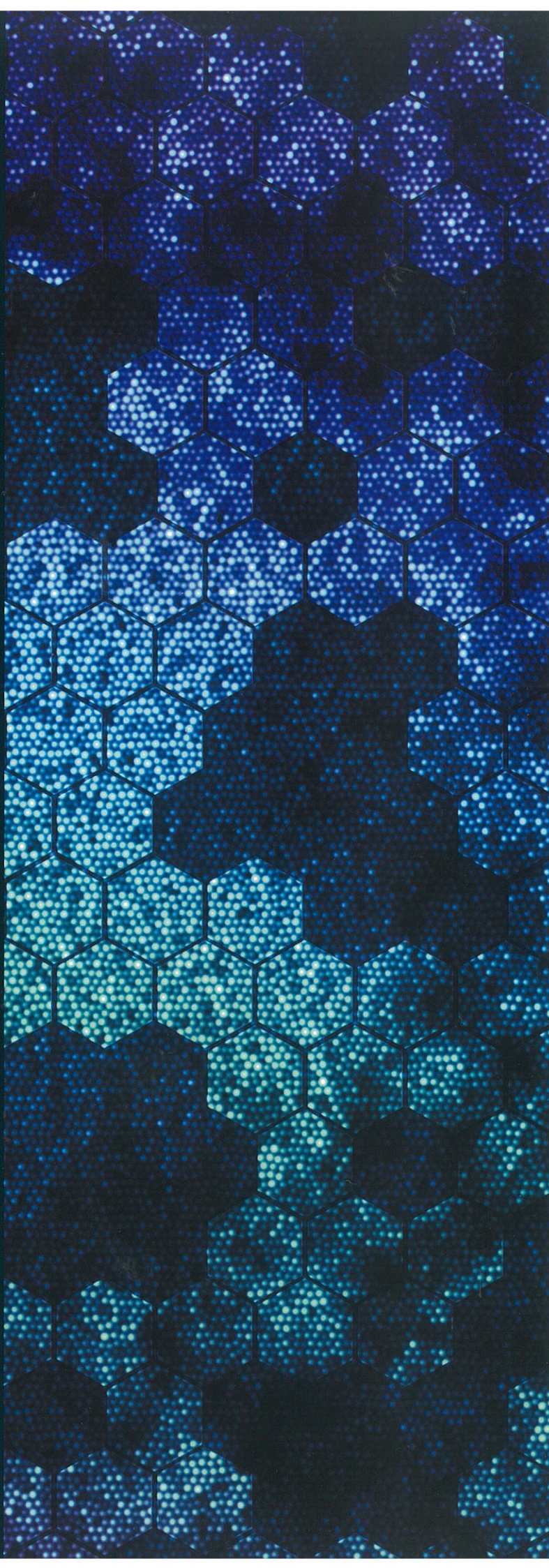


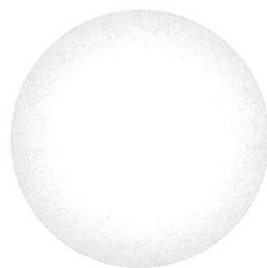


## HS and CL-K

Adsorption Resin and Granular Activated Carbon

*Ajinomoto Fine-Techno Co., Inc.*





## Adsorption Resin, Granular Activated Carbon

Adsorption resins manufactured by Ajinomoto Fine-Techno Co., Inc. are porous polymer beads having the adsorptive properties of both activated carbons and ion-exchange resins.

They can be utilized repeatedly through regeneration by means of a simple process using a chemical solution.

The special activated carbon "CL-K" has an extremely high adsorption performance compared with normal granular carbons, and in particular it exhibits a superior decolorization performance. In combination with adsorption resins, it enables purification treatment at a higher level.

### Handling of resins

1. Avoid exposure to direct sunlight, wind, rain and frost, and store them indoors within a temperature range of 0 to 40 °C.
2. Keep packages sealed and take special care not to allow the resins to dry out.
3. Regenerate the resins for conditioning so you can use them in a more stable manner.
4. Be sure to wear protective equipment (dust-proof glasses, protective gloves, masks, etc.) when coming into contact with resins during filling and extracting work.

\*If the resins get into your eyes, wash them with copious amounts of water and see a doctor.

5. If the resins come into contact with chemicals having a strong oxidation property, such as nitric acid, there is a risk of an explosive reaction, so take extra care to prevent this.

6. For further details, please see the Material Safety Data Sheet (MSDS).

### Conditioning of resins and activated carbons

A very minute amount of impurities (organic matter, fine powder, etc.) are present in adsorption resin and granular activated carbon products. If they are to be used in an environment where precision is required, such as in food purification and pharmaceutical purification, we recommend that you first remove the impurities.

For the conditioning of resins, see Section 1-5 "Regeneration method and life," and for the conditioning of granular activated carbons, see Section 2-5 "Conditioning."

We offer a range of technical materials on decolorization, deodorization, exhaust gas treatment, wastewater treatment, separation and purification, and provide technical services.

We also have a great depth of experience regarding the design, installation and maintenance of adsorption equipment, so please feel free to consult us at any time.

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## ■ 1. Adsorption resin "HS" for decolorization (deodorization)

### ■ 1-1 Overview

Adsorption resin "HS" is a porous spherical resin that uses phenol-formal-dehyde resin as its main framework. This resin has been developed mainly for food and pharmaceutical purification, and it has a physisorption behavior just like activated carbons. Furthermore, because a weak acid group (phenolic hydroxyl group) and a basic group (amino group) exist in the adsorption resin as functional groups, the surface polarity changes according to the pH. Therefore, electrostatic adsorption power is generated in the acid and neutral regions, while the adsorbed matter becomes easier to be desorbed in the alkaline region because the adsorption power is reduced.

Thanks to this property, adsorption resin "HS" shows a superior decolorization performance for the purification of foods and pharmaceuticals, including soy sauce and amino acids (see Fig. 1). Also, it can be used repeatedly by carrying out a simple regeneration treatment using acid and alkali.

For details of the regeneration method, see Section 1-5 "Regeneration method and life."

The resin can be used in the same manner as ion-exchange resins. Fill a column with the resin and inject the concentrate solution. For details, see Section 1-4 "Usage method and test method."

Adsorption resin "HS" can also be used as a food additive. Its classification as a food additive is "ion-exchange resin."

