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AOAC Official Methods of AnalysisSM (OMA)

AOAC EXPERT REVIEW PANEL

SOLIDS IN SYRUPS



THURSDAY, MARCH 16, 2017
1:00 PM – 4:00PM

Gaithersburg Marriott Washingtonian Center
9751 Washingtonian Boulevard
Gaithersburg, MD 20878 USA



AOAC OFFICIAL METHODS OF ANALYSISSM

The *Official Methods of AnalysisSM* (OMA) program is AOAC INTERNATIONAL's premier methods program. The program evaluates chemistry, microbiology, and molecular biology methods. It also evaluates traditional benchtop methods, instrumental methods, and proprietary, commercial, and/or alternative methods. In 2011, AOAC augmented the *Official MethodsSM* program by including an approach to First Action *Official MethodsSM* status that relies on gathering the experts to develop voluntary consensus standards, followed by collective expert judgment of methods using the adopted standards.

The OMA program has undergone a series of transitions in support of AOAC's collaborations, evolving technology, and evolving technical requirements. Methods approved in this program have undergone rigorous scientific and systematic scrutiny such that analytical results by methods in the *Official Methods of Analysis of AOAC INTERNATIONAL* are deemed to be highly credible and defensible.

On September 7, 2012, AOAC INTERNATIONAL further clarified the AOAC *Official MethodsSM* program by transitioning the conformity assessment component of the *Official MethodsSM* program into the AOAC Research Institute. The AOAC Research Institute now administers the AOAC *Official MethodsSM* program for all proprietary, single and sole source methods. Methods submitted through the PTM-OMA harmonized process also will be reviewed through the AOAC Research Institute. All methods in the AOAC *Official MethodsSM* program are now reviewed by Expert Review Panels for First Action AOAC *Official Methods of AnalysisSM* status.

EXPERT REVIEW PANEL (ERP)

The AOAC Expert Review Panels (ERPs) are a key part of AOAC INTERNATIONAL's Method Approval Process. AOAC ERPs are authorized to adopt candidate methods as First Action *Official Methods* and to recommend adoption of these methods to Final Action *Official Methods* status. Scientists are recruited to serve on ERPs in a variety of ways. Normally, a call for experts is published at the same time as a call for methods is posted. Interested scientists are invited to submit their *curriculum vitae* (CV) for consideration. Advisory panel, stakeholder panel, and working group members may make recommendations to AOAC for ERP members. All CVs are reviewed and evaluated for expertise by the AOAC Chief Scientific Officer (CSO) and then to the AOAC Official Methods Board for formal review. The composition of the ERP must be fulfilled with qualified subject matter experts representing various perspectives. Please refer to our Call for Experts on the AOAC homepage for further information.

AOAC INTERNATIONAL
2275 Research Blvd, Suite 300
Rockville, Maryland 20850
Phone: (301) 924-7077



**AOAC Official Methods of AnalysisSM (OMA)
Expert Review Panel for Solids in Syrups**

TABLE OF CONTENTS

A. ABOUT AOAC OFFICIAL METHODS OF ANALYSISSM	3
B. AGENDA	7
C. AOAC INTERNATIONAL VOLUNTEER CONFLICT OF INTEREST, STATEMENT OF POLICY.....	9
D. AOAC INTERNATIONAL ANTITRUST POLICY STATEMENT AND GUIDELINES.....	11
E. AOAC INTERNATIONAL POLICY ON THE USE OF THE ASSOCIATION NAME, INITIALS, IDENTIFYING INSIGNIA, LETTERHEAD, AND BUSINESS CARDS.....	15
F. MEETING AND METHOD REVIEW INFORMATION	19
G. AOAC EXPERT REVIEW PANEL PRESENTATION	21
H. PROPOSED MODIFICATION OF AOAC OFFICIAL METHOD 932.14: SOLIDS IN SYRUPS [FIRST ACTION 1932]	
I. AOAC PRELIMINARY REVIEW OF PROPOSED MODIFICATION.....	43
II. AOAC FINAL ACTION OFFICIAL METHOD 932.14: SOLIDS IN SYRUPS [FIRST ACTION 1932]	45
III. AOAC FINAL ACTION OFFICIAL METHOD 988.06: SPECIFIC GRAVITY OF BEER AND WORT	47
III. DETERMINATION OF SOLIDS IN SYRUPS WITH AOAC 932.14 USING DIGITAL DENSITY METER METHOD - SINGLE LABORATORY VALIDATION	49



EXPERT REVIEW PANEL (ERP) FOR SOLIDS IN SYRUPS



[Gaithersburg Marriott Washingtonian Center](#)

9751 Washingtonian Boulevard, Gaithersburg, MD 20878 USA

Thursday, March 16, 2017

1:00 PM – 4:00 PM EST

MEETING AGENDA

I. Welcome and Introductions

Expert Review Panel Co-Chairs

II. Review of AOAC Volunteer Policies & Expert Review Panel Process Overview and Guidelines

Deborah McKenzie, Senior Director, Standards Development and Method Approval Processes, AOAC INTERNATIONAL and AOAC Research Institute

III. Review of Methods

For each method the assigned ERP members will present a review of the proposed collaborative study manuscript, after which the ERP will discuss the method and render a decision on the status for each method.

1) Proposed Modification of AOAC Official Method 932.14: Solids in Syrups [First Action 1932]

Study Directors: Sam Khoury, Cott Corporation, 1011 10th Avenue, Columbus, Georgia 31901

IV. Next Steps and Upcoming Meetings

V. Adjournment



The Scientific Association Dedicated to Analytical Excellence®

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
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 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.



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.



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.

* * * * *



***Official Methods of AnalysisSM* (OMA) Expert Review Panel MEETING AND METHOD REVIEW GUIDANCE**

The AOAC Research Institute administers AOAC INTERNATIONAL's premier methods program, the AOAC *Official Methods of AnalysisSM* (OMA). The program evaluates chemistry, microbiology, and molecular biology methods. It also evaluates traditional benchtop methods, instrumental methods, and proprietary, commercial, and/or alternative methods and relies on gathering the experts to develop voluntary consensus standards, followed by collective expert judgment of methods using the adopted standards. The *Official Methods of Analysis of AOAC INTERNATIONAL* is deemed to be highly credible and defensible.

All Expert Review Panel (ERP) members are vetted by the AOAC Official Methods Board (OMB) and serve at the pleasure of the President of AOAC INTERNATIONAL. In accordance to the AOAC Expert Review Panel Member and Chair Volunteer Role Description all Expert Review Panel members are expected to 1) serve with the highest integrity, 2) perform duties and method reviews, and 3) adhere to review timelines and deadlines.

To assist the ERP Chair and its members, please note the following in preparation for Expert Review Panel meetings and method reviews.

Pre-Meeting Requirements

1. Confirm availability and plan to be present to ensure a quorum of the ERP.
(Please refer to page 25, Quorum Guidelines, [Expert Review Panel Information Packet](#))
2. Ensure that your laptop, CPU or mobile device can access online web documentation.
3. Be prepared for the meeting by reviewing all relevant meeting materials and method documentation.

In-Person Meeting and Teleconference Conduct

1. Arrive on time.
2. Advise the Chair and ERP members of any potential Conflicts of Interest at the beginning of the meeting.
3. Participation is required from all members of the ERP. All members have been deemed experts in the specific subject matter areas.
4. The ERP Chair will moderate the meeting to ensure that decisions can be made in a timely manner.
5. Follow Robert's Rules of Order for Motions.
6. Speak loud, clear, and concise so that all members may hear and understand your point of view.
7. Due to the openness of our meetings, it is imperative that all members communicate in a respectful manner and tone.
8. Refrain from disruptive behavior. Always allow one member to speak at a time. Please do not interrupt.
9. Please note that all methods reviewed and decisions made during the Expert Review Panel process are considered confidential and should not be discussed unless during an Expert Review Panel meeting to ensure transparency.

Reviewing Methods

Prior to the Expert Review Panel meeting, ERP members are required to conduct method reviews. All methods are reviewed under the following criteria, technical evaluation, general comments, editorial criteria, and recommendation status. These methods are being reviewed against their collaborative study protocols as provided in the supplemental documentation. *Note: The method author(s) will be present during the Expert Review Panel session to answer any questions.*



Official Methods of AnalysisSM (OMA) Expert Review Panel MEETING AND METHOD REVIEW GUIDANCE

Reviewing Methods (Cont'd)

- Reviewers shall conduct in-depth review of method and any supporting information.
- In-depth reviews are completed electronically via the method review form. The method review form must be completed and submitted by the deadline date as provided.
- All reviews will be discussed during the Expert Review Panel meeting.
- Any ERP member can make the motion to adopt or not to adopt the method.
- If the method is adopted for AOAC First Action status, Expert Review Panel members must track and present feedback on assigned First Action *Official Methods*.
- Recommend additional feedback or information for Final Action consideration.

Here are some questions to consider during your review based on your scientific judgment:

1. Does the method sufficiently follow the collaborative study protocol?
2. Is the method scientifically sound and can be followed?
3. What are the strengths and weaknesses of the method?
4. How do the weaknesses weigh in your recommendation for the method?
5. Will the method serve the community that will use the method?
6. What additional information may be needed to further support the method?
7. Can this method be considered for AOAC First Action OMA status?

Reaching Consensus during Expert Review Panel Meeting

1. Make your Motion.
2. Allow another member to Second the Motion.
3. The Chair will state the motion and offer the ERP an option to discuss the motion.
4. The Chair will call a vote once deliberations are complete.
5. Methods must be adopted by unanimous decision of ERP on first ballot, if not unanimous, negative votes must delineate scientific reasons. Negative voter(s) can be overridden by 2/3 of voting ERP members after due consideration.
6. All other motions will require 2/3 majority for vote to carry.

