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MICRO SEPAROMETER AND BALL-ON-CYLINDER LUBRICITY EVALUATOR TESTS
OF CORROSION INHIBITOR/LUBRICITY IMPROVER ADDITIVES

Patricia D. Liberio
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September 1989

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19. ABSTRACT (Continue on reverse if necessary and identify by block number) Due to the addition of the Ball-On-Cylinder Lubricity Evaluator (BOCLE) and Test for Water Separation Characteristics of Aviation Turbine Fuels by Portable Separometer to the Military Specification MIL-I-25017, Revision E, all of the Corrosion Inhibitor/Lubricity Improver additives on the Qualified Products List, QPL-25017-15, had to be tested using these methods. During this program each additive was evaluated and the results may be found in this report. The BOCLE testing resulted in two inhibitors being disqualified from the QPL. The Minimum Effective Concentration (MEC) of eight inhibitors shall be increased in the next revision of QPL-25017. Due to the Micro Separometer (MSEP) testing, two inhibitors shall have new Maximum Allowable Concentration (MAC) and one inhibitor shall be removed from the new QPL.					
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FOREWORD

This report was prepared by Patricia D. Liberio of the Fuels Branch, Fuels and Lubrication Division, Aero Propulsion and Power Laboratory, Wright Research and Development Center, Air Force Systems Command (WRDC/POSF), Wright-Patterson Air Force Base, Ohio 45433-6563. The work reported herein was performed under Project 3048, "Fuels and Lubrication," Work Unit 30480585, "Fuel/Fuel System Support Engineering." This report covers testing accomplished in-house from July 1988 to May 1989.

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LIST OF ABBREVIATIONS

AFB	Air Force Base
ASTM	American Society for Testing and Materials
BOCLE	Ball-On-Cylinder Lubricity Evaluator
CRC	Coordinating Research Council
g	gram
g/m^3	gram per cubic meter
MAC	Maximum Allowable Concentration
MEC	Minimum Effective Concentration
mm	millimeter
MSEP	Micro Separometer
QPL	Qualified Products List
REC	Relative Effective Concentration
WRDC	Wright Research and Development Center

SECTION I

INTRODUCTION

A. BACKGROUND

In the past, corrosion inhibitors were used as lubricity improvers in jet fuels. Unfortunately there was no good test to determine how well these inhibitors improve lubricity. In the early 1960s the Ball-On-Cylinder Lubricity Evaluator (BOCLE) was recognized as the best method for measuring the lubricity properties of jet fuels. During the period of November 1985 through November 1986, Pratt & Whitney conducted a program which was directed at refining and standardizing the Ball-On-Cylinder Lubricity Evaluator (BOCLE) by identifying variables suspected of reducing test method precision. The results of this program are reported in AFWAL-TR-87-2041, Standardization of Lubricity Test. As this program concluded, the Coordinating Research Council, Inc. (CRC) supervised a round-robin evaluation of the BOCLE's precision and reproducibility, resolved and defined the details of the apparatus, and established an operating procedure. This work is described in the CRC Report No. 560 entitled Aviation Fuel Lubricity Evaluation. As a result of this work, the BOCLE has emerged as the test apparatus capable of providing a quantitative value for fuel lubricity. Recently Revision E to Military Specification MIL-I-25017, "Inhibitor, Corrosion/Lubricity Improver, Fuel Soluble," has incorporated the BOCLE as the method to test the lubricity of corrosion inhibitors.

Pratt & Whitney, under contract to the Air Force, ran a series of BOCLE tests on all products on QPL-25017-15, "Qualified Products List of Products Qualified Under Military Specification MIL-I-25017". They tested the inhibitors in Isopar M, Clay Treated JP-4, Clay Treated JP-5, and Clay Treated JP-8 using a 500-g load. Their results can be found in AFWAL-TR-88-2036, Evaluation of Corrosion Inhibitors as Lubricity Improvers. In the CRC Report No. 560, a 1000-g load test parameter was concluded to be more reproducible than the 500-g load used by Pratt & Whitney in their test. This is the main reason for the government retesting of the inhibitors.

When MIL-I-25017 was last revised in the Spring of 1989, there were many discussions about how the Minimum Effective Concentration (MEC) should be established using the BOCLE. After reviewing the BOCLE data and relating it to field experience, the MEC was established to be the larger of the following: one and a half (1.5) times the Relative Effective Concentration (REC) or the amount of inhibitor that gives a wear scar diameter of 0.65 mm or less when tested for lubricity using the BOCLE.

Another change to MIL-I-25017 occurred due to the replacement of ASTM D 2550, "Test for Water Separation Characteristics of Aviation Turbine Fuels," with ASTM D 3948, "Method for Determining Water-Separation Characteristics of Aviation Turbine Fuels by

Portable Separometer". All of the inhibitors had to be tested using the Micro Separometer (MSEP) in order to confirm the Maximum Allowable Concentration (MAC) of each inhibitor.

B. PROGRAM OBJECTIVES

The objective of this test program was to test all of inhibitors on the current Qualified Products List (QPL-25017-15) using the MSEP and BOCLE. The approved inhibitors must have a MSEP rating of 70 or above at their MAC. Also, each inhibitor would have to give a wear scar diameter of 0.65 mm or less at a concentration less than the established MAC in order to be a candidate for the QPL. If this concentration is larger than the REC multiplied by 1.5, it will become the inhibitor's MEC.

SECTION II

INHIBITOR EVALUATION

Fifteen inhibitors from the QPL-25017-15 and one inhibitor, ProChem SPEC-AID 8Q21, which has recently completed government approval as a Category 2 additive, were evaluated.

A. Micro Separometer Rating (MSEP)

1. Test Parameters

The 13 corrosion inhibitors which passed the BOCLE were tested using the MSEP according to ASTM D 3948. Samples of these inhibitors at their MAC were sent to McDill AFB to be evaluated. After review of the results, six inhibitors at varying concentrations were retested at McDill because their MSEP ratings were below the acceptable rating of 70. Of these six inhibitors, four, which showed objectionable results after retesting, were sent to Pratt & Whitney to establish at what concentration they would pass the MSEP. ProChem SPEC-AID 8Q21 was also evaluated at Pratt & Whitney so its MAC could be established.

