

ОБЪЕДИНЕННЫЙ
ИНСТИТУТ
ЯДЕРНЫХ
ИССЛЕДОВАНИЙ
ДУБНА



2 344-4
B-55

13/v-74

E12 - 7744

G.-J.Beyer, E.Herrmann

1816/2-74

RAPID ELECTRODEPOSITION
OF RADIOACTIVE RARE EARTH

1974

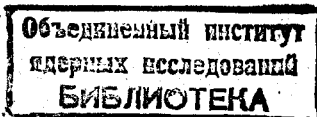
ЛАБОРАТОРИЯ ЯДЕРНЫХ ПРОБЛЕМ

E12 - 7744

G.-J.Beyer, E.Herrmann*

**RAPID ELECTRODEPOSITION
OF RADIOACTIVE RARE EARTH**

Submitted to Radiochemical and Radioana-
lytical Letters



* Sektion Chemie der Technischen Universität
Dresden, Dresden, GDR

Байер Г.-Ю., Херрманн Э.

E12 - 7744

Быстрое электроосаждение радиоактивных редкоземельных элементов

Определены оптимальные условия для быстрого электроосаждения радиоактивных редкоземельных элементов без носителя на вольфраме. Выход достигается за 30 секунд при плотности тока $0,2 \text{ A cm}^{-2}$ из раствора $2 \cdot 10^{-3} \text{ M}$ α -оксиизобутирата аммония.

Препринт Объединенного института ядерных исследований.
Дубна, 1974

Beyer G.-J., Herrmann E.

E12 - 7744

Rapid Electrodeposition of Radioactive
Rare Earth

A method is described for electrodepositing carrier-free radioactive rare earth nuclides onto a small tungsten cathode. The nuclides are deposited to more than 80 per cent in 30 to 40 seconds. The whole operating time including an evaporation step is about two minutes.

Preprint. Joint Institute for Nuclear Research.
Dubna, 1974

INTRODUCTION

Recently, a highly effective and rapid method for isotope separation of carrier-free rare earth and actinide elements has been reported ^{/1, 2/}. An important step of sample preparation for this method is the cathodic deposition of the radioactive products from solutions containing ammonium α -hydroxy-isobutyrate. In order to obtain higher yields of short-lived isotopes, it is desirable the electrolysis period to be reduced. This applies to the preparation of sources for alpha and beta spectrometry, too.

Systematic investigations of electrodeposition of rare earth hydroxides from solutions containing oxycarbonic acids are reported for the lactate system. Novgorodov et al. ^{/3/} obtained a 90 per cent deposition in 20 minutes. Their method is also applicable to the α -hydroxy-isobutyrate (α HiB) system giving good results ^{/1/}. But the removal of the excess of complexing agent by repeated evaporation to near dryness is rather time consuming and requires some experience for obtaining reproducible results.

The aim of these investigations was to simplify the method and to render it safer as well as to optimize the experimental conditions for the electrodeposition of carrier-free rare earth nuclides from solutions containing α HiB.

EXPERIMENTAL

The carrier-free nuclides $^{169-173}\text{Lu}$ and ^{140}Nd MeV obtained from tantalum targets irradiated with 660 mēV protons ^{4/}. Final purification of these elements was achieved by cation exchange chromatography. As eluant a 0.5M buffer solution of α HiB was used. By dilution the eluate with 0.5 M α HiB of pH 4.8 stock solution was prepared.

Three to five drops of the stock solution were evaporated to dryness on a platinum foil in a small depression (about 5 mm dia.). At the end of the evaporation, the complexing agent volatilized forming fumes. After determination of sample radioactivity by using a NaJ(Tl) - scintillation counter, the platinum foil was placed on a metal support connected to the anode. The residue in the depression was dissolved by adding 0.02 ml of α HiB -solution of pH 4.8. The electrolysis was initiated by placing a tungsten cathode on the drop surface a square of 1 mm x 5 mm contacting the surface. A 70 V element supplied the direct current for electrolysis. The micro apparatus used for electrodeposition was similar to that described by Novgorodov et al. ^{3/}. The electrolysis was stopped by lifting the tungsten cathode. The radioactivity of the rare earth deposited onto the tungsten foil and those left on the platinum foil was measured after drying the foils. From the activity balance the magnitude of spraying losses owing to gas formation during the electrolysis was obtained.

