# **Magnetic Separation of Metal Ions**

# K. Chie,† M. Fujiwara,\*,‡ Y. Fujiwara,† and Y. Tanimoto\*,‡

Graduate School of Science, Hiroshima University, Kagamiyama, Higashi-Hiroshima 739-8526, Japan, and Institute for Molecular Science, Myodaiji, Okazaki 444-8585, Japan

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The magnetic separation was investigated for  $\text{Co}^{2+}$  (9500  $\times$  10<sup>-6</sup> cm³ mol<sup>-1</sup>) and  $\text{Fe}^{3+}$  (14600  $\times$  10<sup>-6</sup> cm³ mol<sup>-1</sup>) ions and for  $\text{Cr}^{3+}$  (6200  $\times$  10<sup>-6</sup> cm³ mol<sup>-1</sup>) and  $\text{Al}^{3+}$  ( $-2 \times 10^{-6}$  cm³ mol<sup>-1</sup>) ions. The metal ion solutions were spotted on a silica gel support, and exposed to a magnetic field of 410 kOe² cm<sup>-1</sup> intensity  $\times$  gradient. The  $\text{Co}^{2+}$  ions move farther toward the maximum field than the  $\text{Fe}^{3+}$  ions. The result is explained by the fact that the  $\text{Fe}^{3+}$  ions are adsorbed more strongly on the silica gel surface than the  $\text{Co}^{2+}$  ions. The  $\text{Cr}^{3+}$  ions move farther toward the field center than the  $\text{Al}^{3+}$  ions. This occurs because the  $\text{Cr}^{3+}$  ions are attracted more strongly by the magnetic force than the  $\text{Al}^{3+}$  ions. It is demonstrated that the separation makes effective use of the adsorption activities as well as the magnetic susceptibilities.

## 1. Introduction

In order for particles to be separated by a magnetic field, it is necessary that the magnetic force acting on the particles is more effective than the thermal diffusion induced by a solvent atmosphere. Therefore, the separation has been believed to be possible, when the particles possess larger size so that the magnetic energy is larger than the thermal energy. Many studies have been focused on the separation of paramagnetic particles (>1.0  $\mu$ m size).

Recently, we have succeeded in the separation of  $Cu^{2+}$  (1500  $\times$   $10^{-6}~cm^3~mol^{-1})^1$  and  $Ag^+$   $(-24\times10^{-6}~cm^3~mol^{-1})^2$  ions in a magnetic field. It may be useful to develop the method of separating ions by the magnetic force, since ions have a wide range of paramagnetic (<20000  $\times$   $10^{-6}~cm^3~mol^{-1})$  and diamagnetic (>  $-40\times10^{-6}~cm^3~mol^{-1})$  susceptibilities.

The preset paper deals with the separation of  $Co^{2+}$  (9500  $\times$   $10^{-6}~cm^3~mol^{-1})^1$  and  $Fe^{3+}~(14600\times10^{-6}~cm^3~mol^{-1})^1$  ions and of  $Cr^{3+}~(6200\times10^{-6}~cm^3~mol^{-1})^1$  and  $Al^{3+}~(-2\times10^{-6}~cm^3~mol^{-1})^2$  ions in a magnetic field. The effect of adsorption is discussed for a silica gel support on which the metal ion solutions are spotted.

# 2. Experiment

The magnetic field was applied with a superconducting magnet (Oxford Spectromag 1000). The field direction was horizontal. The field intensity was 80 kOe at the field center, and the field intensity  $\times$  gradient was 410 kOe $^2$  cm $^{-1}$  at  $\pm65$  mm from the field center.

As the metal salts, AlCl<sub>3</sub>·6H<sub>2</sub>O (Wako, >98%), CrCl<sub>3</sub>·6H<sub>2</sub>O (Wako, >99%), FeCl<sub>3</sub>·6H<sub>2</sub>O (Wako, >99%), CoCl<sub>2</sub>·6H<sub>2</sub>O (Wako, >99%), Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O (Wako, >99%), and AgNO<sub>3</sub> (Wako, >99%) were used. The metal ion solutions (1.0 mol dm<sup>-3</sup>) were prepared by dissolving the corresponding metal salts in deionized water. The silica gel support (Wako C-200, 75–150  $\mu$ m, 27 g) was saturated with deionized water (36 cm<sup>3</sup>), and laid in a glass vessel (390 × 40 × 10 mm). For the

separation experiment, the test solutions were made by mixing (1/1) the  $Al^{3+}$  and  $Cr^{3+}$  ion solutions and by mixing (1/1) the  $Fe^{3+}$  and  $Co^{2+}$  ion solutions. The test solutions (50 mm³) were spotted on the silica gel support at  $\pm 100$  mm from the field center. The glass vessel was placed in the magnet bore (374 × ø 50.4 mm), allowed to stand at 295 K for 4 h, and then taken out of the magnet bore. For the adsorption experiment, the test solutions were made by diluting (1/1) the metal ion solutions with deionized water. The test solutions (50 mm³) were spotted on the silica gel support, and allowed to stand under zero field at 295 K for 4 h.

