

ILION TEST METHOD A7

CHEMICAL AND METALLURGICAL CONTROL LABORATORY

DETERMINATION OF MOLYBDENUM

- PURPOSE:** To determine the presence of molybdenum in steel by spot testing.
- SAFETY:**
1. Label all bottles accurately (POISONOUS, ACID ETC.).
 2. Do not mix free KSCN with HCl HNO₃ solution. The fumes released are poisonous.
 3. Do not breathe directly the fumes of the acid reaction on steel.
 4. If any of the solutions contact the skin, wash freely with soap and water.
- PRINCIPLE:** The steel to be tested is electrographically dissolved using hydrochloric (HCl) and nitric acids (HNO₃) as the electrolyte. Potassium thiocyanate (KSCN) reacts with the molybdates to give a yellow-colored compound, and with the ferric ions to produce a red complex precipitate. Upon treatment with stannous chloride (SnCl₂) the yellow compound is reduced to a burning-pink potassium molybdate cyanide (K₂Mo(CNS)₆) and the red ferric complex is reduced to colorless ferrous ions. 83
- STATUS:** This test has been only used in the laboratory, but could readily be adapted for plant use.
- APPARATUS:**
1. Batteries - 2 Eveready #6, 1½ volts each.
 2. Wires - 4 lengths, silver-coated copper wire.
 3. Filter paper - Qualitative grade, 7.0 cm.
 4. Bottles - 4 1/4 oz. screw-top bottles.
- REAGENTS:**
1. Nitric Acid - Use conc. 70.0% commercial grade; Sp. Gr. 1.42.
 2. Hydrochloric Acid - Use conc. 37.0% commercial grade; Sp. Gr. 1.19/0.
 3. Potassium Thiocyanate. Dissolve 20 gm. KSCN in 100 ml. distilled water.
 4. Stannous Chloride. Dissolve 32 gm. SnCl₂·2H₂O in 40 ml. conc. HCl and dilute to 100 ml. with distilled water.
- PROCEDURE:**
1. To the cleaned surface of the steel apply one drop HCl and one drop HNO₃.
 2. Dissolve electrographically for one minute.
 - a. Place the two wire ends in the acid drops on the steel and hold for prescribed interval.
 3. Add one drop KSCN.
 4. Absorb spot of solution onto filter paper.
 - a. Be careful of fingers. Do not touch wet portion of paper.