the replacement of CAC/RM34, 43 and 54 with AOAC 963.26, AOAC 932.12 and AOAC 971.33, respectively, the Committee agreed to request their revocation by CAC40.

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[1] CRD1
[2] CX/MAS 17/38/1
[3] CX/MAS 17/38/2-Rev; Report of the pWG on endorsement of methods of analysis and sampling (CRD2); Comments from Philippines, Kenya, AOAC, IDF, ISO and Mexico (CRD 6), India (CRD 13), Republic of Korea (CRD 18).
[4] CX/MAS 17/38/3; CX/MAS 17/38/3 Add 1; Report of the PWG on endorsement of methods of analysis and sampling (CRD2); comments of Philippines, Kenya, AOAC, IDF, ISO, Mexico and Ghana (CRD 6), Senegal (CRD 14), Nigeria (CRD 15).

Quick frozen French fried potatoes – method for free fatty acids

12. The Committee noted that the methods for the determination of free fatty acids was for fats and oils and not for foods and that a method for fat extraction was necessary prior to the use of the suggested methods.

13. The Committee therefore agreed to request CCPFV to recommend a method for fat extraction.

Sampling plans

14. The Committee did not endorse the sampling plans for ginseng and for quick frozen vegetables since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004) and it was unclear whether the attributes sampling plan actually applied to attributes and not to characteristics that might be described as variable. The Committee noted that a similar question had already been posed to CCPFV with regard to the sampling plan for ginseng and that CCPFV had replied that if the resubmitted sampling plan was not appropriate, CCMAS should develop appropriate sampling plans. The Committee noted the offer of New Zealand (as chair of the EWG on revision of GL50) to develop a template to provide guidance to committees for development of sampling plans, and therefore agreed to defer decision on developing sampling plans at this time.

15. The Committee further noted that similar sampling plans had been endorsed in the past for processed fruits and vegetables and that CCMAS would need to address all sampling plans in a comprehensive way to avoid inconsistencies in *Recommended Methods of Analysis and Sampling* (CODEX STAN 234) and/or commodity standards.

FAO/WHO COORDINATING COMMITTEE FOR ASIA (CCASIA)

Methods of analysis for laver products

16. The Committee did not endorse the methods for acid value and agreed to request clarification from CCASIA whether the provision “acid value” applied to the laver product itself, or the extracted oil. If the method was for the extracted oil, it could be endorsed as Type I.

17. The Committee further noted that the extraction method in the Standard for laver products had been validated for instant noodles and not for laver, and that in this case, a classification as Type IV was recommended, and encouraged CCASIA to submit validation data to CCMAS to reconsider the proposed typing.

18. The Committee did not endorse the sampling plans since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004). It was noted that the sampling plans provided were attribute based. It was questioned whether a sampling plan by variables is more appropriate for certain provisions and requested CCASIA to reconsider the values in line with GL50. The Committee also agreed to inform CCASIA that it would be providing commodity committees with a template for developing sampling plans in case the Committee would like to await developing sampling plans until such time CCMAS would provide the aforesaid template.

COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY PURPOSES

Chromium, molybdenum and selenium

19. The Committee agreed to endorse the new methods for chromium, molybdenum and selenium as Type II and retained or retyped, where necessary, the older methods as Type III. The Committee further agreed to inform CCNFSDU of its concerns that the Type III methods may not all meet the requirements necessary for the determination of analytes at the minimum levels stated in the *Standard for Infant Formula and Formulas for Special Medical Purposes Intended for Infants* (CODEX STAN 72-1981) and that CCMAS could reconsider the endorsement of the Type III methods based on validation data to be submitted CCMAS at its next session.

Total fatty acids

20. The Committee endorsed the AOAC 2012.13|ISO 16958|IDF 231 for total fatty acids, noting that the provision was correct as stated in CODEX STAN 72.

Trans fatty acids

21. The Committee agreed to forward information on the methods identified by CCNFSDU on the matrices and levels for which they had been validated for their consideration (Appendix II, part 3).

FAO/WHO Coordinating Committee for Africa (CCAFRICA)

22. The Committee endorsed all methods submitted by CCAFRICA for the provisions in the proposed draft Standard for unrefined shea butter with the exception of the methods for arsenic, lead and iron as there were no provisions for these contaminants in the Standard.

Committee on Spices and Culinary Herbs (CCSCH)

Cumin and thyme: methods for insect damage, mammalian excreta and mould damage

23. The Committee noted the concern expressed by a delegation with regard to the endorsement of certain national methods (FDA method) rather than internationally validated methods. It was clarified that while internationally validated methods were desirable, the FDA methods had been agreed upon by CCSCH and were fit for purpose, and no other internationally validated methods had been identified or were available at this time.

Sampling plans

24. The Committee did not endorse the sampling plans since the values in the table did not correspond to those recommended in the *General Guidelines on Sampling* (CAC/GL 50-2004). It was unclear whether the sampling plan provided were being applied to attributes or variable characteristics and requested CCSCH to reconsider the values in line with GL50. The Committee also agreed to inform CCSCH that it would be providing commodity committees with a template for developing sampling plans in case the Committee would like to await developing sampling plans until such time CCMAS would provide the aforesaid template.

Other matters

Presentation of methods in CODEX STAN 234

25. The Committee clarified the presentation of multiple methods for a provision in CODEX STAN 234. When methods were identical and/or collaboratively developed, the references for these methods were separated by a vertical bar |, whereas when methods were technically identical, but were formatted or written differently, then the references for these methods were separated by a forward slash /. In the latter case, these methods could be typed as Type I as the methods were technically identical and would produce the identical analytical results. The Committee decided to ask the EWG on CODEX STAN 234 to consider defining the forward slash (/) and advise the Committee at the next meeting.

Process for timely information on endorsement of methods

26. The Committee noted the need for a procedure to ensure that information to assist in the endorsement work of the PWG is provided in a timely manner. The USA, as chair of the WG, informed the Committee that he was in consultation with the Codex Secretariat to address this matter. Ways were being explored to deliver methods for endorsement to SDOs earlier to allow feedback to the PWG co-chairs in advance so that a preparatory document could be circulated to all delegates prior to the session.

Presentation of methods of analysis by committees

27. The Committee agreed to remind committees that when methods are submitted to CCMAS for endorsement, these methods should indicate also the principle as well as proposed typing for the methods.

Conclusion

28. The Committee agreed to send:

- the methods of analysis, as endorsed, to CAC40 for adoption (Appendix II, Part 1),
- the methods for revocation to CAC40 (Appendix II, Part 2); and
- the information on the methods for trans fatty acids to CCNFSDU for their consideration (Appendix II, Part 3).

29. Uruguay expressed their reservation to the decision on the methods of analysis for quick frozen vegetables, as the methods of analysis presented for endorsement (Appendix I, CX/MAS 17/38/3) had been omitted from the Spanish version of the document. Uruguay was therefore not in a position to examine the methods prior to the session.

30. The Committee agreed to re-establish the PWG on methods of analysis and sampling, chaired by USA and co-chaired by Australia, working in English only, to meet immediately prior to the next session.

GUIDANCE ON THE CRITERIA APPROACH FOR METHODS WHICH USE A "SUM OF COMPONENTS" (Agenda item 4)[5]

31. The United Kingdom, as Chair of the EWG, introduced the item. The Delegation reminded the Committee of the decision of CCMAS37 for the work to continue and that this session would take a decision on how to take this work forward[6].

32. The Delegation indicated that overall the EWG agreed that the approaches available in developing criteria approaches for methods that use a sum of components were complex and need to be addressed on a case-by-case basis. In order to take the work forward the Delegation suggested that firstly Note 2 to the *Working Instructions for the Implementation of the Criteria Approach in Codex* of the Procedural Manual be revised to reinforce the complexity of the issues involved and secondly, Appendix 1 of CX/MAS 17/38/4 be converted into an Information Document format for publication on the Codex website so that the information and guidance developed were readily accessible to users wishing to develop numeric method performance criteria for methods that are a sum of components.

33. The Committee recognized that there were numerous ways in which methods and limits that involve a sum of components could be converted into numeric method performance criteria and that approaches taken needed to be developed and decided on a case-by-case basis and would be influenced by several factors including but not limited to whether: (i) components are equally weighted, (ii) there is a known natural-abundance of the components, (iii) measured values for individual components are correlated or uncorrelated, etc. The Committee also noted that consideration of some of relevant information was under the remit of other committees

34. The Committee thus agreed that it would not be appropriate to develop a criteria approach for methods which use a "sum of components" but rather (i) to amend Note 2 (*Working Instructions for the Implementation of the Criteria Approach in Codex*) to improve clarity on the implementation of the criteria approach when developing numeric method performance criteria for approaches that involve a "sum of components" and (ii) to provide information to Codex committees and CCMAS on a variety of (non-exhaustive) issues they may wish to consider when developing numeric method performance for approaches that involve sum of components as well as examples of such approaches and to place this information in an Information Document.

35. The Committee made a number of adjustments to Appendix 1 of CX/MAS 17/84/4 to improve the clarity and accuracy of the information provided. The EU and its member states asked whether the Information Document could be referenced in the proposed amendment to Note 2 in the Procedural Manual. The Codex Secretariat commented that this was not possible as information documents are not formally adopted by the Commission, but they could be made available on the Codex website for consultation.

Conclusion

36. The Committee agreed:

- to forward the revised Note 2 to the *Working Instructions for the Implementation of the Criteria Approach in Codex* to the Commission for adoption and inclusion in the Procedural Manual (Appendix III); and
- to make the Information Document available on the Codex website (Appendix IV).

CRITERIA FOR ENDORSEMENT OF BIOLOGICAL METHODS USED TO DETECT CHEMICALS OF CONCERN (Agenda item 5)[7]

37. The Delegations of Chile and France, co-chairs of the EWG, presented the report of the WG (CX/MAS 17/38/5) and explained the process followed by the WG and the key outcomes; which were a modified list of biological methods (Part I) and biological methods and their validation criteria (Part II).

38. The chairs of the EWG recommended that the Committee consider the recommendations and agree on a way forward.

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5 CX/MAS 17/38/4; comments from Philippines, Kenya, EU, Mexico and Ghana (CRD 7), Senegal (CRD 14), Nigeria (CRD 15), Ecuador (CRD 17; Information document proposal by UK (CRD20).

6 REP16/MAS, paras. 62-63

7 CX/MAS 17/38/5; comments from the EU and Mexico (CRD 8), Senegal (CRD 14), Ecuador (CRD 17).

Part I

39. The Committee noted that while many currently used microbiological methods to quantify vitamins may be replaced by HPLC methods, there were still some microbiological methods considered useful for the quantification of vitamin B12, folates and pantothenic acid in foods. A list of biological methods had been prepared by the EWG with proposals for possible new methods and proposals to either retype or remove the microbiological methods.

Conclusion

40. The Committee agreed to request CCNFSDU to consider the proposed methods and whether they wished to retain the currently used microbiological methods (Appendix V) The replies from CCNFSDU would be considered by the PWG on endorsement of methods of analysis (see Agenda item 3) at CCMAS39.

Part II

41. The Committee considered whether to proceed with the development of criteria for biological methods.

42. Delegations in favour of proceeding with the work were of the opinion that not all the *General Criteria for Selection of Methods of Analysis* were applicable to biological methods; and specific criteria were needed for the review in a consistent and scientific manner of the currently endorsed biological methods in CODEX STAN 234 and for any biological methods that might be introduced in future.

43. These delegations also explained that biological methods continued to be used in their countries and that chemical methods were not always available to replace these methods.

44. Delegations opposing to proceed with further work, expressed the opinion that the *General Criteria for Selection of Methods of Analysis* in the Procedural Manual were applicable also to biological methods and therefore additional criteria were not necessary; and if numerical criteria were needed, these could be considered on a case-by-case basis.

45. These delegations further expressed the view that priority should be given to the extensive work currently being undertaken on the review and update of CODEX STAN 234, especially since biological methods were increasingly being replaced by newer chemical methods and that it was unlikely that many new biological methods would be developed in future.

Conclusion

46. The Committee agreed to continue work on biological methods criteria and to establish an EWG chaired by Chile and Mexico, working in English and Spanish:

- to use the *General Criteria for the Selection of Methods of Analysis* laid down in the Procedural Manual and other related Procedural Manual referenced documents for the validation of methods of analysis to assess methods in which potency of a substance is measured by the response of living organisms or living systems,
- to determine which criteria would not apply and propose some other criteria that might be necessary for biological methods which are currently endorsed by Codex.

47. The Committee further agreed that the work should be discontinued if the EWG does not produce a concrete result for consideration by CCMAS39.

REVIEW AND UPDATE OF METHODS IN CODEX STAN 234-1999 (Agenda Item 6)[8]

48. Brazil, Chair of the EWG and the PWG on the review and update of methods of analysis and sampling in CODEX STAN 234, presented the item and highlighted the key points of discussion and recommendations of the PWG held prior to the session (points 1-5 of CRD4).

49. The Committee considered the report of the PWG as follows:

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8 CL 2017/4-MAS; CX/MAS 17/38/6; CX/MAS 17/38/6-Add.1 (Comments of Argentina, Canada, Japan, New Zealand and Switzerland); summary report of the PWG on the review and update of methods in CODEX STAN 234-1999 (CRD4); IDF (CRD5); Kenya, Peru, EU, Mexico, Ghana and Egypt (CRD9); Senegal (CRD14); Nigeria (CRD15); Ecuador (CRD17).

Codex general methods

50. The Committee agreed that at this stage there was no need for a definition nor a separate section to list Codex general methods in CODEX STAN 234. Update of such methods would be done on a case-by-case basis by the PWG on Endorsement as work on the review progresses (including those general methods related to additives and contaminants as described in *General Methods of Analysis for Food Additives* (CODEX STAN 239-2003) and *General Methods of Analysis for Contaminants* (CODEX STAN 228-2001), respectively).

Structure of CODEX STAN 234-1999

51. The Committee agreed that new work on the standard would address the preamble, scope, structure and other relevant information aimed at facilitating the reading of the methods listed in CODEX STAN 234.

52. The Committee noted that such information did not refer to intellectual property associated to the methods in CODEX STAN 234 (e.g. performance data that may not be available or may be proprietary), but rather to complementary information such as description of CAC/RMs when no internationally validated methods from SDOs had been identified to replace these methods or performance criteria of methods as endorsed by CCMAS.

53. The Committee agreed that this work would constitute new work for approval by CAC40.

Follow-up work on the review and update of CODEX STAN 234-1999

54. The Committee agreed that it would continue to work on the workable packages for the review and update of CODEX STAN 234-1999 as described in CX/MAS 17/38/6. The workable packages will be prepared by the EWG on the review and updated of CODEX STAN 234-1999 and will be sent to the Codex Secretariat in order to be considered by the PWG on endorsement and CCMAS. Depending on the complexity of the issues associated to the workable package a circular letter (CL) could be issued by the Codex Secretariat to seek specific comments from Codex members and observer organizations.

55. The Committee recognized that the above approach would not preclude the Codex Secretariat from already proceeding with the editorial update of CODEX STAN 234 and/or commodity standards in those cases where (i) inconsistencies had been identified between the methods endorsed in CODEX STAN 234-1999 and the methods listed in the commodity standards for the same provision(s) and (ii) the inclusion of CAC/RMs that have been confirmed by CCMAS in the absence of other international references. This work will be done in close collaboration with the Chair of the EWG on the review and update of CODEX STAN 234-1999 and submitted to CCMAS for information and to CAC for adoption as editorial amendments.

56. The Committee further acknowledged that some work could already be advanced in parallel with work on the workable package by addressing methods of analysis for groups of products. This could alleviate the work envisaged on some of the workable packages and could also lead to enhance cooperation with SDOs in the review and update of the methods for other food groups.

57. The Committee agreed that the above work (including consideration of Codex general methods) may imply confirmation, removal, retyping or reassignment of the method to a specific food or group of foods.

58. The Observer of IDF in partnership with ISO and AOAC expressed their willingness to consider all the dairy-related methods as one pack and provide CCMAS with updated references for consideration by CCMAS39.

59. The Observer from AOCS referred to the discussion held at the IAM meeting (Agenda item 10) in regard to the review and update of methods of analysis and sampling plans in CODEX STAN 234-1999. The Observer conveyed the views of the SDOs that updating method references in CODEX STAN 234 should be the responsibility of each SDO to ensure that references and harmonization information are correct though this work will likely take several years. The Committee further agreed (i) to continue to work on the workable packages as well as to pilot an update of all methods related to dairy products with the assistance of IDF, ISO and AOAC and (ii) that the Codex Secretariat will closely work with the Chair of the EWG on the review and update of CODEX STAN 234 on those editorial amendments identified in paragraph 55 that can be presented for information to CCMAS39 and editorial amendments to CAC41.

Future work on database for Codex methods of analysis and sampling plans

60. The Committee noted the importance of having a searchable database with information specific to CCMAS to manage the regular review process and a general interface with information on methods of analysis and sampling adopted by CAC for Codex members and observers available on the Codex website. In the meanwhile, CCMAS can work with an informative document to track the review process.

Conclusion

61. The Committee agreed:

- To start new work on a new format for CODEX STAN 234-1999 subject to approval of CAC40 (Appendix VI).
- To continue work on the review and update of methods of analysis and sampling plans in CODEX STAN 234-1999 through the workable packages.
- To establish an EWG, chaired by Brazil and Uruguay, working in English, to carry out the work indicated in the bullet points above.
- To proceed with the review and update of methods of analysis for dairy products in CODEX STAN 234-1999 by IDF, ISO and AOAC.

INFORMATION DOCUMENT ON PRACTICAL EXAMPLES ON THE SELECTION OF APPROPRIATE SAMPLING PLANS (Agenda item 7)[9]

62. The Delegation of Germany, chair of the eWG on the development of practical examples for the selection of appropriate sampling plans, presented the paper (CX/MAS 17/38/7) and sought approval of the Committee to publish the information document. The Committee agreed on the content of the information document (Appendix VII), which will be made available on the Codex website.

PROPOSAL TO AMEND THE GUIDELINES ON MEASUREMENT UNCERTAINTY (CAC/GL 54-2004) (Agenda item 8)[10]

63. Germany, Chair of the EWG on the review of CAC/GL54, introduced the item and recalled that CCMAS37 agreed to establish an EWG to (i) identify areas for improvements and amendments to CAC/GL 54, (ii) recommend procedures if necessary for determining uncertainty of measurement results including sub-sampling, sample processing and analysis and (iii) avoid overlapping with the *Guidelines on Estimation of Uncertainty of Results* (CAC/GL 59-2006) and to proceed with work based on CRD26 presented at CCMAS37.

64. The Delegation informed the Committee on the output of the work of the EWG in order to keep CAC/GL 54 as simple as possible as follows: (i) the explanatory notes have been relieved from redundancies and are now integrated into the main texts, (ii) a new chapter with recommended procedures for determining uncertainty of measurement results has been introduced based on the document contained in CRD26, (iii) the examples have been revised to be in line with the cited standards and international guidelines, and (iv) the tables of the anticipated measurement uncertainties is now harmonized with the Procedural Manual, Section II, Chapter 1.3. Apart from these changes, all the aspects of general importance of measurement of uncertainty (MU) of CAC/GL 54 were maintained. The proposed revised CAC/GL54 with the changes indicated in points (i) – (iv) are presented in Appendix I to CX/MAS 17/38/8.

65. The Delegation also explained that the proposed introductory text in the proposed revised CAC/GL 54 was necessary to clarify why MU is important in its influence on sampling plans (i.e. on the procedure of lot assessment) and its role in conformity assessment of a particular analytical test sample. Therefore, the proposed revised CAC/GL 54 explains the influence of MU on sampling plans and the corresponding decisions of lot compliance and contain a reference to the concerning ISO standards on sampling.

66. The Delegation further clarified that MU deals with laboratory samples and not with the homogeneity of the lot (i.e. CAC/GL 54 do not address sampling uncertainties). MU of laboratory samples can however influence the sampling plans and the subsequent lot acceptance and conformity assessment of the product with the specification in the standards.

67. The Committee noted that CAC/GL54, as all Codex standards and related texts, are primarily targeted to Codex member countries and as such to any stakeholder in government (e.g. laboratories dealing with MU in the particular case of CAC/GL 54).

[9] CX/MAS 17/38/7; comments from Kenya, Mexico (CRD 10); Senegal (CRD14); Ecuador (CRD).

[10] CX/MAS 17/38/8; comments from Kenya, Peru, EU, IDF, Mexico and Ghana (CRD 11), Senegal (CRD 14), Nigeria (CRD 15), Ecuador (CRD 17).

68. The Committee noted that the proposed revision to CAC/GL 54 would envisage new work for CCMAS and that a clear outline of what the work would entail should be given in a project document for consideration by CCMAS39. Besides, the recommended procedures for estimating MU (new addition) would be better developed as an information document and that it would address examples of procedures for estimating MU. The Committee reasserted that such examples were of illustrative nature and by no means were limited to nor restricted to those to be described in the information document. The Committee also noted that the new work should focus on measurement uncertainty and not deal with sampling uncertainty.

Conclusion

69. The Committee agreed to establish an EWG chaired by Germany and working in English only with the following TOR:

- Preparation of a project document that indicates which amendments and improvements should be identified and used in GL54.
- Revision of GL54 considering the identified areas of improvement and technical and other amendments taking into account the need to simplify the content.
- Elaboration of an information document with examples of procedures for estimating measurement uncertainty.

70. The Committee further agreed that the above work will be developed on the basis of the document presented in Appendix I to CX/MAS 17/38/8.

PROPOSAL TO AMEND THE GENERAL GUIDELINES ON SAMPLING (CAC/GL 50-2004) (Agenda item 9)[11]

71. The Delegation of New Zealand, chair of the EWG, introduced the paper (CX/MAS 17/38/9) and explained that there was wide support in the EWG to undertake new work on simplifying/updating CAC/GL 50-2004.

72. The Delegation highlighted some of the general and technical areas of improvements that could be considered in the revision. Some of the improvements will be developed to assist understanding of the principles of sampling, i.e. (i) an initial section discussing the principles of acceptance sampling and how it works, and how to determine a sampling plan for a particular application; (ii) sampling of materials sold in bulk, and (iii) especially about the use of the terms ‘consumers’ risk’ and ‘producers’ risk’.

73. The Delegation further pointed out that there might be a need for assistance from outside technical experts in undertaking the work.

74. The Delegation recommended that the Committee consider the review paper and agree on a method to achieve the work, in particular its prioritisation and the means of undertaking the first priority work, whereafter a project document could be prepared.

Discussion

75. The following views were expressed:

- The current CAC/GL50 was very theoretical and needed simplification and therefore the future revision should avoid inclusion of additional theoretical information;
- The review document was a good starting point to update CAC/GL 50, but work proposed was considerable and prioritization was necessary as was the need for assistance from external experts;
- The revision of CAC/GL 50 would be extensive and it was premature to embark on the new work. An outline of the possible revised CAC/GL50 would assist in taking a decision on new work.

76. The Codex Secretariat emphasized that the revision should aim at providing a simple and understandable guidance and avoid the overuse of statistical information; that consideration should be given to cross-referencing existing guidance on sampling developed by other internationally recognized standards organisations and the use of examples within the revised document should be avoided to the extent possible.

Conclusion

77. The Committee noted that it was not in a position to request approval at this stage, and agreed to re-establish an EWG chaired by NZ, working in English, to:

- prepare a project document with a clear scope of the work to be undertaken; and

[11] CX/MAS 17/38/9; Comments from Kenya, Peru, EU and Ghana (CRD 12), Senegal (CRD 14), Nigeria (CRD 15), Ecuador (CRD 17); draft Project document prepared by NZ (CRD19).

- an outline of a new CAC/GL 50; and
- prioritization of technical and other improvements; and
- timeframes for the different phases of the work.

REPORT OF AN INTER-AGENCY MEETING ON METHODS OF ANALYSIS (Agenda item 10)[12]

78. The Observer of the American Oil Chemists' Society (AOCS), as chair of the Interagency Meeting (IAM), introduced the report of the IAM and highlighted the various issues discussed in the IAM with respect to the work of CCMAS and other related matters.

79. The Committee noted that several of the issues raised in CRD 16 had been considered under the relevant agenda items.

80. The Committee also noted that a revised version of the proposed ISO Technical Specification for the Assessment of Qualitative Methods will be circulated by ISO/TC 34/SC16 for comment shortly and the guidance document on the validation of non-targeted methods of analysis for detecting adulteration by USP/FCC are under review for publication in late 2017.

81. In relation to timely and extensive review of methods of analysis for endorsement by CCMAS, the Committee noted that IAM agreed to provide feedback to the PWG on endorsement of methods of analysis and sampling where documents are available at least 4 weeks prior to meeting of the PWG.

82. The Committee thanked the members of IAM for their contribution to the work of the Committee.

OTHER BUSINESS AND FUTURE WORK (Agenda item 11)

83. The Committee noted that no other business had been put forward during the adoption of the Provisional Agenda.

DATE AND PLACE OF NEXT SESSION (Agenda item 12)

84. The Committee was informed that the 39th Session would take place in Budapest, Hungary, within the next 18 to 24 months, the final arrangements being subject to confirmation by the host country and the Codex Secretariat.

[12] Report of the 29th IAM (CRD16).

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Appendix II

PART 1. METHODS OF ANALYSIS FOR ADOPTION BY THE 40TH CODEX ALIMENTARIUS COMMISSION

A. Committee on Processed Fruits And Vegetables

B. Coordinating Committee For Asia

C. Committee on Nutrition And Foods For Special Dietary Uses

D. Coordinating Committee For Africa

E. Committee on Spices And Culinary Herbs

F. Committee on Fats And Oils

PART 2. METHODS OF ANALYSIS FOR REVOCATION BY THE 40TH CODEX ALIMENTARIUS COMMISSION

PART 3. METHODS OF ANALYSIS ON TRANS FATTY ACIDS FOR CCNFSDU

PART 1. METHODS OF ANALYSIS FOR ADOPTION BY THE 40TH CODEX ALIMENTARIUS COMMISSION

A. COMMITTEE ON PROCESSED FRUITS AND VEGETABLES

Methods of analysis for quick frozen vegetables

Product	Provision	Method	Principle	Type
Quick frozen fruits and vegetables	Thawing procedure	Method CAC/RM 32 to be placed in CODEX STAN 234	Thawing	I
Quick frozen fruits and vegetables: Vegetables	Cooking procedure	Method CAC/RM 33 to be placed in CODEX STAN 234	Cooking	I
Quick frozen fruits and vegetables (non-glazed)	Net weight	AOAC 963.26	Weighing	I
Quick frozen peas	Solids, alcohol insoluble	Method CAC/RM 35 to be placed in CODEX STAN 234	Gravimetry	I
Quick frozen green and wax beans	Tough strings	Method CAC/RM 39 to be placed in CODEX STAN 234	Stretching	I
Quick frozen fruits and vegetables: Berries, Whole kernel corn and Corn-on-the-cob	Soluble solids, total	AOAC 932.12	Refractometry	I
Quick frozen fruits and vegetables: Berries, leek and carrot	Mineral impurities	AOAC 971.33	Gravimetry	I
Quick frozen fruits and vegetables: Peaches and berries	Drained fruit/drained berries	AOAC 953.15	Draining	I
Quick frozen spinach	Dry matter, Sodium chloride-free	Method described in CODEX STAN 77-1981 is to be moved to CODEX STAN 234	Weighing	I
Quick frozen French fried potatoes	Moisture	AOAC 984.25	Gravimetry (convection oven)	I

B. COORDINATING COMMITTEE FOR ASIA

Methods of analysis for laver products

Provision	Method	Principle	Type
Moisture content	AOAC 925.45B	Gravimetry, drying at atmospheric pressure	IV

Method of analysis for Tempe

Provisions	Method	Principle	Type
Lipid Content	AOAC 963.15	Gravimetry (Soxhlet Extraction)	I

C. COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Methods of analysis for infant formula

Provisions	Method	Principle	Type
Vitamin C	AOAC 2012.22 | ISO/DIS 20635	HPLC-UV	II
Chromium (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	II
	EN 14082	Graphite furnace atomic absorption after dry ashing	III
Molybdenum (Section B of CODEX STAN 72-1981 only)	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	II
	EN 14083	Graphite furnace AAS after pressure digestion	III
Selenium	AOAC 2011.19 | ISO 20649 | IDF 235	ICP-MS	II
	EN 14627	Hydride generation atomic absorption spectrometry (HGAAS)	III
Vitamin B12	AOAC 986.23	Turbidimetric	III
	AOAC 2011.10 | ISO 20634	HPLC	II
Myo-Inositol	AOAC 2011.18 | ISO 20637	LC-pulsed amperometry	II
Vitamin E	AOAC 2012.10 | ISO 20633	HPLC	II
Total fatty acids	AOAC 996.06	Gas chromatography	III
	AOAC 2012.13 | ISO16958 | IDF231	Gas chromatography	II

D. COORDINATING COMMITTEE FOR AFRICA

Methods of analysis for unrefined shea butter

Provision	Method	Principle	Type
Moisture content	ISO 662	Gravimetry	I
Free fatty acid content: acid value and acidity	ISO 660 AOCS Cd 3d-63	Titrimetry	I
Relative density	AOCS Cc 10c-95/ ISO 6883	Pycnometry	I
Saponification value	ISO 3657 / AOCS Cd 3d-25	Titrimetry	I
Iodine value	AOAC 993.20 / ISO 3961 / AOCS Cd 1d-92/ NMKL 39	WijsTitrimetry	I
Peroxide value	AOCS Cd 8b-90/ ISO 3960 / NMKL 158	Titrimetry	I
Unsaponifiable matter	ISO 3596 / AOCS Ca 6a-40	Gravimetry	I
Insoluble impurities content	ISO 663 / AOCS Ca 3a-46	Gravimetry	I
Melting point	ISO 6321 AOCS Cc 3b-92	Open ended capillary tube	I

E. COMMITTEE ON SPICES AND CULINARY HERBS

Methods of analysis for cumin

Provision	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total ash	ISO 928	Gravimetry	I
Acid-insoluble ash	ISO 930	Gravimetry	I
Volatile oils	ISO 6571	Distillation / Volumetric	I
Extraneous vegetable matter	ISO 927	Visual examination / Gravimetry	I
Foreign matter	ISO 927	Visual examination / Gravimetry	I
Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
Mammalian excreta	AOAC 993.27 (for ground)	Enzymatic Detection method	IV
Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV

Methods of analysis for thyme

Provision	Method	Principle	Type
Moisture	ISO 939	Distillation	I
Total ash	ISO 928	Gravimetry	I
Acid-insoluble ash	ISO 930	Gravimetry	I
Volatile oils	ISO 6571	Distillation / Volumetric	I
Extraneous vegetable matter	ISO 927	Visual examination / Gravimetry	I
Foreign matter	ISO 927	Visual examination / Gravimetry	I
Insect damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV
Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (for whole)	Visual examination	IV
	AOAC 993.27 (for ground)	Enzymatic Detection method	IV
Mould damage	Method V-8 Spices, Condiments, Flavors and Crude Drugs (Macroanalytical Procedure Manual, FDA Technical Bulletin Number 5) http://www.fda.gov/Food/FoodScienceResearch/LaboratoryMethods/ucm084394.htm#v-32	Visual examination	IV

Methods of analysis for black, white and green pepper

Provision	Method	Principle	Type
Bulk density	ISO 959-1 Annex B (black) ISO 959-2 Annex A (white)	Gravimetry	IV
Light berries	ISO 959-1 Annex A (black)	Flotation	IV
Extraneous vegetable matter	ISO 927	Visual examination / Gravimetry	I
Foreign matter	ISO 927	Visual examination / Gravimetry	I
Black berries	Physical separation and weighing ISO 959-2	Visual examination	IV
Broken berries	Physical separation and weighing ISO 959-2	Visual examination	IV
Mouldy berries	Macroanalytical procedure manual USFDA technical bulletin V.39 B	Visual examination	IV
Insect damage	Macroanalytical procedure manual USFDA technical bulletin V.39 B	Visual examination	IV
Pinheads or broken berries	Physical separation and weighing ISO959-1	Visual examination	IV
Mammalian excreta	Macroanalytical procedure manual USFDA technical bulletin V.39 B (For Pepper Whole)	Visual examination(For whole pepper)	IV
Mammalian excreta	AOAC 993.27 (for ground pepper)	Enzymatic Detection method (For ground pepper)	I
Moisture content	ISO 939	Distillation	I
Total ash	ISO 928	Gravimetry	I
Non-volatile ether extract	ISO 1108	Soxhlet extraction	I
Volatile oils	ISO 6571	Distillation	I

Provision	Method	Principle	Type
Piperine content	ISO 5564	Spectrophotometry	I
Acid- Insoluble ash	ISO 930	Gravimetry	I
Crude Fibre	ISO 5498	Gravimetry	I

F. COMMITTEE ON FATS AND OILS

Methods of analysis for fish oils

Provisions	Method	Principle	Type
P-Anisidine value	EuropeanPharmacopeia 2.5.36 / AOCS Cd 18-90 / ISO 6885	Spectrophotometry	I
Phospholipids	USP-FCC10 2S (Krill oil): Phosopholipids, Nuclear Magnetic Resonance, Appendix IIC	NMR Spectroscopy	IV
Triglycerides	USP 40-NF35 (Omega-3 Acid Triglycerides): Content of oligomers and partial glyceride;	HPLC-RI	III
	European Pharmacopoeia 1352 (Omega3 acid triglycerides): Oligomers and partial glycerides	HPLC-RI	III
	AOCS Cd 11d-96	HPLC-ELSD	III

PART 2. METHODS OF ANALYSIS FOR REVOCATION BY THE 40TH CODEX ALIMENTARIUS COMMISSION

Methods of analysis for quick frozen vegetables

CAC/RM 34 (Determination of net weight in quick frozen fruits and vegetables (non-glazed))

CAC/RM 43 (Determination of soluble solids, quick frozen fruits and vegetables; berries; total in whole kernel corn and Corn- on-the-cob)

CAC/RM 54 (Determination of mineral impurities in quick frozen fruits and vegetables: Berries, leek and carrot)

PART 3. METHODS OF ANALYSIS ON TRANS FATTY ACIDS FOR COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

Additional Information: Determination of TFA in Collaborative Studies for each method/matrix[1]

Product	Method		
	ISO 16958/IDF 231/ AOAC 2012.13 (g/100g of product)	AOCS Ce 1h-05 and AOAC 996.06	AOCS Ce 1j-07 and Ce 2b-11/Ce 2c-11 (g/100g of sample)
Dairy and ruminant products/fats	**TFA Range: 0.17–5.06 g/100 g (n=5)**: • Cheese (extracted fat), 5.06 g/100 g • Butter, 4.24 g/100 g • Cream, 1.62 g/100 g • Milk powder, 1.03 g/100 g • Liquid milk, 0.17 g/100 g	Not validated	**TFA Range: 0.32–7.27% of total fatty acids (n=5):** • Cheese powder, 7.27% • Anhydrous milk fat, 5.11% • Butter, 2.49% • Evaporated milk, 0.33% • Yogurt, 0.32%
Adult nutritionals	**TFA Range: 0.006–0.010 g/100 g (n=3):** • High protein RTF, 0.009 g/100 g • High fat RTF, 0.010 g/100 g • Milk-based powder, 0.006 g/100 g	Not validated	Not validated
Infant formula	**TFA Range: 0.010–0.073 g/100 g (n=4):** • Milk-based powder, 0.073 g/100 g • Milk-based RTF, 0.027 g/100 g • Milk-based powder, 0.012 g/100 g • Soy-based powder, 0.010 g/100 g	Samples unknown	**TFA Range: 0.15% of total fatty acids (n=1)** • DHA/EPA-fortified infant formula, 0.15%
Samples containing vegetable oils	Not validated	**TFA Range: 0.06–45.01% of total fatty acids (n=10):** • Vegetable shortening, 45.01% • Canola oil, 26.27% and 26.55% • Margarine, 11.62% • Hydrogenated lard, 1.00% • Lard, 0.90% • Sunflower oil, 0.17% • Coconut oil, 0.10% and 0.11% • Cocoa butter, 0.06%	Not validated
Samples containing marine oils or other oils with long chain polyunsaturated fatty acids	Not validated	Not validated	**TFA Range: 0.00–0.68% of total fatty acids (n=2):** • Encapsulated DHA/EPA, 0.68% • DHA/EPA-fortified orange juice, 0.00%

[1] Tyburczy et al., Anal. Bioanal. Chem. (2013), 405, 5759

Samples with unknown fat sources		Not validated	**TFA Range: 0.00–0.68% of total fatty acids (n=14):** • Tallow, 7.14% • Chocolate-cake mix, 0.90% • Whole-egg powder, 0.43% • Frozen cheese pizza, 0.37% • Extruded dog food, 0.31% • Creamy ranch-dressing, 0.24% • Potato chips, 0.22% • Peanut butter, 0.06% • Oatmeal cookie, 0.05% • Canned cat food, 0.05% • Full-fat soy flour flakes, 0.02% • Dry cereal fortified with flax, 0.00% • Horse feed, 0.00% • Gamebird feed, 0.00%

Appendix III

AMENDMENTS TO THE PROCEDURAL MANUAL

(For adoption by CAC)

(note: the amendments are in <u>bold underlined</u> font)

Principles for the Establishment of Codex Methods of Analysis

Section II: Elaboration of Codex standards and related text

Principles for the Establishment of Codex Methods of Analysis

Working Instructions for the Implementation of the Criteria Approach in Codex

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. **<u>There are numerous ways in which methods and limits that involve a sum of components can be converted into method performance criteria but this should be undertaken with care on a case-by-case basis.</u>**

Appendix IV

INFORMATION DOCUMENT ON

CRITERIA APPROACHES FOR METHODS WHICH USE A 'SUM OF COMPONENTS'

(For publication on Codex website)

INTRODUCTION

1. The Procedural Manual of the Codex Alimentarius Commission provides extensive instructions detailing how a Codex Committee may propose an appropriate method of analysis for determining the analyte and/or develop a set of criteria to which a method used for the determination must comply. In either case the specified maximum / minimum level, any other normative level or the concentration range of interest has to be stated.

