GUIDANCE DOCUMENT O: GUIDELINES FOR AWPA ANALYTICAL METHOD STANDARDS

Jurisdiction: Technical Committee P-5

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This Guidance Document is not an AWPA Standard. These are nonmandatory guidelines presented to aid the user in understanding the basic testing requirements for wood protection systems and to assist the AWPA Technical Committees in the development of AWPA Standards. The testing of products in accordance with this Guidance Document does not constitute conformance to any AWPA Standard. No product can be considered to conform to an AWPA Standard until it has been subjected to complete technical review and voting by AWPA's Technical Committees, and procedural review and final action by the AWPA Executive Committee pursuant to the AWPA Technical Committee Regulations.

1.0 Table of Contents

2.0 Introduction

This document provides general provisions and style guidelines to those submitting analytical methods for standardization by AWPA through Sub-committee P-5. These guidelines would also apply when proposing a modification to an existing AWPA analytical method standard or when reaffirming analytical standards.

The AWPA Analytical Method Standards cover a wide range of procedures from simple penetration tests to complex instrumental analyses. Some elements may not be applicable to all methods and in certain instances additional information may be required.

2.1 General Submission Information

Proposed standards based on established chemistries and common instrumental methods will not require extensive documentation. Lesser known or newly established methods may require a proponent to provide the chemistry, stoichiometry, and principle of the analytical procedure. Literature references will also be useful. Examples of chromatograms and spectra from calibration standards and samples should also be supplied, if applicable.

3.0 General Format and Style Guidelines

Formatting recommendations for analytical methods are modifications from the 2018 ASTM Blue Book (ASTM, 2018a). The standard should use numbered sections and subsections generally including the sections listed below recognizing that some of the sections may not be applicable to all methods. Additional sections may be added as needed. Avoid excessive subsection levels and orphaned subsection numbers. Use the International System of units (SI) as the standard units of measurement.

3.1 Title

The title should be clear and concise stating the analytes, matrix, and methodology.

3.2 Scope

Describe the purpose of the method, including what types of analytes or attributes are being measured, and the technology employed. Mention all applicable matrices. Also include limitations and interferences.

3.3 Summary of the Method

For more complex methods, provide a brief summary of the major steps in the method. These details will prepare the analyst on what will be necessary to apply the method.

3.4 Safety

Discuss health and safety issues specific to the method beyond the scope of routine laboratory practices. This includes information regarding specific toxicity of analytes and reagents and personal protective equipment required for performing the method.

3.5 Equipment and Supplies

List method-specific equipment and supplies, without mentioning a specific vendor whenever possible. Include the phrase "or equivalent" as appropriate when vendor-specific instrumentation or supplies are listed. If specific equipment is necessary based on method studies, clearly state what equipment and supplies were tested. If necessary, include sufficient information for locating and purchasing the correct equipment.

Common laboratory apparatus, e.g., beakers, flasks, stirring bars, graduated cylinders, etc., should not be mentioned, unless there is a specific need for one with an unusual or non-standard characteristic, e.g., a specific chemical-resistant coating for a stirring bar, tinted glass flasks, Class A graduated cylinders, etc. All other apparatus should be mentioned in this section, e.g., pH meter, hot plate stirrer, analytical balance, etc. Generally, mention only the more expensive and unique equipment (>$500).

3.6 Reagents and Standards

Provide sufficient detail on necessary reagent grades, concentrations, and the preparation of all reagents and standards to allow the work to be duplicated. A general statement can be made regarding the purity of reagents (e.g. all chemicals are reagent grade). List reagents before standards, particularly if the standards are to be prepared from the reagents. Include all standards and reagents mentioned by the method in any section. Be consistent with the standard or reagent name throughout the method (and the AWPA Standards as possible).

3.7 Sampling

Indicate or reference the procedures used to obtain samples for the method and sample size requirements (volumes of solutions, mass of timber). List special storage containers, temperature, hold times, etc. as necessary.

3.8 Sample Processing

Detail the processing procedures including milling and drying and indicate temperature limitations, particle size requirements, etc.

3.9 Calibration and Standardization

Describe initial calibration procedures as appropriate with details on how to do them. Indicate acceptance limits for the calibration. Provide guidance on what to do if the relevant performance criteria are not met. As appropriate, describe calibration verification and verification frequency.

3.10 Procedure

Provide detailed step-by-step instructions for using the method. Include a description of analytical steps for sample preparation and instrumental or physical analysis. Include those steps that are essential to the procedure and avoid unnecessary restrictive instructions. Include verification and QC steps such as blanks, matrix spikes, and replication as necessary.

3.11 Calculations

Describe quantitative and qualitative information for deriving final sample results from the data. Include the formulas for all calculations including prepared reagents and dilution factors, reagent purity, etc. Clearly indicate the expected results and units in which they are to be reported.

3.12 Precision & Bias Statements

All analytical methods submitted to AWPA for adoption into the Book of Standards must include a precision statement. This requirement applies to both new submissions as well as reaffirmation proposals for existing methods. Statements of Bias are required if comparison against a referee method or against spike recoveries shows a quantifiable or significant bias of the data.

3.12.1 Recommended Elements for Precision Statements

Analytical standards should strive to meet or exceed the minimum number of elements described below. These recommendations for precision testing are modified from the ASTM E691-18 method (ASTM 2018b).

