

## AN EVALUATION OF A LABORATORY AND PLANT PROCEDURE FOR PRESERVATION OF CUTTING FLUIDS\*

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**Summary.** A sample laboratory model for the evaluation of cutting fluid formulations and biocides has been devised by studying performance in the plant at the same time as a derived laboratory sample was being examined. The study was carried out for twelve weeks in which pH, total aerobic count, and sulfate reducers were checked. It was possible to conclude that the laboratory system paralleled the plant results so that future studies could be limited to such a laboratory model.

**Une évaluation d'un procédé de laboratoire et d'installation industrielle pour préserver des fluides de coupe.** On a inventé un modèle dans le laboratoire pour évaluer des formules des fluides de coupe et des biocides, en étudiant le fonctionnement de l'installation et, en même temps, étudiant un échantillon préparé dans le laboratoire. On a effectué cette étude pendant douze semaines, et pendant ce temps on a vérifié le pH, le compte aérobie total et des réducteurs sulfatés. On pouvait conclure que les résultats obtenus dans le laboratoire étaient pareils à ceux de l'installation, afin qu'on puisse borner des études futures à un tel modèle dans un laboratoire.

### Introduction

Although several methods have been published during the past twenty years (Bennett, 1957; Himmel-farb and Scott, 1968; Pivnick and Fabian, 1953) which utilized rather simple laboratory procedures for evaluating stability of cutting fluid formulations and germicides for their preservation, none of these methods was carried out in conjunction with parallel field studies. It was always necessary to rely on co-relative predictability. In the present study a series of individual machines were compared on a weekly basis with the same cutting fluid maintained in the laboratory in a more convenient volume.

### Materials and Methods

Six machines, all containing individual 300-gallon sumps and described in detail in Table 1, were selected after consultation with the appropriate plant personnel. At the beginning of the study, 1 gallon of cutting fluid was taken from each machine and subsequently was used to set up the parallel laboratory evaluation system. For a period of 12 weeks the laboratory gallon was treated the same way as its progenitor plant system, unless otherwise noted. In some instances the parallelisms were exact; in others, it was the best approximation. For example, in the 6 machines, 3 germicides were being evaluated. The time of addition of germicide was about 3 days earlier in the plant than in the laboratory, since we had to await the arrival of communiques from plant personnel on levels added.

**Ein Abschätzung einer Laboratoriums- und Betriebsanlagehand-lungsweise für die Bewahrung von Schneideflüssigkeiten.** Ein Musterlaboratoriumsmodell für die Abschätzung von Schneide-flüssigkeiten formulierungen und Keimtötoren ist erfunden worden durch das Studieren von der Ausführung in der Betrieb-sanlage, indem man gleichzeitig eine abgeleitene Laboratoriums-probe untersucht wurde. Das Studium dauerte zwölf Wochenlang, währenddessen man pH, Gesamt-aerobikzahl und Sulfatabsch-wächer geprüft wurden. Es war möglich zu entscheiden daß das Laboratoriumssystem den Betriebsanlageer- gebnisse gleich waren, sodaß man zukünftige Untersuchung in einem solchen Laboratoriumsmuster ausführen könnte.

**Una Evaluación de un procedimiento del laboratorio y de las plantas para la conservación de los fluidos cortantes.** Se ha ideado una muestra de un modelo de laboratorio para la evaluación de las formulaciones de fluidos cortantes y biocidas, y se ha hecho estudiando el funcionamiento de la planta al mismo tiempo que se examinaba una muestra derivada del laboratorio. Se prosiguió el estudio durante doce semanas, en las que se registraron pH, la enumeración aeróbica total, y los reductores de sulfato. Se pudo concluir que el sistema del laboratorio se asemejaba muy bien a los resultados en las plantas de tal manera que en el porvenir se podrán limitar los estudios a tal modelo de laboratorio.

The type of soluble oil used for make-up in the plant was also used in the laboratory but in a more orderly but artificial replacement scheme. Individual machines were maintained at a constant level with replacement fluid to make up for loss due to carry off, spillage and evaporation. There was complete turn over about weekly. It was necessary to dump most machines approximately every 2 weeks due to the accumulation of swarf, fine and hydraulic oil. However, the machines were not cleaned prior to recharging and the sludge on the tank and tool surfaces served as inoculum for the new charge of fluid. To account for occasional addition of organic load in the plant, weekly additions of 1% cottonseed mash, 0.50% hydraulic fluid (tramp oil) were made. The 1-gallon sample was added to a 4-liter beaker containing 75 grams of cast-iron chips and was subjected to an aeration regimen of 5 days of continuous bubbling and 2 days without. At the conclusion of the quiescent period, which is supposed to simulate weekend shutdown in the plant, evaporation loss was made up with de-ionized water. At this time aliquots were removed for total aerobic bacterial counts, sulfate reducer counts, and pH determinations. Twenty five per cent of the total volume was removed from the beaker and replaced with fresh emulsion corresponding to that used in the plant. This total regimen was repeated for a period of 12 weeks.

