

Chapter 1. Uniform Corrosion

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Chapter 1. Uniform Corrosion

General Description

Test specimens (coupons) of each alloy to be tested, are engraved with a unique identification code, measured to determine dimensions, cleaned to remove grease and oxidation films, rinsed in distilled water and dried. Each coupon is weighed and immersed in a test solution in glass jars with screw caps and placed in an incubator at the test temperature, undisturbed for 90 days. The coupons are rinsed to remove residual test solution and loose corrosion products, cleaned with the appropriate solution and dried. Each coupon is weighed and the change in weight during immersion is used to determine the corrosion rate.

Preparation of Fire Chemical Products

Except for tests specifically requiring aged or stored chemicals, all test solutions should be freshly prepared, usually 24 hours before use. Fire chemical products are prepared by stirring the wet components (when testing the component) or by adding the wet or dry components to water (when testing mixed retardant) according to manufacturer's directions.

The viscosity, refractometer values, density, and pH are measured and compared to the manufacturer's submission information.

If retardant quality control values are acceptable, the retardant is measured into one-quart capacity glass jars. Each jar contains 800 milliliters of test product for a total immersion test or 400 milliliters of test product for a partial immersion test. See figure 5.1.



Figure 5.1—The retardant is measured.

Coupon Description and Preparation

Coupons of four alloys (2024-T3 aluminum, 4130 steel, yellow brass, and Az31B magnesium) are used for evaluation against the Forest Service wildland fire chemical specifications. See figure 5.2. Coupons are purchased from a commercial source (Corrosion Test Supply, French Settlement, Louisiana). Each coupon is nominally 1 inch by 4 inches by 1/8 inch (2.5 cm by 10.2 cm by 0.3 cm) with a hole (13/64 inch [0.5 cm] in diameter), centered, 1/2 inch (1.3 cm) from one end of the coupon. All coupons are purchased unmarked and are stored in the original wrappings.

In the laboratory, each coupon is marked, above the hole, with a unique identification code using a vibrating engraver.

Each coupon is then measured, to the nearest 0.001 cm, in each of the 3 dimensions. The width and thickness are measured about 1 inch (2.5 cm) from the end that does not have the hole.

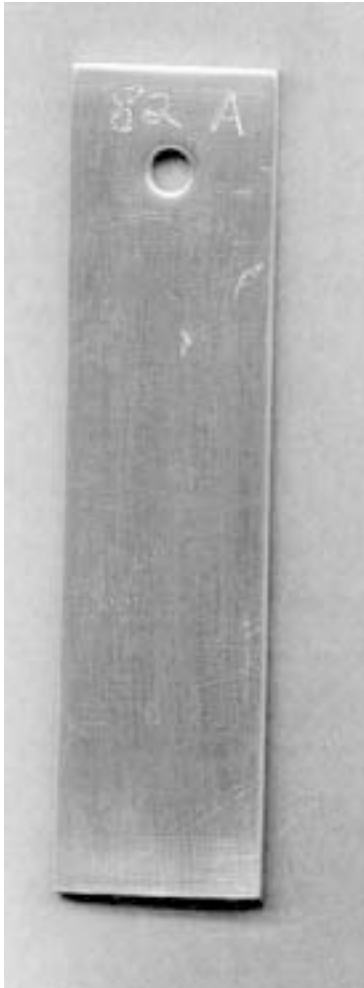


Figure 5.2—Coupon.

Coupon Degreasing and Initial Cleaning

Scotch-Brite scouring pads are cut into strips about 1 inch by 3 inches (2.5 cm by 7.6 cm) and conditioned to remove excess abrasive by scrubbing a clean coupon reserved for that purpose. The conditioning coupon is the same alloy as the test coupons to be cleaned.

Coupons are degreased by scrubbing with a Scotch-Brite scouring pad and Formula 409

cleaner. They are then rinsed under cold tap water and cleaned in the appropriate solution shown in table 5.1.

