

TEST FOR PHOSPHATE CONTAMINATION OF HIGH STRENGTH SCREWS

Application

This method is used to inspect the surfaces of high strength screws for P_2O_5 contamination, which results in brittleness.

Note: This method is based on the reduction of molybdate-phosphate with ascorbic acid producing a blue complex (molybdenum blue) which is stable at $23 \pm 2^\circ C$ for about four hours.

Apparatus and Reagents Required

1. **Test Solution** - Ammonium-molybdate/Sulphuric acid

Preparation: Dissolve 10 g $(NH_4)_6 Mo_7 O_{24} \cdot 4 H_2O$ A.R. (ammonium-molybdate) in distilled water and make up to 100 mL. Add the solution to 300 mL of H_2SO_4 (50 % by weight) and cool to $23 \pm 2^\circ C$.

2. **Test Salt** - Ascorbic Acid A.R.

3. 1 N H_2SO_4

4. 1 N NaOH

5. **Standard Solution**

Preparation: Dissolve 0.1917 g KH_2PO_4 A.R. (potassium-dihydrogenphosphate) in 1000 mL of deionized water. This standard solution contains 100 mg/L P_2O_5 .

6. Graduated Cylinders (10 mL, 50 mL, 5 x 100 mL); beakers (250 mL tall form, 400 mL short form); graduated pipettes; 200 mL volumetric flask; lifting tongs or forceps.

Conditioning and Test Conditions

All test values indicated herein are based on material conditioned in a controlled atmosphere of $23 \pm 2^\circ C$ and $50 \pm 5\%$ relative humidity for not less than 24 h prior to testing and tested under the same conditions unless otherwise specified.

Date	Action	Revisions
2001 01 31	Revised	Editorial – no technical change A. Cockman
2000 04 25		Editorial – no technical change A. Cockman
1994 07 22		

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Procedure

1. Measure 25 mL of 1 N H_2SO_4 into a 400 mL beaker.
2. Immerse a sufficient number of screws to give a test surface area of 15 cm^2 in the 1 N H_2SO_4 for about 10 seconds at $23 \pm 2^\circ\text{C}$.
3. Add 25 mL of 1 N NaOH to neutralize the solution.
4. Take the screws out of the solution using forceps or lifting tongs allowing the solution to drop off the screws.
5. Pipette 8 mL of test solution (ammonium-molybdate/sulphuric acid) into the neutralized solution and add 0.2 g of test salt (ascorbic acid).
6. Shake the beaker for a short time, and then heat the solution to boiling for about 1 minute.
7. Cool the solution and transfer it to a 100 mL graduated cylinder.
8. Compare the developed blue coloring of the solution with the color of the reference solutions which are prepared as described below.

Note: If this method indicates more than 0.1 mg P_2O_5 on the surface of the screws (15 cm^2), it must be assumed that the screws have not been sufficiently cleaned to remove phosphate contamination.

Preparation of the Reference Solutions

1. Pipette 20 mL of the standard solution (potassium-dihydrogenphosphate solution) into a graduated 200 mL flask.
2. Dilute the solution with about 100 mL of distilled water.
3. Add 8 mL of the test solution (ammonium-molybdate/sulphuric acid) using a graduated pipette, and then add about 0.2 g of the test salt (ascorbic acid).
4. Shake the volumetric flask for a short time and then heat the solution and hold at boiling point for about 1 minute. (The color of the solution turns to blue.)
5. Cool the solution and fill the volumetric flask with distilled water up to the standard mark.