Inorganica Chimica Acta, 110 (1985) 25–26

Production of Microgram Quantities of Einsteinium-253 by the Reactor Irradiation of Californium

S. A. KULYUKHIN, L. N. AUERMAN, V. L. NOVICHENKO, N. B. MIKHEEV, I. A. RUMER, A. N. KAMENSKAYA
Institute of Physical Chemistry, USSR Academy of Sciences, Moscow, U.S.S.R.

L. A. GONCHAROV and A. I. SMIRNOV
Kurchatov Institute of Atomic Energy, Moscow, U.S.S.R.

Received January 4, 1985

Abstract

Einsteinium-253 was obtained by irradiation of 100–200 μg of californium-252 by the flux of thermal neutrons 2–5 × 10^{14} neutrons cm^{-2} s^{-1} during 500–900 h. For the separation of einsteinium from californium and neodimium, which was used as a carrier, the chromatographic method of separation on the Aminex Q-15S resin with particles of 20 to 25 μm was applied.

Einsteinium was eluated by a solution of α-oxyisobutirate of ammonium (0.14 M) with pH = 4.95 at room temperature. The coefficient of separation of einsteinium and californium under these conditions is equal to 1.62. The coefficient of purification of einsteinium from californium is *ca.* 10^5.

Es-253 is used to synthesize the elements placed in the end of the Periodic system, such as fermium and mendelevium. Besides this, it can be used as an energy source.

Es-253 is obtained when irradiating Cf-252 by thermal neutrons according to Scheme 1 [1]:

$$\begin{array}{lcl} {}^{252}\mathrm{Cf}\,(\eta,\gamma) & {}^{253}\mathrm{Cf} & \sigma[{}^{253}\mathrm{Cf}] = 17.6\ \mathrm{b} \\ & \Big| & \sigma_f[{}^{253}\mathrm{Cf}] = 1300\ \mathrm{b} \\ \sigma[{}^{252}\mathrm{Cf}] = 20.4\ \mathrm{b} & \beta^- \Big| & \\ \sigma_f[{}^{252}\mathrm{Cf}] = 32\ \mathrm{b} & \Big\downarrow & \\ & {}^{253}\mathrm{Es} & \sigma[{}^{253}\mathrm{Es}] = 155\ \mathrm{b} \end{array}$$

Scheme 1.

As seen from the scheme an unfavourable factor impeding the accumulation of Es-253 is a large fission cross section for Cf-253 which equals 1300 barns. This is why there is an optimal irradiation period for every flow of thermal neutrons, during which the maximum output of Cf-253 is achieved. After irradiation the accumulation of Es-253 in the target occurs, which is accounted for by the relatively long half-life period of Cf-253. The rate of accumulation depends on the ratio of Cf-253 *vs.* Es-253 quantities, as available at the end of irradiation. Therefore, in order to obtain the maximum possible quantity of Es-253 it is worthwhile to provide the separation of Es from the target in 10 to 20 days after irradiation. Also a significant lowering of the γ-radiation value occurs, which is accounted for by the decay of the short-lived fission products.

In order to obtain Es-253 we carried out a series of irradiations of 100–250 μg of Cf-252 using the MR reactor of the Kurchatov Institute of Atomic Energy. The flow of thermal neutrons was 2–5 × 10^{14} neutrons cm^{-2} s^{-1}. According to the calculations, the maximum output of Cf-253 under these conditions is achieved in about 900 h of irradiation. In our experiments the irradiations of Cf-252 targets were 500–900 h long, and their processing to obtain Es-253 was conducted 15–20 days after the end of irradiation.

Because of the spontaneous fission of Cf-252, large amounts of fast neutrons appear (2.4 × 10^6 neutrons s^{-1} $\mu\mathrm{g}^{-1}$). This is why the preparation of a Cf-252 target as well as the extraction of Es-253 from it thereafter was conducted in a box provided with neutron protection and with master-slave manipulators. The box as equipped at the Institute of Physical Chemistry enabled us to operate with Cf-252 quantities of up to 250 μg.

