

## B. GENERAL TESTS

### Coloring Matter Aluminum Lake Tests

The Coloring Matter Aluminum Lake Tests are used for the purity tests and assays of Coloring Matter Aluminum Lakes.

#### 1. Hydrochloric Acid- and Ammonia-Insoluble Substances

Hereinafter in the Monographs, such a specification as "not more than 0.5 % (Coloring Matter Aluminum Lake Tests)" indicates that when determined as directed in the following procedure, the content of the hydrochloric acid- and ammonia-insoluble substance is not more than 0.5 %.

**Procedure** Dry a crucible type glass filter (IG4) at 135 °C for 30 minutes, allow to cool in a desiccator, and weigh it accurately.

Weigh accurately about 2 g of a sample, mix with 20 ml of water, add 20 ml of hydrochloric acid, and stir well, add 300 ml of boiling water, and shake well. Cover with a watch glass, and heat on a water bath for 30 minutes, cool, and filter the supernatant with the glass filter prepared above. Transfer the insoluble substances in the container into the filter with about 30 ml of water, wash twice the insoluble substances in the container and on the filter with 5 ml of water each time, further wash the insoluble substances on the glass filter with 1% ammonia solution until the washings become almost colorless, and wash with 10 ml of diluted hydrochloric acid (1 : 35), and then with water until the washings do not respond to silver nitrate solution (1 : 50). Dry the insoluble substances together with the glass filter at 135 °C for 3 hours, allow to cool in a desiccator, and weigh it accurately.

#### 2. Iodide

Hereinafter in the Monographs, such a specification as "not more than 0.20 % (Coloring Matter Aluminium Lake Tests)" indicates that when determined as directed in the following procedure, the content of sodium iodide is not more than 0.20 %.

**Procedure** Weigh accurately about 60 mg of a sample, add 10 ml of water exactly measured, shake occasionally for about 30 minutes, and filter through a dry filter paper, and use the filtrate as the test solution. Measure 0.5 ml, 1 ml, 10 ml, and 50 ml of Iodide Ion Standard Stock Solution, add water to make exactly 100 ml each, and use these solutions as the standard solutions. Proceed as directed under Ion Chromatography under the following operating conditions, using 100 µl of the test solution, the standard solutions and the standard stock solution. Determine the peak heights or peak areas of the iodide ions of the standard solutions and the standard stock solution to make the calibration curve. Determine the peak height and peak area of the iodide ion of the test solution, and obtain the iodide ion content using the calibration curve. Multiply the iodide ion content by 1.18 to obtain the concentration of the sodium iodide of the test solution, and thus determine its content in the sample.

## B. GENERAL TESTS

Avoid direct sunlight during the procedure. Use a light-resistant container for the preparation of the test solution, and perform the test immediately after its preparation.

### *Operating Conditions*

Detector: Conductometer.

Packing material of column: Porous anion exchanger.

Column: Stainless or plastic tube (4.6 - 6.0 mm in internal diameter, 5 - 10 cm in length).

Guard Column: A column with the same internal diameter and the same packing material as the above column.

Eluant: A solution including 2.5 mmol/l phthalic acid and 2.4 mmol/l tris(hydroxymethyl) amino methane (pH 4.0).

Temperature: 40 .

Flow rate: 1.5 ml/min.

### 3. Heavy Metals

Hereinafter in the Monographs, such a specification as “ not more than 50  $\mu\text{g/g}$  as Zn (Coloring Matter Aluminum Lake Tests, Heavy Metals (1)) ” indicates that when determined as directed in the following procedure (1), the content of heavy metals is not more than 50  $\mu\text{g/g}$  as Zn.