**Bacteriology:** Total aerobic counts were done in plate count agar (Difco) and sulfate reducer counts

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TABLE 1—Description of plant machines

Machine No.	1	2	3	4	5	6
Speed of operation	Slow	Slow	Slow	High	Medium	Medium
Type of operation	Broaching	Broaching	Broaching	Machining	Machining	Machining
Capacity of pit (gallons)	300	300	300	300	300	300
Bacterial inhibitor used	Q	G	D	None	None	None
Nature of coolant	Heavy duty soluble oil (sulfurized fat type)	Heavy duty soluble oil (sulfurized fat type)	Heavy duty soluble oil (sulfurized fat type)	General purpose soluble oil (sulfurized)	General purpose soluble oil (sulfurized)	Heavy duty soluble oil (chlorinated)

D = -1-(3-chloro allyl)-3, 5, 7-triaza-1-azonia-adamantane

G = -Hexahydro-1, 3, 5-tris-2-hydroxyethyl-(5)-triazine

Q = -N-alkyl dimethyl benzyl ammonium chloride

TABLE 2—Medium for the enumeration of sulfate-reducing bacteria

Reagents	Amounts
$\text{KH}_2\text{PO}_4$	0.5 g
$\text{NH}_4\text{Cl}$	1.0 g
$\text{Na}_2\text{SO}_4$	1.0 g
$\text{CaCl}\cdot 6\text{H}_2\text{O}$	1.0 g
$\text{MgSO}_4\cdot 7\text{H}_2\text{O}$	2.0 g
Na Lactate	3.5 g
Yeast extract	1.0 g
Ascorbic Acid	0.1 g
Thioglycolic Acid	0.1 g
$\text{FeSO}_4\cdot 7\text{H}_2\text{O}$	0.5 g
Agar	15 g/liter

The pH is adjusted to 7.6 with NaOH. The medium is diluted to 1000 ml with distilled water, autoclaved for 15 minutes at 15 lb./in<sup>2</sup>, held between 40—44°C, and added aseptically to tubes containing not more than a 15% volume of the sample being tested. Mix, allow to set, and seal with a 1.5-cm plug of agar to prevent access of air to the inoculated portion; incubate in air until the number of black colonies (sulfate-reducing bacteria) shows no further increase (3 to 16 days).

were done in the medium devised by Postgate (1963) (Table 2) and prepared in 20ml screwcap tubes. These were done from plant as well as laboratory samples as close together in time as possible; pH's were measured on a Beckman Model G pH Meter.

*Inhibitors:* The 3 inhibitors used were N-alkyl dimethyl benzyl ammonium chloride (Q), Hexahydro-1,3,5-tris-2-hydroxyethyl-(s)-triazine (G), and 1-(3-chloro allyl)-3,5,7-triaza-1-azonia-adamantane (D) There was always about a 3-day lapse between additions in the plant and in the laboratory, since we had

to wait for the appropriate communique. Additions were made by plant personnel based on their subjective evaluations and not on recommendations from our laboratory. The concentrations used were in accordance with manufacturers' recommendations. Thus based on bacteriological results, these additions might not always appear so rational. Concentrations of each inhibitor are listed in Table 3 and are based on the amounts added at that time. The continual replacement of loss with fresh fluid and the regular disposal of the entire tank prevented build up of inhibitor.

TABLE 3—Weekly inhibitor additions with final concentration in ppm

	Week				
	1—4	5	6—10	11	12
Machine 1, Laboratory model 1 & 4	0	1800 Q <sup>1</sup>	900 Q	1000 G	2500 G
Machine 2, Laboratory model 2 & 5	0	5200 G <sup>2</sup>	2500 G	1000 G	2500 G
Machine 3, Laboratory model 3 & 6	0	2200 D <sup>3</sup>	1100 D	1000 G	2500 G

1. N-alkyl dimethyl benzyl ammonium chloride
2. Hexahydro-1, 3, 5,-tris-2-hydroxyethyl-(5)-triazine
3. 1-(3-chloro allyl)-3,5,7-triaza-1-azonia-adamantane

**Results and Discussion**

Machines 1, 2, and 3 were charged with one of the germicides at week 5, while machines 4, 5, and 6 had no germicidal additions during the course of the study. The parallel laboratory systems for the latter three machines had germicidal additions made to them from the 6th week on, as indicated with each figure.