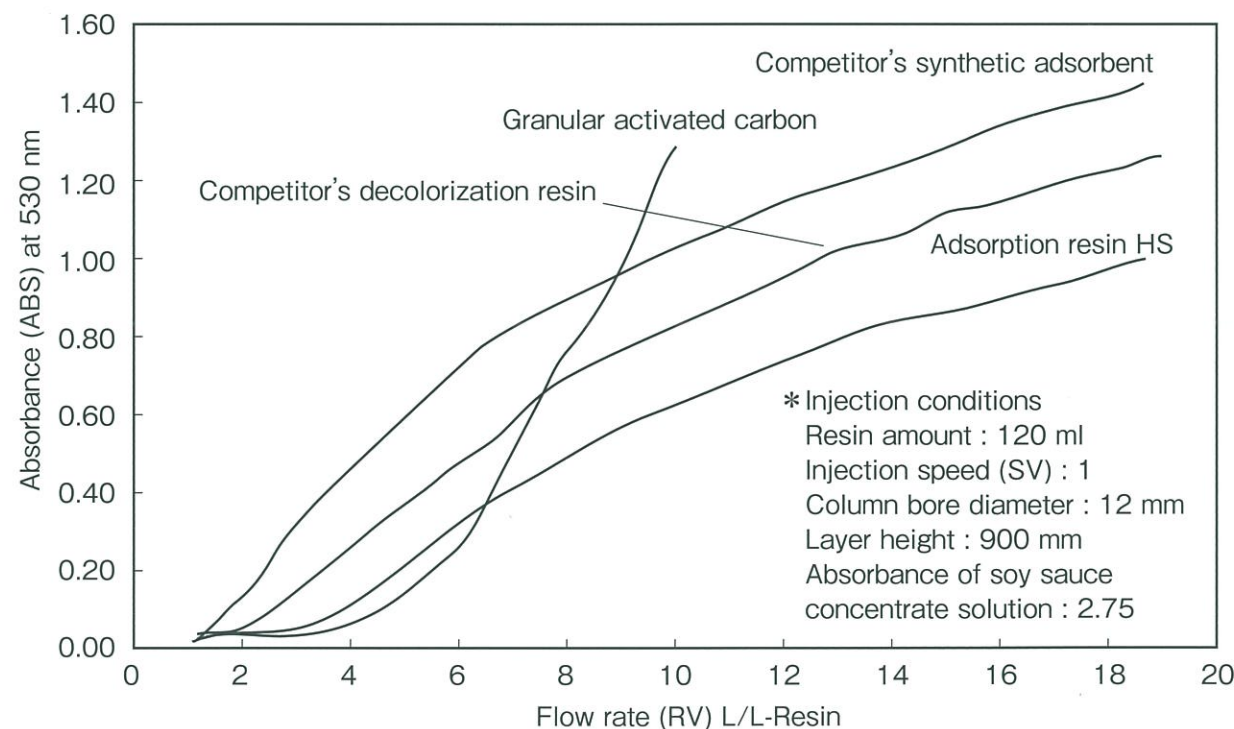


Fig. 1. Injection decolorization curve of soy sauce by various adsorbents

## ■ 1-2 Properties and applications

	Adsorption resin HS
Appearance and shape	Black and spherical
Water content (%)	65 to 75
Apparent density (g/L-R)	700 to 780
Particle size (1.40 to 0.355 mm)	85 % or more
Effective diameter (mm)	0.3 to 0.5
Uniformity coefficient	2.6 or less
Functional group (ion-exchange group)	R $\begin{cases} \text{OH} \\ \text{NH}_2 \end{cases}$
Swelling rate 1N HCl/1N NaOH (new product)	3 vol% increase (alkaline immersion)
Chemical resistance	Deteriorates with concentrated alkali Dissolves with oxidant
Solvent resistance	Good
Usage temperature range (°C) *1	5 to 50
Effective usage pH range *2	2 to 8
Main applications *3	Decolorization and deodorization of amino acids, soy sauce, starch syrup, glucose, honey and sugars

\*1 The resin is normally used at room temperature, but it may be used within this temperature range.

\*2 For efficient decolorization, normally a pH around the treatment solution's isoelectric point is selected. It cannot be used with alkali.

\*3 In addition, we offer adsorption resin KS, the pores of which are adjusted for the deodorization and decolorization of lactose, soy sauce, refined sake, etc. Please contact us for details.

Note : Due to the nature of the resin, a very minute amount of formaldehyde may be eluted from the resin depending on usage conditions. Please use it within the range of specified conditions wherever possible to prevent problems. Please consult us if you are going to use it outside the range of specified conditions.

Table 1. Properties of adsorption resin HS

### ■ 1-3 Difference between adsorption resins and activated carbons

Adsorption resins and activated carbons are both porous solids, but they differ in various points such as pore structure and functional group. Therefore, the adsorption performances of activated carbons and adsorption resins are different.

As an example, the decolorization curves for seasoning liquid are shown in Fig. 2. In this case, the decolorization performance of the adsorption resin is greater than for the activated carbon. Also, as shown in Table 2, as the adsorption resin's adsorption capacity of aromatic amino acids is lower than that of activated carbon, so there is an advantage in that the loss of amino acids is reduced when treating seasoning liquid.

Decolorization curves for monosodium L-glutamate solution are shown in Fig. 3. It can be seen that the decolorization power of the adsorption resin is greater than that of the activated carbon in the region with a smaller amount of adsorbent additive. At the same time, as the activated carbon is highly effective in decolorizing light-colored solutions, in cases where coloring is particularly unwanted, you can use the adsorption resin in a first treatment and the activated carbon in a second treatment. In this fashion, if you use a combination of the two taking their properties into consideration, you will be able to carry out decolorization more efficiently than if you were to use them independently.

For the test method for decolorization, see Section 1-4 "Usage method and test method."

	Tryptophan (g/L)	Phenylalanine (g/L)
Adsorption resin HS	3.4	3.4
Activated carbon	15 to 17	10

Table 2. Amount of adsorbed aromatic amino acids by adsorption resin and activated carbon (approximate value)

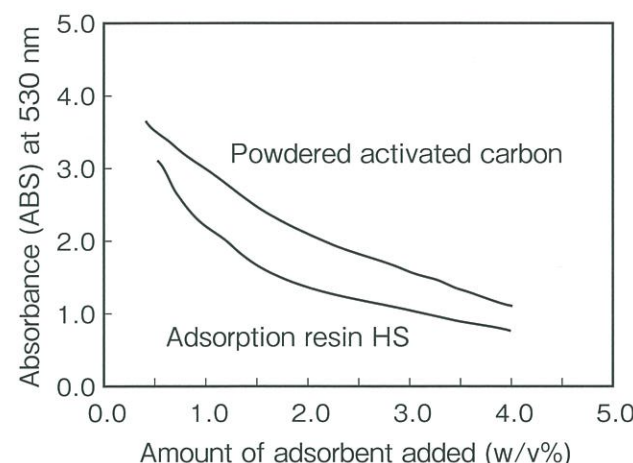


Fig. 2. Decolorization of seasoning liquid

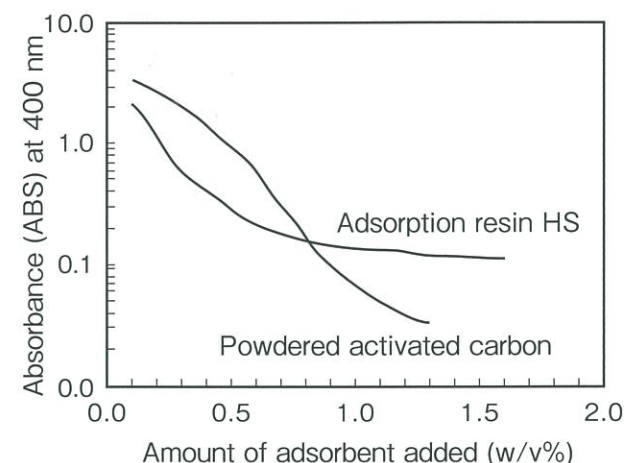


Fig. 3. Decolorization of monosodium L-glutamate solution

#### 1-4 Usage method and test method

The usage method of adsorption resins is basically the same as that for ion-exchange resins. Fill a column (resin tower) with the resin, and inject the concentrate solution for treatment into it at a constant speed. However, as optimum conditions vary depending on the concentrate solution for treatment, we recommend that you carry out the following experiment in order to determine conditions such as the resin amount, the flow rate speed and treatment temperature.

For usable temperature and pH, see Section 1-2 "Properties and applications," and for details of the tests, see the Appendices at the end of the document.

##### 1) Batch adsorption test (⇒ Appendix 2)

Confirm whether decolorization of the concentrate solution for treatment is possible using the adsorption resin.

Measure a required quantity of the resin pulverized in a wet state, add it to the concentrate solution for treatment, stir it for about an hour and filter it. Compare the color of the filtrate and the concentrate solution, and check whether it can actually be used.

##### 2) Injection and regeneration tests using a small column (⇒ Appendices 3 and 4)

Fill two-thirds of a small column (made of glass, bore diameter : 15 to 30 mm, height : 0.6 to 1 m) with the adsorption resin, and run the concentrate solution for treatment through the column at a set speed. Collect the treated concentrate solution by increments at the column exit, confirm the change in colors, and examine the relation between the resin amount and treatment amount in detail. Stop the injection when the color of the treated

concentrate solution becomes dark and the adsorption power becomes reduced, then carry out regeneration in accordance with Section 1-5 "Regeneration method and life." The above process is defined as one cycle.

Repeat the tests with different flow rates, temperatures and regeneration conditions to determine the optimum conditions and calculate the required resin amount.

##### 3) Scale-up test (⇒ Appendix 5)

Implement scale-up test based on the data obtained in 1) and 2), and ascertain whether there are any problems. We recommend that you conduct the test by using a column that allows a resin layer height of 1 m or more, in order to obtain results that are closer to actual conditions.

#### 1-5 Regeneration method and life

Regeneration is carried out so as to recover the adsorption power, or for conditioning before usage or after storage.

Standard regeneration conditions are shown in Table 3.

Order	Operation	SV	Time	Purpose
1	Water driving	0.5	1	Collection of concentrate solution for treatment
2	Alkaline elution (3 to 5% NaOH)	1	2	Elution of adsorbed matter
3	Hot-water (water) washing	1 to 3	1 to 3	Elution of adsorbed matter and removal of alkali
4	Acid neutralization (1% HCl)	1 to 2	1 to 2	Neutralization
5	Water washing	1 to 3	1 to 3	Removal of acids and salts
6	Backwashing	LV 5 m/hr	0.5	Prevention of channelling

Table 3. Standard regeneration method of adsorption resins (decolorization of soy sauce)

##### 1) Water driving

Collect the treatment solution inside the column. When suspended matter is accumulated on the adsorption resin layer, remove the suspended matter by backwashing after water driving. In some cases, air stirring is recommended before backwashing.