Inside the box there was equipment for separating Es from Cf, consisting of a chromatographic column, disposable pipettes for taking samples and replacing them, as well as a centrifuge, electric heater, *etc.* The behavioral control for Cf in the process of preparing the target and its processing after irradiation was carried out by a neutron counter, also placed inside the box.

The Cf-252 target was prepared for irradiation as follows. Cf hydroxide was precipitated from an HCL solution containing 100–250 μg of Cf and 1 mg of Nd^{3+} used as a carrier. Then the hydroxide was wash-

0020-1693/85/$3.30

ed and dissolved in several drops of HCl. The obtained solution was removed into a special quartz tube with a tight cap and dried at 90 ℃ in a flow of air. Then the tube was put in an aluminium container for further irradiation. The use of a quartz tube was possible thanks to the low heat formation coming from the fission of Cf-252 under the neutron effect, as well as to a rather high radiation stability of quartz. Nd, which was chosen as a carrier, activates insignificantly under thermal neutron irradiation, since the basic γ-radioactivity at its irradiation is accounted for by the ^{147}Nd isotope ($t_{1/2}$ = 11 days) formed from ^{146}Nd (σ = 1.3 barns) whose content in a natural Nd isotope mix is 17%. The presence of Nd does not interfere with the chromatographic separation of Cf and Es.

After irradiation and required cooling the aluminium container was cut open in the box by means of a diamond disc. The same device was used for opening the quartz tube in case of difficulties with removing the cap. The irradiated substance containing Cf, Es, Nd and fission products was dissolved in 0.5–1.0 ml of HCl (0.1 M). The solution was placed in a tube containing 100 mg of the cation-exchange resin Aminex SB in the NH_4^+ form (the size of the particles was 20–25 μm) with the following sorbtion of isotopes in the static conditions which prevented the peak broadening during the elution process. The resin was washed twice by a hot NH_4Cl (0.1 M) solution, twice by water and then was removed to the upper part of the column (h = 9 cm, d = 0.5 cm) filled with the same resin. The isotopes were washed out by ammonium α-oxiisobutyrate (0.14 M) with pH = 4.95 at 25 ℃. The speed of elution was 1 ml cm^{-2} min^{-1}. Figure 1 shows the wash-out curve for Es and Cf. For the given column the value of the theoretical plate for Es was 0.34 mm and for Cf 0.20 mm. The coefficient of separation of Es from Cf in those conditions was 1.62. The coefficient of purification of Es from Cf was *ca.* 10^5. In some cases unwanted premix of Cf-252 in Es remained. For further purification the Es fraction was added with HCl up to pH = 1÷2. Then the sorbtion and washing were provided according to the above described scheme. Thus, a deep purification of Es from Cf could be achieved.

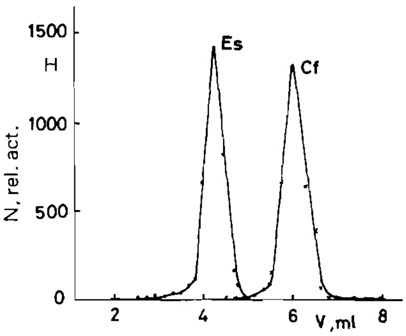

Fig. 1. Eluation curve of Es and Cf.

The extraction of Es from the solution of ammonium α-oxiisobutyrate was made as follows. The solution was added with HCl up to pH = 1 and then passed through the column with a small quantity of the Aminex SB resin (h = 3 cm, d = 0.5 cm). So Es was totally sorbed on the resin. The resin was washed with water and then Es was eluted by a small amount of 4–6 M HCl or nitric acid. The amount of Es-253 obtained was 0.1–0.5 μg, depending on the amount of the irradiated Cf and the duration of the irradiation.

Since after the separation of Es the Cf keeps on accumulating Es-253 due to the decay of Cf-253, a second extraction of Es-253 can be conducted some time later. This enables additional quantities of einsteinium to be obtained.

References

1 S. H. Eberle, Y. Reinhardt, E. Gantuer and Ch. Krückeberg, *KFK-1338*, 1 (1971).