Test Method L-14415

TEST METHOD FOR MULTIMETAL CORROSION INHIBITORS (ENGINE APPLICATIONS)

This test is based on the ASTM Standard D 1384-93 titled : " Standard Test Method for Corrosion Test for Engine Coolants in Glassware ". The purpose of this modification of this test is to study the efficiency of liquid corrosion inhibitors formulated to protect multimetal systems found in engines.

CONTENTS

I - SCOPE

This test method allows the evaluation of a corrosion inhibitor efficiency in the system in which it will be introduced. By the way, it will be possible to determine the minimum dosage of inhibitor for a good protection, in various liquids used in engines : fuels, lubricants, coolants, In consequence, this test shall be performed with the concerned system in the specific test conditions inherent to this one.

The values are stated in International Systems units and are to be regarded as the test.

II- REFERENCED DOCUMENTS

Referenced documents come from following ASTM Standards :

- D 1384-93 : Standard Test Method for Corrosion Test for Engine Coolants in Glassware.
- D 1176 : Test Method for Sampling and Preparing Aqueous Solutions of Engine Coolants or Antirusts for Testing Purposes.
- G 1 : Practice for Preparing, Cleaning and Evaluating Corrosion Test Specimens.

III- SUMMARY OF TEST METHOD

In this test method, the three specimens of metal chosen can reproduce the kind of phenomenon which happens in engines, that is to say a galvanic corrosion due to the presence in the same corrosive middle of different types of metal.

Specimens are totally immersed in the system to be tested, continuously aerated and maintained to a specific temperature (it depends on the system, from 40 to 90°C). The test duration is 336 h.

The corrosion inhibitive properties are evaluated on the basis of the weight changes incurred by the specimens. Each is run in duplicate at least, and the average weight change is determined for each metal. For each tested corrosion inhibitor, the procedure shall provide a control-test without inhibitor.

IV- OTHER APPLICATIONS

By this test, it is possible to determine the effectiveness of corrosion inhibitors in simple-metal systems, as production lines or storage plants.

V- APPARATUS. (fig.1)

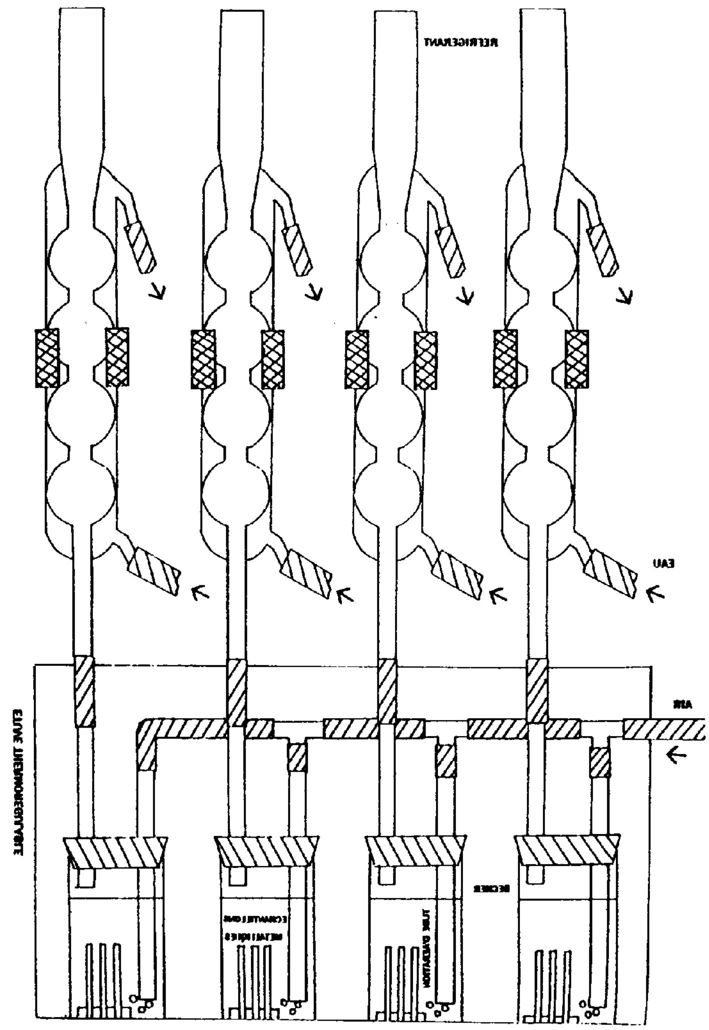

FIGURE 1 : ASSEMBLY

- Heater : an oven with a thermoregulation and a temperature control system, with a volume of 15 litters ;

- Container : a 150 ml, tall-form, spotless beaker, made of heat-resistant glass, 95 mm high, diameter 54 mm, for containing the test solution and specimens. The beaker shall be tightly closed with a rubber stopper, having drill holes to accommodate a water condenser and an aerator tube as shown in figure 1.

