

Writing and Approval of Methods

PART I—DEFINITIONS

1. *Official Method*—An official method is a method that has been approved by the sponsoring technical committee, undergone a collaborative study with at least 8 successful collaborating laboratories, and been approved by the Uniform Methods Committee.
2. *Recommended Practice*—A Recommended Practice is a method that may be of interest or value, but does not have official method status. These may include methods lacking the required level of collaborative study results.
3. *AOCS Standard Procedure*—A Standard Procedure is a method that relies on the use of specific apparatus in accordance with the manufacturer's instructions. These procedures are reviewed and studied to meet official method status. In cases where the technology may be used in place of an official method, parallel equivalence studies are also performed.
4. *AOCS Analytical Guidelines*—Analytical Guidelines provide both a list of methods applicable to the determination of key parameters in a matrix and a description of their applicability together with pertinent notes and comments.
5. *Precision*—The precision of a method expresses the closeness of agreement (degree of scatter) between a series of measurements obtained from multiple sampling of the same homogeneous sample under the prescribed conditions. Precision is expressed in terms of standard deviation of the repeated measurements, both as is and expressed as a percent relative standard deviation (also called *coefficient of variation*) (see AOCS Procedure M 1-92).
6. *Repeatability*—Repeatability is the precision of a method determined from results obtained with the same method by the same operator using the same equipment in the same laboratory within short intervals of time.
7. *Reproducibility*—Reproducibility is the precision of a method determined from results obtained with the same method by different operators on different equipment in different laboratories.
8. *Accuracy*—The accuracy of a method expresses the closeness of agreement between the value produced by the method and the value which is accepted as the true value or an accepted reference value. This is sometimes termed trueness. In the absence of a reference material of known value, accuracy is often determined by spiking a known amount of added analyte into a matrix and determining recovery of the added amount.

PART II—FORMAT FOR AOCS METHODS

Methods proposed for inclusion in *Official Methods and Recommended Practices of the AOCS* should conform in style and be in keeping with the foregoing definitions and the guidelines which follow. The method should be written in AOCS Methods format prior to the collaborative study. An outline of methods format follows the guideline section.

Format Guidelines

Note—When selecting a method for purchase online, purchasers see only the Title, Definition, and Scope. It is important the information in those sections is adequate for correct method selection.