RESULTS AND DISCUSSION

By the evaporation of the α HiB containing solution to dryness no losses in radioactivity could be detected. The residue that was practically invisible is completely soluble in diluted α HiB solutions of pH 4.8. The yield of the electrodeposition of rare earth hydroxides, however, depends on the α HiB concentration. Figure 1 shows that there is a deposition maximum if the α HiB concen-

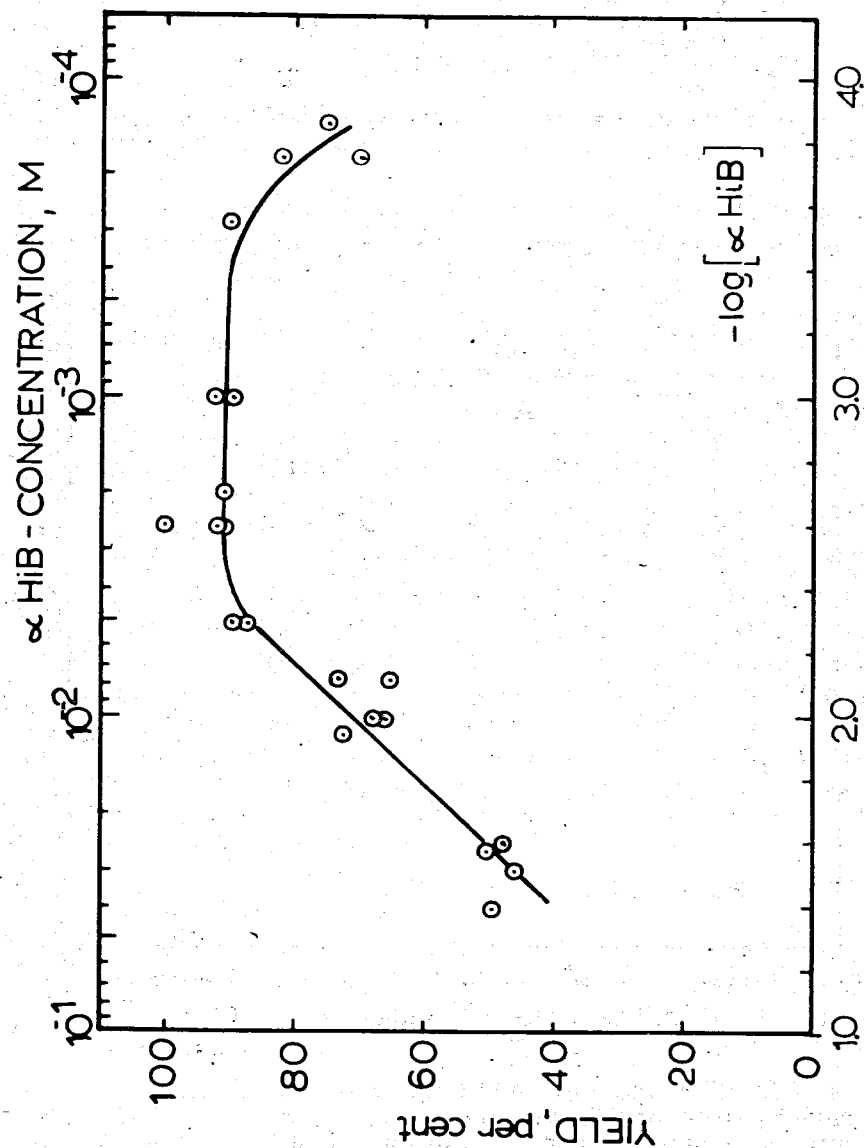
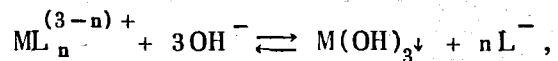


Fig. 1. Effect of the α HiB concentration of the yield of the electrodeposition of lutetium (pH 4.8, electrolyte volume 0.02 ml, depositing duration 60 seconds, current density 0.25 A/cm²).