Table 5.1—Cleaning Procedures Table

| ALLOY | CHEMICAL ¹ | MINUTES | TEMP | REMARKS |
|-----------|---|---------|------------------|--|
| Aluminum | 70% HNO ₃ (concentrated) | 2-3 | Room | Follow with light scrub using non-metallic brush or scrubbing pad ² . |
| Brass | 15-20% HCl (half strength) | 2-3 | Room | Follow with light scrub using non-metallic brush or scrubbing pad. |
| Steel | 50g SnCl ₂ + 20g SbCl ₃ in 1000 mL of concentrated HCl | 3-5 | Cold ice bath | Follow with light scrub using non-metallic brush or scrubbing pad. |
| Magnesium | 15g CrO ₃ + 1g AgCrO ₄ in 84 mL of distilled H ₂ O | 15 | Boiling | Follow with light scrub using non-metallic brush or scrubbing pad. |

¹ Cleaning solutions should be discarded as they become used or discolored. If in doubt, replace it. When cleaning exposed coupons, special care is needed to prevent erroneous results and in the case of the magnesium solution, fresh chemical should be used for each coupon.

² If corrosion film resists cleaning by this procedure alternate with a solution of 2g CrO₃ + 5g H_3PO_4 in 93 mL of distilled water heated to 175-185 °F (79-85 °C) for 10 minutes.

Each batch of six coupons (aluminum, mild steel, or brass) is immersed in the specified cleaning solution shown in table 5.1 in a 600-milliliter plastic beaker. The coupons are arranged, leaning against the inside of the beaker, so that the coupons do not touch each other.

Each magnesium coupon is immersed in cleaning solution in an 8-inch (20 cm) test tube. Several test tubes are placed in a 1-liter beaker, with about 4 inches (10 cm) of water and glass beads or boiling chips, and the water heated to boiling on a hot plate.

Following required immersion in the cleaning solution, the coupons are removed from the solution and scrubbed with a fresh, conditioned scouring pad. The six coupons in a batch are scrubbed with same scouring pad. The pad is then used for degreasing new coupons of the same alloy or discarded.

**Each scouring pad is used for
only one alloy.**

Clean coupons are rinsed in distilled water, surface water removed by wiping with a lint free towel, and the coupons suspended in a drying oven warmed to 125 °F (52 °C). Do not dry in the same oven (incubator) used to maintain temperature for uniform corrosion testing. The released chemical vapors may adversely affect the final corrosion rates.

**Clean coupons must be handled with
tongs or gloved hands only.**

Dry coupons are removed from the oven, allowed to cool for about 15 minutes, and then weighed to the nearest tenth of a milligram. Coupons should be immersed in test solution soon after cleaning.

Coupon Immersion

A strip of fiberglass-reinforced tape is attached across the opening of the jar and half way down each side.

A length of braided dacron fishing line, about 16 inches long, is attached to each coupon by doubling the fishing line and running the loop through the hole in the coupon and then running the cut ends through the loop. A 15- to 20-pound-test fishing line is firm enough for easy handling but not so firm as to make it difficult of tie.

The dacron line with the attached coupon is twisted over the center of the tape so that the coupon is suspended near the center of the jar. The loose ends of the fishing line are pulled over the side of the jar and held in place with tape. See figure 5.3.

One coupon is immersed in the test solution in each jar. Coupons for total immersion tests are positioned so that they are completely covered with solution but not touching the bottom of the jar. Coupons for partial immersion tests are positioned so that the lower 2 inches of the coupon are immersed in the retardant. A small grease pencil tic mark on the edge of the coupon indicates the proper immersion level. See figure 5.4.

The pulp-lined, Bakelite lid is screwed firmly (hand tight) into place on the jar. Each jar is labeled with the name and lot number

of the test product, the identification of the coupon immersed in the product, and the date of the immersion.



Figure 5.3—Coupon in retardant solution.



Figure 5.4—Coupons in solution.

The jars are then placed into preheated incubators at the proper temperature, 70 °F or 120 °F (21 °C or 49 °C), and left undisturbed during the 90-day exposure period. See figure 5.5.



Figure 5.5—Jars in the incubator.

End of the Test Exposure

At the end of the test period, the jars are removed from the incubators, the jars and contents inspected, and the lids removed. Notes are made of any cracked jars, evaporated test solution, or coupons that are destroyed or have pieces missing.