The  $Al^{3+}$  ions were colored red  $(Al(C_{22}H_{13}O_9)_3)$  by a spray of 0.1% ammonium aurintricarboxylate (Wako) solution, and the Fe<sup>3+</sup> ions brown (Fe(C<sub>9</sub>H<sub>6</sub>ON)<sub>3</sub>) by a spray of 0.5% 8-quinolinol (Wako, >99%) ethanol solution. The Cr<sup>3+</sup> ions were colored green (Cr(OH)<sub>3</sub>), and the Co<sup>2+</sup> and Cu<sup>2+</sup> ions blue ([Co(NH<sub>3</sub>)<sub>6</sub>]<sup>2+</sup> and [Cu(NH<sub>3</sub>)<sub>4</sub>]<sup>2+</sup>) by a spray of ammonia solution (Kanto, 28–30%). The Ag<sup>+</sup> ions were colored gray (Ag metal) by irradiation of an ultrahigh-pressure mercury lamp (Ushio UI-501C, 500 W). The distributions of the metal ions were recorded with a camera (Nikon Nikomat FTN) on photographs, which were copied with a scanner (Epson GT-5000ART) into a personal computer (Apple 7600/132). The color-density profiles of the metal ions were analyzed with an image-processing program (NIH Image 1.55).

### 3. Results and Discussion

3.1. Separation of Co<sup>2+</sup> and Fe<sup>3+</sup> Ions and of Cr<sup>3+</sup> and Al<sup>3+</sup> Ions. The separation in the magnetic field was studied for  $\text{Co}^{2+}$  (9500  $\times$  10<sup>-6</sup> cm<sup>3</sup> mol<sup>-1</sup>)<sup>1</sup> and Fe<sup>3+</sup> (14600  $\times$  10<sup>-6</sup> cm<sup>3</sup> mol<sup>-1</sup>)<sup>1</sup> ions and for  $\text{Cr}^{3+}$  (6200  $\times$  10<sup>-6</sup> cm<sup>3</sup> mol<sup>-1</sup>)<sup>1</sup> and Al<sup>3+</sup> (-2  $\times$  10<sup>-6</sup> cm<sup>3</sup> mol<sup>-1</sup>)<sup>2</sup> ions. The metal ion solutions were spotted on the silica gel support at 100 mm from the field center, and exposed to the magnetic field of 410 kOe<sup>2</sup> cm<sup>-1</sup> intensity  $\times$  gradient.

3.1.1. The Distributions. The distributions  $[c_M(z)]$  of the  $Co^{2+}$  and  $Fe^{3+}$  ions measured in the field direction [z] from the spot position are shown in Figures 1a and 2a. The  $Co^{2+}$  ions move to a larger distance toward the field center than the  $Fe^{3+}$  ions. The average moving distances  $[\int_{-\infty}^{\infty} z c_M(z) \ dz/\int_{-\infty}^{\infty} c_M(z) \ dz]$ 

<sup>\*</sup> Corresponding authors. E-mail: fujiwara@sci.hiroshima-u.ac.jp; tanimoto@ims.ac.jp.

<sup>†</sup> Graduate School of Science, Hiroshima University.

<sup>&</sup>lt;sup>‡</sup> Institute for Molecular Science.

are 58 mm for the Co<sup>2+</sup> ions and 27 mm for the Fe<sup>3+</sup> ions. When the whole area is divided with a line at 45 mm, the purity of the  $Co^{2+}$  ions is  $\sim$ 82 mol % in the region of >45 mm, and the purity of the Fe<sup>3+</sup> ions is  $\sim$ 82 mol % in the region of <45 mm. It is noted that the separation of the Co<sup>2+</sup> and Fe<sup>3+</sup> ions is

The distributions of the Cr3+ and Al3+ ions are given in Figures 1b and 2b. The Cr<sup>3+</sup> ions move with sharp width toward the field center, while the Al<sup>3+</sup> ions follow them with tailing. The average moving distances are 57 mm for the Cr<sup>3+</sup> ions and 34 mm for the Al<sup>3+</sup> ions. The purity of the Cr<sup>3+</sup> ions is  $\sim$ 77 mol % in the region of >51 mm, and the purity of the  $Al^{3+}$ ions  $\sim$ 77 mol % in the region of <51 mm. The separation of the Cr<sup>3+</sup> and Al<sup>3+</sup> ions is possible.