2. When a Codex Committee decides that a set of criteria should be developed, in some cases the Committee may find it easier to recommend a specific method and request the Committee on Methods of Analysis and Sampling (CCMAS) to "convert" that method into appropriate criteria. The Criteria will then be considered by CCMAS for endorsement and will, after the endorsement, form part of the standard. Methods are evaluated on the characteristics of:

- Selectivity
- Accuracy
- Precision
- Limit of detection
- Sensitivity
- Practicability
- Applicability.

3. It also allows for the establishment of other criteria as required and offers some guidance on choosing between different methods.

4. The Procedural Manual allows for the "Criteria Approach" as an alternative to the endorsement of a specific method (ibid). The Criteria Approach enables the establishment of a set of criteria (numeric values) which must be met by a method in order for the method to be applicable (i.e. "fit for purpose") to a specific standard. The Criteria Approach is applicable to fully validated Type II and III methods, except for methods such as PCR and ELISA; it is not applicable to Type I methods. The Criteria Approach currently requires information on Applicability, Minimum Applicable Range, Limit of Detection and Quantitation, Precision (with requirements for reproducibility relative standard deviation), Recovery and Trueness.

5. Two approaches for establishing criteria are described in the Procedural Manual. The first utilizes the specified limit (maximum or minimum limit) to establish numeric criteria for the characteristics mentioned above and the second involves the conversion of a specific method to establish numeric criteria. Although the method should be validated and appropriate for the analyte and commodity, there is not a specific requirement that the method be endorsed prior to being "converted" to criteria.

6. The Guidelines for Establishing Numeric Values for Criteria in the Procedural Manual were developed considering only single analyte determinations and not determinations that involve a sum of components. That is, methods where the concentration of a specific analyte is measured and that determination is assessed against a specification. As such, the approach detailed in the Procedural Manual can be inappropriate for determinations that involve a sum of components i.e. where multiple analytes are determined and summed and the sum is assessed against a specification.

7. This Information Document provides information to Codex Committees and the CCMAS on a variety of (non-exhaustive) issues they may wish to consider when developing numeric method performance criteria for approaches that involve a summation of components.

BACKGROUND

8. There are numerous ways in which methods and limits that involve a sum of components can be converted into numeric method performance criteria. Two example approaches are shown in Annex A but these are not the only approaches available. Approaches taken need to be developed and decided on a case-by-case basis and will be influenced by a number of factors including whether, for example:

- the components are equally or unequally weighted;
- there is a known natural-abundance of the components (e.g. Fumonisins B1 and B2 are determined together where the typical ratio of B1:B2 in naturally contaminated samples is 5:2 but the (maximum limit) ML is a total value of B1+B2);
- measured values for individual components are correlated or uncorrelated. The presence of correlation (for example due to multiple components measured on the same instrument at the same time) can have a substantial effect on the precision of the resulting summed values compared to the precision available when components are measured independently;
- the MLs or methods involving the use of toxic equivalents (TEQs) or toxic equivalent factors (TEFs); or,
- the specification contains multiple MLs for both a single analyte and a sum of components.

9. It is unsurprising that there is currently no single mechanism for converting maximum limits that involve a sum of components into method performance criteria as it is complex. With the assessment of future methods and method developers taking into consideration a 'sum of components' approach, Codex may find future compliance less problematic. Further, as analytical technology capability improves the identification and lower quantitation of multi-components of a provision in a commodity may become feasible when historically this was not the case. Alternatively, individual components may be specified as a 'marker' for the 'total components' e.g. benzo[a]pyrene for polynuclear aromatic hydrocarbons in drinking-water. So some options in the 'sum of components' criteria applied by Codex, plus reviews by Codex Committees in cases where there is a 'sum of components' standard specification, may have to occur together to achieve the best outcome.

TOXIC EQUIVALENT FACTORS

10. For certain commodities or analytes there are specifications where the individual concentrations of multiple analytes are determined by a single method, the concentrations are converted to a "toxic equivalent" using a toxic equivalency factor (TEF) and the specification is a limit based on the sum of equivalents. One example of this approach is the determination of the saxitoxin group in the *Standard for Live and Raw Bivalve Molluscs* (CODEX STAN 292-2008). The specification is for the concentration of saxitoxin equivalents which is determined from 12 saxitoxin congeners each multiplied by a TEF and summed. TEFs are also used in other determinations, such as dioxins and dioxin-like PCBs. The current Criteria Approach in the Procedural Manual was not developed considering specifications which use TEF or a sum of toxic equivalents.

RECOMMENDATIONS

1. It is important to note that when developing a Criteria Approach, it is the competent authority (Government, Codex Committee) that is responsible for specifying the range of concentrations for each analyte. Consideration of the ratio of components, toxicity, and properties of matrices (commodities) are outside of the terms of reference of CCMAS, but rather fall under the responsibilities of Codex Commodity Committees or individual Governments.
2. There are numerous ways in which methods and limits that involve a sum of components can be converted into method performance criteria but this should be undertaken with care and also on a case-by-case basis. CCMAS is available to advise Codex Committees if they wish to develop numeric method performance criteria for methods or limits that involve a summation of components.
3. If methods of analysis that employ a summation of components have been collaboratively trialled on a 'sum of components' basis then these can be converted directly into criteria.

11. For MLs that involve use of TEQs/TEFs or other toxicological potencies it is recommended that the MLs themselves are not converted to method performance criteria. In such instances the second approach detailed within the Procedural Manual (i.e. the conversion of a specific method to establish numeric criteria) may be appropriate where numeric criteria may be developed on using untransformed method performance data (i.e. raw data that has not been converted into TEQs) assuming the method has been suitably validated. This was the approach taken when an amendment was made to the *Standard for Live and Raw Bivalve Molluscs* (CODEX STAN 292-2008) where un-weighted numerical performance criteria (i.e. TEFs not applied) were established from the various approved methods.

12. For provisions that contain MLs for both single components and also a sum of components, a combination of approaches may be appropriate. For example, using approaches laid down within the Procedural Manual for the single components and a sum of components approach for MLs that involve a summation of components.

ANNEX A - EXAMPLE APPROACHES

APPROACH 1: THE ML IS A SUM OF COMPONENTS THAT ARE EQUALLY WEIGHTED

For multi-analyte analyses where all components are weighted equal, *n* is the number of components/analytes. The criteria for multi-analyte (and single analyte, *n*=1) would then be as given in Table 1.

Table 1: Guidelines for establishing numeric criteria if the ML is a sum of components that are equally weighted.

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 the individual components[1]:	For ML/*n* ≥ 0.1 mg/kg, [ML/*n* - 3 s_R, ML + 3 s_R] For ML/*n* < 0.1 mg/kg, [ML/*n* - 2 s_R, ML + 2 s_R] NB: the upper level is above the ML for the individual components.			
Limit of Detection (LOD) for the individual components:	For ML/*n* ≥ 0.1 mg/kg, LOD ≤ ML/*n* · 1/10 For ML/*n* < 0.1 mg/kg, LOD ≤ ML/*n* · 1/5			
Limit of Quantification (LOQ) for the individual components:	For ML/*n* ≥ 0.1 mg/kg, LOQ ≤ ML/*n* · 1/5 For ML/*n* < 0.1 mg/kg, LOQ ≤ ML/*n* · 2/5			
Precision for the individual components:	For ML/*n* ≥ 0.1 mg/kg, HorRat value ≤ 2 For ML/*n* < 0.1 mg/kg, the RSD_R < [44%]. RSD_R = relative standard deviation of reproducibility.			
Recovery (R) for the individual components:	**Concentration**	**Ratio**	**Unit**	**Recovery (%)**
	100	1	100% (100 g/100g)	98-102
	≥10	10^{-1}	≥10% (10 g/100g)	98-102
	≥1	10^{-2}	≥1% (1 g/100g)	97-103
	≥0.1	10^{-3}	≥0.1% (1 mg/g)	95-103
	0.01	10^{-4}	100 mg/kg	90-107
	0.001	10^{-5}	10 mg/kg	80-110

[1] For multi-analyte analyses where all components are weighted equal, *n*=number of components/analytes.

	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. For the evaluation of trueness preferably certified reference material should be used.			

Worked Example

Substance X, consisting of 4 analytes, x_1, x_2, x_3 and x_4, in matrix Y.

The ML (i.e. $x_1 + x_2 + x_3 + x_4$) = 20 µg/kg,

As there are 4 analytes, n = 4,

ML/n = 20/4 µg/kg = 5 µg/kg

Using the NMKL Excel spreadsheet**Error! Bookmark not defined.** the following are established:

Minimum Applicable **Range for the individual components:**	0.003* - 0.029** mg/kg = 3 - 29 µg/kg *corresponding to ML/n = 5 µg/kg **corresponding to ML = 20 µg/kg
Limit of Detection (LOD) for the individual components:	1 µg/kg
Limit of Quantification (LOQ) for the individual components:	2 µg/kg
Precision for the individual components:	$RSD_R \leq 44\%$
Recovery for the individual components (R):	40-120%

Issues for consideration

1. It is important to note that throughout this approach the actual ML (for compliance purposes) remains unchanged.
2. The concept of minimum applicable range is clear and can be applied for testing compliance with a specification. However, it might be misinterpreted in cases of food contaminants where the analytical results are used for assessment of exposure to the substances analysed and consumers' risk (e.g. mycotoxins, dioxins PCBs, etc.). For this purpose, the results of measurements of low concentrations at or above the technically achievable LOQ are important. Especially for the most toxic analytes of the sum to be determined.
3. Using this approach the LOD and LOQ criteria may be too strict; especially when "n" is large (e.g. $n >> 5$). In such instances the developers of numeric method performance criteria need to consider the manner in which it considers methods that involve the summation of multiple components (e.g. sterols and PAHs) but where there is only ever likely to be a few components actually present. In such instances the calculated LOD/LOQ may be far too strict for practical purposes and an alternative approach may be more appropriate. For example, in such instances it may be appropriate for n to equal the number of analytes of 'interest' rather

than the total number of components. Alternatively, it may be appropriate to leave the individual minimum applicable range, the LODs and LOQs if already stipulated without taking into account the number of congeners or components of the sum.

APPROACH 2: THE ML IS A SUM OF COMPONENTS WHERE THERE IS A KNOWN NATURAL ABUNDANCE/RATIO OF COMPONENTS.

For multi-analyte analyses where there is a known natural abundance/ratio of components, *f* is the ratio factor. The criteria for multi-analyte (and single analyte, *f*=1) would then be as given in Table 2.

Table 2: Guidelines for establishing numeric criteria if the ML is a sum of components where there is a known natural abundance/ratio of components.

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 <u>the individual components</u>:**	For ML · *f* ≥ 0.1 mg/kg, [ML · *f* - 3 s_R , ML + 3 s_R] For ML · *f* < 0.1 mg/kg, [ML · *f* - 2 s_R , ML + 2 s_R] s_R = standard deviation of reproducibility			
Limit of Detection (LOD) for <u>the individual components</u>:	For ML · *f* ≥ 0.1 mg/kg, LOD ≤ ML · *f* · 1/10 For ML · *f* < 0.1 mg/kg, LOD ≤ ML · *f* · 1/5			
Limit of Quantification (LOQ) for <u>the individual components</u>:	For ML · *f* ≥ 0.1 mg/kg, LOQ ≤ ML · *f* · 1/5 For ML · *f* < 0.1 mg/kg, LOQ ≤ ML · *f* · 2/5			
Precision for <u>the individual components</u>:	For ML · *f* ≥ 0.1 mg/kg, HorRat value ≤ 2 For ML · *f* < 0.1 mg/kg, the RSD_R < [44%] RSD_R = relative standard deviation of reproducibility.			
Recovery (R) for <u>the individual components</u>:	**Concentration**	**Ratio**	**Unit**	**Recovery (%)**
	100	1	100% (100 g/100g)	98-102
	≥10	10^{-1}	≥10% (10 g/100g)	98-102
	≥1	10^{-2}	≥1% (1 g/100g)	97-103
	≥0.1	10^{-3}	≥0.1% (1 mg/g)	95-103
	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. For the evaluation of trueness preferably certified reference material should be used.			

Worked Example

Substance X, consisting of 2 analytes, x_1 and, x_2, in matrix Y. It is known that analytes x_1 and x_2 are typically found in a ratio of 5:3 in naturally-contaminated samples.

The ML = 5000 µg/kg,

As the 2 analytes are normally found in the ratio of 5:3

$f_1 = 5/8 = 0.625$ and,

$f_2 = 3/8 = 0.375$

For analyte x_1

$ML \cdot f_1 = 5000 \cdot 0.625$ µg/kg = 3125 µg/kg and,

For analyte x_2

$ML \cdot f_2 = 5000 \cdot 0.375$ µg/kg = 1875 µg/kg

Using the NMKL Excel spreadsheet[2] the following are established:

Analyte x_1

Minimum Applicable Range for Analyte x_1:	1.862* - 6.883** mg/kg = 1860 - 6880 µg/kg *corresponding to ML · *f* = 3125 µg/kg **corresponding to ML = 5000 µg/kg
Limit of Detection (LOD) for Analyte x_1:	313 µg/kg
Limit of Quantification (LOQ) for Analyte x_1:	625 µg/kg
Precision for Analyte x_1:	$RSD_R \leq 27\%$
Recovery (R) for Analyte x_1:	80-110%

Analyte x_2

Minimum Applicable Range for Analyte x_2:	1.056* - 6.883** mg/kg = 1060 - 6880 µg/kg *corresponding to ML · *f* = 1875 µg/kg **corresponding to ML = 5000 µg/kg
Limit of Detection (LOD) for Analyte x_2:	188 µg/kg
Limit of Quantification (LOQ) for Analyte x_2:	375 µg/kg
Precision for Analyte x_2:	$RSD_R \leq 29\%$
Recovery (R) for Analyte x_2:	80-110%

Issues for consideration

It is important to note that throughout the above process the actual ML (for compliance purposes) remains unchanged.

[2] www.nmkl.org under “How to get method criteria based on ML”

Appendix V

METHODS OF ANALYSIS FOR CONSIDERATION BY THE CODEX COMMITTEE ON NUTRITION AND FOODS FOR SPECIAL DIETARY USES

VITAMIN B3: NICOTINAMIDE

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Special foods	Nicotinamide for milk-based foods	AOAC 944.13	Microbioassay	II	Yes (III)	HPLC method like EN 15652 (Type II)

VITAMIN B3: NIACIN

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Infant formula	Niacin	AOAC 985.34 (niacin (preformed) and nicotinamide)	Microbioassay And turbidimetry	III	No	HPLC method like EN 15652 (Type II)

VITAMIN B5: PANTOTHENIC ACID

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Follow-up formula	Pantothenic acid	AOAC 992.07 Measures total pantothenate : free pantothenic acid + bounded forms	Microbioassay	II	II or III	AOAC 2012.16/ISO 20639 UHPLC MS/MS (Type I or II)

VITAMIN B6: PYRIDOXINE

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Infant formula	Vitamin B6	AOAC 985.32	Microbioassay	III	---	HPLC-Fluorescence like AOAC 2004.07 or EN 14164 (Type II)
Infant formula	Vitamin B6	CEN 14166 (Aggregates free and bound pyridoxal, pyridoxine and pyridoxine and measures as pyridoxine)	Microbioassay	III	----	HPLC – Fluorescence like AOAC 2004.07 or EN 14164 (Type II)

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Special foods	Vitamin B6	AOAC 961.15	Microbioassay	II	type III	HPLC-Fluorescence like AOAC 2004.07 or EN 14164 (Type II) and EN 14663 (includes glycosylated forms) (Free and bound hosphorylated and glycosylated forms measured as the individual forms pyridoxal, pyridoxine and pyridoxamine), HPLC fluorometric method, (Type III)

VITAMIN B12: COBALAMIN

Commodity	Provision	Method	Principle	Type	Propose to remove or change	Possible method proposed
Special foods	Vitamin B12	AOAC 952.20	Microbioassay	II	Type III	HPLC-UV AOAC 2011.10 / ISO 20634 (Type II)
Infant Milk formula	Vitamin B12	AOAC 986.23	Bioassay-Turbidimetric	II	Type III	HPLC UV AOAC 2011.10 / ISO 20634 (Type II)

VITAMIN D: ERGOCALCIFEROL (D2) & cholecalciferol (D3), OTHERS

Commodity	Provision	Method	Principle	Type	Propose to remove or Change	Possible method proposed
Special foods	Vitamin D	AOAC 936.14	Rat bioassay	IV	----	HPLC method like EN 12821(Type II)

Appendix VI

PROJECT DOCUMENT FOR NEW WORK ON THE NEW FORMAT TO CODEX STAN 234-1999

RECOMMENDED METHODS OF ANALYSIS AND SAMPLING

1. Purpose and scope of the proposed standard

The purpose of the proposed new work is to amend the Recommended Methods of Analysis and Sampling (CODEX STAN 234-1999) to the normal format for a standard, including a preamble and other relevant information, scope and use of the Standard.

2. Relevance and timeliness

The methods of analysis listed in Codex standards are primarily intended as methods for the verification of provisions in Codex standards. In this context, it is critical to keep updating the methods of analysis in a single document or a single database, which would allow a simplified and effective search for method as well as a permanent and dynamic revision system. The CCMAS supported CODEX STAN 234 as a single reference for methods of analysis and proposed that CODEX STAN 234 be amended to the normal format for a standard, i.e. to include a preamble and other relevant information as to the scope and use of the Standard. The *General Standard for Contaminants and Toxins in Food and Feed* (CODEX STAN 193-1995) or the *General Standard for Food Additives* (CODEX STAN 192-1995) could be used as examples for the amendment.

3. The main aspects to be covered

A number of changes will be considered such as the inclusion of a preamble, scope and other relevant information to the use of the standard as well as establishing a new structure with a format that allows cross references with commodities standards.

4. An assessment against the *Criteria for the Establishment of Work Priorities*

General Criterion: Consumer protection from the view of health, food safety, ensuring fair practices in food trade and taking into account the identified needs of developing countries.

The proposed work falls under the general criterion for establishment of work priorities, because the use of the Code will strengthen protection of consumers by ensuring food safety. This work also seeks to promote fair practices in food trade taking into account the identified needs of developing countries.

The proposed work is directed primarily to provide a trusted source of information regarding methods of analysis in a single document or a single database, which would allow the verification of provisions in Codex standards.

Criteria applicable to general subjects:

a) Diversification of national legislations and apparent resultant or potential impediments to international trade:

It is covered by the preceding paragraph.

b) Scope of work and establishment of priorities between the various sections of work:

See above section on purpose and scope.

c) Work already undertaken by other international organizations in this field and/or suggested by the relevant international intergovernmental body(ies):

No other similar work has been undertaken by other international organizations.

d) Amenability of the subject of the proposal to standardisation:

It is amenable to standardization since the CODEX STAN 234-1999 is already adopted, and the revisions will be simply to streamline information and make it readily available. Thus, there should be no problem with standardization.

e) Consideration of the global magnitude of the problem or issue:

It is covered by the preceding paragraph.

5. Relevance to Codex strategic objectives

The proposed work falls under 3 Codex Strategic Goals:

Strategic goal 1. Establish international food standards that address current and emerging food issues: the standard intents to verify the provisions in Codex standards.

Strategic goal 2. Ensure the application of risk analysis principles in the development of Codex standards: this work will help in risk management activities, providing a single source of methods of analysis in case of dispute and for inspection and control program.

Strategic goal 4. Implement effective and efficient work management system and practices: making readily available a single trusted source of Methods of Analysis.

6. Information on the relation between the proposal and other existing Codex documents

This standard will build on the Procedure Manual and the CODEX STAN 234-1999 Recommended Methods of Analysis and Sampling

7. Identification of any requirement for and availability of expert scientific advice

Additional scientific advice is not necessary at this moment.

8. Identification of any need for technical input to the standard from external bodies so that this can be planned for

There is no need for additional technical input from external bodies.

9. Proposed timeline for completion of the new work, including the start date, the proposed date for adoption at Step 5, and the proposed date for adoption by the Commission; the timeframe for developing a standard should normally not exceed five years

Work to start in 2018 with adoption at Step 5 and final adoption in 2020.

Appendix VII

INFORMATION DOCUMENT ON PRACTICAL EXAMPLES OF SAMPLING PLANS

(For publication on Codex website)

This Information Document provides help in choosing appropriate sampling plans. These sampling plans are examples and should not be regarded as prescriptive. Each example is one option for the particular situation. Commodity committees may find alternative plans that are more appropriate.

Therefore, they do not present fixed values but give reference to correspondent passages of the standards.

The justification of the choice ("why") of the individual sampling plans and the corresponding decision criteria ensues from the standards to be used in the individual situations. Usually the determination of the appropriate sampling plan is unambiguous, a fact, which will help avoid future conflicts between importing and exporting countries.

The given examples are intended for institutions specializing in sampling and compliance assessment. These institutions are familiar with the quoted standards (ISO, OIML, ICMSF, etc.) and should be able to understand the text in spite of the highly condensed presentation.

Sampling and decision concepts include wrong acceptance and wrong rejection of a lot, which are interrelated.

Examples of Sampling Plans:

The following Table 1 presents the matrix combinations versus measure / provision with the reference codes of the corresponding examples (Table 2). The third dimension of product form of marketing (packages/bulk material/foodstuff for consumption) is implemented into the particular examples.

Table 1: Code of Examples

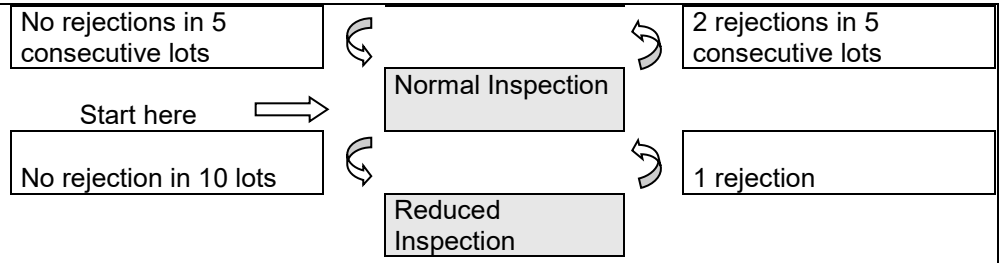
	Fruits/ vegetables	fats/oil	fish/fishery products	milk/milk products	meat/meat products	natural mineral waters	cereals
Qualitative/quantitative characteristics/sensory inspection	FV-Q	FO-Q	F-Q	MI-Q	M-Q	MW-Q	C-Q
food hygiene	FV-FH	n.r.	F-FH	MI-FH	M-FH	MW-FH	n.r.
pesticide residues	FV-P	FO-P	n.r.	MI-P	M-P	n.r.	C-P
contaminants	FV-C1/2	FO-C	F-C	MI-C	M-C	MW-C	C-C
residues of veterinary drugs	n.r.	FO-R	F-R	MI-R	M-R	n.r.	n.r.

n.r. = not relevant

Table 2: Example sampling plans

Example	Criteria	Type of Sampling Plan	Sampling and Decision Reference	
			Isolated Lots	**Continuous series of lots**
FV-Q	Visible defects in fruits	Attribute Plan Sampling uncertainty not applicable	**Consumer:** CAC/GL 50 section 3.1, see specifically ISO 2859-2:1985 Sampling: Procedure A: A plan is identified by the lot size, limiting quality (LQ) and the inspection level (unless otherwise specified, level II shall be used). The sampling size (n) is given in table A. Procedure B: A plan is identified by the lot size, limiting quality (LQ) and the inspection level (unless otherwise specified, level II shall be used). The sampling size (n) is given in table B1 to B10. Decision: For given limiting quality (LQ) and number of samples *n*, a lot is compliant if the number of items with visible defects is less than the Rejection number Re (Tables A, D4). **Producer:** ISO 2859-2:1985: Sampling: see “Consumer” Decision: For given LQ corresponding to AQL of consumer sampling plan from ISO 2859-1 if applicable, Table D5) and number of samples *n*, a lot is compliant if the number of items with visible defects does not exceed the Acceptance number Ac (Table A).	**Consumer:** CAC/GL 50 section 4.2 (table 10) see specifically: NMKL Procedure No 12, Annex – Section 4 (table 5) and Fig.1 (see below) and ISO 2859-1:1999:Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection Sampling: Normal inspection: use of a sampling plan with an acceptance criterion that has been devised to secure the producer a high probability of acceptance when the process average of the lot is better than the acceptance quality limit. Normal inspection is used when there is no reason to suspect that the process average differs from an acceptable level. The sample size is taken from Table 1 and Table 2-A. Tightened inspection: use of a sampling plan with an acceptance criterion that is tighter than that for the corresponding plan for normal inspection. Tightened inspection is invoked when the inspection results of a predetermined number of consecutive lots indicate that the process average might be poorer than the AQL. The sample size is taken from Table 1 and Table 2-B. Reduced inspection: use of a sampling plan with a sample size that is smaller than that for the corresponding plan for normal inspection and with an acceptance criterion that is comparable to that for the corresponding plan for normal inspection. The discriminatory ability under reduced inspection is less than under normal inspection.

				Reduced inspection may be invoked when the inspection results of a predetermined number of consecutive lots Indicate that the process average is better than the AQL. The sample size is taken from Table 1 and Table 2-C. Switching rules: When normal inspection is being carried out, tightened inspection shall be implemented as soon as two out of five (or fewer than five) consecutive lots have been non-acceptable on original inspection (that is, ignoring resubmitted lots or batches for this procedure). When tightened inspection is being carried out, normal inspection shall be re-instated when five consecutive lots have been considered acceptable on original inspection. The outline of the switching rules is shown in Figure 1. Decision: For given inspection level, Acceptable Quality Level (AQL) and number of samples *n*, a lot is compliant if the number of items with visible defects is less than not the Rejection number Re (Tables 1 and 2 e.g. for single sampling). **Producer:** ISO 2859-1:1999: Sampling procedures for inspection by attributes — Part 1: Sampling schemes indexed by acceptance quality limit (AQL) for lot-by-lot inspection Sampling: see “Consumer” Decision: For given inspection level and Acceptable Quality Level (AQL, a lot is compliant if the number of items with visible defects does not exceed the Acceptance number Ac (e.g. Tables 1 and 2 for single sampling).
			NMKL procedure no 12. (Annex - Section 4): Figure 1: Levels of inspection and the switching between those. Tighten Inspection	

			
MI-Q	Fat content in Milkproducts	Variables Plan Prerequisites: 1. The lots have not been screened previously for nonconforming items. 2. Continuing series of lots of discrete products all supplied by one producer using one production process 3. quality characteristic must be measurable on a continuous scale 4. the measurement error is negligible, i.e. with a standard deviation σ_μ no more than 1/10 of the sample standard deviation s or process standard deviation σ. In the case that the measurement error is significant, the sampling number n should be increased by $n^* = n(1+\gamma^2)$ where $\gamma = \sigma_\mu/\sigma$ ISO 3951-1:2013, Annex O)	**Consumer and Producer:** ISO 3951-1:2013: Sampling procedures for inspection by variables – Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL Sampling: For the “s” method acceptance sampling plan the sample standard deviation is used, for the “σ” method acceptance sampling plan the presumed value of the process standard deviation is used. If there is sufficient evidence from the control charts (e.g. ´autocontrol´) that the variability is in statistical control, consideration should be given to switching to the “σ” method. If this appears advantageous, the consistent value of s (the sample standard deviation) shall be taken as σ. Normal inspection is used at the start of inspection (unless otherwise designated) and shall continue to be used during the course of inspection until tightened inspection becomes necessary or reduced inspection is allowed. Tightened inspection shall be instituted when two lots on original normal inspection are not accepted within any five or fewer successive lots. Reduced inspection may be instituted after ten successive lots have been accepted under normal inspection, provided that these lots would have been acceptable if the AQL had been one step tighter, production is in statistical control. In case that switching rules are not applicable, a particular consumer’s risk quality (CRQ) associated with a consumer’s risk should be fixed (e.g. Table K1 or K2). In case of very short series of lots, ISO 2859-2:2010 might be applied, where the fat content of the sample items with respect to the limit (taking into account the measurement uncertainty) might be classified as attribute (see example FV-Q). Summary table 1 directs users to the paragraphs and tables concerning any situation with which they may be confronted. Sample sizes are given in table A2 for the sample size letters given in Clause 23, Chart A (for agreed and fixed AQL at 95 % probability of acceptance and LQ at 10 % probability of acceptance). This should be verified by inspecting the OC curve from among Clause 24, Charts B to R relating to this code letter and AQL. For the “s” method (CAC/GL 50 section 4.3 (table 14) and NMKL Procedure No 12, Annex – section 5 (table 6) see specifically (ISO 3951-1:2013, Clause 15), the procedure for obtaining and implementing a plan is as follows.