AOAC Expert Review Panels An Orientation

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

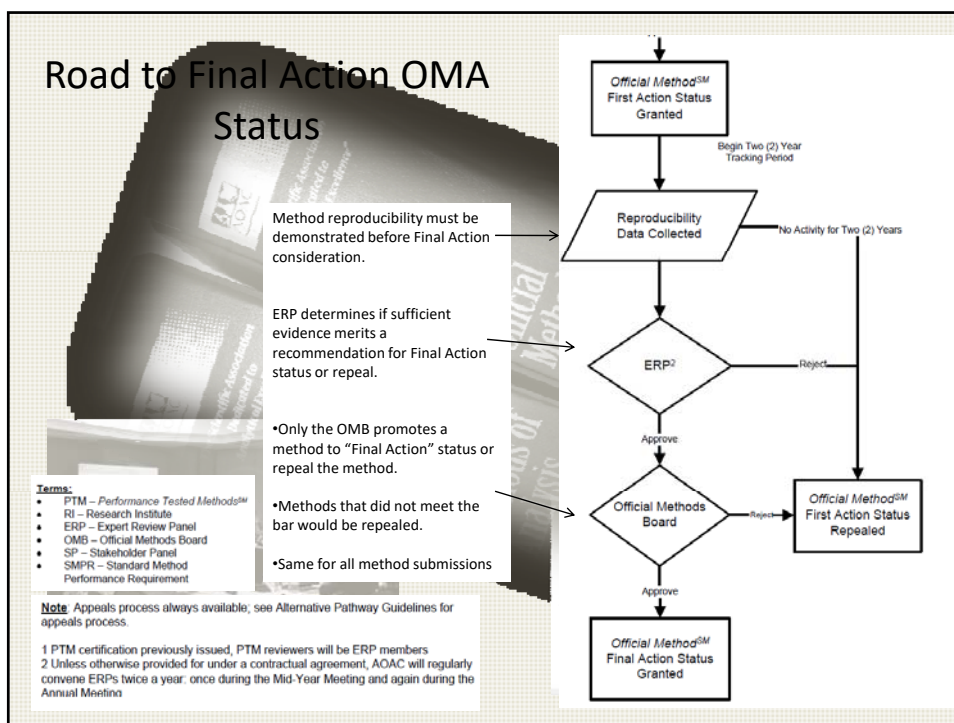
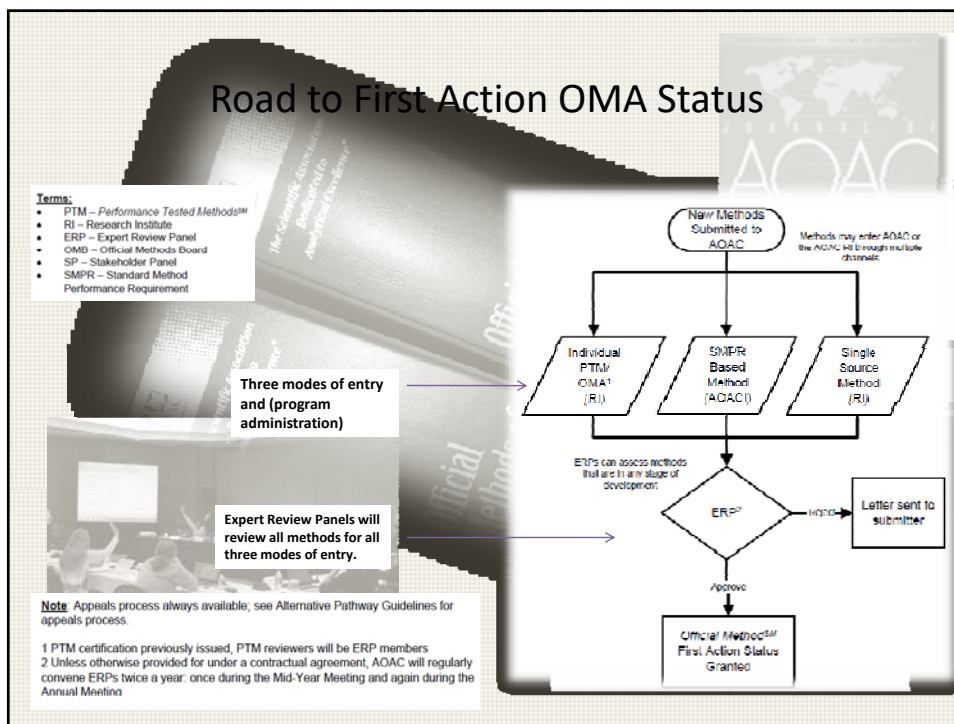
Policy on Volunteer Conflict of Interest

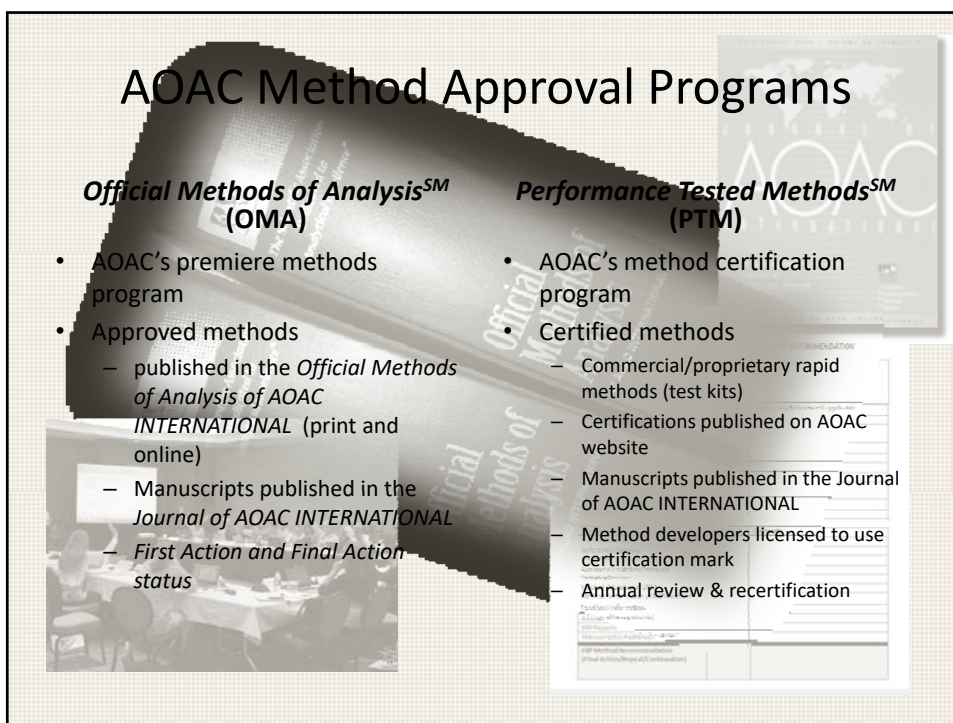
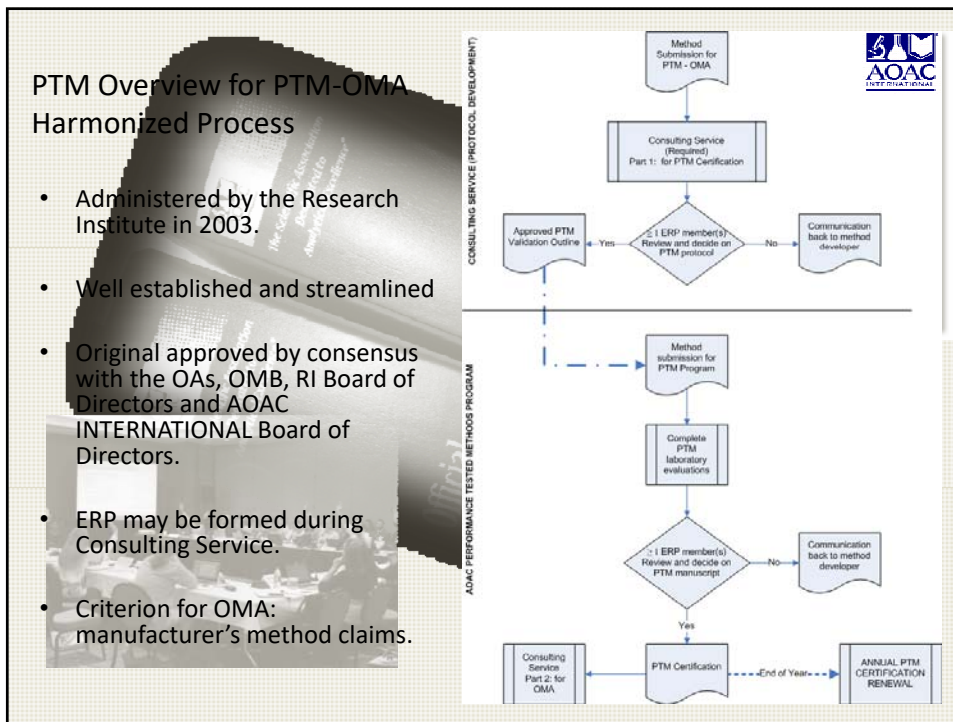
Expert Review Panel Policies and Procedures

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 MethodsSM Program*





Qualifications for ERP Membership

Candidate must meet one of the following:

