PROTOCOL

Standard Test Method for Evaluating the Effectiveness of Cleaning Agents

Tested Formulas:

EcoCompounds 2861518
(Industrial Floor, Gnarly, Rig Wash)

Tested Competitive Name Brand:

ZEP
Product No(E0516228B1)

PREPARED FOR

EcoCompounds Inc.
1536 Eastman Avenue, Suite A
Ventura, CA 93003

PERFORMING LABORATORY

Capco Analytical Services
1536 Eastman Avenue, Suite B
Ventura, CA 93003

PREPARED BY

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DATE

03/10/2017

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Evaluating the Effectiveness of Cleaning Agents

SPONSOR: EcoComponds Inc.
1536 Eastman Avenue, Suite A
Ventura, CA 93003

TEST FACILITY: Capco Analytical Services
1536 Eastman Avenue, Suite B
Ventura, CA 93003

PURPOSE
The purpose of this study is to evaluate the efficacy of EcoCompounds Formula to a Name Brand competitive product per ASTM G121 and G122.

1. Scope

1.1 This test method covers a procedure for evaluating the capability of cleaning agents and processes to remove contamination to the desired level.

1.2 The test coupons provide a relatively rough surface to which contamination can easily adhere.

1.3 The capability of a particular cleaning agent depends upon the method by which it is used and the characteristics of the article being cleaned, such as size, shape, and material. Final evaluation of the cleaning agent should include testing of actual products and production process.

1.4 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.5 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 and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Note 2 .

2. Documents

2.1 *ASTM Standards:*[2]
D1193 Specification for Reagent Water
E177 Practice for Use of the Terms Precision and Bias in ASTM Test Methods
E691 Practice for Conducting an Inter-laboratory Study to Determine the Precision of a Test Method
G94 Guide for Evaluating Metals for Oxygen Service
G121 Practice for Preparation of Contaminated Test Coupons for the Evaluation of Cleaning Agents

2.2 *ANSI Standard:*[3]
D46.1 Surface Texture (Surface Roughness, Waviness, Lay)

3. Terminology

3.1 Definitions:

3.1.1 *Cleaning effectiveness factor (CEF), n*—the fraction of contaminant removed from an initially contaminated test coupon and determined by gravimetric techniques.

3.1.2 *residual contamination, R_C, n*—the absolute mass of contaminant remaining after the cleaning process and ex- pressed in milligrams per square centimeter of area or option- ally as milligrams per square foot.

3.1.3 *Surface roughness, R_A, n*—the arithmetic average deviation of the surface profile from the centerline, normally reported in micrometers.

4. Summary of Test Method

4.1 This test method provides quantitative results as to the ability of a specific cleaning agent/process for removing selected contaminants from standard coupons. The coupons used for testing are prepared in accordance with Practice G121. Cleaning is performed using a cleaning tank with or without ultrasonic agitation; elevated temperature or other cleaning enhancement features and depends on the manufacturer's instructions. The effectiveness of the cleaning process is represented as CEF, the cleaning effectiveness factor that is the fraction of the contaminant removed as determined by gravimetric techniques. A control coupon is used to account for any corrosion or material removal effects due to the cleaning agent/process.

5. Significance and Use

5.1 The purpose of this test method is to define a procedure for evaluating the capability of cleaning agents to clean metallic coupons. Based on the outcome of the testing, suitable cleaning agents may be selected for cleaning in general and for oxygen service in particular.

5.2 The cleaning parameters can be changed and the test method can be repeated. The usual cleaning parameters include cleaning agent concentration, temperature, and time; type and strength of ultrasonic energy or agitation, if used, and others.

NOTE 1—usual cleaning parameters are based on the manufacturer's recommendations.

6. Apparatus

6.1 Materials:

6.1.1 *Test Coupon,* prepared in accordance with Practice G121. The mass of the coupon is approximately 30 to 45 g but will vary significantly for each selected material. Typical materials used in oxygen systems are described in Guide G94.

6.1.2 *Control Coupon*—This is uncontaminated and is sub- jected to the identical cleaning procedure as the contaminated coupons and serves to evaluate corrosion and erosion of the test coupons.