2. Test Data

Table 1 lists the 13 corrosion inhibitors tested at McDill AFB and their ratings. Note that these inhibitors were tested at their MAC listed in the QPL-25017-15. The six inhibitors that failed the first MSEP evaluation are listed in Table 2. This table shows the concentration they were retested at and the new MSEP rating. The corrosion inhibitors tested at Pratt & Whitney are listed in Table 3 with their test results.

3. Conclusions

Eleven of the thirteen inhibitors which passed the MSEP test at the MAC listed in the QPL-25017-15 are: PRI-19, DCI-4A, HITEC 580, NALCO 5403, TOLAD 245, UNICOR J, IPC 4410, IPC 4445, MOBILAD F800, NUCHEM PCI-105, and WELCHEM 91120. Due to the MSEP₃ testing at Pratt & Whitney, DCI-6A will have a new MAC of 9 g/m³ on the next revision of the QPL. Nalco 5405 did not give consistent results when tested at McDill AFB and Pratt & Whitney, so it was evaluated extensively at an independent Approved Testing Laboratory using the MSEP₃. The results of this testing gave Nalco 5405 a new MAC of 11 g/m³. The newly approved Category 2₃ additive, ProChem SPEC-AID 8Q21 will have a MAC of 22.5 g/m³. These results are summarized on Table 4.

TABLE 1. MSEP Ratings of Thirteen Inhibitors

Inhibitor Name	Concentration of Inhibitor (g/m ³)	MSEP Rating
PRI-19	22.5	68*
DCI-4A	22.5	98
DCI-6A	22.5	55*
HITEC 580	22.5	73
NALCO 5403	22.5	69*
TOLAD 245	31.5	70
UNICOR J	22.5	77
IPC-4410	22.5	88
IPC-4445	22.5	80
MOBILAD F800	22.5	69*
NALCO 5405	22.5	50*
NUCHEM PCI-105	18	71
WELCHEM 91120	22.5	65*

* Failure

TABLE 2. MSEP Ratings of Six Inhibitors Retested

Inhibitor Name	Concentration (g/m ³)	MSEP Rating
PRI-19	22.5	52*
DCI-6A	20	53*
NALCO 5405	20	90
NALCO 5403	22.5	64*
WELCHEM 91120	22.5	92
MOBILAD F800	22.5	91

* Failure

TABLE 3. Pratt & Whitney MSEP Results

Inhibitor Name	Concentration of Inhibitor (g/m ³)	MSEP Rating (average)
PRI-19	22.5	77
DCI-6A	9.0	80
NALCO 5403	22.5	82
NALCO 5405*	13.5	64
	18.0	69
	22.5	70
	22.5	64
	31.5	67
	31.5	62
PROCHEM SPEC-AID 8Q21	22.5	85

* As a result of this data, this additive was evaluated at an independent Approved Testing Laboratory

TABLE 4. Conclusions of MSEP Testing

Inhibitor Name	MAC* (g/m ³)	MSEP Rating at MAC
PRI-19	22.5	77
DCI-4A	22.5	98
DCI-6A	9.0	80
HITEC 580	22.5	73
NALCO 5403	22.5	82
TOLAD 245	31.5	70
UNICOR J	22.5	77
IPC-4410	22.5	88
IPC-4445	22.5	80
MOBILAD F800	22.5	91
NALCO 5405	11.0	74
NUCHEM PCI-105	18.0	71
WELCHEM 91120	22.5	92
PROCHEM SPEC-AID 8Q21**	22.5	85

* MAC to be reflected on next revision of QPL

** New addition to QPL

B. Ball-On-Cylinder Lubricity Evaluator (BOCLE)

1. Test Parameters

Each inhibitor was tested using the BOCLE method described in Appendix Y of the CRC Report No. 560. The applied load used was 1000-g as established in the CRC round-robin. All Falex Rings used during this test program were calibrated using the two standard reference fluids recommended by the Pratt & Whitney standardization work: neat Isopar M and Isopar M with 30 ppm DCI-4A. Test temperatures were at 25°C. Each inhibitor was tested in Isopar M at a minimum of three different concentrations.

Isopar M was used as the solvent for three main reasons. Through the testing that Pratt & Whitney performed, Isopar M showed equivalent results to those of JP-4, JP-5 and JP-8. It is also a standard available fluid which is easily obtained at a reasonable price.

2. Test Data

Table 5 lists all the inhibitors and the wear scars achieved at the specified concentrations of the inhibitor in Isopar M. All data points met the repeatability standards defined by the CRC report. Figures 1 through 16 show these data points plotted, wear scar₃ diameter (mm) versus concentration of inhibitor in Isopar M (g/m³). Also included in the figures are the equations of the curves calculated using a second order polynomial curvefit program and the correlation coefficient of the curve. These equations were used to calculate the concentration of the inhibitor needed to create a 0.65 mm wear scar diameter. These calculated concentrations are listed on Table 6.

3. Conclusions

All of the concentrations calculated from the BOCLE data were rounded up to establish the new MEC. Table 7 lists the new MEC and old MEC for all of the inhibitors. The MEC for DCI-4A, DCI-6A, and Nuchem PCI-105 shall be determined by 1.5 times the REC since the BOCLE resulted in a concentration lower than 1.5 times the REC. The MEC of five inhibitors, DCI-4A, DCI-6A, Unicor J, IPC-4410 and Nuchem PCI-105 shall remain the same as the old QPL. The remaining 10 inhibitors shall see an increase in the MEC. Note that the increase in the MEC for Lubrizol 541 and Tolad 249 results in the MEC being higher than the MAC. Hence these two inhibitors shall be deleted from the new₃QPL. ProChem SPEC-AID 8Q21's MEC was calculated to be 11 g/m³.

