Designation: F 862 – 93 (Reapproved 2002)

Standard Test Method for pH and Chloride-ion Concentration of Aerospace Hydraulic Fluids[1]

This standard is issued under the fixed designation F 862; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the measurement of the pH and chloride ion of water extraction of aerospace hydraulic fluids.

1.2 *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.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

3. Summary of Method

3.1 The aerospace hydraulic fluid sample is shaken with water, and the pH and the quantity of chloride ion are determined from the water layer.

3.1.1 The pH is measured using a glass electrode and pH meter.

3.1.2 The quantity of the chloride ion is determined using a silver/sulfide electrode and a specific ion meter.

4. Significance and Use

4.1 On application of the hydraulic fluid within the mechanical fluidic system, the fluid may become contaminated with acid and chloride ion. Mechanical shearing of the hydraulic fluid in the presence of the minute quantity of water and residual amount of organic solvents, used in cleaning, may initiate formation of acid and chloride ion. Measurements are desired to control and maintain the cleanliness and noncorrosiveness of the fluidic system.

5. Apparatus

5.1 *pH Meter*, with expanded millivolt scale and a sensitivity of 1 mV. The method can be adopted for use with a selective-ion meter.[3]

5.2 *Glass Electrode*—The pH response shall be ±0.05 pH.

5.3 *Reference Electrode*—A calomel, silver/silver chloride, or other reference electrode of constant potential.

5.4 *Silver/Silver Sulfide Electrode.* [4]

5.5 *Double Junction Reference Electrode*, sleeve-type.[5]

5.6 *Separatory Funnel*, 250 mL.

5.7 *Buret*, 10-mL capacity.

5.8 *Titration Stand*, preferably built as an integral part of the meter housing and provided with support for the electrodes and electrical stimer, all connected to ground.

5.9 *Gran's Plot Paper.* [6]

6. Reagents

6.1 *Water*, reagent grade, Type IV, in accordance with Specification D 1193.

6.2 *Reference Buffer Solutions,* Standards 185, 186, and 187—Materials supplied by the National Bureau of Standards with pH values.

6.3 *Silver Nitrate Solution, Standard* (2.82×10^{-3} *M*)—Dissolve 4.7909 g of silver nitrate dissolved in 1 L of water and diluted 1:10.

7. Standardization of pH Meter

7.1 Turn on the instrument, allow it to warm up thoroughly, and bring it to electrical balance in accordance with the manufacturer's instructions. Wash the glass and reference electrodes with three changes of water. Form a fresh liquid junction if a sleeve-type reference junction is used.

7.2 Fill the sample cup with the first reference buffer solution and immerse the electrodes. If the pH meter is of the zero-null type, set the dial of the meter to equal pH value of the reference buffer solution. Engage the operating button and rotate the standardization knob until the meter is brought to balance. If the pH meter is of the direct-reading type, engage the operating button, turn the range switch to the proper position, and rotate the asymmetry-potential knob until the reading corresponds to the pH of the reference buffer. Empty the sample cup and repeat until two successive readings are obtained without adjustment of the controls.

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[1] This test method is under the jurisdiction of ASTM Committee F07 on Aerospace and Aircraft and is the direct responsibility of Subcommittee F07.07 on Qualification Testing of Aircraft Cleaning Materials.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] The Orion Model 901 has been found satisfactory.

[4] The Orion Research Model has been found satisfactory.

[5] The Orion Model 90-01 has been found satisfactory.

[6] Gran's plot paper is available from Orion Research, Inc., 380 Putnam Ave., Cambridge, MA 02139.

7.3 Wash the electrodes and the sample cup three times with water. Place the second reference buffer solution in the sample cup and measure the pH, either by simply reading the dial or by adjusting the balance point, depending on the type of pH meter. Adjust the slope control until the reading corresponds to the temperature corrected value of the second reference buffer solution. Use additional portions of the second reference buffer solution, as in 7.2.

7.4 The assembly is operating satisfactorily if the pH reading obtained for the second reference buffer solution agrees with its assigned pH value within 0.05 units.

PART A—pH MEASUREMENT

8. Procedure

8.1 Add a 50-mL sample of hydraulic fluid and 50 mL of reagent water to a 250-mL separatory funnel.

8.2 Mix thoroughly. Allow for the water and oil layers to separate completely.

8.3 Drain the water layer from the funnel into a 100-mL beaker.

8.4 Standardize the pH-meter assembly with two reference buffer solutions as described in 7.1-7.4. Wash the electrodes with three changes of water. Equip the beaker with a small laboratory-type mechanical stirrer having a glass agitator or with a magnetic stirring unit having an inert, plastic-covered impeller. Insert the electrodes and determine the pH value; measure the water sample until two readings 30 s apart do not differ by more than 0.05-pH unit. Record the pH values.

9. Calculation

9.1 The pH of the sample is the average of the last two readings.

10. Report

10.1 Report the pH of the water extraction of hydraulic fluid to the nearest 0.1 unit.

11. Precision and Bias

11.1 A program to establish the precision and bias of this test method will be conducted at a later date.

PART B—CHLORIDE-ION CONCENTRATION MEASUREMENT

12. Procedure

12.1 Add a 100-mL sample of hydraulic fluid and 100 mL of reagent water to a 250-mL separatory funnel.

12.2 Mix thoroughly. Allow for the water and oil layers to separate completely.

12.3 Drain the water layer from the funnel into a 250-mL beaker.

12.4 Equip the beaker with a small laboratory-type mechanical stirrer having a glass agitator or with a magnetic stirring unit having an inert, plastic-covered impeller. Immerse the double-junction reference electrode and the silver/silver sulfide electrode in the solution.

12.5 Add 5.0 mL of silver nitrate standard solution from a 10-mL buret. Record millivolt reading when stable.

12.6 Add 1.0-mL increments of the silver nitrate standard solution and record the millivolt reading when stable after each addition until a total of five have been added.

13. Calculation

13.1 On Gran's plot paper, each major division on the vertical axis shall correspond to 5 mV. The millilitre value shall be plotted on the horizontal axis in accordance with the whole integers on the paper.

13.2 Calculate the chloride-ion concentration.

13.2.1 Plot on the Gran's plot paper the millilitres of standard silver nitrate solution required for titration along the horizontal axis of the plot paper and the corresponding millivolt readings along the vertical axis.

13.2.2 Draw a line through all points and extend to intersect the horizontal baseline. The point at which the plotted line intersects the baseline is equivalent to the numerical quantity of chloride ion in the oil samples in parts per million.

14. Report

14.1 Report the chloride-ion concentration to the nearest whole integer.

15. Precision and Bias

15.1 A program to establish the precision and bias data will be conducted at a later date.

16. Keywords

16.1 chloride ion; hydraulic fluid; pH