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An American National Standard

Standard Practice for Selecting Antimicrobial Pesticides for Use in Water-Miscible Metalworking Fluids[1]

This standard is issued under the fixed designation E2169; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice provides recommendations for selecting antimicrobial pesticides (microbicides) for use in water-miscible metalworking fluids (MWF). It presents information regarding regulatory requirements, as well as technical factors including target microbes, efficacy, and chemical compatibility.

1.2 This guide is not an encyclopedic compilation of all the concepts and terminology ~~uses~~used by chemists, microbiologists, toxicologists, formulators, plant engineers, and regulatory affairs specialists involved in antimicrobial pesticide selection and application. Instead, it provides a general understanding of the selection process and its supporting considerations.

1.3 The values in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate ~~safety~~ safety, health, and ~~health~~environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

D1067 Test Methods for Acidity or Alkalinity of Water
D1293 Test Methods for pH of Water
D3519 Test Method for Foam in Aqueous Media (Blender Test) (Withdrawn 2013)[3]
D3946 Test Method for Evaluating the Bacteria Resistance of Water-Dilutable Metalworking Fluids (Withdrawn 2004)[3]
D4478 Test Methods for Oxygen Uptake (Withdrawn 1994)[3]
D5465 Practices for Determining Microbial Colony Counts from Waters Analyzed by Plating Methods
E686 Method for Evaluation of Antimicrobial Agents in Aqueous Metal Working Fluids (Withdrawn 2004)[3]
E1302 Guide for Acute Animal Toxicity Testing of Water-Miscible Metalworking Fluids
E1326 Guide for Evaluating Non-culture Microbiological Tests
E1497 Practice for Selection and Safe Use of Water-Miscible and Straight Oil Metal Removal Fluids

[1] This practice is under the jurisdiction of ASTM Committee E34 on Occupational Health and Safety and is the direct responsibility of Subcommittee E34.50 on Health and Safety Standards for Metal Working Fluids.

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] The last approved version of this historical standard is referenced on www.astm.org.

E2144 Practice for Personal Sampling and Analysis of Endotoxin in Metalworking Fluid Aerosols in Workplace Atmospheres

2.2 *Government Standards:*

29 CFR 1910 Occupational Safety and Health Standards[4]

40 CFR 152 Pesticide Registration and Classification Procedures[4]

40 CFR 158 Pesticide Programs Data Requirements for Registration[4]

49 CFR 100-180 Research and Special Programs Administration, Department of Transportation[4]

PR Notice 2000-1 Applicability of the Treated Articles Exemption to Antimicrobial Pesticides

Directive 98/8/EC of the European Parliament and of the Council of 16 February 1998 concerning the placing of biocidal products on the market

3. Terminology

~~3.1 *active ingredient (a.i.), n*—the chemical component or components of an antimicrobial pesticide that provides its microbicidal performance.~~

3.1 *Definitions:*

3.1.1 *active ingredient (a.i.), n*—the chemical component or components of an antimicrobial pesticide that provides its microbicidal performance.

3.1.2 *activity spectrum, n*—variety or range of microbes against which an antimicrobial pesticide is effective.

3.1.3 *antimicrobial pesticide, n*—chemical additive, registered under 40 CFR 152, for use to inhibit growth, proliferation, or both of microorganisms.

3.1.3.1 *Discussion—*

Antimicrobial pesticides are registered for one or more end-use applications, or sites, for use within an approved dose range.

3.1.4 *bactericide, n*—antimicrobial pesticide specifically or primarily effective against bacteria.

3.1.5 *bioburden, n*—the level of microbial contamination (biomass) in a system.

3.1.5.1 *Discussion—*

Typically bioburden is defined in terms of either biomass or numbers of cells per unit volume or mass or surface area material tested (g biomass/mL sample; g biomass/g sample; cell/mL sample; colony forming units (CFU)/mL; and so forth).

3.1.6 *biocide, n*—any chemical intended for use to kill or inhibit organisms.

3.1.6.1 *Discussion—*

Biocide is a term commonly used synonymously with the preferred *antimicrobial pesticide* or *microbicide*.

3.1.7 *biodeterioration, n*—the loss of commercial value, performance characteristics, or both of a product (metalworking fluid) or material (coolant system or finished parts) through biological processes.

3.1.8 *biofilm, n*—a film or layer composed of microorganisms, biopolymers, water, and entrained organic and inorganic debris that forms as a result of microbial growth, proliferation, and excretion of polymeric substances at phase interfaces (liquid-liquid, liquid-solid, liquid-gas, and so forth). (Synonym: *skinnogen layer*.)

3.1.9 *bioresistant, adj*—able to withstand biological attack.