		5. production is stable (under statistical control) and the quality characteristic x is distributed according to a normal distribution or a close approximation to the normal distribution	a) With the inspection level given (normally this will be II) and with the lot size, obtain the sample-size code letter using Table A.1. b) For a single specification limit, enter Table B.1, B.2 or B.3 as appropriate with this code letter and the AQL, and obtain the sample size n and the acceptability constant k. For combined control of double specification limits when the sample size is 5 or more, find the appropriate acceptance curve from among Charts s-D to s-R. c) Take a random sample of size n, measure the characteristic x in each item and then calculate x, the sample mean and s, the sample standard deviation (see Annex J). Where a contract or standard defines an upper specification limit U, a lower specification limit L, or both, the lot can be judged unacceptable without even calculating s if x is outside the specification limit(s). For the "σ" method (CAC GL 50 section 4.3 (table 17) and NMKL Procedure No 12, Annex – section 5 (table 7)), see specifically (ISO 3951-1:2013, Clause 16) the procedure for obtaining and implementing a plan is as follows. a) From Table A.1 the sample-size code letter is obtained. b) Depending on the severity of inspection, enter Table C.1, C.2 or C.3 with the sample-size code letter and the specified AQL to obtain the sample size n and acceptability constant k. c) Take a random sample of this size, measure the characteristic under inspection for all items of the sample and calculate the mean value. The sample standard deviation s should also be calculated, but only for the purpose of checking the continued stability of the process standard deviation (see ISO 3951-1:2013, Clause 19). <u>Decision:</u> a lot is compliant if the average fat content of sample items does not fall below the minimum value fixed by AQL and LQ taking into account the corresponding standard deviation (s or σ) and acceptability constant K. The acceptability constant is given in tables B1 to B3 (s-method) and C1 to C3 (σ-method). If single upper or lower specification limits (U or L) are given, calculate the quality statistic $Q_U=(U-x)/s$ or $Q_L=(x-L)/s$ where x the sample mean and s, the sample standard deviation. The lot is acceptable if $Q_U \geq k$ or $Q_L \geq k$ respectively. For the "σ" method, s must be replaced by σ
FO-Q	water content in butter	Variables Plan Prerequisites: see example MI-Q	**Consumer and Producer:** see MI-Q <u>Sampling:</u> see example MI-Q <u>Decision:</u> A lot is compliant if the average water content of sample items does not exceed the maximum value fixed by AQL taking into account the corresponding standard deviation (s or σ) and acceptability constant k. See also example MI-Q

F-Q	Net weight in prepackaged fish	Special Plan	**Consumer and Producer:** OIML R 87 (Edition 2004)[b)]: Quantity of product in prepackages Sampling: see Table 1: Sampling plans for prepackages Decision: for fixed 'Risk Type' (according to fixed AQL given in OIML R 87) the lot is accepted if all of the following criteria are met: 1. The average actual quantity of product in a package is at least equal to the nominal quantity, which is evaluated in the following way: The total error of the quantity of product in a package is given by the sum of the differences between the individual product weights and the nominal weight. The average error is given by that total error divided by the sample size. The lot is accepted if the average error is a positive number. In case of a negative number, the lot is accepted if the standard deviation of the individual product weights times the sample correction factor of Table 1 is higher than the absolute value of the average error. 2. The number of packages containing an actual quantity less than the nominal quantity minus the tolerable deficiency (Table 2) is less or equal the Number of packages in a sample allowed to exceed the tolerable deficiencies (Table 1). 3. No package contains an actual quantity less than the nominal quantity minus twice the tolerable deficiency.
M-Q	Nonmeat Protein in Meat products	Variables Plan Prerequisites: see example MI-Q	**Consumer and Producer:** see MI-Q Sampling: see example MI-Q Decision: A lot is compliant if the average content of nonmeat protein of sample items does not exceed the maximum value fixed by AQL taking into account the corresponding standard deviation (s or σ) and acceptability constant k. See also example MI-Q
MW-Q	Sodium content of prepackaged Mineral Water	Variables Plan Prerequisites: see example MI-Q	**Consumer and Producer:** see MI-Q Sampling: see example MI-Q Decision: A lot is compliant if the average sodium content of sample items does not exceed the maximum value fixed by AQL taking into account the corresponding standard deviation (s or σ) and acceptability constant k. See also example MI-Q
C-Q	Moisture in rice grains	Variables Plan on Bulk Material Sampling uncertainty implemented	**Consumer and Producer:** CAC/GL 50 section 5, see specifically: ISO 10725:2000: Acceptance sampling plans and procedures for the inspection of bulk materials / ISO 11648-1:2003: Statistical aspects of sampling from bulk materials — Part 1: General principles / ISO 24333:2009 Cereals and

			cereal products -- Sampling Sampling: see example C-C Decision: for a given maximum limit, the lot is accepted if the sample grand average of these results $\bar{x}$ is lower than an upper acceptance value $\bar{x} = m_L + \gamma D$
FV-FH	*E. coli* in Frozen vegetables and fruits	Three-class attributes Plan	CAC/GL 50 section 3.2 and NMKL procedure no 12 Annex sampling plans, Section 3, Table 3 and Table 4. See specifically: ICMSF (1986)[a)]: Chapter 18 Sampling plans for vegetables, fruits, and nuts Sampling: See Table 28: Sampling plans and recommended microbiological limits for vegetables, fruits, nuts, and yeast Decision: The lot is accepted if not more than 2 items of 5 samples show the presence of *E. coli* with a concentration between 100 and 1000 CFU/g. The lot is rejected in the opposite case.
M-FH	*Staphylococcus aureus* in fresh or frozen poultry meat	Three-class attributes Plan	**Consumer and Producer:** CAC/GL 50 section 3.2 and NMKL Procedure No 12, Annex – section 3 (tables 1 and 2), see specifically: ICMSF (1986)[a)]: Chapter 13 Sampling Plans For Poultry And Poultry Products Sampling: see Table 22: Sampling plans and recommended microbiological limits for poultry and poultry products Decision: The lot is accepted if not more than 1 item of 5 samples shows the presence of *Staphylococcus aureus* with a concentration between 1000 and 10.000 CFU/g. The lot is rejected in the opposite case.
F-FH	*Listeria monocytogenes* in smoked fish – ready-to-eat	Two-class attributes Plan	**Consumer and Producer:** CAC/GL 50-2004 section 3.2 and NMKL Procedure No 12, Annex – section 3 (tables 3 and 4), see specifically CODEX STAN 311-2013 *Standard for smoked fish, smoke-flavoured fish and smoke-dried fish*, section 6.4. Sampling:See CAC/GL 61-2007 Guidelines on the application of general principles of food hygiene to the control of listeria monocytogenes in foods - Annex II Table 1 and 2 Decision: See CAC/GL 61-2007 Guidelines on the application of general principles of food hygiene to the control of listeria monocytogenes in foods - Annex III
MI-FH	*Staph. aureus* in Cheese, 'hard' and 'semi-soft' types	Two-class attributes Plan	**Consumer and Producer:** CAC/GL 50 section 3.2, see specifically: ICMSF (1986)[a)]: Chapter 15 Sampling plans for milk and milk products Sampling: see Table 24: Sampling plans and recommended microbiological limits for dried milk and cheese Decision:

			The lot is accepted if no item out of 5 samples show the presence of *Staph. aureus* in 1g, where the concentration is higher than 10.000 CFU/g. The lot is rejected in the opposite case.
MW-FH	Microorganisms in Natural Mineral Water	Two-class attributes Plan	**Consumer and Producer:** CAC/RCP 33-1985: *Code of Hygienic Practice for Collecting, Processing and Marketing of Natural Mineral Waters* (see also ICMSF (1986)[a)]: Chapter 25: Sampling plans for natural mineral waters, other bottled waters, process waters, and ice.) Sampling and Decision: Annex I: Microbiological Criteria, Table: Microbiological Criteria, Point of application: at source, during production and end product. Assuming a log normal distribution and an analytical standard deviation of 0.25 log cfu/ml, the sampling plans would provide 95% confidence that a lot of water containing a defined not acceptable geometric mean concentration of specific microorganisms would be detected and rejected based on any of five samples testing positive.
FV-P	Pesticides Residues in Apples for Compliance with MRL	Variables Plan sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL33-1999: *Recommended Methods Of Sampling For The Determination Of Pesticide Residues For Compliance With MRLS* Sampling: The minimum number of primary samples to be taken from a lot is determined from Table 1b. The primary samples must contribute sufficient material to enable all laboratory samples to be withdrawn from the bulk sample. The position from which a primary sample is taken in the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot. The primary samples should be combined and mixed well, if practicable, to form the bulk sample. The minimum size of each laboratory sample is given by Table 4, 1.2. The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. Decision: The lot complies with a MRL (Pesticide Residues in Food and Feed, Codex Pesticides Residues in Food Online Database, FAO and WHO 2013) where the MRL is not exceeded by the analytical result(s). Where results for the bulk sample exceed the MRL, a decision that the lot is non-compliant must take into account: (i) the results obtained from one or more laboratory samples, as applicable; and (ii) the accuracy and precision of analysis, as indicated by the supporting quality control data.
FO-P	Pesticides Residues in vegetable oils	Variables Plan sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL33-1999: *Recommended Methods Of Sampling For The Determination Of Pesticide Residues For Compliance With MRLS* Sampling: The minimum number of primary samples to be taken from a lot is determined from Table 1b. The primary samples must contribute sufficient material to enable all laboratory samples to be withdrawn from the bulk sample. The position from which a primary sample is taken in

			the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot. The primary samples should be packaged units, or units taken with a sampling device. They should be combined and mixed well, if practicable, to form the bulk sample. The minimum size of each laboratory sample (0.5 l or 0.5 kg) is given by Table 4, 5.4. The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. Decision: see FV-P
MI-P	Pesticides Residues in Cheeses, including processed cheeses units 0.3 kg or greater	Variables Plan sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL33-1999: *Recommended Methods Of Sampling For The Determination Of Pesticide Residues For Compliance With MRLS* Sampling: The minimum number of primary samples to be taken from a lot is determined from Table 1b. The primary samples must contribute sufficient material to enable all laboratory samples to be withdrawn from the bulk sample. The position from which a primary sample is taken in the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot. Whole unit(s) or unit(s) of the primary samples should be cut with a sampling device. Cheeses with a circular base should be sampled by making two cuts radiating from the centre. Cheeses with a rectangular base should be sampled by making two cuts parallel to the sides. The minimum size of each laboratory sample (0.5 kg) is given by Table 5, 3.3. The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. Decision: see FV-P
M-P	Fat soluble Pesticides Residues in cattle carcass for Compliance with MRL	Variables Plan Sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL33-1999: *Recommended Methods Of Sampling For The Determination Of Pesticide Residues For Compliance With MRLS* Sampling: The minimum number of primary samples to be taken from a lot is determined from Table 1a, or Table 2 (in the case of a suspect lot). The position from which a primary sample is taken in the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot. Each primary sample is considered to be a separate bulk sample. The Minimum size of each laboratory sample is given in Table 3, 2.1. The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. Decision: see FV-P

C-P	Pesticides Residues in rice grains		**Consumer and Producer:** CAC/GL33-1999: *Recommended Methods Of Sampling For The Determination Of Pesticide Residues For Compliance With MRLS* Sampling: The minimum number of primary samples to be taken from a lot is determined from Table 1b. The primary samples must contribute sufficient material to enable all laboratory samples to be withdrawn from the bulk sample. The position from which a primary sample is taken in the lot should preferably be chosen randomly but, where this is physically impractical, it should be from a random position in the accessible parts of the lot. Sampling devices required for grain are described in ISO recommendations. The primary samples should be combined and mixed well, if practicable, to form the bulk sample. The minimum size of each laboratory sample (1 kg) is given by Table 4, 2. The analytical sample should be comminuted, if appropriate, and mixed well, to enable representative analytical portions to be withdrawn. The size of the analytical portion should be determined by the analytical method and the efficiency of mixing. Decision: see FV-P
FV-C1	Aflatoxin in ready-to-eat Treenuts	Variables Plan on Bulk Material Sampling, sample preparation, and analytical variances used to compute operating characteristic curves	**Consumer and Producer:** CODEX STAN 193-1995: General Standard For Contaminants And Toxins In Food And Feed Sampling: See ANNEX 2. Each lot, which is to be examined for aflatoxin, must be sampled separately. Lots larger than 25 tonnes should be subdivided into sublots to be sampled separately. If a lot is greater than 25 tonnes, the number of sublots is equal to the lot weight in tonnes divided by 25 tonnes. It is recommended that a lot or a sublot should not exceed 25 tonnes. The minimum lot weight should be 500 kg. Representative sampling should be carried out from the same lot. In the case of *static lots* of treenuts contained either in a large single container or in many small containers, it is not ensured that the contaminated treenut kernels are uniformly dispersed throughout the lot. Therefore, it is essential that the aggregate sample be the accumulation of many small incremental samples of product selected from different locations throughout the lot. The minimum number of incremental samples, the minimum incremental sample size and the minimum aggregate sample size depend on the lot weight and are given by Table 1. In the case of *dynamic lots*, the samples are taken from a moving stream of treenuts. The size of the aggregate sample depends on the lot size, the flow rate of the moving stream and the parameters of the sampling device. Two laboratory samples each of 10kg are taken from the aggregate sample. The laboratory samples should be finely ground and mixed thoroughly. The test portions taken from the comminuted laboratory samples by a random process should be approximately 50 grams. Decision: If the aflatoxin test result is less than or equal to 10 µg/kg total aflatoxin in the test samples from both laboratory samples, the lot is accepted.

FV-C2	Total Aflatoxins in Peanuts intended for further Processing	Variables Plan on Bulk Material Sampling, sample preparation, and analytical variances used to compute operating characteristic curves	**Consumer and Producer:** CODEX STAN 193-1995: *General Standard For Contaminants And Toxins In Food And Feed* Sampling: See AFLATOXINS TOTAL, ANNEX 1: Each lot which is to be examined must be sampled separately. Large lots should be subdivided into sublots to be sampled separately. The weight or number of sublots depend on the lot size and is laid down in Table 1. The number of incremental samples to be taken depends also on the weight of the lot, with a minimum of 10 and a maximum of 100 (Table 2). For the sampling procedure see example FV-C1. The weight of the incremental samples should be approximately 200 grams or greater, depending on the total number of increments, to obtain an aggregate sample of 20 kg. The laboratory sample may be a portion of or the entire aggregate sample. If the aggregate sample is larger than 20 kg, a 20 kg laboratory sample should be removed in a random manner from the aggregate sample. A minimum test portion size of 100 g should be taken from the finely ground and mixed laboratory sample. Decision: If the aflatoxin test result is less than or equal to 15 µg/kg total aflatoxin in the test sample, the lot is accepted.
FO-C	Erucic acid in vegetable Oil (bulk)		**Consumer and Producer:** CAC/GL 50 section 5, see specifically: ISO 10725:2000: Acceptance sampling plans and procedures for the inspection of bulk materials / ISO 11648-1:2003: Statistical aspects of sampling from bulk materials — Part 1: General principles Sampling: see example C-C Decision: see example C-C for a given maximum limit m_L, the lot is accepted if the sample grand average of these results $\bar{x}$ is lower than an upper acceptance value $\bar{x} = m_L + \gamma D$.
F-C	Dioxins and dioxin like PCB´s in Fish (individual packages or units)	Variables Plan Sampling uncertainty implemented	**Consumer and Producer:** ISO 3951-1:2013: Sampling procedures for inspection by variables – Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL Sampling: Since the Dioxin content usually is not process controlled, for the “s” method (CAC/GL 50 section 4.3 (table 14) and NMKL Procedure No 12, Annex – section 5 (table 6)) see specifically (ISO 3951-1:2013, Clause 15), the procedure for obtaining and implementing a plan is as follows. a) With the inspection level given (normally this will be II) and with the lot size, obtain the sample-size code letter using Table A.1.

			b) For a single specification limit U (the ML for Dioxins and dioxin like PCB's), enter Table B.1, B.2 or B.3 as appropriate with this code letter and the (usually low) AQL, and obtain the sample size n and the acceptability constant k. c) Take a random sample of size n, measure the characteristic x in each item and then calculate $\bar{x}$, the sample mean and s, the sample standard deviation (see Annex J). Decision: calculate the quality statistic $Q_U=(U-\bar{x})/s$ The lot is acceptable if $Q_U \geq k$
MI-C	Aflatoxin M1 in Milk (bulk)		**Consumer and Producer:** CAC/GL 50 section 5, see specifically: ISO 10725:2000: Acceptance sampling plans and procedures for the inspection of bulk materials / ISO 11648-1:2003: Statistical aspects of sampling from bulk materials — Part 1: General principles CODEX STAN 193-1995: *General Standard For Contaminants And Toxins In Food And Feed* Sampling: see example C-C Decision: see example C-C for the given maximum limit m_L=0.5 μg/kg (CODEX STAN 193-1995: *General Standard for Contaminants and Toxins in Food and Feed*), the lot is accepted if the sample grand average of these results $\bar{x}$is lower than an upper acceptance value $\bar{x} = m_L + \gamma D$.
M-C	benzo(a)pyrene in meat	Variables Plan Sampling uncertainty implemented	**Consumer and Producer:** ISO 3951-1:2013: Sampling procedures for inspection by variables – Part 1: Specification for single sampling plans indexed by acceptance quality limit (AQL) for lot-by-lot inspection for a single quality characteristic and a single AQL Sampling: see Mi-Q Sample sizes are given in table A2 for the sample size letters given in Clause 23, Chart A (for agreed and fixed AQL at 95 % probability of acceptance and LQ at 10 % probability of acceptance). This should be verified by inspecting the OC curve from among Clause 24, Charts B to R relating to this code letter and AQL. 3. For the “s” method (CAC/GL 50 section 4.3 (table 14) and NMKL Procedure No 12, Annex – section 5 (table 6)) see specifically (ISO 3951-1:2013, Clause 15), The procedure for obtaining and implementing a plan is as follows. a) With the inspection level given (normally this will be II) and with the lot size, obtain the sample-size code letter using Table A.1. b) Enter Table B.1, B.2 or B.3 as appropriate with this code letter and the AQL, and obtain the sample size n and the acceptability constant k.

			c) Take a random sample of size n, measure the characteristic x in each item and then calculate $\bar{x}$, the sample mean and s, the sample standard deviation (see Annex J). Where a contract or standard defines an upper specification limit U, the lot can be judged unacceptable without even calculating s if $\bar{x}$ exceeds the specification limit. For the "σ" method (CAC GL 50 section 4.3 (table 17) and NMKL Procedure No 12, Annex – section 5 (table 7)), see specifically (ISO 3951-1:2013, Clause 16) the procedure for obtaining and implementing a plan is as follows. 4. 5. a) From Table A.1 the sample-size code letter is obtained. 6. 7. b) Depending on the severity of inspection, enter Table C.1, C.2 or C.3 with the sample-size code letter and the specified AQL to obtain the sample size n and acceptability constant k. 8. c) Take a random sample of this size, measure the characteristic under inspection for all items of the sample and calculate the mean value. The sample standard deviation s should also be calculated, but only for the purpose of checking the continued stability of the process standard deviation (see ISO 3951-1:2013, Clause 19). <u>Decision:</u> calculate the quality statistic $Q_U=(U-\bar{x})/s$ The lot is acceptable if $Q_U \geq k$ For the "σ" method, s must be replaced by σ
MW-C	Arsenic in Natural Mineral Water	Variables Plan on Bulk Material Sampling uncertainty implemented	**Consumer and Producer:** CAC/GL 50 section 5, see specifically: ISO 10725:2000: Acceptance sampling plans and procedures for the inspection of bulk materials / ISO 11648-1:2003: Statistical aspects of sampling from bulk materials — Part 1: General principles CODEX STAN 193-1995: *General Standard For Contaminants And Toxins In Food And Feed* <u>Sampling:</u> see example C-C <u>Decision:</u> see example C-C for the given maximum limit m_L=0.01 mg/kg (CODEX STAN 193-1995: *General Standard for Contaminants and Toxins in Food and Feed*), the lot is accepted if the sample grand average of these results $\bar{x}$is lower than an upper acceptance value $\bar{x} = m_L + \gamma D$.
C-C	Cadmium content in wheat	Variables Plan on Bulk Material Sampling uncertainty implemented	**Consumer and Producer:** CAC/GL 50 section 5, see specifically: ISO 10725:2000: Acceptance sampling plans and procedures for the inspection of bulk materials / ISO 11648-1:2003: Statistical aspects of sampling from bulk materials — Part 1: General principles/ ISO 24333:2009 Cereals and

			cereal products -- Sampling Sampling: sampling from a commodity is classified into two different procedural types: • sampling of bulk materials for the accurate estimation of an average value of the quality characteristic assessed in the lot by suppliers • inspection procedure for bulk materials for making a decision concerning lot acceptance by consumers. ISO 11648 is an International Standard for the first type of procedure, ISO 10725 for the second type, which is based on the assumption that the value of the individual standard deviation of the specified quality characteristic is known and stable. The sample size can be estimated using Tables 3 - 22 of the standard ISO 10725:2000 with fixed producer's risk α and consumer's risk α and fixed cost ratio level from the relative standard deviations $d_I = \sigma_I/D$ and $d_T = \sigma_T/D$ (ISO 10725:2000, 6.3.4) with the sampling increment standard deviation σ_I and test sample standard deviation σ_T. The number $2n_I$ increment samples should be taken from the lot and each two of them should be pooled to two composite samples. From each of the two composite samples $2n_T$ test samples should be prepared (e.g. homogenized). For imprecise standard deviations, one measurement per test sample should be performed (ISO 10725:2000, 6.3.2.2). As an alternative, the number and size of the increment samples and of the test samples are given in ISO 24333 Table 1 or Table 2 for flowing or static bulk material respectively. That standard also gives information on suitable sampling devices. Decision: As emphasized above, prerequisite is the determination of the estimation standard deviation σ_E (ISO 10725:2000, 6.2.7 / ISO 11648-1:2003) by monitoring of the cadmium content and to assess that it is stable. It is permitted to use the values of standard deviations specified by an agreement between the supplier and the purchaser (e.g. ´autocontrol´) (ISO 10725:2000, 6.2.1). Taking into account the discrimination interval $D = (K_\alpha + K_\beta)\,\sigma_E$ (formula C6 in C.4.2) and assuming that the measurement standard deviation is negligible compared to σ_E (which should be proven), the following four quantities might be fixed by agreement: the acceptance quality limit for the lot mean m_A (corresponding to AQL, producers' risk), the probability α of wrongly rejecting a conforming lot, the non-acceptance quality limit for the lot mean m_R (corresponding to LQ, consumers' risk), and the probability α of wrongly accepting a nonconforming lot. For a given acceptance quality limit m_A, the lot is accepted if the sample grand average of these results $\bar{x}$ is lower than an upper acceptance value $\bar{x} = m_A + \gamma D$ with the constant for obtaining the acceptance value $\gamma = K_\alpha/(K_\alpha + K_\beta)$.
FO-R	Residues of Veterinary Drugs in Fat	Variables Plan sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL71-2009: *Guidelines For The Design And Implementation Of National Regulatory Food Safety Assurance Programme Associated With The Use Of Veterinary Drugs In Food*

			Producing Animals Sampling: See example F-R, The minimum quantity required for laboratory samples is 500 g (Table A II Group 031). Decision: see example F-R
F-R	Residues of Veterinary Drugs in Packaged Fish	Variables Plan Sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL71-2009: *Guidelines For The Design And Implementation Of National Regulatory Food Safety Assurance Programme Associated With The Use Of Veterinary Drugs In Food Producing Animals* Sampling: For non-suspect lots a statistically-based, unbiased sampling program is recommended (sampling is conducted at random throughout the lot under inspection, although often systematic sampling is employed). In stratified random sampling the consignment is divided into non-overlapping groups or strata e.g. geographical origin, time. A sample is taken from each stratum. In systematic sampling units are selected from the population at a regular interval (e.g., once an hour, every other lot, etc.). Where non-compliant results are detected it is possible to derive a crude estimate of the likely prevalence in the general product population (e.g. ´autocontrol´). The number of primary samples required to give a required statistical assurance can be read from Appendix A, Table 4. For exact or alternative probabilities to detect a non-compliant residue, or for a different incidence of non-compliance, the number of samples n to be taken may be calculated from: $n = \ln(1-p) / \ln(1-i)$ Where p is the probability to detect a non-compliant residue (e.g. 0.95), it is the supposed incidence of non-compliant residues (e.g. 0.10) in the lot. In biased or estimated worst case sampling, investigators use their judgment and experience regarding the population, lot, or sampling frame to decide which primary samples to select. Such directed or targeted sampling protocols on a sub-population (biased sampling) are designed to place a greater intensity of inspection/audit on suppliers or product considered to possibly have a greater potential than the general population of being non-compliant. If compliant results from biased sampling confirm non-biased program results, they provide increased assurance that the system is working effectively. The canned or packaged product should not be opened for sampling unless the unit size is at least twice the amount required for the final laboratory sample. The final laboratory sample should contain a representative portion of juices surrounding the product. The minimum quantity required for laboratory samples is 500 g of edible tissue (Table C VII Class B – Type 08, A). Decision: For purposes of control, the maximum residue limit for veterinary drugs (MRLVD) is applied to the residue concentration found in each laboratory sample taken from a lot. Lot compliance with a MRLVD is achieved when the mean result for analysis of the laboratory test portions does not indicate the presence of a residue, which exceeds the MRLVD. Regulatory action is only taken on samples containing residues, which can be demonstrated to exceed the regulatory action limit with a defined statistical confidence.

Mi-R	Residues of Veterinary Drugs in Raw Milk	Variables Plan on Bulk Material Sampling uncertainty not applicable	**Consumer and Producer:** CAC/GL71-2009: *Guidelines For The Design And Implementation Of National Regulatory Food Safety Assurance Programme Associated With The Use Of Veterinary Drugs In Food Producing Animals* Sampling: See example F-R, The minimum quantity required for laboratory samples is 500 mL (Table B I Group 033). Decision: See example F-R
M-R	Residues of Veterinary Drugs in Meat/Meat products	Variables Plan sampling uncertainty not applicable	Consumer and Producer: CAC/GL71-2009: *Guidelines For The Design And Implementation Of National Regulatory Food Safety Assurance Programme Associated With The Use Of Veterinary Drugs In Food Producing Animals* Sampling: See example F-R, The minimum quantity required for laboratory samples is 500 g (Table A I Group 030). Decision: See example F-R

[a)] Microorganisms in Foods 2. Sampling for microbiological analysis: Principles and specific applications. 1986. 2nd Ed. International Commission on Microbiological Specifications for Foods.

[b)] International Organization of Legal Metrology (OIML), Bureau International de Métrologie Légale 11, rue Turgot - 75009 Paris - France, Publication OIML R 87 Edition 2004 (E)

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item 3 – AOAC OMB Awards**

OMB has four awards for which candidates will be discussed. There are two (2) team awards and two (2) individual awards.

The awards are:

1. Award in Recognition of Technical and Scientific Excellence (team award)
2. Expert Review Panel of the Year (team award)
3. Technical Service Award (individual award)
4. Method of the Year (individual award)

Nominations for these awards are determined as follows:

1. Nominations for the Recognition award come from the Official Methods Board itself and will be discussed during the meeting.
2. Nominations for the Expert Review Panel of the Year are based on ERPs that have met over the past three (3) years (2014-2016).
3. Nominations for the Technical Service Award are based on solicitations to the leadership of stakeholder panels, ERPs, technical and advisory committees, Communities, and AOAC Sections.
4. Nominations for the Method of the Year are based on the First Action or Final Action methods reviewed during the past three (3) years (2014-2016). Information for Method of the Year award nominations was sent out via email on Monday, June 18, 2017.

Enclosures:

1. AOAC Awards Policy and Procedures Document
2. Eligible ERPs
3. Eligible and nominated candidates for Technical Service Award
4. Listing of Eligible methods

***OFFICIAL METHODS*[SM] PROGRAM AWARDS**

Contents

AWARD IN RECOGNITION OF TECHNICAL AND SCIENTIFIC EXCELLENCE

Selection Criteria

The purpose of this award is for the Official Methods Board (OMB) to recognize a team, stakeholder panel or working group that has published a major document or other body of work that demonstrates a unique or particularly noteworthy level of technical and scientific expertise.

The minimum criteria for selection are:

a. The body of work includes major initiatives or technical guidelines accepted, completed or published within the last three years.
b. The team has been instrumental in developing or modifying technical guidelines or method validation processes.
c. The team product demonstrates significant merit as to the scope of the project, the involvement of a diverse and/or international group of stakeholders or an innovative approach to difficult analytical challenges.
d. The award recognizes teamwork that enhances the reputation of the Association and fosters the mission of AOAC INTERNATIONAL.

Selection Process:

a. The chair of the OMB solicits the OMB members for nominees.
b. Written recommendations and supporting information will be submitted to the OMB chair. The information will be distributed to the members of the OMB.
c. The OMB selects the recipient of this award. The winner is selected by a 2/3 vote. If necessary, the OMB chair may cast the tie-breaking vote.

Award

An appropriate letter of appreciation and thanks will be sent to the recipient(s) of this award. The winner will be announced at the appropriate session of the AOAC INTERNATIONAL annual meeting, with presentation of an award. All members participating in the winning team will be acknowledged at the annual meeting, receive an award and a letter of appreciation. The name of the winner, with supporting story, will be carried in the announcement in the *ILM*.

EXPERT REVIEW PANEL OF THE YEAR

Selection Criteria

The minimum criteria for selection are:

a. The expert review panel must have completed a significant milestone (e.g. First Action Method, Final Action Method, method modification) within the last three years.
b. Generally, some unique or particularly noteworthy aspect of the ERP's work is highlighted as making the ERP worthy of the award, such as innovative technology or application, breadth of applicability, critical need, difficult analysis, or timeliness.
c. The panel report demonstrates significant merit as to the scope of the project, the involvement of a diverse and/or international group of recognized experts or an innovative approach to difficult analytical challenge.

Selection Process:

a. AOAC staff lists all eligible panels for consideration and forwards that list along with the ERP report to the Chair of the Official Methods Board (OMB).
b. The OMB Chair forwards the list along with any supporting information to the OMB.
c. The OMB selects the Expert Review Panel of the Year. Winner is selected by a 2/3 vote. If necessary, the OMB chair may cast tie-breaking vote.

Award

An appropriate letter of appreciation and thanks will be sent to the members of the winning Expert Review Panel. The winning panel will be announced at the appropriate session of the AOAC INTERNATIONAL annual meeting, with presentation of an award. All panelists participating in the winning panel will be acknowledged at the annual meeting, receive an award and a letter of appreciation. The name of the winning ERP, with supporting story, will be carried in the announcement in the *ILM*.

TECHNICAL SERVICE AWARD

More than one volunteer may be selected in this category each year. In each case the area of expertise should be noted at the time of presentation of the award.

Selection Criteria includes:

a. Has demonstrated timely, competent, and continuous service in an exemplary manner to a Stakeholder Panel (SP), Expert Review Panel (ERP), Working Group (WG), Section, Community, and Committee and/or to the Official Methods Board (OMB).
b. Has donated this service within the three years prior to nomination.
c. Gives outstanding expert guidance and support in all technical aspects as needed and requested.

Additional support for selection is exemplary performance in one or more of the areas below:

a. Has provided guidance on safety, statistical, technical matters, or process expertise.
b. Has been instrumental in developing, modifying or validating a high quality method for publication in the Official Methods of Analysis.
c. Communicates related activities through the appropriate channels, either through the panel/group/community chairs, the Committee on Statistics or Safety or through the Chief Scientific Officer or other staff designees.
d. Contributes significantly to AOAC INTERNATIONAL over a period of years with other accomplishments related to his/her area of expertise (e.g symposium presentations, poster presentations, publications, workshops, meetings).
e. Contributes to the development and improvement of AOAC INTERNATIONAL guidelines, OMA methods, statistics or safety programs.
f. Helps guide AOAC in the decision-making process to make the organization a leader in the field of analytical science.

Selection Process

a. The Official Method Board (OMB) will solicit the Chairs of the Stakeholder Panels, Expert Review Panels, Working Groups, Committees, Community, and the Association membership for nominees. Recommendations based on input from anyone qualified to discuss the contribution of the nominee can be submitted.
b. Written recommendations and supporting information must be submitted to the OMB Chair. The OMB chair will distribute the information to the members of the OMB.
c. The OMB selects the winner(s) of the Technical Service Award by a 2/3 vote. If necessary, the OMB chair may cast tie-breaking vote.

Award

An appropriate letter of appreciation and thanks will be sent to the recipient(s) of this award. The winner will be announced at the appropriate session of the AOAC INTERNATIONAL annual meeting, with presentation of an award. The recipient(s) will be acknowledged at the annual meeting, receive an award and a letter of appreciation. The name of the winner, with supporting story, will be carried in the announcement in the *ILM*.

METHOD OF THE YEAR

OMB may select more than one method in this category each year.

Selection Criteria

The minimum criteria for selection are:

a. The method must have been approved for first or final action within the last three years.
b. Generally, some unique or particularly noteworthy aspect of the method is highlighted as making it worthy of the award, such as innovative technology or application, breadth of applicability, critical need, difficult analysis, and/or range of collaborators.
c. The method demonstrates significant merit in scope or is an innovative approach to an analytical problem.

Selection Process:

a. AOAC staff lists all eligible methods for consideration and forwards that list with supporting documentation (e.g. ERP chair recommendation(s)) to the Chair of the Official Methods Board (OMB).
b. The Chair forwards the list along with any supporting information to the members of the OMB.
c. The OMB selects the Method of the Year. The winner is selected by 2/3 vote. If necessary, the OMB chair may cast tie-breaking vote.

Award

An appropriate letter of appreciation and thanks will be sent to the author(s) of the winning method. The corresponding author will be announced at the appropriate session of the AOAC INTERNATIONAL annual meeting, with presentation of an award. All authors will be acknowledged at the annual meeting, will receive an award and a letter of appreciation. The name of the winner(s), with supporting story, will be carried in the announcement in the *ILM*.

2014 – 2016 AOAC EXPERT REVIEW PANEL PROFILES

ERP Profile Summaries Only

AOAC SPIFAN Expert Review Panels

AOAC ERP for SPIFAN Nutrient Methods **(2015 ERP of the Year)**

AOAC ERP for SPIFAN Pesticide Contaminants **(2016 ERP of the Year)**

AOAC ERP for SPIFAN Whey Protein

AOAC SPDS Expert Review Panels

AOAC ERP for Dietary Supplement Methods – Chondroitin, PDE5 Inhibitors, & Anthocyanins

AOAC ERP for Dietary Supplement Methods - Ashwagandha, Folin C, & Kratom

AOAC ERP for Dietary Supplement Methods – Aloin & Tea

AOAC ERP for Dietary Supplement Methods – Lutein & Turmeric

AOAC SPSFAM Expert Review Panels

AOAC ERP for Ethanol in Kombucha Methods

AOAC ERP for SPSFAM Heavy Metal Methods

AOAC ERP for SPSFAM Select Food Allergen Methods

AOAC Research Institute OMA Expert Review Panels

AOAC ERP for Dietary Starch Methods

AOAC ERP for Fertilizer Analysis Methods

AOAC ERP for Food Allergens – Gluten

AOAC ERP for Microbiology Methods for Foods and Environmental Surfaces **(2014 ERP of the Year)**

AOAC ERP for PAH Methods

AOAC ERP for Pesticide Residue Methods

AOAC ERP for Veterinary Drug Residue Methods **(2015 ERP of the Year)**

2017 NOMINEES FOR TECHNICAL SERVICE AWARD

2016 OMB TECHNICAL SERVICE AWARD NOMINEES	Nominee #1	Nominee #2
Name of Nominee	Yvonne Salfinger	Brendon Gill
Nominee's Affiliation	American Ppublic Health Laboatories / Association of Food and Drug Officials	Fonterre Cooperative Group
Nominee's Title		
Nominee's Email Address	yhale@aol.com	Bredon.Gill@Fonterra.com
a. Has the nominee provided guidance on the development of standards and methods in addition to safety, statistical, technical matters, or process expertise?	YES	YES
b. Has the nominee been instrumental in developing, modifying standards or validating a high quality method(s) for publication in the Official Methods of Analysis?	YES	YES

2016 OMB TECHNICAL SERVICE AWARD NOMINEES	Nominee #1	Nominee #2
c. Has the nominee communicated related activities through the appropriate channels, either through the stakeholder panel/working groups/expert review panels/community chairs, the Committee on Statistics or Safety or through the Chief Scientific Officer or other staff designees?	YES	YES
d. Has the nominee contributed significantly to AOAC INTERNATIONAL over a period of years with other accomplishments related to his/her area of expertise (e.g symposium presentations, poster presentations, publications, workshops, meetings)?	YES	YES
e. Has the nominee contributed to the development and improvement of AOAC INTERNATIONAL standards, guidelines, OMA methods, statistics or safety programs?	YES	YES
f. Has the nominee helped to guide AOAC in the decision-making process to make the organization a leader in voluntary consensus standards development and analytical science?	YES	YES

2017 NOMINEES FOR TECHNICAL SERVICE AWARD

2016 OMB TECHNICAL SERVICE AWARD NOMINEES	Nominee #1	Nominee #2
Written recommendations and supporting information must be submitted. Please provide a recommendation in support of your nomination. Also, if you answered "YES" to any of the above, please provide details in support of your nomination and recommendation.	Yvonne is a most worthy nominee for the AOAC OMB Technical Service Award. She has chaired the Committee on Safety since 2013 and served the Micro ERP as the Safety Advisor during the same period. Her technical assessments of the methods reviewed by the ERP have been extremely thorough and went beyond just the safety aspects. Her input was always timely and insightful, helping the ERP achieve successful resolution to the methods submitted for 1st and Final Action.	Brendon has been an invaluable member of the AOAC SPIFAN ERP. He always performs very thorough method reviews and he consistently provides the ERP with valuable comments and guidance with these methods. Brendon bring to AOAC a combination of scientific expertise and industry experience, and he uses both of these in making significant contributions to the SPIFAN ERP. In addition to participating the the SPIFAN ERP, Brendon has lead two successful MLT studies for SPIFAN; one for a method measuring
Name & Title of Nominator	Michael Brodsky & Wendy McMahon	Darryl Sullivan
Nominator's Affiliation	Brodsky Consultants & Merieux MutriSciences	Covance Laboratories

SPDS METHODS REVIEWED IN 2014 – 2016

AOAC 2016.09	Aloin A, Aloin B, and Aloe emodin in Raw Materials and Finished Products
AOAC 2016.10	Theanine in Tea Dietary Ingredients and Supplements
AOAC 2016.16	Curcuminoids in Turmeric Roots and Supplements
AOAC 2015.11	Chondroitin Sulfate Content in Raw Materials and Dietary Supplements
AOAC 2015.12	Phosphodiesterase Type 5 Inhibitors in Dietary Ingredients and Supplements
AOAC 2015.17	Estimation of Withanolides in *Withania somnifera*

SPIFAN METHODS REVIEWED 2014 - 2016

AOAC 2016.02 Biotin by Liquid Chromatography Coupled with Immunoaffinity Column Cleanup Extraction

AOAC 2016.03 Chloride in Milk, Milk Powder, Whey Powder, Infant Formula, and Adult Nutritionals by Potentiometric Titration

AOAC 2016.05 Vitamins D_2 and D_3 in Milk Powders, Infant Formulas, and Adult Nutritionals by LC-MS/MS

AOAC 2016.06 Fructans in Infant and Adult/Pediatric Nutritional Formula by High-Performance Anion-Exchange Chromatography with Pulsed Amperometric Detection

AOAC 2016.11 Biotin in Infant, Pediatric, and Adult Nutritionals by HPLC and Fluorescence Detection

AOAC 2016.13 Lutein and Beta-Carotene in Infant Formula and Adult Nutritionals, Reversed-Phase Ultra-High-Performance Liquid Chromatography

AOAC 2016.14 Fructans in Infant Formula and Adult Nutritionals

AOAC 2015.06 Minerals and Trace Elements in Infant Formula and Adult Nutritionals

AOAC 2015.07 Chloride in Infant Formula

AOAC 2015.08 Chloride in Infant Formula and Adult/Pediatric Nutritional Formula

AOAC 2015.09 Vitamin K_1 in Infant, Pediatric, and Adult Nutritionals

AOAC 2015.10 Choline and Carnitine in Infant Formula and Adult Nutritionals

AOAC 2015.14 Vitamins B_1, B_2, and B_6 in Infant Formula and Related Nutritionals

AOAC 2014.02 Vitamin B_{12} in Infant Formula and Adult/Pediatric Formulas

AOAC 2014.04 Choline/Carnitine in Infant Formulas and Adult Nutritional Products

AOAC 2011.10 Vitamin B_{12} in Infant Formula and Adult/Pediatric Formula
(ERP Chair Nomination for 2016 & 2017 Method of the Year)

AOAC 2011.18 Myo-Inositol in Infant Formula and Adult/Pediatric Formula

AOAC 2011.19 Cr, Mo, and Se in Infant Formula and Adult/Pediatric Formula

AOAC 2011.20 Nucleotides in Infant Formula and Adult/Pediatric Formula

AOAC 2012.10 Vitamin A & Vitamin E in Infant Formula and Adult/Pediatric Formula

AOAC 2012.13 Labeled Fatty Acid Content in Infant Formula and Adult/Pediatric Formula

AOAC 2012.15 Total Iodine in Infant Formula and Adult/Pediatric Nutritional Formula
(ERP Chair Nomination for 2016 & 2017 Method of the Year)

AOAC 2012.16 Pantothenic acid in Infant Formula and Adult/Pediatric Formula

AOAC 2012.22 Vitamin C in Infant Formula and Adult/Pediatric Formula

AOAC 2016.15 Quantification of Whey Protein Content in Infant Formulas by Sodium Dodecyl Sulfate-Capillary Gel Electrophoresis

AOAC 2015.02 Sodium Monofluoroacetate in Dairy Powders

AOAC 2015.03 Sodium Fluoroacetate in Infant Formula

AOAC 2015.04 Monofluoroacetate in Powdered Nutritional Products

SPSFAM METHODS REVIEWED IN 2014 - 2016

AOAC 2016.12 Ethanol in Kombucha
AOAC 2016.04 Four Arsenic Species in Fruit Juice
AOAC 2015.01 Heavy Metals in Food

RI CHEMICAL CONTAMINANTS SOLE-SOURCE OR PROPRIETARY METHODS REVIEWED IN 2014 - 2016

AOAC 2014.08 Multiclass Pesticide Residues in Tea **(2015 Method of the Year)**

AOAC 2014.09 Polycyclic Aromatic Hydrocarbons (PAH) in Seafood **(2015 Method of the Year)**

AOAC 2012.25 Triphenylmethane Dyes and Their Metabolites in Aquaculture Products **(2016 Method of the Year)**

RI FEEDS & FERTILIZERS - SOLE-SOURCE OR COMMERCIAL/PROPRIETARY METHODS REVIEWED IN 2014 – 2016

AOAC 2015.18	Phosphorus and Potassium in Commercial Inorganic Fertilizers
AOAC 2015.15	Nitrogen, Phosphorus, and Potassium Release Patters in Controlled-Release Fertilizers
AOAC 2014.10	Dietary Starches in Animal Feeds and Pet Food

RI GLUTEN - SOLE-SOURCE OR PROPRIETARY METHODS REVIEWED IN 2014 – 2016

AOAC 2015.05 Partially Hydrolyzed Gluten in Fermented Cereal-Based Products

AOAC 2015.16 Gluten in Processed and Nonprocessed Corn

AOAC 2014.03 Gluten in Rice Flour & Baked Rice Products

AOAC 2012.01 Gliadin as a Measure of Gluten in Corn and Rice Products

RI MICROBIOLOGY SOLE-SOURCE OR PROPRIETARY METHODS REVIEWED IN 2014 – 2016

AOAC 2016.01 *Salmonella* spp. in Select Foods and Environmental Surfaces

AOAC 2016.07 *Listeria* in Select Foods and Environmental Surfaces

AOAC 2016.08 *Listeria monocytogenes* in Variety of Foods and Select Environmental Surfaces

AOAC 2015.13 Enumeration of Aerobic Bacteria in Food

AOAC 2014.01 *Salmonella* in Selected Foods

AOAC 2014.05 Enumeration of Yeast and Mold in Food

AOAC 2014.06 *Listeria* species in Selected Foods and Environmental Surfaces

AOAC 2014.07 *Listeria monocytogenes* in Selected Foods and Environmental Surfaces

AOAC 2013.01 *Salmonella* in a Variety of Foods and Environmental Surfaces

AOAC 2013.02 *Salmonella* species in a Variety of Foods

AOAC 2013.09 *Salmonella* in Selected Foods

AOAC 2013.10 *Listeria* in a Variety of Foods and Environmental Surfaces **(2014 Multi-Laboratory Study of the Year – Microbiology – join with 2013.11)**

AOAC 2013.11 *Listeria monocytogenes* in Variety of Foods **(2014 Multi-Laboratory Study of the Year – Microbiology – join with 2013.10)**

AOAC 2013.14 Identification of *Salmonella* spp. From Colony Picks **(2014 Award in Recognition of Technical and Scientific Excellence for co-authors)**

AOAC 2012.02 Gram Positive Microbial Identification ***(ERP Chair Nomination for 2017 Method of the Year)***

AOAC INTERNATIONAL
The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item 4 – ERP Final Action Method Recommendations**

BACKGROUND

There are two (2) methods that have been recommended by ERPs for Final Action *Official Methods*^{SM} status.