TABLE 5. BOCLE Test Data

CORROSION INHIBITOR	Wear Scar Diameter (mm) at							
	0	4.5	6.8	9.1	11.3	15.1	22.7	31.8
	g/m ³	g/m ³	g/m ³	g/m ³	g/m ³	g/m ³	g/m ³	g/m ³
DCI-4A	0.84	0.71	0.68		0.60		0.52	
HITEC 580	0.85		0.76		0.67		0.58	
PRI-19	0.83		0.77		0.71	0.66	0.61	
DCI-6A	0.81		0.66	0.63	0.62	0.59	0.58	
LUBRIZOL 541	0.85		0.77		0.69	0.66	0.62	
NALCO 5403	0.85		0.76	0.70	0.68		0.57	
NALCO 5405	0.85	0.74	0.70	0.67			0.57	
TOLAD 245	0.84			0.76		0.72		0.63
TOLAD 249	0.84		0.79		0.80	0.75	0.65	0.63
UNICOR J	0.82	0.73		0.64			0.55	
IPC-4410	0.82	0.75	0.66		0.60	0.59	0.56	
IPC-4445	0.85		0.78		0.72	0.66	0.60	
MOBILAD F800	0.84	0.76	0.73		0.63		0.56	
WELCHEM 91120	0.83		0.72		0.64	0.63	0.57	
NUCHEM PCI-105	0.83		0.74		0.69	0.68	0.59	
PROCHEM SPEC-AID 8Q21	0.82	0.76	0.70	0.65	0.65	0.58	0.54	