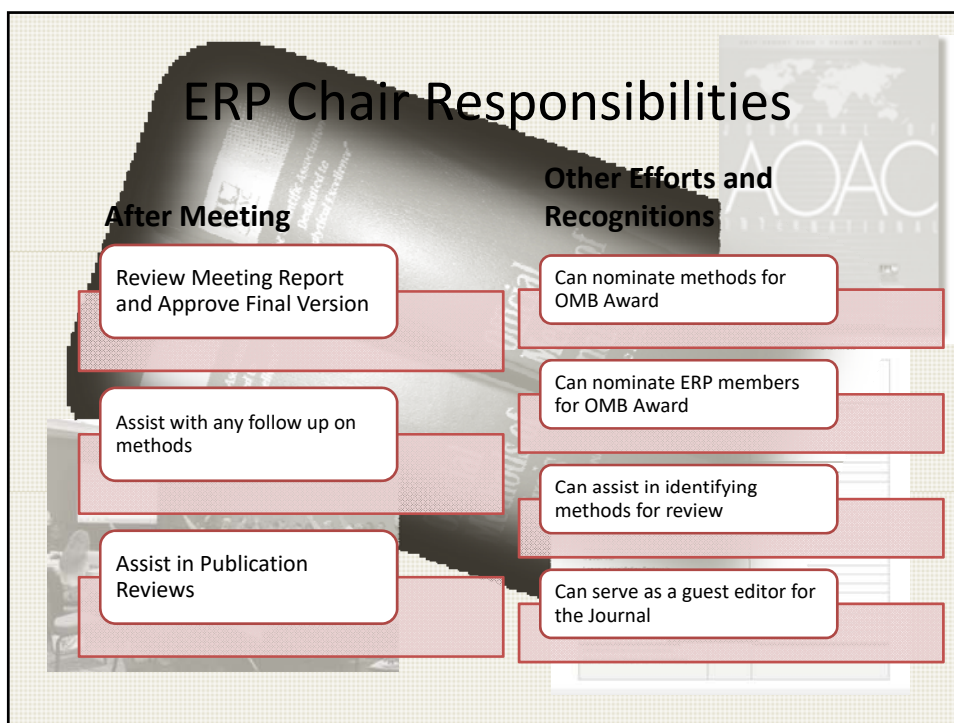
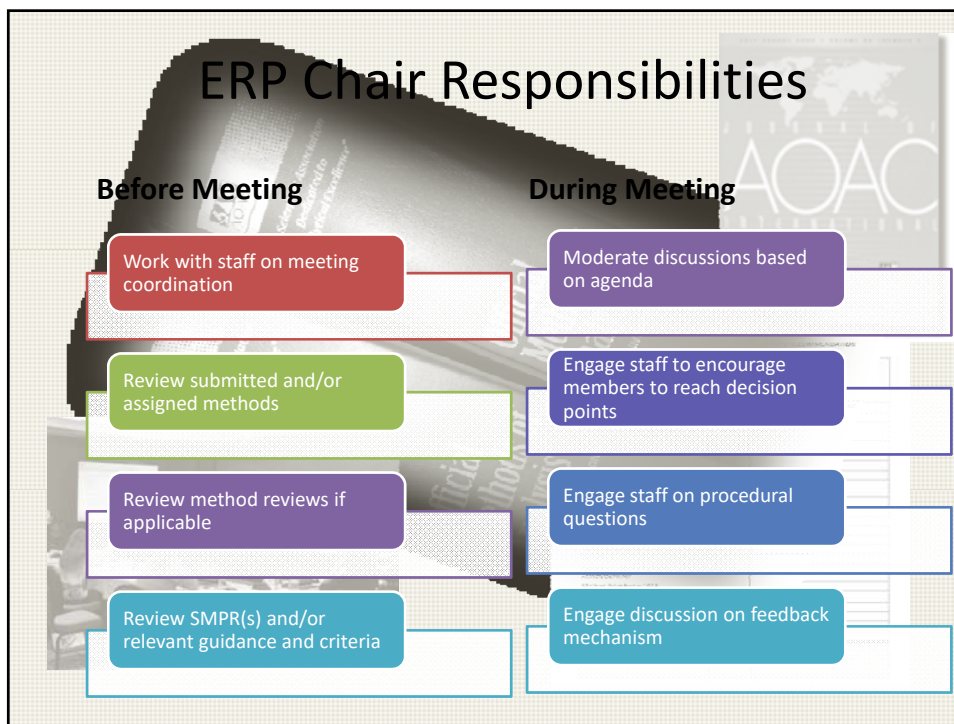
- Demonstrated knowledge in the appropriate scientific disciplines.
- Demonstrated knowledge regarding data relevant to adequate method performance.
- Demonstrated knowledge of practical application of analytical methods to bona fide diagnostic requirements.

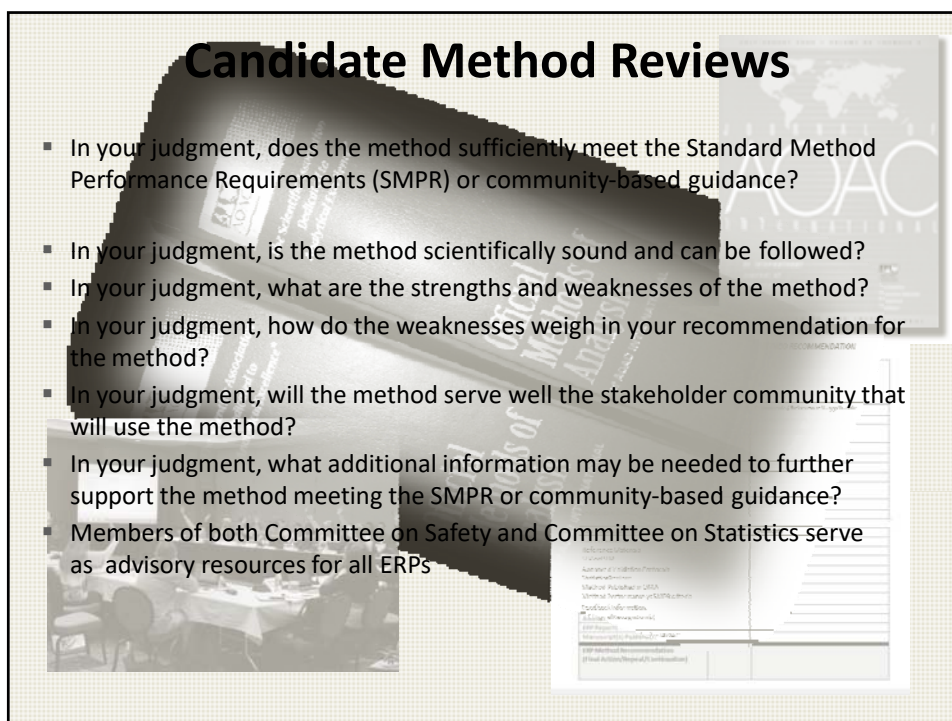
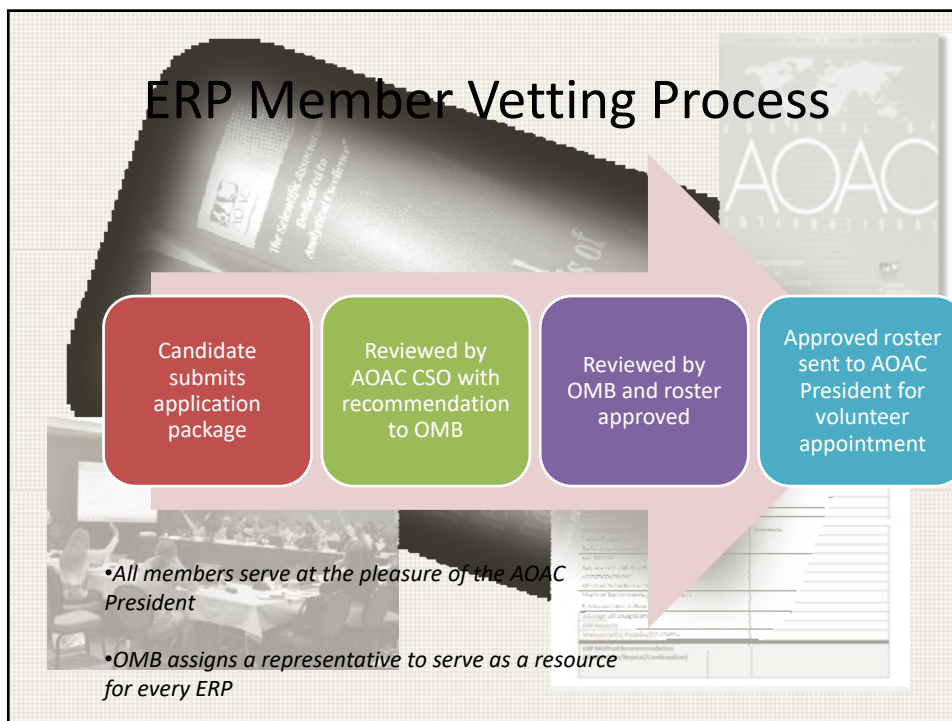
Candidate application package includes:

- Statement of Expertise
- Current Abridged CV or Resume

Experts and Methods

- AOAC issues
 - Call for Methods (*Stakeholder affiliated methods*)
 - Call for Experts
- Sole Source/Individual Method Submissions
 - Applications to Research Institute





ERP Meetings

- ERPs will meet in person at a minimum of twice a year and up to four times per year:
 - AOAC Mid-Year meeting (DC metro area)
 - AOAC Annual Meeting.
 - 2 additional designated times for proprietary method Organizational Affiliates
- At the ERP meeting:
 - Reviews will be presented and a primary or secondary reviewer can make a motion/recommendation to the ERP whether or not to adopt the method as First Action OMA.
 - ERP discusses the method.
 - ERP renders a decision on First Action status.
 - ERP renders decisions on modifications to First Action methods only.
- If the method is adopted
 - ERP decides on what additional information is needed to recommend the method for Final Action status

ERP Meetings

Quorum

Presence of 7 vetted ERP members **OR** Presence of 2/3 vetted ERP members

WHICHEVER IS GREATER

IF NO QUORUM, NO OFFICIAL MEETING

Method Review Overview

- Method authors may be invited to make a presentation on their method
- REVIEWERS PRESENT THEIR REVIEWS AND MAY INITIATE A MOTION TO ADOPT THE METHOD IF THEY CHOOSE
 - Chair recognizes each reviewer
 - Primary and secondary reviews are presented.
 - If in favor, they may make and second a motion to adopt or not adopt the method
 - Chair can then entertain discussion on the method
 - Chair can call for a vote once deliberation is complete

Consensus – First Action Adoption

- First Action Official Methods status is granted:
- Method must be adopted by unanimous decision of ERP on first ballot, if not unanimous, negative votes must delineate scientific reasons.
- Negative voter(s) can be overridden by 2/3 of voting ERP members after due consideration.
- Method becomes First Action on the date when ERP decision is made.

Consensus – First Action to Final Action

- The ERP may then reach consensus on any additional information that it needs to review to be able to make a recommendation for Final Action *Official Methods* status.

