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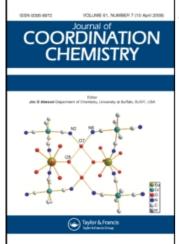
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Norio Koine^a

^a Department of Industrial Chemistry, Faculty of Engineering Ehime University, Matsuyama, Ehime, Japan

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PREPARATION OF A NEW STRONG ANION-EXCHANGER DERIVED FROM TOYOPEARL GEL AND ITS APPLICATION TO THE OPTICAL RESOLUTION OF NEUTRAL AND CATIONIC COBALT(III) COMPLEXES

NORIO KOINE

Department of Industrial Chemistry, Faculty of Engineering Ehime University, Bunkyo-cho, Matsuyama, Ehime 790, Japan (Received April 18, 1988)

Abstract A new anion-exchanger with a quarternary ammonium type of exchange group has been derived from Toyopearl (Fractogel TSK) gel. The complete optical resolution of mer-[Co(β -ala)₃] and [Co(en)₃]³⁺ was achieved on a column of [Sb₂(d-tart)₂]²⁻ form exchanger with much higher efficiency compared with the previous methods.

Keywords: Anion-exchanger, Toyopearl gel, quaternary ammonium

INTRODUCTION

Column chromatography is one of the most useful techniques for the optical resolution of neutral and cationic metal complexes in the preparative scale. 1,2 So far, commercial Sephadex ion-exchangers have been almost exclusively used as adsorbents, although d-tartrate derivatives of Sephadex or Toyopearl gel have been also applied successfully. 3 However, the separation efficiency of these exchangers is still unsatisfiable, because it takes a long elution time in excess of a day. The purpose of the present work is to develop an exchanger with higher resolution efficiency.

PREPARATION OF THE EXCHANGER

The anion-exchanger was prepared from Toyopearl HW-50S (20-40 μ m) according to the scheme below. The ion-exchange capacity was 0.47 mmol/ml for [Co(edta)] ion in H₂O. The exchanger of [Sb₂(d-tart)₂]²⁻ form was used for the present optical resolution.

OPTICAL RESOLUTION

The Toyopearl exchanger was applied to the optical resolution of two cobalt(III) complexes, $mer-[Co(\beta-ala)_3]$ and $[Co(en)_3]^{3+}$, of which the complete resolution has been already achieved chromatographically using other exchangers. The elution curves of the complexes are shown with the resolving conditions in Figures 1 and

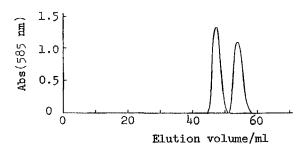


FIGURE 1 Elution curve of mer-[Co(β-ala)3].

Column: 1.0^{ϕ} x 70 cm ([Sb₂(d-tart)₂]²⁻ form) Sample: 25 mg, Eluent: 50 % (v/v) EtOH Flow rate: 0.12 ml/min (peristaltic pump)

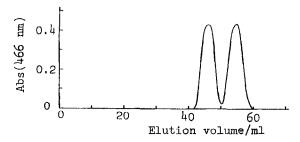


FIGURE 2 Elution curve of $[Co(en)_3]^{3+}$.

Column: 1.0° x 70 cm ([Sb₂(d-tart)₂]²- form) Sample: 20 mg, Eluent: 0.02 M [Sb₂(d-tart)₂]²- Flow rate: 0.17 ml/min (peristaltic pump)

2. As seen in Figure 1, 25 mg of $mer-[Co(\beta-ala)_3]$ was completely resolved in 8.2 h from the beginning of the elution. This complex has been completely resolved by the same method as the present one using QAE-Sephadex A-25 exchanger. 4 The time required is about 50 h for the resolution of 50 mg of the complex on a column of the exchanger (2^{ϕ} x 54 cm). The complete resolution of $[Co(en)_3]^{3+}$ (20 mg of chloride) was achieved in 6.5 h in the present work (Figure 2). On the other hand, the complete resolution of this complex (40 mg) on a column of d-tartrate derivative of Toyopearl HW40F (1.6 $^{\circ}$ x 39 x 3 (recycling)) using a 0.3 M Na₂SO₄ solution as the eluent took about 10 h for the elution of only the desired complex.³ These facts clearly indicate that the separation efficiency of the present exchanger is better than those of the previous ones. Furthermore, the present exchanger carries the favorable properties of the original Toyopearl gel: small variation of volume due to the change of composition of the eluents; insensitivity to degradation by microorganisms. Thus, the present exchanger can be readily prepared from Toyopearl gel and has excellent properties for efficient and easy operation of chromatographic resolution. One disadvantage is that a reduced pressure (-0.5 kg/cm^2) is required for the elution.

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