1. *Title*—Each method should have a specific, descriptive title describing the analysis that is being done and the matrices where the method is applicable.
2. *Definition*—The definition should be short and concise, and indicate what analyte is determined by the test, how the determination is made (GC, HPLC, AA, solvent extraction, etc.). Additional special information should be in the definition only when it is required for determining applicability of the method.
3. *Scope*—The scope should describe the products and/or matrices to which the method applies. Limitations, applicable concentrations and any known interferences (matrix, spectral, etc.) should be noted. The reader should be able to quickly judge from the Scope whether the Method is appropriate to their product or matrix. Indicate interferences briefly in this section.
4. *Apparatus*—Methods restricted to specific vendors should be minimized and equipment should be generically described, followed by possible vendor sources only if needed. All appearances of endorsement must be avoided. 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 a numbered list. List each reagent with a typed number followed by a full stop (.). Do not rely on the autonumbering feature of your word processing program. The use of CAS Registry Numbers is encouraged. Grades of chemicals should be indicated in the method in keeping with both Section H of *Official Methods and Recommended Practices of the AOCS* and the guidelines that follow. In general, the quality of chemicals used in testing must be high. Quality designations vary by manufacturer, where possible the method should not use proprietary designations for chemical quality. 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. Do not abbreviate the designation.
6. **Solutions**—In some methods, special solutions and/or standards may be prepared. This should be noted in separate lettered and numbered subsections in the Reagents section titled “Preparation of Solutions” and “Preparation of Standards.” Explicit instructions for making solutions and preparing standards should be provided.
 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. They should provide an unambiguous set of instructions on how to perform the method, not a just a description. Start each step with a typed step number followed by a full stop (.); do not rely on the autonumbering feature of your word processing program. Each method must be complete, without the need to refer to another method. If part of the method is carried out according to another method, the latter can be referred to in the References section.
 8. **Calculations**—All calculations should be shown, including explanation of derivation of all factors, whether they be chemical or mathematical. The exact manner in which analyte concentration is to be calculated must be specifically and clearly stated in this section. Any instructions for reporting results also need to be clearly stated in this section.
 9. **Precision**—All AOCS methods require precision data; the precision and accuracy (see Part I, “Definitions,” 5 and 8) data should be generated during the collaborative study. Unless the method is being adopted from another organization the collaborative study must be designed and carried out as outlined in AOCS Procedure M 4-86. The precision (and accuracy if possible) of the method must be presented as shown in Table 1 in AOCS Procedure M 1-92. 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.
 10. **Notes**—Important instructions for the steps required to carry out the Procedure should never be given in notes, they should part of the Procedure. The notes should be numbered and contain other 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 “see *AOCS Laboratory Safety*.”
 - (b) Sensitivity—Response per unit concentration.
 - (c) Limit of Detection—minimum measurable concentration above the background.
 - (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.
 - (f) Authors are free to add any comments they think will be useful in the Notes, including a lengthy description of theory if that is appropriate.
 11. **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. The reference section must include the 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.
 12. **Figures and images**—all figures and images must be supplied in high resolution (at least 300 dpi) in separate, individual .jpg or .eps documents. Figures of sufficient resolution can be extracted from a .pdf if the resolution of the original image was at least 300 dpi. They cannot be extracted from Word documents at sufficient resolution. Chromatograms, spectra, diagrams of specialized equipment and other technical illustrations should be included wherever possible.
 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 mood, meaning each instruction should be in the form of a command.
 - (c) Reference standards for accuracy determination—if reference 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.

- (d) The Method number and book section are assigned by the Editor-in-Chief. If the method revises or replaces an existing method, the present number should be indicated.
 - (e) Numerical data in tables must be set to align on the decimal. Numerical data include LOD, LOQ, Collaborative study data, spikes, etc.
14. After the collaborative study and statistical analysis are completed, the validation data and collaborative study will be added in a section at the end of the method. This validation section should include a complete description of the types of samples analyzed and the results that were achieved. The society may encourage publication of reports of its validation studies in either the Journal of the American Oil Chemists' Society or INFORM.
 15. Committee responsibility—The technical committee or subcommittee that reviewed the method and recommended adoption to the Uniform Methods Committee 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.

PART III—DESCRIPTION OF AOCS METHODS

AOCS Official Methods

A method will be granted AOCS Official Method status only after (a) approval of the draft of the method by the appropriate technical committee/subcommittee, (b) an acceptable collaborative study carried out as outlined in AOCS Procedure M 4-86, (c) proper statistical criteria have been met, (d) approval of the procedure and the results of the collaborative study by the Uniform Methods Committee, and (e) the collaborative study results have been published, or at least submitted for publication. An Official Method requires a minimum of 8 successful collaborating laboratories. A method lacking good results from at least 8 laboratories cannot become an Official Method but can be designated a Recommended Practice, at the discretion of the Uniform Methods Committee. Methods intended for international use, such as adoption as ISO methods, require successful collaborative study results from 8 laboratories in at least 5 countries.

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. The usual reason a method becomes a Recommended Practice is that there were insufficient successful laboratories in the collaborative study (less than 8) and an additional collaborative trial is not planned.

AOCS Standard Procedures

AOCS Standard Procedures require the use of specific equipment, often from a single manufacturer, according to the manufacturer's instructions. The review of AOCS Standard Procedures, including a collaborative study conforming to AOCS Procedures M 4-86, is carried out in the same manner and to the same standard as Official Methods. Standard Procedures require approval by an AOCS Methods Subcommittee and the Uniform Methods Committee.