AOAC 2016.02 Biotin in Infant Formula and Adult/Pediatric Formula
AOAC 2016.05 Vitamin D in Infant Formula and Adult/Pediatric Formula

ATTACHMENTS

ERP recommendations

ERP SUMMARY FOR FIRST TO FINAL ACTION METHOD RECOMMENDATION

AOAC 2016.02	Determination of biotin by liquid chromatography coupled with immunoaffinity column clean-up extraction

GUIDANCE FOR AOAC ERPS - APPENDIX G[1]	Considered?	Comments/Reference if applicable
Method Applicability	Yes	Meets SMPR
ERP First Action to Final Action recommendations & improvements	Yes	
Draft Final Action method reviewed by ERP	Yes	Completed in June 2017
Safety Concerns	Yes	
Reference Materials	Yes	NIST 1849a and SPIFAN Materials
Single Laboratory Validation	Yes	Joseph et al.: Journal AOAC Int Vol. 99, 4 (2016) p.1110
Reproducibility/Uncertainty and Probability of Detection	Yes	In publication, pending review
Comparison to SMPR (SMPR criteria met?)	Yes	Meets AOAC SMPR 2014.005
Feedback from Users of Method	Yes	All Addressed

DOCUMENTATION	Available?	Comments
Safety Evaluation	Yes	
Reference Materials	Yes	NIST 1849a and SPIFAN Materials
SLV or PTM	Yes	Joseph et al.: Journal AOAC Int Vol. 99, 4 (2016) p.1110
Approved Validation Protocols	Yes	OMA Appendix L
Statistics Review	Yes	
Method Published in OMA	Yes	
Method Performance vs SMPR criteria	Yes	Meets AOAC SMPR 2014.005
Feedback Information	Yes	All addressed
Additional Recognition(s)	Yes	Method is a working item in ISO
ERP Reports	Yes	3-16-2016; 9-20-2016; 3-16-2017
Manuscript(s) Published in JAOAC	Yes	Joseph et al.: Journal AOAC Int Vol. 99, 4 (2016) p.1110 (SLV); MLT publication in process
ERP Method Recommendation (Final Action/Repeal/Continuation)		

[1] *Official Methods of Analysis of AOAC INTERNATIONAL*, Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis, p.3 "*First Action to Final Action Methods: Guidance for AOAC Expert Review Panels.*"

Determination of total biotin by liquid chromatography coupled with immunoaffinity column clean-up extraction: Multi Laboratory Testing, Final Action 2016.02

First Action 2016
Final Action 2017

George Joseph and Ranjani Devi
AsureQuality Ltd, PO Box 41, Shortland Street, Auckland 1140, New Zealand

Elaine C. Marley and David Leeman
R-Biopharm Rhône Ltd, West of Scotland Science Park, Glasgow, Scotland G20 0XA

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A collaborative study was carried out on AOAC First Action Official Method 2016.02: Determination of total biotin by liquid chromatography coupled with immunoaffinity column clean-up extraction. Subsequent to the successful method optimisation and analysis practice samples by 12 laboratories covering 10 different countries, 9 laboratories completed the analysis 12 pairs of blind duplicates before the due date of the study. Carefully selected SPIFAN matrices were used for the multi laboratory testing.

The sample is dispersed in phosphate buffered saline (PBS) and autoclaved at 121±2^{0}C for 25 minutes. The sample is cooled to room temperature and then diluted to 100mL in a volumetric flask. The extract is centrifuged and filtered using a Whatman glass microfiber filter paper (GE Healthcare Life Sciences, Buckinghamshire, UK). Clear filtrate is collected for clean-up and extraction. Biotin immunoaffinity column is mounted onto a SPE manifold. A disposable syringe barrel is connected to the immunoaffinity column as a reservoir. The buffer in the affinity column is drained and the sample filtrate is loaded through the reservoir and allowed to flow through by gravity. The column is washed with PBS followed by water. Air is passed through the column to remove residual liquid.

Biotin and Biocytin from the column is eluted with methanol and collected in a reacti-vial (Cat. No. 13223, Thermo Scientific). The eluent is evaporated to dryness using a heating block set at 85±5°C under a gentle stream of nitrogen and the sample is re-constituted in 1mL of water. The biotin / biocytin in the reconstituted sample are analysed simultaneously by HPLC using a PDA set at 200nm. Identification of peaks is based on absolute retention time. Quantification was by multipoint external calibration using peak area responses of the analytes. Spectrum scan (200 nm to 350 nm) can be used for the purity and identity confirmation as required.

Biotin / Vitamin H / Vitamin B_7 is a B group vitamin involved in the production of energy and functions as a coenzyme in bicarbonate-dependent carboxylation reactions. Biotin exists as free biotin and in protein-bound forms in foods. Signs of biotin deficiency are quite evident in humans who consume raw egg white over long periods. Raw egg white contains avidin, a protein that has shown to bind biotin in the small intestine and prevent its absorption. Clinical findings of biotin deficiency include dermatitis, conjunctivitis, alopecia and central nervous system abnormalities.

Biotin (Hexahydro-2-oxo-1H-thieno[3,4-d]imidazol-4-pentanoic acid; CAS 58-85-5) is the fusion of an imidazolidone ring with a tetrahydrothiophen group linked to a valeric acid side chain. The chemical formula of biotin is $C_{10}H_{16}N_2O_3S$ with a molecular weight of 244.31.

Biocytin (N6-[5-[(3aS,4S,6aR)-Hexahydro-2-oxo¬-1H-thieno[3,4-d]imidazol-4-yl]-1-oxopentyl]-L-lysine; CAS 576-19-2) is a bound form of biotin (the linked molecule is lysine) is also biologically active in human system. The chemical formula of biocytin is $C_{16}H_{28}N_4O_4S$ with a molecular weight of 372.48.

Naturally active forms of biotin in foodstuffs are d-biotin and d-biocytin though the fortified form is exclusively d-biotin. These two forms should therefore be determined in order to estimate the total biotin nutritional value.

In view of the absence of a standard analytical procedure for total biotin, the biotin working group of SPIFAN activity mooted by AOAC International has developed a Standard Method Performance Requirements (SMPR 2014.005) in search for a candidate method to be considered as a global dispute resolution standard.

The AsureQuality Auckland Laboratory has developed a method to facilitate a specific, precise, accurate and robust procedure for the analysis of biotin and biocytin from Infant Formula and Adult / Pediatric Nutritional Formulas (1-8). The method meets the SMPR and also has an assured limit of quantification of 0.1µg/100g (1ppb) based on a simple mathematical relationship between lowest standard and dilution. The method involves immunoaffinity column (R-Biopharm Rhone, EASI-EXTRACT biotin column or equivalent) clean-up and extraction followed by LC - UV set at 200nm. The laboratory has carried out extensive Single Laboratory Validation (SLV) study following SPIFAN SLV guidelines (Appendix L: AOAC International) using SPIFAN kit and NIST SRM 1849a. The SLV along with the method was reviewed by the AOAC SPIFAN expert review panel (ERP) in 2016 and approved for First Action Official Method of Analysis (OMA) 2016.02.

Collaborative Study (Multi Laboratory Testing)

The objective of the Multi Laboratory Testing (MLT) study was to establish the precision and accuracy of the method in various laboratories to demonstrate the suitability of the method as an international reference method with endorsement as AOAC Final Action, and possible adoption by ISO and Codex.

The study was chaired by Dr George Joseph from AsureQuality New Zealand and is monitored by AOAC International (SPIFAN) Official Method Board through its delegated personnel. The study is divided in two parts: method set up and qualification of participants (Part 1) and multi laboratory testing by the qualified participants (Part 2).

Extensive campaign was carried out through AOAC and ISO/IDF meetings / contacts to recruit potential laboratories for the MLT. Formal invitation has been sent to more than 30 laboratories around the world by the study director and 15 laboratories agreed to participate. Delivery of SPIFAN matrices to a laboratory in Thailand was failed / cancelled due to issues with the recipient's / country's import clearance requirements. Two laboratories could not progress at all with the MLT within the time frame due to inadequate resources or alternative problems. The details of the remaining 12 laboratories from 10 different countries participated in the MLT study (Table 2016.02A)

Table 2016.02A: MLT Participants

Lab #	Name of the laboratories	Physical Address	Country
1	Abbott Laboratories	3300 Stelzer Road, Columbus, Ohio 43219	United States
2	Aquanal Laboratoire Aquitaine Analyses	151 bis, Avenue Jean Jaures, Pessac 33600	France
3	AsureQuality New Zealand Limited	131 Boundary Road, Auckland 0600	New Zealand
4	AsureQuality Singapore Pte.	29 Tai Seng Avenue 534119	Singapore
5	DTS Food Laboratories	71 Boundary Road, Melbourne VIC 3051	Australia
6	FirstSource Laboratory Solutions LLP	IDA, Nacharam Cross Rd, Hyderabad-500076	India
7	Fonterra Co-operative Group Limited	Cnr No.1 Rd & SH26, Waitoa 3341	New Zealand
8	Merieux NutriSciences	3600 Eagle Nest Dr, Crete IL 60417	United States
9	R-Biopharm Rhône Ltd	45 Acre Road, Glasgow G20 0XA	Scotland
10	Mead Johnson, China	Xia Yuan Rd, Dongji ID, Guangzhou, 510730	P.R. China
11	Mead Johnson, Netherlands	Middenkampweg2, Nijmegen, 6545CJ	Netherlands
12	Nestle Research Centre, Switzerland	Case Postale 44, Lausanne 26, CH-1000	Switzerland

The study was carried out using SPIFAN II matrices, which represent most of the products in the scope of the project (Infant Formula and Adult Nutritionals made from any combination of milk, soy, rice, whey, hydrolysed protein, starch and amino acids, with and without intact protein). Fourteen different product types were provided to each laboratory which includes two practice samples and 12 samples for multi laboratory testing over two separate days. Two split samples of each matrix were included in the package as blind coded duplicates to analyse on the same day. The sample details are summarised in Table 2016.02B

Table 2016.02B: AOAC SPIFAN Matrices

Samples	Product Description	Batch / Lot #	Blind Duplicate Codes	
Practice 1	Infant Formula Powder Partially Hydrolysed Soy Based	410457651Z	SWUO667	SWUO667
Practice 2	Infant Formula Powder FOS/GOS Based	50350017W1	URTF231	URTF231
MLT - 1	Infant Formula Powder Partially Hydrolysed Milk Based	410057652Z	KDOX966	ATAN351
MLT - 2	Infant Elemental Powder	00795RF	ECHL425	UOPM297
MLT - 3	Infant Formula RTF Milk Based	EV4H2R	XKIP216	HYJU890
MLT - 4	Adult Nutritional RTF High Fat	00729RF00	DYLB360	ZMQM883
MLT - 5	Infant Formula Powder Milk Based	4044755861	NSRB999	JSDT587
MLT - 6	Infant Formula Powder Soy Based	E10NWZC	TJHR217	OACN211
MLT - 7	NIST SRM 1849a	CLC10-b	KGSZ273	LTCT316
MLT - 8	Adult Nutritional Powder Low Fat	00859RF00	LYNY751	PZGP859
MLT - 9	Child Formula Powder	00866RF00	RQXQ518	GVPE615
MLT - 10	Toddler Formula Powder Milk-Based	4052755861	EFXN778	BFA0941
MLT - 11	Infant Formula Powder Milk Based	K16NTAV	CULF358	GBZC169
MLT - 12	Adult Nutritional RTF High Protein	00730RF00	FPTE312	DOMY545

Homogeneity assessment of AOAC SPIFAN product matrices were performed by analysing several active nutritional ingredients. The testing was performed by Covance Laboratories Inc. in one of their facilities. The homogeneity report was provided to the study director which is carefully evaluated before commencing the MLT programme.

The participating laboratories were requested to analyse two practice samples in duplicate using the first action method provided and report the results to the study director. It was communicated to the laboratories that any deviation, such as necessity to substitute reagents, columns, apparatus or instruments, must be duly recorded and reported. Electronic templates were provided to the participants for data reporting. Moreover raw data was requested wherever necessary. After review, the study director identified the laboratories which have the capability to run the analysis successfully. The study director also had discussion with those laboratories produced unacceptable data to see any technical reasons which can be resolved within reasonable time frame to include them in the second part of the study.

The qualified laboratories were then asked to analyse the MLT samples on two different days following a carefully designed protocol provided. The results were submitted to the study director for evaluation.

Unless otherwise specified in the protocol, all powdered samples were analysed on a reconstituted basis, using 25 grams of sample into 225 grams with water, as stated in the method. An electronic template was provided for data reporting including system suitability, linearity, peak areas of the standard curve as well as of the sample extracts. Furthermore, detailed information on the different weights and volumes used during sample preparation as indicated in the method, as well as raw data (chromatograms of standards and samples) were requested. Laboratories are asked to report final biotin and biocytin results in µg/100 g to two decimal places.

After data collection, outliers were detected using Cochran and Grubbs tests. The number and coded identity of statistical outlier laboratories is included in the final report. Average biotin concentrations, standard deviations of repeatability (Sr) and relative standard deviations of repeatability (RSDr) were estimated from blind duplicates in MLT samples. The blind coded duplicates were analysed on the same day. Standard deviations of reproducibility (SR), relative standard deviations of reproducibility (RSDR), and HorRat values (RSDR/predicted RSDR) were also determined.

The analytical method provided to the laboratories for the MLT is the same as it is published in Journal of AOAC Volume 99, Number 4, 2016, pages 1110 to 1112 which is codified as First Action AOAC 2016.02. Extensive details of the sample preparation, chromatography, calculation, reporting criteria were specified in the method and the protocol provided to the laboratories. None of the laboratories that participated in the MLT recorded any modifications or deviations from the documented procedure. This information was requested to assess the suitability of the method for further approval as Final Action AOAC method.

AOAC Official Method 2016.02

Determination of total biotin by liquid chromatography coupled with immunoaffinity column clean-up extraction

First Action 2016
Final Action 2017

Applicable to the determination of total biotin in all forms of infant, adult, and/or pediatric formula (powders, ready-to-feed liquids and liquid concentrates)

Caution: Refer safety data sheets for all chemicals prior to use. Ensure all appropriate personal protective equipment and follow good laboratory practices.

A. Principle / Methodology

The sample is dispersed in phosphate buffered saline (PBS) and autoclaved at 121±2^{0}C for 25 minutes. The sample is cooled to room temperature and then diluted to 100mL in a volumetric flask. The extract is centrifuged and filtered using a Whatman glass microfiber filter paper (GE Healthcare Life Sciences, Buckinghamshire, UK). Clear filtrate is collected for clean-up and extraction. Biotin immunoaffinity column is mounted onto a SPE manifold. A disposable syringe barrel is connected to the immunoaffinity column as a reservoir. The buffer in the affinity column is drained and the sample filtrate is loaded through the reservoir and allowed to flow through by gravity. The column is washed with PBS followed by water. Air is passed through the column to remove residual liquid.

Biotin / Biocytin from the column is eluted with methanol and collected in a reacti-vial (Cat. No. 13223, Thermo Scientific). The eluent is evaporated to dryness using a heating block set at 85±5°C under a gentle stream of nitrogen and the sample is re-constituted in 1mL of water. The biotin / biocytin in the reconstituted sample are analysed simultaneously by HPLC using a PDA set at 200nm. Identification of peaks is based on absolute retention time. Quantification was by multipoint external calibration using peak area responses of the analytes. Spectrum scan (200 nm to 350 nm) can be used for the purity and identity confirmation as required.

B. Chemicals

1. Laboratory Reagent Grade Water
2. Sodium Dihydrogen Phosphate Dihydrate
3. Disodium Hydrogen Phosphate Dihydrate
4. Sodium Hydroxide
5. Methanol, HPLC grade
6. Acetonitrile, HPLC grade
7. Ortho-phosphoric acid, 85%
8. PBS - pH 7.4 (Cat. No. 10010031 Life Technologies / Thermo Scientific or equivalent)
9. Biotin - Purity ≥ 99% (Cat. No. B4501 Sigma Chemical Co., St. Louis, MO, USA, or equivalent)
10. Biocytin - Purity ≥ 98% (Cat. No. B4261 Sigma Chemical Co., St. Louis, MO, USA,or equivalent)

C. Reagents

(a) ***Sodium Hydroxide*****, 2M. -** Weigh 80g of sodium hydroxide in a 1L volumetric flask, then dissolve in water and make up to the mark.

(b) ***Sodium Phosphate Buffer*****, 0.15 M. -** Weigh 9.15g of sodium dihydrogen phosphate dihydrate and 16.31g of disodium hydrogen phosphate dihydrate in a 1L volumetric flask, then dissolve in water and make up to the mark. Adjust the pH to 7 with 2M sodium hydroxide.

(c) ***Phosphoric acid*****, 0.1%. -** In a 1L volumetric flask, add 500 mL water. Add 1.2 mL of ortho-phosphoric acid. Mix and make up to the mark with water.

D. Apparatus

(a) Whatman Glass Microfiber Filters. - CAT No. 1820-125.
(b) R-Biopharm Rhone Easy Extract Biotin Immunoaffinity Column Pack. - P82/P82B or equivalent.
(c) SPE Manifold. - With accessories.
(d) Autoclave. - Set at 121°C.
(e) Centrifuge. - Variable speed.
(f) Analytical Balance. - 4 dp.
(g) Amber glass screw-cap bottle. - 100mL.
(h) Horizontal shaker.
(i) Volumetric flasks. - 1L and 250mL, 100mL and 10mL.
(j) Pipettors. - Calibrated, 10.0mL, 5.0mL, 1.0mL and 200µL, 100µL and 50µL.
(k) Measuring cylinder. - 100mL, 50mL.
(l) Reacti-Vials
(m) Reacti-therm heating block. - With nitrogen blow down (Thermo Scientific).
(n) Ultrasonic bath. - Set at 50ºC.
(o) Centrifuge tubes. - 50mL.
(p) Vortex mixer.
(q) Syringe filter. - PTFE 0.45µm (Cat. No. 13HP045AN; Advatec Syringe Filters, Cole Parmer, Vernon Hills, IL, USA).
(r) Disposable syringes. - 10mL and 1mL.
(s) HPLC vials. - 2mL with 200µL glass inserts.

E. Sample Preparation

Note: For weight and loading volumes for the different ranges of product, see Table 2016.02A. Slurry may be used wherever product heterogeneity is expected.

For the slurry, reconstitute the 25 g powder with warm water (~50°C) to a total weight of 200 g. Mix thoroughly on a horizontal shaker for 15 min and then sonicate at 50°C for 10 min. Cool to room temperature. For liquid samples, mix well to ensure homogeneity of the sample portion and weigh the specified quantity.

(a) Weigh sample/slurry into a 100 mL amber glass screw-cap bottle. See Table 2016.02A.
(b) Add 0.15 M sodium phosphate buffer to a volume of 50 mL.
(c) Swirl gently to mix.
(d) Autoclave the sample preparation at 121°C for 25 min.
(e) Cool the sample to room temperature. Quantitatively transfer the extracts into a 100 mL volumetric flask and make up to the mark with 0.15 M sodium phosphate buffer, mixing well.
(f) Transfer extracts into centrifuge tubes and centrifuge the samples at 4000 rpm for 15 min.
(g) Filter the samples using Whatman glass microfiber filter paper and collect the filtrate.
(h) Set up the SPE manifold. Attach the immunoaffinity column connected to a 10 mL reservoir. Drain off buffer just above the gel.
(i) Load the sample filtrate onto the column as per Table 2016.02A and initialize the flow with the help of a vacuum pump.
(j) Let the solution pass through the column by gravity at a rate of one drop per second.
(k) Wash the column by passing 10 mL PBS through the column, followed by 10 mL water (initialize the flow with the help of vacuum at every step and leave it for gravity).
(l) Remove any residual liquid from the column by introducing gentle vacuum.
(m) Introduce a Reacti-Vial and elute the analyte under gravity with 2 mL methanol. Elute further with an additional 1 mL methanol. Backflush at least three times when eluting and this can be achieved by gentle up and down motion of the syringe plunger to maximize the elution.

(n) Evaporate the eluent to dryness using a heating block set at 85 ± 5°C, under a gentle nitrogen blow down.

(o) Cool down to room temperature by keeping it outside for about 15 min

(p) Redissolve with 1 mL water and then cap the Reacti-Vials and vortex for 30 s. Filter by using a syringe filter in a clean glass insert for the HPLC analysis

Table 2016.02A. Sample Preparation

Product (µg/100g)		Sample Preparation				Conc (µg/100mL)	
Min	Max	Weight (g)	Volume (mL)	Load (mL)	Final	Min	Max
0.1	0.5	20	100	50	1 mL	1	5
0.5	1.0	10	100	20	1 mL	1	2
1.0	5.0	10	100	10	1 mL	1	5
5.0	50.0	2.0 (Slurry 16g)	100	10	1 mL	1	10
50.0	100.0	1.0 (Slurry 8g)	100	10	1 mL	5	10
100.0	400.0	0.5 (Slurry 4g)	100	5	1 mL	2.5	10

F. Standard Preparation

(a) Stock Standard Biotin (100 µg/mL).—Weigh 25 mg biotin reference material in a 250 mL amber volumetric flask. Add 150 mL water and sonicate at room temperature for 90 min with occasional shaking. Make up to volume with water.

(b) Stock Standard Biocytin (100 µg/mL).—Weigh 10 mg biotin reference material in a 100 mL amber volumetric flask. Add 60 mL water and sonicate at room temperature for 90 min with occasional shaking. Make up to volume with water.

(c) Mixed intermediate standard (100 µg/100 mL).—Dilute 1 mL each of stock standards to 100 mL with water.

(1) Standard 1 (1.0 µg/100 mL).—Dilute 100 µL mixed intermediate standard to 10 mL with water.

(2) Standard 2 (2.5 µg/100 mL).—Dilute 250 µL mixed intermediate standard to 10 mL with water.

(3) Standard 3 (5.0 µg/100 mL).—Dilute 500 µL mixed intermediate standard to 10 mL with water.

(4) Standard 4 (7.5 µg/100 mL).—Dilute 750 µL mixed intermediate standard to 10 mL with water.

(5) Standard 5 (10 µg/100 mL).—Dilute 1 mL mixed intermediate standard to 10 mL with water.

(6) Standard 6 (20 µg/100 mL).—Dilute 2 mL mixed intermediate standard to 10 mL with water

Note: The concentrations given above are indicative only; calculate the actual concentrations of biotin and biocytin in each calibration standards using the following formula.

Biotin / Biocytin (µg/100mL) = (W1 x P x 10 x Vis) ÷ (V x 10)

W1= Weight of biotin or biocytin (mg)
P = Percentage purity from the certificate of analysis
Vis = Volume of mixed intermediate standard used for the calibration standard (mL)
V = Volume of stock standard (250mL for biotin and 100mL for biocytin)

G. Chromatographic Conditions

(a) Mobile phase A. - 0.1% Phosphoric acid
(b) Mobile phase B. - 100% Acetonitrile
(c) Mobile phase C. - 80% Acetonitrile
(d) Column: Kinetex Phenyl-Hexyl (Cat. No. 00F-4495-E0; Phenomenex, Torrance, CA, USA), 150 × 4.6 mm × 2.6 µm × 100 Å.
(e) Column temperature. - 25±2ºC
(f) Retention times. - Biocytin 4.5 to 5.5 minutes and biotin 16 to 17 minutes
(g) Run time. - 27 minutes
(h) Detector. – Photodiode array detector operating at 200 nm (spectrum scan 200–350 nm)
(i) Injection volume. - 100µL

For gradient program see Table 2016.02B.

Table 2016.02B. Gradient Program

Time (min)	Flow Rate (mL/min)	Mobile Phase A (%)	Mobile Phase B (%)	Mobile Phase C (%)
0.0	0.6	90	10	0
18.0	0.6	90	10	0
18.5	0.8	0	0	100
24.0	0.8	0	0	100
24.5	0.6	90	10	0
27.0	0.6	90	10	0

H. Quality Control

(a) Check system suitability by injecting Standard 3 five times. RSD should be ≤2%.
(b) Run the calibration standards at the beginning and end of the sequence (slope drift ≤2%).
(c) The six-point calibration should give a correlation coefficient ≥0.997.
(d) Test one in five samples in duplicate. The duplicates should be within the method repeatability.
(e) Inject one of the calibration standards after every five sample injections.
(f) Analyze a reference sample (e.g., National Institute of Standards and Technology Standard Reference Material 1849a) in duplicate.
(g) Identification of biotin peak is based on absolute retention time. Spectrum scan can be used for peak purity confirmation if required.
(h) Perform three high level recoveries with every batch of immuno affinity columns

I. Calculation and Reporting

The chromatography software will automatically calculate the concentration of the sample in µg/100g, provided the concentration of the standards in (µg/100mL), sample weight (g) and dilution are entered correctly.

Manual calculation can also be performed by using the following equation:

$$\text{Biotin or Biocytin (µg/100g)} = \frac{\text{(Sample Area x Dilution)}}{\text{(Slope x Sample weight in grams)}}$$

Dilution = 10 (100 x 1 ÷ 10) Sample made up to 100mL, 10mL used for IAC clean-up to a final volume of 1mL with water for HPLC analysis. The dilution will be 20 if 5mL is used for IAC clean-up.

Slope = Valid slope calculation based on concentration on X-axis and peak area on Y-axis

Sample weight: Calculate powder equivalent in grams using the following equation for reconstituted powder samples.

Sample weight (powder equivalent) in grams = (W1 x W3) / W2

For ready to feed liquid samples, the sample weight used for extraction is used for the calculation.

Report results to three significant figures, using microgram-per-100-gram units or convert to other units as required.

J. Repeatability

The difference between the results of duplicate portions of the same sample tested at the same sequence should not exceed 6% of the mean result.

K. Reproducibility

The difference between the results of duplicate determinations tested on different days should not exceed 12% of the mean result

L. Uncertainty of Measurement

Uncertainty of the method was calculated as 7%, using appropriate statistical procedure (square root of the sum of squares of the errors expressed as a percentage).

M. Limit of Quantitation

The LOQ was calculated based on the lowest working standard and dilution factor,

Limit of Quantitation (LOQ) was calculated based on the lowest working standard and the dilution factor.

LOQ = (1 x 100) / (20 x 50) = 0.1µg/100g (1ppb)

1 = 1µg/100mL lowest standard
100 = Volume (mL)
20 = 20g sample
50 = Volume (mL) loaded on immunoaffinity column
1= Final volume (mL)

RESULTS AND DISCUSSION

The laboratories were requested to analyse biotin and biocytin in the samples, however none of the samples were found to have biocytin and only biotin results were reported and evaluated in the study. All the biotin results presented in the report are expressed as *µg/100g* of sample as received basis from AOAC International.

Three of the twelve laboratories (Labs 10, 11 and 12) could only report the results of practice samples prior to the due date of the study, 27 Jan 2017. In general, the results from Lab 7 is showing low bias which was investigated further. The possible reasons for the low bias could be the practice followed in sample preparation especially the steps related to loading of immunoaffinity column and the elution technique where triple backflush is specified. The investigation from Lab 7 reported that the analysts did not follow the backflush technique effectively and that may have caused the incomplete elution of biotin from the cartridges. The laboratory also reported that they had deviated from the protocol in reconstitution of powder samples by taking 10g of powder in 80mL. The results from Labs 7, 10, 11 and 12 are included in the MLT report and for statistical evaluation of the results to calculate precision of the analytical method.

Linearity and Range

The retention time of biocytin ranges from 3.3 minutes to 6.1 minutes and that of biotin from 13.8 min to 22.5 minutes. The retention times reported from Lab 9 stands out from other laboratories though the chromatographic conditions were followed as documented. The variation is likely due to difference in the hardware configuration of the liquid chromatographs used for the analysis. The information was not available from Lab 10 at the time of the data processing.

All the participants used more or less the same calibration range 1 to 20 µg/100mL for biocytin and biotin as specified in the method. The range showed excellent correlation coefficient among the participants of not less than 0.999, confirming the linearity of the method over the calibration range (Table 2016.02C).

Table 2016.02C: Retention times and correlation coefficients

Lab #	Retention Time (Min)		Correlation Coefficient (r^2)	
	Biocytin	Biotin	Biocytin	Biotin
1	4.6	16.1	0.9981	0.9995
2	3.8	15.9	0.9996	0.9999
3	4.9	16.2	0.9987	0.9993
4	5.9	18.1	0.9983	0.9997
5	4.4	15.5	0.9997	0.9993
6	4.9	16.5	0.9995	0.9995
7	3.3	14.5	0.9990	0.9990
8	4.1	13.8	0.9991	0.9973
9	6.1	22.5	0.9998	0.9995
11	4.9	15.1	0.9995	0.9988
12	4.1	12.9	0.9990	0.9993

MLT Results and Statistical Evaluation

The biotin results from practice and MLT samples (Day 1 and 2) are given in Tables 2016.02D, E and F respectively. Fourteen different product types were analysed in duplicate by nine laboratories which includes two practice samples and 12 samples for MLT over two separate days. The practice samples were analysed in duplicates and the blind coded duplicates were analysed during MLT on the same day by the participants. Laboratories 10, 11 and 12 submitted results for practice samples only.

The results are closely comparable between the duplicates and among all the participants and all the data have been used for statistical evaluation to calculate repeatability, reproducibility and HorRat. The statistical analysis of the data was carried out using *International Study Workbook Version 2.1 for Blind (Unpaired) Replicates* from AOAC International. The data as presented in the report as *µg/100g* were used as such for statistical calculation with a Factor for Units of Measurement of 1.00E-08. The summary of relative standard deviation of repeatability (RSD_r) and reproducibility (RSD_R) along with HorRat performance ratios is tabulated in Table 2016.02G.

Relative Standard Deviation of Repeatability (RSD_r)

Repeatability expresses the precision under the same operating conditions (intra-assay) over a short interval of time. The AOAC SPIFAN SMPR 2014.005 for the biotin analysis specifies a maximum repeatability standard deviation of not more than 6% for biotin levels greater than 1 µg/100g. The mean RSD_r of the matrices analysed in the study is 4.6% and is well within the limit of the SMPR. However MLT samples 5 and 9 recorded slightly higher RSD_r of 6.79% and 7.03% respectively. The duplicate results from Lab 8 are causing the outlier for MLT sample 5 and removing the data from the lab bring the RSD_r down to 5.68%. Similarly, the removal of duplicate data from Lab 9 will bring the RSD_r of MLT sample 9 down to 5.81%. The removal of these values still leaves statistically significant duplicate sets (8 sets) for a valid RSD_r calculation. However the values are kept in the report as the overall repeatability meets the SMPR criteria and the SLV demonstrated the repeatability precision before the present study.

Relative Standard Deviation of Reproducibility (RSD_R)

Reproducibility expresses the precision among the laboratories. The relative standard deviation of reproducibility (RSD_R) exceeds the SMPR criteria of not more than 12% for all the 14 matrices analysed in the study confirming the precision of the analytical method. The maximum RSD_R noticed was 9.5% in the MLT sample 1, which is partially hydrolysed milk based infant formula matrix. The reproducibility precision confirms the suitability of the SPIFAN method AOAC 2016.02 as a strong candidate as a reference method for global dispute resolution hence the inter-laboratory variation is minimal and exceeds the expectation of the SMPR limits.