##### 2) Alkaline elution and water washing

Next, adsorbed matter is eluted using alkaline aqueous solution, which causes some swelling of the adsorption resin. Normally, for alkaline regeneration, 3 to 5% aqueous sodium hydroxide is used and the treatment is conducted at room temperature. However, depending on the treatment solution, the treatment can be carried out at a temperature other than room temperature (i.e. 60 to 80°C). After injecting alkaline aqueous solution, carry out water washing or hot-water washing to remove the eluted adsorbed matter as well as excess alkali.

### 3) Acid neutralization and water washing

After washing with sufficient water, neutralize the resin using dilute acid. Around 1% hydrochloric acid is normally injected, however, organic acid such as acetic acid and aqueous ammonium chloride solution can also be used instead of hydrochloric acid. Furthermore, neutralization can be carried out by stirring the resin with a small amount of acid\*4. When neutralization is finished, implement water washing to remove salts and excess acids produced in the course of neutralization.

\*4 Add hydrochloric acid at one-tenth of the resin amount, with a slightly higher concentration than that of the injected solution (i.e. about 7%), and stir it. This approach is used for the treatment of a neutral pH solution, for example, for the decolorization of sugars and the deodorization and decolorization of proteins.

### 4) Backwashing

Lastly, carry out backwashing to prevent channelling, and even out the resin to complete the entire process.

As the optimum regeneration process varies depending on the concentrate solution for treatment and usage conditions, we recommend that you consider regeneration conditions based on the injection test. During regeneration, alkaline wastewater containing adsorbed matter will be produced as effluent. The total amount of regeneration wastewater will be around 10 to 25 times as much as the original resin volume per regeneration if a standard regeneration method is used. Please handle the produced wastewater in accordance with the relevant effluent standards.

The adsorption performance of an adsorption resin will decline gradually during repeated adsorption-regeneration cycles because adsorbed matter will be deposited inside the resin. In addition, the swelling rate will increase gradually, and the strength of resin grains will decrease gradually.

Fig. 4 shows the rate of decrease of resin's performance when it is used for the decolorization of seasoning liquid. Considering the adsorption performance and strength of resin, normally the resin should be changed after being used for 60 to 80 cycles, although this may vary depending on the treatment solution and usage conditions.

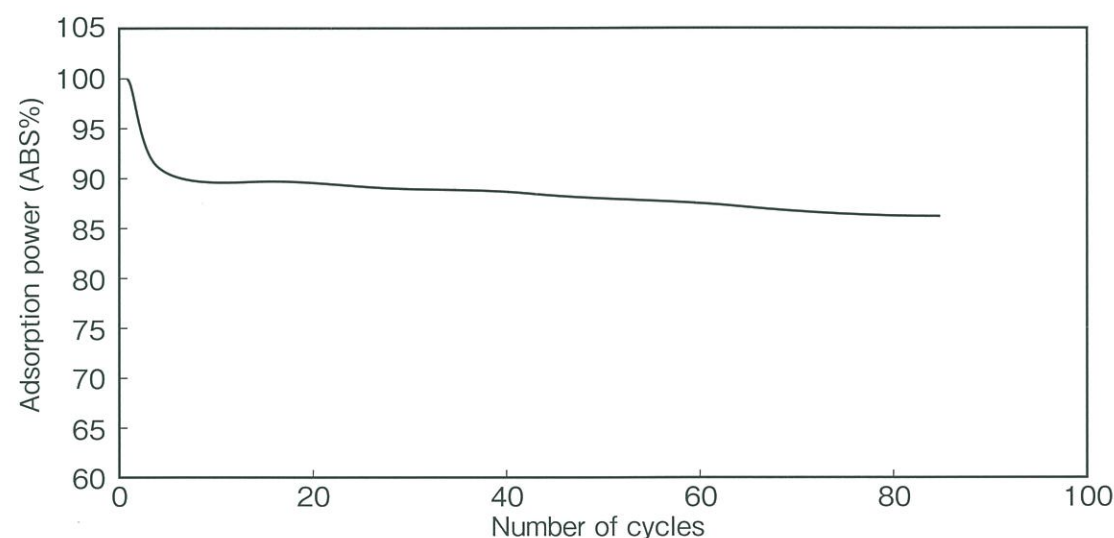


Fig. 4. Relation between adsorption power and number of cycles of the adsorption resin HS for seasoning liquid

## 1-6 Storage method

A minute amount of organic matter, which cannot be removed completely even although regeneration is carried out, is deposited in the resin after usage. In particular, if the resin is used in the food industry, micro organism may develop using this organic matter as its nutrient, contaminating the resin, so please be sure to keep the cautions below.

When storing the used resin, you must store it at pH 2 or less after carrying out regeneration, and store it in a wet state. If the resin is dried, its porosity will be lost and its adsorption performance will decline significantly. Also, if you intend to use it again after storage, be sure to carry out conditioning before use.

Particularly, if the resin is left inside the column without being used for several months, carry out backwashing or conditioning regularly during the storage period. If flow treatment is not carried out on the resin inside the column, flocks of resin grains will form and a blockage may result. Also, if the adsorption resin is stored repeatedly for long terms, its lifetime will be slightly shortened.

## 1-7 Column (adsorption resin tower) equipment

An example of equipment for handling adsorption resins is shown in Fig. 5. In addition to an "adsorption tower" consisting of a column filled with the resin, a pump for moving the treatment solution and clear water, a tank for keeping regeneration acid and alkali, as well as tubes, valves, a manometer and a flowmeter are required.

As acid and alkali are used during regeneration, be sure that the tubes and other equipment that come into contact with chemical solutions are made of materials that are resistant to these chemical solutions. Specifically, acid-resistant materials must be used for the column. Also, the materials must be able to withstand the temperatures reached during use. When using at room temperature, a column for general ion-exchange resins can be used.

At the bottom of the resin tower, place a butt strap or a strainer on a supporting bed paved with gravel or glass beads to support the resin load, while preventing resin leakage. Set the resin layer height (column filling height) at 1 to 2 m for each column (tower) after considering factors such as the strength of the resin. When sufficient decolorization is not possible using a single tower, such as in cases where the color of the concentrate solution for treatment is especially dark or a great deal of treatment is required, two to three towers are generally used in series to carry out regeneration sequentially from the column that has reached adsorption equilibrium. When setting up the equipment, we recommend that you determine the required resin amount, flow direction and rate, usage temperature, etc. after implementing the injection test specified in Section 1-4 "Usage method and test method."

If the column's height is not tall enough, the resin may blow out of it during backwashing due to the momentum of the water, and the resin may flow out from the top of the column. Calculate the height allowing sufficient margin based on the backwashing expansion rate shown on the next page.

