ELECTRODEPOSITION OF CARRIER-FREE $^{95}\text{Zr}$ FROM AN AQUEOUS BATH

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The electrodeposition of carrier-free $^{95}\text{Zr}$ for nuclear spectroscopic studies has been successfully achieved using urea-oxalate electrolyte solution. Employing a current density of 40 mA per cm$^2$, over 80% of radioactive zirconium was deposited within 40 hours. Attempts to electroplate $^{95}\text{Zr}$ from aqueous baths containing various other inorganic and organic acids, bases, or their salts, were unsuccessful.

It is still a problem to electrodeposit radioactive nuclides of refractory elements, such as zirconium, hafnium, tantalum, etc. A more or less complete study of these elements from fused salt baths was reported by MELLORS and SENDEROFF$^{1-3}$ and was reviewed by SENDEROFF.$^4$

HOLT$^5$ reviewed the previous work on the electrodeposition of zirconium from aqueous and non-aqueous baths, and came to the conclusion that it was not possible to electroplate this element. Further attempts$^6-8$ of a similar nature also met with failure. However, by employing a mixed type of bath containing hydride and borohydride, BRENNER et al.$^9$ were able to co-deposit zirconium with titanium, containing about 45% zirconium.

STEPHEN$^{10}$ reported that these refractory elements cannot be electroplated from aqueous solutions, but can only be used as substrates.

These methods of depositing zirconium from fused baths, or its co-deposition with titanium, are not suitable for the electroplating of radioactive zirconium nuclides required for nuclear spectroscopic studies. The problem was, therefore, studied to discover a suitable method for the electrodeposition of radioactive zirconium.

Experimental

Reagents and chemicals

All the reagents used were of AnalaR grade; they were used without further purification.

Urea oxalate. 120 g of urea ($\simeq 2$ moles) was dissolved in a minimum amount of hot distilled water ($\simeq 50^\circ C$). In another beaker 126 g of oxalic acid ($\simeq 1$ mole) was dissolved in a similar way. The urea solution was then added slowly to the hot oxalic acid solution with constant stirring, when the urea-oxalate separated. The temperature of the mixture was raised to dissolve all the precipitated urea-oxalate. The solution was then allowed to cool slowly. Needle shaped crystals were formed, which were filtered and recrystallized from distilled water. The process was repeated twice. Finally, the crystals of urea-oxalate were filtered off and dried. The melting point was $168^\circ C$ (uncorrected) (lit. m. p.: $170^\circ C$).
The purified product was analyzed for oxalic acid by titration with potassium permanganate. The analysis gave the formula of the substance as \([\text{CO(NH}_2\text{)}_2\text{(COOH)}_2\text{]}_2 \) or \(\text{C}_4\text{H}_{10}\text{O}_6\text{N}_4\). Blank runs indicated urea having no effect on the assay of oxalic acid by potassium permanganate.

The zirconium tracer used in the experiment was \(^{95}\text{Zr}\) supplied by Bhabha Atomic Research Centre (BARC), Trombay. This was purified from its daughter \(^5\text{Nb}\) by the method of Huffman et al.\textsuperscript{11}

**Apparatus**

*Electrolysis cell.* The cell used was similar to that described by Casto\textsuperscript{12} and Dodson et al.\textsuperscript{13} It is shown in Fig. 1 dismantled, and in Fig. 2 assembled.

![Electrolysis cell diagram](image)

*Electrodes.* Various metal foils such as copper, stainless steel, platinum, tantalum, etc. were used. Tantalum foil was supplied by ICI Ltd., Birmingham, England. These were used as cathodes. The anode was made of a platinum strip.

A 6 V battery was the source of emf. An ammeter with a wire-wound potentiometer connected in series was used to measure and control the current in the cell.

A gamma-ray scintillation spectrometer and NaI(Tl) crystal were employed to measure the radioactivity of both the solution and the cathode.