

**AOCS Procedure M 2-65**

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# Writing and Approval of Methods

**PART I—ADOPTION AND PUBLICATION OF METHODS**

Article VIII, Section 1 of the Bylaws of the American Oil Chemists' Society reads as follows: "Official Methods and Recommended Practices." The society shall investigate, adopt and publish such uniform methods of analysis in the field of oils, fats, and related materials as may appear to be in the public interest, convenience or necessity." This publication shall be called *Official Methods and Recommended Practices of the American Oil Chemists' Society*.

To facilitate careful consideration and orderly adoption of methods of analysis, the Uniform Methods Committee (UMC) has established the following procedure:

1. Every method must be assigned to a technical committee, subcommittee or task group and have committee approval before the method can be recommended to the Uniform Methods Committee.
2. The technical committee chairperson will obtain this approval by a ballot of all committee members, furnishing the members with a copy of the method and supporting data.
3. Approval shall consist of a two-thirds affirmative vote of those voting on the method. A negative ballot must be accompanied by a reason for the negative vote. To be a legal ballot, 60% of the ballots sent out must be returned.
4. The technical committee chairperson will submit to the AOCS Technical Services copies of all methods and supporting data sent out with the committee ballot. These will be sent to the members of the Uniform Methods Committee. Any comments by UMC members will be sent to the AOCS Technical Services, who will pass them on to the technical committee chairperson for consideration.
5. A method receiving no negative ballots shall immediately be recommended to the Uniform Methods Committee.
6. If a method receives the necessary two-thirds affirmative ballots but has one or more negative ballots, the committee chairperson may (a) recommend the method (accompanied by all negative ballots) be forwarded to the Uniform Methods Committee or (b) hold the method for immediate discussion and resolution of the negative ballots. All members casting negative ballots shall be contacted by the committee chairperson.
7. The Uniform Methods Committee members shall have one month to consider and return the ballots. All negative ballots must be accompanied by reasons for negative vote.
8. Methods receiving no negative ballots will be submitted directly to the editor of *Official Methods and Recommended Practices of the American Oil Chemists' Society* for publication without further discussion by the Uniform Methods Committee.
9. Methods receiving one or more negative ballots will be reviewed with the committee chairperson.
10. A negative ballot may be resolved by (a) withdrawal of the negative ballot by the member casting the ballot or (b) a vote of two-thirds of the committee members to override the negative ballot.
11. If the negative ballots are resolved by the Uniform Methods Committee, the method is submitted to the editor for publication. If the negative ballots are not resolved, the method is returned to the technical committee for further study.
12. To achieve its objectives of developing and validating analytical methods, the society may encourage publication of reports of its validation studies in either the *Journal of the American Oil Chemists' Society* or *INFORM*. It issues the resulting officially adopted methods in *Official Methods and Recommended Practices of the AOCS*.

**PART II—REVIEW AND UPDATING OF EXISTING METHODS**

Article VIII, Section 3 of the Bylaws of the American Oil Chemists' Society reads as follows: "All Official Methods and Recommended Practices shall be updated yearly through Additions and Revisions to Methods. In addition, all methods shall be completely reviewed at five-year intervals by qualified persons (known as 'associate method editors')." "

The methods published by the society are used for referee purposes and, therefore, must be the most accurate and precise methods available. To keep the Official Methods in line with the changing technology in analytical determinations, the Uniform Methods Committee has established the following procedure:

1. Every method now published in *Official Methods* is assigned to either a technical committee or an associate methods editor.
2. Each year, the technical committee chairperson (or the designated subcommittee chairperson) or the associate methods editor will review the assigned methods to ascertain their status in light of current technological developments.
3. A revision in a method must be submitted to the AOCS Technical Services for submission to the Uniform Methods Committee.
4. All AOCS methods shall be reviewed every five years. The Uniform Methods Committee and the editor shall "reapprove" the methods. The reapproval date is to be noted on the method.

**PART III—DEFINITIONS**

1. Official Method—an official method is a method that has been approved by the sponsoring technical committee and the Uniform Methods Committee and adopted by the Governing Board of the AOCS.