The test solution is poured from the jar and discarded.

Final Coupon Cleaning

Each coupon is rinsed with a forceful stream of cold tap water to remove residual test solution and loose corrosion products. The coupons are then allowed to air dry while still suspended from the dacron fishing line. After the coupons are dry, usually overnight, the fishing line is removed. See figure 5.6.



Figure 5.6—Test solution jars with coupons.

After the coupons are dried, any built-up corrosion products are removed from the coupons by scraping with the flat edge of a stainless steel spatula. In some cases the corrosion inhibitor film must be gently chipped or peeled from the coupon, once more using the flat edge of a spatula. After the corrosion products and inhibitors are removed the coupons are cleaned in chemical solutions.

Each batch of coupons, no more than six, is cleaned along with a control coupon that is cleaned in the same manner as the test coupons. The aluminum, mild steel, and brass coupons are immersed in the cleaning solution, leaning against the sides of the container, a 600-milliliter plastic beaker, in such a way that the coupons do not touch each other.

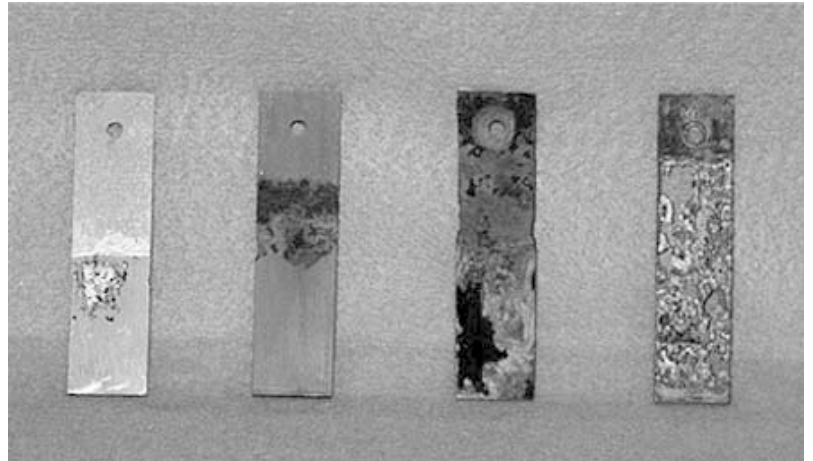


Figure 5.7—Final coupons.

Following soaking as specified table 5.1, each coupon is removed from the solution and scrubbed with a fresh, conditioned scouring pad. Clean coupons are rinsed in distilled water, wiped with a lint free towel, and suspended in the drying oven as previously described. See figure 5.7.

Care must be taken to scrub and handle each coupon in the same way. Magnesium coupons are immersed in individual, 8-inch (200 mm) test tubes containing the cleaning solution. Several test tubes are then placed in a 1-liter beaker containing water and glass beads or boiling chips. The contents of the beaker are heated on a hot plate. Coupons are scrubbed, rinsed, and dried as described previously. Since corrosion to magnesium coupons often makes the identification marks difficult to read, care must be taken to maintain the identity of each coupon during the cleaning and drying processes.

If persistent residues remain, the coupons may be cleaned a second time, again using a control coupon to determine the weight lost during cleaning. After drying the coupons are cooled and then weighed to the nearest 0.001 milligram.

Control Coupons

Control coupons reserved for this purpose are not exposed to any of the test solutions. The control coupons are cleaned and weighed

before use; cleaned with a set of exposed coupons; and then weighed again in order to determine the weight loss of the coupon that should be attributed to the cleaning method rather than the exposure to test solution. Each control coupon must be weighed before and after cleaning.

Calculations of Corrosion Rate

The corrosion rate is calculated for each coupon using the initial and final weights, control weight loss, coupon surface area, and density of the alloy and exposure time in the formula.

$$Cr = \frac{534 [(Wt_i - Wt_f) - (Wt_{ci} - Wt_{cf})]}{A t d}$$

Where:

Wt_i = coupon initial weight, mg

Wt_f = coupon final weight, mg

Wt_{ci} = control coupon initial weight, mg

Wt_{cf} = control coupon final weight, mg

A = surface area of the coupon, in²

t = exposure time, hr

d = density of the alloy, g/cm³

2.77 g/cm³ for 2024-T3 aluminum

7.86 g/cm³ for 4130 steel

8.53 g/cm³ for yellow brass

1.77 g/cm³ for Az31B magnesium

Replicates

Three replicates will be performed for most tests on fresh material; however, magnesium corrosion tests performed for performance information will not be replicated.