AOCS Analytical Guidelines

AOCS Analytical Guidelines are guidance documents detailing the AOCS methods suitable for analysis of key parameters in a particular matrix and giving a description of their applicability together with pertinent notes and comments.

PART IV—DEVELOPMENT AND SUBMISSION OF METHODS TO AOCS

A proposer or developer of a new method obtains a copy of the AOCS Methods Template, a copy of "Proposer steps for Official Methods and Recommended Practices of the AOCS.docx," and a copy of this Procedure from the AOCS Technical Department and drafts their method as outlined in them. The method draft is sent to the AOCS Technical Director for review. The review determines whether the draft is sufficiently clear and complete to allow a laboratory that is unfamiliar with the method to carry it out. If the method draft does not meet this criterion, the AOCS Technical Department advises the method proposer of the deficiencies and returns the draft for revision. The method proposer returns a revised version to AOCS, which is reviewed by the Technical Director; this process is repeated until a satisfactory draft is developed.

The method draft is sent to the appropriate Subcommittee of the AOCS Uniform Methods Committee for review and approval. This review is expected to be finished within one month. The method must be written in its final form (similar in appearance to published Official Methods) and approved by the Subcommittee before the collaborative trial can begin.

The proposed experimental design for the collaborative study is sent to the chairperson of the UMC before the full-scale collaborative study can begin; approval by the chairperson is required before beginning the collaborative study.

After consultation with the method developer and AOCS staff, a study director is appointed. The study director, with the assistance of AOCS staff, is responsible for finding labs for the collaborative study, preparing at least five test samples for each collaborative study participant, overseeing the collaborative study to ensure it is carried out as directed in AOCS Procedure M 4-86, and compiling and reporting the results to AOCS. A precollaborative study among the labs intending to participate in the full collaborative study is recommended but is not required. Precollaborative studies are often carried out with a limited number (1–4) of samples. A precollaborative study can discover errors and detect labs that are not following the procedures prior to the full collaborative study.

It is recommended that study director seek at least 12 labs when planning a collaborative study. An AOCS Official Method requires successful completion of the collaborative trial by no fewer than eight laboratories, with a minimum of five test samples sent to each laboratory. International methods (such as those adopted by ISO) require successful results from at least 8 laboratories in at least 5 countries (see ISO standard 5725, part 2).

Collaborative samples are sent by the study director, with a deadline for returning results. It is recommended that a reference, practice, or performance evaluation sample be included with instructions not to analyze the collaborative samples until a specified degree of recovery, repeatability and/or other attribute has been achieved.

After the Study Director compiles and reports the results to AOCS, AOCS carries out statistical analysis as outlined in AOCS Procedure M 4-86. Results of the statistical analysis are examined and approved by the study director. The statistical results are incorporated into the method draft, which is then reviewed by the technical committee or subcommittee. A report in which the identities of the other laboratories are identified only by numbers may be issued to collaborative study participants.

Approval of the subcommittee indicates that the method is ready to be recommended to the Uniform Methods Committee for adoption by AOCS.

