Test Method for CRB Series of Boron Selective Resins

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Test method for DIAION CRB03, 05 of boron selective resins

1 Preparation of stock solution

Prior to experiments, remove suspended solids, oils, and oxidizers from the stock solution brought into contact with CRB03 or 05.

2 Simple test method

There are two methods for roughly checking adsorption behavior: the batch method and the column method. Each of these methods is described below.

2.1 Batch method

Testing using the batch method is a simple technique as for determining, before more in-depth studies, whether or not selective adsorption is feasible.

Place about 10 mL of conditioned resin into an Erlenmeyer flask with stopper, and pour in the specified amount of stock solution. While stirring or shaking strong enough that the resin floats up, allow to react for 12–24 hours. Then check the adsorption effect by filtering the supernatant from the resin slurry and analyze the filtrate. When using a magnetic stirrer, be careful because this may cause crushing of the resin in some cases.

The CRB reaction rate is slower than ordinary ion exchange resin. Therefore, to find the complete equilibrium adsorption capacity, please react for about 100 hours. It is also affected by pH, so the stock solution should be adjusted to the optimal pH.

2.2 Column method

Fill a column with 10–20 mL of the conditioned resin, and allow stock solution to flow at a flow velocity of SV 5–20. Sample a fixed amount of the treated liquid, analyze, and check effectiveness. If concentration of the adsorbed component is high, allow to flow at a slow flow velocity.

For details on conditioning, see section 3.3.

3 Checking and setting column liquid passage conditions

Conduct a column liquid passage test to check liquid passage conditions for industrialization purposes and to optimize regeneration conditions.

3.1 Experimental equipment

In the case of a small-scale experiment for resin selection, as indicated above, it is convenient to use a 10–15 mm (dia.) glass column for the experiment, but for this experiment, use a column with a diameter of about 20 mm or more, and set the height of the layer filled with resin to 60 cm or higher. In addition, when performing backwashing, the column must have backwashing space roughly the same as the resin layer height.

(If the purpose is not to collect design data, then a scale smaller than the above conditions is fine, and there is no need to perform backwashing.)

Fix the column perpendicular to the support stand etc. Fig. 1 shows an example of the experimental equipment.

If there is no pump, the stock solution container can be placed at a location higher than the column, and liquid allowed to flow due to gravity.

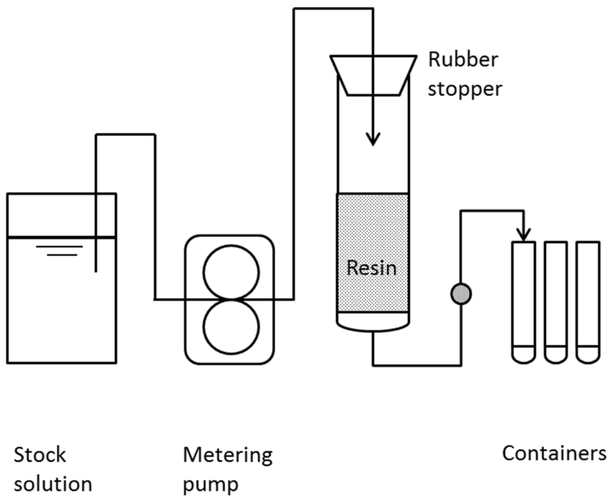

Fig. 1: Equipment

3.2 Resin filling method

When filling resin into the column, take the specified amount of resin, obtained by accurately measuring volume in water beforehand using a graduated cylinder and the tap method, and add while in the slurry state into the column filled with water (Fig. 2).

At this time, be careful not to allow air bubbles into the resin layer. If air bubbles get in, insert a rod or similar tool, and remove the air bubbles.

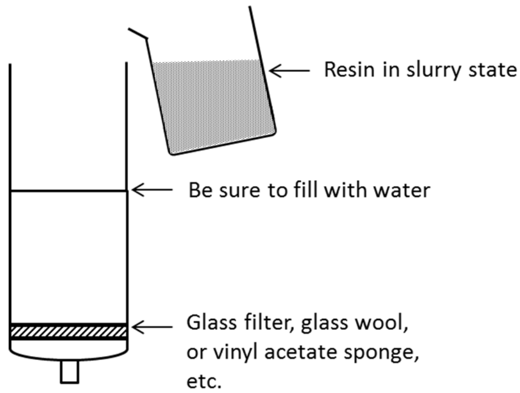

Fig. 2: Resin filling method

3.3 Resin conditioning

The ion form of CRB03 or 05 is a free base form. Resin conditioning must be performed before use to set to an ion form ratio suitable for liquid passage treatment.

The conditioning method is the same as 3.3.1 elution–3.3.6 regeneration processes described below.

3.3.1 Elution process

This is the process for eluting boron after passing liquid. (In the case of new resin, boron has not been adsorbed, but be sure to perform this step as pre-processing to prepare the ion form in the regeneration step.)

Concentration: 1mol/L HCl or 0.5 mol/L H_2SO_4

Amount used: 1.5–3.0 mol/L-R

Liquid passage flow velocity: SV 1–3

3.3.2 Eluent expulsion process

Allow pure water to flow at the same flow velocity as in the elution step, about 3–5 BV.