- This is a separate motion.



Road to First Action OMA Status

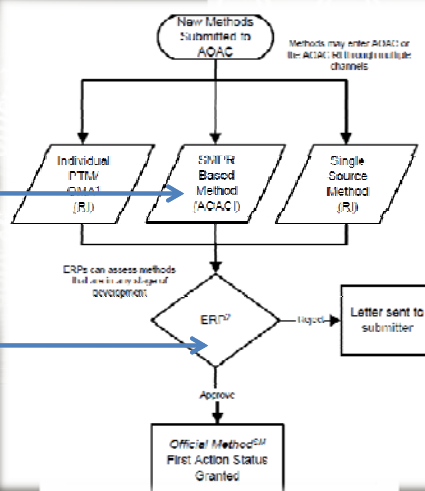
- Terms:**
- PTM – Performance Tested MethodSM
 - RI – Research Institute
 - ERP – Expert Review Panel
 - OMB – Official Methods Board
 - SP – Stakeholder Panel
 - SMPR – Standard Method Performance Requirement

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



ERP Meetings – Review for First Action

METHOD AUTHOR: present any method and any resulting changes to the method since submission for review, summary of SLV and/or reproducibility evaluation, any recognitions (from AOAC or external) and, final draft of method proposed for decision

ERP CHAIR & MEMBERS: present reviews and discuss any resulting issues or questions on the method, review and agree upon final draft of method proposed for decision, and chair calls for ERP decision in accordance to procedures.

CONSENSUS: Method must be adopted by unanimous decision of ERP on first ballot. If not unanimous, negative votes must delineate scientific reasons. Negative voter(s) can be overridden by 2/3 of non-negative voting ERP members after due consideration.
Abstentions do not count towards vote; in case of multiple abstentions the results will need to be evaluated. Staff will monitor and record consensus voting.

STAFF: Will organize and coordinate meeting, record ERP actions and decisions, draft ERP report and distribute after chair approval, work with chair and OMB liaison to complete checklist and assemble recommendation package for OMB.

ERP Methods Review & Approval

Methods should be scientifically sound with demonstrating that it will meet the needs of those using the method (evidenced by meeting the standard, or other acceptance criteria)

ERPs have approved methods with evidence of high potential to First Action and request additional work or support be submitted for review prior to ERP convening to recommend an action to OMB

OMB requires a justification or rationale for methods that are deemed acceptable and adopted but may not fully meet the standard set or acceptance criteria.

OMB Expectations for First Action

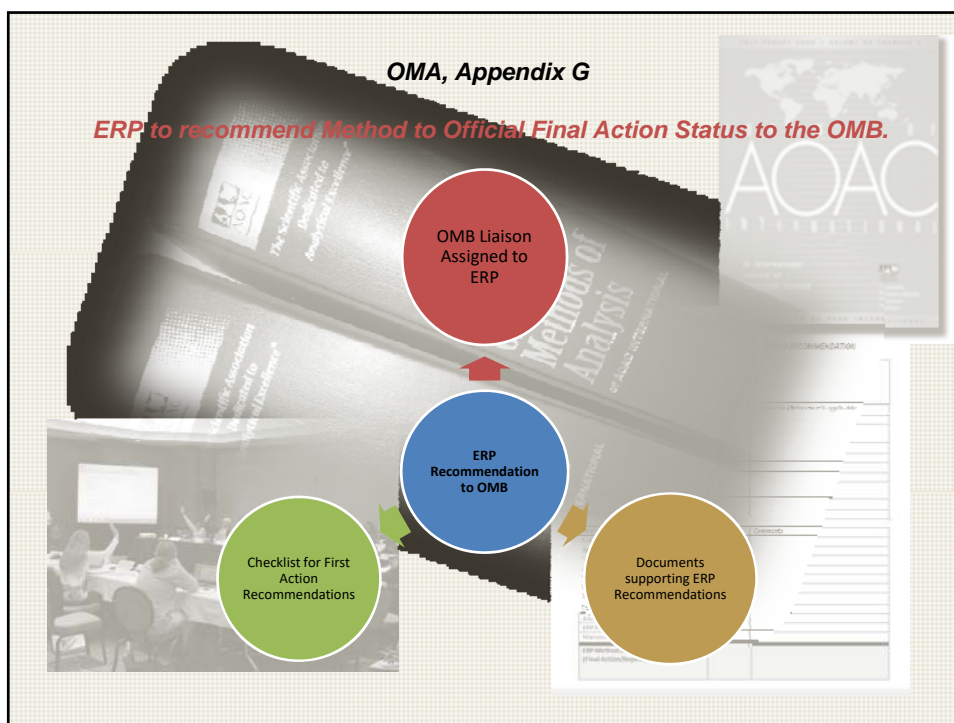
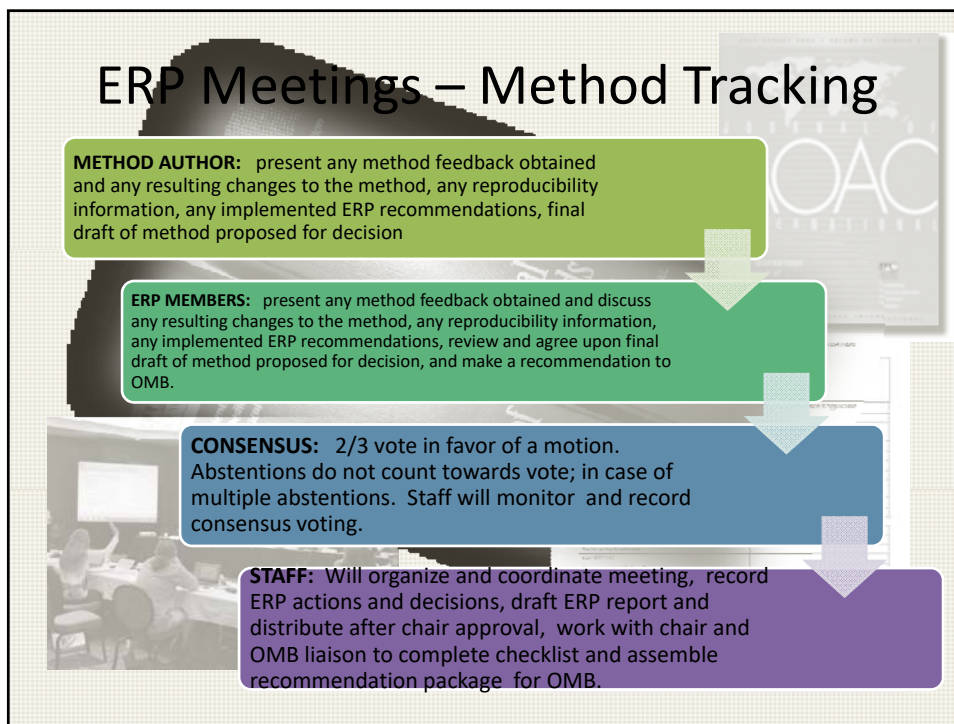
- Safety review needed prior to First Action status
- SLV type of supporting information available per the SMPR
 - Applicability, Method Performance Requirements Table, System Suitability, Reference Materials, and Validation Guidance
- Comparison to SMPR
 - Documented method performance versus a SMPR
 - Document reasons for acceptability if method does not meet the SMPR

Publication of First Action Methods

- Any approved method(s) along with supporting manuscript(s) and documentation sent to AOAC Publications after the meeting.
 1. Method incorporating ERP revisions (preferably in AOAC Format)
 2. Method Manuscript incorporating specified ERP revisions (in AOAC Format)
 3. Signed AOAC Copyright Authorization form

NO OMA NUMBER ASSIGNED UNTIL ALL DOCUMENTATION SUBMITTED

- Method and method manuscript prepared for publication in the *Official Methods of Analysis of AOAC INTERNATIONAL* and in *Journal of AOAC INTERNATIONAL*
- Updates on methods approved or status changes are published in the *Inside Laboratory Management* magazine and on the AOAC website



OMA, Appendix G

Further data indicative of adequate method reproducibility (between laboratory performance to be collected. Data may be collected via a collaborative study or by proficiency or other testing data of similar magnitude.

- ERP is looking to verify if method reproducibility has been appropriately assessed and satisfactorily demonstrated

The diagram illustrates the OMB Expectations for ERPs regarding method reproducibility. It starts with a central box labeled "OMB Expectations for ERPs" with "Reproducibility" highlighted in yellow. This box branches into two categories: "Qualitative Methods" and "Quantitative Methods".

- Qualitative Methods** leads to the outcome: "probability of detection or equivalent".
- Quantitative Methods** leads to the outcome: "demonstrated method reproducibility and/or uncertainty".

The background of the slide features a blurred image of a meeting and a document titled "Official Methods of Analysis" with the AOAC logo.

OMA, Appendix G

Two years maximum transition time (additional year(s) if ERP determines a relevant collaborative study or proficiency or other data collection is in progress).

The diagram details the transition process for a method. It is divided into two main sections:

- 2 yr tracking of method:**
 - ERP verification of any changes to the method
 - ERP recommendations implemented successfully
 - ERP evaluation of any feedback on method and its performance
- ERP Recommendations:**
 - Move method to Final Action OMA status
 - Repeal method from OMA
 - Continuance of First Action OMA status

The background of the slide features a blurred image of a meeting and a document titled "Official Methods of Analysis" with the AOAC logo.

OMA, Appendix G

Method removed from Official First Action and OMA if no evidence of method use or if no data indicative of adequate method reproducibility available at the end of the transition time.