Horwitz Ratio (HorRat)

The Horwitz ratio (HorRat) is a normalized performance parameter indicating the acceptability of methods of analysis with respect to among-laboratory precision (reproducibility). It is the ratio of the observed relative standard deviation among laboratories calculated from the actual performance data, RSD_R, to the corresponding predicted relative standard deviation ($PRSD_R$) calculated from the Horwitz equation. The formula for the Horwitz ratio as presented in the *International Study Workbook* of AOAC International is applicable only when the concentration is in the unit/unit form (e.g., µg/µg or g/g, etc.). When the analyte concentration is a mass fraction amount as µg/100g, the appropriate factor for unit of measurement should be selected to generate correct HorRat values. The factor selected in the study for the calculation is 1.00E-08.

Under reproducibility conditions, the acceptable HorRat value range is 0.5 to 2 as per AOAC International. The HorRat values obtained in this study were within 0.3 to 0.5 in all SPIFAN matrices except the NIST SRM 1849a. The performance requirements of SPIFAN methods are generally tighter than routine analytical methods and therefore $PRSD_R$ used in the formula may be slightly higher pushing the HorRat values down. Nevertheless, the lower Horwitz ratios confirm the enhanced performance of the method and homogeneity of the matrices used for the study. The NIST SRM 1849a recorded the lowest HorRat value of 0.21 and undoubtedly the most homogeneous certified standard reference material used in the MLT.

Accuracy (NIST SRM 1849a)

The accuracy of an analytical procedure expresses the closeness of agreement between the value which is accepted either as a conventional true value or an accepted reference value and the value found. The established procedure for accuracy assessment is by analysing samples with known concentrations such as certified reference materials. The SPIFAN SMPR 2014.005 recommends NIST SRM 1849a for accuracy evaluation and the SRM is included in the SPIFAN matrices as blind coded duplicates.

The NIST SRM 1849a results were all within the certified limits of biotin confirming the accuracy of the method. It has been noticed that one of the duplicate results of NIST reference sample was slightly on the low bias (185.41µg/100g) for Lab 7 but this was investigated as explained before. The NIST SRM 1849a has comparatively higher biotin content and homogeneous therefore unlike other powder samples it was not reconstituted with water and 1g powder was directly weighed for the analysis.

Matrix recoveries were carried out during SLV at different levels of calibration range and the recoveries were within 95 to 105%.

Selectivity (Comparison with LCMS/MS)

Selectivity / specificity is the ability to assess unequivocally the analyte in the presence of components which may be expected to be present. Typically these might include impurities, degradants, matrix, etc.

Selectivity of the method is confirmed by analysing the SPIFAN matrices by LCMS/MS method by one of the laboratories participated in the MLT study (Lab 5). The method is a modified version of AOAC Official Method 2012.16 for pantothenic acid and uses a UPLC platform with triple quadrupole mass spectrometer. The biotin results by LCMS/MS method were closely comparable to the MLT results for the range of SPIFAN matrices used in the study. Please refer to Table 2016.02H for comparison data.

Table 2016.02D: Biotin (µg/100g) Practice Samples

Lab #	PRACTICE Sample 1		PRACTICE Sample 2	
	A	B	A	B
1	39.28	39.28	15.50	15.50
2	37.76	38.74	14.69	15.68
3	40.21	40.21	15.11	15.11
4	39.40	39.40	15.17	15.17
5	42.90	37.30	13.20	15.90
6	39.33	39.92	14.73	15.79
7	31.73	32.26	12.38	11.50
8	40.49	40.38	15.98	16.48
9	40.83	40.61	14.41	15.53
10	38.58	38.58	14.73	14.73
11	39.80	39.10	15.10	15.30
12	34.29	35.89	16.00	14.87
Mean	**38.72**	**38.47**	**14.75**	**15.13**
%RSD	**7.75**	**6.17**	**7.14**	**8.19**

Table 2016.02E: Biotin (µg/100g) MLT samples (Day 1)

Lab #	MLT Sample 1		MLT Sample 2		MLT Sample 3		MLT Sample 4		MLT Sample 5		MLT Sample 6	
	A	B	A	B	A	B	A	B	A	B	A	B
1	32.96	31.42	68.94	72.48	3.45	3.88	70.84	73.68	24.42	24.78	46.79	43.44
2	39.47	38.48	81.71	83.53	4.33	4.49	78.33	68.43	27.42	28.91	51.02	48.20
3	33.38	33.21	80.37	81.62	4.03	4.22	69.58	72.92	26.25	27.38	46.18	45.42
4	35.41	34.62	81.88	80.43	3.79	3.74	71.65	70.80	28.39	24.24	44.15	44.03
5	37.49	34.70	84.88	79.87	3.84	4.37	77.67	74.07	26.12	25.93	47.15	43.55
6	29.09	35.06	77.44	81.29	4.43	4.05	74.44	74.98	29.29	32.95	41.88	47.62
7	33.45	32.33	76.96	75.88	4.29	3.96	57.51	59.17	24.82	24.66	39.25	41.15
8	27.85	30.34	75.70	79.96	3.66	4.00	72.86	70.76	25.36	30.22	39.91	45.41
9	34.96	37.95	81.26	81.41	4.33	4.43	74.23	74.41	27.94	26.37	42.89	47.41
Mean	**33.78**	**34.23**	**78.79**	**79.61**	**4.02**	**4.13**	**71.90**	**71.02**	**26.67**	**27.27**	**44.36**	**45.14**
%RSD	**10.89**	**8.04**	**5.95**	**4.24**	**8.68**	**6.37**	**8.53**	**6.94**	**6.31**	**10.72**	**8.57**	**5.16**

Table 2016.02F: Biotin (µg/100g) MLT Samples (Day 2)

Lab #	MLT Sample 7		MLT Sample 8		MLT Sample 9		MLT Sample 10		MLT Sample 11		MLT Sample 12	
	A	B	A	B	A	B	A	B	A	B	A	B
1	195.27	200.37	270.20	265.83	171.08	155.73	10.69	9.80	44.26	44.68	48.66	56.39
2	200.63	191.30	271.16	266.46	180.38	159.53	11.11	10.68	46.01	43.08	52.02	53.47
3	196.99	196.48	265.19	272.41	179.63	168.93	10.40	10.24	42.19	43.96	54.56	54.37
4	191.31	205.32	251.11	265.49	171.89	162.31	11.19	9.20	44.02	44.66	54.54	54.92
5	196.80	196.60	260.67	247.84	170.46	161.75	9.76	9.37	40.82	40.13	52.48	49.79
6	193.53	186.75	263.54	248.41	184.86	161.43	9.88	9.89	43.31	42.48	53.84	56.11
7	185.41	190.38	219.30	213.21	153.16	140.19	10.05	9.15	38.43	39.03	50.09	50.14
8	201.99	199.82	268.69	254.35	175.94	163.60	9.36	9.59	40.44	42.14	52.00	44.45
9	202.58	205.62	278.04	274.41	183.98	158.33	11.02	10.72	46.05	46.44	55.47	54.89
Mean	**196.06**	**196.96**	**260.88**	**256.49**	**174.60**	**159.09**	**10.38**	**9.85**	**42.84**	**42.96**	**52.63**	**52.73**
%RSD	**2.81**	**3.33**	**6.64**	**7.37**	**5.55**	**5.02**	**6.35**	**6.02**	**6.05**	**5.42**	**4.24**	**7.39**

Table 2016.02G: Statistical evaluation biotin results from SPIFAN matrices

Samples	Product Description	p	Mean	%RSD_r	%RSD_R	HorRat
Practice 1	Infant Formula Powder Partially Hydrolysed Soy Based	12	38.59	3.18	6.98	0.38
Practice 2	Infant Formula Powder FOS/GOS Based	12	14.94	4.92	7.74	0.36
MLT - 1	Infant Formula Powder Partially Hydrolysed Milk Based	9	34.01	5.53	9.48	0.50
MLT - 2	Infant Elemental Powder	9	79.20	2.64	5.14	0.31
MLT - 3	Infant Formula RTF Milk Based	9	4.07	5.52	7.59	0.29
MLT - 4	Adult Nutritional RTF High Fat	9	71.46	3.88	7.75	0.46
MLT - 5	Infant Formula Powder Milk Based	9	26.97	6.79	8.76	0.45
MLT - 6	Infant Formula Powder Soy Based	9	44.75	5.77	6.96	0.39
MLT - 7	NIST SRM 1849a	9	196.5	2.38	3.04	0.21
MLT - 8	Adult Nutritional Powder Low Fat	9	258.7	2.81	7.03	0.51
MLT - 9	Child Formula Powder	9	166.8	7.03	7.03	0.47
MLT - 10	Toddler Formula Powder Milk-Based	9	10.12	5.74	6.65	0.29
MLT - 11	Infant Formula Powder Milk Based	9	42.90	2.26	5.72	0.31
MLT - 12	Adult Nutritional RTF High Protein	9	52.68	5.14	5.89	0.33

p = total number of laboratories
RSD_r = relative standard deviation of repeatability
RSD_R = relative standard deviation of reproducibility
HorRat = Horwitz Ratio (RSD_R / $PRSD_R$)

Table 2016.02H: Comparison with LCMS/MS technique

Sample #	Product Description	Blind Duplicates	Biotin (µg/100g)		
			AOAC 2016.02	LCMS/MS	Difference
MLT 1	Infant Formula Powder Partially Hydrolysed Milk Based	A	33.78	35.55	-1.77
		B	34.23	39.10	-4.87
MLT 2	Infant Elemental Powder	A	78.79	82.45	-3.66
		B	79.61	81.15	-1.54
MLT 3	Infant Formula RTF Milk Based	A	4.02	3.73	0.29
		B	4.13	3.84	0.29
MLT 4	Adult Nutritional RTF High Fat	A	71.90	66.06	5.84
		B	71.02	77.49	-6.47
MLT 5	Infant Formula Powder Milk Based	A	26.67	26.99	-0.32
		B	27.27	25.25	2.02
MLT 6	Infant Formula Powder Soy Based	A	44.36	43.00	1.36
		B	45.14	42.27	2.87
MLT 7	NIST SRM 1849a	A	196.06	186.58	9.48
		B	196.96	181.50	15.46
MLT 8	Adult Nutritional Powder Low Fat	A	260.88	263.90	-3.02
		B	256.49	246.17	10.32
MLT 9	Child Formula Powder	A	174.60	171.69	2.91
		B	159.09	162.77	-3.68
MLT 10	Toddler Formula Powder Milk-Based	A	10.38	9.87	0.51
		B	9.85	10.30	-0.45
MLT 11	Infant Formula Powder Milk Based	A	42.84	41.66	1.18
		B	42.96	39.77	3.19
MLT 12	Adult Nutritional RTF High Protein	A	52.63	54.05	-1.42
		B	52.73	51.57	1.16

SUMMARY AND CONCLUSION

The SLV and the MLT data provide systematic scientific evidence for a simple, selective, accurate and precise method as a potential candidate reference method for dispute resolution for the determination of total biotin in all forms of infant, adult, and/or pediatric formula. The method fully meets the intended purpose and applicability statement by complying with standard method performance requirement outlined in AOAC SPIFAN SMPR 2014.005.

The method was applied to a cross section of matrix types and established acceptable precision and accuracy. The analytical platform is inexpensive and the method can be used in almost any labs worldwide with basic facilities. The immunoaffinity column (IAC) clean-up extraction is the key step to successful analysis. Although R-Biopharm IAC was used for the MLT, alternative IAC was compared during SLV with comparable results. The performance parameters of the method are compared with AOAC SPIFAN SMPR 2014.005 and the key points are summarised in the Table 2016.02I.

Table 2016.02I: Comparison of method performance with SMPR 2014.005

Parameters	SMPR 2014.005	AOAC 2016.02	Comments
Analytical Range	0.1 - 150 µg/100g	0.1 - 300 µg/100g	Analytical range of the proposed method is wider based on the sample weight and loading volume for IAC extraction.
Limit of Quantitation	≤0.1 µg/100g	≤0.1 µg/100g	The proposed method meets the LOQ of 0.1 µg/100g as required by SMPR 2014.005.
Repeatability (RSD_r)	>1 µg/100g: ≤6%	>1 µg/100g: ≤5%	Repeatability of the proposed method is better than the SMPR 2014.005.
Reproducibility (RSD_R)	>1 µg/100g: ≤12%	>1 µg/100g: ≤10%	Reproducibility of the proposed method is better than the SMPR 2014.005.
Horwitz Ratio (HorRat)	0.5 to 2.0	0.3 to 0.5	Excellent precision performance rating.
Recovery (>1µg/100g)	90 to 110%	95 to 105%	The spiked recoveries by the proposed method are better than the recovery range specified by SMPR 2014.005.
NIST SRM 1849a	199 ± 13	197 ± 10	NIST 1949a results are within the certified limits.

RECOMMENDATIONS

A complete AOAC International MLT study workbook along with the statistical report and draft copy of the study report summarising the outcomes of this collaborative study were submitted with the recommendation that the AOAC First Action Official Method 2016.02 be accepted as a SPIFAN endorsed AOAC Official Final Action Method.

NOTES FROM THE STUDY DIRECTOR

I would like to place on record my learnings and experience with the collaborators during the course of the study. I believe that ideas for improvement, feedback and challenges are valuable information for future enhancement to the method in some way or other.

Filtrate for IAC Clean-up: One of the participants commented that the filtrate for IAC clean-up is not clear, suggested that if the same extraction would be also efficient if performed at pH 4, which would precipitate many of the proteins, yielding a clear filtrate. My laboratory tested few SPIFAN matrices using sodium acetate solution 0.4M, pH 4.0 (In to a 2000mL volumetric flask, weigh 108.8 g sodium acetate trihydrate. Add about 1800mL water. Dissolve. Add 50mL acetic acid, and adjust pH to 4.0 with acetic acid, dilute to volume with water). The filtrate was clear, IAC clean-up was faster and the biotin results were comparable to the current method. However this change was not advised to any of the collaborators and the participant was advised to proceed with current method as published as First Action Official AOAC 2016.02.

IAC loading speed: Two laboratories obtained lower biotin results for practice samples initially were advised to follow IAC loading by gravity as documented in the method and the results came back higher and comparable to other labs.

Elution of biotin from IAC: While assisting a participant investigating low biotin results, it became apparent that the "back flush" technique was inexactly followed, mainly it seems because the exact description of this technique was not given. So, rather than performing a true "back flush", the 3mL methanol eluate was recycled through the IA cartridge three times. It is possible that this may partly explain slightly low recovery for some samples, given that the IA cartridge may well have retained a small portion of this eluent fraction / biotin. The details of the back flush using a syringe plunger / reverse pressure is now detailed in the method.

Syringe Filter: At least two laboratories experienced interference with chromatography. The investigation isolated the issue with syringe filters with extractable chromophores. It was advised to test the filters as part of the reagent blank preparation

Quality Water: Quality of water**,** especially the water used for the final extraction is very important to get clean chromatogram. Type 1 water is recommended and this information will be updated in the method.

UPLC Platform: The availability of stationary phase (solid-core, kinetex phenyl hexyl) in smaller particle size for UPLC platforms is encouraging. This would significantly reduce analysis time and improve throughput, my laboratory is doing further work to validate the change.

Calculation errors: The calculation error is a common problem which was noticed. It was advised to participants to use reference samples to prompt any issues with calculation before reporting the results.

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1. *Karen Schimpf and Linda Butler Thompson*, Abbott Laboratories, United States
2. *Isabelle Malaviole*, Aquanal Laboratoire Aquitaine Analyses, France
3. *Michael Hodgson,* George Joseph and Ranjani Devi, AsureQuality Limited, New Zealand
4. *Craig Macfadzean, Xinping Huo and Felicia Lim*, AsureQuality Limited, Singapore
5. *Jirong Wang, Trevor Gill and Lee Kennedy,* DTS Food Laboratories, Australia
6. *Sudhakar Yadlapalli*, FirstSource Laboratory Solutions LLP, India
7. *Harvey Indyk, Brendon Gill, Mumtaz Dole and Cesar Yin*, Fonterra Co-operative, New Zealand

8. *Sneh Bhandari and Tiffany Gallegos-Peretz*, Merieux NutriSciences, United States
9. *Elaine Marley and Dave Leeman*, R-Biopharm Rhône Ltd, Scotland
10. *Hua Chen and Yan Green,* Mead Johnson, China
11. *Maurice Seegers and Nico Dekker*, Mead Johnson, Netherlands
12. *Esther Campos Gimenez,* Nestle Research Centre, Switzerland
13. *David Wollard,* Eurofins, New Zealand
14. *Adrienne McMahon,* Wyeth Nutrition Askeaton, Ireland

Chromatograms

Chromatograms of calibration standard and a SPIFAN MLT sample (00859RF00 / PZGP859) are given below in Figure 1 and 2 respectively.

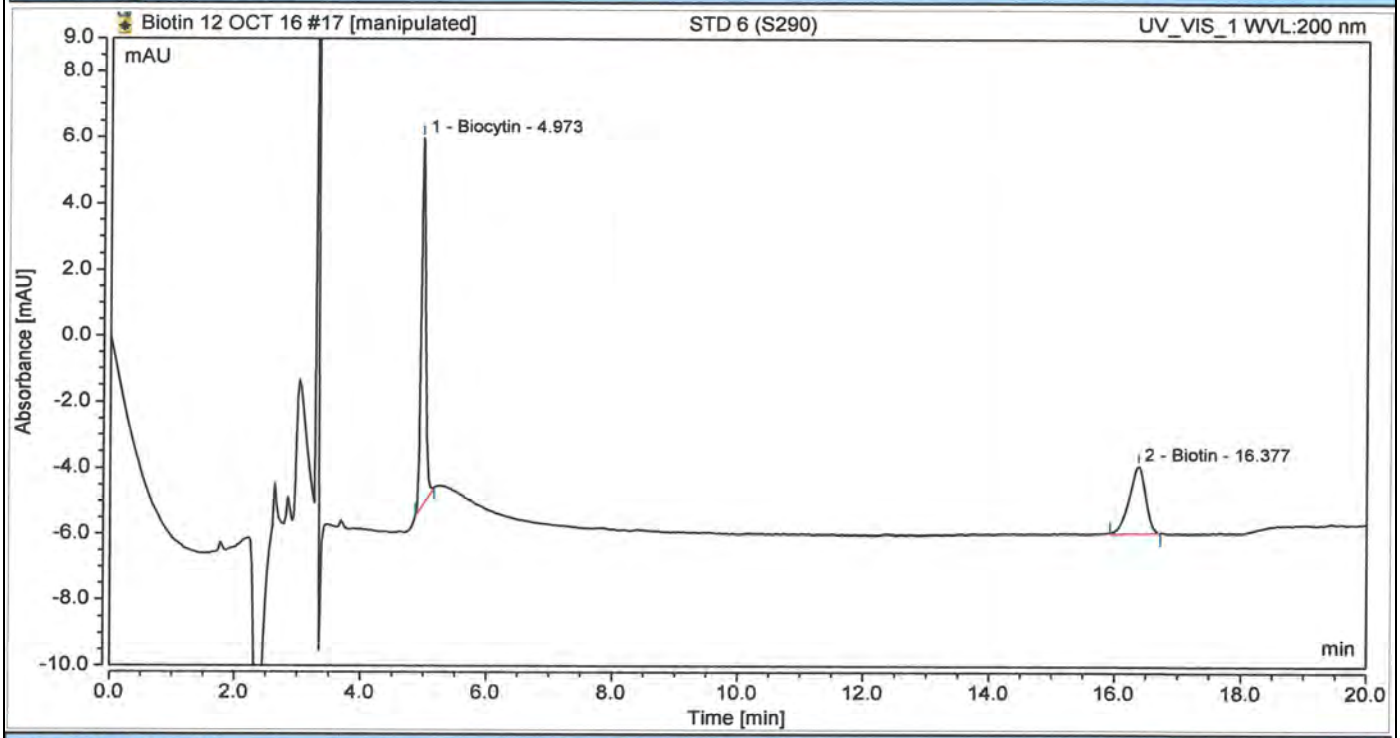
Figure 1: Calibration standard

Chromatogram and Results

Injection Details

Injection Name:	**STD 6 (S290)**	*Run Time (min):*	**27.00**
Vial Number:	**RA6**	*Injection Volume:*	**100.00**
Injection Type:	**Calibration Standard**	*Channel:*	**UV_VIS_1**
Calibration Level:	**06**	*Wavelength:*	**200.0**
Instrument Method:	**Biotin 11 Aug 15 ver 8**	*Bandwidth:*	**4**
Processing Method:	**Biotin**	*Dilution Factor:*	**1.0000**
Injection Date/Time:	**13-Oct-16 00:25**	*Sample Weight:*	**1.0000**

Chromatogram



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount ug/100mL
1	Biocytin	4.973	0.892	11.102	59.09	84.42	19.8967
2	Biotin	16.377	0.618	2.049	40.91	15.58	19.6813
Total:			**1.510**	**13.151**	**100.00**	**100.00**	

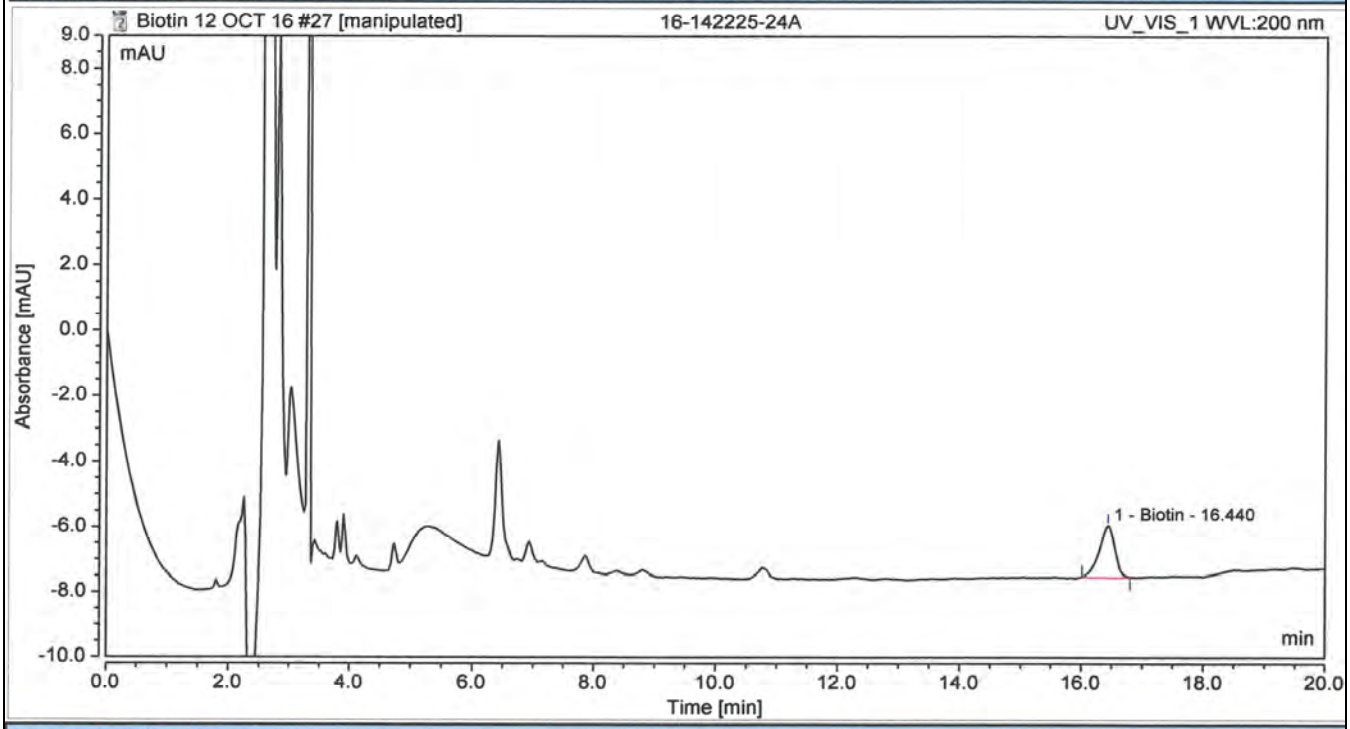
Figure 2: MLT Sample 8B (00859RF00 / PZGP859)

Chromatogram and Results

Injection Details

Injection Name:	**16-142225-24A**	*Run Time (min):*	**27.00**
Vial Number:	**RB7**	*Injection Volume:*	**100.00**
Injection Type:	**Unknown**	*Channel:*	**UV_VIS_1**
Calibration Level:		*Wavelength:*	**200.0**
Instrument Method:	**Biotin 11 Aug 15 ver 8**	*Bandwidth:*	**4**
Processing Method:	**Biotin**	*Dilution Factor:*	**20.0000**
Injection Date/Time:	**13-Oct-16 05:12**	*Sample Weight:*	**1.1154**

Chromatogram



Integration Results

No.	Peak Name	Retention Time min	Area mAU*min	Height mAU	Relative Area %	Relative Height %	Amount ug/100mL
n.a.	Biocytin	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
1	Biotin	16.440	0.477	1.599	100.00	100.00	272.4113
Total:			**0.477**	**1.599**	**100.00**	**100.00**	

AOAC SMPR 2014.005

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

Intended Use: Reference Method for Dispute Resolution

1 Applicability

Determination of total biotin in all forms of infant, adult, and/or pediatric formula (powders, ready-to-feed liquids, and liquid concentrates).

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 in liquid form, which may constitute the sole source of nourishment [AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN); 2010], made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

d-Biotin.—5-[(3aS,4S,6aR)-2-oxohexahydrothieno[3,4-d] imidazol-4-yl]pentanoic acid (*see* Figure 1).

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).

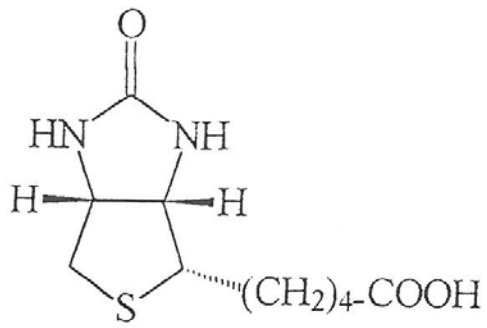


Figure 1. d-Biotin.

Table 1. Method performance requirements[a]		
Analytical range	0.1–150[b]	
Limit of quantitation (LOQ)	≤0.1[b]	
Repeatability (RSD_r)	0.1–1[b]	≤8%
	>1[b]	≤6%
Recovery	0.1–1[b]	80 to 120% of mean spiked recovery over the range of the assay
	>1[b]	90 to 110% of mean spiked recovery over the range of the assay
Reproducibility (RSD_R)	0.1–1[b]	≤16%
	>1[b]	≤12%
[a] Concentrations apply to (a) "ready-to-feed" liquids "as is"; (b) reconstituted powders (25 g into 200 g of water); and (c) liquid concentrates diluted 1:1 by weight.		
[b]		

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

See Table 1.

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 analytical range.

6 Reference Material(s)

National Institute of Standards and Technology (NIST) Standard Reference Material® (SRM) 1849a 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 1849a consists of 10 packets, each containing approximately 10 g of material. Certified value of NIST 1849a is 1.99 ± 0.13 mg/kg biotin.

7 Validation Guidance

Recommended level of validation: *Official Methods of Analysis*SM.

8 Maximum Time-to-Result

No maximum time.

Approved by AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN). Final Version Date: March 18, 2014.

ERP SUMMARY FOR FIRST TO FINAL ACTION METHOD RECOMMENDATION

AOAC 2016.05	Analysis of Vitamin D2 and Vitamin D3 by LC MS/MS in Milk Powders, Infant Formulas, and Adult Nutritionals

GUIDANCE FOR AOAC ERPS - APPENDIX G[1]	**Considered?**	**Comments/Reference if applicable**
Method Applicability	Yes	Meets SMPR
ERP First Action to Final Action recommendations & improvements	Yes	
Draft Final Action method reviewed by ERP	Yes	Completed in June 2017
Safety Concerns	Yes	
Reference Materials	Yes	NIST 1849a and SPIFAN Materials
Single Laboratory Validation	Yes	Gill et al, Journal of AOAC Int 99, 5 (2016) p.1321
Reproducibility/Uncertainty and Probability of Detection	Yes	In publication pending review.
Comparison to SMPR (SMPR criteria met?)	Yes	AOAC 2011.004
Feedback from Users of Method	Yes	All Addressed

DOCUMENTATION	**Available?**	**Comments**
Safety Evaluation	Yes	
Reference Materials	Yes	NIST 1849a and SPIFAN Materials
SLV or PTM	Yes	Gill et al, Journal of AOAC Int 99, 5 (2016) p.1321
Approved Validation Protocols	Yes	OMA Appendix L
Statistics Review		
Method Published in OMA	Yes	
Method Performance vs SMPR criteria	Yes	Meets the AOAC SMPR 2011.004
Feedback Information	Yes	All addressed
Additional Recognition(s)	Yes	Is a working item in ISO
ERP Reports	Yes	3-16-2016; 9-20-2016; 3-16-2017
Manuscript(s) Published in JAOAC	Yes	Gill et al, Journal of AOAC Int 99, 5 (2016) p.1321 (SLV); MLT publication in process
ERP Method Recommendation (Final Action/Repeal/Continuation)		

[1] *Official Methods of Analysis of AOAC INTERNATIONAL*, Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis, p.3 "*First Action to Final Action Methods: Guidance for AOAC Expert Review Panels.*"

Analysis of Vitamin D_2 and Vitamin D_3 in Infant and Adult Nutritional Formulas by Liquid Chromatography–Tandem Mass Spectrometry: A Multi-Laboratory Testing Study

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Abstract

A multi-laboratory testing study was conducted on AOAC First Action Method 2016.05: Analysis of Vitamin D_2 and Vitamin D_3 in Fortified Milk Powders, Infant Formulas, and Adult/Pediatric Nutritional Formulas by Liquid Chromatography–Tandem Mass Spectrometry. Nine laboratories participated in the analysis of duplicate samples of 20 nutritional products. The samples were saponified at high temperature with lipid-soluble components extracted into isooctane; an aliquot was washed, and vitamin D derivatized with 4-phenyl-1,2,4-triazoline-3,5-dione to form a high molecular mass, easily ionizable adduct, extracted into acetonitrile and analyzed by reversed phase liquid chromatography-tandem mass spectrometry. Stable isotope-labeled internal standards were used for quantitation to correct for losses in extraction and variation in derivatization and ionization efficiencies. Acceptable precision as relative standard deviation (RSD) was demonstrated; repeatability ranged from 1.9 to 5.8% RSD_r and reproducibility values ranged from 6.4 to 12.7% RSD_R, with samples meeting the precision limits specified in the vitamin D Standard Method Performance Requirements and the guidelines recommended for the Horwitz ratio. Method accuracy was assessed using the NIST 1849a SRM, with the *p*-value of 0.32 indicating an absence of bias against the certified value. As expected, placebo samples not fortified with vitamin D returned negligible results.

Introduction

Vitamin D is not a true vitamin as individuals with adequate skin exposure to UV radiation produce vitamin D from a precursor, 7-dehydrocholesterol. However, dietary supplementation of vitamin D is necessary for many, with infant formulas typically fortified with vitamin D_3 and, less commonly, vitamin D_2. Both vitamin D_2 (ergocalciferol) and vitamin D_3 (cholecalciferol) are metabolized in the liver to their respective 25-hydroxy vitamers, which are the dominant circulating forms in blood. The main biological function of vitamin D is calcium homeostasis, controlling the absorption, transport, and deposition of calcium and phosphorus as part of bone mineralization (1).

Rapid, high-throughput analytical methods for vitamin D are needed for routine testing to meet product specifications, and reference methods utilizing contemporary techniques are needed to demonstrate product compliance with strict global regulations. Given that the internationally accepted multi-dimensional LC–UV method for vitamin D, AOAC 2002.05 (2), has a long total analysis time, an updated reference method for vitamin D was identified by the Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) as a priority. We previously developed a liquid chromatography–tandem mass spectrometry (LC–MS/MS) method that incorporates saponification prior to solvent extraction, with 4-phenyl-1,2,4-triazoline-3,5-dione (PTAD) derivatization prior to instrumental analysis (3). The method subsequently underwent a comprehensive single laboratory validation (SLV) study using the SPIFAN kit (4), a set of infant formula and adult nutritional products that were selected as a representative sub-sample of the wide range of commercially available products, and the results were compared with the standard method performance requirements (SMPR) (5, 6). In March 2016, this LC–MS method was reviewed by the SPIFAN expert review panel (ERP), was approved for Official First Action status as AOAC Method 2016.05 (7), and was recommended to advance to a multi-laboratory testing (MLT) study for evaluation of reproducibility.

Multi-Laboratory Testing Study

The participating laboratories represented a wide range of food-testing laboratories including governmental agencies, infant formula manufacturers, and contract analytical services. Prior to commencement of the MLT study, each collaborator received a detailed study protocol to allow familiarization with the technique and an opportunity to communicate any difficulties.

The SPIFAN kit and a candidate Standard Reference Material (SRM) NIST 1869 (National Institute of Standards and Technology, Gaithersburg, MD), a soy-based infant/adult nutritional formula fortified with both vitamin D_2 and vitamin D_3, were used in this study. A practice sample, NIST 1849a, was run by participants and, when acceptable results had been obtained, approval to proceed to the analysis of the SPIFAN kit samples was given. The SPIFAN kit was tested over 2 separate days as blind-coded duplicate pairs.

All data were statistically analyzed using the harmonized guidelines for collaborative studies to establish overall mean, intra-laboratory repeatability (S_r), repeatability relative standard deviation (RSD_r), inter-laboratory reproducibility (S_R), reproducibility relative standard deviation (RSD_R), and Horwitz ratio (HorRat) (8). Cochran (p = 0.025, one-tail) and Grubbs (single and double, p = 0.025, two-tail) tests were utilized to determine outliers.

Method

The method protocol sent to the collaborating laboratories was as described in AOAC First Action Method 2016.05 with the minor modification of removal of the drying step for acetone.

AOAC Official Method 2016.05

Analysis of Vitamin D_2 and Vitamin D_3 in Fortified Milk Powders, Infant Formulas, and Adult/Pediatric Nutritional Formulas by Liquid Chromatography–Tandem Mass Spectrometry

Final Action 2017

[Applicable to the determination of vitamin D_2 and vitamin D_3 in fortified milk powders, infant formulas, and adult/pediatric nutritional formulas.]

Caution: Refer to the Material Safety Data Sheets for all chemicals prior to use. Use all appropriate personal protective equipment and follow good laboratory practices.

A. Principle

Samples are saponified at high temperature; then lipid-soluble components are extracted into isooctane. A portion of the isooctane layer is transferred and washed, and an aliquot of 4-phenyl-1,2,4-triazoline-3,5-dione (PTAD) is added to derivatize vitamin D to form a high-molecular mass, easily ionizable adduct. The vitamin D adduct is then extracted into a small volume of acetonitrile and analyzed by reverse-phase liquid chromatography (LC). Detection is by tandem mass spectrometry (MS/MS) using multiple reaction monitoring. Stable isotope-labeled (SIL) d6-vitamin D_2 and d6-vitamin D_3 internal standards are used for quantitation to correct for losses in extraction and any variation in derivatization and ionization efficiencies.

B. Apparatus

(a) *Ultra-high performance LC (UHPLC) system.*—Nexera (Shimadzu, Kyoto, Japan) or equivalent LC system consisting of a dual pump system, a sample injector unit, a degasser unit, and a column oven.

(b) *Triple-quadrupole mass spectrometer.*—Triple Quad 6500 (Sciex, Framingham, MA) or equivalent MS/MS instrument.

(c) *Column.*—Kinetex C_{18} core-shell, 2.6 µm, 2.1 mm × 50 mm (Phenomenex, Torrance, CA) or equivalent.

