Ion Exchange Resins

Definition Ion Exchange Resins occur as granules, powders, and suspensions called Ion Exchange Resin (granule), Ion Exchange Resin (powder), and Ion Exchange Resin (suspension), respectively.

Ion Exchange Resin (granule)

Description Ion Exchange Resin (granule) occurs as a black, brown, light red-brown, or white, spherical, massive, or granular substance. It is almost odorless.

Identification Perform the following tests (I) and (II) to identify whether the resin is cation exchange resin or anion exchange resin.

(Ⅰ) Cation exchange resin Make a resin column pouring 5 ml of Ion Exchange Resin (granule) with water into a glass tube for chromatography (about 1 cm in internal diameter). Flow 25 ml of diluted hydrochloric acid (1 → 10) at a rate of about 5 ml per minute, and wash by flowing 100 ml of water at the same rate. Flow 25 ml of potassium hydroxide solution (1 → 15) at the same rate, and wash again by flowing 75 ml of water at the same rate. To 5 ml of the last washings, add 2 ml of diluted acetic acid (1 → 20), and add 3 drops of sodium cobaltinitrite TS. No yellow turbidity appears. Transfer 2 ml of the resin of the resin column into a test tube, add 5 ml of diluted hydrochloric acid (1 → 10), shake well for 5 minutes, filter. Wash the resin on the filter paper with water, and combine the filtrate and the washings to make about 5 ml. Add 4 ml of sodium hydroxide solution (1 → 25) to the solution, shake, add 2 ml of diluted acetic acid (1 → 20), and add 3 drops of sodium cobaltinitrite TS. A yellow precipitate is formed.

(Ⅱ) Anion exchange resin Make a resin column pouring 5 ml of Ion Exchange Resin (granule) with water into a glass tube for chromatography (about 1 cm in internal diameter). Flow 25 ml of diluted hydrochloric acid (1 → 10) at a rate of about 5 ml per minute, and wash by flowing 100 ml of water at the same rate. To 5 ml of the last washings, add 1 ml of diluted nitric acid (1 → 10), and add 3 drops of silver nitrate solution (1 → 50). No white turbidity appears. Transfer 1 ml of the resin of the resin column into a test tube, add 3 ml of sodium hydroxide solution (1 → 25), shake well for 5 minutes, and filter. Wash the resin on the filter paper with water, and combine the filtrate and the washings to make about 5 ml. Add 3 ml of diluted nitric acid (1 → 10) to

the solution, and add 3 drops of silver nitrate solution (1 → 50). A white precipitate is formed.

Purity Prepare the sample of the cation exchange resin or the anion exchange resin by (I) or (II) as appropriate, given below, immerse thoroughly in water, and blot the adhering water with a filter paper, and use as sample A.

(I) Cation exchange resin Prepare the sample (H form) as follows: Measure 30 ml of Ion Exchange Resin (granule), transfer into a glass tube for chromatography (about 3 cm in internal diameter), flow 1,000 ml of diluted hydrochloric acid (1 → 10) at a rate of 15—20 ml per minute, and wash by flowing water at the same rate. Measure 10 ml of the washings, and perform the test for Chloride. Wash with water until the amount is not more than the amount equivalent to 0.3 ml of 0.01 mol/l hydrochloric acid.

(II) Anion exchange resin Prepare the sample (OH form) as follows: Measure 30 ml of Ion Exchange Resin (granule), transfer into a glass tube for chromatography (about 3 cm in internal diameter), flow 1,000 ml of sodium hydroxide solution (1 → 25) at a rate of 15—20 ml per minute, and wash by flowing water at the same rate. Wash with water until the washings become neutral with phenolphthalein TS.

(1) Solids Not less than 25%.

Weigh 10.0 g of sample A. In the case of the cation exchange resin, dry at 100℃ for 12 hours, and weigh again; in the case of the anion exchange resin, dry at 40℃ for 12 hours in a vacuum desiccator at 4 kPa, and weigh again.

(2) Water-soluble substances Not more than 0.50%.

Weigh 10.0 g of sample A, transfer into a cylindrical filter (28 mm in internal diameter, 100 mm in length), suspend in 1,000 ml of water, and extract for 5 hours while shaking occasionally. Measure 50 ml of the extract, evaporate carefully, and dry at 110℃ for 3 hours. Weigh the amount of the residue. Perform a blank test in the same manner, and make any necessary correction.