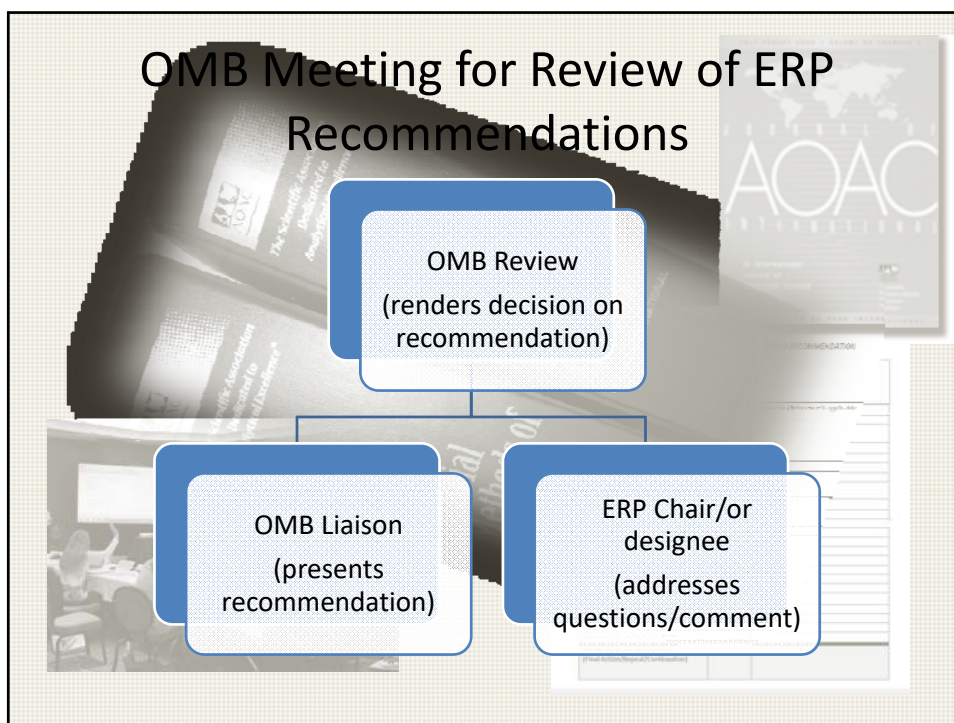
First Action OMA Tracking

- Tracking period is ≤ 2 years and begins on the date of the ERP's decision to adopt a method for OMA First Action status.

No Use in 2 Years

- Repeal from OMA





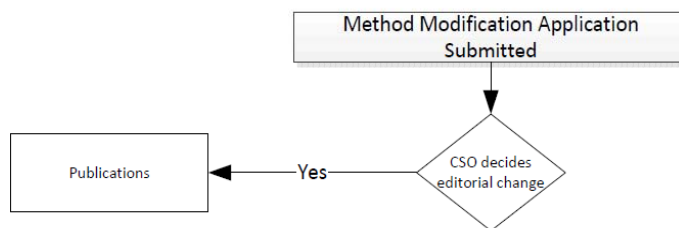
Modifications to Official Methods

- 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

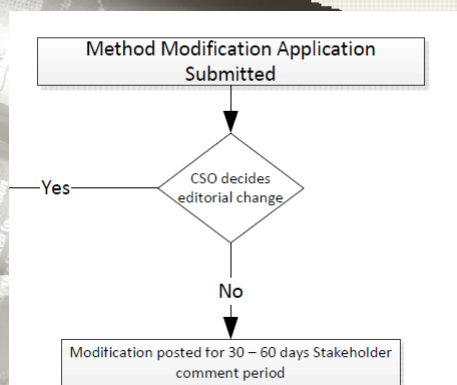
- Results in no changes to the current validated performance. There is no significant effect to the results. The method will retain the original number.
- Supporting data to justify the proposed modification must be submitted. Equivalency data is required unless adequate Justification to exclude this data is provided.
- Examples include: Reagent change, a change in a column or consumables that do not impact the validated method performance.

Major Modifications

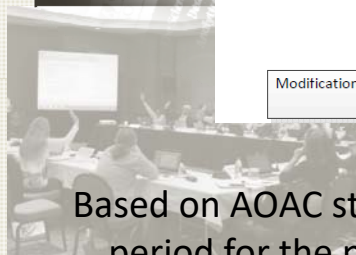
- Results in a change to the current validated performance of the method.
- This level of modification will result in a new method as part of AOAC standards development and will receive a new method number.
- Examples include: significant change to the technology, sample preparation, or chemistry.

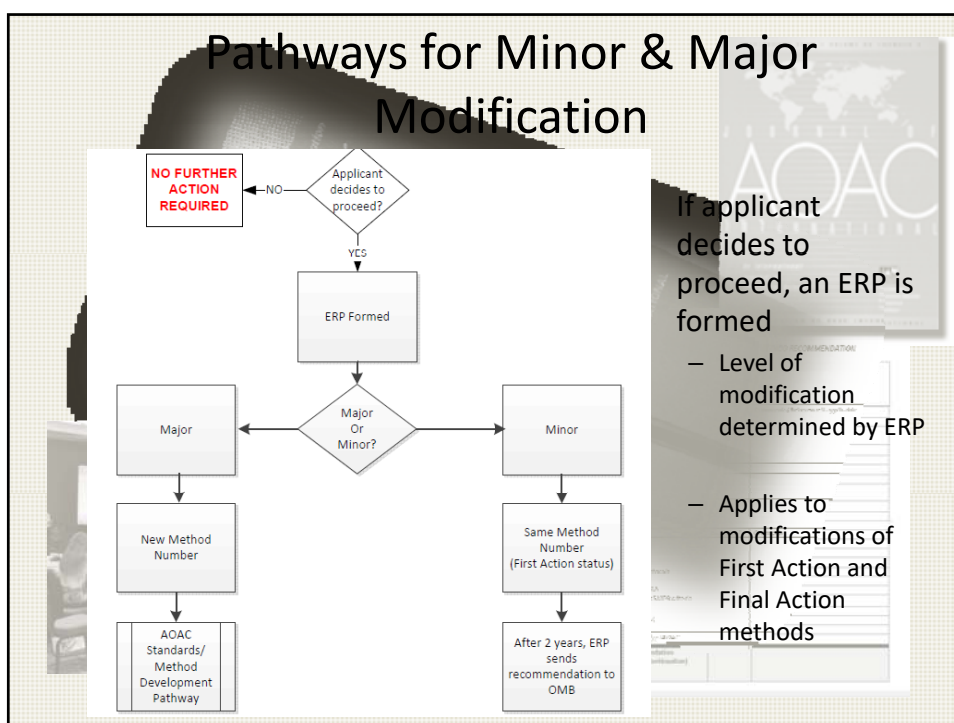
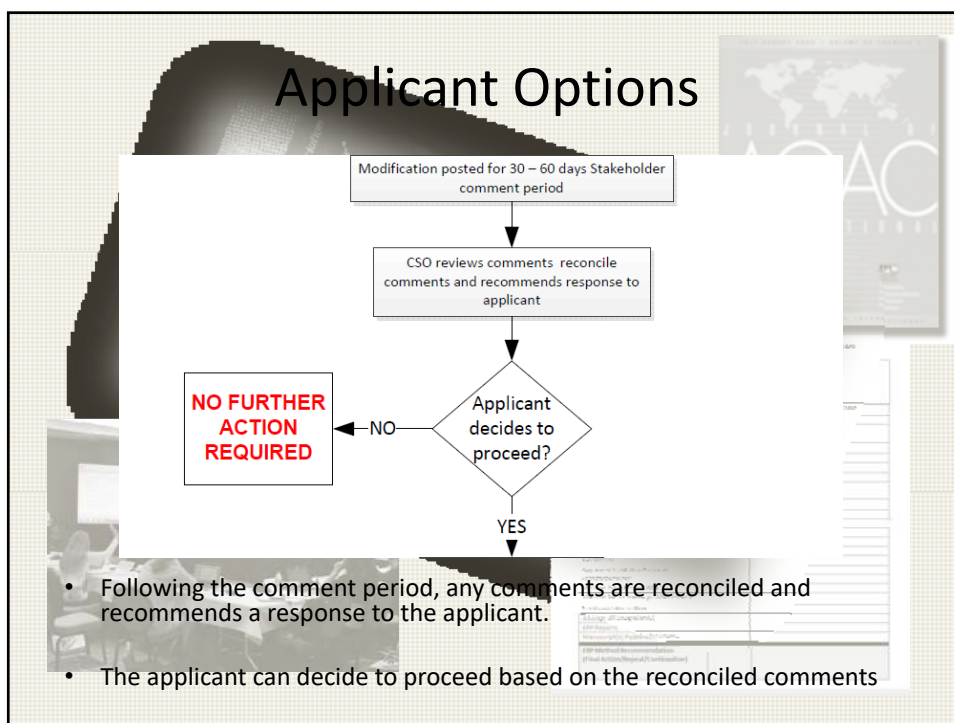


Minor & Major Modifications



Based on AOAC staff review, a public comment period for the proposed modification is required.





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 MethodsSM status.
- Track First Action Official MethodsSM 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

Second Review

Method Number: 932.14

Method Name: Solids in syrups

Status: First Action 1932; Final Action undated

Reviewer: Scott Coates, Chief Science Officer, AOAC INTERNATIONAL

Review Date: February 3, 2014

Documents Reviewed:

- 1) AOAC Official Method 932.14 (attachment 1)
- 2) Revised AOAC Official Method 932.14 (attachment 2)
- 3) DRAFT Determination of Solids in Syrups with AOAC Method 932.14 Using Digital Density Meter Method: Single- Laboratory Validation; Sam G. Khoury, M.S., MBA; Cott Corporation, 1001 10th Avenue, Columbus, GA 31901; January 2014. (attachment 3)

Summary of Proposed Modification:

Addition of a 4th measurement option using the digital density meter with a U-tube oscillator used in OMA 988.06.

Purpose of Modification:

Method modernization.

Classification of Modification:

The proposed modification is a change (an additional measuring option) in the procedure of the method. Such a modification requires the generation and submission of data; review of data; revision of the method in the OMA w/ editorial review; and preparation of a manuscript describing the revisions and supporting verification data. Equivalent to Level 2 minor modification.

Status

As requested:

A verification study was completed and submitted that includes: side-by-side comparison of common samples using the original method versus the proposed modified method; and intermediate precision studies of both methods.

