Evaluation Manual for the Authorisation of plant protection products and biocides

EU part

Biocides

Chapter 7 Efficacy

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Authors:
Lonne Gerritsen, PhD

Co-ordination:
Werner Pol, MSc, BSc

Lay-out:
Jiske de Wolf

ctgb

Board
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Chapter 7 Efficacy
Category: biocides

Main group 2 Preservatives
PT13 Metalworking fluids

Contents

GENERAL INTRODUCTION

This chapter describes the data requirements for the essesment of the efficacy of a biocide and the active substance within PT 13, and which evaluation methodologies are applied for the EU framework. This chapter is derived from the TNsG on product evaluation (appendix to chapter 7 on Product Type 13 metal working fluid preservatives).

In December 2012 a new chapter of the TNsG was endorsed describing requirements for the main group of preservatives (PT6 to PT13). See this chapter for more recent requirements. Where the chapter below is in contradiction with the general chapter the general chapter will prevail.

PRODUCT TYPE 13 - METALWORKING FLUID PRESERVATIVES

1 INTRODUCTION

1.1 Spectrum of biological activity (including target organisms)

1.1.1 Biological activity

Metalworking fluids that contain water may be spoiled by both bacteria and/or fungi, including yeasts. Metalworking fluid preservatives must therefore be capable of adequately protecting metalworking fluids from microbial attack. This may mean total elimination and exclusion of micro-organisms from the metalworking fluid. Alternatively, and more realistically, this may mean preventing the numbers of micro-organisms in the metalworking fluid from reaching levels high enough to cause deleterious effects.

1.1.2 Target organisms

Micro-organisms that spoil metalworking fluids may be free living in the water phase, and metabolise compounds that migrate from the oil phase. In addition, biofilms may develop within a system. The open nature of systems employing metalworking fluids provides many opportunities for microbial contamination.

Oil-in-water emulsion metalworking fluids are normally alkaline (*ca*. pH 8-9), and as they are circulated and thus aerated, the initial microbial contamination is normally by aerobic Gram-negative bacteria such as *Pseudomonas* spp (e.g. *P. aeruginosa*), *Actinobacter* spp. and *Aerobacter* spp. However, Gram positive bacteria such as *Bacillus* spp., and even some fungi may also be present even at relatively high pH values. As the flora within a system increases, oxygen deficiencies occur. Eventually some areas in a system become sufficiently anaerobic to allow sulfate-reducing anaerobic bacteria to proliferate. Anaerobic conditions can be made worse by weekend or other more prolonged plant shutdowns.

Semi-synthetic formulations often contain glycols as a partial replacement for oil. These formulations have a tendency to be more prone to fungal rather than bacterial attack. Fungi that will proliferate under such conditions include common moulds and yeasts.

Changes in pH and temperature will influence the type of microbial flora that proliferates in a particular metalworking fluid. For example, bacterial contamination may lead to a fall in pH, which will allow a secondary fungal attack.

1.2 Areas of use and sites of application

Metalworking fluids are applied to metal being worked (cutting, grinding, rolling, drawing etc). Because of this, metalworking fluids are widely used throughout the engineering industry, and in large quantities.

In a typical machine tool set-up, metalworking fluid is held in a tank. When the machine is operating, the metalworking fluid is pumped via pipes to the tool, where it is applied. The precise way in which the metalworking fluid is applied varies, but common methods are continuous jet, spray or mist. The metalworking fluid then returns to the storage tank. Metalworking fluids may also be applied to tools by hand dispensers.

Wherever metalworking fluids are being used, metalworking fluid preservatives will be required to control microbial attack.

1.3 Methods of application

Preservatives used to protect metalworking fluids from microbial attack may be added to the concentrate by the manufacturer, or to the dilution at the tank side.

Levels of preservatives in the metalworking fluid must be kept sufficiently high in order to maintain efficacy against micro-organisms. Therefore it will be necessary to add additional preservative from time to time, to replace that 'used up' or lost.

1.4 Instructions for use

The instructions for use define the precise way in which the product is used and will typically include:

- Preparation of the formulation for use
- Dilution rate

2 AVAILABLE DATA

2.1 Introduction

Laboratory-based simulated-use tests may be limited in their ability to predict field use-levels of preservatives in metalworking fluids. This is because the many plant variables that will influence overall preservative efficacy cannot be fully reproduced in the laboratory. These plant variables include machine characteristics, the metal being worked, changing fluid characteristics during use, and the particular micro-organisms present. Laboratory tests can be used to rank preservatives, but this ranking will only be relevant to the conditions prevalent during the study. Therefore, the laboratory study of preservatives in metalworking fluids can only give an indication that a particular biocide or combination of biocides will have at least some activity against micro-organisms in the field.

Field or in-use tests may be capable of predicting field use levels, but the recommendations stemming from a particular study will only be relevant for the plant conditions tested, and cannot be reliably extrapolated to other field situations. However, positive results from a number of field studies will provide a good indication that the biocide will be efficacious in other situations.