PART V—ADOPTION AND PUBLICATION OF METHODS

Article XII, Section 1 of the Bylaws of the American Oil Chemists' Society reads as follows: "Official Methods and Recommended Practices. The Society investigates, adopts, and publishes such 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 is 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 draft submitted to AOCS for consideration as an Official Method, Standard Procedure, or Recommended Practice must be assigned to a technical committee, subcommittee, or task group, which reviews the method draft before the collaborative trial is carried out.
2. Following the collaborative trial and statistical analysis, the technical committee chairperson will obtain approval by a ballot of the subcommittee, furnishing the subcommittee with a copy of the method and supporting data. The subcommittee shall have one month to consider and return the ballots to the subcommittee chairperson.
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. Sixty percent of the ballots sent out must be returned for the evaluation to be complete. Two-thirds of the votes received must indicate that the collaborative study results show that the method is ready for review by the UMC.
4. A method receiving no negative ballots in the technical committee vote shall immediately be recommended to the Uniform Methods Committee. If a method receives the necessary two-thirds affirmative ballots but has one or more negative ballots, all members casting negative ballots shall be contacted by the chairperson of the technical committee or subcommittee. The 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 in the technical committee or subcommittee.
5. The Uniform Methods Committee shall vote on approval of the method. The Uniform Methods Committee shall have one month to consider and return the ballots to the Uniform Methods Committee chairperson. Failure to vote by a UMC member shall count as an abstention. The Uniform Methods Committee can reject the method, or approve it by a two-thirds majority vote. If the method is rejected, the Subcommittee may make recommendations to change parts of the method and conduct another collaborative study. All negative UMC ballots must be accompanied by reasons for negative vote.
6. 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.
7. Methods receiving one or more negative ballots will be reviewed by the UMC and subcommittee chairpersons. Any reasons for a negative vote by UMC members will be sent to AOCS Technical Services, who will pass them on to the technical committee chairperson for consideration.
8. 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 UMC to override the negative ballot.

9. 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.
10. Officially adopted methods are published in the *Official Methods and Recommended Practices of the AOCS*.

PART VI—ADOPTION OF METHODS FROM OTHER ORGANIZATIONS

Methods of other organizations may be adopted as AOCS Official Methods if approved by the organization owning the method, 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 the procedure outlined in Part V.

PART VII—REVIEW AND UPDATING OF EXISTING METHODS

Official Methods and Recommended Practices are updated through Additions and Revisions to Methods. In addition, methods are completely reviewed at regular 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. Methods in need of revision are brought to the attention of the AOCS Technical Services department.
2. The chairperson of the relevant technical committee/subcommittee collaborates with the person pointing out the need for revision to evaluate scope of changes to the method required by 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. AOCS methods reviewed prior to the publication of a new edition of *Official Methods and Recommended Practices of the AOCS* shall be reapproved by the Uniform Methods Committee and the editor. The reapproval date is to be noted on the method.

This page is intentionally blank.

All rights reserved. No part of this method may be reproduced or transmitted in any form or by any means without written permission of the publisher.

AOCS Laboratory Safety

INTRODUCTION

The following sections do not contain complete listings of all the elements involved in laboratory safety. These precautionary notes serve as a reminder of possible hazards involved in the use of particular operations or substances, especially those items and materials frequently used in AOCS methods. The user of these methods should refer to standard texts on laboratory safety for a more complete treatment of the subject. Follow safety requirements and rules issued by voluntary organizations and government agencies [Occupational Safety and Health Administration (OSHA), in particular] expert in the field of laboratory safety.

EQUIPMENT

Blenders, grinders, electrical equipment—Motors on high-speed blenders used to mix flammable solvents with other material should be rated for use with the materials in question and in the class and division rating of the lab where the work is being performed. Blend toxic or flammable liquids in an effective fume-removal area. Accidents involving electrical equipment may result in mechanical injury, e.g., fingers are being caught in chopping mill knives or grinders; electrical shock, which may be due to lack of or improper grounding, defective equipment, exposed wiring, or inadequate maintenance; and fire through ignition of flammable vapors by electrical sparks. Ground all electrical equipment. Installation, maintenance, and repair operations should be performed by qualified electricians.

Atomic absorption spectrophotometer—Use effective fume-removal device to remove gaseous effluents from burner. Use specially designed exhausts when nitrous oxide (N_2O) is used as a fuel oxidant. If instrument has drain trap, check to ensure it is filled with water before igniting burner. Explosions of fuel gas accumulated through drain traps have been reported.

Compressed gas cylinders—Identify contents (by means of attached decal, stencil or tag) of compressed gas cylinders by name of gas contained in the cylinder rather than by color codes. Secure cylinders in upright position by means of strap, chain, or nontip base. Use only correct pressure gauges, pressure regulator, and flow regulator for each size of gas cylinder and type of gas, as specified by supplier. Use toxic gases only in effective fume-removal areas. When burning gas or performing a reaction, use back flow prevention device in gas line to prevent flame or reaction from being sucked back into cylinder.