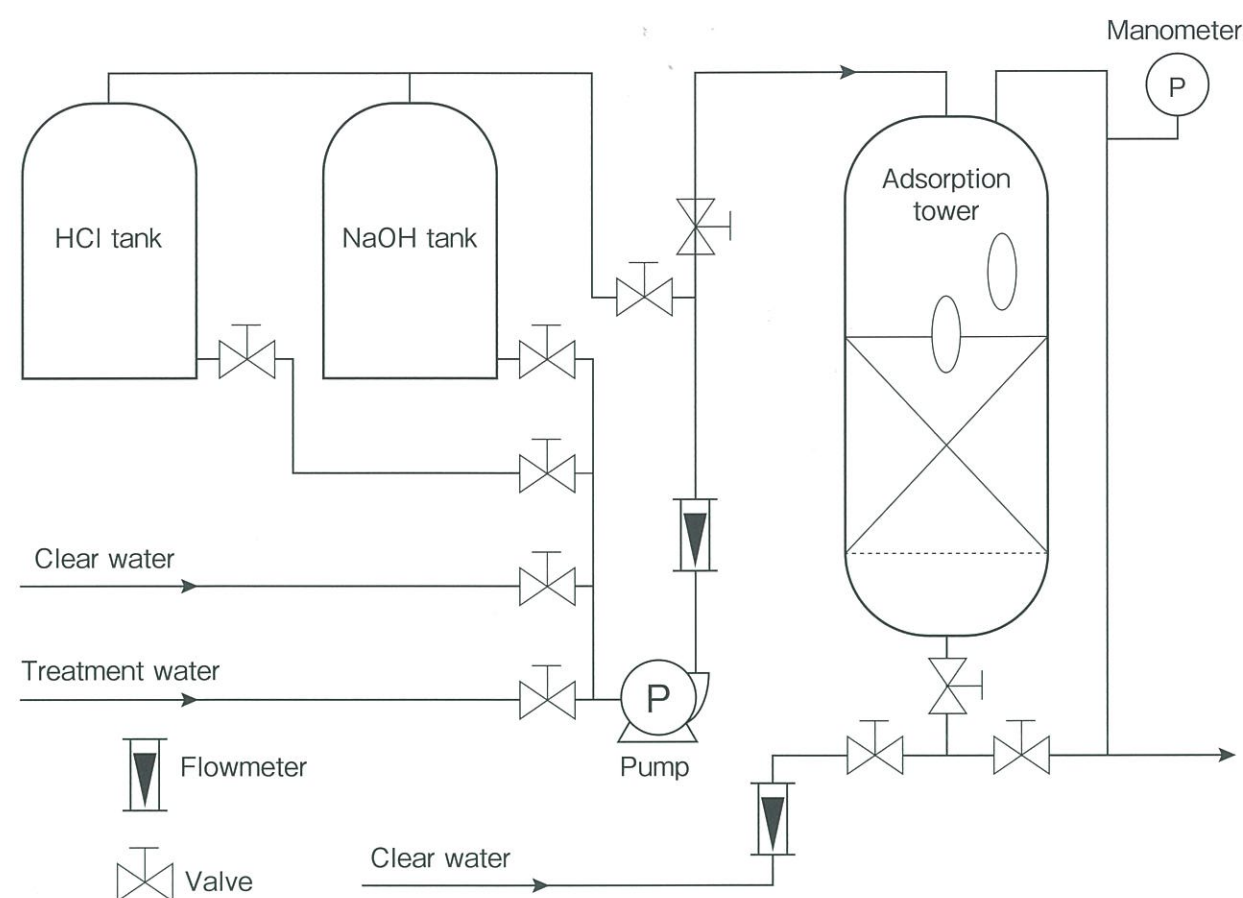


Fig. 5. Example of adsorption resin tower setup

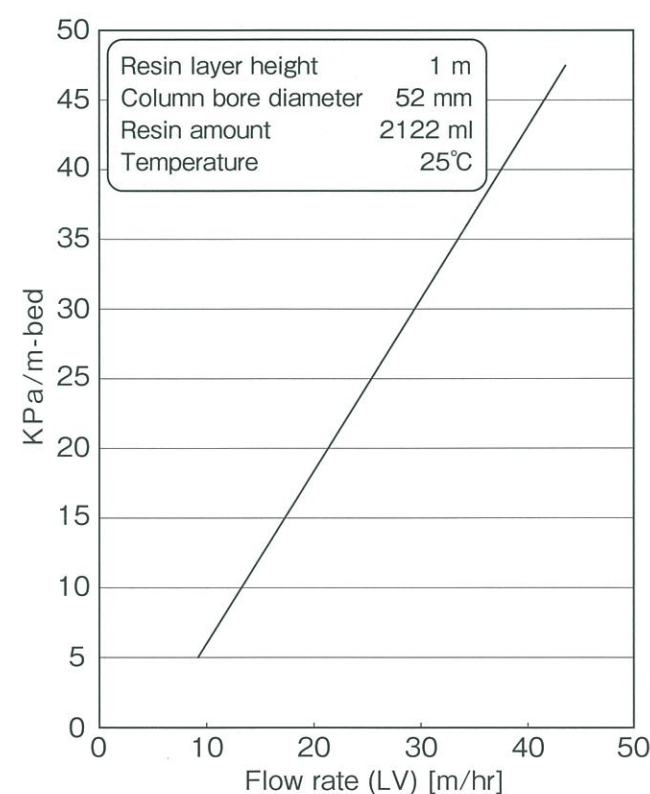


Fig. 6. Pressure loss of adsorption resin HS

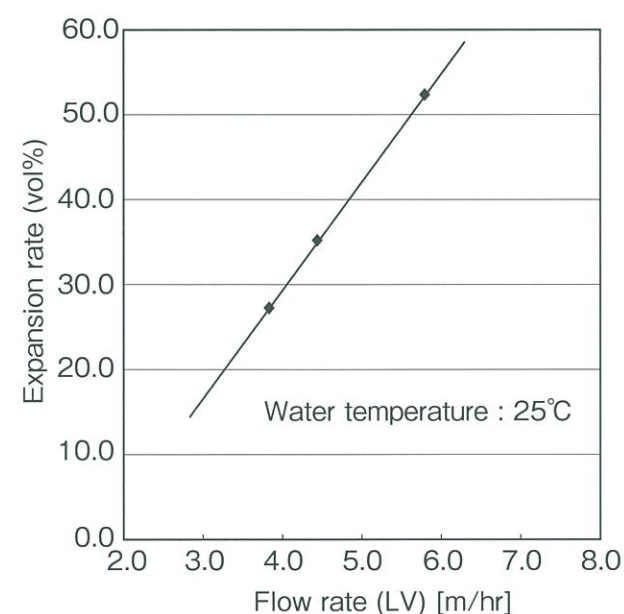


Fig. 7. Backwashing expansion rate of adsorption resin HS

## ■ 2. Granular activated carbon "CL-K" regenerable with chemical solutions

### ■ 2-1 Overview

As the specially activated carbon "CL-K" has an excellent adsorption performance and high purity, it is particularly effective in applications that require a high level of purification in the food and pharmaceutical industries. As it is highly effective in decolorizing light-colored solutions, a better effect can be expected if you use the resin for pretreatment (see the previous section), and CL-K at the exit in combination.

CL-K has two features: "regenerable with alkaline solutions" and "higher adsorption performance than normal granular activated carbons." As it is regenerable with alkaline solutions, a regeneration process necessary for normal granular carbons is not required, and it is very economical.

When adsorption performance has declined significantly, regeneration with heat treatment is required. Contact us for details.

### ■ 2-2 Applications

- Purification of sugars (fructose, glucose, sucrose, starch syrup, honey, etc.)
- Purification of chemical seasonings (monosodium glutamate, amino acid, inosinic acid, licorice)
- Decolorization, deodorization, purification and separation of industrial chemicals and pharmaceuticals
- Decolorization, deodorization and adsorption of organic matter (BOD and COD) of industrial water and city water
- Decolorization, deodorization and adsorption of organic matter (BOD and COD) at sewage plants
- Decolorization or adsorption of organic matter (BOD and COD) of dye effluent
- Decolorization and deodorization of alcohol
- Decolorization of dry cleaning solvents

### ■ 2-3 Properties

- General properties

Special activated carbon CL-K		
	Standard value	Example of analyzed value
Particle size (%) 10 to 32 mesh (1.70 to 0.50 mm)	90 or more	99.2
Hardness (%)	60 or more	77.9
Apparent density (g/ml)	0.16 to 0.27	0.24
pH	5.0 to 9.4	7.7
Iodine adsorption performance (mg/g)	1200 or more	1580
Methylene blue adsorption performance (ml/g)	—	280
Calcium content (mg/kg)	5000 or less	380

Table 4.

○ Food additive test

Analyzed item	Compositional standard	Passed/Failed
Appearance	Tasteless and odorless black grains	Passed
Confirmation test	Passed	Passed
Chloride (%)	0.53 or less	Passed
Sulfate (%)	0.48 or less	Passed
Zinc (%)	0.10 or less	Passed
Lead ( $\mu\text{g/g}$ )	5 or less	Passed
Arsenic ( $\mu\text{g/g}$ )	3 or less	Passed

Table 5.

## 2-4 Usage method and test method

The usage method of CL-K is basically the same as that for adsorption resin HS. Fill a column (resin tower) with CL-K and inject the concentrate solution for treatment at a constant speed.

When considering treatment conditions, carry out tests in accordance with the test method for adsorption resin HS (Section 1-4 and Appendices 2 to 5) to determine optimum conditions. Also, when using them in combination as shown in Section 2-8 "Example of combined usage," we recommend that you conduct tests using multiple towers, by connecting the columns in series as shown in the figure in Appendix 3.

## 2-5 Conditioning

CL-K is an activated carbon, and fine powder of carbon will be produced as a result of the friction among particles when filling a column. When you use CL-K for the first time, be sure to carry out backwashing. Backwashing is normally carried out at around  $\text{LV} = 20 \text{ m/h}$ .

## 2-6 Regeneration method

Standard regeneration method of CL-K using alkaline solution is shown below.

Order	Operation	SV	Time	Purpose
1	Water driving	0.5	1	Collection of concentrate solution for treatment
2	Alkaline elution (3 to 5% NaOH)	1	2	Elution of adsorbed matter
3	Hot-water (water) washing	1 to 3	1 to 3	Elution of adsorbed matter and removal of alkali
4	Acid neutralization (1% HCl)	1 to 2	1 to 2	Neutralization
5	Water washing at room temperature	1 to 3	1 to 3	Removal of acids and salts

Table 6.