tration in the electrolyte is about $3 \cdot 10^{-4}$ to $3 \cdot 10^{-3}$ M. At higher concentrations, the yield of deposition decreases, because the equilibrium of the reaction



where M - rare earth ion, L - α HiB ion, is shifted to the left-hand side. At lower concentrations of the

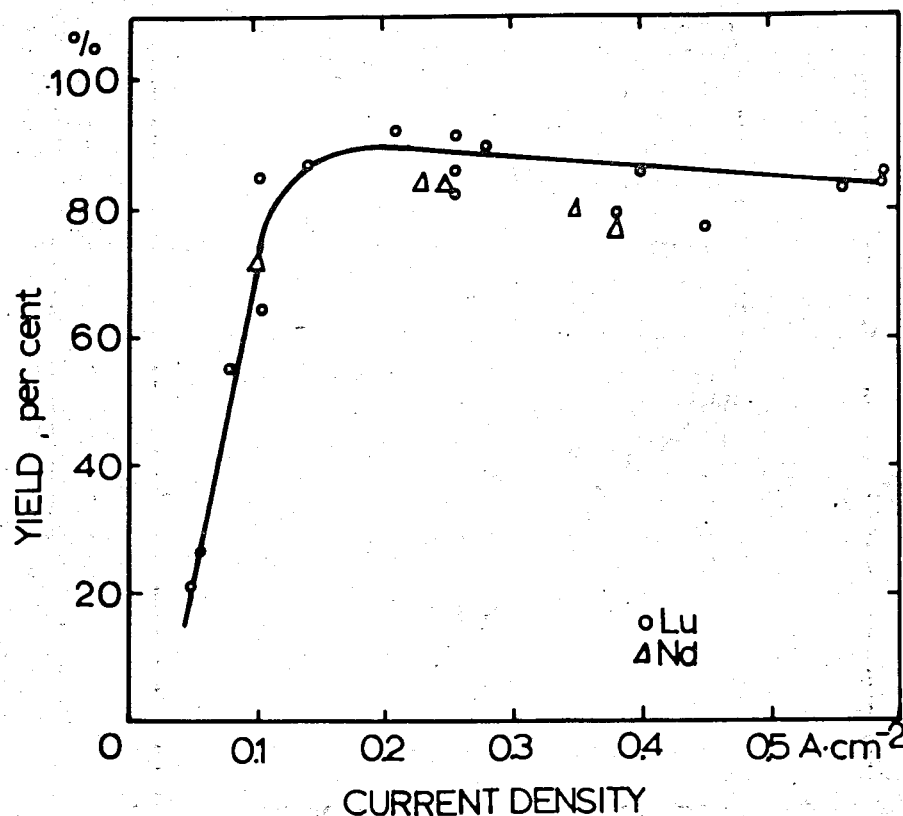


Fig. 2. Effect of the current density on the yield of the electrodeposition of neodymium and lutetium (electrolyte: 0.02 ml of $2 \cdot 10^{-3}$ M α HiB solution, pH 4.8, depositing duration 60 seconds).

complexing agent radiocolloids are likely to exist that are not electrodeposited.

The effect of current density on deposition is shown in Fig. 2. To produce an adequately high pH-value near the cathode a minimum current density is necessary^{/5/}. The yield decreases if the current density is too high. This is due to the increase spraying losses caused by the increased gas formation. The optimum current density is about 0.2 to 0.3 A/cm².

The effect of varying the depositing time is shown in Figure 3. After 30 seconds the depositing equilibrium is nearly reached and the deposition yield is 80 per cent and over.

Furthermore, Figures 2 and 3 show that lutetium as a representative of heavy rare earth elements is deposited with higher yields than neodymium, which was chosen as an example for lighter rare earth elements. This can be explained by the higher solubility product of neodymium hydroxide.

The optimum electrodeposition of carrier-free rare earth nuclides was obtained for the following conditions: The eluate of the cation exchange separation (2 to 3 drops) was evaporated to dryness. The residue was dissolved in 0.02 ml of 10^{-3} M α HiB solution of pH 4.8 and the electrodeposition carried out for 30 to 40 seconds at a current density of 0.2 to 0.3 A/cm². The whole time for the source preparation including the evaporation step is about two minutes, the yield being 80 per cent and better.