6.1.3 *Cleaning Agent,* prepared according to the manufacturer's instructions. Specification D1193 Type II water shall be used for preparing aqueous solutions.

6.2 Equipment:

6.2.1 *Cleaning Tank,* A vessel of sufficient size to conduct a number of evaluations simultaneously. Testing is enhanced by having automatic temperature and time controls. A cleaning tank with ultrasonic may be used.

6.2.2 *Balance,* accuracy to 0.1 mg. However, 0.01 mg accuracy is desirable to detect contamination levels of 10 mg/m^2 (1 mg/ft^2) or less.

6.2.3 *Beaker Holder*—A device to support beakers in the ultrasonic cleaner tank such that the beakers do not contact the bottom and sides of the tank.

7. Test Procedure

7.1 Prepare a minimum of six test coupons by Practice G121.

7.2 Indicate the masses of coupons in grams as *MXy* where *X* is the coupon designation (number, letter, or name) and *y* = 1 indicates a clean coupon, *y* = 2 indicate a contaminated coupon and *y* = 3 indicate a coupon after cleaning.

7.3 Designate one coupon as the control coupon to undergo cleaning without contamination.

7.4 Measure the mass of the control and test coupons (recording them as *MX*1 as previously defined).

7.5 Contaminate five test coupons in accordance with Practice G121.

7.6 Measure the mass of all contaminated test coupons (recording them as *MX*2 as previously defined).

7.7 Process the control coupon in the test cleaning solution separately from the contaminated test coupons.

7.8 The contaminated test coupons can be processed in independent beakers held in the cleaning tank or as a batch in a single beaker.

7.9 Clean the test and control coupons in the candidate cleaning agent by the manufacturer's procedure or selected procedure.

7.9.1 Prepare the cleaning agent in accordance with the manufacturer's recommendations.

7.9.2 Select beakers of suitable size to accommodate the test coupons and fit the beaker holder.

7.9.3 Wash the beakers thoroughly with a solution of liquid, surface-active cleaning agent in hot water and rinse with type II water.

7.9.4 Fill the beakers with the cleaning agent solution to a level that will ensure the test coupons are submerged.

7.9.5 Fill the cleaning tank to its operating level with the transfer fluid and preheat to desired test temperature.

7.9.6 Place the beakers in the beaker holder in the tank so that the liquid levels in the tank and beakers are approximately equal.

7.9.7 Allow the temperatures of the tank fluid and cleaning agent in the beakers to equilibrate at the desired temperature.

7.9.8 Suspend the test coupons and control coupon in the cleaning agent, using a wire hook of the same material as the coupon or a compatible material. Position the coupons such that they do not touch the beaker or one another.

7.9.9 Begin agitation or sonication in the cleaning process and start the timer.

7.9.10 Upon completing the required cleaning time, discontinue the agitation or sonication, and remove the coupons from the cleaning agent.

7.9.11 Rinse the test coupon in accordance with the manufacturer's recommendations.

7.9.12 Allow the suspended coupons to dry overnight or in a forced convection oven for one hour.

7.9.13 Determine the final mass of each test coupon (record- ing them as *MX*3 as previously defined), including the control coupon.

8. Calculation

8.1 *Validation of Procedure*—Examine the control coupons to determine whether they lost mass (such as might occur if there was corrosion occurring, if the coupons were dissolving, or if the standard cleaning procedure used prior to contamination had left residue on the coupons); gained mass (such as might occur if the solution was plating a material on their surfaces, or was depositing contaminant rather than removing it) or exhibited the same mass. The simplest valid test procedure is one in which there is no change in the control- coupon's mass to within the measurement error of the balance.

8.1.1 If the control coupon is designated *MC*, and, if | *MC*3− *MC*1 | < balance error, then the experiment is valid. Proceed to calculate a cleaning effectiveness factor.

8.1.2 If | *MC*3− *MC*1 | is greater than the balance error, the test may be considered to be suspect and the reason for the mass change should be investigated.