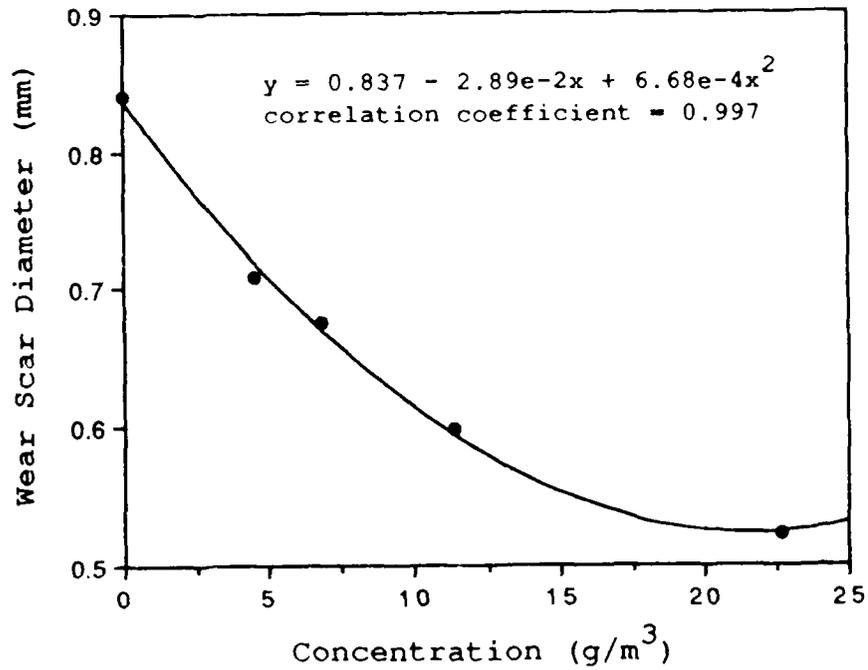


Figure 1. DCI-4A BOCLE Data

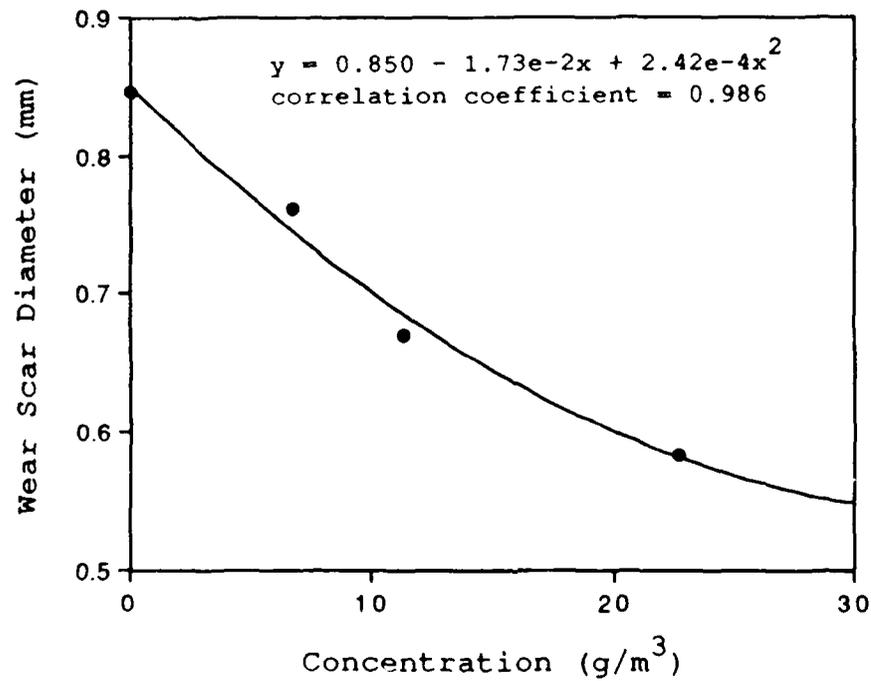


Figure 2. HITEC 580 BOCLE Data

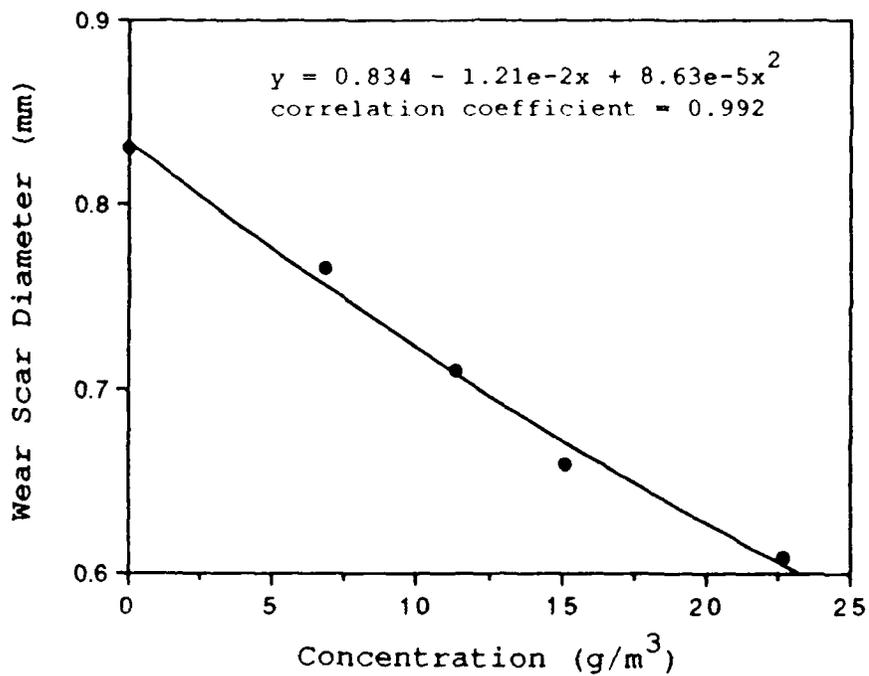


Figure 3. PRI-19 BOCLE Data

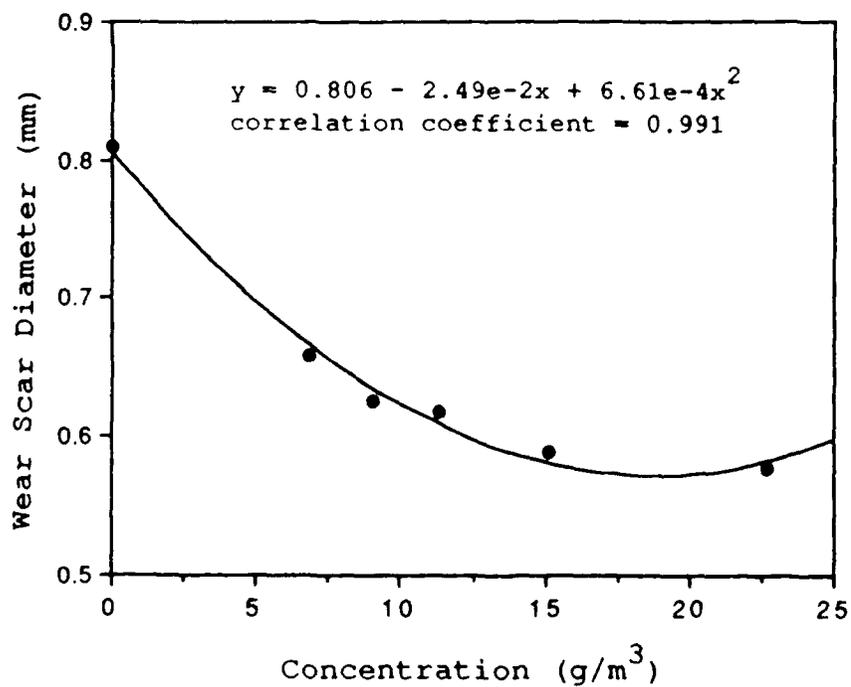


Figure 4. DCI-6A BOCLE Data

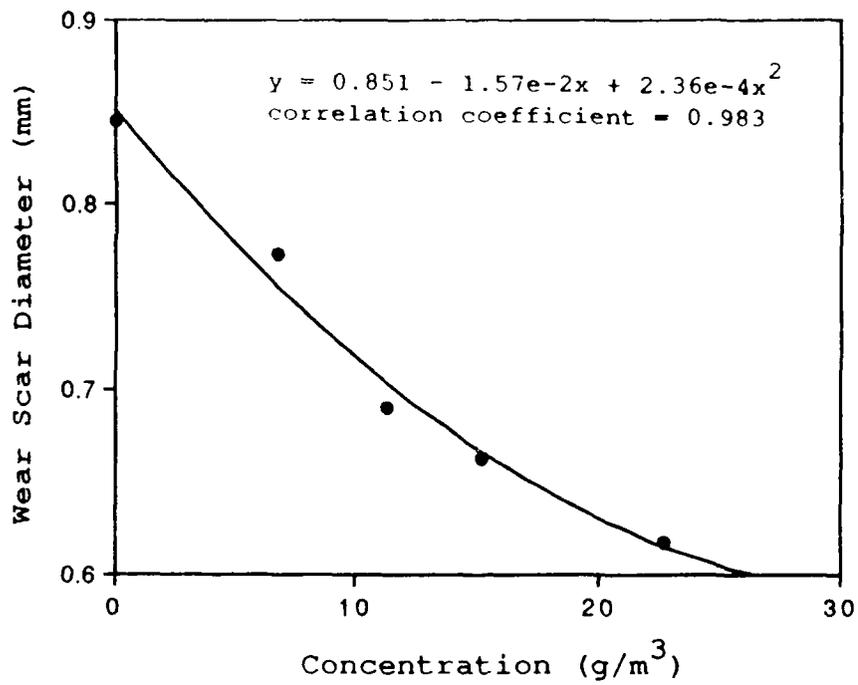


Figure 5. LUBRIZOL 541 BOCLE Data

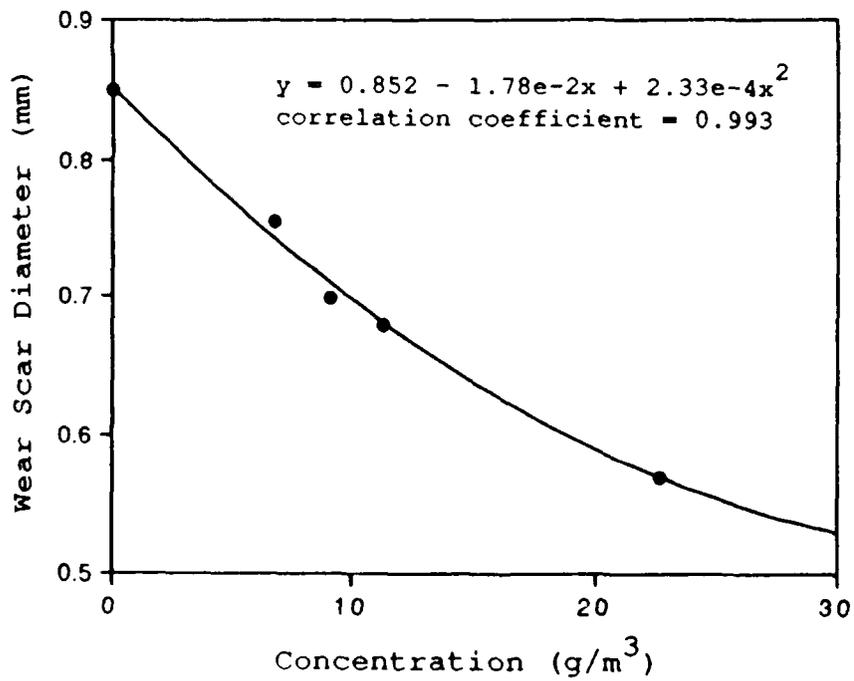


Figure 6. NALCO 5403 BOCLE Data

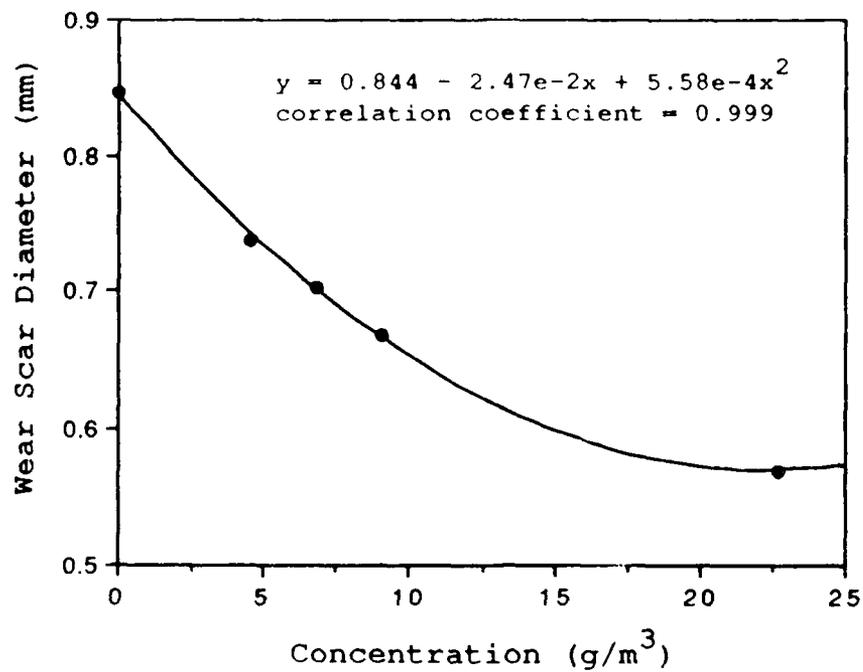


Figure 7. NALCO 5405 BOCLE Data

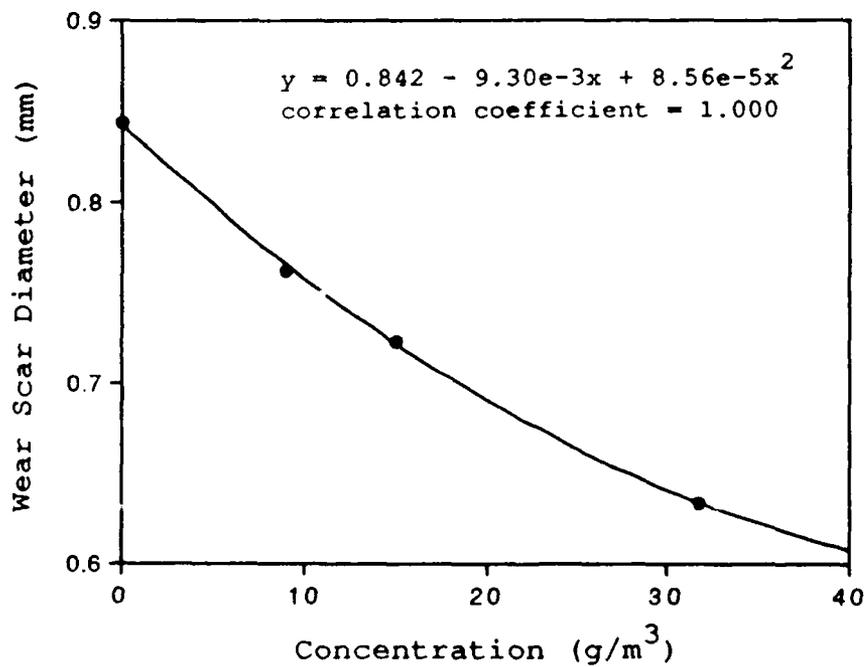


Figure 8. TOLAD 245 BOCLE Data

