Format for AOAC *Official Methods of Analysis*

The language of the method should be concise and completely free from ambiguity. Conciseness is desirable, both to ensure clarity and to save space. Whenever there is a conflict between clarity and style, clarity is more important.

Present Tense and Imperative Mode

- Check sentences that do not begin with a verb and change them, if feasible, to the imperative mode (e.g. Pipet 10 mL..., Stir..., etc.). Exceptions are: use of adverb modifier ("Accurately weigh..."), prepositional clause ("For refined sugars, use..."), permissive statements ("Ferric hydroxide may be used..."), and statements in the "Principle" section.

Abbreviations

- Most abbreviations are the same as those used by Chemical Abstracts. Do not use abbreviations in titles and headings. See the *Definitions of Terms and Explanatory Notes*.

Repetition and Redundancy

- Eliminate repetition and redundancy as far as possible; use only for emphasis. Do not use "distilled" with water, "concentrated" with common acids, "95%" with alcohol, or "ACS" with reagents covered by ACS specifications. These are understood by definition.

Terminology, Formulae and Chemical Names

- For names of chemical compounds, use the spelling, hyphenation, and word division given in Chemical Abstracts. Use a national pharmacopeia for names for drugs. Use ISO nomenclature for pesticides and Codex nomenclature for names of food additives and color additives.

Consistency

- Watch for internal contradictions in the text: volumes that do not add up or that exceed the capacity of the container; too abrupt a transition from one operation to another (a line may be omitted); and impractical or impossible numbers (e.g., 100 g NaCl will not dissolve in 100 mL water).

Cross-references

- All new AOAC methods should be written as complete and self-contained as practical. Do not refer to other AOAC methods. If part of a procedure in an *Official Method*^{SM} is taken from material previously published elsewhere, incorporate those steps in the method rather than referring the analyst to another publication.

Definitions

- The section "Definition of Terms and Explanatory Notes," *Official Methods of Analysis of AOAC INTERNATIONAL*, is the basic guide to conventions and consistency.

Illustrations and Tables

- If symbols are used on the figure, include an explanation in the caption or text. Provide descriptive titles for tables. Explain any obscure headings in a footnote.

Bibliographic References

- Check all references for accuracy. Use standard Chemical Abstracts abbreviations for *Journal* titles. In general avoid references in method. Cite background references in the "Introduction" or "Discussion" section of the collaborative study manuscript -- not in the method. If part of a procedure in an *Official Method*^{SM} is taken from material previously published elsewhere, incorporate those steps in the method rather than referring the analyst to another publication.

Safety

- All methods must be reviewed for safety and potential hazards. Methods should automatically incorporate cross-references to the safety statement(s), or present questioned conditions to the attention of the Committee on Safety for resolution.
- Decisions regarding inclusion of safety statements should be practical, recognizing that overuse will be self-defeating.
- Methods that create toxic, obnoxious or environmentally hazardous fumes and wastes should contain practical directions for disposal.

Checking Edited Copy and Proofreading

- The author must review a copy of the original version and edited copy to ensure that there has been no change in meaning, to correct typographical errors, and to answer any questions posed by the editor. The author must review the typeset method for accuracy.

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Online Technical Resources

Method Development, Optimization & Validation

- OMA - Appendix F - Guidelines for Standard Method Performance Requirements
- Homogeneity
- Guide for Writing Methods in AOAC Format
- Statistics Protocol Review Form
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix G: Procedures and Guidelines for the Use of AOAC Voluntary Consensus Standards to Evaluate Characteristics of a Method of Analysis
- OMA - Appendix I: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Biological Threat Agent
- Methods and/or Procedures
- OMA - Appendix J: AOAC INTERNATIONAL Methods Committee Guidelines for Validation of Microbiological Methods for Food and Environmental Surfaces
- OMA - Appendix K: Guidelines for Dietary Supplements and Botanicals
- OMA - Appendix L: AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation
- OMA - Appendix M - Validation Procedures for Quantitative Food Allergen ELISA Methods: Community Guidance and Best Practices
- Safety Checklist

Method Review

- Examples of Statistical Analysis
- Statistics Manuscript Review Form
- OMA - Appendix A: Standard Solutions and Reference Materials
- OMA - Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis
- OMA - Appendix H: Probability of Detection (POD) as a Statistical Model for the Validation of Qualitative Methods