The separation of the  $Cu^{2+}$  (1500  $\times$  10<sup>-6</sup> cm<sup>3</sup> mol<sup>-1</sup>)<sup>1</sup> and  $Ag^+$   $(-24 \times 10^{-6} \text{ cm}^3 \text{ mol}^{-1})^2$  ions has been reported recently.<sup>3</sup> The  $Cu^{2+}$  ions move by  $\sim$ 48 mm toward the field center, but the Ag<sup>+</sup> ions stay at the spot position. The purity of the Cu<sup>2+</sup> ions is  $\sim$ 90 mol % in the region of >10 mm, and the purity of the  $Ag^+$  ions  $\sim 90$  mol % in the region of < 10 mm.

3.1.2. Magnetic Movement. The movement of the  $Co^{2+}$  (9500  $\times 10^{-6} \, \text{cm}^3 \, \text{mol}^{-1})^1$ ,  $\text{Cr}^{3+} \, (6200 \times 10^{-6} \, \text{cm}^3 \, \text{mol}^{-1})^1$ , and  $\text{Cu}^{2+}$  $(1500 \times 10^{-6} \text{ cm}^3 \text{ mol}^{-1})^1$  ions is understood by the magnetic force acting on these paramagnetic metal ions. Since the magnetic force works on the paramagnetic metal ions in the direction where the field intensity increases, they are attracted toward the field center.

The observation that the Fe $^{3+}$  (14600  $\times$  10 $^{-6}$  cm $^3$  mol $^{-1}$ ) $^1$ ions move to a smaller distance in the magnetic field than the Co<sup>2+</sup> ions is not explained by the fact that the Fe<sup>3+</sup> ions have a larger paramagnetic susceptibility than the Co<sup>2+</sup> ions. The Fe<sup>3+</sup> ions must be attracted more strongly by the magnetic force than the Co<sup>2+</sup> ions. The discrepancy between the movement and susceptibility for the Fe3+ ions is resolved, if the effect of the adsorption of the silica gel support is taken into account (section 3.2). The Fe<sup>3+</sup> ions are adsorbed more strongly on the support, and cannot move to a larger distance in the field than the  $Co^{2+}$  ions.

The movement of the Al<sup>3+</sup>  $(-2 \times 10^{-6} \text{ cm}^3 \text{ mol}^{-1})^2$  ions in the magnetic field seems to be curious, because they are diamagnetic. When only the Al<sup>3+</sup> ions are exposed to the field, they do not move. However, it has been shown that ions with a paramagnetic susceptibility move by the magnetic force, not as a single particle, but as a large group composed of the paramagnetic ions and water molecules.4 When the group is formed out of the Cr<sup>3+</sup> ions, Al<sup>3+</sup> ions, and water molecules, it becomes paramagnetic as a whole and is attracted toward the field center.

The result that the Ag<sup>+</sup>  $(-24 \times 10^{-6} \text{ cm}^3 \text{ mol}^{-1})^2$  ions stay at the spot position in the magnetic field is understandable for two reasons. First, the Ag+ ions have a small diamagnetic susceptibility, and are not affected by a weak magnetic force.<sup>3</sup> Second, the Ag<sup>+</sup> ions are adsorbed most strongly on the silica gel support, and do not move in any direction (section 3.2).

3.2. Adsorption on Silica Gel Support. The adsorption on the silica gel support was studied for the Cu<sup>2+</sup>, Ag<sup>+</sup>, Co<sup>2+</sup>, Fe<sup>3+</sup>, Cr<sup>3+</sup>, and Al<sup>3+</sup> ions. The metal ion solutions were spotted on the silica gel support, and allowed to stand at zero field. The distributions  $[c_D(r)]$  of the Cu<sup>2+</sup>, Ag<sup>+</sup>, Co<sup>2+</sup>, Fe<sup>3+</sup>, Cr<sup>3+</sup>, and  $Al^{3+}$  ions measured in the radial direction [r] are shown in Figures 3 and 4.