Distillations, extractions, evaporations—For flammable liquids, perform operations behind safety barrier with hot water, steam, or electric mantle heating. Do not use open flames to heat flammable liquids. Use effective fume-removal device to remove flammable vapors as they are produced. Set up apparatus on firm supports and secure all connections. Leave ample headroom in flask and add boiling chips before heating begins. All controls, unless vapor sealed, should be located outside vapor area. For toxic liquids, use effective fume-removal device to remove toxic vapors as they are produced. Avoid contact with skin and inhalation of vapors. Store and dispose of toxic solvents in the manner prescribed by the Environmental Protection Agency (EPA) and OSHA.

Vacuum—Any apparatus to be used under vacuum shall be coated, taped, or otherwise treated to minimize effects of possible implosion, and a safety shield in place during operation. Vacuum pump drive belts must have effective guards.

ACIDS

Use effective acid-resistant fume-removal device whenever heating acids or performing reactions which liberate acid fumes. When diluting acids, always add acid to water, unless otherwise directed in a method. Keep acids off skin, and protect eyes when working with acids. If acids come in contact with skin or eyes, wash immediately with large amounts of water. Do not store oxidizing acids (perchloric, nitric, sulfuric) near organic materials. Mixing organic materials with these acids, particularly perchloric, could result in an explosion.

Acetic acid in the pure state is moderately toxic by ingestion and inhalation. It is a strong irritant to skin and tissue. The TLV in air is 10 ppm.

Hydrochloric acid is a strong acid and will cause severe burns. Protective clothing should be worn when working with this acid. It is toxic by ingestion and inhalation and a strong irritant to eyes and skin. The use of a properly operating fume hood is recommended. When diluting the acid, always add the acid to the water, never the reverse.

Hydrogen bromide gas and hydrobromic acid are toxic by inhalation and strong irritants to eyes and skin. Use a properly operating fume hood when working with these compounds.

Nitric acid is a highly corrosive and toxic oxidizing agent. Use effective acid-resistant fume-removal device whenever heating acids or performing reactions that liberate acid fumes. When diluting acids, always add acid to water unless otherwise directed in a method. Keep acids off skin and protect eyes when working with acids. If acids come in contact with skin or eyes, wash immediately with large amounts of water. Do not store oxidizing acids (perchloric, nitric, sulfuric) near organic materials. Mixing organic materials with these acids, particularly perchloric, could result in an explosion.

Peroxic acid is an oxidizing agent and is dangerous in contact with organic materials. It is a strong irritant. It decomposes at 130°C. Do not use cork or rubber stoppers on storage bottles.

Sulfuric acid is a strong acid and will cause severe burns. Protective clothing should be worn when working with this acid. It is a dehydrating agent and should not be stored in the vicinity of organic materials. Use great caution in mixing with water due to heat evolution that can cause explosive spattering. Always add the acid to water, never the reverse.

ALKALIES

Alkalies can burn skin, eyes, and respiratory tract severely. Wear heavy rubber gloves and face shield to protect against concentrated alkali liquids. Use effective fume-removal device or gas mask to protect respiratory tract against alkali dusts or vapors. When working with extremely caustic materials, like sodium hydroxide and potassium hydroxide, always add pellets to water and not vice versa. These alkalies are extremely exothermic when mixed with water. Take precautions to contain the caustic solution in the event the mixing container breaks from the extreme heat generated.

Potassium hydroxide can severely burn skin, eyes, and respiratory tract. Wear heavy rubber gloves and face shield to protect against concentrated alkali liquid splash. Use effective fume-removal device or gas mask to protect respiratory tract against alkali dusts or vapors. When working with extremely caustic materials, such as potassium hydroxide, always add the pellets to the water and not the reverse.

