

Test Method for Chelating Resin CR11

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1 Properties of CR11

Exchange group: Iminodiacetic acid (product is in Na form)

Exchange capacity: Cu 0.5 mmol/mL-R or higher

Effective pH: 4–10

The adsorption rate for divalent metal ions is highest at around pH 5. At a higher pH, ions may be precipitated as hydroxides, resulting in an increased ion leakage rate. Therefore, the stock solution should be adjusted to the optimal pH. Some metal ions can be slightly adsorbed in an acidic solutions, in such a case, CR11 is available at a pH lower than 4.

Ion form: Na form

In terms of the ion form of the exchange group, 100% Na form exhibits the largest through-flow exchange capacity. However, due to the high pH inside the resin particles, precipitation may occur in the column even if pH of the stock solution is adjusted. In this case, it is necessary to reduce the percentage in the Na form, leave a portion in the H form, and lower the pH of the resin particles prior to experiments.

2 Pretreatment of resin

Resin can be used as is without pretreatment for preliminary experiments and other rough experiments.

- 2.1 Accurately measure the necessary amount of resin, immersed in water, in a graduated cylinder (tap method) and transfer to a beaker.
- 2.2 After removing the water through decantation, add 1 mol/L HCl in an amount about 3 times the amount of resin (BV) and stir for about 10 minutes (do not use a magnetic stirrer as it will crush the resin).
- 2.3 Remove the HCl through decantation, add about 3 BV of desalinated water and discharge after stirring. Add about 3 BV of desalinated water and wash in the same way again.
- 2.4 Following the same procedure as 2.2, convert the resin to the Na form using about 3 BV of 1 mol/L NaOH.

2.5 Following the same procedure as 2.3, wash three times with about 3 BV of desalinated water.

3 Batch adsorption test (simple method for determining whether adsorption is feasible)

3.1 Accurately measure about 10 mL of resin, immersed in water, in a graduated cylinder. After draining the water, place resin into an Erlenmeyer flask.

3.2 Add stock solution containing 6 mmol of metal, adjusted to pH 4–5, and shake for 2 hours.

3.3 After shaking, measure the concentration of the metal in the aqueous solution and find the adsorption amount.

The adsorption amount varies depending on the pH, so measure the pH as a reference value.

3.4 If precipitation of metal hydroxide occurs, then before shaking add HCl (2.4–6.0 mmol) equivalent to 20–50% of the amount of carboxylic acid in the resin (about 1.2 meq/mL).

4 Column liquid passage test

Used column diameter: 15 mm (dia.) or larger

Resin layer height: 300 mm or higher

Liquid passage flow velocity: SV 10–30 (hr^{-1})

Note: When collecting design data, use a column with a diameter of at least 20 mm and a layer height of 800 mm or higher.

4.1 Pass stock solution adjusted to pH 4–5 through the column filled with pretreated resin. Stop passing liquid when the specified concentration of metal has leaked into the pre-treatment liquid.

4.2 After expelling the stock solution with 1.5–2 BV of desalinated water, regenerate according to the following regeneration method.

4.3 For cycle 2 and cycle 3, pass liquid through in the same way.

4.4 For through-flow exchange capacity, use the average values of cycle 2 and cycle 3, omitting the complete regeneration of cycle 1.

If precipitation of metal hydroxide occurs, lower the Na form conversion rate by decreasing the amount of NaOH in 5.4 of the following regeneration method. In this case, use a method such as air bubbling to mix the resin layer after rinsing with water.

5 Regeneration method

5.1 After passing liquid, expel stock solution in the column with 1.5 BV of desalinated water.

5.2 Provide a flow of 2-3 BV of 2 mol/L HCl or 1 mol/L H₂SO₄ at a flow velocity of SV 2 to elute the metal.(*1)

5.3 Provide a flow of 2 BV of desalinated water at the same flow velocity and expel the HCl or H₂SO₄.

5.4 Provide a flow of 2 BV of 1 mol/L NaOH from the bottom of the column(*2) at a flow velocity of SV 2 and convert the resin to Na form.

5.5 Provide a flow of 1.5 BV of desalinated water at the same flow velocity and expel the NaOH.

5.6 Provide a flow of 10 times the amount of desalinated water at the flow velocity of the stock solution to wash the NaOH.

*1 If elution of metals is insufficient, increase the concentration and amount of HCl or H₂SO₄. For alkaline earth metals such as Ca, precipitation of sulfates will occur if H₂SO₄ is used. For other heavy metals, regeneration is easier using H₂SO₄. Due to the difficulty of regeneration in the case of Hg and trivalent ions (Cr, Fe, etc.), regeneration must be done using highly concentrated H₂SO₄. With Hg complete regeneration is achieved with 3 BV of a mixed liquid of NH₄Cl (5%) and NH₃ (28%).

*2 When the R-H form becomes an R-Na form, resin volume swells by about 1.3 times. If a

small diameter column is used, the resin will not be able to expand when liquid is passed from the top of the column, and this may result in damage to the glass column from the expansion pressure of the resin.