AACC Approved Methods – Approval Process, Instructions, and Helpful Hints

What Is an AACC Approved Method?

AACC Approved Methods are methods that have been evaluated in a collaborative trial/study by a number of independent laboratories according to a systematic plan. A collaborative trial/study is designed to gather information on the performance of an analytical method in practical applications in real-world settings. If a collaborative study is deemed satisfactory on the basis of statistical analysis, the AACC Technical Committee and the AACC Approved Method Technical Board may then approve the method.

AACC Approved Methods were originally intended to be used to determine an accurate measure of the analyte of interest (a substance whose chemical constituents are being identified and measured) in food and feed matrices. An example of this type of method is: 14-60.01 Total Carotenoid Content of Cereal Grains and Flours. However, there now exist methods that are either qualitative (e.g., 04-28.01 Free or Combined Tartaric Acid—Qualitative Method) or that measure physical properties (e.g., 56-36.01 Firmness of Cooked Pulses). Guideline documents that are based on AACC Approved Methods also form part of the AACC Approved Methods of Analysis (e.g., 55-60.01 Guideline for Determination of Particle Size Distribution).

AACC Approved Methods are also primary methods that can be used to determine a reference value and to calibrate quick methods.

Many AACC Approved Methods are recognized by the Codex Alimentarius Commission as international standards for food and feed analyses.

Introduction to the Approved Method Development Process

You may have identified that your industry or service sector needs a new method, but where do you begin to progress the idea, get it written, and get it approved? Do you have a suggestion for a new method that will help you gain greater market acceptance for your product, make it easier for suppliers to deliver a high-quality product, or assist with meeting a procurement or regulatory requirement?

The process of proposing and eventually approving a new method can seem overwhelming, particularly if you have never before considered yourself as an author. However, the Cereals & Grains Association would like to offer the tools and advice required to get you started and assist you through each stage of the process.

The process consists of four phases: exploratory, planning, collaborative, and completion. A high-level overview of the process can be found here. This Helpful Hints document is meant to guide you through each phase of the process, providing gateways that should all be checked (☐) before you are ready to move on to the next stage of each phase.

The Cereals & Grains Association receives a variety of applications for development/approval of new methods. It should be noted that not all requests come to fruition in the end, as there may be insufficient interest from stakeholders, other methods that already exist that meet the particular need, etc.

Getting Started

A company, organization, trade association, professional society, university, government department, research group, or single person may propose a method and/or start the request for approval of a new method. To suggest a new AACC Method and begin the approval process, contact the AACC Approved Methods Board (AMB) or connect with a Technical Committee Chair.

A variety of information should be provided to request approval of a new method. The information is used to evaluate the level of interest and support needed to organize the activity, facilitate consensus between the activity participants, develop an efficient strategy for development of the method, promote business across the industry or profession, and develop successful marketing plans for the activity.

An established method of analysis currently recognized as a standard method from a different standards organization or used for different matrices can also be considered for adoption as an AACC Approved Method. In this case, documents used for method validation (collaborative trial reports or equivalent) by other organizations should be presented to the AACC AMB for review. However, the Cereals & Grains Association will not accept a method from another standards organization unless approval to co-publish is discussed and can be provided.

The method proposer should be prepared to join the assigned Technical Methods Committee (TMC) for discussions about the proposed method. They will act as an advocate for the method and provide supporting information, such as a description of the purpose and scope of the method of analysis and a description of potential users for which the method will be relevant. This phase can be challenging, and the proposer should plan accordingly.

Before the AACC AMB and the TMC review the new method, the proposer should clearly understand the value of AACC Method approval and describe any other discussions they may have held with other groups, such as standards organizations. The proposer needs to understand that the Cereals & Grains Association will hold copyright on all methods validated and approved within the AACC Method approval process. Cross-approval with other method organizations is considered on a case-by-case basis.

All methods need potential users and may require a sponsor. The assigned TMC must assess the importance and value of the method in cereal, grain, and pulse sciences. A sponsor for a new method may be needed, as there may be associated costs that need to be identified in consultation with the TMC Chair. Be aware that agreements may need to be put in place and that staff is available to help with this process.