Two replicates will be performed on most test materials following one-year storage tests. Six replicates will be performed on the partial immersion tests with mild steel and high temperature aluminum.

Manufacturers may request that additional replicates be included, at their expense, for any test condition.

Rejection of Individual Tests

After all tests are completed the results are calculated. Spurious results may be rejected if there is supporting information (based on the inspection of individual tests) or statistical basis for the action, i.e., the average without a value is more than three standard deviations from the average with the value included.

Averaging of Results

The results of all tests (excluding those rejected as described above) with the same alloy, temperature, and immersion conditions will be averaged to determine the final corrosion rate.

At the request of the supplier, additional replicates may be performed if quantities of test material allow. The results of **all of the tests** (except those rejected as described above) for the same alloy, temperature, and immersion conditions will be included in the final average.

Reporting of Results

The results of all tests, including those rejected, and the average will be reported in writing to the manufacturer. The reasons for including or rejecting a suspect value will be included in the report.

Chapter 2. Intergranular Corrosion Test

Mixed retardant shall be tested for intergranular corrosion. At least one coupon for each exposure and temperature from the uniform corrosion tests on the specified alloys shall be sliced as shown in figure 5.8. The coupon will be mounted, polished to 0.3-micron alumina finish, and etched with Keller's reagent using standard metallurgical techniques.

Coupon Examination

The coupon will then be examined microscopically with a magnification of 500. Any attack of a metal at the grain boundary in the exposed uniform corrosion alloy coupon specimens shall not be acceptable.

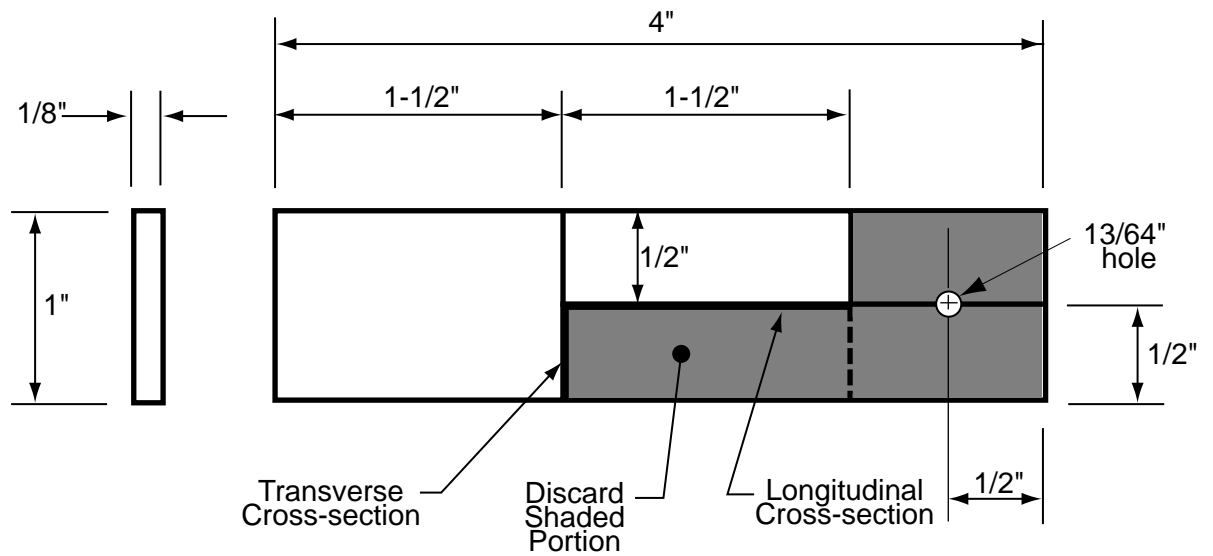


Figure 5.8—Coupon drawing from specification.