When suspended matter causes clogging of the activated carbon bed, carry out backwashing before alkaline elution (implement backwashing with an expansion rate of 30%). The elution of adsorbed matter will be improved if the water temperature is higher (50 to 80°C) and a longer time is taken. Basically follow the instructions above with conditions appropriate for the specific case.

During regeneration, alkaline wastewater containing adsorbed matter will be produced as

effluent. The total amount of regeneration wastewater will be around 10 to 25 times as much as the filled activated carbon volume per regeneration if a standard regeneration method is used. Please handle the produced wastewater in accordance with the relevant effluent standards.

## 2-7 Storage method

A minute amount of organic matter, which cannot be removed completely even although regeneration is carried out, is deposited in CL-K after usage. In particular, if CL-K is used in the food industry, micro organism may develop using this organic matter as its nutrient, contaminating CL-K. When storing CL-K, you must carry out regeneration and inject a small amount of water continuously without stopping. If continual injection is difficult, you may dip it in water and store it at pH 2 or less. In this case, however, slight deterioration may be caused by acid adsorption.

If you intend to use it again after storage, be sure to carry out regeneration before use.

## 2-8 Example of combined usage

### ○ Decolorization by combining adsorption resin HS and CL-K

As adsorption properties vary depending on the type of activated carbons — adsorption resins and ion-exchange resins — it is expected that the degree of purification will be improved depending on their combination. In combination with our adsorption resin HS and iron-removal resin PF\*, the recovery of CL-K's adsorption capacity by alkaline regeneration will be facilitated further.

The figure below shows an example of injection decolorization using adsorption resin HS and CL-K for the decolorization of monosodium glutamate.

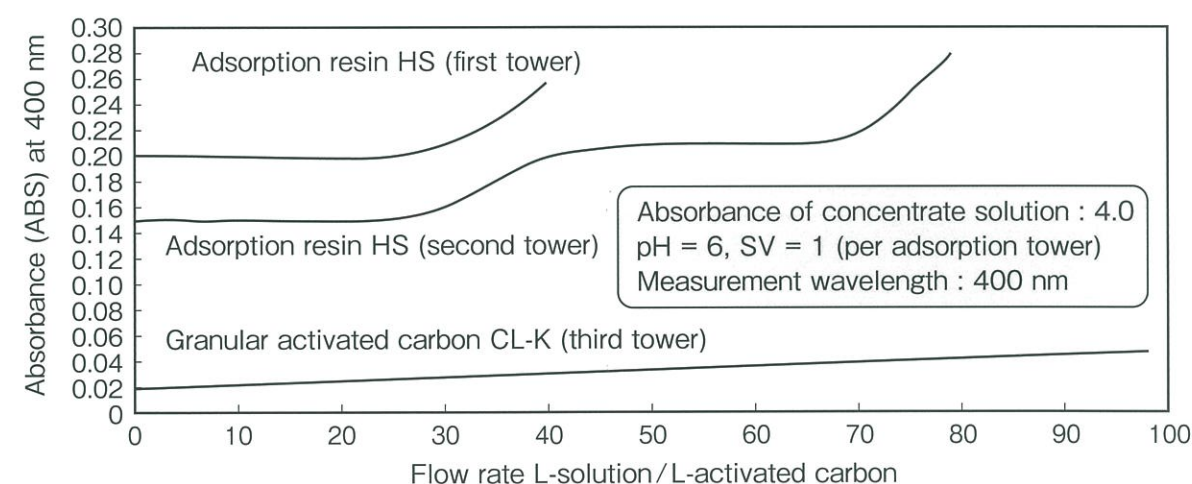


Fig. 8.

\*Iron-removal resin PF is a resin for removing iron from aqueous solutions. When colorization is caused by iron, if you use PF and remove iron in advance, the operation will become more efficient because the load on the decolorization process will be reduced. Request a catalog for details.

2-9 Water injection resistance and backwashing expansion rate

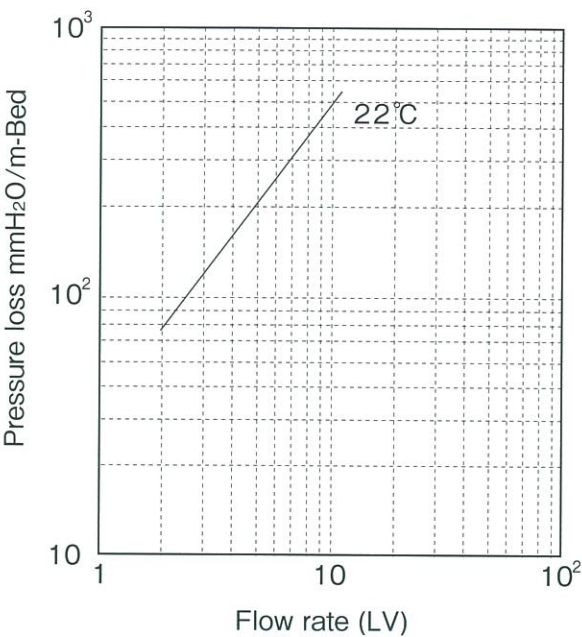


Fig. 9. CL-K's pressure loss

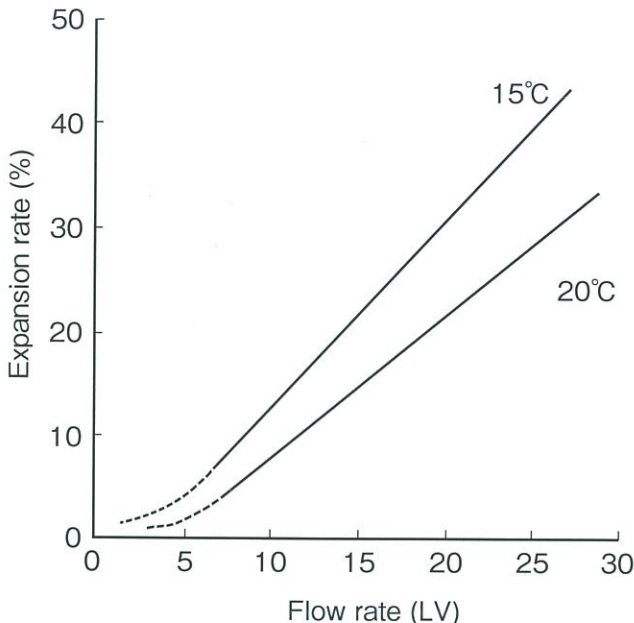


Fig. 10. Backwashing expansion rate

Appendix

1 Glossary of terms related to adsorption resins

Term	Meaning and description
Swelling rate	A value that represents the change in volume accompanied with the change in the characteristic of liquid. Where the volume of the resin when it is dipped in 1N HCl aqueous solution is $V_a$ and the volume of the resin when it is dipped in 1N NaOH aqueous solution is $V_b$ , the value can be obtained as follows : swelling rate (%) = $(V_b - V_a) / V_a$ . Normally if the swelling rate becomes higher, the resin's strength declines, so this value indicates the resin's time for replacement.
SV	This term stands for space velocity. It represents the amount of fluid run per hour to the volume of the column filling. For example, when fluid is run at 2 ml/min in a column filled with 60 ml of resin, 120 ml of fluid is flowed per hour, so $SV = 120 / 60 = 2 [h^{-1}]$ .
Flow rate (RV)	It represents how many times the fluid is run to the volume of the column filling. For example, when 1500 ml of fluid is run in a column filled with 100 ml of resin, $RV = 1500 / 100 = 15$ .
LV	This term stands for linear velocity. It represents the distance that fluid travels toward the injection direction in a unit of time. For example, when injection is carried out at 100 ml/h in a column with a cross-sectional area of 20 $cm^2$ , $LV = 100 / 20 = 5 \text{ cm/h}$ .

Table 7.

2 Batch adsorption test

○ Required equipment

Mortar, conical flask, rubber plug, filter paper, funnel and stirrer or shaker

1) Pulverization and measurement of the resin

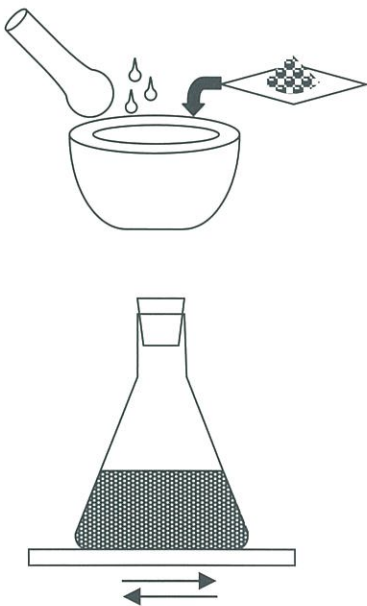
Place an appropriate amount of resin in a mortar, add a small amount of water and pulverize thoroughly.