ACKNOWLEDGEMENT

The authors are grateful to V.A.Khalkin for the stimulation of this work and his helpful interest.

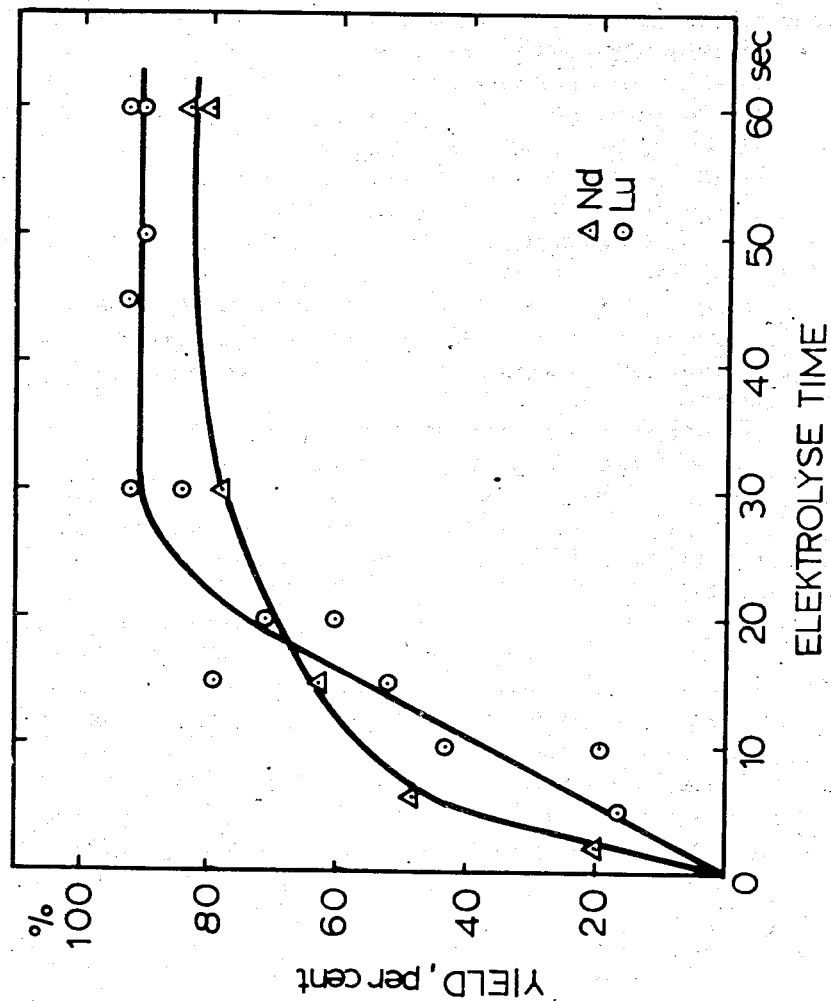


Fig. 3. Effect of time on the deposition of lutetium and neodymium (electrolyte: 0.02 ml of $2 \cdot 10^{-3}$ M aHiB solution, pH 4.8, current density 0.25 A/cm^2).

REFERENCES

1. G.J.Beyer, E.Herrmann, A.Piotrowski, V.J.Raiko, H.Tyrroff. Nucl. Instr. and Meth., 96, 437 (1971).
2. G.J.Beyer, E.Herrmann, F.Molnar, V.I.Raiko, H.Tyrroff. Radiochim. Radioanal. Letters, 12, 259 (1972).
3. A.F.Novgorodov, V.L.Kochetkov, N.A.Lebedev, V.A.Khalkin. Radiochimiya (Russ.), 6, 73 (1964).
4. M.Vobecky, A.Mastalka. Collection Czech. Chem. Commun., 28, 709 (1963).
5. P.G.Hansen, J.Inorg. Nucl.Chem., 12, 30 (1959); 17, 232 (1961).

Received by Publishing Department
on February 22, 1974.