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The Foam Separation of Radioruthenium

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NOTE

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Summary

Foam separation of radioruthenium as Ru(IV) and RuNO(III) by the surface-active agents dodecylamine and gelatin from aqueous solutions has been studied. The multistage separation of Ru(IV) with dodecylamine was effective and more than 99% of Ru(IV) has been separated from the solution.

INTRODUCTION

The method of the foam separation has been employed for separation and concentration of a range of ions, radioactive elements, and colloidal particles (1-7). The principle of the method consists in adding a surface-active agent to the solution containing ions or colloidal particles meant for separation. This surface-active agent binds the separated component electrostatically and creates an insoluble complex. In our work the method of foam separation has been used for separating the ionic and colloidal forms of Ru(IV) and RuNO(III) from solutions.

EXPERIMENTAL

Solutions

We have been working with 50 ml $10^{-6}M$ solution of Ru(IV) chloride or RuNO(III) nitrate and the corresponding colloidal hydroxides were obtained through pH adjustment (8). From the cation-

active, anion-active, and non-ionic surface-active agents tried, dodecylamin and gelatin were chosen as the most suitable after preliminary examination. Chemicals for the preparation of solutions were of A.R. purity and both the surface-active agents were pure. The $10^{-6} M$ solutions of Ru(IV) chloride or RuNO(III) nitrate labeled with ^{106}Ru were obtained by diluting more concentrated solutions prepared by a previously described method (8).

Equipment

For the foam separation a glass vessel of about 50 ml capacity was made. The lower part was provided with a fritted glass (mean size of pores, 15–40 μ) for the bubbling of nitrogen, and the upper part with an overflow tube as a foam outlet. Nitrogen was purified with a solution of potassium hydroxide before coming into the solution. In order to be able to choose the surface-active agent with a proper charge and the correct conditions for separation, we determined the charge and electrophoretic mobility of particles of the separated colloidal hydroxides Ru(IV) and RuNO(III) by means of an electrophoretic ultramicroscope with an electrophoresis cell consisting of two parallel tubes (9, 10).

Working Procedure

A surface-active agent was added to 50 ml of solution containing radoruthenium in an ion or colloidal form, pH was adjusted, and during the time of separation nitrogen was bubbled through. In the alkaline sphere the pH was adjusted by sodium hydroxide; in the acid sphere by hydrochloric acid for Ru(IV) and by nitric acid for RuNO(III). In the case of Ru(IV) the period of separation with both surface-active agents was 1 hr; for RuNO(III) it was 2 hr. After separation had been completed, the radioactivity in the solution was determined. The results are expressed as per cent of the component being separated. In the case of the Ru(IV) colloid the charge and electrophoretic mobility were measured at the 10^{-5} and $10^{-6} M$; in the case of the RuNO(III) colloid they were measured at the $10^{-5} M$ concentration only because at a concentration of $10^{-6} M$ the particles were not clearly visible. On the ocular scale of the ultramicroscope it was possible to read the distance covered by the particle in a given time, and from the value of the potential gradient at a given cell length of the electrophoretic mobility could be calculated.

RESULTS AND DISCUSSION

A number of factors influencing the foam separation were determined to find the optimum efficiency conditions. The optimum velocity of the nitrogen flow was 0.36 liter/min for Ru(IV) with dodecylamine and 0.9 liter/min with gelatin. For RuNO(III) the optimum nitrogen velocity was 0.22 liter/min for dodecylamine and 0.72 liter/min for gelatin. An increase in nitrogen flow led to an increase in separation but due to considerable foaming the losses of the solution were higher than 5%, which was the limiting value for our purposes. The concentration of the surface-active agent was also determined. It had to be chosen with regard both to the separation efficiency and to the losses caused by foming. Under the requirement of losses lower than 5%, the highest separation was reached for Ru(IV) with 5 mg of dodecylamine (92%) or 2 mg of gelatin (65%), and for RuNO(III) with 7.5 mg of dodecylamine (55%) or 5 mg of gelatin (41%). Higher concentrations of surface-active agents led to considerable foaming and losses of the solution.