Miscellaneous

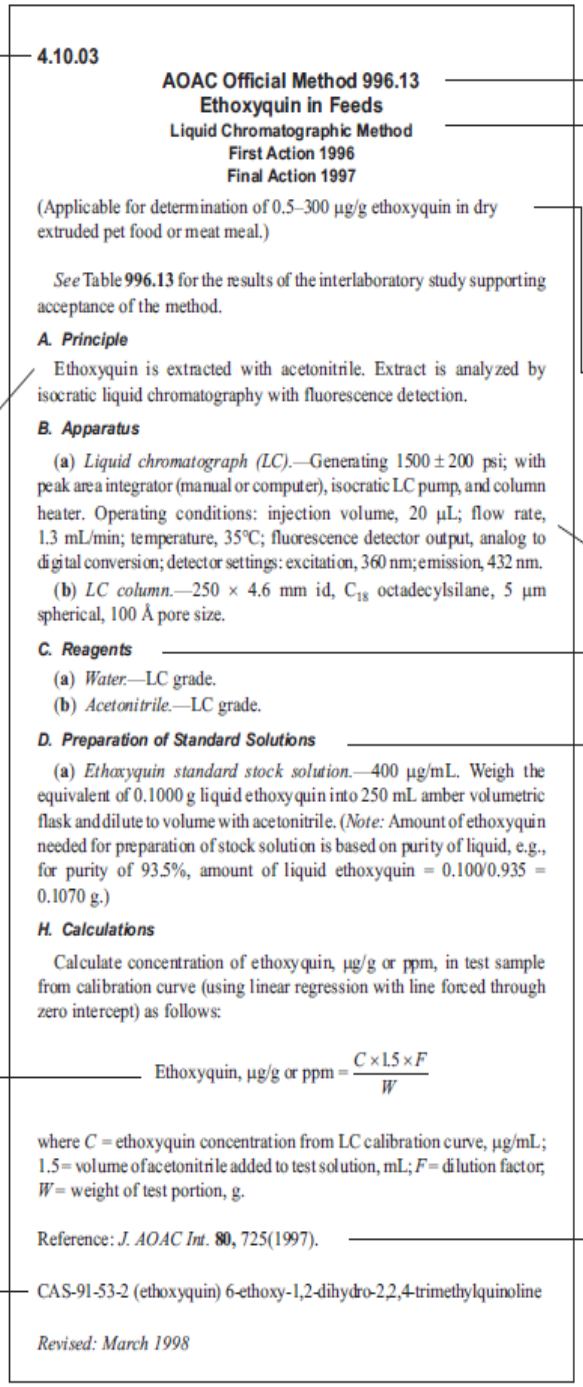
- Definition of Terms and Explanatory Notes
- OMA - Appendix B: Laboratory Safety
- OMA - Appendix E: Laboratory Quality Assurance
- OMA - Appendix C: Reference Tables

All resources are accessible at
http://www.aoac.org/vmeth/guidelines.htm

For questions, please contact:
P 301-924-7077 x157 E dmckenzie@aoac.org

Guide to Method Format

(Method shown is incomplete to allow space for description.)



4.10.03

AOAC Official Method 996.13
Ethoxyquin in Feeds
Liquid Chromatographic Method
First Action 1996
Final Action 1997

(Applicable for determination of 0.5–300 µg/g ethoxyquin in dry extruded pet food or meat meal.)

See Table **996.13** for the results of the interlaboratory study supporting acceptance of the method.

A. Principle

Ethoxyquin is extracted with acetonitrile. Extract is analyzed by isocratic liquid chromatography with fluorescence detection.

B. Apparatus

(**a**) *Liquid chromatograph (LC).*—Generating 1500 ± 200 psi; with peak area integrator (manual or computer), isocratic LC pump, and column heater. Operating conditions: injection volume, 20 µL; flow rate, 1.3 mL/min; temperature, 35°C; fluorescence detector output, analog to digital conversion; detector settings: excitation, 360 nm; emission, 432 nm.

(**b**) *LC column.*—250 × 4.6 mm id, C_{18} octadecylsilane, 5 µm spherical, 100 Å pore size.

C. Reagents

(**a**) *Water.*—LC grade.

(**b**) *Acetonitrile.*—LC grade.