3.2.1. Thermal Diffusion and Magnetic Movement. The Ag<sup>+</sup> ions are adsorbed most strongly in Figure 4 on the silica gel support, and do not leave the spot position. The average diffusion distances  $\left[\int_0^\infty rc_D(r) \ 2\pi r \ dr/\int_0^\infty c_D(r) \ 2\pi r \ dr\right]$  are 6.4 mm

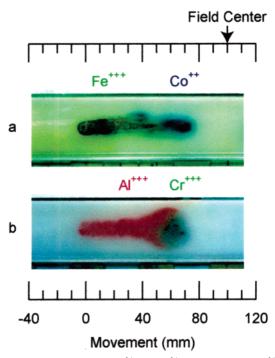


Figure 1. Separation of (a) Co<sup>2+</sup> and Fe<sup>3+</sup> ions and of (b) Cr<sup>3+</sup> and Al<sup>3+</sup> ions on silica gel support. The metal ion solutions were spotted at the 0 mm position which was 100 mm from the field center.

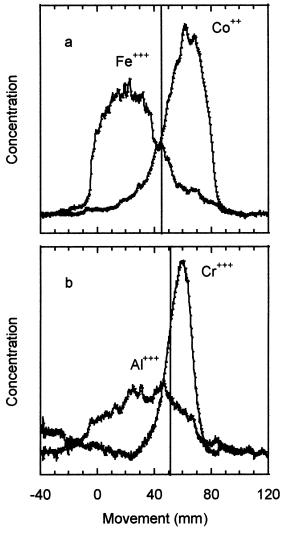
for the Ag<sup>+</sup> ions and 12.2 mm for the Cu<sup>2+</sup> ions. The finding is consistent with the fact that the Ag+ ions stay at the spot position, when the Cu<sup>2+</sup> ions move toward the maximum field.<sup>3</sup>

The Fe<sup>3+</sup> ions are adsorbed more strongly on the silica gel support and diffuse less easily by the thermal fluctuation than the Co<sup>2+</sup> ions. The average diffusion distances are 11.1 mm for the Fe<sup>3+</sup> ions and 14.9 mm for the Co<sup>2+</sup> ions. The result accounts for the observation that the Fe<sup>3+</sup> ions move to a smaller distance in the magnetic field than the Co<sup>2+</sup> ions (Figure 2), though the Fe<sup>3+</sup> ions have a larger paramagnetic susceptibility than the Co<sup>2+</sup> ions. It should be mentioned that, in the separation of the Co<sup>2+</sup> and Fe<sup>3+</sup> ions, the adsorption on the silica gel support is more effective than the attraction by the magnetic field.

The Al<sup>3+</sup> ions are not adsorbed strongly on the silica gel support, and diffuse most easily in Figure 4. The average diffusion distances are 16.2 mm for the Al<sup>3+</sup> ions and 11.5 mm for the Cr<sup>3+</sup> ions. This explains why the diamagnetic Al<sup>3+</sup> ions follow the Cr<sup>3+</sup> ions easily in the magnetic field (Figure 2).

3.2.2. Adsorption Activities and Magnetic Susceptibilities. The smaller diffusion distances [Ag<sup>+</sup> (6.4 mm)  $\leq$  Fe<sup>3+</sup> (11.1 mm) < Al<sup>3+</sup> (16.2 mm)] by the stronger adsorption in Figure 4 correspond to the smaller moving distances [Ag<sup>+</sup> ( $\sim$ 0 mm) <  $Fe^{3+}$  (27 mm)  $\leq Al^{3+}$  (34 mm)] by the magnetic force in Figure 2 and ref 3. Surprisingly, the paramagnetic Fe<sup>3+</sup> ions move to a smaller distance in the magnetic field than the diamagnetic Al<sup>3+</sup> ions, though the Co<sup>2+</sup> ions (which are followed by the Fe<sup>3+</sup> ions) move to a larger distance than the Cr<sup>3+</sup> ions (followed by the Al<sup>3+</sup> ions). The adsorption pulls the Fe<sup>3+</sup> ions back from the Co<sup>2+</sup> ions. The result would be extended to general applications; the silica gel support is effective to separate paramagnetic ions with different adsorption activities in the magnetic field.