**Procedure** Weigh 2.5 g of a sample, place in a quartz or porcelain crucible, moisten with a little quantity of sulfuric acid, heat gradually and almost incinerate at a temperature as low as possible, cool, add again 1 ml of sulfuric acid, and heat gradually until the white fumes of sulfuric acid are no longer evolved. Place in an electric furnace, ignite at 450 - 550 to incinerate, and cool. Add 5 ml of hydrochloric acid and 1 ml of nitric acid, crush the lumps thoroughly, and evaporate to dryness on a water bath. Add again 5 ml of hydrochloric acid, crush the lumps, and evaporate to dryness on a water bath. Add 10 ml of diluted hydrochloric acid (1 4) to the residue, dissolve by heating, cool, and filter through a filter paper (5-C) for quantitative analysis. Wash the residue on the filter paper with about 30 ml of diluted hydrochloric acid (1 4), combine the filtrate and the washings, and evaporate to dryness on a water bath. Then add 10 ml of diluted hydrochloric acid (1 4) to the residue, dissolve by heating, cool, and filter. Wash the crucible and the residue on the filter paper with 5 ml of diluted hydrochloric acid (1 4) and with 5 ml of water, combine the filtrate and the washings, and add water to make 50 ml. Use this solution as the sample solution. Prepare the blank test solution in the same manner as the sample solution.

#### (1) Zinc

**Test Solution** Measure 10.0 ml of the sample solution, add 10 ml of diluted hydrochloric acid (1 4), add water to make 50 ml.

**Control Solution** Measure 10.0 ml of the blank test solution, add 2.5 ml of Zinc

## B. GENERAL TESTS

Standard Solution and 10 ml of diluted hydrochloric acid (1 : 4) and water to make 50 ml.

For the test solution and the control solution, proceed as directed under Atomic Absorption Spectrophotometry under the following operating conditions. The absorbance of the test solution does not exceed that of the control solution.

### *Operating Conditions*

Light Source: Zinc hollow cathode lamp.

Wavelength: 213.9 nm.

Supporting gas: Air.

Combustible gas: Acetylene.

### (2) Iron

*Test Solution* Measure 4.0 ml of the sample solution, add 10 ml of diluted hydrochloric acid (1 : 4) and water to make 50 ml.

*Control Solution* Measure 4.0 ml of the blank solution, add 5 ml of Iron Standard Solution, 10 ml of diluted hydrochloric acid (1 : 4) and water to make 50 ml.

For the test solution and the control solution, proceed as directed under Atomic Absorption Spectrophotometry under the following operating conditions. The absorbance of the test solution does not exceed that of the control solution.

### *Operating Conditions*

Light source: Iron hollow cathode lamp.

Wavelength: 248.3 nm.

Supporting gas: Air.

Combustible gas: Acetylene.

### (3) Other heavy metals

*Test Solution* Measure 20 ml of the sample solution, place into a Nessler tube, adjust its pH to about 4 with ammonium acetate solution (1 : 10), add water to make 50 ml.

*Control Solution* Measure 20 ml of the blank test solution and 2.0 ml of Lead Standard Solution, place into a Nessler tube, and prepare the control solution in the same manner as the test solution.

Add 2 drops of sodium sulfide TS to each solution, shake well and allow to stand for 5 minutes. The color of the test solution is not darker than that of the control solution.

## 4. Barium

Hereinafter in the Monographs, such a specification as "not more than 500 µg/g as Ba (Coloring Matter Aluminum Lake Tests)" indicates that when determined as directed in the following procedure, the content of barium is not more than 500 µg/g as Ba.

### Procedure

## B. GENERAL TESTS

*Test Solution* Weigh accurately about 1 g of a sample, place into a platinum crucible, moisten with a little quantity of sulfuric acid, heat gradually until the sample is almost incinerated at a temperature as low as possible. Cool, add again 1 ml of sulfuric acid, and heat gradually until the white fumes of sulfuric acid are no longer evolved. Ignite the residue at 450 - 550 °C for 3 hours in an electric furnace, and cool. Add 5 g of anhydrous sodium carbonate to the residue, mix well, cover the crucible with a lid, and fuse by heating. Continue heating for 10 minutes, cool, add 20 ml of water, and heat on a water bath until the fused substance is dissolved. Cool, filter through a filter paper (5C) for quantitative analysis, and wash the residue on the filter paper with water until the washings are free from sulfate. Then transfer the residue together with the filter paper into a beaker, add 30 ml of diluted hydrochloric acid (1 : 4), shake well, boil, and cool. Filter the content, wash the residue on the filter paper with 10 ml of water, combine the filtrate and the washings, and evaporate to dryness on a water bath. Dissolve the residue with 5 ml of water, filter if necessary, add 0.25 ml of diluted hydrochloric acid (1 : 4), and mix well. Add water to make 25 ml.