A revised method to replace the existing 932.14 was submitted

Additional Comments:

1. The modernization of this method in and of itself justifies the approval of the modifications provided that comparability and repeatability results are reasonable.
2. The results demonstrate that the modernized method is equivalent to the original method.
3. All documents requested in the first review have been submitted.
4. **Submit to OMB for approval.**

44.1.04

AOAC Official Method 932.14
Solids in Syrups
First Action 1932
Final Action 20XX

A. By Means of Spindle

(Accurate only when applied to pure sucrose solutions, but extensively used for approximate results with liquid sugar products containing invert sugar and other nonsucrose solids.)

(a) *Direct.* – Density of juices, syrups, etc. is conveniently determined with Brix or Baumé hydrometer, preferably former as scale graduations agree closely with percent total solids. Table for comparison of degrees Brix (percent by weight of pure sucrose in pure solutions), degrees Baumé (modulus 145), specific gravity at 20/4°C, is given in [942.33](#) (see *Appendix C*).

Use Brix spindle graduated in tenths and appropriate range, and cylinder of sufficient diameter (≥ 12 mm larger than spindle bulb) to permit spindle to come to rest without touching sides. Solution should be at room temperature. If this varies $>1^\circ\text{C}$ from temperature at which spindle was graduated (20°C), apply correction according to [900.03](#) (see *Appendix C*). Before taking reading, let solution stand in cylinder until all air bubbles escape and all fatty or waxy materials come to top and are skimmed off. (Air bubbles may be conveniently removed by applying vacuum to cylinder by means of tube passing through stopper inserted in top of cylinder.) Lower spindle slowly into syrup; do not let syrup on spindle reach above syrup level.

(b) *Double dilution.* – If syrup is too dense to determine density directly, dilute weighted portion with weighted amount of H_2O , or dissolve weighted portion and dilute to known volume with H_2O . In first instance, percent total solids is calculated by following formula:

$$\text{Solids in undiluted material, \%} = \frac{W \times S}{w}$$

where S = percent solids in diluted material; W = weight diluted material; and w = weight syrup taken for dilution.

When dilution is made to definite volume, use following formula:

$$\text{Solids in undiluted material, \%} = \frac{V \times D \times S}{w}$$

where V = volume diluted solution at given temperature; D = specific gravity of diluted solution at same temperature; S = percent solids in diluted solution at same temperature; and w = weight syrup taken for dilution.

Calculation is simplified by mixing equal weights sugar product and H_2O , and multiplying Brix of solution by 2.

B. By Means of Pycnometer

(a) *Specific gravity (in vacuo or in air).* – Determine specific gravity of solution at 20/4°C, 20/20°C in vacuo, or 20/20°C in air as in [945.06C](#) (see 26.1.06), using either pycnometers described in [945.06A\(b\)](#) (see 26.1.06) or other suitable type. Apply air buoyancy correction to specific gravity in air and determine percent by weight of solids as sucrose from appropriate table, [942.33](#) (see *Appendix C*) or [962.37](#) (see *Appendix C*). When density of substance is too high for direct determination, dilute and then calculate sucrose content of original material as **A(b)**.

(b) *Specific gravity of molasses.* – Use special calibrated 100 mL volumetric flask with neck ca 8 mm id. Weigh empty flask and then fill with molasses, using long-stem funnel reaching below graduation mark, until level of molasses is up to lower end of neck of flask. (Flow of molasses may be stopped by inserting glass rod of suitable size into funnel so as to close stem opening.) Carefully remove funnel to prevent molasses from coming in contact with neck, and weigh flask and molasses. Add H_2O almost to graduation mark, running it down side of neck to prevent mixing with molasses. Let stand several h or overnight for bubbles to escape. Place flask in constant temperature water bath. Preferably at 20°C , and leave until it reaches bath temperature. Dilute to volume at temperature with H_2O . Weigh. Reduce weight molasses to in vacuo and calculate density. Obtain corresponding Brix or Baumé reading from [942.33](#) (see *Appendix C*).

Example:

X , weight H_2O content of flask at 20°C in vacuo = 99.823 g

Y , weight molasses at 20°C in vacuo = 132.834 g

Z , weight of molasses and H_2O at 20°C in vacuo = 137.968 g

$X - (Z - Y)$ = weight H_2O occupying space of molasses in vacuo = 94.689 g

$$\frac{132.834}{94.689} = 1.403 \text{ specific gravity } \left(\frac{20^\circ}{20^\circ} \right) \text{ molasses}$$

References: *JAOAC* **15**, 195 (1932); **18**, 83 (1935)

C. By Means of Refractometer

(Applicable only to liquids containing no undissolved solids.)

Soluble solids by refractometric method is that concentration by weight of sucrose in solution that has same refractive index (n) as solution analyzed. Use instrument with scale graduated at least in 0.001 units or 0.5% sucrose, permitting estimation to 0.0002n or 0.25% respectively. Adjust instrument to read n of 1.3330 or 0% sucrose with H_2O at 20°C .

Determine refractometer reading of solution at 20°C and obtain corresponding percent dry substance from either direct reading, if sugar refractometer is used, or from [990.35](#) (see *Appendix C*), if instrument gives readings in terms of refractive index. Circulate H_2O at constant temperature, preferably 20°C , through jackets of refractometer or through trough of immersion instrument, long enough to let temperature of prisms and of syrup each equilibrium, continuing circulation during observations and taking care that temperature is held constant.

If determination is made at temperature other than 20°C , or if humidity causes condensation of moisture on exposed faces of prisms, make measurements at room temperature and correct readings to standard temperature of 20°C from [990.36](#) (see *Appendix C*). If solution is too dark to be read in instrument, dilute with concentrated sugar solution; never use H_2O for this purpose. Mix weighted amounts of solution under examination and solution of pure sugar of about same strength, and calculate percent dry substance in former = $[(W + B)C - BD]/W$, where W = weight (g) syrup mixed with B ; B = weight (g) sugar solution used in dilution; C = percent dry substance in mixture $W + B$ obtained from refractive index; and D = percent dry substance in pure sugar solution obtained from its refractive index.

For liquid products containing invert sugar, correct percent solids obtained from [990.35](#) (see *Appendix C*) by adding 0.022 for each percent invert sugar in product.

References: *JAOAC* **15**, 79 (1932); **16**, 81 (1933); **17**, 74 (1934); **41**, 621 (1958); **73**, 124 (1990).

D. By Means of U-Tube Oscillation – Digital Density Meter Method

(a) *Direct.* – Specific Gravity of juices.

(b) *Principal*

Natural frequency of hollow oscillator varies with density of material that fills tube. Electronic measurement of a time period is converted to digital display of density, specific gravity and Brix.

(c) *Apparatus and Reagents*

(1) *Digital density meter:* Anton Paar® GmbH, Anton-Paar-Str.20, A-8054 Graz, Austria – Europe; (www.anton-paar.com). DMA 4500M digital analyzer with U-shaped, oscillating sample tube.

(2) *Hypodermic syringe.* As recommended in density meter instructions.

(d) *Calibration of Density Meter*

Calibrate digital density meter at $20.0 \pm 0.1^\circ\text{C}$ with air and H_2O . Use hypodermic syringe to inject liquids into analysis tube. Fill tube completely so no gas or solids are entrapped

(e) *Determination*

Inject test sample, if carbonated beverage (ensure decarbonation), into filling tube. Be sure no gas is entrapped in tube. Let thermostated analysis tube reach thermal equilibrium. Obtain specific gravity measurement if desired result are Brix then convert to Brix using **AOAC942.33** – Degrees Brix, specific gravity, and degrees Baume of sugar solutions (Plato Table).

References: Anton Paar® GmbH. *J. of AOAC Intl*, Vol XXX, No. XX (20XX). *J. Am. Soc. Brew. Chem.* **36**, 118 (1978). *Methods of Analysis* (1992) 8th Ed., American Society of Brewing Chemists, 3340 Pilot Knob Rd, St. Paul, MN 55121, USA, Beer 2-B.

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27.1.06

**AOAC Official Method 988.06
Specific Gravity of Beer and Wort**

**Digital Density Meter Method
First Action 1988
Final Action 2008**

ASBC–AOAC Method

A. Principle

Natural frequency of hollow oscillator varies with density of material that fills tube. Electronic measurement of a time period is converted to digital display of density.

B. Apparatus and Reagents

(a) *Digital density meter*.—Mettler Toledo, Inc. DMA 46 (current Models DE40, DE51, DE45), or equivalent (Mettler Toledo, Inc., 1900 Polaris Pkwy, Columbus, OH 43290, USA; www.mt.com).

(b) *Hypodermic syringe*.—As recommended in density meter instructions.

C. Calibration of Density Meter

Calibrate digital density meter at 20.0 ± 0.1 °C with air and H₂O. Use hypodermic syringe to inject liquids into analysis tube. Fill tube completely so no gas or solids are entrapped.

D. Preparation of Test Sample

Decarbonate beer samples as in 920.49 (see 27.1.01). Beer must be brilliantly clear and free from any particulate matter, and must be at 20 °C test temperature. Filter if required.

E. Determination

Inject clear, decarbonated test sample into filling tube. Be sure no gas is entrapped in tube. Let thermostated analysis tube reach thermal equilibrium (0.5–4 min). Obtain specific gravity measurement.

References: *J. Am. Soc. Brew. Chem.* **36**, 118(1978).

Methods of Analysis (1992) 8th Ed., American Society of Brewing Chemists, 3340 Pilot Knob Rd, St. Paul, MN 55121, USA, Beer 2-B.