8.2 Cleaning Effectiveness Factor (CEF):

8.2.1 The cleaning effectiveness factor indicates the fractional contaminant that was removed during cleaning (for example, *CEF* = 0.9 indicates that 90 % of the contaminant was removed).

$$CEF = \frac{Mx2 - Mx3}{Mx2 - Mx1}$$

where:

MX2 − MX3 = the mass of contaminant removed, and
MX2 − MX1 = the mass of contaminant applied.

8.2.2 Calculate the *CEF* for each test coupon.

8.2.3 Calculate the average *CEF* by arithmetic mean.

9. Report

9.1 Because of the many variables involved in conducting a cleaning test program, it is necessary that all data be carefully documented.

9.2 Report the following information, as applicable:

9.2.1 Date of test,

9.2.2 Technician,

9.2.3 Contaminant identification, and

9.2.4 Coupon Data Refer to Practice G121*:*

9.2.4.1 Identification number of each coupon,

9.2.4.2 Material,

9.2.4.3 Surface roughness, (*RA)* micrometers, and

9.2.4.4 Coupon Contaminated surface areas.

9.2.5 Cleaning Data:

9.2.5.1 Cleaning agent identification,

9.2.5.2 Concentration of cleaning agent,

9.2.5.3 pH of diluted cleaning agent,

9.2.5.4 Ultrasonic, soak, or agitation,

9.2.5.5 Time,

9.2.5.6 Temperature,

9.2.5.7 Level of ultrasonic frequency (kHz), and

9.2.5.8 Power density in watts per liter.

9.2.6 Rinsing Data:

9.2.6.1 Agent,

9.2.6.2 Time,

9.2.6.3

9.2.6.4

9.3

9.3.6.1 Temperature,

9.3.6.2 Number of rinses, and

9.3.6.3 Agitation method (if any).

9.2.7 Drying Data:

9.2.7.1 Method,

9.2.7.2 Time, and

9.2.7.3 Temperature.

9.2.8 Test Data:

9.2.8.1 Initial mass of each coupon, including control coupon, *MX*1 and *MC*1,

9.2.8.2 Mass of each coupon with contaminant, *MX*2,

9.2.8.3 Mass of each cleaned coupon after cleaning, *MX*3, and

9.2.8.4 Mass of control coupon after cleaning, MC3.

9.2.8.5 Report | *MC*3− *MC*1 | and give comparison to balance error.

9.2.9 Report (*MX*2− *MX*3) and (*MX*2− *MX*1) and *CEF f*or each test coupon.

9.2.9.1 Report average *CEF*.

10. Precision and Bias

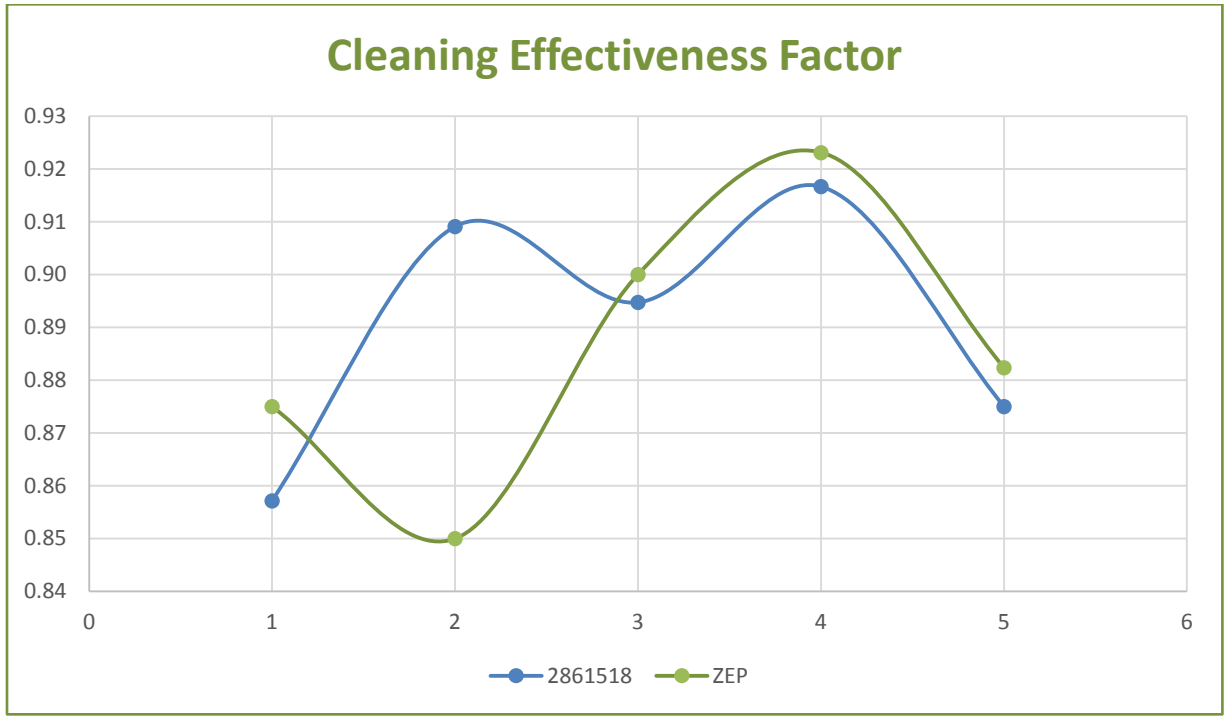
10.1.1 The terms repeatability limit and reproducibility limit in Table 1 are used as specified in Practice E177.