In the dependence of separation on pH, in the case of Ru(IV) the highest separation was achieved with dodecylamine at a pH of 5.6 and with gelatin at 5.5 (Fig. 1). For RuNO(III) the best separation took place at a pH of 7.3 with dodecylamine and at 8.5 with gelatin (Fig. 1). These pH values were used in other determinations. A change in

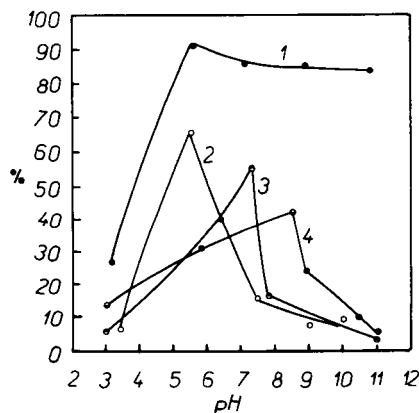


FIG. 1. Dependence of Ru(IV) and RuNO(III) separation on pH. Curve 1, Ru(IV) with dodecylamine. Curve 2, Ru(IV) with gelatin. Curve 3, RuNO(III) with dodecylamine. Curve 4, RuNO(III) with gelatin.

pH influences the form of Ru(IV) and RuNO(III); they pass from the ion form in acid medium into colloidal hydroxides with an increase in pH (8). The pH values at which the highest separation was achieved are not in agreement with pH values at which predominantly Ru(IV) and RuNO(III) colloids are formed (8). The value of pH also influences the solubility of the complex which is formed from the surface-active agent and Ru(IV) or RuNO(III). By the charge and mobility measurements with the electrophoretic ultramicroscope, a change in pH changes the sign of the charge of the colloidal hydroxide Ru(IV), its isoelectric point being at a pH of 4.8 (Fig. 2). The isoelectric point was not determined for colloidal hydroxide RuNO(III) since the particles of this hydroxide were not visible in the slightly acid range, but in analogy to other metal hydroxides, a change of the sign of its charge is probable. With dodecylamine, which is a cation surface-active agent and has a positive charge, the maximum separation of radio-colloids Ru(IV) and RuNO(III) takes place at a pH where their charge is negative and the prevalent mechanism is electrostatic attraction. With gelatin, which is an ampholyte with an isoelectrical point of 4.85 (11), the maximum separation was reached at the same charges both of colloidal hydroxides and of the gelatin, which has a negative charge after a pH of 4.85. In this case the mechanism of separation corresponds to the formation of an insoluble complex.

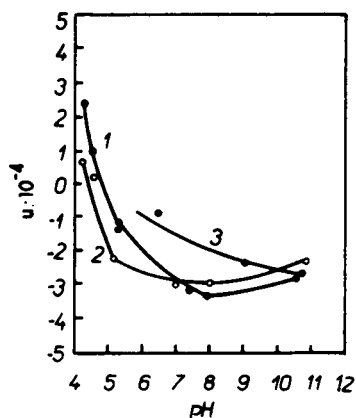


FIG. 2. Dependence of the electrophoretic mobility of colloidal particles Ru(IV) and RuNO(III) on pH. u -Mobility of particles, cm/sec/V/cm. Curve 1, mobility for $10^{-5} M$ Ru(IV). Curve 2, mobility for $10^{-6} M$ Ru(IV). Curve 3, mobility for $10^{-5} M$ RuNO(III).