3.1.9.1 *Discussion—*

Bioresistant, or recalcitrant, chemicals are not readily metabolized by microorganisms.

3.1.10 *biostatic, adj*—able to prevent existing microbial contaminants from growing or proliferating, but unable to kill them.

3.1.10.1 *Discussion—*

Biostatic additives may be registered antimicrobial pesticides or unregistered chemicals with other performance properties. The difference between biocidal and biostatic performance may be attributed to dose, chemistry, or both.

[4] Code of Federal regulations available form United States Government Printing Office, Washington, DC.

3.1.11 *contamination control, n*—maintenance of bioburden at an operationally defined level, at or below which the bioburden does not affect the fluid or system adversely.

3.1.12 *demand, n*—the sum of all factors that contribute to decreasing the effective concentration of antimicrobial pesticide.

3.1.12.1 *Discussion—*

Processes contributing to demand include, but are not limited to: reaction with microbes, reactions with other chemicals in the fluid, adsorption onto surfaces, absorption into materials, and temperature.

3.1.13 *dose, n*—concentration of antimicrobial pesticide added to treated solution.

3.1.13.1 *Discussion—*

Dose is generally expressed as either ppm active ingredient (a.i.) or ppm as supplied (a.s.).

3.1.14 *fungicide, n*—antimicrobial pesticide specifically or primarily effective against fungi.

3.1.15 *half-life ($T_{½}$), n*—time required for concentration of a microbicide to diminish to one half its initial concentration.

3.1.16 *lethal dose, n*—concentration at which treatment kills at least one of test subjects.

3.1.16.1 *Discussion—*

The LD_{50} is the term used in toxicology defining the dose that kills 50 % of the test population.

3.1.17 *microbicide, n*—synonymous with antimicrobial pesticide.

3.1.18 *minimum inhibitory concentration (MIC), n*—lowest treatment dose that will prevent test population from growing, proliferating, or otherwise contributing to biodeterioration.

~~3.2 *activity spectrum, n*—variety or range of microbes against which an antimicrobial pesticide is effective.~~

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The LD_{50} is the term used in toxicology defining the dose that kills fifty percent of the test population.

3.17 *microbicide, n*—synonymous with antimicrobial pesticide.

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4. Summary of Practice

4.1 Microorganisms can grow in all water-miscible metalworking fluids including water-miscible metal removal fluids, a subset of the broader class of metalworking fluids. Consequences of uncontrolled microbial contamination in metalworking fluids may include biodeterioration, rancidity, and aerosolization of potentially pathogenic microbes and toxic or allergenic microbial cell constituents. Consequently, microbial contamination control is desirable from both operational and industrial hygiene perspectives.

4.2 Antimicrobial pesticides are used to prevent biodeterioration and may also reduce the risk of disease associated with the use of water-miscible metalworking fluids. They may be used in-drum, on-site, or both. Antimicrobial pesticides work either by killing microbes, inhibiting specific undesirable microbial activities, or both in the treated fluid. Antimicrobial pesticides used in metalworking fluids include representatives from a number of chemical groups. Consequently, antimicrobial pesticides vary widely in their mode of action, compatibility with other fluid components, and other performance properties.

4.3 The process of selecting an antimicrobial pesticide for use in metalworking fluids shall include, minimally, confirmation that the product is (*1*) approved for the intended application; (*2*) compatible with other fluid and system constituents; and (*3*) effective. Other considerations including, but not limited to intended application, target microbes, desired speed of action, performance persistence, handling precautions, toxicological properties, water and oil miscibility, and waste treatability may affect microbicide selection.

4.4 Microbicide selection begins with a fundamental understanding of the coolant formulation chemistry, biodeterioration control strategy, and specific customer needs. General background information[5] regarding MWF system management is available in

[5] Organization Resources ~~Counselors.~~Counselors, *Management of the Metal Removal Fluid ~~Environment.~~Environment,* ~~Web site:~~ http://www.aware-services.com/orc/ .2000.

Practice E1497 and elsewhere. Armed with this information, candidate microbicides can be selected for further evaluation. Products that meet all of the selection criteria are ultimately tested in field application. Since antimicrobial pesticide efficacy can diminish over time, the selection process may be viewed as cyclic. Moreover, since microbicides can be toxic, they require rigorous and competent product stewardship throughout their use cycle.

5. Significance and Use

5.1 This practice summarizes the steps in the antimicrobial pesticide selection process, reviewing technical and regulatory considerations inherent in the process. It complements and amplifies information provided in Practice E1497.

5.1.1 Steps in the antimicrobial selection process include: needs identification, use strategy selection, efficacy testing, chemical compatibility testing, regulatory consideration review, handling, and disposal issue review.