Things to Consider and Prepare from the Start

☐ Create an outline of the method. There is no better time than at the start of the process to draft an outline of a proposed method. The content will need to be distributed to the AACC AMB and appropriate TMC to decide whether the method is required, worthwhile, suitable, and feasible. This method outline does not need to be the final detailed document—it can be revised in several drafts as the method progresses through the approval process. This outline should be marked as a draft document (watermark) and have the Cereals & Grains Association copyright in the footnote.

☐ As a method proposer, you may start to lobby interest for potential volunteers who will take part in your collaborative trial.

- A collaborative trial requires at least eight laboratories for statistical validation of the performance parameters. It is advisable to initially identify 12 labs. Often some of the labs will withdraw from a collaborative trial at various stages for unforeseen reasons.
- The involvement of many labs in the collaborative trial enables proper statistical analysis to demonstrate the reproducibility of the method and provides opportunities for improvements of each step in the method protocol.
- It is advisable to identify labs that will use your method in the future or labs that have a responsibility related to the analytical problem.
- It is beneficial to invite labs that employ personnel experienced in the basic techniques involved in the method under development; however, personnel are not required to have experience with the specific method for a lab to be selected. It is advisable to choose labs certified or accredited for similar methods.
- Use personal contacts, technical societies, trade associations, literature searches, or advertisements to identify collaborators.
- It is advisable to choose collaborators from diverse laboratories that have an interest in development of the method, such as regulatory agencies, industry, and institutions.
- It is acceptable to invite different laboratories from the same organization if their individual operations are independent of each other and they each have their own instruments, standards, reagents, and supervision.

☐ Your method is likely to involve equipment and/or instrumentation. For methods where the result produced can only be generated by specific equipment or instrumentation, all members of the collaborative trial will need to use the same equipment or instrumentation specified in your draft method in order to completely follow your method steps and optimize repeatability of the results. Where possible, different software versions should be investigated, as tools/features may differ and affect the collection of the same results.

☐ When instrumentation is used, it is recommended that your draft method be sent to an expert in the use of the equipment/instrument or to the manufacturer to determine if your proposed method can be optimized further. Often an expert is only sought after data is collected to assist in resolving anomalies. At this point, the expert may well have lost the opportunity to suggest improvements to the method and its outcome. If this is the case, you will wish you had sought assistance at the outset rather than face the possibility of having to rerun the collaborative trial to collect meaningful results.

People Required

- ☐ Method Proposer – A company, organization, trade association, professional society, university, government department, or single person may propose a new method. A proposer should prepare an outline of the method, contact the AACC AMB, and be prepared to provide appropriate information for a decision regarding starting the approval process.

  The Method Proposer should be part of the collaborative trial discussions and help find sufficient international laboratories for the trial.

- ☐ Method Leader(s) – A Method Proposer could be a Method Leader. However, the TMC Chair or another appointed person could also take this role. Appointing a coleader may also help with many tasks throughout the process. Method Leader(s) are responsible for coordinating all phases/steps of the method approval process, including the collaborative trials.

- ☐ Technical Methods Committee Chair – The TMC Chair arranges for the method to be discussed in GroupZones (the community site used for committee discussions) with committee members and other experts, appoints a Method Leader, supports the entire process, etc.

- ☐ Statistician – The Statistician provides guidance on the number of samples and replicates required for statistically significant data and provides statistical analysis of data after completion of the collaborative study. The Statistician is appointed by the chair of the Statistics Committee once the method is approved to move forward in the process.

- ☐ Trial Collaborators – Trial Collaborators participate in the collaborative trial.

- ☐ AACC Approved Methods Technical Board – The AMB is made up of all TMC Chairs/Co-chairs, the *Cereal Chemistry* Approved Methods Associate Editor, the Chair of the Statistics Committee, and the AMB Coordinator. The AMB receives the proposed method, makes a preliminary evaluation, and assigns the proposed method to an appropriate TMC.

PHASE I—The Exploratory Phase

This phase in the approval process is the period of due diligence during which information is gathered about the industry and the need for the proposed method. At this point the Cereals & Grains Association, through the AACC AMB, will establish whether there is enough support to proceed.

Some information gathering is essential to progress effectively at the beginning. The first step is to explore whether a standard method already exists for use in the field—contacts in trade associations, government agencies, or other standards development organizations, with the assistance of Cereals & Grains Association personnel, can be used to help in the search. Doubling efforts and parallel standardization activities lead to confusion in the market and deplete stakeholder resources for development of standard methods.