(3) Heavy metals Not more than 20 μg/g as Pb (Sample A 1.0 g, Method 2, Control solution Lead Standard Solution 2.0 ml).

(4) Arsenic Not more than 4.0 μg/g as As_2O_3 (Sample A 0.50 g, Method 3, Apparatus B).

Total Ion Exchange Capacity Perform the test for cation exchange resin or anion exchange resin by (I) or (II) as appropriate.

(I) Cation exchange resin Not less than 1.0 milliequivalent/g.

Weigh accurately about 5 g of sample A prepared for the Purity Tests. Add 500 ml of 0.2 mol/l sodium hydroxide, exactly measured, and allow to stand for 12 hours while shaking occasionally. Measure exactly 10 ml of the supernatant, and titrate with

0.05mol/l sulfuric acid (indicator: 3 drops of methyl orange TS). Perform a blank test in the same manner, and calculate the total ion exchange capacity by the formula

$$\text{Total ion exchange capacity} = \frac{\text{Volume (ml) of 0.05mol/l sulfuric acid consumed in the blank test} - \text{Volume (ml) of 0.05mol/l sulfuric acid consumed in this test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 5 \text{ (milliequivalent/g)}$$

(II) Anion exchange resin Not less than 1.0 milliequivalent/g.

Weigh accurately about 5 g of sample A prepared for the Purity Tests. Add 500 ml of 0.2 mol/l hydrochloric acid, exactly measured, and allow to stand for 12 hours while shaking occasionally. Measure exactly 10 ml of the supernatant, and titrate with 0.1 mol/l sodium hydroxide (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same manner, and calculate the total ion exchange capacity by the formula

$$\text{Total ion exchange capacity} = \frac{\text{Volume (ml) of 0.1 mol/l sodium acid consumed in the blank test} - \text{Volume (ml) of 0.1 mol/l sodium acid consumed in this test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 5 \text{ (milliequivalent/g)}$$

Ion Exchange Resin (powder)

Description Ion Exchange Resin (powder) occurs as a black, brown, light red-brown, or white powdery substance. It is almost odorless.

Identification Perform the following tests (I) and (II) to identify whether the resin is cation exchange resin or anion exchange resin.

(I) Cation exchange resin Make a resin layer pouring 2 g of Ion Exchange Resin (powder) with water into a pressure filter (about 7.5 cm in internal diameter) equipped with a membrane filter (1μm in pore diameter). Flow 25 ml of diluted hydrochloric acid (1 → 10) at a rate of about 5 ml per minute, and wash by flowing 100 ml of water at the same rate. Flow 25 ml of potassium hydroxide solution (1 → 15) at the same rate, and wash again by flowing 75 ml of water at the same rate. To 5 ml of the last washings, add

2 ml of diluted acetic acid (1 → 20), and add 3 drops of sodium cobaltinitrite TS. No yellow turbidity appears. Transfer 0.5 g of the resin layer into a test tube, add 5 ml of diluted hydrochloric acid (1 → 10), shake well for 5 minutes, and filter. Wash the resin on the filter paper with water, and combine the filtrate and the washings to make about 5 ml. Add 4 ml of sodium hydroxide solution (1 → 25) to the solution, shake, add 2 ml of diluted acetic acid (1 → 20), and add 3 drops of sodium cobaltinitrite TS. A yellow precipitate is formed.

(II) Anion exchange resin Make a resin layer pouring 2 g of Ion Exchange Resin (powder) with water into a pressure filter (about 7.5 cm in internal diameter) equipped with a membrane filter (1μm in pore diameter). Flow 25 ml of diluted hydrochloric acid (1 → 10) at a rate of about 5 ml per minute, and wash by flowing 100 ml of water at the same rate. To 5 ml of the last washings, add 1 ml of diluted nitric acid (1 → 10), and add 3 drops of silver nitrate solution (1 → 50). No white turbidity appears. Transfer 0.5 g of the resin layer into a test tube, add 3 ml of sodium hydroxide solution (1 → 25), shake well for 5 minutes, filter, wash the resin on the filter paper with water, and combine the filtrate and the washings to make about 5 ml. Add 3 ml of diluted nitric acid (1 → 10) to the solution, and add 3 drops of silver nitrate solution (1 → 50). A white precipitate is formed.

