



Test Method for Synthetic Adsorbents

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Test method for synthetic adsorbents

1 Preparation of stock solution

Prior to experiments, remove suspended solids (ss), oils, and oxidizers and air bubbles from the stock solution that will come into with synthetic adsorbents.

2 Selection of resin brand

In adsorption by synthetic adsorbents, the material to be adsorbed must be diffused into the pores of the adsorbent and reach the adsorption surface. Therefore the resin must have a pore diameter sufficient and appropriate for that purpose.

Select the optimal adsorbent by taking the following characteristics into consideration.

2.1 Pore size

SP850<SP825<SP70<SP700<HP21, SP207<HP20, HP2MG

For material with a molecular weight in the hundreds, use the SP800 Series. The HP20 Series is suitable for weights in the thousands and higher.

2.2 Adsorption power

HP2MG<HP20, HP21, SP70, SP700, SP825, SP850<SP207

Due to the high adsorption power of SP207, it is recommended for previously difficult adsorption systems (water-soluble materials with low molecular weight) and adsorption of light yellow pigments that cannot be removed even with activated charcoal. However, the reverse side of its high adsorption power is its characteristic of being difficult to elute. So when eluting, please consider the eluent concentration and type. Please try HP2MG in systems (highly hydrophobic macromolecules) where synthetic adsorbents previously could not be used because conventional elution was impossible.

2.3 Magnitude of true specific gravity

HP20, HP21, SP70, SP700, SP825, SP850<HP2MG<SP207

SP207 has a high true specific gravity. Therefore backwashing is easy, and settling down can be done quickly. It is particularly effective for treatment of liquids with high specific gravity and upward flow treatment in system containing SS components.

2.4 Swelling characteristics

HP2MG<SP207, SP70, SP700, SP825, SP850<HP20, HP21

With the HP20 Series, there is a 20–30% change in volume when changing from a water system to a solvent system. Therefore, please consider the possibility of column damage or compaction due to pressure when volume expands.

2.5 Pore distribution sharpness (chromatographic performance)

HP20<HP21, SP207<SP70, SP700<SP825, SP850

If high separability is required, the SP800 Series is a good selection due to its sharp pore distribution.

3 Test method for selection of resin brand

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

3.1 Batch method

Testing using the batch method is a simple technique for selecting an effective product in the initial stages of review. In the end, this is only a guide for roughly determining brands which suit the intended purpose.

Place about 10 mL of pretreated resin into a beaker or an Erlenmeyer flask and pour in the specified amount of stock solution. While stirring or shaking strong enough that the resin floats up, allow to react for 0.5–2 hours. Then check the effect by filtering out the supernatant from the resin slurry, and analyzing the filtrate. At this time, adsorption speed can be determined by conducting analysis at fixed intervals.

When using a magnetic stirrer, be careful because this may cause crushing of the resin in some cases.

3.2 Column method

Fill the column with 10–20 mL of pretreated resin, and allow stock solution to flow at a flow velocity of SV 0.5–5. Sample a fixed amount of the treated liquid, analyze, and check effectiveness.

Use a slow flow velocity if the molecular weight of the adsorbed material is high, or if the viscosity of the liquid is high.

*For details on pre-treatment, see Section 4.2.

4 Checking and setting column liquid passage conditions

After the brand has been selected, conduct a column liquid passage test to check liquid passage conditions for industrialization purposes and to optimize regeneration conditions. (As a special case, industrialization may be done using the batch method.)

4.1 Experiment 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. When water is replaced with organic solvent, synthetic adsorbent increases in volume by 10–30%. If the change in volume is large, there is a risk of column damage due to swelling pressure of the resin, so please consider to use a large-diameter glass column in such a case. Care is needed when using a PVC or acrylic column because there are restrictions on factors such as solvents used and experiment temperature. Fasten the column perpendicularly to a support stand, etc. The following diagram shows an example of the experiment equipment (Fig. 1).

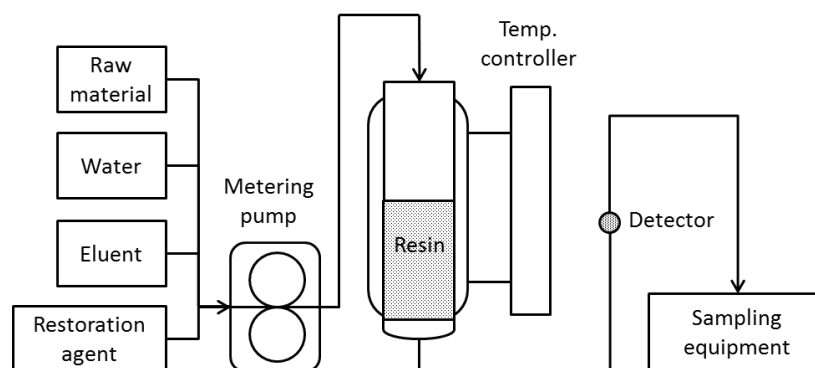


Fig. 1: Equipment

4.2 Pre-treatment (gas removal)

To remove gas in resin, first accurately measure volume of resin in water beforehand using a measuring cylinder. Put resin into a beaker, immerse in methanol (or the organic solvent being used), stir with a rod or similar tool, and remove air.

