

## D. MONOGRAPHS

### Alginic Acid

[ 9005-32-7 ]

**Content** Alginic Acid, when dried, contains 91.0 - 104.5% of alginic acid.

**Description** Alginic Acid occurs as a white to light yellow filamentous, granular or powder. It has a slight characteristic odor and taste.

**Identification** To 0.25 g of Alginic Acid, add 50 ml of sodium hydroxide solution (4.3 100) and dissolve. Use this solution as the test solution. To 10 ml of the test solution, add 2 ml of calcium chloride solution (2.5 100). A gelatinous precipitate is formed immediately. To 10 ml of the test solution, add 5 ml of ammonium sulfate saturated solution. No precipitate is formed.

**Purity** (1) Specific rotation  $[\alpha]_D^{20} = -80 - -180^\circ$  (0.50 g, sodium hydroxide solution (4.3 100), 100 ml, on the dried basis ).

(2) pH 2.0 - 3.4 ( 3% suspension ).

(3) Sulfate Not more than 0.96% as SO<sub>4</sub>.

**Test Solution** Weigh about 0.10 g of Alginic Acid accurately, dissolve it in 20 ml of sodium hydroxide solution (4.3 100), and neutralize with diluted hydrochloric acid (1 4). Then, add 1 ml of hydrochloric acid, shake well, heat in a water bath for several minutes, cool, and filter. Wash the container three times with 10 ml of water each time, filter the washings through the filter paper used above, combine the filtrates, and add water to make 50 ml. Measure 10 ml of this solution, and add water to make 50 ml.

**Control Solution** To 0.40 ml of 0.005 mol/l sulfuric acid, add 1 ml of diluted hydrochloric acid (1 4) and water to make 50 ml.

(4) Phosphate Weigh 0.10 g of Sodium Alginate accurately, dissolve it 20 ml of sodium hydroxide solution (4.3 100), and neutralize with diluted nitric acid (1 4), and make it uniformly. Cool, add 5 ml of diluted nitric acid (1 4) and 20 ml of Ammonium Molybdate TS, and warm. No yellow precipitate is formed.

(5) Heavy metals Not more than 40 µg/g as Pb (0.50 g, Method 2, Control solution Lead Standard Solution 2.0 ml).

(6) Lead Not more than 10 µg/g as Pb (1.0 g, Method 1).

(7) Arsenic Not more than 4.0 µg/g as As<sub>2</sub>O<sub>3</sub> (0.50 g, Method 3, Apparatus B).

**Loss on Drying** Not more than 15.0% (105 °C, 4 hours).

**Residue on Ignition** Not more than 10.0% (calculated on the dried basis).

**Microbial Limits** Proceed as directed under the Microbial Limit Tests. The total viable aerobic count is not more than 5,000/g and no *Escherichia coli* is observed.

**Assay** (1) Apparatus The apparatus is outlined in the figure given in the



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acid (1      120) and several pieces of zeolite, and connect the flask to reflux condenser F with moistening its spherical ground-glass joint with a little amount of phosphoric acid. Raise the mercury in mercury valve B about 5 cm into its tube by feeding air with a pressure from three-way stop cock M, shut the cock, and confirm for a few minutes that the top of the mercury raised above does not descend. Flow air-free carbon dioxide into the apparatus with suction at the flow rate of about 3 - 6 L per one hour, boil gently for 2 minutes while heating in mantle heater E, and allow to cool for 15 minutes. Place 23 ml of hydrochloric acid in drop tube G, add exactly 25 ml of 0.25 mol/l sodium hydroxide immediately after removing absorption tube J from the apparatus, add 5 drops of *n*-butanol, and connect again absorption tube J to the apparatus. Flow air-free carbon dioxide into the apparatus with suction at the flow rate of about 2 L per one hour, transfer the hydrochloric acid placed in drop tube G into reaction flask D, and boil the Alginic Acid placed in the flask for 3 hours while heating with mantle heater E. Then, stop heating and sucking, and transfer slowly the 0.25 mol/l sodium hydroxide solution placed in absorption tube J into conical flask K through three-way stop cock M using compressed air. Remove flask K from the apparatus, add 10 ml of barium chloride solution (1      10), stopper, shake gently for about 2 minutes, add 2 drops of phenolphthalein TS, and titrate with 0.1 mol/l hydrochloric acid. Perform a blank test in the same manner.

1 ml of 0.1 mol/l hydrochloric acid = 25.00 mg of Alginic Acid