Sodium hydroxide can severely burn skin, eyes and respiratory tract. Protective clothing should be worn when working with this alkali. Wear heavy rubber gloves and face shield to protect against concentrated alkali liquid splash. Use effective fume-removal device or gas mask to protect respiratory tract against alkali dusts or vapors. When working with extremely caustic materials, such as sodium hydroxide, always add pellets to water and not the reverse.

SOLVENTS

Vapors from some volatile solvents are highly toxic. Several of these solvents are readily absorbed through the skin. Do not let vapors concentrate to a flammable level in the work area, because it is nearly impossible to eliminate all chances of sparks from static electricity, even though electrical equipment is grounded. Use effective fume-removal device to remove solvent vapors as they are liberated.

Acetone is a highly flammable solvent. Forms explosive peroxides with oxidizing agents. Use effective fume-removal device. Do not mix with chloroform.

Acetonitrile is a flammable solvent. There is toxic action by skin absorption and inhalation. A fume hood should be used at all times when using acetonitrile.

Aniline is an organic chemical compound consisting of a benzene ring attached to an amino group. Acute exposure can cause upper respiratory tract irritation and congestion.

Benzene is a highly toxic and highly flammable solvent. Avoid contact with the skin. Do not breathe vapors. Use effective fume-removal device. Decomposes violently in the presence of strong oxidizing agents. Reacts violently with chlorine. Benzene is a cancer-causing agent.

Carbon disulfide is a colorless, highly-flammable poisonous liquid. It is harmful either by inhalation, prolonged or repeated skin contact, or by ingestion. Chronic poisoning may ensue from repeated exposure to vapor. It is a dangerous fire and explosion risk, and can be ignited by friction. Extreme precautions should be taken when using this solvent. A fume hood should be used at all times when handling this solvent.

Carbon tetrachloride is a known carcinogen. It is toxic by ingestion, inhalation, and skin absorption. It is a narcotic. It should not be used to extinguish fires. It decomposes to phosgene gas at high temperature. It reacts violently with alkali metals. A fume hood should be used at all times when handling this solvent.

Chlorobenzene is a colorless flammable liquid. It has a moderate fire risk. Explosive limits are 1.8–9.6% in air. Avoid inhalation and skin contact. The TLV is 75 ppm in

air. Chlorobenzene and trichlorobenzene are toxic by ingestion and inhalation. Use a properly operating fume hood when working with this solvent.

Chloroform is a known carcinogen. It is toxic by inhalation and has anesthetic properties. Avoid contact with the skin. Prolonged inhalation or ingestion can lead to liver and kidney damage and may be fatal. It is nonflammable, but will burn on prolonged exposure to flame or high temperature, forming phosgene gas when heated to decomposition temperatures. Can react explosively with aluminum, lithium, magnesium, sodium, potassium, disilane, N_2O_5 , and sodium hydroxide + methanol. The TLV is 10 ppm in air. A fume hood should be used at all times when using chloroform.

Cyclohexane is a highly flammable liquid. It may be fatal if swallowed or inhaled, and can cause skin irritation. Use effective fume-removal device. Can react vigorously with strong oxidizing agents.

Dichloromethane (methylene chloride) is toxic and a carcinogen that will emit highly toxic fumes and phosgene gas when heated. The TLV is 100 ppm in air. A fume hood should be used at all times when using methylene chloride.

Diethyl ether is an extremely flammable liquid, and a severe fire and explosion hazard when exposed to heat or flame. It is a central nervous system depressant by inhalation and skin absorption. Store protected from the light. It will form explosive peroxides upon exposure to light. Handle empty containers, particularly those from which ether has evaporated, with extreme caution. Explosive limits in air are 1.85–48%. The TLV is 400 ppm in air. Can react explosively when in contact with chlorine, ozone, lithium aluminum hydride, or strong oxidizing agents. A fume hood should be used at all times when using ethyl ether. Avoid static electricity.