2. Recommended Practice—a Recommended Practice is a method that may be of interest or value, but does not have official method status.
  3. Quality Assurance—assurance is the overall concept by which the quality of analytical results generated by a laboratory is assured. This concept encompasses such mechanisms as choice of methods, skills of personnel, calibration of equipment, sample handling and preservation, maintenance of records, reporting of results, quality control considerations, laboratory facilities, etc.
  4. Quality Control—control is one of the mechanisms of quality assurance and encompasses such laboratory activities as maintenance of quality control charts, standard additions, calibration curves, recovery studies by additions and dilutions, participation in proficiency testing or collaborative studies, replication of analyses, split sampling, equivalency testing, preservation of samples, use of certified standards, editing of results, etc.
  5. Precision—the precision of a method usually is associated with inherent random errors and is expressed in terms of its standard deviation or, preferably, relative standard deviation (coefficient of variation). Precision usually refers to the repeatability of a result, or the “grouping” of results that would reveal “scatter” around a mean value which, with close grouping, would indicate a method of high precision (see AOCS Procedure M 1-92).
  6. Accuracy—the accuracy of a method usually is described in terms of its systematic error but is measured in terms of its ability to reflect the “true” value of a system, or its ability to hit the targeted value. An analytical method may have poor precision and yet be accurate; conversely, it may be inaccurate and have good precision. However, the ideal method would have relatively high precision grouped around the targeted value and limited random and systematic errors.
4. Apparatus—sources of apparatus and equipment must be indicated and, with the exception of a sole source supplier, all appearances of endorsement must be avoided. A general description of the apparatus may be used and is preferred. Cases of custom-made equipment and apparatus should be avoided if at all possible, but, if these are necessary, full specifications, drawings or diagrams should be provided.
  5. Reagents—all reagents used in the method must be listed. In some methods, special solutions and/or standards may be prepared. This should be noted in separate lettered and numbered sections titled “Preparation of Solutions” and “Preparation of Standards.” Grades of chemicals should be indicated in the method in keeping with both Section H of *Official Methods* and the guidelines that follow.
 

Chemicals are made in several grades based on quality and purity. These qualities are designated by names which vary somewhat among manufacturers. In general, the quality of chemicals used in testing must be high. It will be necessary for the purchaser to read the quality designations in the catalog being used, as designations vary considerably with manufacturers. Although not complete, the following list will give some idea of the grades of chemicals available:

    - (a) Primary standards—primary standards of high precision assay are suitable as references and in the preparation of standard solutions.
    - (b) ACS reagent grade—for some chemicals, the American Chemical Society has established standards. Most manufacturers making chemicals meeting these standards list them as “ACS Reagent Grade.”
    - (c) Reagent grade—this designation is used by some manufacturers to label chemicals of high quality. Many chemicals of reagent grade carry the manufacturer’s analysis on the label.
    - (d) USP grade—USP-grade chemicals meet the specifications of the *United States Pharmacopeia*, a publication establishing standards for chemicals and drugs used as pharmaceuticals.
    - (e) Other grades—there are other designations of quality such as “Spectro,” “Highest Purity,” or the manufacturer’s name grade, all of which may be very high quality, but will necessitate reading the quality grade designations in the preface of most catalogs to determine the qualities listed.

#### PART IV—FORMAT FOR AOCS METHODS

Methods proposed for inclusion in *Official Methods* should conform in style and be in keeping with the foregoing definitions and the guidelines which follow. An outline of methods format follows the guideline section.

##### Format Guidelines

1. Method numbers—each method should carry a page heading to show the section of the book for which it is intended. Specific method numbers will be assigned by the editor, but if the method revises or replaces an existing method, the present number should be indicated.
  2. Title—each method should have a specific, descriptive title.
  3. Definition—the definition should indicate what analyte is determined by the test, how the determination is made (GC, HPLC, AA, solvent extraction, etc.) and any special principle(s) involved that may be necessary for a better understanding of the method (balanced equations, color changes, wavelengths, special chromatographic columns, etc.).
  4. Scope—the scope should describe the product and/or matrix to which the method applies and any similar products or matrices. Limitations, applicable concentrations and any known interferences (matrix, spectral, etc.) should be noted.
7. Procedure—instructions in the procedure should be clear and succinct, usually with no more than one thought to a line and following in logical continuity to the conclusion.
  8. Sample requirements—where appropriate.
  9. Calculations—all calculations should be shown, including explanation of derivation of all factors, whether they be chemical or mathematical.
  10. Precision—All AOCS methods require precision data. The precision (and accuracy if possible) of the method must be presented as shown in Table 1 in AOCS Procedure M 1-92. The precision and accuracy (see