10.2 *Bias*—since there is no accepted reference material suitable for determining the bias for the procedure for measuring the CEF in this test method, bias has not been determined.

11. Conclusion

11.1.1.1 EcoCompounds formula number 2861518 (Rig Wash, Industrial Floor) when compared to ZEP (Product No.E0516228B1) in on par in terms of Cleaning Effectiveness Factor (CEF) as detailed in our lab data below:

9.2.2	Technician	FF	FF	FF	FF	FF	FF	FF	FF	FF	FF
9.2.3	Oil Contaminant (CO) Crude Oil	co	co	co	co	co	co	co	co	co	co
9.2.4.1	Coupon Number	2016-31	2016-32	2016-33	2016-34	2016-35	2016-41	2016-42	2016-43	2016-44	2016-45
9.2.4.2	Material (Glass) G	G	G	G	G	G	G	G	G	G	G
9.2.4.3	Surface Roughness R_A NM	2	2	2	2	2	2	2	2	2	2
9.2.5	Cleaning Data										
9.2.5.1	CLEANING AGENT	2861518	2861518	2861518	2861518	2861518	ZEP	ZEP	ZEP	ZEP	ZEP
9.2.5.2	Concentration of agent	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3	0.3
9.2.5.3	pH of diluted agent	6	6	6	6	6	6	6	6	6	6
9.2.5.4	(U,S,A) Ultra, Soak, Agitation	A	A	A	A	A	A	A	A	A	A
9.2.5.5	Time (Min) 10	7:10	7:10	7:20	7:20	7:30	6:00	6:10	6:20	6:30	6:40
9.2.5.6	Temperature (F)	78	78	78	78	78	78	78	78	78	78
9.2.6	Rinsing Data N/A	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a	n/a
9.2.7	Drying Data										
9.2.7.1	Method (A) air dry	A	A	A	A	A	A	A	A	A	A
9.2.7.2	Time (hrs)	24	24	24	24	24	24	24	24	24	24
9.2.7.3	Temperature (F) =Ambient	78	78	78	78	78	78	78	78	78	78
9.2.8	Test Data										
9.2.8.1	Coupon Standard Wht. Gms MX1 and MC1	5.236	5.136	5.204	5.121	5.173	5.213	5.195	5.215	5.230	5.232
9.2.8.2	Coupon Mass w/Contaminant MX2	5.243	5.147	5.223	5.133	5.181	5.229	5.208	5.225	5.243	5.249
9.2.8.3	Coupon Mass cleaned MX3	5.237	5.137	5.206	5.122	5.174	5.215	5.197	5.216	5.231	5.234
9.2.8.4	Control Coupon mass MC3	5.089	5.089	5.089	5.089	5.089	5.089	5.089	5.089	5.089	5.089
9.2.8.5	Report MC3 - MC1	-0.1	0.0	-0.1	0.0	-0.1	-0.1	-0.1	-0.1	-0.1	-0.1
9.2.8.6	Report MX2 - MX3	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9.2.8.6.1	Report MX2 - MX1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
9.2.8.6.2	Cleaning Effectiveness Factor (CEF)	0.86	0.91	0.89	0.92	0.87	0.88	0.85	0.90	0.92	0.88
9.2.8.7	Average CEF	0.89					0.89				


