METHOD 1110A

CORROSIVITY TOWARD STEEL

1.0   SCOPE AND APPLICATION

    1.1   This method measures the corrosivity toward steel of both aqueous and nonaqueous liquid wastes.

2.0   SUMMARY OF METHOD

    2.1   This test exposes coupons of SAE Type 1020 steel to the liquid waste to be evaluated and, by measuring the degree to which the coupon has been dissolved, determines the corrosivity of the waste.

3.0   INTERFERENCES

    3.1   In laboratory tests, such as this one, corrosion of duplicate coupons is usually reproducible to within 10%. However, large differences in corrosion rates may occasionally occur under conditions where the metal surfaces become passivated. Therefore, at least duplicate determinations of corrosion rate should be made.

4.0   APPARATUS AND MATERIALS

    4.1   An apparatus should be used, consisting of a kettle or flask of suitable size (usually 500 to 5,000 mL), a reflux condenser, a thermowell and temperature regulating device, a heating device (mantle, hot plate, or bath), and a specimen support system. A typical resin flask set up for this type of test is shown in Figure 1.

    4.2   The supporting device and container shall be constructed of materials that are not affected by, or cause contamination of, the waste under test.

    4.3   The method of supporting the coupons will vary with the apparatus used for conducting the test, but it should be designed to insulate the coupons from each other physically and electrically and to insulate the coupons from any metallic container or other device used in the test. Some common support materials include glass, fluorocarbon, or coated metal.

    4.4   The shape and form of the coupon support should ensure free contact with the waste.

    4.5   A circular specimen of SAE 1020 steel of about 3.75 cm (1.5 in.) diameter is a convenient shape for a coupon. With a thickness of approximately 0.32 cm (0.125 in.) and a 0.80-cm (0.4-in.)-diameter hole for mounting, these specimens will readily pass through a 45/50 ground-glass joint of a distillation kettle. The total surface area of a circular specimen is given by the following equation:

$$A ' \frac{3.14 \ (D^2 \ \& \ d^2)}{2} \ \% \ (t)(3.14)(D) \ \% \ (t)(3.14)(d)$$

where:

    t = thickness.
    D = diameter of the specimen.
    d = diameter of the mounting hole.

If the hole is completely covered by the mounting support, the last term in the equation, (t)(3.14)(d), is omitted.

      4.5.1   All coupons should be measured carefully to permit accurate calculation of the exposed areas. An area calculation accurate to ± 1% is usually adequate.

      4.5.2   More uniform results may be expected if a substantial layer of metal is removed from the coupons prior to testing the corrosivity of the waste. This can be accomplished by chemical treatment (pickling), by electrolytic removal, or by grinding with a coarse abrasive. At least 0.254 mm (0.0001 in.) or 2-3 mg/cm^2 should be removed. Final surface treatment should include finishing with #120 abrasive paper or cloth. Final cleaning consists of scrubbing with bleach-free scouring powder, followed by rinsing in distilled water and then in acetone or methanol, and finally by air-drying. After final cleaning, the coupon should be stored in a desiccator until used.

      4.5.3   The minimum ratio of volume of waste to area of the metal coupon to be used in this test is 40 mL/cm^2.

5.0 REAGENTS

    5.1   Sodium hydroxide (NaOH), 20%: Dissolve 200 g NaOH in 800 mL Type II water and mix well.

    5.2   Zinc dust.

    5.3   Hydrochloric acid (HCl): Concentrated.

    5.4   Stannous chloride (SnCl$_2$).

    5.5   Antimony chloride (SbCl$_3$).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

This method does not provide sample collection, preservation, and handling guidelines.

7.0 PROCEDURE

    7.1   Assemble the test apparatus as described in Sec. 4.0, above.

    7.2   Fill the container with the appropriate amount of waste.

7.3   Begin agitation at a rate sufficient to ensure that the liquid is kept well mixed and homogeneous.

7.4   Using the heating device, bring the temperature of the waste to 55 EC (130 EF).

7.5   An accurate rate of corrosion is not required; only a determination as to whether the rate of corrosion is less than or greater than 6.35 mm per year is required. A 24-hr test period should be ample to determine whether or not the rate of corrosion is > 6.35 mm per year.

7.6   In order to determine accurately the amount of material lost to corrosion, the coupons have to be cleaned after immersion and prior to weighing. The cleaning procedure should remove all products of corrosion while removing a minimum of sound metal. Cleaning methods can be divided into three general categories: mechanical, chemical, and electrolytic.

7.6.1   Mechanical cleaning includes scrubbing, scraping, brushing, and ultrasonic procedures. Scrubbing with a bristle brush and mild abrasive is the most popular of these methods. The others are used in cases of heavy corrosion as a first step in removing heavily encrusted corrosion products prior to scrubbing. Care should be taken to avoid removing sound metal.

7.6.2   Chemical cleaning implies the removal of material from the surface of the coupon by dissolution in an appropriate solvent. Solvents such as acetone, dichloromethane, and alcohol are used to remove oil, grease, or resinous materials and are used prior to immersion to remove the products of corrosion. Solutions suitable for removing corrosion from the steel coupon are:

Solution	Soaking Time	Temperature
20% NaOH + 200 g/L zinc dust	5 min	Boiling
Conc. HCl + 50 g/L $SnCl_2$ + 20 g/L $SbCl_3$	Until clean	Cold

7.6.3   Electrolytic cleaning should be preceded by scrubbing to remove loosely adhering corrosion products. One method of electrolytic cleaning that can be employed uses:

Solution: 50 g/L H_2SO_4

Anode: Carbon or lead

Cathode: Steel coupon

Cathode current density: 20 amp/cm^2 (129 amp/in.^{2})

Inhibitor: 2 cc organic inhibitor/liter

Temperature: 74 EC (165 EF)

Exposure Period: 3 min.