Wrap the pulverized resin in filter papers and remove the surface moisture thoroughly.

Using a scale, take out a set amount (example : 5 g).

2) Adsorption test

Place the pulverized resin and 50 ml of concentrate solution for treatment in a 100 ml conical flask and seal it with a rubber plug. Stir it for an hour or agitate it for an hour at around 120 rpm (This is normally conducted at room temperature).



### 3) Filtration and analysis

Carry out filtration using a filter paper that does not let the resin powder pass.

Dispose of the initial flow (about 10 ml) of filtrate and use the subsequent filtrate for analysis.

When conducting a decolorization test, generally, absorbance is measured using a spectrophotometer (if the color is dark, measurement is carried out after diluting and the measured result is multiplied by the dilution rate).

### 4) Calculation

Based on the result of 3), determine whether the resin can be used for the decolorization of the concentrate solution.



### <Q&A>

**Q-1** Why do you add water before pulverization?

**A-1** When the adsorption resin is dry, the resin's porosity will be lost, resulting in a significant decrease in adsorption performance. Therefore, you should pulverize it after adding water to prevent it from drying out, remove excess moisture later, and measure its weight.

**Q-2** We have implemented decolorization using an activated carbon. How can we compare the resin's performance with the activated carbon's performance?

**A-2** As the adsorption resin contains about 70% of moisture, the amount of resin added in a decolorization test cannot be compared with that of other adsorbents without modification. When comparing the resin with dry adsorbents such as activated carbons, use a value calculated by multiplying the resin weight by 0.3 (dry weight).

**Q-3** Why do you dispose of the initial 10 ml of filtrate when carrying out filtration?

**A-3** During the initial stage of filtration, the pigments are adsorbed onto the filter paper, so the color of the initial filtrate will be lighter than that of the actual filtrate. This operation is carried out to prevent overestimation of adsorption performance.

**Q-4** What is the acceptable extent of decolorization?

**A-4** The extent of decolorization varies depending on the concentrate solution, so it is not appropriate to make a sweeping judgment, but for example, in case of 50 ml of soy sauce, if 5.5 g of resin (15 v/v%) is added, the absorbance at 530 nm will be reduced from 5.9 to around 1.3.

**Q-5** Even if we conduct an adsorption test, we cannot achieve the desired chromaticity. What should we do?

**A-5** If your desired absorbance is 0.5 or less, a large amount of resin may be required for decolorization. In this case, as explained in Section 1-3, consider combined usage of an activated carbon that is good at decolorization of light-colored solutions. As CL-K specified in this catalog is regenerable with chemical solutions, it can share the same equipment as the adsorption resin.

### 3 Injection test (small column)

○ Required equipment

Column (made of glass and tapered, bore diameter: 10 to 30 mm, height: about 1 m), stand, funnel, tubes, beaker, graduated cylinder and metering pump (fraction collector).

Schematic diagram of equipment for an injection test is shown below.

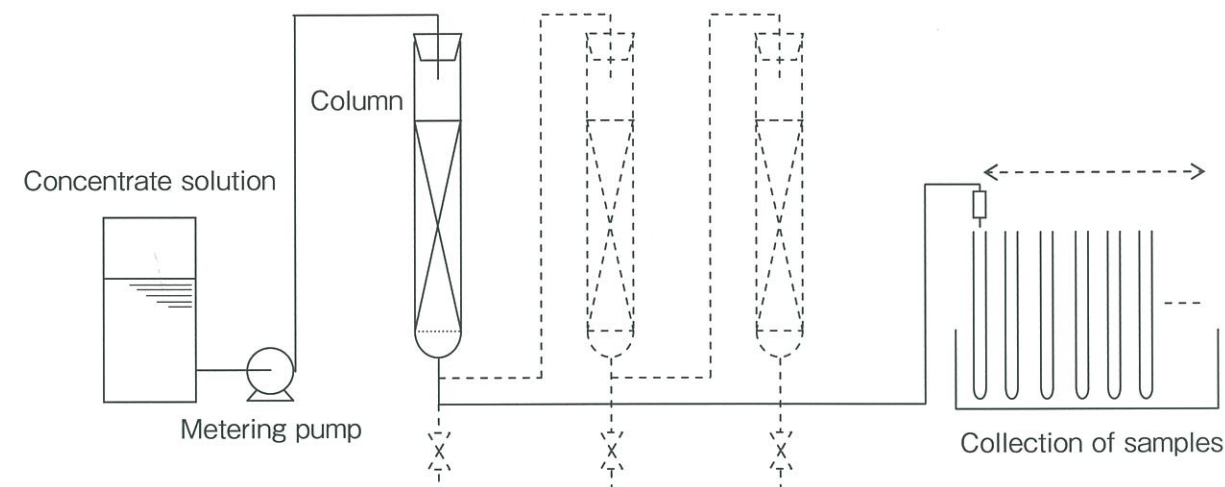
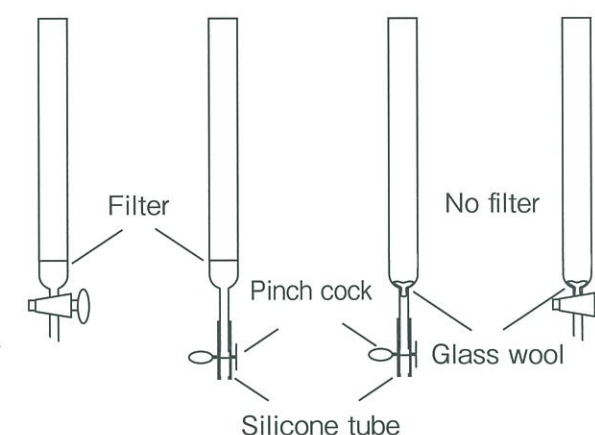


Fig. 11. Column injection test equipment diagram (dotted lines show usage of multiple towers)

#### 1) Column preparation

Place the column vertically on a stand. If the column has a filter, use it as is, but if the column does not have a filter, place glass wool or absorbent cotton at the exit to prevent the resin grains from passing through. If there is no cock, fit a silicone tube over the thin tube at the exit, and clip the tube using a pinch cock.



#### 2) Calculation of required resin amount

Resin should be filled to a half or two-thirds the height of the column. Calculate the required resin amount (volume) based on bore diameter and filling height.

Example: the resin amount (volume) when filling a column with a bore diameter of 2 cm for a height of 50 cm will be  $1 \times 1 \times \pi \times 50 = 157$  ml.

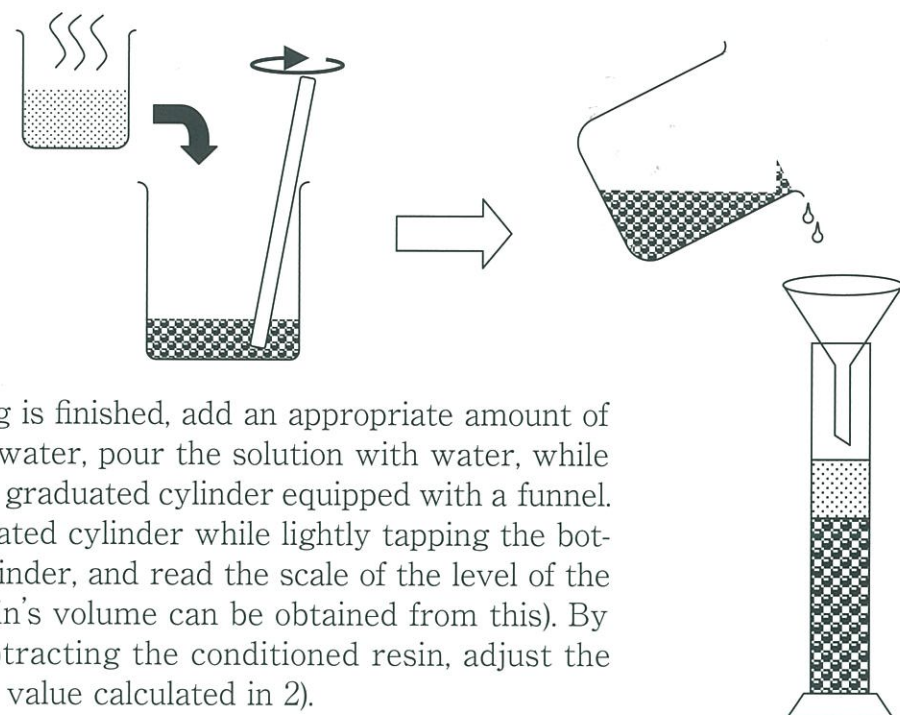
#### 3) Calculation of injection speed

Injection speed varies depending on the concentrate solution for treatment, but normally the test is conducted with an SV of 0.5 to 1 (for deodorization, SV = 2). In general, if the SV is smaller, the contact time is longer, so the efficiency of decolorization will be improved, but the treatment amount per unit of time will decrease.