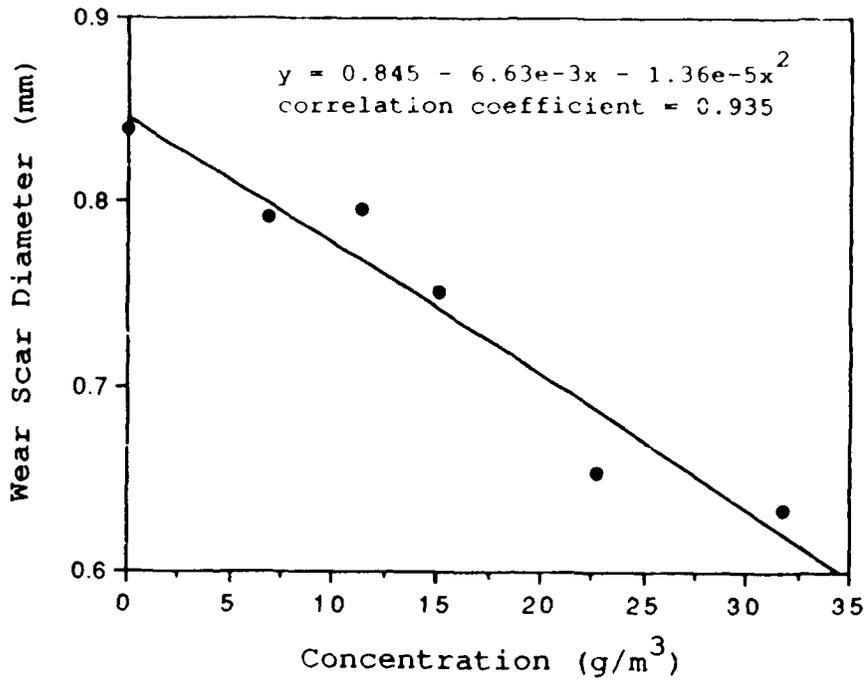


Figure 9. TOLAD 249 BOCLE Data

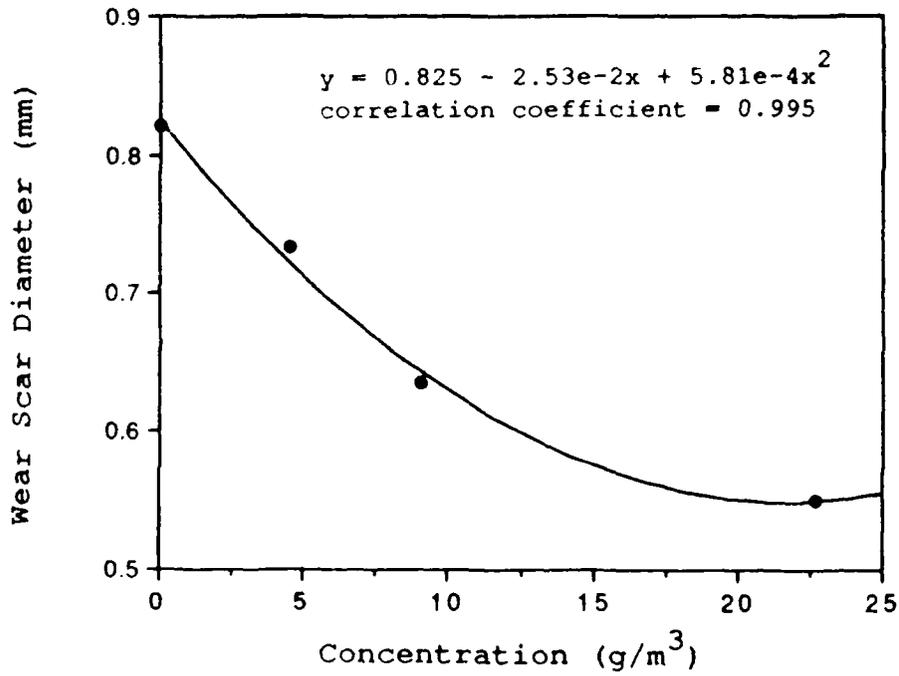


Figure 10: UNICOR J BOCLE Data

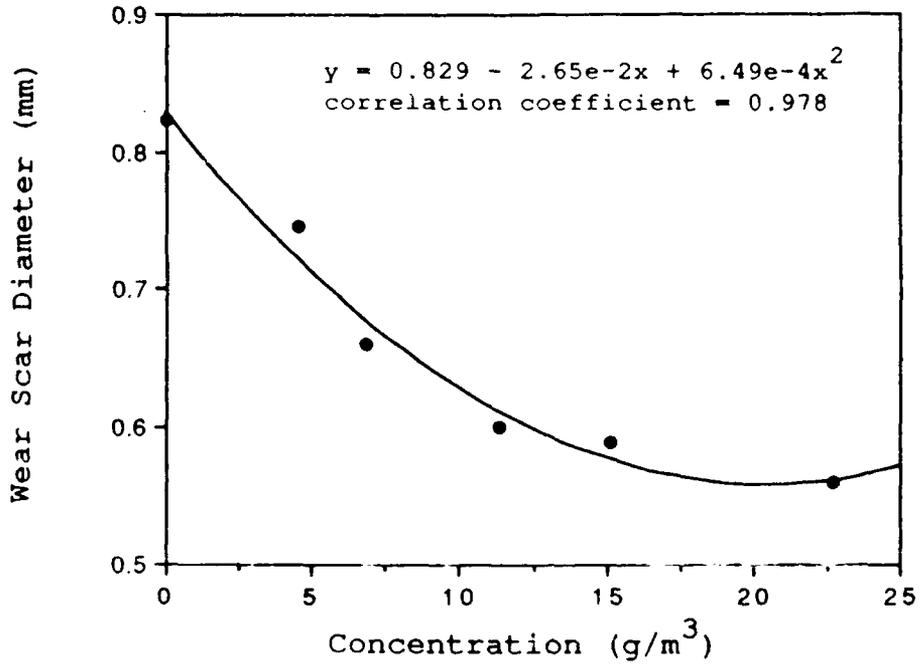


Figure 11: IPC-4410 BOCLE Data

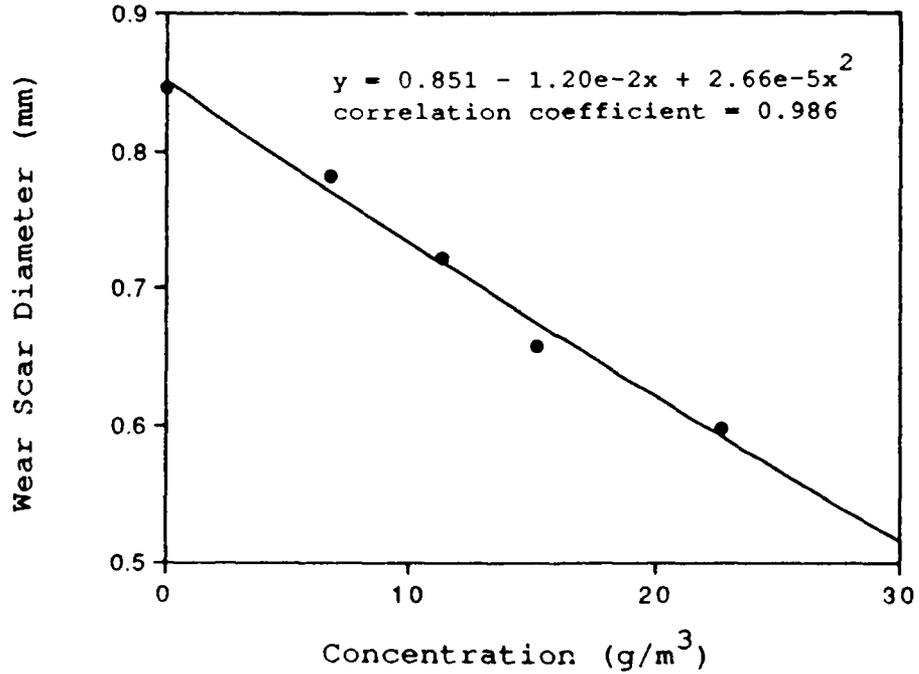


Figure 12: IPC-4445 BOCLE Data

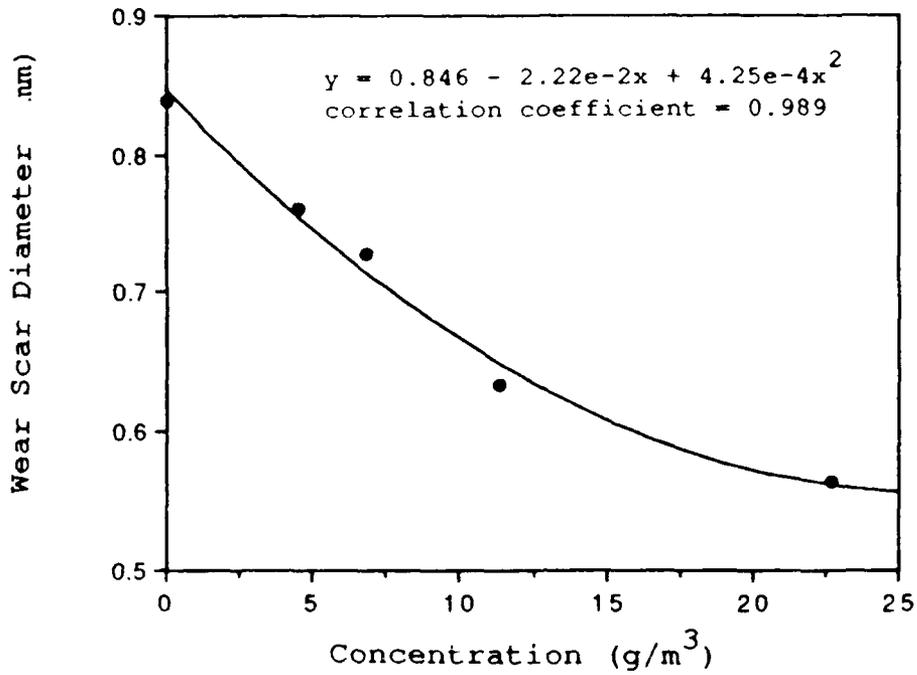


Figure 13: MOBILAD F800 BOCLE Data

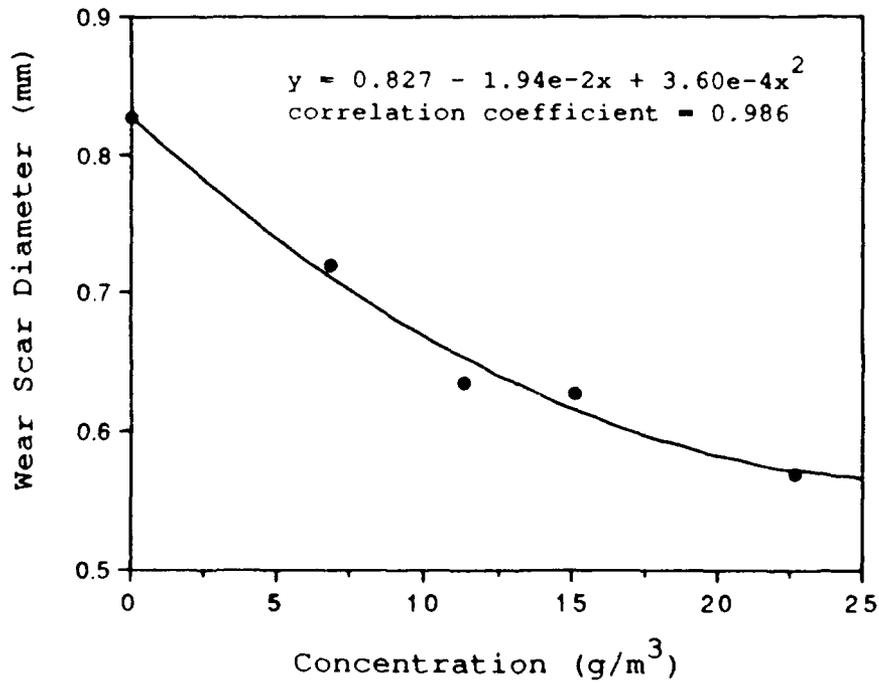


Figure 14: WELCHEM 91120 BOCLE Data

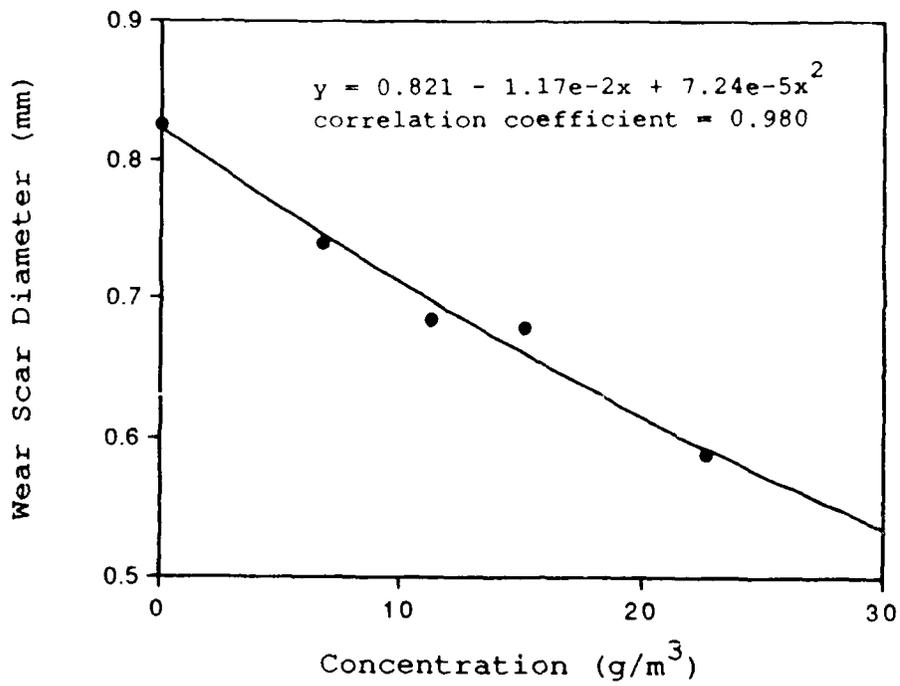


Figure 15: NUCHEM PCI-105 BOCLE Data

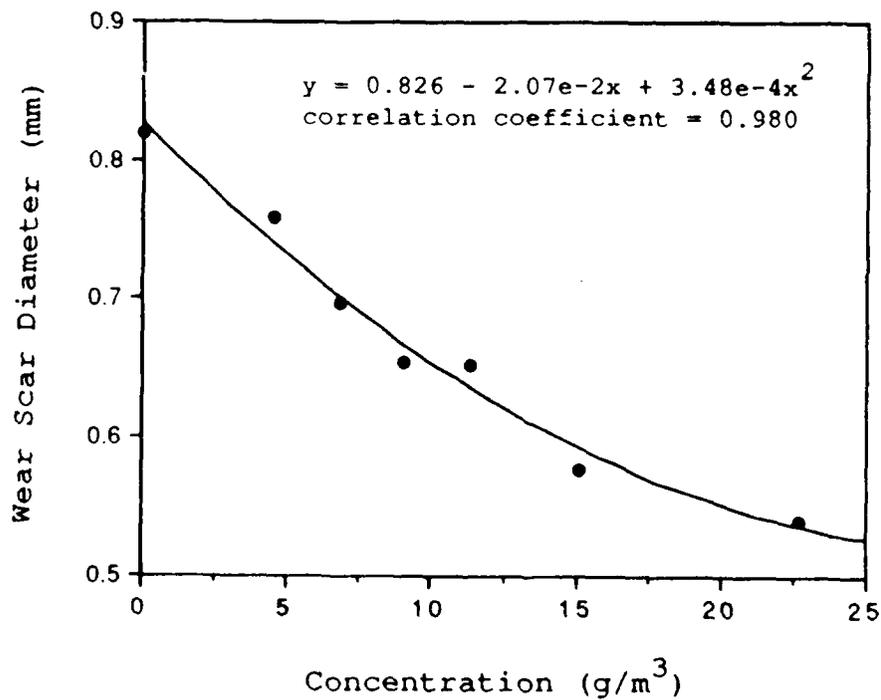


Figure 16: PROCHEM SPEC-AID 8Q21 BOCLE Data