Purity Prepare the sample of the cation exchange resin or the anion exchange resin by (I) or (II) as appropriate, given below, immerse thoroughly in water, and blot the adhering water with a filter paper, and use as sample A.

(I) Cation exchange resin Prepare the sample (H form) as follows: Weigh 30 g of Ion Exchange Resin (powder), transfer into a pressure filter (7.5 cm in internal diameter) equipped with a membrane filter (1 μm in pore diameter), flow 1,000 ml of diluted hydrochloric acid (1 → 10) at a rate of 15—20 ml per minute, and wash by flowing water at the same rate. Measure 10 ml of the washings, and perform the test for Chloride. Wash with water until the amount is not more than the amount equivalent to 0.3 ml of 0.01 mol/l hydrochloric acid.

(II) Anion exchange resin Prepare the sample (OH form) as follows: Weigh 30 g of Ion Exchange Resin (powder), transfer into a pressure filter (about 7.5 cm in internal diameter) equipped with a membrane filter (1 μm in pore diameter), flow 1,000 ml of sodium hydroxide solution (1 → 25) at a rate of 15—20 ml per minute, and wash by flowing water at the same rate. Wash with water until the washings become neutral with phenolphthalein TS.

(1) Solids Not less than 25%.

Proceed as directed under Purity (1) in Ion Exchange Resin (granule).

(2) Water－soluble substances Not more than 0.50%.

Weigh 10.0 g of sample A, suspend by adding 1,000 ml of water, and extract for 5 hours while stirring occasionally. Filter the suspension through a pressure filter (about 7.5 cm in internal diameter) equipped with a membrane filter (1 μm in pore diameter). Measure 50 ml of the filtrate, evaporate carefully, and dry at 110℃ for 3 hours. Weigh the amount of the residue.

(3) Heavy metals Not more than 20 μg/g as Pb (Sample A 1.0 g, Method 2, Control Solution Lead Standard Solution 2.0 ml).

(4) Arsenic Not more than 4.0 μg/g as As_2O_3 (Sample A 0.50 g, Method 3, Apparatus B).

Total Ion Exchange Capacity Perform the test for cation exchange resin on anion exchange resin by (I) or (II) as appropriate.

(I) Cation exchange resin Not less than 1.0 milliequivalent/g.

Weigh accurately about 5 g of sample A prepared for the Purity Tests. Add 500 ml of 0.2 mol/l sodium hydroxide, exactly measured, allow to stand for 12 hours while shaking occasionally. Filter the suspension through a pressure filter (7.5 cm in internal diameter) equipped with membrane filter (1 μm in pore diameter). Measure exactly 10 ml of the filtrate, and titrate with 0.05 mol/l sulfuric acid (indicator: 3 drops of methyl orange TS). Perform a blank test in the same manner, and calculate the total ion capacity by the formula

$$\text{Total ion exchange capacity} = \frac{\text{Volume (ml) of 0.05mol/l sulfuric acid consumed in the blank test} - \text{Volume (ml) of 0.05mol/l sulfuric acid consumed in this test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 5 \text{ (milliequivalent/g)}$$

(II) Anion exchange resin Not less than 1.0 milliequivalent/g.

Weigh accurately about 5 g of sample A prepared for the Purity Tests. Add 500 ml of 0.2 mol/l hydrochloric acid, exactly measured, allow to stand for 12 hours while shaking occasionally. Filter the suspension through a pressure filter (7.5 cm in internal diameter) equipped with membrane filter (1 μm in pore diameter). Measure exactly 10 ml of the filtrate, and titrate with 0.1 mol/l sodium hydroxide (indicator: 3 drops of phenolphthalein TS). Perform a blank test in the same manner, and calculate the total ion capacity by the formula

Total ion exchange capacity

$$= \frac{\text{Volume (ml) of 0.1 mol/l sodium acid consumed in the blank test} - \text{Volume (ml) of 0.1 mol/l sodium acid consumed in this test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 5 \text{ (milliequivalent/g)}$$

Ion Exchange Resin (suspension)

Description Ion Exchange Resin (suspension) is a brown, light red-brown, or white suspension. It is almost odorless.