The AACC AMB will identify the TMC with the closest interest for the analyte(s) and/or property measurement or application of the method to be able to provide expert review of the concept/considerations and make the necessary connections.

Next, the assigned TMC and the Method Proposer will identify key stakeholders and contact them to ensure that there is agreement on the market relevance of the identified standards field and that the stakeholders are committed to participating in the project.

Once the TMC Chair has given approval for the creation/approval of a new method, the first draft will be drawn up by the Method Leader.

The Method Leader can access additional resources on requirements of Method documents on the Cereals & Grains Association website.

Once completed, draft documents are transmitted to the TMC Chair for sharing on the community site.

Steps and Checklist

☐ **The Method Proposer describes the analyte(s)/property(ies) to be measured and defines the purpose and scope of the method. The proposer also identifies the need and value of the proposed method to the cereals, grains, and pulses community.**

A method can be designed to measure an analyte in a specific material (e.g., (AACC Approved Method 08-12.01 Ash in Farina and Semolina) or in various materials (e.g., AOAC Intl. Official Method 991.42 Insoluble Dietary Fiber in Foods and Foods Products). AOAC Method 991.42 was tested in 22 different materials, and the performance results are listed for each.

The material (sometimes called the matrix) is the medium that contains the analyte or possesses the physical property to be measured. If the proposed method is applicable to multiple matrices, several materials are eventually required for a collaborative study. The materials used as unknowns in the collaborative study should represent common matrices that the method is likely to be used to analyze.

It may be that at this early stage it is possible to measure several parameters of interest with your method. It is better to propose a wider scope and parameter collection (if available) at this early stage. At the point at which collaborative results are gathered and data is analyzed, the scope may need to be narrowed if certain parameters are not repeatable. If this is necessary, you will have the ability to retain the "best" parameters, which will be included in your final method. Once the collaborative tests have been performed, you will not be able to add parameters to or expand the scope of your method unless you repeat the collaborative trial.

It is important to decide on the range of analyte concentrations for which the method will be applicable. The collaborative study must test the full range of concentrations as indicated in the method's scope. Eventually, you will need to prepare samples with various concentrations of the analyte. For example, if the method is to be used to estimate protein concentration in flours from 10 to 16% protein, then the collaborative study must be conducted with flours containing 10 to 16% protein. If zero is in the area of interest, prepare blanks and consider a series of tests to determine the levels of detection and quantitation. Measuring near zero has some special considerations for statisticians and will need to be discussed with your statistician. Also, different procedures should be used for qualitative (i.e., present/absent, high/satisfactory) testing. Blanks used for calibration or practice runs are not considered as one of the five materials. Materials with naturally occurring concentrations are preferred over spiked samples whenever possible. Materials must be homogeneous and stable in order to withstand shipping to collaborators. Heterogeneity can cause outliers and will increase variance estimates that are not due to the intrinsic method variability.

☐ **The Method Proposer contacts the AACC AMB with their proposal to obtain the AMB's initial expression of interest and referral to the appropriate TMC.**

☐ **The assigned TMC considers and evaluates the proposal and confirms the proposed method meets a defined need for cereals, grains, and pulses and is practical. The TMC approves the start of the method approval process. The TMC assigns the Method Leader.**

☐ **A Statistician is appointed by the chair of the Statistics Committee to mentor and guide the validation steps and the eventual collaborative study, as well as to provide statistical analysis of data upon completion of the collaborative study.**

☐ **The Method Leader, with the help of the TMC, finalizes the proposal/document describing the analytes/properties to be measured and the purpose and scope of the method. All Word documents should now be marked with a Cereals & Grains Association copyright and a "draft" watermark.**

☐ **A draft of the written version of the proposed method is prepared, and validation steps are planned by the Method Leader and TMC.**

- The method steps are identified, listed, and clearly described; it is advisable to use a format and style close to those that will be used for eventual publication.
- The use of imperative directions is advisable.
- Critical points and convenient stopping points should be identified.
- Photographs and illustrations are useful to avoid misinterpretation and minimize improvisation.
- Exact matrices and range of concentrations are identified.
- Accuracy, precision, repeatability, specificity, detection limit, and quantitation limit are defined.
- Safety aspects should be considered, and safety precautions should be clearly described.
- The written version of the method should be edited for completeness and clarity.