Where the resin filling amount is 150 ml and SV = 0.5, the injection speed will be 75 ml/h or 1.25 ml/min.

#### 4) Simple conditioning and measurement of the resin

Place an amount of resin slightly larger than the resin amount calculated in 2) in a beaker, add hot water, and stir well. Fine particles will float to the surface, so remove them with hot water by decantation. Next, add tap water and wash the resin several times in the same manner.



When washing is finished, add an appropriate amount of ion-exchange water, pour the solution with water, while stirring, into a graduated cylinder equipped with a funnel. Fill the graduated cylinder while lightly tapping the bottom of the cylinder, and read the scale of the level of the resin (the resin's volume can be obtained from this). By adding or subtracting the conditioned resin, adjust the volume to the value calculated in 2).

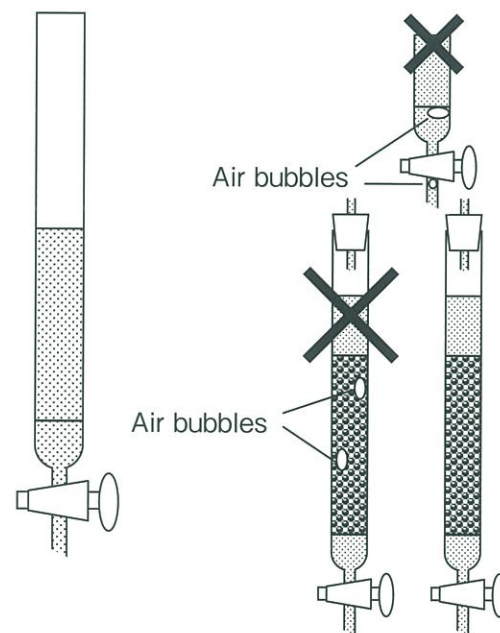
#### 5) Filling the column

Pour the ion-exchange water into the column of 1) up to about half its height.

Do not leave any air at the bottom of the filter or the exit tube (when air remains, move water from the bottom to remove the air).

Next, place a funnel at the top of the column, gradually pour the resin measured in 2) with ion-exchange water, while stirring. You have to be careful not to let the water and resin overflow and not let air bubbles get into the resin layer.

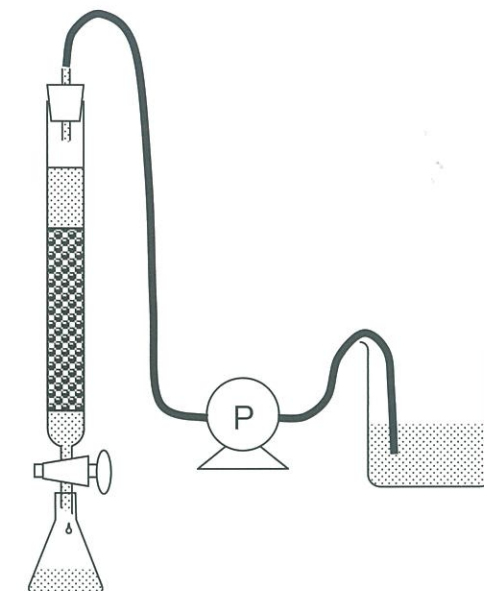
When the amount of water becomes too great, open the cock and discharge water accordingly, and be careful to leave a water layer at all times over the top of the resin. When a full amount of resin is filled, discharge water so that the height of the water layer over the resin becomes 5 to 10 cm.



#### 6) Preparation of injection

Pour ion-exchange water into a beaker and connect tubes as shown in the figure. After putting a rubber plug into the top of the column, open the cock at the bottom and operate the pump.

Next, stop the pump and check for leaks (when the pump is stopped, the discharge of fluid will stop after a few drops).



#### 7) Adjustment of injection speed

Start the pump again and collect the fluid discharged from the exit for a set period of time to obtain the current injection speed based on that value. Adjust the speed by tuning the pump's volume to the value calculated in 3).

#### 8) Implementation of injection

Replace the beaker content with the concentrate solution for treatment and start injection. At the same time, start collecting the discharged fluid by increments (automatic collection is possible if you have a fraction collector). However, measurement should be carried out from the moment when the water remaining inside the column is completely discharged. When precision is required, measure values such as salt concentration and refractivity, and check that the content is virtually replaced with the concentrate solution.

#### 9) Completion of injection

Complete the process as the color tone of the discharged fluid becomes darker gradually and when its value obviously becomes several times greater than the target value.

#### 10) Analysis

Measure the color tone of the discharged fluid using a spectrophotometer. Indicate the injection amount using flow rate (RV) and graph out the relation of the color tone of the discharged fluid.

In actuality, you should seek optimum conditions by repeating the test above at different temperatures and flow rates. If you wish to change the treatment temperature, use a column with a jacket, and run warm water or cold water outside the column at desired temperature.

## <Q&A>

**Q-1** We cannot prepare for a column.

**A-1** As this test also serves as a test for the plant engineering design, it is desirable that you conduct the test using a column, but if you absolutely cannot, you can carry out the test by using a transparent plastic pipe made of materials such as acrylic, blocked with absorbent cotton at one end, and fitted with rubber plugs with glass pipes at both ends, as shown in the figure to the right.

However, as fluid may accumulate at the bottom, you cannot expect the precise test results compared with a test using a column.

If you also implement a regeneration test, use materials whose chemical resistance has been confirmed. If you cannot obtain these tools either, please consult us.

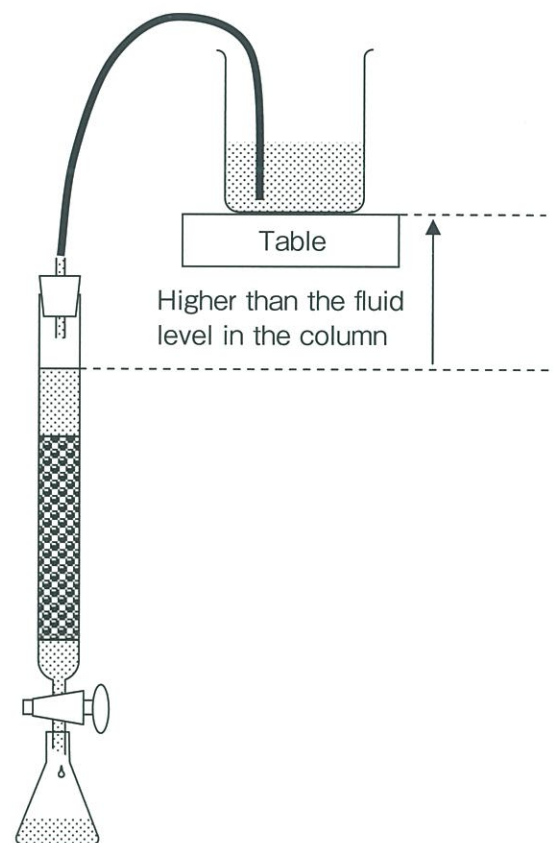


**Q-2** We cannot provide a metering pump.

**A-2** If a metering pump is unavailable, let the fluid drop under its own weight.

In Step (6) "Preparation of injection," as shown in the figure to the right, after inserting a rubber plug into the top of the column, place the beaker at a position that is higher than the top surface of the resin, and open the cock at the bottom of the column.

Adjust the flow velocity mainly using the restriction of the cock. In the case of a pinch cock, use a pinch cock whose tightness can be adjusted.



**Q-3** Roughly speaking, when should we start the measurement?

**A-3** In the initial stage of injection, ion-exchange water accumulated in the column will be discharged. Therefore, if you know the amount of this ion-exchange water, you will be able to know when to start the measurement. As the amount of water in the section filled with resin is about a half of the volume of the entire filled section, you can obtain the total amount of discharge irrelevant to the measurement by adding this amount of water to the amount of water in the column and the tube. However, as this is just a rough guide, when precision is required, measure values such as salt concentration and refractivity and check that the content is replaced with concentrate solution.

**Q-4** What should we do to reduce the amount of the light-colored solution that is discharged in the initial stage?

**A-4** If there is a large amount of water above the resin, the amount of light-colored solution

will become greater. In order to reduce this, we recommend that you:

① Remove the rubber plug at the top of the column and reduce the water inside the column to a level slightly higher (0.5 to 1 cm) than that of the resin, and close the cock at the bottom.