5.2 This practice provides stakeholders in the microbicide selection process an overview of its complexities, including the process of obtaining pesticide registration from cognizant governing bodies.

5.3 Personnel responsible for antimicrobial pesticide selection will be able to use this practice as a roadmap through the process.

5.4 Personnel responsible for industrial hygiene, product or plant management will gain insight to the tradeoffs attendant with antimicrobial use and selection.

6. Needs Information

6.1 The first step in the microbicide selection process is the recognition of a need. Recognition may come as a consequence of new metalworking fluid formulation development or evolving requirements in one or more fluid end-use applications.

6.1.1 Antimicrobial pesticide needs typically fall into either or both of the following categories:

6.1.1.1 *Biodeterioration Prevention*—The various strategies used to enhance coolant life.

6.1.1.2 *Health and Safety*—Reducing the risk of employee exposure to potentially pathogenic microbes or allergenic microbial constituents such as endotoxins (Practice E2144).

6.2 Once the need has been recognized, the next step is to define the need operationally. This is achieved by determining the answers to the needs analysis questions, for example:

6.2.1 What type of metalworking fluid formulation requires microbicidal augmentation? Antimicrobials vary in their respective oil and water solubilities. Moreover, chemical incompatibilities exist between certain antimicrobials and other metalworking fluid constituents. Microbicides that are deemed inappropriate based on their incompatibility with the other formulation components need not be considered further. (See 9.1.)

6.2.2 What are the desired performance-life and biodegradability criteria for the finished formulation? Bioresistance and biodegradability need to be balanced. Waste treatability and extended sump life are both important considerations. (See Section 8.)

6.2.3 What respective roles should antimicrobial pesticides and bioresistant performance additives play in achieving those criteria? Metalworking fluid formulators can select from a growing number of bioresistant corrosion inhibitors and other performance additives that confer greater overall formulation bioresistance. Two caveats affect bioresistant additive selection:

6.2.3.1 Bioresistant additives should have some demonstrable performance benefit other than inhibiting biodeterioration.

6.2.3.2 The toxicological (for example, those described in Guide E1302) and environmental fate profiles of a bioresistant, putatively non-biocidal, performance additive shall be more benign than those of the microbicides they are replacing.

6.2.4 What are the target microbes? (See 7.3.)

6.2.5 Will the microbicide be added into the formulation, tankside, or both? (See 7.1.)

6.2.6 Will the microbicide, either in-formulation or as tankside additive be used at a single or multiple end-use sites? Approved chemical lists vary among companies conducting metalworking operations. Antimicrobials to be considered for use should be listed on prospective users' approved chemicals lists.

6.2.7 Will the microbicide, either in-formulation or as tankside additive be used domestically only, or will it be traded internationally? Industrial pesticide regulations differ around the world. Not all products approved by the U.S. EPA are approved in Canada, Europe, or other industrialized regions or vice versa. Moreover, registration and reporting requirements vary amongst nations. Global acceptability may be an important consideration (see Section 10).

6.3 Completion of this needs analysis step will facilitate the balance of the microbicide selection process.

7. Antimicrobial Pesticide Use Strategies

7.1 Microbicides may be added either in-formulation, tankside, or both. Users, understanding how the metalworking fluids they use are formulated, should select an appropriate pesticide use strategy for each end-use application.

7.1.1 In-formulation microbicide use means that antimicrobial(s) are formulated into coolant concentrate.

7.1.1.1 Microbicide addition at this stage may reduce or eliminate the requirement for subsequent tankside addition. It also protects ~~high-water-content~~ high-water-content formulations from spoilage during storage and transport.

7.1.1.2 When formulated into coolant, microbicides are added at concentrations sufficient to provide adequate a.i. once the formulation has been diluted to end-use strength. In-drum demand may reduce the residual microbicide concentration available by the time coolant concentrate is diluted for end use.

7.1.1.3 With coolants intended for a variety of end-use applications, each requiring different final coolant concentrations, it may be difficult to blend a single microbicide concentration in-drum. For example, assume that the target end-use microbicide concentration is ~~1 000~~1000 ppm and the expected coolant finished dilution range is 5 to 10 %. Blending microbicide into the formulation at 2 % will yield the desired ~~1 000~~1000 ppm when the coolant is diluted to 5 % and ~~2 000~~2000 ppm when the coolant is diluted to 10 %. The latter concentration may exceed the maximum microbicide concentration permitted under the microbicide's U.S. EPA pesticide registration. Using less microbicide in the concentrate might result in ineffective end-use strength.

7.1.1.4 Adding microbicides in-formulation requires a series of assumptions regarding antimicrobial pesticide demand during storage and in application.