(d) *UV spectrophotometer.*—Capable of digital readout to 3 decimal places.

(e) *Centrifuge tubes.*—Polypropylene, 15 mL.

(f) *Boiling tubes.*—Glass, 60 mL.

(g) *Water baths.*—Cold 20°C, hot 70°C.

(h) *Disposable syringes.*—1 mL.

(i) *Syringe filters.*—PTFE, 0.2 µm, 13 mm.

(j) *Centrifuge.*—Suitable for 60 mL boiling tubes and 15 mL centrifuge tubes.

(k) *Pasteur pipette.*—Glass, ~140 mm.

(l) *Horizontal shaker.*

(m) *Microcentrifuge vials.*—2 mL.

(n) *Filter membranes.*—0.45 µm nylon.

(o) *Cryogenic vials.*—2 mL.

(p) *Schott bottles.*—1 L.

(q) *HPLC vials, septa, and caps.*

C. Reagents

(a) *Vitamin D_2 (ergocalciferol).*—CAS No. 50-14-6, purity: ≥99%.

(b) *Vitamin D_3 (cholecalciferol).*—CAS No. 67-97-0, purity: ≥99%.

(c) *d6-Vitamin D_2.*— (26,26,26,27,27,27-d6 ergocalciferol), CAS No. 1311259-89-8, enrichment: ≥99%, purity: ≥99%.

(d) *d6-Vitamin D_3.*— (26,26,26,27,27,27-d6 cholecalciferol), CAS No. 118584-54-6, enrichment: ≥99%, purity: ≥99%.

(e) *PTAD (4-phenyl-1,2,4-triazoline-3,5-dione).*—Reagent grade (store in desiccator at 2–8°C).

(f) *Formic acid.*—LC–MS grade.

(g) *Potassium hydroxide.*—Reagent grade.

(h) *Pyrogallol.*—Reagent grade.

(i) *Ethanol.*—LC grade.

(j) *Methanol.*—LC–MS grade.

(k) *Isooctane (2,2,4-trimethylpentane).*—LC grade.

(l) *Acetone.*—LC grade.

(m) *Acetonitrile.*—LC–MS grade.

(n) *Water.*—Purified with resistivity ≥18 MΩ.

D. Reagent Preparation

(a) *PTAD solution (10 mg mL^{-1}).*—To a 5 mL volumetric flask, add 50 mg PTAD, then add 4 mL acetone, and dissolve; dilute to volume with acetone. Expiry: 1 day.

(b) *Potassium hydroxide solution (50%, w/v).*—Dissolve 100 g potassium hydroxide in 200 mL water. Expiry: 1 month.

(c) *Ethanolic pyrogallol solution (1%, w/v).*— Dissolve 5 g pyrogallol in 500 mL ethanol. Expiry: 1 day.

(d) *Mobile phase A (formic acid; 0.1%, v/v).*—To 500 mL water, add 0.5 mL formic acid. Expiry: 1 week.

(e) *Mobile phase B (methanol; 100%, v/v).*—500 mL methanol. Expiry: 1 month.

E. Standard Preparation

Vitamin D is sensitive to light; perform all steps under UV-shielded lighting. If vitamin D_3 is exclusively required for analysis, then standards pertaining to vitamin D_2 need not be used and vice versa.

(a) *Stable isotope-labeled vitamin D_2 or vitamin D_3 stock standard (SILD$_2$SS or SILD$_3$SS; ~10 µg mL^{-1}).*—

(1) Dispense the contents of a 1 mg vial of d6-vitamin D_2 or a 1 mg vial of d6-vitamin D_3 into separate 100 mL volumetric flasks.

(2) Dissolve in ~90 mL ethanol. To promote dissolution, sonicate if necessary. Mix thoroughly; dilute to volume with ethanol.

(3) Measure the absorbance of an aliquot of SILD$_2$SS or SILD$_3$SS at 265 nm. The spectrophotometer should be zeroed against an ethanol blank solution. Calculate and record the concentration.

(4) Immediately dispense aliquots of SILD$_2$SS or SILD$_3$SS (~1.3 mL) into cryogenic vials and freeze at ≤15°C.

(b) *Stable isotope-labeled internal standard (SILIS; ~1 µg mL^{-1}).*—

(1) Prepare an adequate volume of SILIS for the daily sample numbers. For every 15 samples (or part thereof) in an analytical run, remove one vial of SILD$_2$SS and one vial of SILD$_3$SS from the freezer and allow to warm to room temperature.

(2) Pipette 1.0 mL each of SILD$_2$SS and SILD$_3$SS into the same 10 mL volumetric flask (use a separate 10 mL volumetric flask for each set of 15 samples). Dilute to volume with acetonitrile and mix thoroughly.

(3) Pool all 10 mL volumetric flasks together and mix thoroughly.

(4) Make fresh daily.

(c) *Non-labeled vitamin D_2 or vitamin D_3 stock standard (NLD$_2$SS or NLD$_3$SS; ~1 mg mL^{-1}).*—

(1) Accurately weigh approximately 50 mg vitamin D_2 or vitamin D_3 into separate 50 mL volumetric flasks.

(2) Dissolve in ~40 mL ethanol. To promote dissolution, sonicate if necessary. Mix thoroughly; dilute to volume with ethanol. Store in a freezer at ≤15°C for a maximum of 3 months.

(d) *Non-labeled vitamin D_2 or vitamin D_3 purity standard (NLD$_2$PS or NLD$_3$PS; ~10 µg mL^{-1}).*—

(1) Pipette 1.0 mL NLD$_2$SS or NLD$_3$SS into separate 100 mL volumetric flasks. Dilute to volume with ethanol.

(2) Measure the absorbance of an aliquot of each solution at 265 nm. The spectrophotometer should be zeroed against an ethanol blank solution. Record the absorbance and calculate the concentration.

(3) Make fresh daily.

(e) *Non-labeled working standard (NLWS; ~1 µg mL^{-1}).*—

(1) Pipette 1.0 mL NLD_2PS and 1.0 mL NLD_3PS into a single 10 mL volumetric flask. Dilute to volume with acetonitrile.

(2) Make fresh daily.

(f) *Calibration standards (CS).*—Make fresh daily. See **Table 2016.05A** for concentrations of the calibration standard solutions.—

(1) *Calibration standard 1 (CS1).*—Pipette 10 µL NLWS and 250 µL SILIS into a 25 mL volumetric flask.

(2) *Calibration standard 2 (CS2).*—Pipette 50 µL NLWS and 250 µL SILIS into a 25 mL volumetric flask.

(3) *Calibration standard 3 (CS3).*—Pipette 250 µL NLWS and 250 µL SILIS into a 25 mL volumetric flask.

(4) *Calibration standard 4 (CS4).*—Pipette 500 µL NLWS and 250 µL SILIS into a 25 mL volumetric flask.

(5) *Calibration standard 5 (CS5).*—Pipette 1250 µL NLWS and 250 µL SILIS into a 25 mL volumetric flask.

(6) To each calibration standard, add 5 mL acetonitrile and 75 µL PTAD solution; shake to mix.

(7) Leave the calibration standards in the dark for 5 min.

(8) Add 6.25 mL water to each calibration standard and then dilute to volume with acetonitrile; shake to mix.

(9) Transfer ~1 mL of each calibration standard to an HPLC vial ready for analysis.

Table 2016.05A Nominal concentrations of calibration standards

Calibration standard	Concentration, ng mL^{-1}	
	Vitamin D	SIL d6-vitamin D
CS1	0.4	10
CS2	2.0	10

CS3	10	10
CS4	20	10
CS5	50	10

F. Sample Preparation

Vitamin D is sensitive to light; perform all steps under UV-shielded lighting. Sample preparation step (b) is optional for powder samples for which homogeneity may be an issue.

(a) *Powder sample preparation.*—Accurately weigh 1.8–2.2 g powder sample into a boiling tube. Record the weight.

(b) *Slurry sample preparation.*—

(1) Accurately weigh 19.0–21.0 g powder into a disposable slurry container. Record the weight.

(2) Accurately weigh ~80 mL water into the container. Record the weight.

(3) Shake thoroughly until mixed. Place in the dark at room temperature for 15 min and shake to mix every 5 min.

(4) Accurately weigh 9.5–10.5 g slurry or reconstituted powder sample into a boiling tube. Record the weight.

(c) *Liquid sample preparation.*—Accurately weigh 10.0 mL liquid milk into a boiling tube. Record the weight.

G. Extraction and Derivatization

(a) To a powder, slurry, or liquid sample in a boiling tube, add 10 mL ethanolic pyrogallol solution, then add 0.5 mL SILIS, and then cap and vortex mix.

(b) Add 2 mL potassium hydroxide solution to the boiling tube; cap and vortex mix.

(c) Place the boiling tube in a water bath at 70°C for 1 h; vortex mix every 15 min.

(d) Place the boiling tube in a water bath at room temperature until cool.

(e) Add 10 mL isooctane to the boiling tube; cap the boiling tube tightly and place on a horizontal shaker for 10 min.

(f) Add 20 mL water to the boiling tube and invert the tube 10 times; place in a centrifuge at ≥250 × *g* for 15 min.

(g) Transfer a 5 mL aliquot of the upper isooctane layer into a 15 mL centrifuge tube using a Pasteur pipette, taking care not to transfer any of the lower layer.

(h) Add 5 mL water to the centrifuge tube; cap and vortex mix; then place in a centrifuge at 2000 × *g* for 5 min.

(i) Transfer 4–5 mL upper isooctane layer to a new 15 mL disposable centrifuge tube using a disposable pipette, taking care not to transfer any of the lower layer.

(j) Add 75 µL PTAD solution to the centrifuge tube; cap and immediately vortex mix.

(k) Allow to stand in the dark for 5 min to allow the derivatization reaction to complete.

(l) Add 1 mL acetonitrile to the centrifuge tube; cap and vortex mix; then place in a centrifuge at 2000 × *g* for 5 min.

(m) Using a variable volume pipette, transfer 500 µL lower layer into a microcentrifuge vial, taking care not to transfer any of the upper layer.

(n) Add 167 µL water to the microcentrifuge vial; cap and vortex mix.

(o) Using a syringe filter, transfer an aliquot from the microcentrifuge vial to an amber HPLC vial; then cap.

H. Chromatography

(a) Set up the UHPLC system with the configuration shown in **Table 2016.05B.**

Table 2016.05B Chromatographic instrument settings

Instrument parameter	Value
Mobile phase A	Formic acid, 0.1%
Mobile phase B	Methanol, 100%
Column	Kinetex C_{18}
Oven temperature	40°C
Chiller temperature	15°C
Injection volume	3 µL
Initial flow rate	0.6 mL min^{-1}

(b) Form gradients by high-pressure mixing of the two mobile phases, A and B, using the procedure shown in **Table 2016.05C**.

Table 2016.05C Gradient procedure for chromatographic separation

Time, min	Flow rate, mL min^{-1}	Mobile phase composition	
		% A	% B
0	0.6	25	75
3.3	0.6	0	100
3.7	1.0	0	100

4.8	1.0	0	100
4.9	0.6	25	75
5.5	0.6	25	75

I. Mass Spectrometry

(a) Set up the mass spectrometer with the instrument settings shown in **Table 2016.05D**.

Table 2016.05D Mass spectrometer instrument settings[a]

Instrument parameter	Value
Ionization mode	ESI^+
Curtain gas	30 psi
Nebulizer gas GS1	40 psi
Heater gas GS2	40 psi
Collision gas	N_2
Source temperature	300°C
Ion spray voltage	5500 V

[a] These settings are suitable for the 6500 triple-quadrupole mass spectrometer (Sciex). Optimal settings on alternative instruments may differ.

(b) The specific compound parameters to be used are shown in **Tables 2016.05E** and **2016.05F**.

Table 2016.05E Compound parameters (vitamin D_2 instrument method only)

Vitamin D_2 ion[a]	Precursor ion, m/z	Product ion, m/z	DP[b], V	EP[c], V	CE[d], V	CXP[e], V	Dwell time, ms
Analyte quantifier	572.2	298.0	81	10	23	22	120
Analyte qualifier	572.2	280.0			39	16	80
Internal standard quantifier	578.2	298.0			23	22	120
Internal standard qualifier	578.2	280.0			39	16	80

[a] Analyte = vitamin D_2–PTAD adduct, Internal standard ion = d6-vitamin D_2–PTAD adduct.
[b] DP = declustering potential, [c] EP = entrance potential, [d] CE = collision energy, [e] CXP = collision cell exit potential.

Table 2016.05F Compound parameters (vitamin D_3 instrument method only)

Vitamin D_3 ion[a]	Precursor ion, m/z	Product ion, m/z	DP[b], V	EP[c], V	CE[d], V	CXP[e], V	Dwell time, ms
Analyte quantifier	560.2	298.0	151	10	21	18	120
Analyte qualifier	560.2	280.0			37	18	80
Internal standard quantifier	566.2	298.0			21	18	120
Internal standard qualifier	566.2	280.0			37	18	80

[a] Analyte = vitamin D_3–PTAD adduct, Internal standard ion = d6-vitamin D_3–PTAD adduct.
[b] DP = declustering potential, [c] EP = entrance potential, [d] CE = collision energy, [e] CXP = collision cell exit potential.

J. Calculations

(a) *Concentration of stable isotope-labeled vitamin D_2 in stock standard SILD$_2$SS.*—

$$SILD_2SS_{D2conc} = \frac{SILD_2SS_{abs(\lambda max)}}{E^{1\%}_{1cm}} \times 10000$$

where: $SILD_2SS_{D2conc}$ = concentration of d6-vitamin D_2 in stock standard (µg mL^{-1}); $SILD_2SS_{abs(\lambda max)}$ = UV absorbance of stock standard at 265 nm (cm^{-1}); $E^{1\%}_{1cm}$ = extinction coefficient for vitamin D_2 in ethanol (461 dL g.cm^{-1}); 10000 = concentration conversion factor (g dL^{-1} to µg mL^{-1}).

(b) *Concentration of stable isotope-labeled vitamin D_3 in stock standard SILD$_3$SS.*—

$$SILD_3SS_{D3conc} = \frac{SILD_3SS_{abs(\lambda max)}}{E^{1\%}_{1cm}} \times 10000$$

where: $SILD_3SS_{D3conc}$ = concentration of d6-vitamin D_3 in stock standard (µg mL^{-1}); $SILD_3SS_{abs(\lambda max)}$ = UV absorbance of stock standard at 265 nm (cm^{-1}); $E^{1\%}_{1cm}$ = extinction coefficient for vitamin D_3 in ethanol (485 dL g.cm^{-1}); 10000 = concentration conversion factor (g dL^{-1} to µg mL^{-1}).

(c) *Concentration of stable isotope-labeled vitamin D_2 in internal standard SILIS.*—

$$SILIS_{D2conc} = SILD_2SS_{D2conc} \times \frac{1.0}{10} \times 1000$$

where: $SILIS_{D2conc}$ = concentration of d6-vitamin D_2 in internal standard (ng mL^{-1}); $SILD_2SS_{D2conc}$ = concentration of d6-vitamin D_2 in stock standard (µg mL^{-1}); 1000 = concentration conversion factor (µg mL^{-1} to ng mL^{-1}).

(d) *Concentration of stable isotope-labeled vitamin D_3 in internal standard SILIS.*—

$$SILIS_{D3conc} = SILD_3SS_{D3conc} \times \frac{1.0}{10} \times 1000$$

where: $SILIS_{D3conc}$ = concentration of d6-vitamin D_3 in internal standard (ng mL^{-1}); $SILD_3SS_{D3conc}$ = concentration of d6-vitamin D_3 in stock standard (µg mL^{-1}); 1000 = concentration conversion factor (µg mL^{-1} to ng mL^{-1}).

(e) *Concentration of non-labeled vitamin D_2 in purity standard NLD_2PS.*—

$$NLD_2PS_{D2conc} = \frac{NLD_2PS_{abs(\lambda max)}}{E^{1\%}_{1cm}} \times 10000$$

where: NLD_2PS_{D2conc} = concentration of vitamin D_2 in purity standard (µg mL^{-1}); $NLD_2PS_{abs(\lambda max)}$ = UV absorbance of purity standard at 265 nm (cm^{-1}); $E^{1\%}_{1cm}$ = extinction coefficient for vitamin D_2 in ethanol (461 dL $g.cm^{-1}$); 10000 = concentration conversion factor (g dL^{-1} to µg mL^{-1}).

(f) *Concentration of non-labeled vitamin D_3 in purity standard NLD_3PS.*—

$$NLD_3PS_{D3conc} = \frac{NLD_3PS_{abs(\lambda max)}}{E^{1\%}_{1cm}} \times 10000$$

where: NLD_3PS_{D3conc} = concentration of vitamin D_3 in purity standard (µg mL^{-1}); $NLD_3PS_{abs(\lambda max)}$ = UV absorbance of purity standard at 265 nm (cm^{-1}); $E^{1\%}_{1cm}$ = extinction coefficient for vitamin D_3 in ethanol (485 dL $g.cm^{-1}$); 10000 = concentration conversion factor (g dL^{-1} to µg mL^{-1}).

(g) *Concentration of non-labeled vitamin D_2 in working standard NLWS.*—

$$NLWS_{D2conc} = NLD_2PS_{D2conc} \times \frac{1.0}{10} \times 1000$$

where: $NLWS_{D2conc}$ = concentration of vitamin D_2 in working standard (ng mL^{-1}); NLD_2PS_{D2conc} = concentration of vitamin D_2 in purity standard (µg mL^{-1}); 1000 = concentration conversion factor (µg mL^{-1} to ng mL^{-1}).

(h) *Concentration of non-labeled vitamin D_3 in working standard NLWS.*—

$$NLWS_{D3conc} = NLD_3PS_{D3conc} \times \frac{1.0}{10} \times 1000$$

where: $NLWS_{D3conc}$ = concentration of vitamin D_3 in working standard (ng mL^{-1}); NLD_3PS_{D3conc} = concentration of vitamin D_3 in purity standard (µg mL^{-1}); 1000 = concentration conversion factor (µg mL^{-1} to ng mL^{-1}).

(i) *Concentrations of vitamin D_2 and vitamin D_3 in calibration standards CS1–CS5.*—

$$CS1_{Dconc} = NLWS_{Dconc} \times \frac{0.01}{25}$$

$$CS2_{Dconc} = NLWS_{Dconc} \times \frac{0.05}{25}$$

$$CS3_{Dconc} = NLWS_{Dconc} \times \frac{0.25}{25}$$

$$CS4_{Dconc} = NLWS_{Dconc} \times \frac{0.5}{25}$$

$$CS5_{Dconc} = NLWS_{Dconc} \times \frac{1.25}{25}$$

where: $CS1–CS5_{Dconc}$ = concentration of vitamin D_2 or vitamin D_3 in calibration standards (ng mL^{-1}); $NLWS_{Dconc}$ = concentration of vitamin D_2 or vitamin D_3 in working standard (ng mL^{-1}).

(j) *Concentrations of stable isotope-labeled d6-vitamin D_2 and d6-vitamin D_3 in calibration standards CS1–CS5.—*

$$CS1\text{-}5_{Dconc} = SILIS_{Dconc} \times \frac{0.25}{25}$$

where: $CS1–CS5_{Dconc}$ = concentration of d6-vitamin D_2 or d6-vitamin D_3 in calibration standards (ng mL^{-1}); $SILIS_{Dconc}$ = concentration of d6-vitamin D_2 or d6-vitamin D_3 in internal standard (ng mL^{-1}).

(k) *Mass of powder in slurried sample.—*

$$S_{mass} = \frac{D_{mass}}{(D_{mass} + W_{mass})} \times A_{mass}$$

where: S_{mass} = the mass of powder in slurried sample (g); D_{mass} = the mass of dry powder used to make the slurry (g); W_{mass} = the mass of water used to make the slurry (g); A_{mass} = the mass of the aliquot of slurried sample used in the analysis (g).

(l) Determine the linear regression curve $y = mx + c$ (using the "least squares" method) for the ratio of peak areas (non-labeled vitamin D/stable isotope-labeled d6-vitamin D) vs. the ratio of concentrations (non-labeled vitamin D/stable isotope-labeled d6-vitamin D) for five calibration standards with the y-intercept forced through zero.

(m) The concentration (*w/w*) of vitamin D_2 or vitamin D_3 in dry powders is calculated as:

$$Result\ D = \frac{PA_{NLD}}{PA_{SILD}} \times \frac{SILIS_{Dconc}}{L} \times \frac{SILIS_{alqt}}{S_{mass}} \times \frac{100}{1000}$$

where: Result D = vitamin D_2 or vitamin D_3 concentration in sample (µg hg^{-1}); PA_{NLD} = peak area of vitamin D_2 or vitamin D_3 in sample; PA_{SILD} = peak area of d6-vitamin D_2 or d6-vitamin D_3 in sample; $SILIS_{Dconc}$ = concentration of d6-vitamin D_2 or

d6-vitamin D_3 in SILIS (ng mL^{-1}); L = slope of calibration curve; $SILIS_{alqt}$ = volume of SILIS aliquot spiked into sample (0.5 mL); S_{mass} = mass of sample (g); 1000 = concentration conversion factor (ng g^{-1} to µg g^{-1}); 100 = concentration conversion factor (µg g^{-1} to µg hg^{-1}).

(n) The concentration (*w/v*) of vitamin D_2 or vitamin D_3 in ready-to-feed (RTF) liquids is calculated as:

$$\text{Result D} = \frac{PA_{NLD}}{PA_{SILD}} \times \frac{SILIS_{Dconc}}{L} \times \frac{SILIS_{alqt}}{S_{vol}} \times \frac{100}{1000}$$

where: Result D = vitamin D_2 or vitamin D_3 concentration in sample (µg dL^{-1}); PA_{NLD} = peak area of vitamin D_2 or vitamin D_3 in sample; PA_{SILD} = peak area of d6-vitamin D_2 or d6-vitamin D_3 in sample; $SILIS_{Dconc}$ = concentration of d6-vitamin D_2 or d6-vitamin D_3 in SILIS (ng mL^{-1}); L = slope of calibration curve; $SILIS_{alqt}$ = volume of SILIS aliquot spiked into sample (0.5 mL); S_{vol} = volume of sample (mL); 1000 = concentration conversion factor (ng mL^{-1} to µg mL^{-1}); 100 = concentration conversion factor (µg mL^{-1} to µg dL^{-1}).

(o) The concentration of vitamin D_2 or vitamin D_3 as IU hg^{-1} in the sample is calculated as:

$$\text{Result(IU hg}^{-1}\text{)} = \text{Result(}\mu\text{g hg}^{-1}\text{)} \times 40$$

where: 40 = dietary conversion factor (µg hg^{-1} to IU hg^{-1}).

K. Data Handling

Report result as µg hg^{-1} to one decimal place or as IU hg^{-1} to zero decimal places.

Results and Discussion

The initial phase of method evaluation within the participating laboratories involved the analysis of a practice sample. The NIST 1849a SRM was selected as the practice sample as it provided confidence that the method was implemented appropriately within each laboratory and that accurate results could be obtained.

A total of 12 laboratories agreed to participate as part of this study; however, only 9 laboratories were able to submit data for evaluation prior to the submission deadline, with 2

laboratories not reporting any data and 1 laboratory unable to achieve acceptable results for the practice sample as defined by the SPIFAN ERP (4).

Upon completion of the analysis of all samples, each participating laboratory reported measured results as well as additional information such as sample identification, weights, volumes, UV absorbances, and peak areas. Participants were also asked to document any deviation from the method and any other pertinent comments based on their experiences in adapting the method into their laboratory. The results received from participants were tabulated and are summarized in Table 1. All 9 collaborating laboratories returned acceptable standard calibration parameters based on linear regression correlation coefficients (r^2: ≥ 0.998).

Only a single pair of results for vitamin D_2 from Lab 6 was excluded as Cochran outliers; no other outliers were identified and all other results were used in the generation of precision values.

Repeatability ranged from 1.9 to 5.8% RSD_r and reproducibility values ranged from 6.4 to 12.7% RSD_R (Table 2), with $HorRat_R$ values for the method ranging from 0.2 to 0.6 (expected range 0.5–2.0) (8).

Method accuracy was assessed in accordance with SPIFAN procedures (4), based on results from the NIST 1849a SRM (Table 3). The *p*-value of 0.32 indicates that no bias against the certified value was found. As expected, placebo samples not fortified with vitamin D returned negligible results.

The method has demonstrated its compliance with the applicability statement of vitamin D SMPR, 2011.004 (5), and has been demonstrated to be suitable for the analysis of vitamin D in a wide range of infant formulas and nutritional products, as illustrated with the range of different matrices used in the SLV study (6) and this MLT study.

A summary of each laboratory's performance was sent to participants, along with an invitation to make comments on the performance of the method in their laboratory. In general, comments were positive with respect to the ease of use of the method. Lab 3 and Lab 9 found that the centrifugation of the samples at 250 × *g* did not give a good separation between the two layers and recommended that a higher centrifuge speed be used. It was noted by Lab 4 that the drying step for acetone was not necessary and did not use it. This had also been noted by the authors and all participants were advised not to dry acetone during the MLT study. Lab 6 used vials in a heating block rather than boiling tubes in a water bath because of limitations of equipment and the results did not appear to have been compromised.

Safety concerns with this method were evaluated and there were no major hazards beyond those typically found in chemistry laboratories. Users of the method are directed to use appropriate safety equipment when handling acids, bases, and solvents, and to refer to Material Safety Data Sheets for detailed safety instructions for each chemical used.

The described protocol provides an accurate, precise, rapid, and robust method for the analysis of vitamin D that is suitable both for routine compliance testing and as a reference method to demonstrate product compliance against global regulations.

Conclusion

An MLT study of AOAC First Action 2016.05, an LC–MS/MS method for the analysis of vitamin D_2 and vitamin D_3 in infant formulas and nutritional products, was undertaken. The method was implemented in 9 laboratories and demonstrated acceptable precision and accuracy.

Recommendation

A study report summarizing the outcomes of this multi-laboratory collaborative study was submitted with the recommendation that AOAC First Action Method 2016.05 be accepted as

a SPIFAN endorsed AOAC Final Action method. The SPIFAN ERP evaluated the study data in March 2017 and endorsed the recommendation, which was subsequently approved by the AOAC Official Methods Board.

Acknowledgments

The Study Director would like to thank the following laboratories and individuals for their participation in this study: Andrew Todd and Mark Reynolds (Fonterra, New Zealand); Carolyn Burdette and Melissa Phillips (NIST, USA); Danny Samson and Maurice Seegers (Mead-Johnson, Netherlands); Emeline Tissot, Frédéric Martin, and Esther Campos-Giménez (Nestlé, Switzerland); Greg Hostetler and Steve Tennyson (Perrigo Nutritionals, USA); Isabelle Malaviole and Marlène Daminato (Aquanal, France); Shane Wei (Fonterra, New Zealand); Tiffany Gallegos-Peretz and Sneh Bhandari (Merieux Nutrisciences); Vicky Manti (Danone Nutricia, Netherlands); Yang Zhou (Eurofins, USA).

The assistance of Melissa Phillips (NIST, USA), for the supply of NIST 1869, included as part of this study, and Shellie Stassi (Covance, USA), for arranging the shipping of the SPIFAN kit to participating laboratories, is greatly appreciated.

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Table 1. Tabulated raw data for replicate analyses of SPIFAN kit

Lab # [a]	Vitamin D_3, µg hg^{-1}											
	SRM NIST 1849a				Partially hydrolyzed soy-based infant formula powder		Infant elemental powder		High-protein adult nutritional ready-to-feed		Soy-based infant formula	
3	10.78	10.19	10.52	10.05	7.89	8.93	9.21	8.96	0.97	0.98	11.54	10.36
4	9.92	10.55	10.61	10.51	8.69	9.11	8.02	7.95	0.97	0.94	10.28	10.45
6	10.69	10.94	10.48	10.85	9.13	9.53	8.61	8.29	1.10	1.08	10.62	10.33
7	10.22	10.58	10.13	10.28	9.07	8.83	8.33	8.53	0.98	0.92	9.81	10.02
8	10.04	10.13	10.09	9.87	8.11	8.26	8.04	8.14	0.93	0.92	9.76	9.64
9	11.11	11.73	12.18	11.34	9.22	9.74	9.07	9.00	1.21	1.11	11.43	10.77
10	9.83	9.39	9.44	9.46	8.26	8.38	7.50	7.52	0.85	0.94	9.38	9.45
11	9.58	9.12	9.27	9.61	8.49	8.21	8.03	7.64	1.02	0.89	8.78	9.77
12	9.60	8.92	10.20	10.14	7.34	6.90	7.40	6.87	0.77	0.74	7.82	7.54

[a] Data for Labs 1, 2, and 5 not submitted prior to MLT submission deadline.

Table 1 (continued). Tabulated raw data for replicate analyses of SPIFAN kit

Lab # [a]	Vitamin D_3, µg hg^{-1}											
	High-protein adult nutritional ready-to-feed (unfortified)		Infant formula powder		High-fat adult nutritional ready-to-feed (unfortified)		Milk-based infant formula ready-to-feed		High-fat adult nutritional ready-to-feed		Milk-based infant formula ready-to-feed (unfortified)	
3	0.00	0.00	9.10	9.00	0.00	0.00	0.70	0.72	1.30	1.34	0.00	0.00
4	0.02	0.01	8.61	8.86	0.10	0.16	0.68	0.69	1.38	1.24	0.02	0.01
6	0.00	0.00	8.70	9.00	0.00	0.00	0.72	0.71	1.39	1.46	0.00	0.00
7	0.01	0.00	8.59	8.71	0.01	0.02	0.67	0.66	1.26	1.28	0.00	0.00
8	0.00	0.00	7.82	7.93	0.00	0.00	0.67	0.65	1.19	1.16	0.00	0.00
9	0.00	0.00	8.44	8.87	0.08	0.05	0.81	0.73	1.30	1.24	0.01	0.01
10	0.00	0.00	7.95	7.92	0.02	0.02	0.59	0.59	1.23	1.25	0.00	0.00
11	0.00	0.00	8.57	7.44	0.00	0.00	0.63	0.68	1.23	1.27	0.00	0.00
12	0.00	0.00	7.66	6.84	0.00	0.00	0.51	0.52	1.18	1.13	0.00	0.00

[a] Data for Labs 1, 2, and 5 not submitted prior to MLT submission deadline.

Table 1 (continued). Tabulated raw data for replicate analyses of SPIFAN kit

Lab # [a]	Vitamin D_3, µg hg^{-1}											
	Milk-based child formula powder		Partially hydrolyzed milk-based infant formula powder		Child elemental powder		Milk-based infant formula powder		FOS/GOS[b]-based infant formula powder		Infant elemental powder (unfortified)	
3	7.93	7.75	9.05	8.86	9.60	9.36	10.35	10.63	7.16	7.44	0.00	0.00
4	8.32	8.09	9.29	9.27	8.94	9.28	10.36	10.25	7.17	7.00	0.50	0.30
6	8.21	8.65	9.25	9.35	9.36	9.15	10.58	10.67	7.01	6.83	0.00	0.00
7	7.36	7.37	9.04	8.76	9.06	9.24	10.11	10.23	6.95	6.66	0.01	0.04
8	7.78	7.78	8.32	8.41	8.72	8.44	9.77	10.03	6.70	6.77	0.00	0.00
9	7.26	7.52	9.90	9.63	9.87	9.67	11.86	10.78	8.09	7.93	0.03	0.03
10	7.50	7.50	8.47	8.39	8.40	8.32	9.48	9.61	6.57	6.49	0.01	0.00
11	6.82	6.77	8.85	7.75	7.02	7.45	8.78	9.79	5.75	6.23	0.00	0.00
12	7.43	7.64	8.32	8.29	8.09	7.41	8.10	9.66	7.04	5.80	0.00	0.00

[a] Data for Labs 1, 2, and 5 not submitted prior to MLT submission deadline.

[b] FOS—Fructooligosaccharide, GOS—Galactooligosaccharide.

Table 1 (continued). Tabulated raw data for replicate analyses of SPIFAN kit

Lab # [a]	Vitamin D_3, µg hg^{-1}				Vitamin D_2, µg hg^{-1}			
	Milk-based child formula powder (unfortified)		Low-fat adult nutritional powder		Candidate SRM NIST 1869			
3	0.00	0.00	3.20	3.02	15.07	14.76	13.14	14.05
4	0.06	0.04	3.74	3.55	13.62	14.49	13.75	14.44
6	0.00	0.00	3.28	3.56	*21 09	*18.01	14.24	14.10
7	0.01	0.02	3.35	3.45	12.79	12.80	13.19	12.72
8	0.00	0.00	2.64	2.83	13 20	13.34	12.60	12.64
9	0.03	0.04	4.06	3.81	16.75	17.54	17.61	17.34
10	0.00	0.01	3.15	3.13	14.30	13.30	13.30	13.40
11	0.00	0.00	2.98	2.83	15.44	15.49	13.06	11.93
12	0.00	0.00	2.89	3.25	10.49	11.14	12.21	11.99

[a] Data for Labs 1, 2, and 5 not submitted prior to MLT submission deadline.

* Results removed as Cochran outlier prior to precision calculation.

AOAC SMPR 2011.004

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

Intended Use: Global Dispute Resolution Method

1 Applicability

Determination of total vitamin D_2 and vitamin D_3 in all forms (powders, ready-to-feed liquids, and liquid concentrates) of infant, adult, and pediatric nutritional formulas. For the purpose of this SMPR, vitamin D_2 is defined as ergocalciferol (CAS 8017-28-5) and its previtamin isomer; and vitamin D_3 is defined as cholecalciferol (CAS 67-97-0) and its previtamin isomer.

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 in 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 SD or RSD calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation (%RSD_R).

Table 1. Method performance requirements[a]

Analytical range	0.12–5.1[b]	
Limit of detection (LOD)	≤0.02[b]	
Limit of quantitation (LOQ)	≤0.12[b]	
Repeatability (RSD_r)	0.12–1.5[b]	≤15%
	>1.5[b]	≤11%
Recovery	0.12–1.5[b]	80–120%
	>1.5[b]	90–110%
Reproducibility (RSD_R)	≤15%	

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

[b] µg/100 g expressed separately as vitamin D_2 and vitamin D_3 in

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

See Table 1.

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 analytical range. A control sample must be included.

6 Reference Material(s)

National Institute of Standards and Technology 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. Certified value of vitamin D_3 in NIST 1849 is 0.251 (±0.027) mg/kg vitamin D_3.

7 Validation Guidance

Recommended level of validation: *Official Methods of Analysis*SM.

8 Maximum Time-to-Signal

No maximum time.

Approved by Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN). Final Version Date: April 5, 2011. Effective Date: June 29, 2011.

AOAC INTERNATIONAL

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MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item Va - Update on the AOAC Committee on Safety**

Yvonne Salfinger will present the Committee revised draft Terms of Reference and provide a verbal update to OMB on the Committee on Safety.

AOAC INTERNATIONAL

TERMS OF REFERENCE

I. NAME:

COMMITTEE ON SAFETY

II. MISSION:

To promote an awareness of safety and health matters within the AOAC membership and to give guidance in that area with particular emphasis on the integration of safety into the methods development process.

III. RESPONSIBILITIES:

To review all methods in process, as Methods Committee Safety Advisors, in order to integrate safety in methods.

To serve as a resource for AOAC membership with regard to safety matters.

~~To submit information on laboratory safety to The Referee.~~

All Committee members, other than those holding *ex officio* appointments, serve at the pleasure of the President. All Committee members are expected to actively participate in the work of the Committee; including, but not limited to, promptly responding to communications, attending and actively participating in meetings, reviewing meeting background materials and agendas prior to meetings, and accepting and following through on assignments. Persons who do not actively participate will be removed from the committee by the President at the request of the chair.