Dimethylformamide is a clear flammable liquid and a strong irritant to skin and tissue. It is toxic by skin absorption. The TLV is 10 ppm in air.

Ethanol (ethyl alcohol) is a clear, colorless, highly flammable liquid. Use effective fume-removal device when heating or evaporating.

Ethyl ether is a highly flammable liquid and a severe fire and explosion hazard when exposed to heat or flame. It is a central nervous system depressant by inhalation and skin absorption. It will form explosive peroxides upon exposure to light. Handle empty containers, particularly those from which ether has evaporated, with extreme caution. Explosive limits in air are 1.85–48%. The TLV is 400 ppm in air. A fume hood should be used at all times when using ethyl ether.

Hexane is a highly flammable solvent and a dangerous fire risk. All work should be performed in a fume hood, with no open flames. The TLV for hexane is 50 ppm in air. OSHA recommends that exposure not exceed 350 ng/M^3 for a time-weighted average. Hexane vapor causes lung irritation and produces neurotoxic effects.

Heptane is a highly flammable liquid and a dangerous fire risk. Vapors may cause lung irritation and may produce neurotoxic effects. A fume hood should be used at all times when using this solvent.

Methanol (methyl alcohol) is flammable liquid, and toxic. Avoid contact with eyes. Avoid breathing vapors. Use effective fume-removal device. Can react vigorously with sodium hydroxide + chloroform, potassium hydroxide + chloroform, and perchloric acid.

Methyl isobutyl ketone (MIBK) is a clear, colorless, and highly flammable liquid and a dangerous fire risk. Explosive limits in air are 1.4–7.5%. Avoid inhalation and ingestion. It is absorbed by the skin. The TLV is 50 ppm in air.

Petroleum ether is the petroleum fraction consisting of aliphatic hydrocarbons in the boiling range 35–60° C. The term *ether* is only figurative, signifying extreme lightness and volatility. It is extremely flammable. The explosive limits in air are 1–6%. Use effective fume-removal device. Avoid static electricity.

Pyridine is a clear liquid with a distinct odor, is highly flammable and a dangerous fire risk. The explosive limits in air are 1.8–12.4%. It is toxic by ingestion and inhalation. The TLV is 5 ppm in air. The danger from crude pyridine is greater than from pure pyridine, the associated homologs and impurities being even more toxic than pyridine itself.

Tetrachloroethylene (perchloroethylene) is a colorless, volatile, nonflammable liquid chlorinated hydrocarbon that will emit toxic fumes of phosgene when exposed to sunlight or flames. It is an irritant to eyes and skin. The TLV is 50 ppm in air.

Tetrahydrofuran is a highly flammable liquid and a dangerous fire risk. The flammable limits in air are 2–11%. It is toxic by ingestion and inhalation. The TLV in air is 200 ppm. It tends to form peroxides upon storage in air.

Toluene is a highly flammable liquid and a dangerous fire risk. Explosive limits in air are 1.27–7%. It is toxic by ingestion, inhalation, and skin absorption. The TLV is 100 ppm in air. A fume hood should be used at all times when using toluene.

Trichloroethane is a synthetic, light-sensitive, volatile, colorless, liquid miscible with many nonpolar organic solvents. It is an irritant to eyes and skin. The TLV is 350 ppm in air.

Xylene is flammable and a dangerous fire risk. The TLV is 100 ppm in air.

CHEMICALS

Chlorine is a poisonous gas. The TLV is 1 ppm in air. It is a strong oxidizing agent and should not be allowed to come in contact with organic materials, hydrogen, powdered metals, and reducing agents. A fume hood should be used at all times when using chlorine.

Gossypol is toxic by ingestion. Avoid contact with particulate matter when working with standards. It is inactivated by heat.