Although the Al<sup>3+</sup> ions are adsorbed less strongly on the silica gel support than the Cr3+ ions in Figure 4, the diamagnetic Al3+ ions move to a smaller distance in the magnetic field than the paramagnetic Cr<sup>3+</sup> ions in Figure 2. As a result, the Al<sup>3+</sup> ions are separated from the Cr3+ ions by the magnetic force. The observation makes a marked difference from the one in the usual



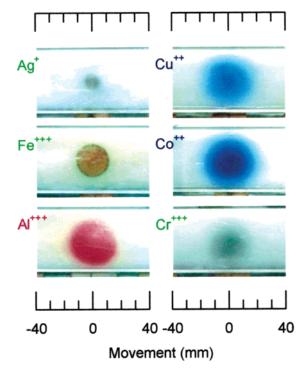
**Figure 2.** Distributions of (a)  $Co^{2+}$  and  $Fe^{3+}$  ions and of (b)  $Cr^{3+}$  and  $Al^{3+}$  ions on silica gel support.

adsorption chromatography, where the less-adsorbed molecules go farther than the more-adsorbed molecules. Generally, it is demonstrated that the magnetic field is effective to separate diamagnetic and paramagnetic ions, when the thermal diffusion of ions is suppressed by contact with the silica gel support.

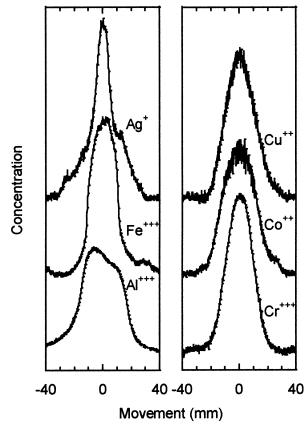
- **3.3. Relationship between Magnetic Movement and Thermal Diffusion.** The separation of the Co<sup>2+</sup> and Fe<sup>3+</sup> ions may be understood by the relationship between the magnetically moving distance and thermal-diffusion distance. The different mechanisms of motions are that the magnetic movement takes place as the motion of a group composed of metal ions and water molecules,<sup>4</sup> but that the thermal diffusion occurs as the motion of a single metal ion.
- 3.3.1. Expressions for the Distances. For a group composed of metal ions and water molecules, the distance z(t) of movement at time t in a magnetic field H(z) measures the frictional coefficient  $f_G$  (of metal ion—water molecule group) and magnetic force F(z), the latter of which is a function of a mole number n and molar susceptibility  $\chi$  of the metal ions.<sup>4</sup>

$$z(t) = \int_0^t [F(z)/f_G] dt$$
$$= \int_0^t (n\chi/f_G) [H(z)\partial H(z)/\partial z] dt$$
(1)

The parameter  $f_G/n\chi$  is calculated by numerical integration.



**Figure 3.** Adsorption of  $Cu^{2+}$ ,  $Ag^+$ ,  $Co^{2+}$ ,  $Fe^{3+}$ ,  $Cr^{3+}$ , and  $Al^{3+}$  ions on silica gel support. The metal ion solutions were spotted at the 0 mm position.



**Figure 4.** Distributions of Cu<sup>2+</sup>, Ag<sup>+</sup>, Co<sup>2+</sup>, Fe<sup>3+</sup>, Cr<sup>3+</sup>, and Al<sup>3+</sup> ions on silica gel support.

Approximately, the frictional coefficient  $f_G$  of a metal ion—water molecule group is inversely proportional to the distance z(t) of movement.

For a single metal ion, the mean distance  $\langle r(t) \rangle$  of diffusion at time t in two-dimensional space is a measure of the diffusion

coefficient D, which is related to the frictional coefficient  $f_S$  (of single metal ion) at temperature T.

$$\langle r(t)\rangle^2 = \pi Dt$$
  
=  $\pi k T t / f_S$  (2)

where k is the Boltzmann constant. The frictional coefficient  $f_S$  of a single metal ion is inversely proportional to the square of the mean distance  $\langle r(t) \rangle$  of diffusion.