Part III, 5 and 6) data must be generated during the collaborative study. The method should contain statements on repeatability and reproducibility limits, similar to the examples given in AOCS Procedure M 1-92. If a method is adopted from another organization, citation must be given to the publication containing the statistical analysis of the organization's collaborative study. See AOCS Official Method Cd 12b-92 as a typical example.

11. Notes—the notes should contain information of particular importance to the method, such as
  - (a) Safety considerations—practices that may involve hazards of a carcinogenic nature, flammability, use of fume hoods or dangers from ingestion or inhalation, etc., should be prefaced “*Caution.*”
  - (b) Sensitivity—response per unit concentration.
  - (c) Detection level—minimum measurable concentration above the background level.
  - (d) Reporting—manner of reporting results, such as units of measurement and significant figures, should be explained.
  - (e) Interferences—interferences are stated briefly in the Scope of the method; however, if methodology exists for either reducing or eliminating the interference, it should be noted.
12. References—references should be included for purposes of background information, with due credit given to the original publication of the method and in consideration of proprietary information. References must include published results of collaborative studies, whether performed by the AOCS or other organizations. If the method is an official method of another organization, reference must also be made to the organization's official methods publication.
13. Other considerations—
  - (a) Authorship—self-explanatory, but is used to provide a contemporary source of background information.
  - (b) Style—the method should be written in the imperative voice.
  - (c) Figures—original black and white drawings or glossy photographic prints must be provided to the editor for any figures to be included in the printed method.
  - (d) Standards for accuracy—if standards exist for checking the accuracy of the method, they should be noted in the method write-up, along with information on where the standards may be obtained.
14. Committee responsibility—the technical committee proposing the method is responsible for its accuracy. It is important that numerical values, spelling, calculations and all laboratory directions be checked carefully. The method should be written in AOCS Methods format prior to the collaborative study (see AOCS Procedure M 4-86).

## PART V—DESCRIPTION OF AOCS METHODS

### *AOCS Official Methods*

A method will be granted AOCS Official Method status only after (a) an acceptable collaborative study, (b) proper statistical criteria have been met, (c) approval of the technical committee, (d) approval of the Uniform Methods Committee and (e) the collaborative study results have been published, or at least submitted for publication. In lieu of (a) and/or (b), a method may be adopted, but only as a Recommended Practice.

### *AOCS Recommended Practices*

A recommended practice is a method that may be of interest, but does not have Official Method status. The method is still subject to the approval process noted in this procedure. A recommended practice may or may not be based on a collaborative study. For example, the statistical evaluation of an attempted collaborative study (or repeat studies of the same method) may reveal the variation of the method to be outside the acceptable performance range (assuming that all other obvious errors have been corrected). If there is a high degree of interest, the method may be adopted still, but only as a Recommended Practice. The interest in such a method may be based on having a simpler, more rapid or qualitative procedure either as an adjunct to an official method or for use in general.

### *AOCS Standard Procedures*

AOCS Standard Procedures are guidance documents or specific methodology that requires the use of specific equipment according to the manufacturer's instructions. Standard Procedures require both pre-collaborative and collaborative study and require approval by an AOCS Methods Subcommittee and the Uniform Methods Committee.

## PART VI—ADOPTION OF METHODS FROM OTHER ORGANIZATIONS

Methods of other organizations may be adopted as AOCS Official Methods, provided criteria are met as follows:

1. The method must be based on a published collaborative study.
2. The collaborative study must, at a minimum, be based on the guidelines noted in AOCS Procedure M 4-86, with acceptable supporting statistical data.
3. The method must be rewritten into AOCS Official Methods format, with supporting statistics included.
4. The AOCS methods write-up must include the journal reference to the collaborative study, as well as the appropriate reference to the other organization's official methods publication.
5. After being rewritten into AOCS format, the method must be approved by AOCS Procedure M 2-65.