In consultation with the Statistician, you may decide to run an unofficial mini-/pre-collaborative study with two or three friendly labs (optional and subject to time and resources) to see if the method has any readily identifiable and fixable problems that were not observed in your own lab during the development of the method and the single-lab validation (SLV). This mini-/pre-collaborative study can also ensure that the samples are homogeneous and stable. The data from this mini-/pre-collaborative study can be included in the analysis of the official collaborative study if no changes or improvements have been made.

Before you proceed with a mini-/pre-collaborative trial ask a colleague or other analyst who is not involved in your method development to review the instructions for clarity and details. It is wise to revise the method at this stage based on questions that were raised or problems encountered by misinterpretation of your written method.

PHASE II – The Planning Phase

☐ **Method Leader prepares a final draft of the method using the AACC Methods style for review within the TMC.**

☐ **Collaborative trial is planned.**

☐ The Statistician is consulted to mentor and guide setup of the collaborative study. Both the Method Leader and the TMC Chair participate in the consultation.

It is recommended that you consult with the Statistician at the earliest opportunity regarding your intended matrix of samples and parameters you wish to collect. Your Statistician can then confirm that you are intending to test the correct number and range of samples as specified in the method "scope." It is far easier for your Statistician to guide you before you have collected a large amount of data, since sometimes the advice that is given can affect the optimization or outcome of your data collection and this advice is not something you wish to receive at a late stage in the method approval process. At this point, it is also advisable to consult the following references:

- Nelsen, T. C., and Wehling, P. (2008). Collaborative studies for quantitative chemical analytical methods. Cereal Foods World 53:285-288. DOI: 10.1094/CFW-53-5-0285.
- AOAC International. (2002). Appendix D: Guidelines for collaborative study procedures to validate characteristics of a method of analysis. *AOAC Official Methods of Analysis*. AOAC International, Rockville, MD.

These references explain in detail about sample choice and preparation and about the statistical analyses that will be performed on your data. It is recommended that you understand the calculations to optimize conversations with your Statistician and understand terms such as accuracy, precision, repeatability, reproducibility, Youden pairs, standard deviation, etc., as you will need to use these in your final report to explain the quality of your data. Your assigned Statistician will be available to help throughout this process.

☐ Statistician approves final collaborative study design.

☐ Trial resources determined and identified (samples, shipping, calibration process, funding required).

- Code/label the test samples in a manner such that it is not obvious which samples are blind duplicates and the samples are not analyzed in a set order.
- Prepare sets of blind or matched duplicates (a pair is considered one material) as unknowns (blind) and code in a random pattern. There is no need to test triplicates; duplicates are sufficient to estimate internal variance. Rather than designing a study with five triplicates, use resources to design a study with seven or eight duplicates.
- Consider the containers that will be used for shipping the samples: extraneous analytes cannot be contributed to the contents, and analytes or other constituents from the matrix (e.g., water) cannot be adsorbed by the containers.
- The materials may need to be stabilized (e.g., freeze, dehydrate, add preservatives or antioxidants). This should be done in such a way that it does not affect the performance of the method. Any changes to the composition of materials must be avoided; consider using protective packaging solutions, such as vapor-tight containers, flushing with an inert gas, refrigeration, or other options.
- Only in exceptional cases (e.g., with unstable materials) should collaborators be allowed to prepare their own materials for analysis. In such instances, it is advisable that an individual not involved in the analysis be asked to prepare the materials.

- It is advisable to prepare more laboratory sample sets (e.g., 25%) than there are study collaborators. Often, some of the packages do not reach collaborators, new laboratories are asked to participate in the collaborative trial, or some sample sets may need to be analyzed again to verify stability during storage.

☐ The Method Leader, with the help of the TMC, identifies collaborators, shares the proposed method with potential collaborators, and confirms their ability to participate in the collaborative study.

- The collaborative study requires at least eight laboratories for statistical validation of the performance parameters. We suggest that you initially identify 12 or more laboratories, as some may not be able to participate within the desired timeframe.

☐ Final collaborators are confirmed and notified of the timeframe for receiving samples, testing, and returning results.

- A formal letter to each collaborator is prepared stating the reason for selecting that lab (e.g., familiarity with the problem or method), listing the estimated number of staff-hours required to complete the study, number of test samples to be received, number of analyses required, expected distribution date for the test samples, and target completion date for the study.
- A copy of the written version of the method is included.
- It is advisable to request a reply to the invitation by a specific date.