In the magnetic movement of the  $Co^{2+}$  and  $Fe^{3+}$  ions, the parameter  $f_G/n\chi$  is estimated from the average moving distances in Figure 2. The denominator  $n\chi$  is assumed to be the same value for the  $Co^{2+}$  and  $Fe^{3+}$  ions, since they move as a group in the magnetic field. The ratio of the frictional coefficients between the  $Co^{2+}$  and  $Fe^{3+}$  ions is

$$f_{G,Co}/f_{G,Fe} = (f_{G,Co}/n\chi)/(f_{G,Fe}/n\chi)$$

$$= (0.84 \times 10^{15} \text{ m}^{-3} \text{ kg s}^{-1})/(1.66 \times 10^{15} \text{ m}^{-3} \text{ kg s}^{-1})$$

$$= 0.51$$

In the thermal diffusion of the  $Co^{2+}$  and  $Fe^{3+}$  ions, the parameter  $f_S/\pi kTt$  is given from the average diffusion distances in Figure 4. The ratio of the frictional coefficients between the  $Co^{2+}$  and  $Fe^{3+}$  ions is

$$f_{S,Co}/f_{S,Fe} = (f_{S,Co}/\pi kTt)/(f_{S,Fe}/\pi kTt)$$
  
=  $(14.9 \text{ mm})^{-2}/(11.1 \text{ mm})^{-2}$   
=  $0.55$ 

3.3.2. Separation of More- and Less-Adsorbed Ions. Suppose that a group (or sphere) contains more-adsorbed metal ions, less-adsorbed metal ions, and water molecules. When the group is moving in a magnetic field, it is attracted as a whole by the magnetic force.<sup>4</sup> The "friction" is considered to arise from the adsorption on silica gel particles.<sup>5</sup> Some part of the surface of the group is covered with the more-adsorbed ions. If the more-adsorbed surface is in contact with the silica particles, the friction is caused by the more-adsorbed ions. The more-adsorbed surface goes slowly and falls out from the group during the movement in the magnetic field. After the magnetic movement, the resultant distribution of the more-adsorbed ions reflects the friction of the more-adsorbed surface. The reverse holds also for the less-adsorbed ions.

The magnetic susceptibility  $n\chi$  for the group is assumed to be the sum  $n_1\chi_1 + n_2\chi_2$  for the component ions 1 and 2, since the group is attracted as a whole by the magnetic force. The frictional coefficient  $f_G$  for the part of the surface is expected to be proportional to the frictional coefficient  $f_S$  for the single ion, since the adsorption activity of the surface is similar to the adsorption activity of the single ion. Then, the expectation is in agreement with the above estimation of the ratios of the frictional coefficients for the  $\text{Co}^{2+}$  and  $\text{Fe}^{3+}$  ions. This indicates that the separation of the  $\text{Co}^{2+}$  and  $\text{Fe}^{3+}$  ions in a magnetic field is achieved by the adsorption on silica gel particles.

The separation of the Cr<sup>3+</sup> and Al<sup>3+</sup> ions may not be explained by the adsorption activities of the metal ions. The observation is that, though the Cr<sup>3+</sup> ions are adsorbed more strongly on silica gel particles than the Al<sup>3+</sup> ions, the paramagnetic Cr<sup>3+</sup> ions move to a larger distance in a magnetic field than the diamagnetic Al<sup>3+</sup> ions. When the Al<sup>3+</sup> ions are adsorbed on the silica particles, they may not be attracted by the magnetic force and may not follow the magnetic movement of the Cr<sup>3+</sup> ions.

#### 4. Conclusion

In the magnetic separation of metal ions, the adsorption activities play an effective role as well as the magnetic susceptibilities. The  $\text{Co}^{2+}$  (9500  $\times$   $10^{-6}$   $\text{cm}^3$   $\text{mol}^{-1}$ ) ions are separated from the Fe³+ (14600  $\times$   $10^{-6}$   $\text{cm}^3$   $\text{mol}^{-1}$ ) ions, since the Fe³+ ions are adsorbed more strongly on the silica gel support than the  $\text{Co}^{2+}$  ions. The  $\text{Cr}^{3+}$  (6200  $\times$   $10^{-6}$   $\text{cm}^3$   $\text{mol}^{-1}$ ) ions are separated from the Al³+ ( $-2\times10^{-6}$   $\text{cm}^3$   $\text{mol}^{-1}$ ) ions, because the Cr³+ ions are attracted more strongly by the magnetic field than the Al³+ ions.

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### **References and Notes**

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- (4) Fujiwara, M.; Chie, K.; Sawai, J.; Shimizu, D.; Tanimoto, Y. J. Phys. Chem. B, submitted.
- (5) The saying comes from the thought that, because the sphere ( $\sim$ 2.4  $\mu$ m size)<sup>4</sup> of metal ion—water molecule group makes a motion around the particle (75–150  $\mu$ m) of silica gel, the particle makes relatively the opposite motion around the sphere.