*Control Solution* To 0.5 ml of Barium Standard Solution, add 0.25 ml of diluted hydrochloric acid (1 : 20) and water to make 25 ml.

Proceed tests for the test solution and control solution as directed under Inductively Coupled Plasma-Atomic Emission Spectrometry. The emission intensity of the test solution is not stronger than that of the control solution.

### 5. Arsenic

Hereinafter in the Monographs, such a specification as “ not more than 4.0 µg/g as As<sub>2</sub>O<sub>3</sub> (Coloring Matter Aluminum Lake tests) ” indicates that when determined as directed in the following procedure, the content of arsenic as As<sub>2</sub>O<sub>3</sub> is not more than 4.0 µg/g.

#### Procedure

*Test Solution* Weigh 0.50 g of a sample, place into a quartz or porcelain crucible, add 20 ml of a solution of magnesium nitrate in ethanol (1 : 10), burn by igniting the ethanol, and heat gradually to incinerate at 450 - 550 °C. If carbonized material still remains, moisten with a small quantity of nitric acid, and ignite again at 450 - 550 °C to incinerate. Cool, add 6 ml of hydrochloric acid to the residue, add about 10 ml of water if necessary, and dissolve by heating on a water bath. Cool, add water to make 25 ml.

*Control Solution* To 2.0 ml of Arsenic Standard Solution, add water to make 25 ml.

Perform tests for the test solution and control solution by the method using Apparatus C as directed under the Arsenic Limit Test. The absorbance of the test solution is not more than that of the control solution.

### 6. Other Coloring Matter Lakes

## B. GENERAL TESTS

Hereinafter in the Monographs, such a specification as “ Coloring Matter Aluminum Lake tests, Other Coloring Matter Lakes (1) ” indicates the test is to be performed as directed in (1) of the following.

**Procedure** (1) Weigh the amount of the sample corresponding to 0.10 g as a coloring matter, add 60 ml of diluted acetic acid (1 : 3), heat to boil, and cool. Add acetone to make 100 ml, mix well, and use its supernatant as the test solution. Measure 2  $\mu$ l of the test solution, and without any control solutions, proceed as directed under Paper Chromatography using a mixture of *n*-butanol, 1% ammonia solution, and absolute ethanol (6 : 3 : 2) as a developing solvent. Use a filter paper (No. 2) for paper chromatography. When the developing solvent ascends about 15 cm, stop the development, air-dry the filter paper, place on a white plate, and observe from above under daylight. Only one spot is observed.

(2) Proceed as directed in (1), using 1% ammonia solution instead of diluted acetic acid (1 : 3) and a mixture of 25 % ethanol and 5 % ammonia solution (1 : 1) as a developing solvent.

(3) Weigh the amount of the sample corresponding to 50 mg as a coloring matter, and proceed as directed in (1).

(4) Proceed as directed in (1), using diluted acetic acid (1 : 20) instead of diluted acetic acid (1 : 3).

### 7. Assay

(1) Weigh accurately the specified quantity of a sample, transfer into a 500-ml wide-mouthed Erlenmeyer flask, and add 20 ml of diluted sulfuric acid (1 : 20). Shake well, add 50 ml of boiling water, and dissolve by heating. Add 150 ml of boiling water and 15 g of sodium citrate. While passing carbon dioxide through this solution and boiling this solution vigorously, titrate with 0.1 mol/l titanium trichloride until the proper color of the sample disappears.

(2) Proceed as directed in (1), using 15 g of sodium hydrogen tartrate instead of sodium citrate.

(3) Proceed as directed in (1), using 15 g of sodium hydrogen tartrate instead of sodium citrate. In this case, use 10 ml of Light Green SF Yellow solution (1 : 1,000) as the indicator, perform the blank test in the same manner as the test solution, and make any necessary correction.