IV. COMPOSITION AND ORGANIZATION:

There shall be a minimum of 5 and a maximum of 12 members, including the chair and past chair. A member shall be appointed for a three-year term, with no maximum limitation. The chair is appointed for one three-year term and may serve one additional year on the Committee as immediate past chair.

All members of the Committee are appointed by the President Elect and assume office immediately following the Annual Meeting.

The Committee shall be composed of members representing a balance of government, industry, and academia as appropriate to the scope of the

Committee. No more than one-half of the members may be from a single agency. Less than one-half of the members must be from industry.

Subcommittees, task forces, and other appropriate subgroups shall be appointed as the needs arise.

V. STAFF LIAISON:

The Executive Director shall assign a member of the staff to serve as staff liaison.

VI. REVIEW SCHEDULE:

Every three years.

VII. DATE ESTABLISHED:

1965

VIII. DATES REVISED:

8/91; 3/99; 9/99

Method Safety & Risk Assessment Guide

- Methods submitted to AOAC *Official Methods*℠ Program are subject to undergo a risk assessment.
  - Appropriate safety instructions (in general or specific terms) must be included in the method if there is a likelihood of exposure to actual or potential hazards when using the method.

- Method authors should complete the safety checklist to assess and expose potential safety hazards. Expert review panels will review methods for safety and all potential or actual hazards must be addressed as a requirement for Final Action *Official Method*℠ status. A safety advisor can serve a resource to address any outstanding concerns.

- The method submitter or Expert Review Panel should make every attempt to be proactive in providing the suitable wording and documentation to address the potential or actual safety hazard.

  - Safety advisors reviewing a method that lacks safety precautions and a suitable wording concerning safety should be suggested for inclusion in the text.
    - May suggest appropriate wording or require additional information.
    - Must clearly state objections if not recommending the method to move forward in the review process until the safety concerns are satisfactorily addressed.

- For methods that contain numerous hazards, the text may be best improved with a comprehensive safety statement, prominently displayed early in the method, e.g. in the materials and methods section of the text A text hyperlink such as http://www.ilpi.com/msds/ may be advantageous, as it provides the user with up-to-date pertinent safety information.

- http://www.cdc.gov/biosafety/publications/bmbl5/index.htm/ is the "Biosafety in Microbiological and Biomedical Laboratories" 2009 Manual. For microbiology methods, it describes the hazardous nature of many pathogens, together with their biosafety level requirements.

- Methods that contain a small number of specific safety hazards may best be improved with a caution in the text immediately following the first mention of the hazard.

  - For example (a modified version of some of the text below may be appropriate):
    - Use effective ventilation equipment when fumes or aerosols are generated.
    - Keep skin exposure to ultraviolet radiation to a minimum.
    - Conduct reactions behind a safety barrier. Wear face shield and gloves.
    - Wear skin, eye, and respiratory protection when handling.
    - Corrosive substance.
    - Biosafety containment level (1, 2 or 3) required with pathogen.
    - Microbiologically contaminated liquid or solid wastes should be sterilized.
    - See Appendix B of the OMA
    - See ~~M~~SDS information for safety precautions.
    - See "Biosafety in Microbiological and Biomedical Laboratories" Manual (2009)

- Following revision, the method should alert the user to hazards / potential hazards in a general or specific way, whichever is considered most appropriate and effective.

Online Technical Resources

Method Development, Optimization & Validation

- OMA - Appendix F - Guidelines for Standard Method Performance Requirements
- Homogeneity
- Guide for Writing Methods in AOAC Format
- Statistics Protocol Review Form
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis
- OMA - Appendix I: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Biological Threat Agent
- Methods and/or Procedures
- OMA - Appendix J: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Microbiological Methods for Food and Environmental Surfaces
- OMA - Appendix K: Guidelines for Dietary Supplements and Botanicals
- OMA - Appendix L: AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation
- OMA - Appendix M - Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices
- Safety Checklist

Method Review

- Examples of Statistical Analysis
- Statistics Manuscript Review Form
- OMA - Appendix A: Standard Solutions and Reference Materials
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix H: Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods

Miscellaneous

- Definition of Terms and Explanatory Notes
- OMA - Appendix B: Laboratory Safety
- OMA - Appendix E: Laboratory Quality Assurance
- OMA - Appendix C: Reference Tables

All resources are accessible at
http://www.aoac.org/vmeth/guidelines.htm

For questions, please contact:
P 301-924-7077 x157 E dmckenzie@aoac.org

Revised October 2013

Safety Checklist Questions

- ✓ Are any materials used or compounds formed that are explosive or flammable?
- ✓ Are there any side reactions that could occur that might produce flammable or explosive products or conditions?
- ✓ Are there any hazards created from electric or mechanical equipment?
- ✓ Are pressure differentials created that could result in an explosion or implosion?
- ✓ Are any substances used or formed which are:
  - o radioactive?
  - o carcinogenic?
  - o mutagenic?
  - o tetratogenic?
  - o abortogenic:?
  - o otherwise a significant health hazard?
- ✓ Would there be increased hazards if the reaction temperature were increased even modestly?
- ✓ Are special procedures required if a spill of the reaction mixture occurs?
- ✓ Is there a risk in producing a dangerous aerosol?
- ✓ Are special procedures required for the disposal of reagents or reaction products?
- ✓ Are there any organisms and/or their products used/present that are:
  - o Pathogenic?
  - o allergenic?
  - o carcinogenic?
  - o mutagenic?
  - o tetratogenic?
  - o otherwise a significant health hazard?
- ✓ Are there any potential hazards in handling or storage of reagents, test samples, or standards?
- ✓ Are there any other hazards that should be addressed regarding the method?
- ✓ Does your method use chlorinated solvents?
- ✓ If "yes" to question13, have non-chlorinated solvents equivalent to chlorinated solvents been investigated?
- ✓ Include appropriate precautionary statements in method write-up.
- ✓ Provide specific information on hazard (MSDS or other supporting documentation)

Online Safety Resources

Useful source information concerning safety hazards is available in:

- Official Methods of Analysis of AOAC INTERNATIONAL Appendix B: http://www.eoma.aoac.org
- US Department of Labor / Occupational Health and Safety Administration (OSHA): http://www.osha.gov/web/dep/chemicaldata/default.asp
- American Chemical Society / Chemical Abstracts Service: http://www.cas.org/
- MSDS Solutions Centre MSDS online : ~~http://www.ilpi.com/msds/~~ ~~http://www.ilpi.com/msds/#Manufacturers~~ http://www.msds.com/
- Biosafety information: http://www.cdc.gov/biosafety/publications/bmbl5/index.htm/
- Public Health Agency of Canada MSDS for pathogens: http://www.phac-aspc.gc.ca/lab-bio/res/psds-ftss/index-eng.php
- ~~IATA transportation information for hazardous materials~~ http://www.iata.org/whatwedo/cargo/dangerous_goods/Pages/index.aspx

Examples of Caution Statements

For example (a modified version of the text given below may be appropriate):

Caution - This procedure uses substances that are neurotoxic, corrosive and hazardous. Care should be taken to avoid ingestion or contact with the skin. Laboratory personnel should follow normal laboratory safety precautions and have ready access to the ~~material~~ safety data sheets (~~MSDS~~, http://www.ilpi.com/msds/) for all hazardous substances used in the test procedure, should work in a well ventilated environment and be provided with appropriate safety protection including clothing, protective gloves and appropriate eye protection (http://www.eoma.aoac.org).

Caution - This procedure uses / detects pathogenic microorganisms and / or their metabolic products. Care should be taken to avoid ingestion, inhalation of potentially infectious aerosols, or contact with the skin. Laboratory personnel should follow normal laboratory safety precautions and have ready access to the appropriate material safety data sheets (~~MSDS~~, http://www.phac-aspc.gc.ca/lab-bio/res/psds-ftss/index-eng.php) should use the appropriate biosafety containment (http://www.cdc.gov/biosafety/publications/bmbl5/index.htm/) and be provided with appropriate safety protection including clothing, protective gloves and appropriate eye protection http://www.eoma.aoac.org.

Revised October 2013

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Sidney Sudberg, Chair – Committee on Statistics / OMB Member**

Subject: **Item Vb – Summary of Activities for the AOAC Committee on Statistics**

1. Progress updates
    a. Statistics for Microbiology methods validation,
        1. Testing/Validating of the new spreadsheets for both Qualitative & Quantitative calculations actively moving forward
    b. Changes to Appendix J (after1.a.1. above complete)
        1. Quantitative methods need changes (performed during &/or after validation)
            1. Candidate minus Reference
            2. New method of calculating the CI for the paired DPod
            3. Use of Z (n) vs. t (n-1)
            4. Removal of reference to Welch/Satterthwaite t-test?
            5. Chi-Square Test Calculator http://lcfltd.com/AOAC/aoac-binary-v2-5.xlsx
    c. Revising of Stat Committee's Terms of Reference (TOR)
        1. Committee should have something to review
    d. Intermediate Precision (IP) Working Group:
        1. Appropriate applicability for use of IP: Completed
            1. SLV
            2. First Action
        2. Committee on Statistics to produce a separate appendix &/or Glossary, i.e. remove statistical details from various appendices?
            1. Appendices J, K & others
    e. Appendix K Revision to begin:
        1. Terms, Definitions & its' Measurement need to be defined
            1. Intermediate Precision
            2. LOD/LOQ
    f. Proficiency Testing Data for:
        1. Collaborative Studies: Chondroitin Collab Study
        2. Validation
        3. TDRM interaction & the use of qualified Reference Standards
    g. Incremental Collab Studies

1. Currently being worked on & reported on in Atlanta in September at our Symposium

h. Method Equivalency: TR 343
    1. Will be discussed in Atlanta in September at our Symposium

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods Board

Subject: Item Vc – AOAC OMB New Member Selection Committee

Only one member of the OMB has a second term that expires in September 2017. To consider new candidates, Shauna Roman proposes that the list of candidates from last year be considered. Please see the List of Candidates below. Roman has reached out to all the candidates on the list and only one of eight (8) candidates has declined in being considered. The working group, chaired by Shauna Roman also consists of Yvonne Salfinger and Melissa Phillips who volunteered during the February meeting. The working group will convene after this meeting to consider the OMB's composition/needs and ultimately bring forward a candidate(s) for OMB's recommendation. Additionally, the working group will consider updates to the policy document for OMB's consideration.

After the OMB makes agrees on a recommended new person, staff will draft the proposed OMB roster for 2017-2018 and will present it to the Board of Directors for their review and appointment.

List of Candidates is as follows:

1. John Austad, Covance Laboratories
2. Gabriel Giancaspro, US Pharmacopeia
3. Adrianne Klijn, Nestle Research Center
4. Dawn Mettler, Rockbridge Laboratories
5. Catherine Rimmer, US NIST
6. Eric Verdon, ANSES
7. Jane Weitzel, Independent Consulting
8. Jon Wong, US FDA

Enclosures:

1. AOAC Policy for New OMB Member Selection

Process for Selecting Members of the Official Methods Board (OMB)

The process begins with the OMB Search Committee.

Composition

The Search Committee shall consist of three (3) members: two members of the current OMB and - the Immediate Past Chair of the OMB who shall serve as chair of the Search Committee.

Purpose

The objective of the Search Committee is to identify and recommend a slate of nominees as potential candidates for membership on the OMB. They shall seek candidates from such sources as the Association Membership, the Communities, and Stakeholders Groups. The OMB will select a nominee from this slate.

Process

Criteria for Member of the OMB

- Must provide a current Curriculum Vitae;
- Must be a member of AOAC INTERATIONAL in good standing;
  - Must have a letter of support from the sponsoring organization [employer/supervisor]
  - Must have an executed AOAC Volunteer Acceptance Form
  - Must provide two letters of recommendation from someone other than an employee, employer or supervisor.
- Should be willing and capable of acting as a Liaison with the standards development and conformity assessment bodies~~Communities~~, Technical Divisions, Research Institute, and other major Stakeholders~~.~~;
- Should possess the minimum of a Bachelor's degree in chemistry, biology, mathematics or a related scientific field;
- Should demonstrate technically competent written and oral communication and networking skills;
- Should demonstrate leadership capabilities through documentation of project management, supervisory experience, or leadership positions within AOAC;
- Should have experience in the AOAC ~~collaborative study process~~standards development and method approval processes;
- Should be familiar with the AOAC ~~Program Manual~~Conformity Assessment programs and the Official Methods of Analysis appendices; and
- Should have successfully completed OMB training in the ~~method validation~~standards development and conformity assessment processes, demonstrate ability to perform adequate review of AOAC methods, ~~collaborative studies~~, and agree to appropriate retraining at least every three years.

Appointment of the Candidate

The nominee shall be contacted by the Chair of the OMB to confirm his/her willingness and ability to serve. Once confirmation has been received, the ~~nominee~~ recommended OMB roster shall be presented to the Board of Directors for their approval and subsequent appointment(s) as needed by the President of the Association.

Composition of The Official Methods Board

The OMB shall be composed of the Chair, Vice Chair, the Chair of the Committee on Safety, the Chair of the Committee on Statistics, and up to 9 more members not to exceed a total of 13 members at any given time. The 9 appointed members are to represent a balance of government, industry, and academia as appropriate to the needs of the Association. No more than one-half of the members of the OMB may be from a single agency and no more than one-half of the members may be from industry.

PROCESS FOR SELECTING THE VICE CHAIR OF THE OFFICIAL METHODS BOARD (OMB)

The process begins with the OMB.

Criteria for the Vice Chair of the OMB

- Must have served for at least one year as a Member of the OMB
- Must fulfill all the criteria for a Member of the OMB

The members of the OMB serve as the search committee for a Vice Chair. They identify and recommend a slate of nominees as potential candidates for Vice Chair. The nominees shall be contacted by the Chair of the OMB to confirm his/her willingness and ability to serve. Once confirmation has been received, the nominee(s) will be presented to the OMB for a vote. An email ballot shall be sent out to the members of the OMB with the slate of nominees. The current Vice Chair collects and tallies the ballots.

The selection of the Vice Chair will be decided by at least a majority vote of the OMB. If there is a tie, the Chair will cast the determinative vote. If no one receives a majority vote, another email ballot will be sent out with the top two nominees who received the highest number of votes.

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item VIa – Conformity Assessment Training and Education**

McKenzie will provide a verbal update.

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods

Subject: Board Item VIb – Volunteer Confidentiality and Nondisclosure

It was brought to my attention that volunteers do not sign off on maintaining confidentiality with review of methods and protocols. The Performance Tested Methods (PTM) program has an agreement that reviewers and staff can sign; however, there is no such thing witn OMA.

Since then, I realize just how much intellectual information is shared. I would like to discuss how we go about encouraging more nondisclosure without impacting transparency and the expert intellectual discussions groups need to make decisions.

AOAC *Official Methods of Analysis*

An Orientation of Sole Source Method Modifications

Deborah McKenzie רב
Sr. Dir., Standards Development
AOAC INTERNATIONAL
Sr. Dir., AOAC Research Institute
Staff Liaison - Official Methods Board

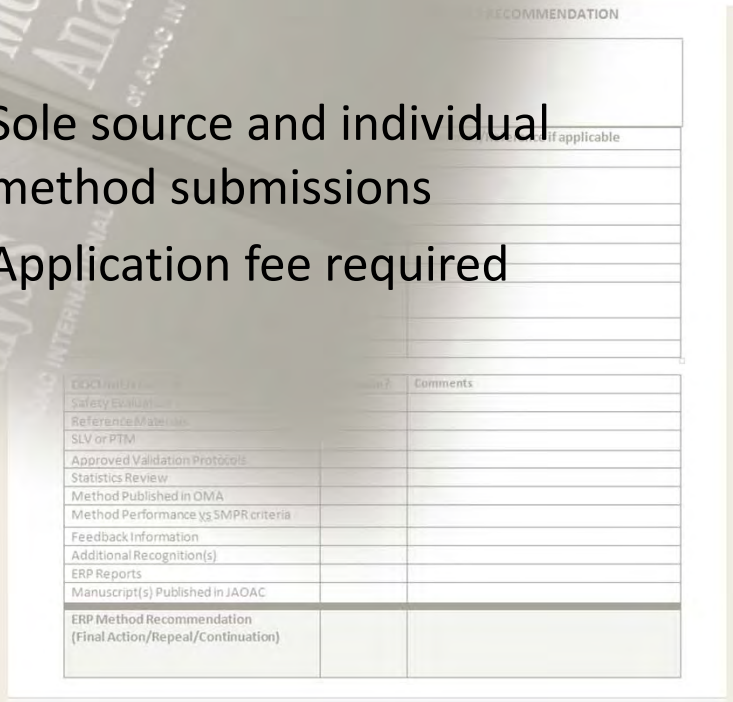
AOAC Method Approval Programs

AOAC INTERNATIONAL

- Administers *Official Methods*^{SM} program based on AOAC standards development activity
- Adoption of methods as *Official Methods* is contingent upon standards development activities
- No application fee required to submit methods in response to Call for Methods
- Method submissions coincide with standards development activities

AOAC Research Institute

- Administers *Official Methods*^{SM} program based on individual submissions
- Sole source and individual method submissions
- Application fee required



AOAC Policies & Procedures

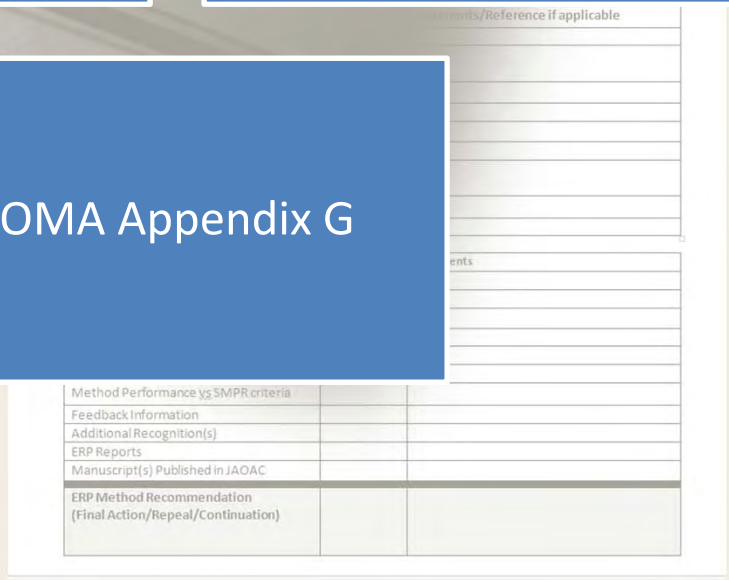
Policy on Antitrust

Policy on Use of Association Name, Identifying Insignia, Letterhead, Business Cards

Policy on Volunteer Conflict of Interest

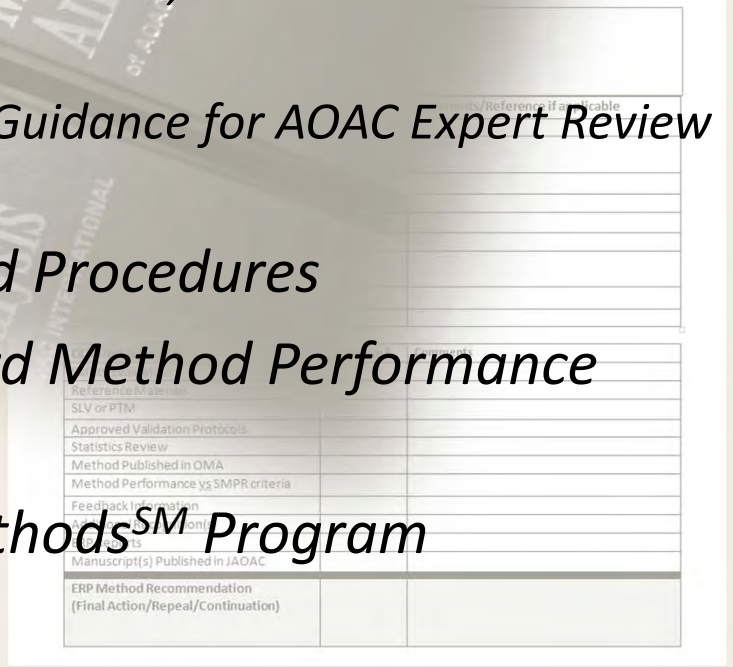
Expert Review Panel Policies and Procedures

OMA Appendix G



Policies and Procedures for Adoption of *Official Methods of Analysis*

- *OMA, Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis*
  - *Expert Review Panels, Official Methods Board, First and Final Action Official Methods*
  - *First Action to Final Action Methods: Guidance for AOAC Expert Review Panels*
- *Expert Review Panels – Policies and Procedures*
- *Appendix F: Guidelines for Standard Method Performance Requirements*
- *OMA, About the AOAC Official Methods℠ Program*



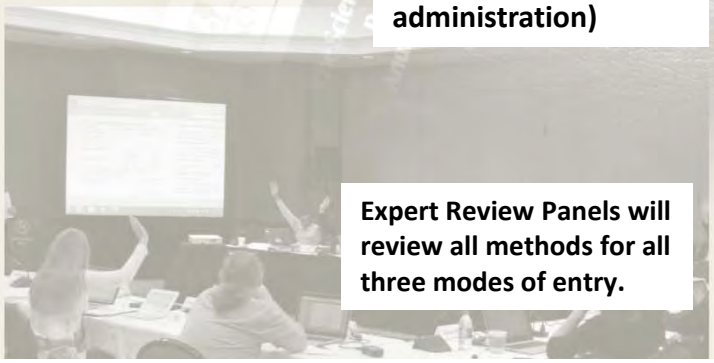
Road to First Action OMA Status

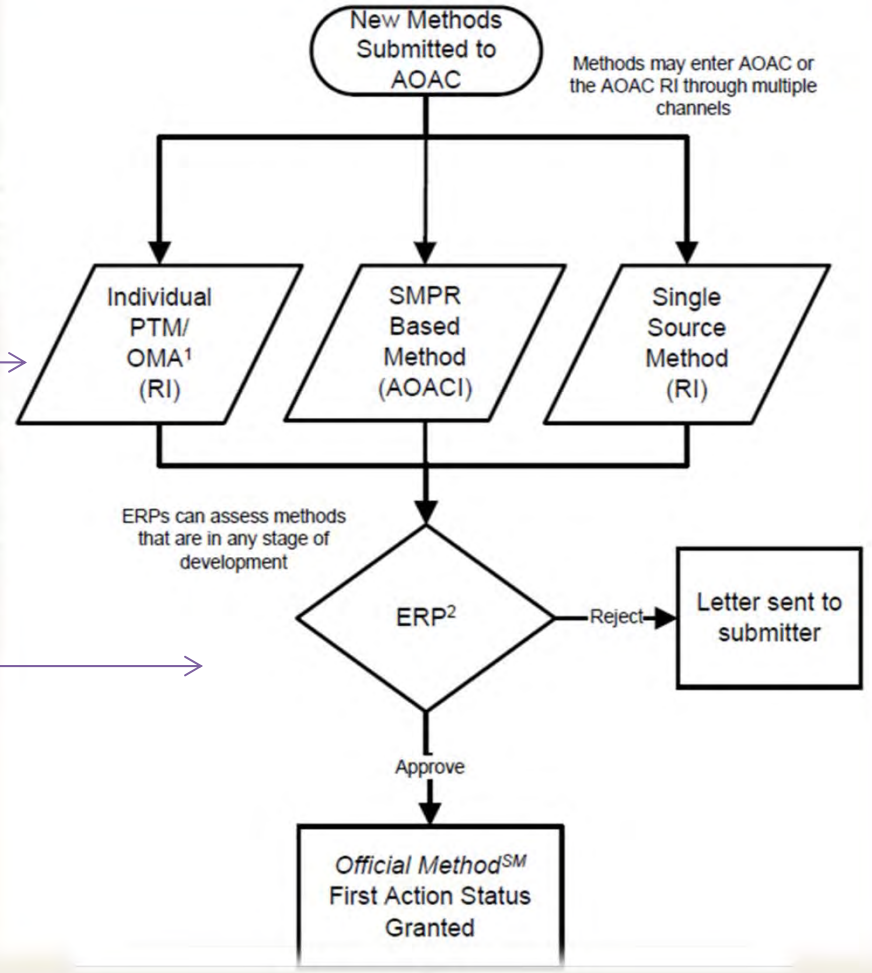
Terms:

- PTM – *Performance Tested Methods℠*
- RI – Research Institute
- ERP – Expert Review Panel
- OMB – Official Methods Board
- SP – Stakeholder Panel
- SMPR – Standard Method Performance Requirement

Three modes of entry and (program administration)

Expert Review Panels will review all methods for all three modes of entry.





Note: Appeals process always available; see Alternative Pathway Guidelines for appeals process.

1 PTM certification previously issued, PTM reviewers will be ERP members
2 Unless otherwise provided for under a contractual agreement, AOAC will regularly convene ERPs twice a year: once during the Mid-Year Meeting and again during the Annual Meeting

Road to Final Action OMA Status

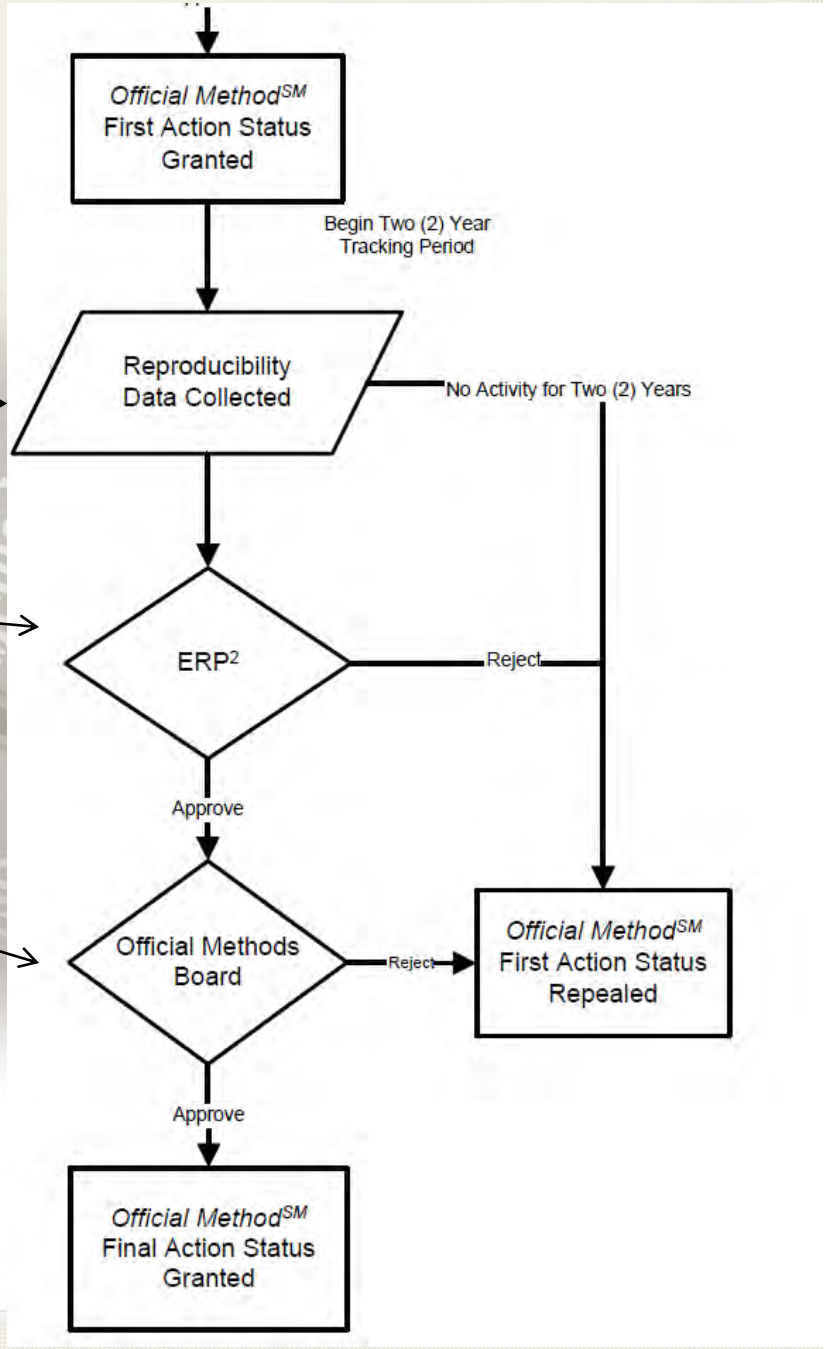
Method reproducibility must be demonstrated before Final Action consideration.

ERP determines if sufficient evidence merits a recommendation for Final Action status or repeal.

•Only the OMB promotes a method to "Final Action" status or repeal the method.

•Methods that did not meet the bar would be repealed.

•Same for all method submissions



Terms:

- PTM – *Performance Tested Methods℠*
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- ERP – Expert Review Panel
- OMB – Official Methods Board
- SP – Stakeholder Panel
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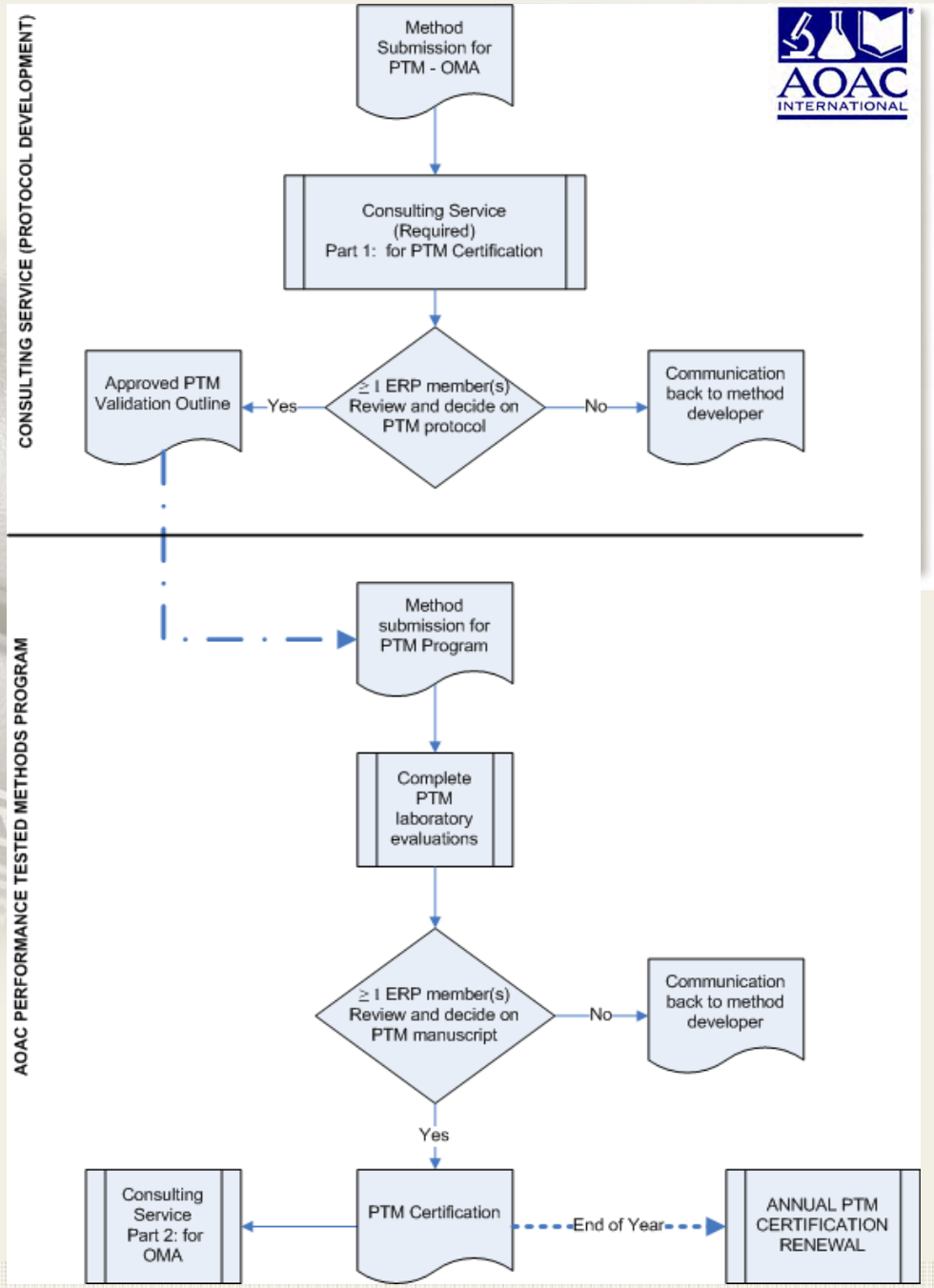
Note: Appeals process always available; see Alternative Pathway Guidelines for appeals process.

1 PTM certification previously issued, PTM reviewers will be ERP members
2 Unless otherwise provided for under a contractual agreement, AOAC will regularly convene ERPs twice a year: once during the Mid-Year Meeting and again during the Annual Meeting

PTM Overview for PTM-OMA Harmonized Process

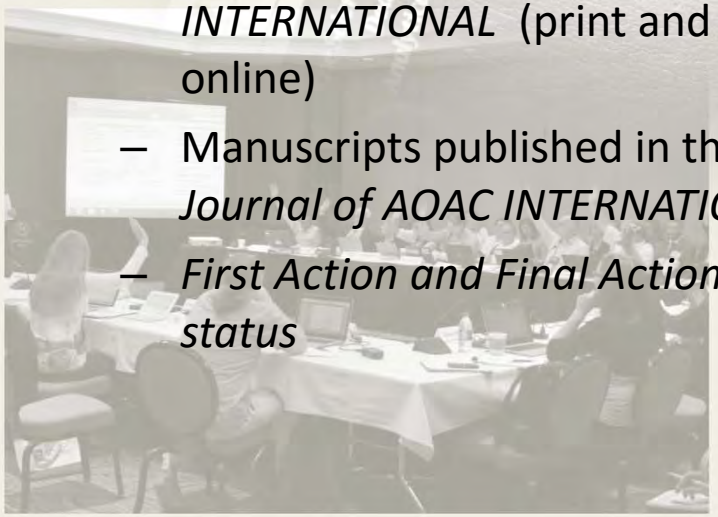
- Administered by the Research Institute in 2003.
- Well established and streamlined
- Original approved by consensus with the OAs, OMB, RI Board of Directors and AOAC INTERNATIONAL Board of Directors.
- ERP may be formed during Consulting Service.
- Criterion for OMA: manufacturer's method claims.