Identification Perform the following tests (I) and (II) to identify whether the resin is cation exchange resin or anion exchange resin.

(I) Cation exchange resin To 0.5 ml of Ion Exchange Resin (suspension), add 5 ml of water and 1 ml of a strongly acidic cation-exchange resin, react for 1 hour while shaking occasionally, and filter through absorbent cotton on a funnel. To the filtrate, add 0.3 g of sodium chloride, shake for 3 minutes, add 1 drop of methyl red TS, and shake. The color of the solution changes to pink.

(II) Anion exchange resin To 0.5 ml of Ion Exchange Resin (suspension), add 5 ml of water and 1 ml of a strongly basic anion-exchange resin, react for 1 hour while shaking occasionally, and filter through absorbent cotton on a funnel. To the filtrate, add 0.3 g of sodium chloride, shake for 3 minutes, add 1 drop of phenolphthalein TS, and shake. The color of the solution changes to pink.

Purity (1) Solids Not less than 4.0%.

Weigh 1.0 g of Ion Exchange Resin (suspension), dry at 105℃ for 5 hours, and weigh again.

(2) Water—soluble substances Not more than 0.50% w/v.

Measure 100 ml of Ion Exchange Resin (suspension), and filter through a pressure filter (about 7.5 cm in internal diameter) equipped with a membrane filter (0.05 μm in pore diameter). Measure 10 ml of the filtrate, evaporate carefully, and dry at 105℃ for 3 hours. Weigh the amount of the residue is not more than 50 mg.

(3) Heavy metals Not more than 20 μg/g as Pb (1.0 g, Method 2, control solution Lead Standard Solution 2.0 ml).

(4) Arsenic Not more than 4.0 μg/g as As_2O_3 (0.50 g, Method 3, Apparatus B).

Total Ion Exchange Capacity Perform the test for cation exchange resin or anion exchange resin by (I) or (II) as appropriate.

(I) Cation exchange resin Not less than 1.0 milliequivalent/g.

Measure accurately the amount of Ion Exchange Resin (suspension) equivalent to about 0.2 g of the solid, pour it into a glass tube for chromatography (about 1 cm in internal diameter) packed with 10 ml of a strongly acidic cation-exchange resin, flow it at a rate of about 2 ml per minute, and flow about 20 ml of water at the same rate. Wash by flowing about 80 ml of water at a rate of 15—20 ml per minute. Transfer all of the eluate and the washings into a beaker, add about 1 g of sodium chloride, and titrate with 0.1 mol/l sodium hydroxide using a pH meter until the pH becomes 7.0. Perform a blank test in the same manner, make any necessary correction, and calculate the total ion exchange capacity by the formula

Total ion exchange capacity

$$= \frac{\text{Volume (ml) of 0.1 mol/l sulfuric hydroxide consumed in this test} - \text{Volume (ml) of 0.1 mol/l sulfuric hydroxide consumed in the blank test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 0.1 \text{ (milliequivalent / g)}$$

(II) Anion exchange resin Not less than 1.0 milliequivalent/g.

Measure accurately the amount of Ion Exchange Resin (suspension) equivalent to about 0.2 g of the solid, pour it into a glass tube for chromatography (about 1 cm in internal diameter) packed with 10 ml of a strongly basic anion-exchange resin, flow it at a rate of about 2 ml per minute, and flow about 20 ml of water at the same rate. Wash by flowing about 80 ml of water at a rate of 15—20 ml per minute. Transfer all of the eluate and the washings into a beaker, add about 1 g of sodium chloride, and titrate with 0.1 mol/l hydrochloric acid using a pH meter until the pH becomes 7.0. Perform a blank test in the same manner, make any necessary correction, and calculate the total ion exchange capacity by the formula

Total ion exchange capacity

$$= \frac{\text{Volume (ml) of 0.1 mol/l hydrochloric acid consumed in this test} - \text{Volume (ml) of 0.1 mol/l hydrochloric acid consumed in the blank test}}{\text{Weight (g) of the sample} \times \frac{\text{Solid (\%)}}{100}} \times 0.1 \text{ (milliequivalent / g)}$$