*Acid adsorbed to the exchange group is hydrolyzed, so the liquid flowing out does not become neutral.

3.3.3 Regeneration process

Ions are converted to the free base form (the form active for boron adsorption) by using an aqueous solution of NaOH, but if the resin is used with 100% free base form, the treated liquid will become alkaline due to an exchange reaction with the salt being treated. If the treated liquid has high pH and problems occur such as precipitation of metal components or deterioration of main components, liquid is passed after setting the resin to a mixture of ion forms: free base form and H_2SO_4 form or HCl form. The optimal rate of conversion to the free base form varies depending on the type and concentration of the salts contained in the treated liquid, but general speaking, the free base form is adjusted in the range of 50–100% of the resin total exchange capacity using a 1 mol/L aqueous solution of NaOH. The liquid passage flow velocity is SV 1–3.

3.3.4 Caustic expulsion process

Allow pure water to flow at the same flow velocity of 1–2 BV.

3.3.5 Mixing process

This is performed to ensure a uniform distribution of the free base form and acid adsorption form, but mixing cannot be done in the experiment column, so remove the resin from inside the column, place it in a beaker, and then refill into the column while evenly mixing with a stirring rod.

3.3.6 Water rinsing process

In order to wash away remaining NaOH, allow pure water to flow at the same flow velocity as the regeneration process, about 5–10 BV.

3.4 Procedure steps

As a typical example of the regeneration/liquid passage procedures, the following shows the procedure steps. For details on each step, see "4. Experiment procedure" below.

Backwashing	Expansion rate is 1.3–1.5 times the resin layer height. 15–30 min
Settling down	5–10 min
Water draining	About 5–10 cm above the resin surface

Elution	See 3.3.1
Eluent expulsion	3–5 BV at same flow velocity as elution step
Regeneration	See 3.3.3
NaOH expulsion	1–2 BV at same flow velocity as regeneration step
Mixing	See 3.3.5
Water rinsing (1)	See 3.3.6
Sweetening on	Allow treated liquid to flow at same flow velocity as in liquid passage step
Liquid passage	See 4.11
Sweetening off	Allow pure water to flow at same flow velocity as in liquid passage step
Water rinsing (2)	Allow pure water to flow at same flow velocity as in liquid passage step

4 Experimental procedure

The following explains the experiment procedure for the column method, but steps 4.1 Backwashing – 4.2 Settling down can be omitted depending on the situation. In addition, if there is no need to recover liquid, there is no need to perform 4.10 Sweetening on and 4.12 Sweetening off.

4.1 Backwashing

This is performed to discharge, to the outside of the column, the suspended solid

(SS) component mixed in during liquid passage and the resin crushed during use, and to improve contact efficiency with the regeneration agent by loosening the compacted resin layer.

Perform backwashing with an upward flow at a flow velocity so that the resin layer expands by 1.3–1.5 times, until the water flowing out is transparent. The backwashing expansion rate varies depending on the water temperature.

4.2 Settling down

This step allows the resin layer, expanded due to backwashing, to naturally settle.

4.3 Water draining

After the settling down step is finished, this process drains off water at the top of the column to a position about 5–10 cm above the resin surface. This is done to avoid dilution of the regeneration agent.

4.4 Elution

This step is for eluting the adsorbed boron.

For the eluent concentration and use amount, please see 3.3.1 above.

4.5 Eluent expulsion

This step is an extension of elution. Here, unreacted regeneration agent remaining in the column is expelled using pure water at the same flow velocity as elution. The amount of pure water used is 3–5 times the amount of resin.

4.6 Regeneration

In this step, exchange groups in the acid adsorption form are converted to the free base form which is active for boron adsorption. For the NaOH concentration and use amount, please see 3.3.3 above.

4.7 NaOH expulsion

This step is for expelling NaOH used for regeneration with pure water at the same flow velocity as regeneration. The amount of water used is 1–2 times the amount of resin.

4.8 Mixing

This is performed to achieve a uniform distribution of the free base form and acid adsorption form. In the column procedure, the resin is removed once into a beaker, and then refilled into the column while evenly with a stirring rod.

4.9 Water rinsing (1)

This is performed to rinse away any NaOH remaining in trace amounts.

4.10 Sweetening on

When performing treatment with a special chemical liquid, water in the resin layer is steadily replaced due to the liquid being passed through. "Sweetening on" refers to this replacement step.

4.11 Liquid passage

Recovery as treatment liquid begins from the point where the chemical liquid at the column outlet reached or exceeded a certain concentration, and continues until ion leakage occurs and the specified liquid quality is no longer obtained. This process is called liquid passage.

Flow velocity during liquid passage is SV 5–20.

4.12 Sweetening off

In treatment with chemical liquid, this is the process of replacing the treated liquid in the column with water after passing through the chemical liquid. The term applies until the outlet concentration is at or below the specified concentration. This portion is recovered as treatment liquid or sweet water.

4.13 Washing (2)

In treatment with chemical liquid, this is performed in the same way as washing after regeneration. The amount of washing water used is about 1/2 that used after regeneration.