Systems 1, 2, and 3 (Fig.'s 1, 2, and 3) show that for the first 5 weeks aerobic and sulfate-reducer counts in the plant and in the laboratory fluctuated to some extent but that the trends for both microbial types in both the systems generally changed in the same direction. In System 2 (Fig. 2), in which additional compound G was begun at week 5, all counts dropped to 0. In one (Fig. 1) the addition of Q from weeks 5-10 had minimal effect. The subsequent addition of G at week 11 produced a sharp drop in total count. However, in System 3 (Fig. 3) D produced a drop in sulfate reducers but not in total aerobic count; the addition of G at week 11 here resulted in an increase in counts, suggesting a possible antagonism and indicated the necessity of knowing previously such interactions before making large-scale commitments.

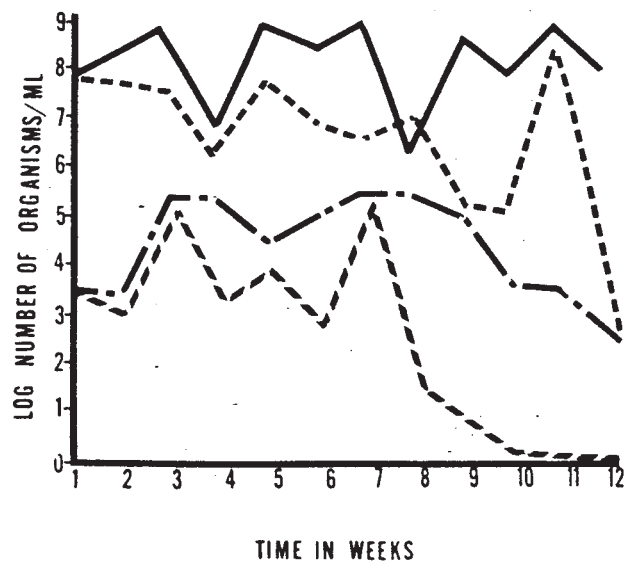
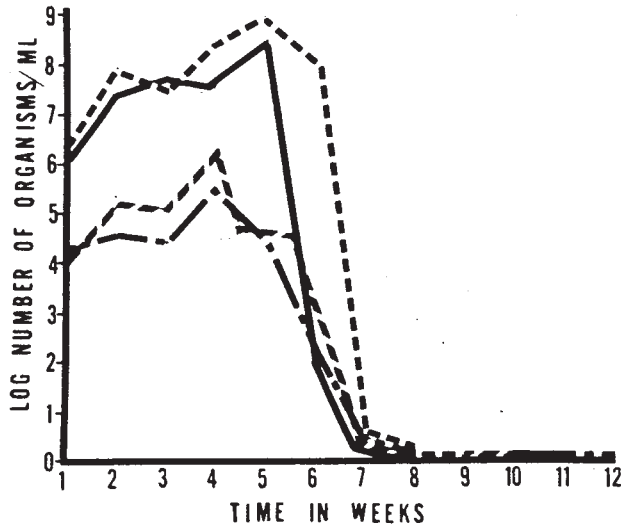
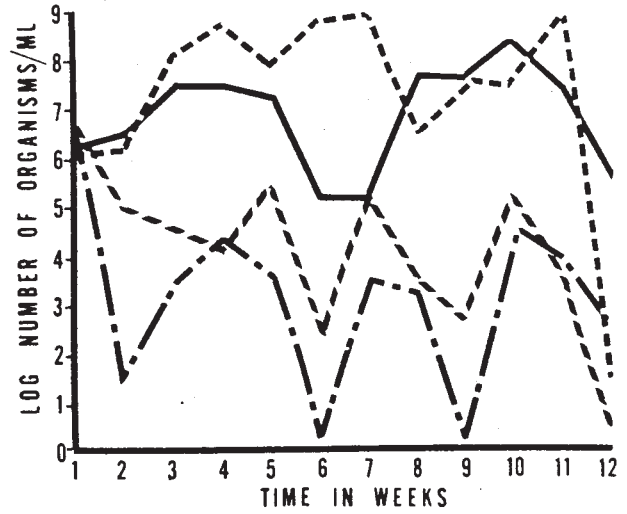