2.2 Simple laboratory tests

Information regarding the innate toxicity of metalworking fluid preservatives against a wide range of target species can be derived from minimum inhibitory concentration (MIC) studies.

Data from simple laboratory tests alone will not be sufficient to successfully support an application for authorisation.

2.3 Laboratory-based simulated-use tests

Simulated-use tests conducted under laboratory conditions attempt, to a lesser or greater extent, to emulate conditions encountered by metalworking fluid preservatives when in service.

Whilst the precise details of the various available standard laboratory-based simulated-use tests vary, in general they all follow a similar pattern.

Metalworking fluid including preservative is placed in a vessel, and may be shaken on a mechanical shaker, or directly aerated, with humidified air if necessary. Alternatively, a pump can be employed to maintain a recirculating flow of metalworking fluid that is allowed to fall back into the vessel under the influence of gravity. The shaker, aeration system or pump can be switched off in the evenings and/or at weekends to simulate plant shutdowns. The metalworking fluid may be maintained at ambient temperature, or heated to reflect normal operating conditions.

The metalworking fluid preservative is challenged by inoculation with a microbial culture at the start of test, and at intervals throughout the study period. The microbial culture may be derived from laboratory cultures, or from spoiled metalworking fluid collected in the field. The origins of all test micro-organisms should be stated.

Metal chips/filings/swarf etc may also be placed in the vessel containing the metalworking fluid.

The metalworking fluid may be monitored on a regular basis for changes in any or all of the following parameters:

- Visual appearance
- pH
- Oxygen uptake
- Presence, level and survival of micro-organisms (using standard sampling techniques)

Many multiples of the system described above can be operated at the same time, and for extended periods. Adequate untreated controls should always be included.

Full details of all tests methods should be available.

2.4 Field or in-use tests

In-use testing involves the antimicrobial evaluation of the product under actual conditions of use. Guidance concerning field testing of metalworking fluid preservatives is limited. However, some criteria may be considered:

- Data from field tests where the conditions provide severe challenges from harmful organisms may provide the strongest support for metalworking fluid preservatives.

- The tests should cover all pertinent factors associated with the intended use pattern(s).

- It may be difficult or impossible to run concurrent negative controls.

2.5 Standard test methods

Standard test methods have been produced by ASTM (US EPA and Canada) in North America. These standard test methods are listed in Appendix 1.

Whilst the use of standard test methods may be valuable for the assessment of the efficacy of metalworking fluid preservatives, use of these standards is not mandatory.

Competent authorities will consider testing strategies based on other national or international standard test methods, or, alternatively, non-standard test data, provided that they are both relevant and robust. It is relatively common for metalworking formulators to develop their own in-house efficacy test methods.

Other known test methods that may be used (following modification in some cases) for evaluation of the efficacy of metalworking fluid biocides are listed in Appendix 2.

APPENDIX 1

STANDARD PROTOCOLS FOR EFFICACY ASSESSMENT OF METALWORKING FLUID BIOCIDES (THIS LIST IS NOT EXHAUSTIVE)

Date	Title, organisation
1991	Standard test method for evaluation of antimicrobial agents in aqueous metal working fluids. ASTM, Philadelphia, USA.
1991	Standard test method for evaluation of antimicrobial agents as preservatives for invert emulsion and other water containing hydraulic fluids. ASTM, Philadelphia, USA.
1992	Anonymous, 1992. Standard test method for evaluating the bacteria resistance of water-dilutable metalworking fluids. ASTM, Philadelphia, USA.

APPENDIX 2

OTHER PROTOCOLS THAT MAY BE USED FOR THE EFFICACY ASSESSMENT OF METALWORKING FLUID BIOCIDES (THIS LIST IS NOT EXHAUSTIVE)

Date	Title, organisation
1987	Evaluation of the biostability of aqueous metalworking fluids. Renault test method No. D551721.
1987	A recirculating test rig for the investigation of metal-working fluid spoilage. In; Industrial microbiological testing. Rawlinson, A.P. and Shennan, J.L., 1987. Edited by Hopton, J.W. and Hill, E.C., 1987. Blackwell Scientific Publications, Oxford. ISBN 0 632 01793 7. pp227-231.
1987	South African standard specification for biocides for use in emulsions of aqueous metal working fluid and aqueous hydraulic fluid. The Council of the South African Bureau of Standards. SABS 1435-1987.
1990	Cutting fluid, soluble, biostable joint service designation ZX-9. UK MOD 91-70 issue.
1993	A standardized screening method for determining the bioresistance of and evaluating biocides in aqueous metalworking fluids. IBRG (draft MWF).

6. References

1 Biocidal directive (BPD) (98/8/EC)

2 Technical Notes for Guidance: TNsG on Product Evaluation; Common principles and practical procedures for the authorisation and registration of products. Available at: http://ecb.jrc.it/biocides/