AOAC Method Approval Programs

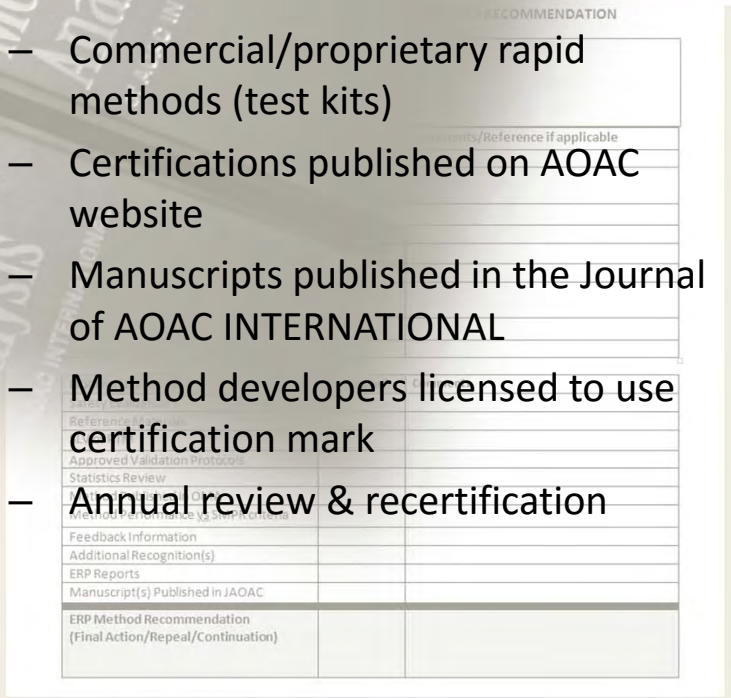
Official Methods of Analysis^{SM} (OMA)

- AOAC's premiere methods program
- Approved methods
  - published in the *Official Methods of Analysis of AOAC INTERNATIONAL* (print and online)
  - Manuscripts published in the *Journal of AOAC INTERNATIONAL*
  - *First Action and Final Action status*



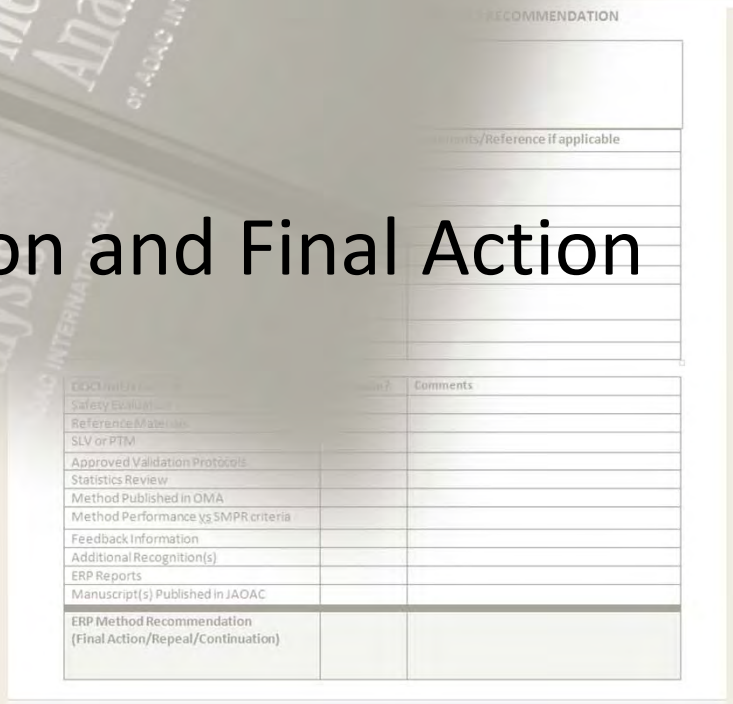
Performance Tested Methods^{SM} (PTM)

- AOAC's method certification program
- Certified methods
  - Commercial/proprietary rapid methods (test kits)
  - Certifications published on AOAC website
  - Manuscripts published in the Journal of AOAC INTERNATIONAL
  - Method developers licensed to use certification mark
  - Annual review & recertification



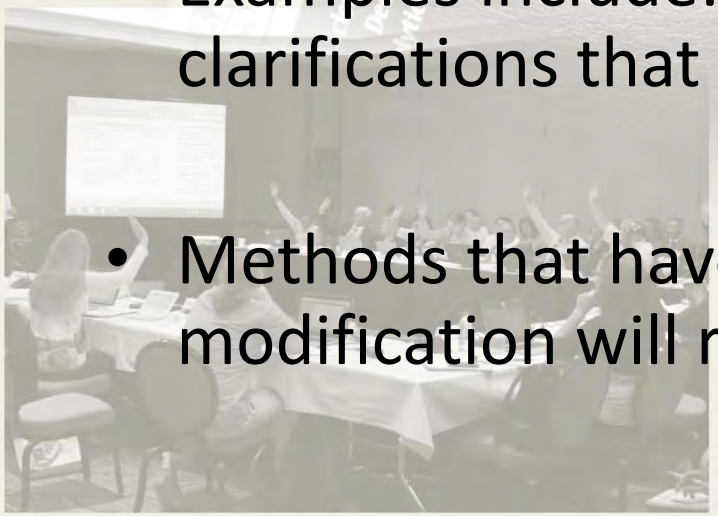
Modifications to Official Methods

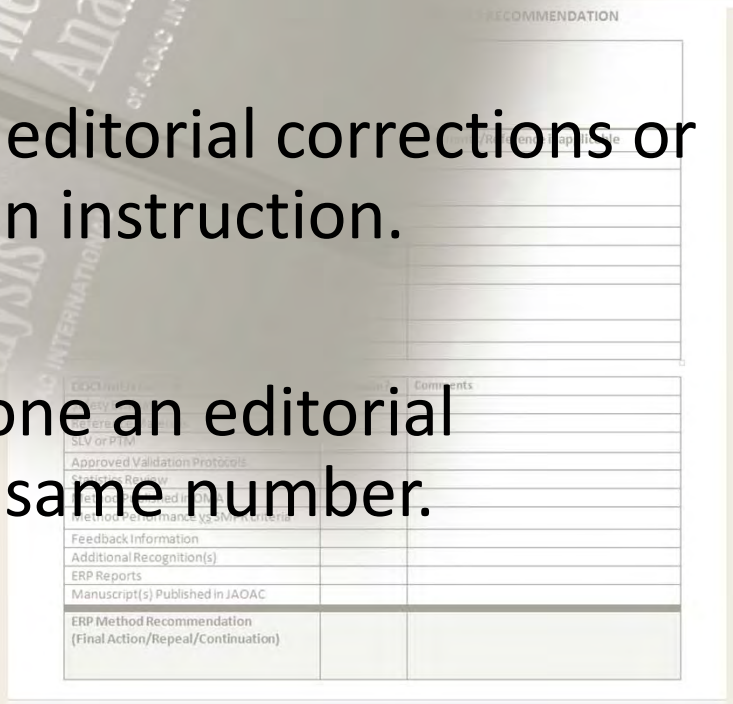
- Types of Modifications
  - Editorial
  - Major
  - Minor
- Applicable to First Action and Final Action OMA
- Relevant to all ERPs



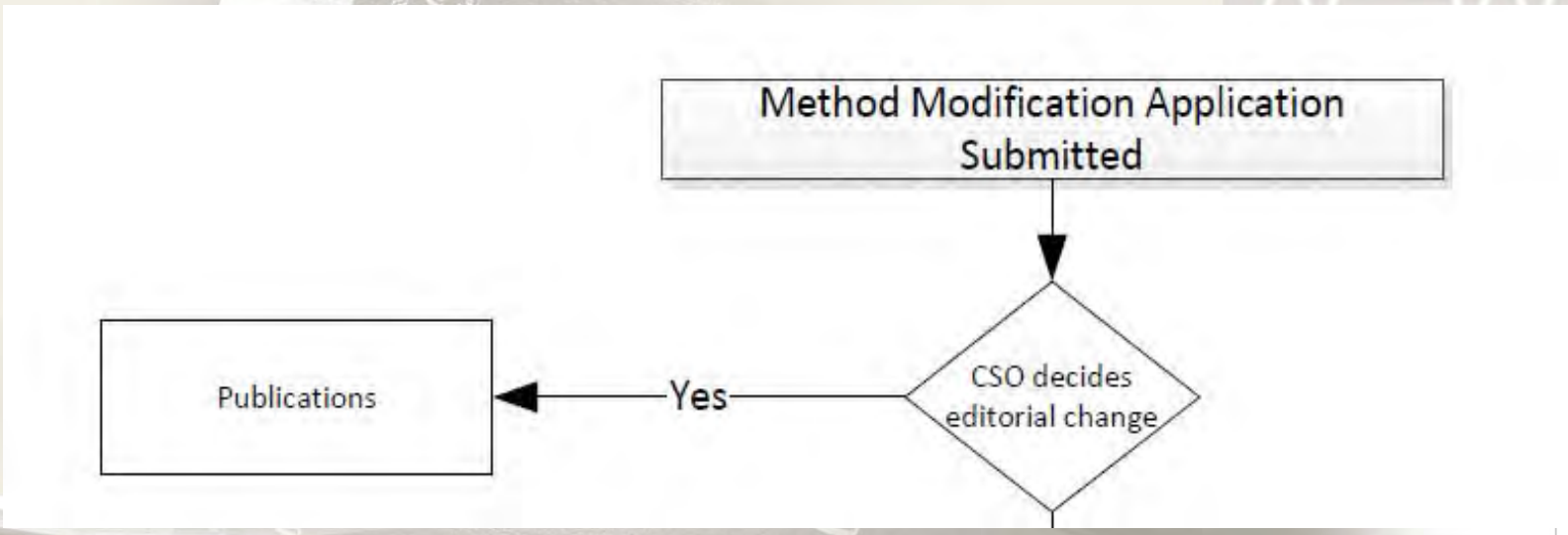
Editorial Modifications

- The applicant must submit a written explanation of the change(s) including a statement that the modification does not alter the validated performance of the method.
- Examples include: Typos or editorial corrections or clarifications that strengthen instruction.
- Methods that have undergone an editorial modification will retain the same number.





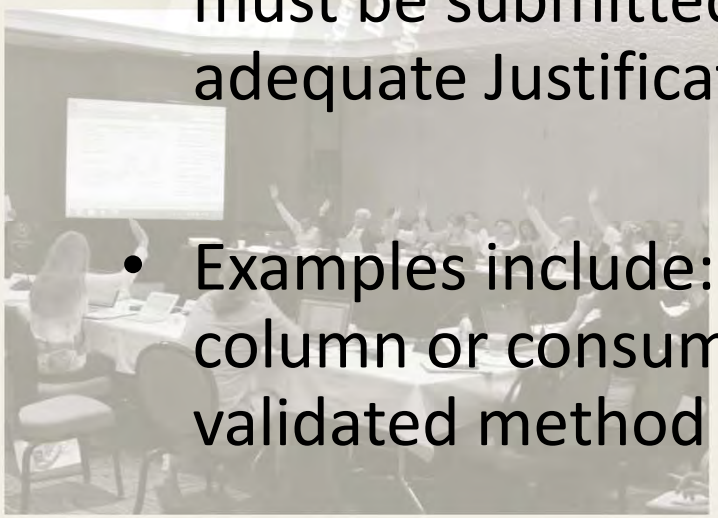
Editorial Changes

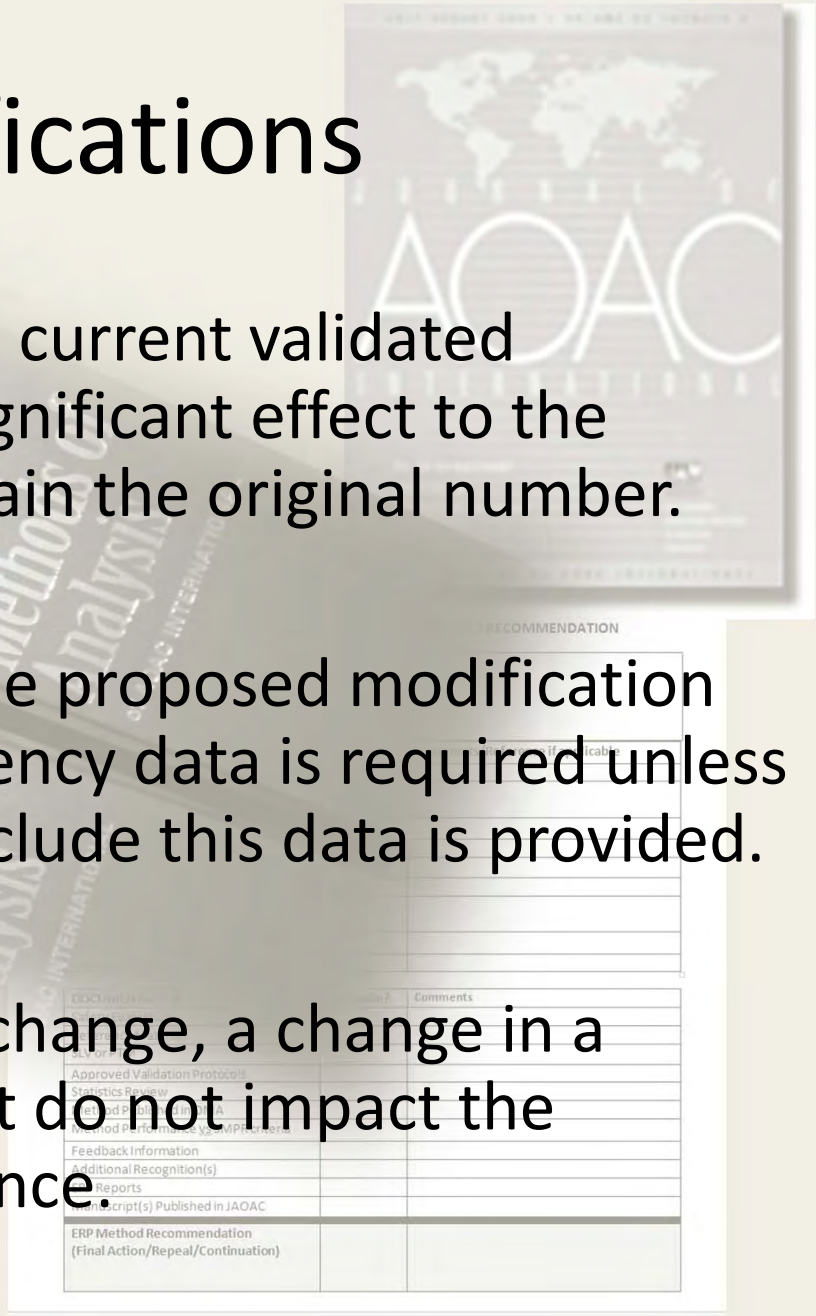


- Editorial changes to methods only require AOAC staff review and the change is made to the OMA with changes noted in next printed edition of OMA.
- A list of the methods with editorial modifications will be published in *Inside Laboratory Management and on* the Website.

Minor Modifications

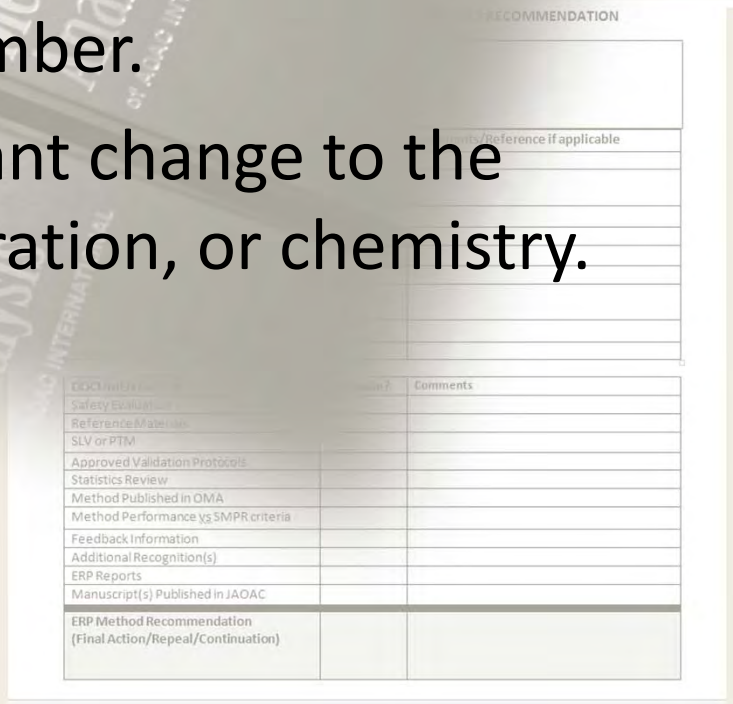
- Results in no changes to the current validated performance. There is no significant effect to the results. The method will retain the original number.
- Supporting data to justify the proposed modification must be submitted. Equivalency data is required unless adequate Justification to exclude this data is provided.
- Examples include: Reagent change, a change in a column or consumables that do not impact the validated method performance.



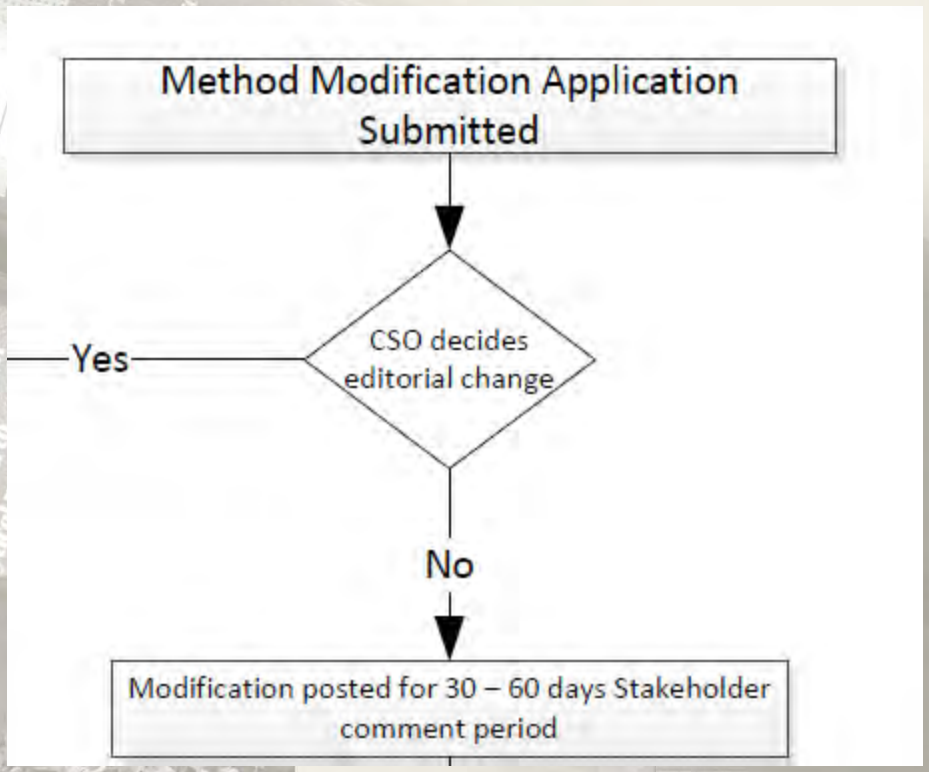


Major Modifications

- Results in a change to the current validated performance of the method.
- This level of modification will result in a new method as part of AOAC standards development and will receive a new method number.
- Examples include: significant change to the technology, sample preparation, or chemistry.

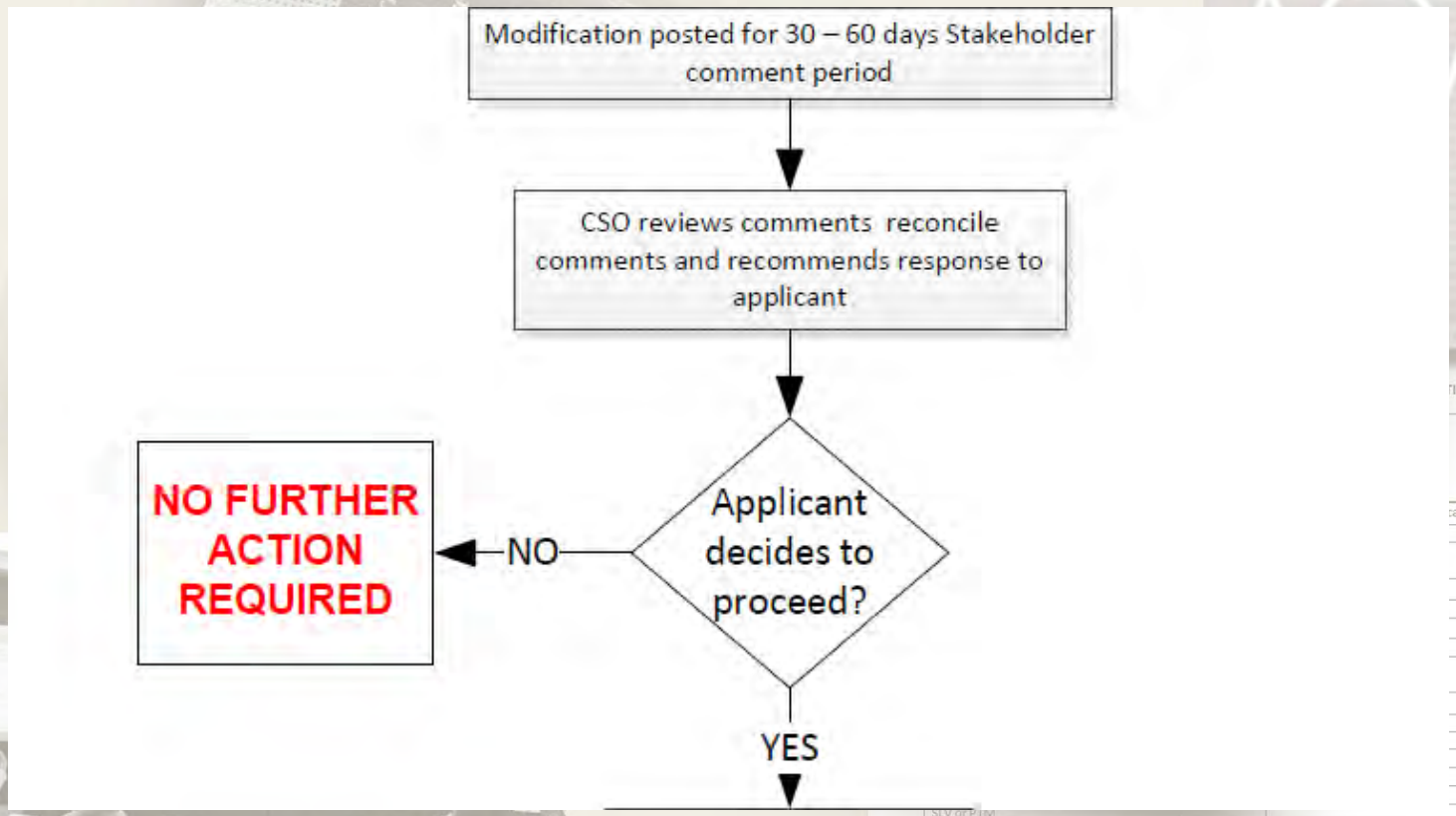


Minor & Major Modifications



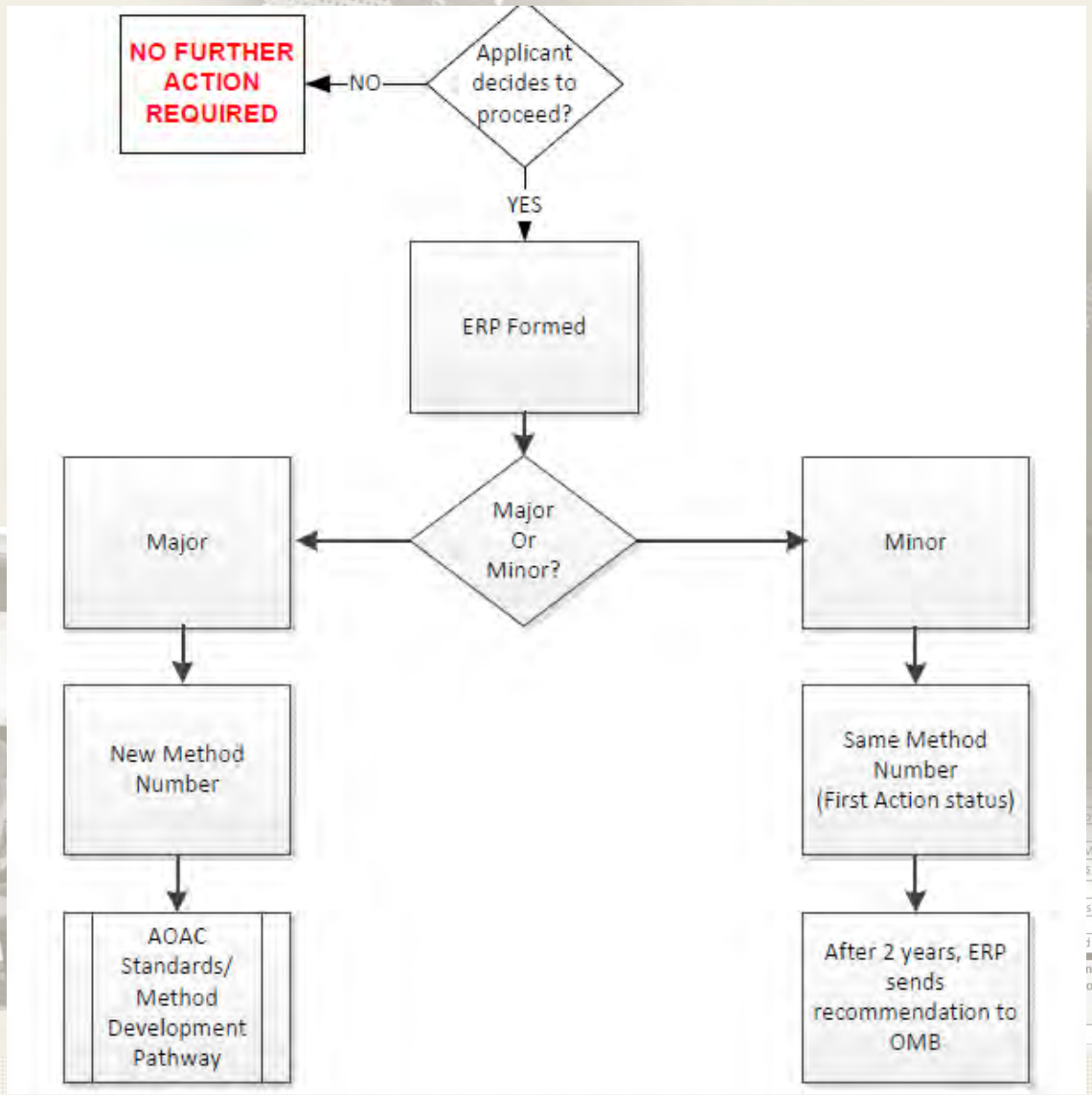
Based on AOAC staff review, a public comment period for the proposed modification is required.

Applicant Options



- Following the comment period, any comments are reconciled and recommends a response to the applicant.
- The applicant can decide to proceed based on the reconciled comments

Pathways for Minor & Major Modification

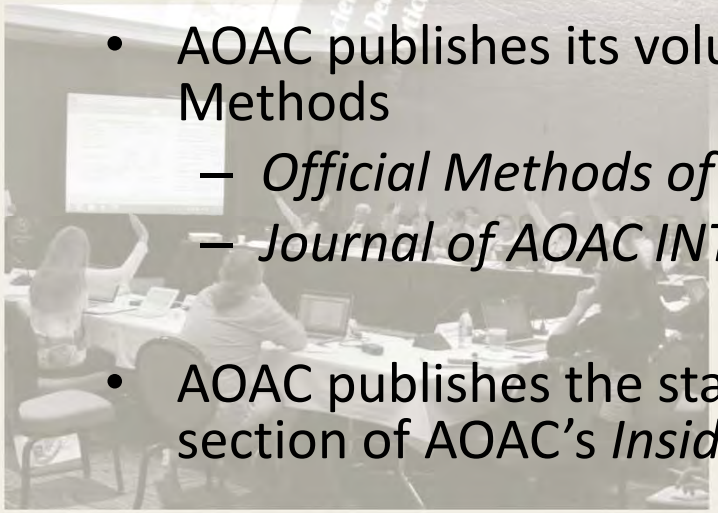


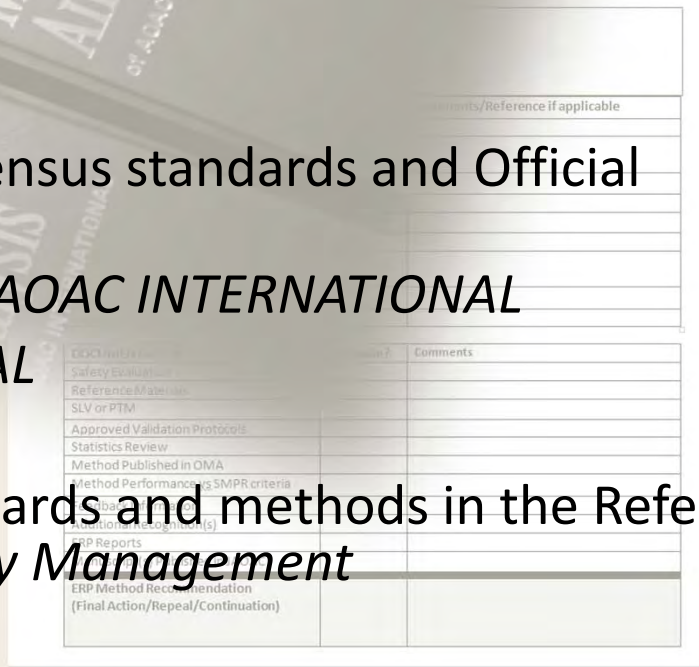
If applicant decides to proceed, an ERP is formed

- Level of modification determined by ERP
- Applies to modifications of First Action and Final Action methods

Documentation and Communication

- AOAC carefully documents the actions of Stakeholder Panel and the Working Groups
- AOAC will prepare summaries of the meetings
  - Communicate summaries to the stakeholders
  - Publish summaries in the *Referee* section of AOAC's *Inside Laboratory Management*
- AOAC publishes its voluntary consensus standards and Official Methods
  - *Official Methods of Analysis of AOAC INTERNATIONAL*
  - *Journal of AOAC INTERNATIONAL*
- AOAC publishes the status of standards and methods in the Referee section of AOAC's *Inside Laboratory Management*





Roles and Responsibilities

AOAC Official Methods Board

- Vet and approve stakeholder panel chair & voting members
- Vet and approve ERP membership and AOAC Experts
- Render decisions on status of First Action methods (Final Action, repeal, etc…)
- Assign a liaison to each stakeholder panel and ERP
- Coordinate OMB Awards

AOAC Expert Review Panels

- Review methods and meet in person to render decisions on methods for First Action Official Methods℠ status.
- Track First Action Official Methods℠ and modify, if necessary
- Recommend First Action methods after 2 years or less to OMB for Final Action, continuance, or Repeal
- Participate in Consulting Service and PTM reviews for OMA and harmonized PTM and harmonized OMA method studies

AOAC Experts

- Review and approve PTM validation testing protocol documentation
- Peer review of PTM validation manuscript and supporting documentation

AOAC Research Institute - PTM Expert Reviewers

- Peer Review of PTM validation manuscripts and supporting documentation

AOAC Research Institute Independent Laboratories

- Conduct independent evaluation of candidate method using AOAC approved testing protocols

AOAC Stakeholder Panels

- Develop voluntary consensus standards
- Assign working groups to draft standards method performance requirements
- Voting members demonstrate consensus on behalf of stakeholders

AOAC Staff

- Coordinate method reviews and method approval activities
- Coordinate OMB meetings
- Provide trainings and orientations
- Maintain website and communication
- Document and publish actions and decisions
- Coordinate standards development activities
- Publish standards and methods

AOAC Research Institute Technical Consultants

- Draft validation protocols in Consulting Service for assigned methods
- Facilitate PTM evaluation of assigned candidate methods
- Facilitate comments/responses for assigned OMA reviews

Questions?

Thank you

☺

ERP – Solids in Syrups (AOAC 932.14)

- Modification to AOAC Official Method 932.14: Solids in Syrups.
- The open public comment period for the proposed modification of AOAC Official Method 932.14 was posted for a minimum of 30 days. The comment period closed on December 30, 2016. Comments were to be compiled, reviewed, and intended to obtain input on the proposed modification. All interested parties had an opportunity to submit comments. At the time of this meeting, we did not receive any comments regarding the proposed method modification.

- Section - D. By Means of U-Tube Oscillation – Digital Density Meter Method
  - Applicability - (Applicable for solutions containing sucrose, liquid sugar products containing invert sugar, other nonsucrose solids and liquids containing no undissolved solids). Determine Brix from specific gravity as measured by AOAC 988.06 using AOAC 942.33 – Degrees Brix, specific gravity, and degrees Baume of sugar solutions (Plato Table).
- Modify AOAC 988.06 applicability to: … is applicable for the determination of Specific Gravity of Beer, Wort and non pulpy fruit juice beverages.
  - Add Journal reference to 988.06
- ERP requirements for AOAC 988.06 for Final Action recommendation
  - Reproducibility data for non-pulpy fruit juice beverages
  - 2) User feedback
- ERP requirements for AOAC 932.14 for Final Action recommendation
  - Reference 998.06 in 932.14 in Section D.

Question for OMB: two methods being modified. Should ERP recommendation for AOAC 988.06 be posted for comment?

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods Board

Subject: Item VIIa – Status of AOAC Standards Development Activities

McKenzie will provide verbal updates

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods Board

Subject: Item VIIb – OMB Liaisons

Verbal Update

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods Board

Subject: Item VIIc – Review and Approval of Stakeholder Panel Representative Voting Members

During the AOAC Mid-Year meeting, there were some stakeholder panels for which registered organizations did not attend or for which organizations that did not register did attend. This did create a temporary offset in the balance of stakeholders represented in a couple of instances. In the immediate term, other representative organizations were invited to sit as voting members and balance was restored. Within OMB, there currently is not an easy way to rectify this in the moment.

For Expert Review Panels, the OMB chair can authorize a new member to sit on ERP in the urgency of not having enough members for a quorum. This is substantiated at the next OMB teleconference or meeting during which the modification to the ERP is disclosed and discussed.

Recommendation:
For the OMB consider a similar type of process for the stakeholder panels. The caveat is what happens when the OMB chair is not accessible.

AOAC INTERNATIONAL

The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: June 22-23, 2017

To: AOAC INTERNATIONAL Official Methods Board

From: Deborah McKenzie, Staff Liaison – AOAC Official Methods Board

Subject: Item VIId – Standard Development Training and Education

McKenzie will provide a verbal update.

AOAC INTERNATIONAL
The Scientific Association Dedicated to Analytical Excellence®

MEMORANDUM

Date: **June 22-23, 2017**

To: **AOAC INTERNATIONAL Official Methods Board**

From: **Deborah McKenzie, Staff Liaison – AOAC Official Methods Board**

Subject: **Item VIII – AOAC Annual Meeting Activities**

Below are the Annual Meeting activities currently for which OMB liaisons are needed. This will be updated as the list of activities are modified.

Date	Time	Event
Friday, Sept. 22, 2017	8:30am – 5:00pm	SPDS Stakeholder Panel (Dietary Supplements)
Saturday, Sept. 23, 2017	8:30am – 5:00pm	SPDS Working Groups
Sunday, Sept. 24, 2017	8:30am – 12:00pm	SPSFAM Stakeholder Panel (Food Panel)
Sunday, Sept. 24, 2017	8:30am – 5:00pm	ISPAM Stakeholder Panel & Working Groups
Sunday, Sept. 24, 2017	1:00pm – 3:00pm	ERP for SPSFAM Ethanol in Kombucha Methods
Monday, Sept. 25, 2017	8:15am – 10:15am	AOAC Board of Directors Meeting
Monday, Sept. 25, 2017	1:30pm – 3:30pm	ERP for SPSFAM Select Food Allergen Methods
Monday, Sept. 25, 2017	4:00pm – 6:00pm	ERP for Microbiology Methods (RI)
Monday, Sept. 25, 2017	5:00pm – 6:30pm	New Member Welcoming Reception
Tuesday, Sept. 26, 2017	8:00am – 10:00am	ERP for Anthocyanins (RI)
Tuesday, Sept. 26, 2017	TBD	ERP for SPIFAN MCPD Methods
Tuesday, Sept. 26, 2017	10:30am – 12:30pm	ERP for Dietary Starches in Feed Methods (RI)
Tuesday, Sept. 26, 2017	1:00pm – 5:00pm	ERP for SPSFAM BPA Methods
Wednesday, Sept. 27, 2017	8:00am – 10:00am	ERP for Gluten Assay Methods (RI)
Wednesday, Sept. 27, 2017	8:15am – 9:45am	Committee on Statistics Symposium
Wednesday, Sept. 27, 2017	10:00am – 12:00pm	Committee on Statistics Meeting
Thursday, Sept. 28, 2017	9:00am – 1:00pm	OMB Meeting

OMB will need to discuss the OMB Meeting time and general content as well as the New Member engagement activities.