PHASE III – The Collaborative Trial Phase

☐ **The Method Leader arranges for the method and test samples to be shipped to collaborators. Collaborators are trained and instructed to follow steps exactly. Timelines are clearly communicated.**

It is advisable to consult the following references for proper conduct of the collaborative trial:

- Nelsen, T. C., and Wehling, P. (2008). Collaborative studies for quantitative chemical analytical methods. Cereal Foods World 53:285-288. DOI: 10.1094/CFW-53-5-0285.
- AOAC International. (2002). Appendix D: Guidelines for collaborative study procedures to validate characteristics of a method of analysis. *AOAC Official Methods of Analysis*. AOAC International, Rockville, MD.

☐ **Collaborative trial is completed. Data is collected from collaborative labs.**

☐ **Data is analyzed.**

☐ **Data is discussed with TMC Chair and Statistician.**

☐ **A draft of the Collaborative Trial Report is prepared.**

- It is advisable at this stage to prepare the report according to the requirements and guidelines of *Cereal Chemistry*.

PHASE IV – The Completion Phase

The method draft must be reviewed and approved by the voting members of the TMC assigned at the beginning of the process. All voting members are expected to contribute to this process. Once approved by the TMC, the method can be submitted to the AMB (and the AMB Coordinator).

☐ **Collaborative Trial Report is reviewed and approved by the assigned TMC.**

- Once approved by the assigned TMC, the report is submitted via Manuscript Central to *Cereal Chemistry*. A copy of the report is also submitted to the AMB Coordinator for record keeping.
- The *Cereal Chemistry* Approved Methods Associate Editor invites two reviewers to conduct in-depth reviews of the Collaborative Trial Report. The comments are sent back to the author(s) to respond to the reviewer(s') comments and revise the manuscript.
- After revisions, the Collaborative Trial Report is sent back via Manuscript Central for final acceptance by the *Cereal Chemistry* Editor-in-Chief.

☐ **AMB reviews the method and Collaborative Trial Report (version accepted for publication in *Cereal Chemistry*) and votes on the method under consideration.**

- Votes and comments are collected and communicated by the AMB Coordinator to the Method Leader and TMC Chair for editing of the method.
- The AMB Coordinator performs the final check of the documents before publication. In case of controversy, the AMB will be consulted.

☐ **Enhancement(s) (calculators, photos, video, etc.) for the method can be submitted along with the final draft for voting or prepared after the approval process.**

- Method enhancements require the approval of the Method Enhancements Committee.

☐ **Publication of the new method in the *AACC Approved Methods of Analysis* manual and publication of the Collaborative Trial Report in *Cereal Chemistry*.**

- The Collaborative Trial Report will be referenced in the published method.

HELP & RESOURCES

Templates, guidelines, and other documents have been designed to help speed the process as you draft a new AACC Approved Method. Visit the Approved Methods of Analysis Contacts and Resources webpage for more available resources.

Nelsen, T. C., and Wehling, P. (2008). Collaborative studies for quantitative chemical analytical methods. Cereal Foods World 53:285-288. DOI: 10.1094/CFW-53-5-0285.

AOAC International. (2002). Appendix D: Guidelines for collaborative study procedures to validate characteristics of a method of analysis. *AOAC Official Methods of Analysis*. AOAC International, Rockville, MD.

Contact Information

info@cerealsgrains.org

Modifying Existing Method Documents

If an AACC Approved Method requires revision, your first step will be to contact the TMC Chair responsible for the method to explain the rationale for the changes you are proposing and to request the proposed revisions be considered.

An electronic Word document containing the AACC Approved Method will be sent to you.

In the document, clearly indicate your proposed changes. The preferred formats for specifying changes are as follows: underline text that is added and ~~strikethrough text~~ that is deleted. Ensure that vertical bars indicating sections for revised text are clearly visible in the page margin.

If changes are suggested for only a small part of the approved method, only the revised section(s) need to be submitted. Please copy and paste the revised section(s) into a separate document and verify that the number and exact year and date for the method are clearly displayed in the revised document.

The approval process for method changes will depend on the degree of the proposed changes. Edits to Notes, minor clarifications, and changes to the Suppliers Guide may simply require approval from the assigned Technical Committee. Changes that are more significant or alter the original method will require the approval of the AMB. Each case will need to be considered individually.