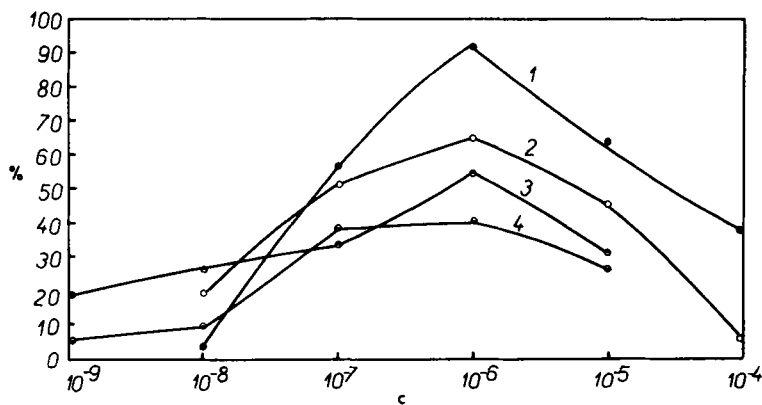


FIG. 3. Dependence of separation on Ru(IV) and RuNO(III) concentration in a solution. c , initial molar concentration of Ru(IV) or RuNO(III). Curve 1, Ru(IV) with dodecylamine. Curve 2, Ru(IV) with gelatin. Curve 3, RuNO(III) with dodecylamine. Curve 4, RuNO(III) with gelatin.

The dependence of separation on the concentration of Ru(IV) and RuNO(III) in the solution for both surface-active agents was also determined. The results are given in Fig. 3 and show that it is most efficient at a concentration of $10^{-6} M$ of the radioactive component. At

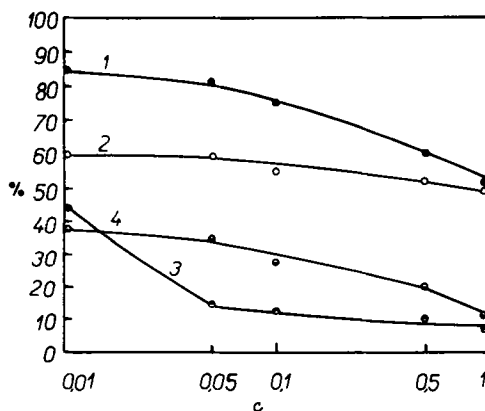


FIG. 4. Influence of increased electrolyte concentration on Ru(IV) and RuNO(III) separation. c , molar concentration of the NaCl or NaNO₃ in solution. Curve 1, Ru(IV) with dodecylamine. Curve 2, Ru(IV) with gelatin. Curve 3, RuNO(III) with dodecylamine. Curve 4, RuNO(III) with gelatin.

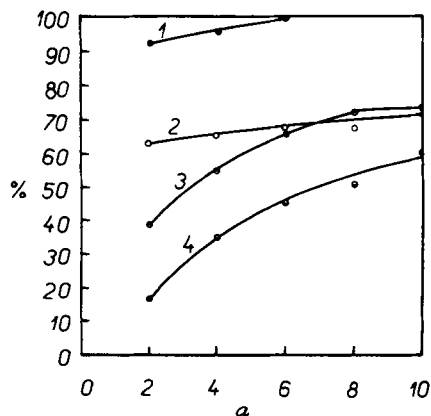


FIG. 5. Multistage separation of Ru(IV) and RuNO(III). a , milligrams of dodecylamine or gelatin in 50 ml of the solution of individual stages. Curve 1, Ru(IV) with dodecylamine. Curve 2, Ru(IV), with gelatin. Curve 3, RuNO(III) with dodecylamine. Curve 4, RuNO(III) with gelatin.

trace concentrations we can expect a lowered efficiency of separation. A higher concentration of electrolyte in the solution was manifested in a lowering of the percentage of separation of Ru(IV) and RuNO(III). The results of this determination with sodium chloride and sodium nitrate are given in Fig. 4.

In order to achieve higher separation without increasing the concentration of the surface-active agent and the flow of nitrogen, a multistage separation was carried out (12). In each stage 2 mg of surface-active agent were used for 50 ml of the solution, the same solution being moved from stage to stage. The results of this procedure, given in Fig. 5, show that in all cases the separation was increased, the efficiency for Ru(IV) with dodecylamine being 99% after the third stage. Another stage was not introduced because foaming after the third stage was so strong that losses of the solution were higher than 5%. In the other cases there were 5 separation stages.

Acknowledgment

The charge and the electrophoretic mobility of colloidal particles Ru(IV) and RuNO(III) have been measured in the Institute "Ruder Bošković" in Zagreb, in the Department of Radiochemistry on the electrophoretic ultramicroscope developed at this institute. The authors would like to express their thanks to Prof. Dr. R. Wolf from

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