Hydrazine sulfate can cause eye, skin, and mucous membrane irritation and liver and kidney damage. This compound is a known carcinogen in laboratory animals, causing lung and liver tumors in rats. It is a suspected human carcinogen. Precautions should be taken in handling this compound—use gloves, eye protection, and respiratory protection. Avoid the inhalation of dust and powder. Dispose of waste material and waste solutions in a proper and safe manner.

Lead acetate is toxic by ingestion, inhalation, and skin absorption.

Mercury vapors and compounds are extremely toxic and cumulative. Hazardous in contact with ammonia, halogens, and alkali. Regard spills on hot surfaces as extremely hazardous and clean up promptly. Powdered sulfur sprinkled over spilled mercury can assist in cleaning up spills. High degree of personal cleanliness is necessary for persons who use mercury. Handle only in locations that can be readily and completely cleaned up. When mercury evaporation is required, use effective fume-removal device. To avoid environmental contamination, dilute liquid remaining in Kjeldahl digestion flasks to about 300 mL with water, cool to room temperature, and add 50 mL 30% hydrogen peroxide. (If Raney powder method is used, 6 mL of hydrogen peroxide is sufficient.) Warm gently to initiate reaction, let reaction go to completion in warm flask, and separate precipitated mercuric sulfide. Reserve precipitate in closed, labeled container for recovery of mercury or disposal by EPA requirements.

Potassium dichromate is toxic by ingestion and inhalation. There is sufficient evidence in humans for the carcinogenicity of chromium [+6], in particular lung cancer. It is a strong oxidizing agent and a dangerous fire risk in contact with organic chemicals.

Sylon BFT is a powerful silylating reagent, composed of mixing 1 part trimethyl chlorosilane with 99 parts of bistrimethylsilyl-trifluoroacetamide, and should be used only in a properly operating fume hood. This reagent is highly flammable.

tert-Butyl methyl ether is extremely flammable and toxic. Avoid inhalation, ingestion, and eye or skin contact. The TLV is 50 ppm in air. OSHA recommends that exposure not exceed 100 mg/M^3 for a time-weighted average. Respiratory irritation, dizziness, and disorientation have been reported. A fume hood should be used at all times when using *tert*-Butyl methyl ether.

Wijs solution, iodine monochloride, causes severe burns, and the vapors can cause lung and eye damage. Use of a fume hood is recommended. Wijs solution without carbon tetrachloride is available commercially.

ADDITIONAL MATERIALS

Castor seeds are poisonous due to the presence of ricin, a highly toxic albumin, and ricinine, a highly toxic alkaloid. Neither pressing nor extraction removes them; hence both hazards remain in the pomace. They also contain an allergenic protein polysaccharide (CB-1A) that is among the most powerful known allergens. It is strongly recommended that workers wear rubber gloves when preparing analytical samples, and that they avoid inhaling any of the dust arising from the castor beans by working near an air exhaust or in a well ventilated laboratory hood.

Fumonisins are hepatotoxic and carcinogenic to rats; effects on humans are not fully known. Wear protective gloves to reduce skin contact with corn extracts. Any laboratory spillages should be washed with a 5% aqueous solution of commercial sodium hypochlorite followed by H_2O . (Dispose of waste solvents according to applicable environmental rules and regulations.)

Mycotoxins should be handled with extreme care because they are highly toxic substances. Perform manipulations under a properly operating fume hood. Take particular precautions, such as the use of a glove box, when toxins are in dry form, because of their electrostatic nature and resulting tendency to disperse in working areas. Swab accidental spills of toxin with 5% NaOCl bleach. Rinse all glassware exposed to toxins with 1% NaOCl bleach solution and then wash thoroughly with warm water.

REFERENCES

Official Methods of Analysis, Association of Official Analytical Chemists, 14th edn., 1984, pp. 1010–1015.

Standard Methods for the Analysis of Oils, Fats and Derivatives, 7th edn., Blackwell Scientific Publications, 1987.

Hawley's Condensed Chemical Dictionary, 11th edn., revised by N. I. Sax and R. J. Lewis, Jr., 1987.