7.1.1.5 Underdosing may select for microbes naturally resistant to the a.i.

7.1.2 A second treatment strategy, tankside use, refers to microbicide addition directly into the diluted coolant, in application. Tankside usage may permit tighter control of coolant system bioburdens. It may also improve targeting and reduce the chances of selection for treatment-resistant microbial communities. However, tankside use requires personnel at the use facility to handle microbicide concentrate and increases the risks associated with unauthorized or insufficiently trained personnel handling microbicides.

7.1.2.1 When used tankside, microbicide should be added to systems at points where mixing and ventilation is adequate and splash risk is minimal.

7.1.2.2 Tankside microbicide addition should be linked to condition monitoring to reduce the risk of overdosing or underdosing.

7.1.2.3 Microbicides should not be added tankside without consulting the coolant formulator. Antagonistic reactions between tankside antimicrobials and coolant constituents might denature the microbicide or cause the release of noxious vapors.

7.1.3 A third treatment strategy is to formulate microbicide into coolant concentrate to provide in-drum and some level of end-use protection, and to augment this with tankside additions, based on condition monitoring data. This approach reduces the amount of tankside microbicide required. It compensates for the uncontrolled variables that affect microbicide demand.

7.2 *Contamination Stage:*

7.2.1 Several tankside dosing strategies may be used to control microbial contamination in metalworking fluids. Microbicide may be added in response to data excursions beyond established control limits. They may be added according to a schedule. They may be added after coolant rancidity makes conditions at end-user facilities intolerable. Regardless of the strategy, antimicrobial pesticide should be added at sufficient concentration to be effective. Moreover, the duration of coolant and system exposure to an effective microbicide concentration should be sufficient to achieve contamination control.

7.2.2 Tankside addition linked to a condition-monitoring program generally provides the most cost-effective control. Data used to determine the need for biocide addition may include, but are not limited to, the parameters listed in Table 1.

7.2.3 At user facilities that lack adequate means for running condition monitoring tests, tankside microbicide treatment may be scheduled. Without data, this strategy creates two potential contamination control risks.

7.2.3.1 If the interval between microbicide additions is too short, a.i. concentration in the coolant may build up to excessive levels.

7.2.3.2 If the interval is too great, bioburdens may overwhelm the treatment, resulting in one or more of the problems listed in 4.1.

7.2.3.3 Minimally, users choosing a tankside microbicide treatment strategy should know the average coolant turnover rate and the relative loss rates due to dragout and evaporation, respectively. Microbicide additions should then be scheduled to maintain a.i. concentrations between upper and lower control limits specified by the manufacturer or coolant supplier.

7.2.4 Tankside microbicide use as a crisis response measure is generally ineffective, and is mentioned here only because it's a common practice within the metalworking industry. Once bioburdens are excessive, microbicide demand is likely to consume the added product before it can reduce the microbial population to acceptable levels. Moreover, shock treating heavily contaminated systems will generally cause masses of slime to slough off of system walls and plug-off filters, spray nozzles, or both. Frequently, treatment at this late stage must be accompanied by system cleanout.

7.3 *Target Microbes:*

7.3.1 Each U.S. EPA and EU registered antimicrobial pesticide has a range of target microbes against which it is particularly effective. Table 2 lists antimicrobials (a.i.) approved for use in metalworking fluids as of 01 January 2016, and Table 3 lists the biocidal substances approved for use in metalworking fluids as of 04 October 2016.

7.3.1.1 From time to time, government ~~agencies,~~agencies such as the U.S. EPA and the European Chemicals ~~Agency,~~Agency add or delete products to their list of antimicrobial pesticides. Tables 2 and 3 are therefore illustrative of the range of active ingredients and activity spectra available, but are not considered as authoritative for regulatory purposes for the United States, European Economic Union, or elsewhere.

TABLE 1 Diagnostic Tests for Determining Microbial Contamination in Metalworking Fluids

Procedure	ASTM Designation
Alkalinity	D1067
Odor[A]	N/A
Bacterial or fungal viable count[B]	D5465
Foaming tendency	D3519
pH	D1293
Two-hour oxygen demand[C]	D4478
Visual inspection[D]	N/A

[A] Musty, putrid, rotten egg, and other atypical odors in the vicinity of MWF systems are symptomatic of uncontrolled microbial contamination.
[B] Alternatives for traditional viable counts may be used. See Guide E1326 for more information.
[C] A significant bioburden typically will deplete at least 50 % of the dissolved oxygen in a coolant sample within 2 h.
[D] Visible slime stringers on machine surfaces and sluice walls provide unequivocal evidence of uncontrolled microbial contamination.