Cleaning Effectiveness Factor
0.93
0.92
0.91
0.90
0.89
0.88
0.87
0.86
0.85
0.84
0
1
2
3
4
5
6
2861518
ZEP

PROCEDURE FOR IDENTIFICATION OF THE TEST SYSTEM

Capco Analytical Services maintains Standard Operating Procedures (SOPs) relative to efficacy testing studies. Efficacy testing is performed in strict adherence to these SOPs which have been constructed to cover all aspects of the work including, but not limited to, receipt, log-in, and tracking receipt and use of chemical reagents. These procedures are designed to document each step of efficacy testing studies.

METHOD FOR CONTROL OF BIAS: *N/A*

STUDY ACCEPTANCE CRITERIA

Control Acceptance Criteria

The study controls must perform according to the criteria detailed in the study controls description section and in accordance with ASTM 121 and ASTM 122.

PROTOCOL CHANGES

If it becomes necessary to make changes in the approved protocol, the revision and reasons for change will be documented, reported to the Sponsor and will become a part of the permanent file for that study. Similarly, the Sponsor will be notified as soon as possible whenever an event occurs that may have an effect on the validity of the study.

Standard operating procedures used in this study will be the current effective revision at the time of the work. Any minor changes to SOPs (for this study) or methods used will be documented in the raw data and approved by the Study Director.

PRODUCT DISPOSITION

It is the responsibility of the Sponsor to retain a sample of the test substance(s). All unused test substance will be discarded following study completion unless otherwise requested by Sponsor.

RECORD RETENTION

Study Specific Documents

All of the original raw data developed exclusively for this study shall be archived at Capco Analytical Services. These original data include, but are not limited to the following:

1. All handwritten raw data for control and test substances including, but not limited to, notebooks data forms and calculations.
2. Any protocol amendments/deviation notifications.

3. All measured data used in formulating the final report.
4. Memoranda, specifications, and other study specific correspondence relating to interpretation and evaluation of data, other than those documents contained in the final study report.
5. Original signed protocol.
6. Certified copy of final study report.
7. Study specific SOP deviations made during the study.

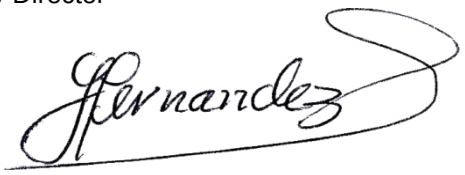
Good Laboratory Practice Compliance Statement

This study meets the U.S. Environmental Protection Agency's good laboratory practice standards and requirements for 40 CFR § 160 with the following exception:

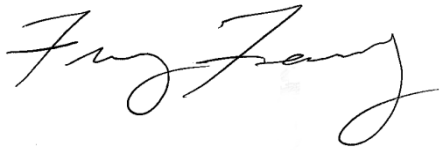
- Records concerning test substance characteristics (i.e., composition, purity, stability, strength, solubility) are maintained by the study sponsor
- Analysis concerning test substance characteristics (i.e., uniformity, solubility stability , etc.) after mixture with dilute were not conducted.

Study Director

Company: Capco Analytical
Name: Franz Fernandez, Ph.D.
Title: Study Director

Signature:  Study Completion Date: 03/10/2016

Company: Capco Analytical
Name: Franz Fernandez Jr.
Title: Laboratory Technician

Signature:  Study Completion Date03/10/2017