TABLE 6. Calculated Minimum Effective Concentration (MEC)

Corrosion Inhibitor	Calculated ₃ Concentration (g/m ³) at 0.65 Wear Scar Diameter

DCI-4A	7.9*
HITEC 580	14.5
PRI-19	17.4
DCI-6A	7.9*
LUBRIZOL 541	17.3**
NALCO 5403	13.9
NALCO 5405	10.2
TOLAD 245	27.7
TOLAD 249	27.8**
UNICOR J	8.6
IPC-4410	8.5
IPC-4445	17.4
MOBILAD F800	11.3
WELCHEM 91120	11.6
NUCHEM PCI-105	16.2*
PROCHEM SPEC-AID 8Q21	10.3

* Lower than 1.5 X Relative Effective Concentration
 ** Higher than Maximum Allowable Concentration (MAC)

TABLE 7. Comparison of Minimum Effective Concentrations (MEC)

Corrosion Inhibitor	New MEC ₃ (g/m ³)	Old MEC ₃ (g/m ³)
DCI-4A	9	9
HITEC 580	15	9
PRI-19	18	9
DCI-6A	9	9
LUBRIZOL 541	18	9
NALCO 5403	14	9
NALCO 5405	11	9
TOLAD 245	28	22.5
TOLAD 249	28	9
UNICOR J	9	9
IPC-4410	9	9
IPC-4445	18	9
MOBILAD F800	12	9
WELCHEM 91120	12	9
NUCHEM PCI-105	18	18
PROCHEM SPEC-AID 8Q21	11	--

SECTION III
RECOMMENDATIONS

Table 8 lists the recommended REC, MEC and MAC for the next revision of the QPL. As a result of this program two inhibitors, Lubrizol 541 and Tolad 249, will be deleted from the QPL. Two inhibitors, DCI-6A and Nalco 5405, will have a lower MAC. Eight inhibitors, Hitec 580, PRI-19, Nalco 5403, Tolad 245, IPC-4445, Nalco 5405, Mobilad F800 and Welchem 91120, will have an increase₃ in their MEC. ProChem₃SPEC-AID 8Q21 shall have a MEC of 11 g/m³ and a MAC of 22.5 g/m³.

TABLE 8. CPL Recommendation

Corrosion Inhibitor	REC ₃ (g/m ³)	MEC ₃ (g/m ³)	MAC ₃ (g/m ³)
DCI-4A	6	9	22.5
HITEC 580	6	15	22.5
PRI-19	6	18	22.5
DCI-6A	6	9	9.0
NALCO 5403	6	14	22.5
NALCO 5405	6	11	11.0
TOLAD 245	15	28	31.5
UNICOR J	6	9	22.5
IPC-4410	6	9	22.5
IPC-4445	6	18	22.5
MOBILAD F800	6	12	22.5
WELCHEM 91120	6	12	22.5
NUCHEM PCI-105	12	18	18.0
PROCHEM 8Q21	6	11	22.5

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