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Determination of Solids in Syrups with AOAC Method 932.14 Using Digital Density Meter Method: Single- Laboratory Validation

Sam G. Khoury, M.S., MBA

Cott Corporation, 1001 10th Avenue, Columbus, GA 31901

AOAC Official MethodSM 932.14, Solids in Syrups, has been used to measure Brix in food manufacturing laboratories since its publication in the Official Methods of Analysis in 1932. The Final Action lists determination of Solids in Syrups in one of three methods; A. By Means of Spindle (Density), B. By Means of Pycnometer (Specific Gravity) and C. By Means of Refractometer (Refractive Index). Since the method's publication, modern technology for measurement of density has emerged in the form of digital density meter with a U-tube oscillator (DDM). To investigate performance of digital density meter with U-tube oscillator, verification studies were conducted to estimate its precision from replicate measurements made in a single laboratory under variable conditions: days, analysts and instruments. A second Intermediate Precision Study was conducted for the existing Density "spindle" method. Both Intermediate Precision studies showed that the proposed method and original method exhibited very little overall variability and both studies' results exhibited statistically similar with-in day repeatability and day-to-day reproducibility. In addition, side-by-side comparisons of common samples were administered using the original spindle method versus the proposed U-tube oscillating DDM method. Side-by-side analyses demonstrated that the overall repeatability and reproducibility of the density meter with a U-tube oscillator were statistically comparable to those of the existing "spindle" method at the 5% level. Recommendations for modification to AOAC Official Method 932.14 are suggested based on statistical analysis of the data and a review of the literature. Modification will entail addition of a 4th measurement option using the digital density meter with a U-tube oscillator as described in OMA 988.06.

Soluble solids, which normally equate to sugar content in juice and sugar sweetened beverages, are measured as specific gravity, refractive index or density and converted to Brix or Baume. Brix is

defined as the percentage of total solids in solution, in grams of solute/100 gram of solution (g/g) (5). Because sugar constitutes most of the soluble solids in sugar sweetened beverages and juices, Brix is accepted as an approximate measure of the actual sugar solids content in sweetened beverages or juices. This unit is widely used in general chemistry and has become the most used unit for sugar content in the juice industry (7). Government regulations outlining percentage juice labeling requirements define minimum single strength Brix standards for each juice type (1). Density using U-Tube densitometer has been proposed as a mean to measure specific gravity (8) and Brix of sugar sweetened beverages and juices. The densitometer has advantages over other methods of measuring Brix in that the DDM is less sensitive to pH, temperature, color and turbidity (7).

Historical Information for AOAC Official Method 932.14 was not published and unavailable for review. Therefore an intermediate precision study for the original spindle method was conducted as part of this research project.

Experimental

Six different studies were selected for this validation; 1) Intermediate Precision Study of the digital density meter (DDM) using an Apple juice matrix. Data was collected using two analysts, four different digital density meters over four days. 2) Intermediate Precision Study of the published "spindle" method. Data was also collected using two analyses, two different spindles over four days. 3) Side-by-side comparison of common samples using the original "spindle" method versus the proposed modified method. Statistical analysis was calculated using SAS JMP® software.

Participants received clearly and unambiguously written instructions detailing the study design, testing protocol, and the reporting forms using MS Excel.

Materials

Four sample matrices were created for study. Sample types selected to represent the wide range of sugar concentrations, acidity, color, turbidity that reflect common liquid sugar beverage types and the diversity of combinations/concentrations of sugars in beverages. The beverages studies include apple juice (AJ), Grape juice (GJ), pear juice (PJ), and cranberry juice cocktail (CJC).

Apparatus

- (a) Digital density meter – Anton Paar DMA 4500M digital analyzer with U-shaped, oscillating sample tube. The instrument controls temperature of the sample at 20.00 °C ±0.05°C.
- (b) Syringes, for use in manual injection, at least 2 mL as recommended in density meter instructions.
- (c) Brix Hydrometer spindle with scale graduations for specific gravity.
- (d) Cylinder of sufficient diameter (≥12 mm larger than spindle bulb) to permit spindle to come to rest without touching sides or the base as per **AOAC 932.14** requirements.
- (e) Certified lab thermometer graduated with Celsius degrees.
- (f) Digital density meter and Brix hydrometer; specific gravity readings are converted to Brix using **AOAC 942.33** - Degrees Brix, specific gravity, and degrees Baume of sugar solutions (Plato Table).

Methods

Intermediate precision study of the digital density meter – Apple juice was mixed in a five gallon container, agitated and samples from which were collected and analyzed for specific gravity and sucrose Brix. Each analysis set was conducted fifteen duplicate times and repeated over four days with two different analysts using four digital density meters.

Intermediate precision study of the original spindle hydrometer – Apple juice was prepared in a five gallon container, agitated and sampled for specific gravity and sucrose Brix. Each analysis set was conducted thirty duplicate times and repeated over four days with two different analysts using two (“certified”) Brix hydrometers.

Side-by-side comparison of common samples using apple juice (AJ), grape juice (GJ), pear juice (PJ), and cranberry juice cocktail (CJC) were analyzed using digital density meter and spindle in repeated sets of thirty each. Results were recorded in specific gravity units and Brix values

were determined by converting the specific gravity results Brix using **AOAC 942.33** tables.

Test Procedure

Intermediate precision study – digital density meter:

- (a) Four Anton Paar 4500M instruments were used for this study. All were calibrated daily prior to testing at 20.0°±0.1°C with air and water.
- (b) Bulk sample of each lot of juice was mixed to ensure an even distribution of solids and then divided into one gallon portions for each day of analysis.
- (c) At least 2 mLs in volume per measurement were injected into each the density meter’s U-tube, analysts ensured that no gas is entrapped in tube.
- (d) Specific gravity and Brix measurements were collected after U-tube and sample had reached 20°C thermal equilibrium.

Intermediate precision study – hydrometer:

- (a) Two spindle hydrometers spindles graduated in tenths of degrees Brix were used for this study.
- (b) Bulk sample of each lot of juice was mixed thoroughly to ensure an even distribution of solids and then divided into one gallon portions for each day of analysis
- (c) Five cylinders of sufficient diameter and height per analyst were filled per day.
- (d) Samples were equilibrated at room temperature (20°C) prior to analysis.
- (e) Readings were taken after the sample was left in cylinder and all air bubbles allowed to escape.
- (f) Spindle was lowered slowly into sample and readings were collected by recording the corresponding graduating on the lower end of the sample meniscus.
- (g) Corrections were applied according to **AOAC 900.03** if temperature varied from 20.0°C.

Statistical Analysis

Each analyst recorded results for the proposed digital density meter as well as spindle method. The data sheets were submitted to the study coordinator at the end of each day of testing.

For each individual type of common samples, duplicates **AOAC 932.14** “By Means of Spindle” were averaged, and these values along with digital density meter results were reported as specific gravity and Brix, and the performance parameters of repeatability (s_r) and reproducibility (s_R) standard deviation, relative standard deviation of repeatability (RSD_r) and reproducibility (RSD_R),

and repeatability and reproducibility values (r and R) were analyzed. In addition, an F-test was conducted to compare variances between methods for repeatability (r_r) and reproducibility (r_R). Both method means were also compared via a post hoc Tukey-Kramer HSD test to distinguish if any testing conditions were significantly different from each others.

Intermediate Precision study analysis was performed using (ANOVA) based on ISO 5725-3:1994, Accuracy (trueness and precision) of measurement methods and results - Part 3: Intermediate measures of the precision of a standard measurement method. Intermediate Precision study was performed for both proposed and exiting methods.

Results

(a) *Precision - Repeatability (within-day variability)*. - The repeatability of the proposed method DDM was evaluated from duplicate analysis of 240 duplicate analyses over the period of four consecutive days. The overall SD of repeatability, S_r was 0.77, which corresponds to an RSD_r 2.44%. These results were exactly the same for original spindle method. *Intermediate reproducibility (between-day variability)* - The RSD of the intermediate reproducibility (RSD_R) in sugar-based juice was calculated at 17.46% for the proposed method versus 17.44% for the original method. (Table 1).

Table 1. Repeatability and Reproducibility comparison between DDM and Spindle

	DDM	Spindle
N^a	240	240
SD^b	1.76×10^{-05}	2.01×10^{-04}
S_r	0.08	0.08
$RSD_r, \%$	8.03	8.02
r^c	0.24	0.24
S_R	0.18	0.18
$RSD_R, \%$	17.46	17.44
R^d	0.51	0.51

^a N = Number of duplicate tests
^b SD = Standard Deviation
^c r = Repeatability values $S_r \times 2.8$.
^d R = Reproducibility values $S_R \times 2.8$.

Comparison of mean values of continuous variables between categories was performed by one-way ANOVA for both methods. (Table 2)

Table 2. Analysis of Variance (ANOVA) for digital density meter and original AOAC method.

	F Ratio	Prop > F
DDM-Day	0.3291	0.8043
DDM- Analyst	0.3852	0.5355
DDM-Instrument	0.2298	0.8756
Spindle-Day	0.1494	0.9300
Spindle-Analyst	0.0266	0.8706
Spindle-Instrument	0.2589	0.6113

Figure 1. DDM means and SD in days

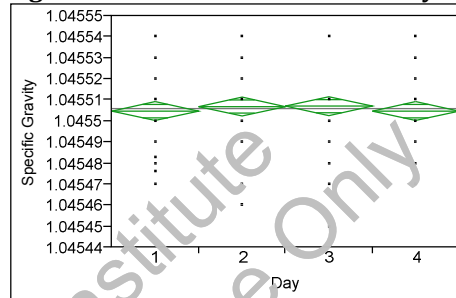


Figure 2. DDM means and SD by Analyst

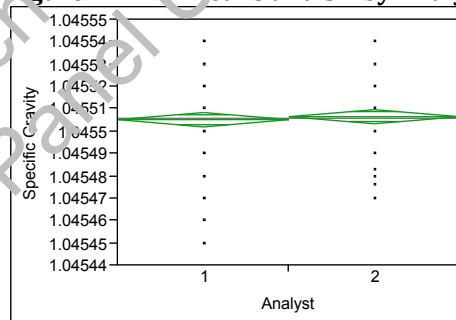


Figure 3. DDM means and SD by Instrument

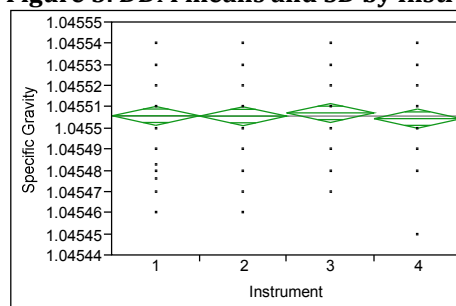


Figure 4. Spindle means and SD in days

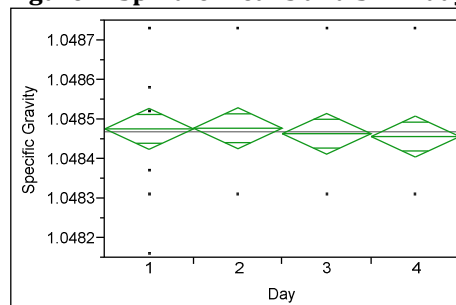
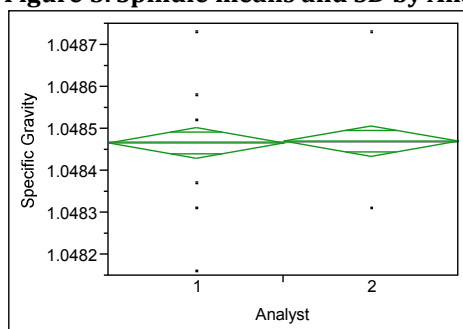
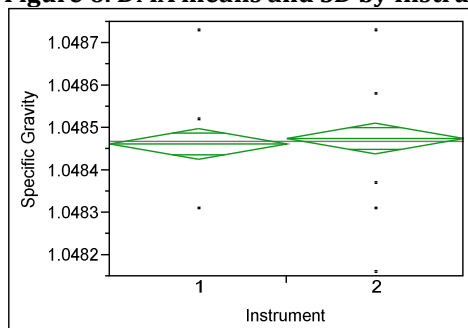