② Replace the water inside the tube that sends the concentrate solution for treatment. Operate the pump to run the concentrate solution for treatment and stop the pump when the concentrate solution comes out from the tip of the tube.

③ Slowly add the concentrate solution for treatment over the resin using a dropper. Continue this process until the height of the concentrate solution layer reaches 5 to 10 cm, and reinsert the rubber plug.

④ Open the cock at the bottom and check if there is any leakage of fluid as in (6) above. When the operations above have been finished, start the injection. However, even if you complete the operations above, as there is an impact of water included in the resin, it is impossible to eliminate the light-colored solution completely.

**Q-5** What is the desirable extent of decolorization?

**A-5** The extent of decolorization varies depending on the concentrate solution for treatment, so it is not appropriate to make a sweeping judgment. However, as a guide, when considering the average chromaticity of the discharged fluid, if the flow rate that exceeds the desired chromaticity in a plotted graph is  $RV = 10$  or more, we recommend that you use an adsorption resin. Also in cases where the flow rate falls below this value, efficient decolorization may be possible if you combine the resin with activated carbon.

## 4 Regeneration test (small column)

○ Required equipment

Same as the injection test (small column)

○ Required reagent

Acid (dilute hydrochloric acid, etc.) and alkali (3 to 5% aqueous sodium hydroxide, etc.)

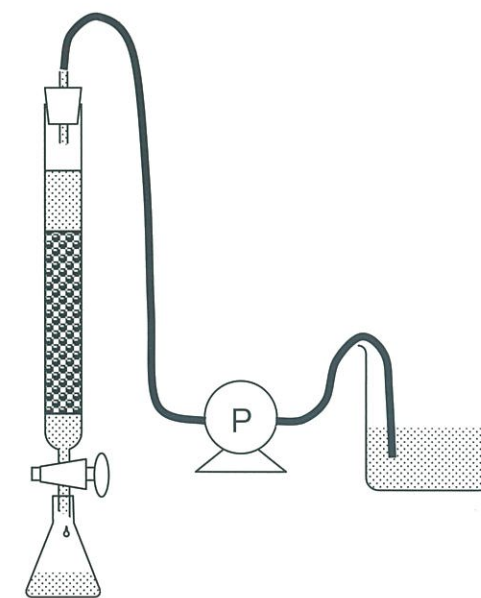
You can carry out this regeneration test immediately after you complete the injection test in the previous section.

### 1) Regeneration

Utilize the column used in the injection test and carry out injection in accordance with the standard regeneration method specified in Table 3 on Page 4.

Place the reagent to be injected in a beaker, adjust the pump's output to achieve the designated flow rate, and implement injection.

Continue collecting the discharged fluid by increments.



## 2) Analysis

Measure the chromaticity and pH of the collected discharged fluid and plot the data as shown in the figure below.

As color is susceptible to the solution's pH, normally you should measure the pH first and then add acid or alkali to adjust the pH so that it becomes the same as that of the concentrate solution, and measure the chromaticity.

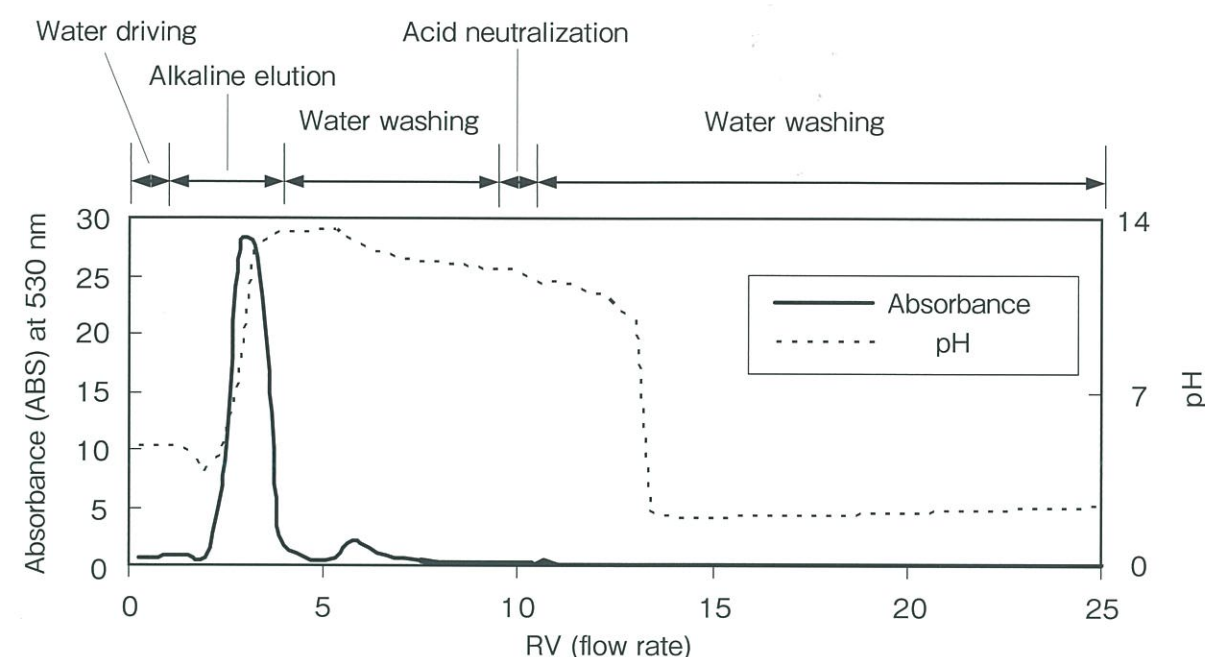


Fig. 12. Example of plotted data of discharged fluid during regeneration

## 3) Refilling

After regeneration is finished, carry out refilling instead of the normal backwashing operation.

Remove equipment such as rubber plugs, turn the column upside down over a receiver such as a 1 L beaker, run water slowly from the column exit, and pour the resin into the receiver.

Add purified water to the resin collected in the receiver, stir it, wait for the resin to sink, and dispose the supernatant solution as in Section 3-4) (if there is suspended matter, repeat this operation until it is eliminated).

Next, pour the full amount into the column as in Section 3-5) and fill the column.

## 4) Adsorption test

Conduct an injection test again after refilling and measure the adsorption power.

By changing the regeneration temperature and the flow rate, you can seek optimum conditions.

## <Q&A>

**Q-1** Why do we collect and analyze the discharged fluid?

**A-1** You should measure the chromaticity to check if the elution of adsorbed matter has been carried out efficiently. Also, you should measure the pH to check whether a pH is achieved where alkaline elution is carried out properly, and whether the desired pH is recovered when regeneration is finished.

**Q-2** Why do we carry out refilling?

**A-2** When regeneration is carried out, the resin swells slightly and then shrinks, so blocking of resin grains may be caused, thereby bringing about a tendency to channeling. In order to prevent this, backwashing is implemented in the end of regeneration, but implementing backwashing in a small column is difficult because of problems related to the column length and the pump. Therefore, refilling is carried out to achieve effects similar to those of back-washing.

## 5 Scale-up test

Carry out scale-up based on the data of 3. Injection test and 4. Regeneration test.

Use 1 to 5 liters of resin, set up equipment as shown in Fig. 5 on Page 6, and implement Tests 3. and 4. Arrange the column so that the height of the resin layer is 1 m or more.

Regarding the injection, downflow (flowing from up to down) as well as upflow are possible. Also, in order to facilitate regeneration, we recommend that you set up the equipment in a manner that enables you to carry out backwashing.

## Product list

Activated Carbon Division		
Classification		Application
Powdered activated carbons	Wood carbon	Purification of pharmaceuticals, amino acids, brewing chemicals and industrial chemicals, and city water and wastewater treatment
	Coconut carbon	City water and wastewater treatment
	Coal carbon	Dioxin removal
Granular activated carbons <For liquid phase>  <For gas phase>	Wood carbon	Removal of colloidal substance
	Coconut carbon	Water clarification, dechlorination of tap water and farming applications
	Coal carbon	Purification of industrial chemicals and foods, and city water and wastewater treatment
	Coconut carbon	Deodorization treatment
	Granulated carbon	Deodorization, solvent recovery and dioxin treatment
	Functional activated carbon	Adsorption and removal of various offensive odor substances
Synthetic adsorbents	Adsorption resin	Decolorization and deodorization of sugars, amino acids, soy sauce and pharmaceuticals
	Iron-removal resin	Removal of iron in refined sake, soy sauce and amino acids
	Chelate resin	Treatment of mercury and other heavy metals
Plant-related		Decolorization and deodorization equipment and wastewater and exhaust gas treatment equipment Solvent recovery equipment and mercury and other heavy metal removal equipment Design and installation of water treatment equipment

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