- Condenser : a water condenser of the reflux, glass tube type, having a 200 mm condenser jacket with a diameter of 38 mm.

- Aerator tube : a system of continue air circulation with an air pump, and with rate regulation.

VI - METAL TEST SPECIMENS

Metal test specimens used in this test are as follows :

- Steel : of the ETA60 type, certified by the supplier.
Size : 40 x 20 mm, thickness : 3 mm.
Chemical composition of the carbon steel is as follows :
C : 0,17 à 0,23 %
Mn : 0,30 à 0,60 %
P : 0,040 % maximum
S : 0,050 % maximum

- Copper : 99,9 % pure, certified by the supplier. Size : 40 x 20 mm, thickness : 3 mm.

- Aluminium : of the 6060 type, certified by the supplier. Size : 40 x 20 mm, thickness : 3 mm.

Other types of metal are used in engine composants. It is possible to perform this test with desired types of specimens if they are more representative to a specific middle.

VII - METAL SPECIMENS ARRANGEMENT. (fig. 2)

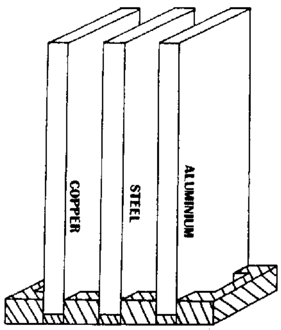

FIGURE 2 : SPECIMENS ARRANGEMENT

Metal specimens are put on heat-resistant polymeric supports. They shall be separated by a space of 5 mm, in the order shown in figure 2. The assembly is placed in the beaker and is totally covered by the test solution.

VIII - TEST SOLUTION

The liquid to be tested shall be mixed with the proper quantity of corrosive water to give a 33 1/3 % volume test solution.
The corrosion inhibitor is added with a the required dosage. Four tests can be run at the same time, one of them will be the control-test (without inhibitor) and the other ones will contain different dosages of inhibitor.
The corrosive water shall contain 150 ppm each of :
- sulfate
- chlorure
- bicarbonate

introduced as sodium salts. The specified corrosive water can be prepared by dissolving the following amounts of anhydrous salts in 1 l of distilled water :
- sodium sulfate : 222 mg
- sodium chloride : 247,5 mg
- sodium bicarbonate : 207 mg.

IX - TEST CONDITIONS

Assembly :

The arrangement of the assembled metal specimens with relation to the aerator tube and other components is shown in figure 1.

Test Temperature :

The required temperature depends on the system to be tested. It is determined in relation to the boiling point of the system. For example, if the studied system is a low-boiling point coolant, this temperature will be 71 °C (ASTM 1384-93).

Aeration Rate :

The aeration rate shall be 30 +/- 5 ml/min. The aerator tube should be located at least 1 cm away from the test assembly to avoid direct contact with the metal specimens.

Test Duration :

The test shall be run continuously for 2 weeks (336 h).

X - PREPARATION OF TEST SPECIMENS

Sand the steel and the aluminium specimens on the cut surfaces with a N°60 grade dry sanding paper. Remove any burrs from coupon edges with a N°320 grade Silicium Carbide sanding paper. Use then a moistened bristle brush.
The copper specimen is scrubbed vigorously, using a moistened bristle brush and pumice until the entire metal area is bright, shiny, and free from any visible oxide film or tarnish.
Rinse the specimens thoroughly with tap water ; then, rinse with acetone, dry, and weigh to the nearest 1 mg.

XI - PROCEDURE

The assembly allows to run four tests at the same time, one of them is the control-test. The test procedure is as follows :

1. Carefully clean the test beaker, condenser, rubber stopper, and aerator tube, and thoroughly rinse with water.

2. After the preparation of metal specimens following the previous procedure, and after weighing them to the nearest 1 mg, bolt them together as shown in figure 2. Place this arrangement in the beaker.