4.3 Column filling

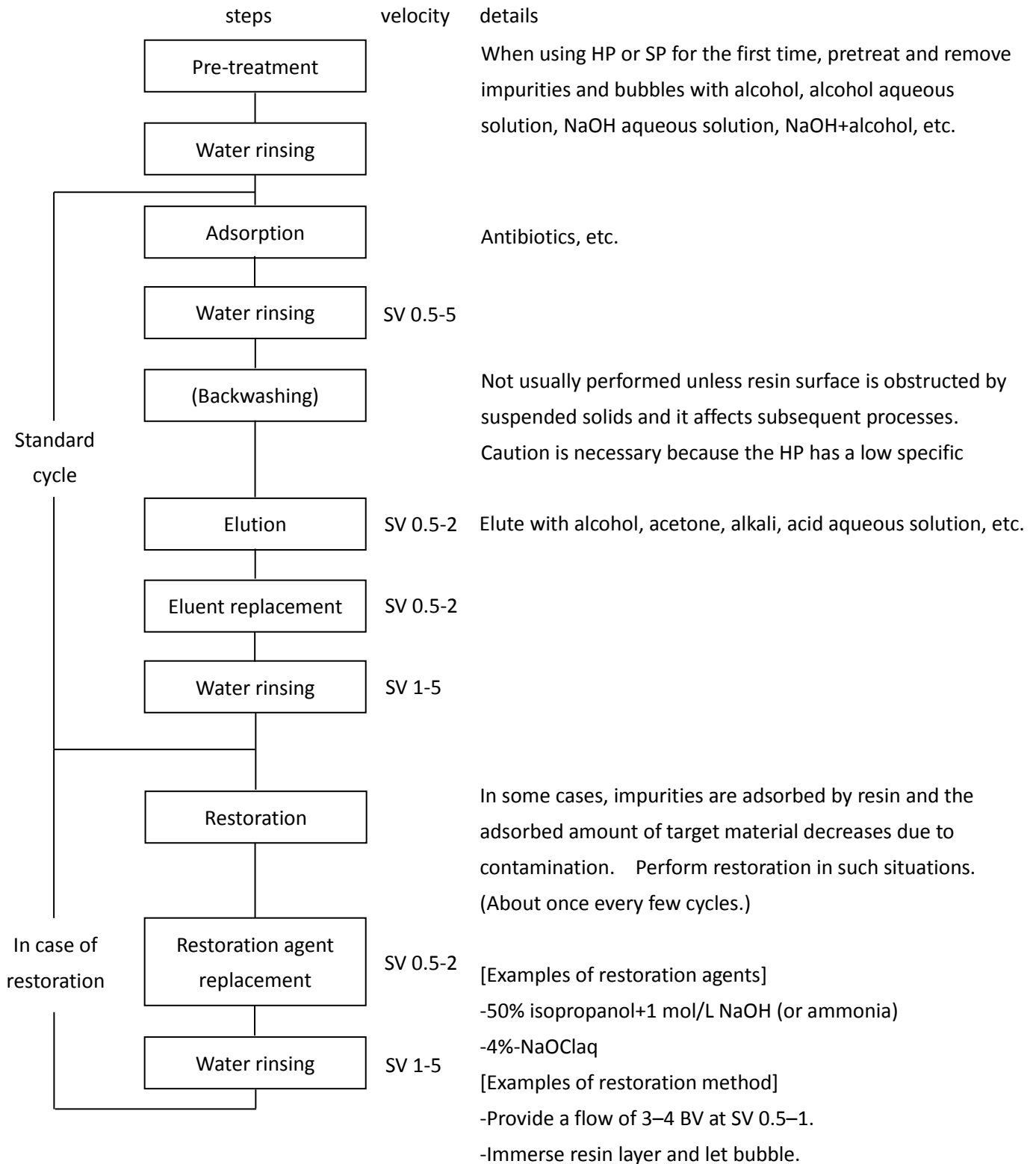
When filling resin into the column, add into the column filled with water while immersed in methanol (or the organic solvent being used).

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

4.4 Adsorbent washing

If trace amounts of eluate will be a problem, remove the eluate by first washing with the organic solvent used in an amount at least 5 times the resin amount at about SV 2, and then rinse with water.

5 Standard cycle for the synthetic adsorbent column method



6 Experimental procedure

A synthetic adsorbent has high adsorption power to the degree that the material is highly hydrophobic (undissociated) as indicated above. Therefore, please set the following conditions after ascertaining the chemical properties of the adsorbed material.

6.1 Adsorption

6.1.1 pH adjustment

In the case of an electrolyte, adjust pH so it is undissociated, and in the case of an amphoteric electrolyte, adjust pH so it is at the isoelectric point.

6.1.2 Salt concentration

When salt is added, there is a reduction in solubility of the adsorbed material due to a salting out effect, and the adsorption amount increases. The adsorption amount may also change due to changing the counter ion.

6.1.3 Change in solvent system, etc.

It is possible in some cases to increase the adsorption amount by reducing solubility of the adsorbed material.

6.2 Elution/Regeneration

Normally, organic solvents such as alcohol and acetone are used for this purpose. Also, in the case of an electrolyte or amphoteric electrolyte, elution can be done by adding acid or alkali, dissociating, and increasing water solubility.

In special cases, elution may be possible using water, hot water, steam, etc.

There are many cases in practice where a combination of organic solvents, acids and alkalis are used.

6.3 Restoration

If the resin irreversibly adsorbs impurities, and there is a drop in performance such as a reduced adsorption amount, or decline in separation performance, then elution (regeneration) is performed using more severe conditions than an ordinary solvent. This procedure is called the restoration. Agents used for restoration include organic solvents (e.g., alcohol, acetone, benzene, carbon tetrachloride), acids, alkalis, surfactants, and in some cases, oxidizers.

One generally used restoration agent is a liquid mixture of alcohol and NaOH. Among alcohols, restoration effectiveness is as follows: isopropyl alcohol > ethanol > methanol. NaOH is mixed as a 2–4% aqueous solution.

Restoration should not be done after performance has totally degraded. It is

more effective if carried out at a stage where degradation is minor (about once every few cycles).