NOTE: Precautions must be taken to ensure good electrical contact with the coupon to avoid contamination of the cleaning solution with easily reducible metal ions and to ensure that inhibitor decomposition has not occurred. Instead of a proprietary inhibitor, 0.5 g/L of either diorthotolyl thiourea or quinolin ethiodide can be used.

7.7   Whatever treatment is employed to clean the coupons, its effect in removing sound metal should be determined by using a blank (i.e., a coupon that has not been exposed to the waste). The blank should be cleaned along with the test coupon and its waste loss subtracted from that calculated for the test coupons.

7.8   After corroded specimens have been cleaned and dried, they are reweighed. The weight loss is employed as the principal measure of corrosion. Use of weight loss as a measure of corrosion requires making the assumption that all weight loss has been due to generalized corrosion and not localized pitting. In order to determine the corrosion rate for the purpose of this regulation, the following formula is used:

Corrosion Rate (mmpy) = (weight loss x 87.60)/(area x time x metal density) (Reference 1), then,

$$\text{Corrosion Rate (mmpy)} = \frac{\text{weight loss x 11.145}}{\text{area x time}}$$

where: weight loss is in milligrams,
              area is in square centimeters,
              time is in hours,
              metal density of SAE type 1020 steel = 7.86 g/cm^3, and
              corrosion rate is in millimeters per year (mmpy).

8.0   QUALITY CONTROL

8.1   All quality control data should be filed and available for auditing.

8.2   Duplicate samples should be analyzed on a routine basis.

9.0   METHOD PERFORMANCE

9.1   No data provided.

10.0 REFERENCES

1.   National Association of Corrosion Engineers, "Laboratory Corrosion Testing of Metals for the Process Industries," NACE Standard TM 01 69 (1972 Revision), NACE, 3400 West Loop South, Houston, TX 77027.
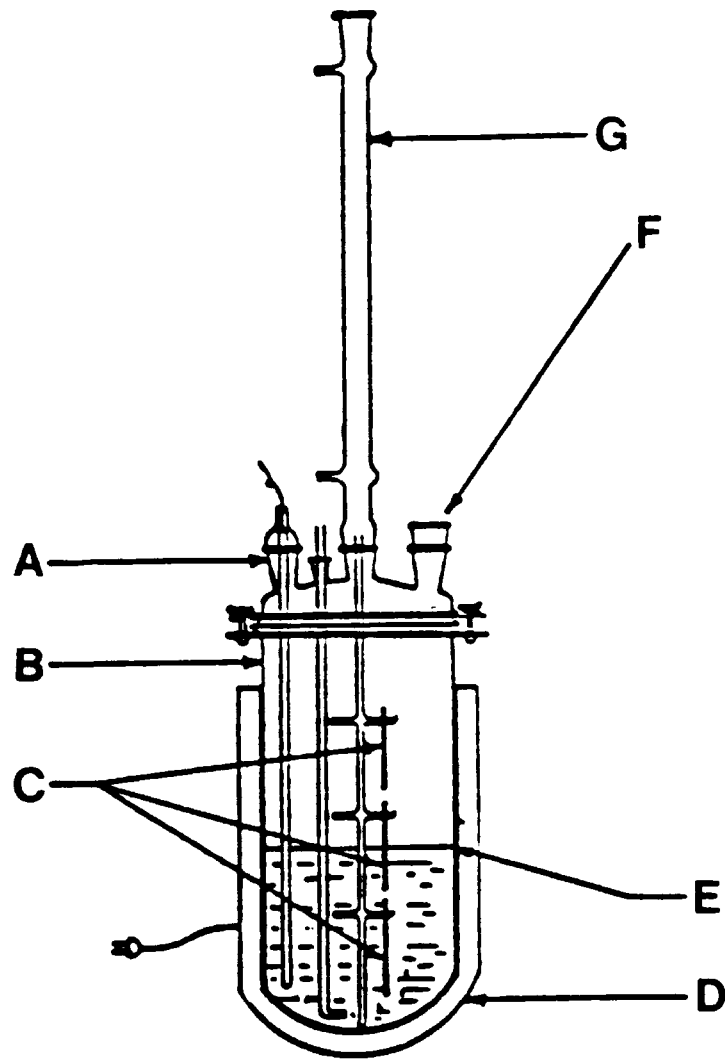
Figure 1. Typical resin flask that can be used as a versatile and convenient apparatus to conduct simple immersion tests. Configuration of the flask top is such that more sophisticated apparatus can be added as required by the specific test being conducted. A = thermowell, B = resin flask, C = specimens hung on supporting device, D = heating mantle, E = liquid interface, F = opening in flask for additional apparatus that may be required, and G = reflux condenser.

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    │  Start  │
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┌──────────────────┐
│   7.1 Assemble   │
│  test apparatus. │
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│ 7.2 Fill container│
│   with waste.    │
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         ▼
┌──────────────────┐
│   7.3 Agitate.   │
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         ▼
┌──────────────────┐
│   7.4 Heat.      │
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         │
         ▼
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       ( A )
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┌──────────────────┐
│ 7.6 Clean coupons│
│  by mechanical,  │
│ chemical, and/or │
│   electrolytic   │
│    methods.      │
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│ 7.7 Check effect │
│   of cleaning    │
│  treatment on    │
│ removing sound   │
│     metal.       │
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┌──────────────────┐
│  7.8 Determine   │
│ corrosion rate.  │
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    ┌─────────┐
    │  Stop   │
    └─────────┘
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