D. Preparation of Standard Solutions

(**a**) *Ethoxyquin standard stock solution.*—400 µg/mL. Weigh the equivalent of 0.1000 g liquid ethoxyquin into 250 mL amber volumetric flask and dilute to volume with acetonitrile. (*Note:* Amount of ethoxyquin needed for preparation of stock solution is based on purity of liquid, e.g., for purity of 93.5%, amount of liquid ethoxyquin = 0.100/0.935 = 0.1070 g.)

H. Calculations

Calculate concentration of ethoxyquin, µg/g or ppm, in test sample from calibration curve (using linear regression with line forced through zero intercept) as follows:

$$\text{Ethoxyquin, µg/g or ppm} = \frac{C \times 1.5 \times F}{W}$$

where C = ethoxyquin concentration from LC calibration curve, µg/mL; 1.5 = volume of acetonitrile added to test solution, mL; F = dilution factor; W = weight of test portion, g.

Reference: *J. AOAC Int.* **80,** 725(1997).

CAS-91-53-2 (ethoxyquin) 6-ethoxy-1,2-dihydro-2,2,4-trimethylquinoline

Revised: March 1998

Locator number identifies method by chapter, subchapter, and sequence within the subchapter for easy cross referencing and access. 4 = chapter 4; .10 = subchapter 10; .03 = the third method found in Chapter 4, subchapter 10. The locator number is not the permanent number and is included only for convenient accessibility.

Chemical names of pesticides and drugs are given at end of pertinent chapter.

Calculation symbols are identified and show correct units.

Chemical Abstracts Service Registry Number. A unique identifier that may be used to search a number of data-retrieval systems.

Method number identifies method by year of adoption or first appearance in *Official Methods of Analysis of AOAC INTERNATIONAL*. 996 = First Action 1996; .13 = sequence of adoption in 1996.

Title may include analyte and matrix, type of method, and official status.

Applicability statement addresses utility and limitations on use of method or other information.

Specifications for necessary laboratory apparatus and reagent preparations. *See also Definition of Terms and Explanatory Notes.*

Method may be divided into several descriptive sections.

References direct the user to the published collaborative study and any subsequent revisions in the method. Other informative references may be included.

The AOAC style used for preparing methods for publication in the *Official Methods of Analysis of AOAC INTERNATIONAL* includes the following essentials:

- ✓ Standardized format that follows the order of laboratory operations.
- ✓ Use of the imperative mode.
- ✓ Cross-references to identical reagents, apparatus, and operations.
- ✓ Use of standardized definitions, terminology, and style.
- ✓ Use of accepted abbreviations and simplifications.
- ✓ Use of SI units
- ✓ Methods should be written as complete and self-contained as practical.
- ✓ Normality should be referred in terms of Molarity.
- ✓ ppm should be changed to mg/kg or mg/L
- ✓ ppb should be changed to ng/g or ng/mL
- ✓ ppt should be changed to pg/g or pg/mL

FORMAT OF AOAC® *OFFICIAL METHODS of ANALYSIS OF AOAC INTERNATIONAL*

Title:
- ❖ Includes analyte being determined, type of matrix (matrices), and analytical technique used for analysis.

Applicability:
- ❖ Includes list of matrix(es) along with specific matrix types and range or limits of determination or detection.

Precautions:
- ❖ Makes an analyst aware of hazardous materials used in analysis.

Data Collection:
- ❖ Table(s) that presents performance parameters including matrices tested in a collaborative study, levels of analyte(s), % recovery, RSD_r, RSD_R, s_r, s_R, HORRAT, number of observations, etc

Principle:
- ❖ Explains scientific premise on which the method is operates specifically the mechanism of the analysis.

Apparatus:
- ❖ Lists the equipment that requires assembly or that has specifications critical to the method performance. Describe equipment in terms of performance characteristics.

Reagents:
- ❖ List the reagents with amounts and appropriate units needed to conduct the analysis and describe the reagents in terms of performance characteristics.

Sample and Test Portion Preparation:
- ❖ Describe the preparation of samples and the test portion.

Determination:
- ❖ Describes the actual analysis.

Calculations:
- ❖ Section that explains how to calculate final results; presented in a form of equation or description.

Other sections as needed

REFERENCING *AOAC® OFFICIAL METHODS*℠

When referencing *AOAC® Official Methods*℠, only the method number should be used as seen in the following example:

(1) *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed., AOAC INTERNATIONAL, Gaithersburg, MD, USA, Official Method **2008.01**

Revised October 2013