*9.1.2. Minimum of 6 participating labs**

10.2.2. Minimum of 3 materials = 3 levels or concentrations*

11.1. Minimum of 3 test results per material.*

**terms are defined in ASTM E691-18§3 and examples of their use in Sections ASTM E691-18 §5.2.1 – 5.2.4.*

3.12.2 Recommended Precision Statement Format

3.12.2.1 Recommended Precision Statement Template

X. Precision Statement: The following statements and table(s) should be used to judge the acceptability of an analysis using the method and the conditions described below. The precision data were developed following guidelines in ASTM E691-18.

X.1 Repeatability: Duplicate test results on the same test material by the same operator using the same equipment should not be suspect at the 95% confidence level if they do not differ from one another by equal to or less than the confidence limits shown in the following table(s).

X.2 Reproducibility: Duplicate test results on the same test material by different operators in different laboratories should not be suspect at the 95% confidence level if they do not differ from one another by equal to or less than the confidence limits shown in the following table(s).

3.12.2.2 Recommended Precision Statement Table Format

The recommended table format includes columns for analyte concentration, confidence limits for repeatability, and confidence limits for reproducibility. The title of the Precision Statement Table should contain the compound name, starting material (wood or treating solution), and other relevant details if parameters vary (e.g. extraction by reflux, extraction by ultrasonic bath)

Title

	Confidence Limits	
Analyte Concentration Range	Repeatability (r)	Reproducibility (R)
Level 1		
Level 2		
Level 3		
Level 4		
Level 5		

3.12.2.3 Recommended Element Statement (Appears Below Each Table)

The above precision statement is based on round robin data from x laboratories, y test results, and z materials covering the retention/concentration (choose one) range of (insert low to high range values and compound name).

Additional statement(s) as needed to describe nature of test result if observation ≠ determination ≠ test result.

3.12.3 Temporary Precision Statement

If the proponent or task group of a proposal for a new analytical standard determines that a delay or modification to the study is required, a temporary statement shall be included which contains repeatability based on the results from a single operator. A temporary statement is permitted until reaffirmation of the standard. Use a statement such as the following:

Precision[1] – The repeatability standard deviation from a single operator has been determined to be (insert table).

[1] The reproducibility of this test method is not provided at this time because (insert the reason or reasons). The reproducibility of this test method is being determined and is expected to be available on or before (insert year).

3.12.4 Modified Precision Statement

If it is not possible at the time of reaffirmation to provide a statement on precision as recommended in 3.12.1, a modified statement may be used such as the following:

3.12.4.1 Modified Precision Statement Template

X. Precision Statement: The following statements and table(s) should be used to judge the acceptability of an analysis using the method and the conditions described below. The precision data were developed following guidelines in ASTM E691-18, but minimum requirements were not met for analyte X.

X.1 Repeatability: Duplicate test results on the same test material by the same operator using the same equipment should not be suspect at the 95% confidence level if they do not differ from one another by equal to or less than the confidence limits shown in the following table(s).

X.2 Reproducibility: Duplicate test results on the same test material by different operators in different laboratories should not be suspect at the 95% confidence level if they do not differ from one another by equal to or less than the confidence limits shown in the following table(s).

3.12.4.2 Modified Precision Statement Table Format

The modified table format includes columns for analyte concentration, confidence limits for repeatability, and confidence limits for reproducibility. The title of the Precision Statement Table should contain the compound name, starting material (wood or treating solution), and other relevant details if parameters vary (e.g. extraction by reflux, extraction by ultrasonic bath)

Title

	Confidence Limits	
Analyte Concentration Range	Repeatability (r)	Reproducibility (R)
Level 1		
Level 2		
Level 3		
Level 4		
Level 5		

3.12.4.3 Modified Element Statement (Appears Below Each Table)

The above precision statement is based on round robin data from x laboratories, y test results, and z materials and thus does not meet the minimum requirements set forth in ASTM E691-18. Materials were tested covering the retention/concentration (choose one) range of (insert low to high range values and compound name). Additional statement(s) as needed to describe nature of test result if observation ≠ determination ≠ test result.

3.12.4.4 Acceptable Reasons for a Modified Precision Statement May Include

- Modification to an existing standard was made that was significant enough to require new precision testing, but the method is no longer widely used.
- The method is a non-referee secondary method that is not widely used.
- There were an insufficient number of laboratories willing to participate that have the required equipment or experience with the method.

3.12.4.5 Unacceptable Reasons for a Modified Or Omitted Statement Includes

- An interlaboratory study has revealed that the precision is poor.
- The standard was written before precision statements were required.
- No attempt was made to meet the requirements.

3.12.5 Non-Quantitative Precision Statement

If the standard is qualitative (non-numerical) or is a preparation procedure where the final precision is determined by another method, use a statement such as the following:

> Precision – This standard does not require a precision statement, as this is a qualitative method only.
>
> Precision – This standard is preparatory/instructional (choose one) in nature. Precision of the analytical result is determined by the final analytical test methodology employed.

4.0 References

ASTM International. (2018a). Form and style for ASTM standards. West Conshohocken, PA: American Society for Testing and Materials.

ASTM International. (2018b). ASTM E691-18. Standard practice for conducting an interlaboratory study to determine the precision of a test method. West Conshohocken, PA: American Society for Testing and Materials.