Electrolytic Amalgamation of Uranium Using an Ion Exchange Membrane

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Summary A novel method for the preparation of uranium amalgam using an ion exchange membrane for adjusting pH during electrolysis is described.

THE preparation of uranium metal directly from aqueous solution has been attempted by various authors.[1] The conventional process of extracting uranyl ion in aqueous solution by sodium amalgam is somewhat tedious and gives rather low yields, probably owing to difficulties in controlling pH which increases during the extraction. The reduction of the uranyl ion to the metallic state is known to proceed according to equation (1). Direct electrolysis

$$UO_2^{2+} + 4H^+ + 6e \longrightarrow U + 2H_2O \qquad (1)$$

using a mercury pool as cathode will unavoidably lead to elevation of pH and consequently to the precipitation of UO_2 and hydroxides. The reaction is thus prevented from proceeding beyond the U^{IV} state.

An electrolytic cell has been designed, which is composed of two compartments divided by a cation exchange membrane (CK-1, Asahi Chemicals): the upper compartment consists of a platinum anode and an acidic solution, while the lower one with a mercury pool as cathode is filled with the uranyl ion solution. The pH of the cathode compartment where the reaction takes place is kept virtually unchanged during the electrolysis by continuously supplying hydrogen ion *via* the ion exchange membrane from the upper compartment. The hydrolysis and precipitation of uranium can thus be prevented. In order to maintain the temperature and the uranyl ion concentration in the cathode compartment constant the solution is circulated between a thermostatted reservoir and the cathode compartment.

We used 15 ml of mercury and 400 ml of 0·1 M uranyl acetate solution, with an anode compartment filled with 1M SO_4^- solution (100 ml, 1 : 2 H_2SO_4–Na_2SO_4). Electrolysis was carried out at 10 V and 3 A for 2 h. One third of the mercury became a soft solid mass which contained as much as 1·1 g U/ml Hg, while the liquid portion contained 7·4 mg U/ml Hg. *Ca.* 70% of the uranyl ion was amalgamated, which is greater than the 50 mg U/ml Hg obtained by Malan *et al.* using sodium amalgam as extractant.[2] Part of the amalgam was thoroughly washed with dil. HCl and H_2O and then heated at 10^{-4} mmHg and 1300 °C. 5 g of uranium metal density 19·2 g cm^{-3}, were recovered.

A similar experiment using ^{239}Np tracer, in which 50% of NpO_2^+ were amalgamated, shows that this method might be useful for the amalgamation of metal-oxy-ions and might find applications in the preparation of the respective metals directly from aqueous solutions.

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[1] G. Bouissières, M. Haissinsky, and Y. Legoux, *Bull. Soc. chim. France*, 1961, 1028; G. Boussières and Y. Legoux, *ibid.*, 1965, 386; E. David and G. Boussières, *Inorg. Nuclear Chem. Letters*, 1968, **4**, 153; J. Malý, *J. Inorg. Nuclear Chem.*, 1969, **31**, 1007.

[2] H. P. Malan, Y. Kobayashi, and T. Ishimori, *J. Inorg. Nuclear Chem.*, 1971, **33**, 3097.