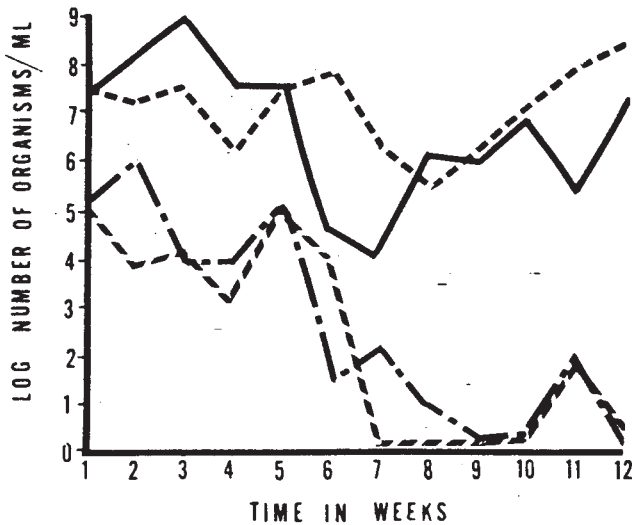
Figure 1 Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. N-alkyl dimethyl benzyle ammonium chloride was added to both from weeks 5 to 10 and Hexahydro-1,3,5-tris-2-hydroxyethyl-(s)-triazine added during weeks 11 to 12. Cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4 to the laboratory model



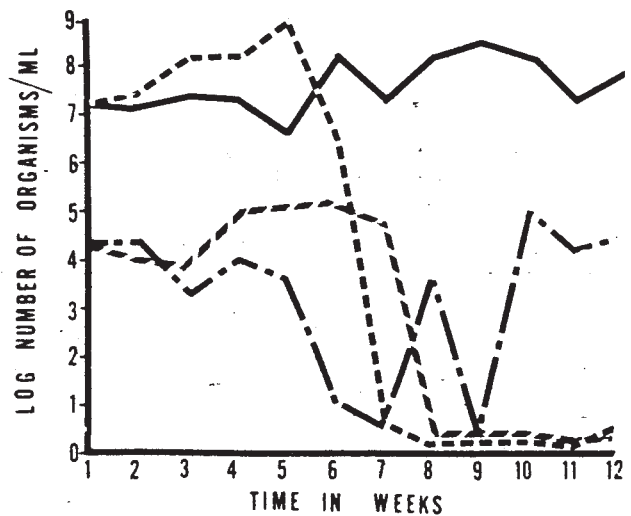
**Figure 2** Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. Hexahydro-1,3,5-tris-2-hydroxyethyl-(s)-triazine was added to both from weeks 5 to 12; cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4 to the laboratory model.



**Figure 4.** Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. The following additions were made to the laboratory model: N-alkyl dimethyl benzyl ammonium chloride was added from weeks 5 to 10 and Hexahydro-1,3,5-tris-2-hydroxy-ethyl-(s)-triazine added during weeks 11 to 12. Cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4.



**Figure 3** Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. 1-(3-chloro allyl) -3,5,7-triaza-1-azonia-adamantane was added to both from weeks 5 to 10 and Hexahydro-1,3,5-tris-2-hydroxyethyl-(s)-triazine added during weeks 11 to 12. Cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4 to the laboratory model.



**Figure 5.** Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. The following additions were made to the laboratory model: Hexahydro-1,3,5-tris-2-hydroxyethyl-(s)-triazine from weeks 5-12; cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4.

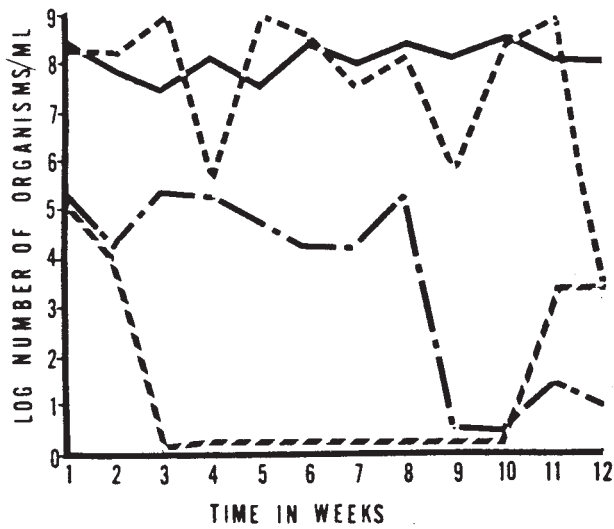


Figure 6. Comparison of bacterial levels in a machine cutting fluid sump with a derived laboratory model. The following additions were made to the laboratory model: 1-(3-chloro allyl)3-, 5, 7-triaza-1-azonia-adamantane was added from weeks 5 to 10 and Hexahydro-1,3,5-tris-2-hydroxy-ethyl-(s)-triazine added during weeks 11 to 12. Cast iron chips and cottonseed mash were added at week 2 and hydraulic oil at week 4.