3. Pour 110 g of the prepared test solution into each beaker.

4. Fit the condenser and aeration tube to the beaker, and set the aeration rate to the required value.

5. Close tightly the oven and raise the temperature at the required value. Pass water through the condenser at a rate sufficient to maintain adequate cooling.

6. Check the tests once a day to ensure proper solution temperature, aeration rate, and solution level. The tests may operate unattented on weekends and holidays. Make up evaporation losses during the corrosion tests by addition of distilled water.

7. At the end of the test, immediately disassemble specimens and brush very lightly with a soft bristle brush and water to remove loosely held corrosion products. To remove the more tenacious corrosion products and films, the individual specimens shall then be subjected to additional cleaning treatments detailed below.

8. Weigh the treated specimens to the nearest 1 mg.

XII - POST-OPERATIVE ADDITIONNAL CLEANING TREATMENTS OF METAL SPECIMENS

Steel treatment :
Use a brass scraper or brass bristle brush, followed by scrubbing with a wet Silicium Carbide sanding paper, N°320 grade, and a wet bristle brush and pumice.

Aluminium treatment :
Dip for 5 min in a water solution containing 10 volume % of orthophosphoric acid (H3PO4 75%) maintained at 80 °C. Rinse with water, then brush very lightly with a soft bristle brush to remove any loose films, and again rinse with water. If a film remains, immerse for 1 min in concentrated nitric acid and repeat the previous step.

Copper treatment :
Dip in a 1 + 1 mixture of concentrated HCl and water for 15 s to remove tarnish films, rinse with tap water to remove acid, and scrub with a wet bristle brush and pumice.

Follow each of these operations noted above by thorough rinsing, first in tap water and then in acetone. Then dry and weigh the specimens to the nearest 1 mg.

It is better to determine cleaning losses obtained on an untested set of metal specimens. Deduct the average cleaning losses from gross weight differences to determine actual corrosion losses.

XIII - DETERMINATION OF CLEANING LOSSES FACTORS

Each untested metal specimen are weighed to the nearest 1 mg, and treated following the post-operative cleaning procedure. They are then weighed to the nearest 1 mg. The difference between both values is the cleaning loss factor fcl. The actual weight change of metal specimen is:

Actual weight change = mesured weight change - fcl

XIV - REPORT

The corrected metal weight changes are stated in mg for a given test duration. To make easy comparisons, all test duration shall be the same. The corrected value found for each metal, each inhibitor dosage, each test number will be reported as follows :

Metal	Weight changes per specimens in mg-Test duration : t				
	Test N°	Control-test	X1% in inhibitor	X2% in inhibitor	X3% in inhibitor
Copper	1				
	2				
	3				
Aluminium	1				
	2				
	3				
Steel	1				
	2				
	3				

A single weight change that appears completely out of line shall be dealt.

DETERMINATION OF CLEANING LOSSES FACTORS FOR COPPER, STEEL AND ALUMINIUM

A – DEFINITION

The cleaning losses factors fcl are the weight changes of metal specimen due to the post-operative additional cleaning treatments described in the "Test Method For Multimetal Corrosion Inhibitors (Engine Application)".

B- PROCEDURE

The metal specimens used are described in the part VI of the test method. Each untested metal specimen is weighed to the nearest 1 mg, and treated following the post-operative cleaning procedure. They are then weighed to the nearest 1 mg. The difference between both values is the cleaning losses factor f_{cl} .

C- RESULTS

Five trials per metal specimen have been performed. Results are given in the following table :

Cleaning losses factors in mg			
Trial	Copper	Aluminium	Steel
1	4	7	2
2	4	7	3
3	3	7	2
4	4	6	2
5	3	7	3

We can determine an average value of the cleaning losses factors :

- Copper : f_{cl} = 4 +/- 1 mg
- Aluminium : f_{cl} = 7 +/- 1 mg
- Steel : f_{cl} = 2 +/- 1 mg

D - USE

These values of fcl can be used to determine actual weight changes in every test report where metal specimens and post-operative cleaning treatments are those described in the test method.

actual weight changes = measured weight changes - f_{cl}