Figure 5. Spindle means and SD by Analyst**Figure 6. DMA means and SD by Instrument**

(b) *Side-by-side* - The mean specific gravity and Brix results for both methods in all side-by-side samples were equivalent. There was no statistical difference in the repeatability (s_r) and reproducibility (s_R) values between both methods for AJ, GJ, PJ and CJC (Table 3 and Table 4). Results for r^2 as correlation coefficient for both methods is 0.99995 indicating good correlation (Table 5). Results for Tukey-Kramer HSD showed that there was no statistical difference between means of results of repeated measurements of specific gravity and sugar Brix in side-by-side juice samples.

Table 3. Specific gravity in a side-by-side comparison of common samples apple juice (AJ) and grape juice (GJ) using DDM and Spindle.

	DDM-AJ	Spindle-AJ	DDM-GJ	Spindle-GJ
<i>N</i>	60	60	60	60
Mean	1.04792	1.04806	1.06533	1.06531
SD	1.96X10 ⁻⁰⁵	3.42X10 ⁻⁰⁴	1.73X10 ⁻⁰⁵	1.09X10 ⁻⁰⁴
s_r	0.08	0.08	0.08	0.08
RSD_r,%	8.02	8.02	7.96	7.96
<i>r</i>	0.24	0.24	0.24	0.24
s_R	0.18	0.18	0.18	0.18
RSD_R,%	17.44	17.44	17.30	17.33
R	0.51	0.51	0.52	0.52

Table 4. Specific gravity in a side-by-side comparison of common samples pear juice (PJ) and cranberry juice cocktail (CJC) using DDM and Spindle.

	DDM-PJ	Spindle-PJ	DDM-CJC	Spindle-CJC
<i>N</i>	60	60	60	60
Mean	1.05419	1.05414	1.05288	1.05285
SD	2.56X10 ⁻⁰⁵	1.71X10 ⁻⁰⁴	8.50X10 ⁻⁰⁵	1.85X10 ⁻⁰⁴
s_r	0.08	0.08	0.08	0.08
RSD_r,%	8.00	8.00	8.01	8.01
<i>r</i>	0.24	0.24	0.24	0.24
s_R	0.18	0.18	0.18	0.18
RSD_R,%	17.39	17.39	17.40	17.40
R	0.51	0.51	0.51	0.510

Table 5. Correlation between DDM and Spindle for common liquid sugar products.

	DDM-Mean	Spindle-Mean
AJ	1.04792	1.04806
GJ	1.06533	1.06531
PJ	1.05419	1.05414
CJC	1.05288	1.05285

Correlation, $r^2 = 0.99995$

Statistical significant was assured at $p < 0.05$. We performed all statistical analysis using JMP® 9.0.0, SAS Institute Inc. software.

Discussion

In this study, the **digital density meter** method was compared to the **AOAC 932.14A** "By Means of Spindle" method for solids in syrups measuring juice products. Four juice types were evaluated by both methods. Two intermediate studies for each method were conducted.

There were no statistically significant differences in the repeatability variance (s_r) or reproducibility variance (s_R) between the digital density meter and reference method in four of the four juice types tested. The proposed digital density method had significantly better measurement standard deviation (SD) in all the lots analyzed in this study. The lower SD is attributed to the differences in the test principle of digital density meter compared to the standard method. The digital density meter is a U-tube oscillation digital method while the standard method is a graduated spindle that relies on the technician for interpretation. Therefore the test principle of the spindle method is inherently more variable than the digital density meter method.

Recommendations for Modifications of AOAC Official Method 932.14

Based on the results of this study, a review of methods and literature. The author recommends the addition measuring option of U-tube oscillator.

The author suggests that the AOAC Official Methods Board consider the following as a minor addition to AOAC Official Method **932.14**:

D. By Means of Digital Density Meter with a U-Tube Oscillator:

References

- (1) Code of Federal Regulation. Part 21 Ch. I (4-1-11 Edition) §101.30(h)(1)
- (2) *Official Methods of Analysis* (2005) 18th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, Method **AOAC 932.14**
- (3) *Official Methods of Analysis* (2005) 18th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, Method **AOAC 988.06**
- (4) *Official Methods of Analysis* (2005) 18th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, Method **AOAC 942.33**
- (5) *Official Methods of Analysis* (2005) 18th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, Method **AOAC 900.01**
- (6) SAS Institute Inc. (2010) JMP®(data analysis software), version 9.0.0. www.jmp.com (accessed October 24, 2013)
- (7) Shachman, Maurice. "The soft drinks companion: a technical handbook for the beverage industry"
- (8) Physical Tests/ <241> Specific Gravity, (2012) 36th Ed. UNITED STATES PHARMACOPEIA



EXPERT REVIEW PANEL (ERP) SOLIDS IN SYRUPS



Is this method recognized or adopted by another agency outside of AOAC?

- 1) No
- 2) USP 841, used density meter with a U-tube oscillator
- 3) yes

USP Specific Gravity

IFU 1a Relative Density of Fruit Juices

- 4) USP (Physical Tests, 841 Specific Gravity, (2012) 36th Ed., UNITED STATES, PHARMACOPEIA also referenced by Reference book (Shachman, Maurice. "The soft drinks companion: a technical handbook for the beverage industry)
- 5) do not know
- 6) USP 841. This method is adopted for measuring sp gravity of liquids using digital density meter..

Does your organization support the proposed modification being submitted to AOAC? Please indicate explanations.

- 1) Yes, my organisation supports the proposed modifications. This modification is logical as the technique is published as AOAC OMA.
- 2) Yes we do.
Statistics domenstrates equivalency.
The densitometer has advantages over other methods of measuring Brix in that the DDM is less sensitive to PH, temperature, color and turbidity.
Also, automation is advantage
- 3) yes

Digital density meter specific gravity analysis is a well established technology and is now routinely used in many industrial laboratories

- 4) Yes
- 5) Yes, the digital density meter is a useful alternative to other density measurement methods. The performance of the digital density meter has been shown to be equivalent to the spindle method for the matrices apple, grape, pear and cranberry juice.
- 6) Based on my experience of 48 years, I have found the spindle and digital density meter are equivalent methods with digital density meter method with more precision w/tighter standard dev than spindle. This has been substantiated by actual data.

Does your organization, have any additional suggestions regarding the modification of this method?

- 1) No
- 2) no



EXPERT REVIEW PANEL (ERP) SOLIDS IN SYRUPS

- 3) It would be useful to have data over a wider range of specific gravity values. The data provided in the study covers only a fraction of the typical range for fruit juices, and includes no data for syrups or concentrates.
- 4) Listed as a part of the official AOAC method
- 5) in the update to Method 932.14 D.

Please add an applicability statement which clarifies the range of juices and their sugar content (i.e. Is it necessary to dilute concentrates?) as well as whether or not the juices may contain undissolved solids such as orange juice with pulp.

Under "Determinations": Add any precautions if pulp or other solids may interfere with the analysis.

- 6) NO



EXPERT REVIEW PANEL (ERP) SOLIDS IN SYRUPS

RECOMMENDATION OF PROPOSED MODIFICATION

- 1) Yes - I agree that the proposed changes can be implemented
- 2) Yes - I agree that the proposed changes can be implemented
- 3) Yes - I agree that the proposed changes can be implemented for OMA 932.14.
- 4) Yes - I agree that the proposed changes can be implemented for OMA 932.14.
- 5) Yes – I agree that the proposed changes can be implemented for OMA 932.14 with the following revisions
- 6) Yes – I agree that the proposed changes can be implemented for OMA 932.14 with the following revisions

Please explain and delineate for scientific reasons

Please explain the additional revisions:

- 1) No additional revision is required
- 2) see suggestions above. While this is definitely a change in technology and thus is technically a major change, I feel that the study conducted provides adequate information on the performance of the digital density meter and it would also be appropriate to make to an alternative technique within Method 932.14, assuming that the application statement includes only those matrices represented in the side-by-side study.
- 3) Include under method name: " specific gravity method of solutions containing sucrose, liquid sugar products, and other nonsucrose solids"
 - a) direct - add "solutions containing sucrose"
 - e) determination- remove "carbonated beverage reference" - redundant

RECOMMENDATION OF PROPOSED MODIFICATION LEVEL

- 1) Editorial Change
- 2) Minor Modification
- 3) Major Modification
- 4) Minor Modification
- 5) Minor Modification
- 6) Minor Modification

REVIEWERS

- 1) George Joseph
- 2) Mohamed Hamad
- 3) Dana Krueger
- 4) SNEH BHANDARI
- 5) Jo Marie Cook
- 6) Prem Virmani