In Systems 4, 5, and 6 (Fig's 4, 5, and 6), no germicidal additions were ever made to the machines in the plant but were made in the laboratory equivalents at the time indicated. These results for the most part reiterate those of Systems 1-3. Compound D (Fig. 6) is successful in lowering sulfate reducers counts but not total aerobic populations. Compound Q (Fig. 4) again proved ineffectual. However, compound G (Fig. 5) reduced the levels of both types of bacteria to undetectable levels.

In Table 4, the pH's for the entire test period are listed. The fluctuation in the machine pH's reflects primarily the dumping and re-charging that took place approximately every 2 weeks. In System 2 (Fig. 1) there is the best correlation between machine and its laboratory model; this is the system with the best microbial control and it emphasizes that drops

Key to figures 1-6

- Sulfate-reducer machine
- Aerobic bacteria machine
- - -○- - - Sulfate-reducer lab model
- - -△- - - Aerobic bacteria lab model

TABLE 4—pH measurements in emulsion systems

		Week—pH											
		1	2	3	4	5	6	7	8	9	10	11	12
1	L	7.4	7.2	7.2	7.5	7.7	7.7	7.7	7.8	7.4	8.3	8.3	8.5
	M	7.4	7.8	7.9	8.0	8.2	6.8	8.1	8.1	7.8	7.6	6.8	7.5
2	L	7.6	7.5	7.4	7.8	7.8	9.1	9.0	9.1	9.1	9.1	9.0	9.0
	M	7.6	7.4	7.6	8.0	8.2	9.2	9.0	9.1	9.2	9.2	8.8	8.5
3	L	7.4	7.8	7.5	8.0	8.1	8.1	8.2	8.2	8.1	8.0	8.1	8.3
	M	7.4	7.5	7.3	8.2	8.3	9.0	6.8	6.6	6.6	7.2	7.4	7.8
4	L	8.1	8.8	7.2	8.2	8.7	8.4	9.3	8.8	8.2	8.4	8.8	8.8
	M	8.1	9.0	8.8	6.8	9.1	9.1	9.2	9.2	8.1	9.2	9.1	9.2
5	L	8.1	8.3	7.8	7.9	8.2	8.1	8.8	9.2	9.2	9.3	9.1	9.0
	M	8.1	9.1	8.1	6.9	9.3	7.9	8.7	9.1	8.6	8.7	8.8	9.2
6	L	8.1	8.1	6.9	8.0	8.5	7.9	8.5	8.5	8.4	8.4	7.2	7.9
	M	8.1	8.1	8.6	6.9	9.1	9.1	9.2	9.2	8.9	9.1	9.1	9.1

L = Laboratory model  
 M = Machine

in ambient cutting fluid pH below 8.5 results from microbial action.

In view of the fact that the machine tanks had to be dumped at regular intervals because of accumulation of tramp (hydraulic) oil, more irregularity might have been expected in the bacterial counts from those cutting fluids. This was apparently not the case, implying that each machine may have its own resident flora which re-infect the new charge of cutting fluid. Since the machines were not cleaned between charges and since there is good indication that growth of microorganisms takes place primarily on surfaces and crevices, the assumption is no doubt a reasonable one. It would be wise, therefore, whenever possible to utilize an inoculum derived from the machines in which the inhibitor is to be subsequently used in evaluating that inhibitor. Although we have no comparable data for extrapolation, the same recommendation should be made for large central recirculating systems that are difficult to control.

At any rate, in the 6 systems evaluated, and in particular systems 1, 2, and 3, only compound G proved effective in reducing bacterial levels in the emulsions. From extensive observation in the laboratory and in the field, we have concluded that total counts must be maintained below  $10^7$ /ml for at least 6 weeks in assessing the capabilities of a biocide; compound G successfully passed that test.

#### Acknowledgement

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