Chemical procedure applied for the identification of Rf/Db produced in the $^{48}\text{Ca} + ^{243}\text{Am}$ reaction

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Summary. A chemical separation procedure for Rf/Db is described which was applied to a long-lived decay product from the nuclear fusion reaction $^{48}\text{Ca} + ^{243}\text{Am}$. A 1.2 mg thick ^{243}Am target was bombarded by 247 MeV ^{48}Ca particles. The recoiling products were collected in a thick Cu catcher for about one day and then subjected to a chemical separation procedure that included an ion exchange from dilute HF solutions. Final samples were prepared on 30 $\mu\text{g}/\text{cm}^2$ thick polyethylene (PE) foils and counted in 4π -geometry for α -particles and spontaneous fission (SF) coincidences. The detector arrays were surrounded by ^3He detectors to also assay prompt neutrons. Decontamination factors from actinides of about 10^5 were achieved. Group 6 (W) to 14 (Pb) elements as models for their heavier homologues were shown to be separated from the Rf/Db fraction with more than 90%. In eight final samples, representing a total beam dose of 3.4×10^{18} particles, 15 SF events were detected. The decay pattern points to a single component with a half-life of ≈ 32 h, which shows a chemical behavior similar to the lighter homologues of group 4 and 5 elements.

1. Introduction

In 2003 an experiment was carried out at the U400 cyclotron (FLNR, JINR Dubna, Russia) aimed at synthesizing element 115 in the $^{243}\text{Am}(^{48}\text{Ca}, xn)^{291-x}115$ reaction. Three decay chains were observed with five consecutive α -emissions, detected within about 20 s, terminated by a spontaneous fission (SF) event after 16–28 h [1]. This SF activity was thus assigned to ^{268}Db or, after EC-decay, to ^{268}Rf originating from the decay of the initial evaporation residue $^{288}115$.

In order to confirm this assignment, a chemical identification of this long-lived descendant seems appropriate. The separation scheme should guarantee a high separation factor from transfer products in the region of heavy actinides like Cf or Fm, which can also decay by SF. Moreover, conditions

had to be selected that allowed to separate all other possible transactinides up to element 114 and to collect both Db and Rf in one final fraction.

The chemical properties of transactinide elements were studied by several authors. Both aqueous and gas phase chemistry experiments were carried out. For a review we refer to [2].

Liquid phase separation was considered optimum due to the requirements concerning the heavy actinide separation. In the past 25 years, several media have been studied for the characterisation of the chemical behaviour of Rf and Db in aqueous solution using for example extraction from HCl containing solutions into triisooctylamine (TIOA) [3, 4] or cation exchange with α -HIB solution [5, 6] and many others. All these systems are not suitable in the present case either due to poor separation factors achieved for actinides or because of a non-similar behaviour of group 5 and 4 elements. In our view, the most promising chemical system for simultaneous separation of elements from the 4th and 5th group is ion exchange from HF containing solution [7–9]. In this medium, all members of group 4 and 5 form anionic fluoride complexes, which are not adsorbed on cation exchangers, whereas lanthanides/actinides can be adsorbed on such columns with high distribution coefficients [10, 11].

In this publication, the chemical aspects of the experiment are described. For an overview of the study we refer to [12, 13]. Additional details of the physics objectives, including a description of the data acquisition system and interpretation of the results are subject of a forthcoming publication [14].

2. Experimental

Irradiation

A 32-cm² rotating target consisting of the enriched isotope ^{243}Am (99.9%) in the oxide form was bombarded by a 247 MeV ^{48}Ca beam (middle of the target) at the U400 cyclotron with an average intensity of 0.85 μA . The 1.2 mg/cm² ^{243}Am target was deposited onto 1.5 μm Ti

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Fig. 1. Construction of the target chamber equipment (see text).

foils. A collimator, 10 mm in diameter, limited the irradiated area. The recoiling reaction products passed through a second 12 mm collimator, 10 mm behind the target, and were stopped in a 50 mm diameter copper catcher positioned 100 mm behind the target (Fig. 1). The collection efficiency of the compound nucleus in such a geometry (collecting angle $\pm 12.5^\circ$) was close to 100%. The range of recoils in the copper catcher was approximately 3–4 μm . Details of this estimate are given in [15].

After the end of bombardment (EOB), the Cu catcher was dismantled and the surface cleaned from sputtered ^{243}Am with cellulose tissues and ethanol. Subsequently, a 7–10 μm layer (corresponding to 120–180 mg of Cu) was removed using a micro-lathe. This layer, in form of copper chips, was subjected to the chemical separation procedure.

Chemical separation

The chemical procedure was designed to separate group 4 and 5 elements with highest possible decontamination factors from actinides, and, additionally, with a sufficient high factor ($\geq 80\%$) from transactinides with $Z \geq 106$ within about 3 hours, ending in nearly weightless final samples. This requires a complete separation from the macro amount of Cu, a minimized volume of the group 4/5 fraction ($< 2\text{ ml}$) and a fast evaporation of the final solution onto thin PE foils.

For the development of the chemical separation procedure, carrier-free tracers ^{177}Ta , $^{92\text{m}}\text{Nb}$, ^{88}Zr , ^{175}Hf , ^{88}Y , ^{167}Tm , and ^{169}Yb were used, which were produced in (α, xn) and spallation reactions with the cyclotrons at JINR, the U200 (FLNR), and the Synchrotron “F” (Laboratory of Nuclear Problems).

For the evaluation of the chemical behaviour of elements with $Z \geq 106$ carrier-free isotopes of Ir, Pt, Au, Hg, Tl, and low-carrier Pb ($< 1\text{ mg}$ per sample) were produced *via* activation with high-energy neutrons using the spallation neutron source SINQ at PSI.

Tracer production

^{177}Ta from α -irradiated Lu_2O_3 , ^{175}Hf from α -irradiated Yb_2O_3 , $^{92\text{m}}\text{Nb}$ from α -irradiated Yb_2O_3

Details of the chemical procedures are described in [16].

^{169}Yb , ^{167}Tm from spallation of Ta

Tantalum was irradiated with 660 MeV protons at the Synchrotron “F” for about 7 h. Ta was then dissolved in conc. HNO_3 /conc. HF solution. 1 mg La in form of a $\text{La}(\text{NO}_3)_3$ solution was added to precipitate the insoluble fluoride. LaF_3 was washed twice to remove traces of Ta, then dissolved in H_3BO_3 containing HCl solution and passed through a cation exchange column ($8 \times 40\text{ mm}$, DOWEX50 $\times$ 8, 150–200 mesh, H^+ -form). Subsequent removal of all rare earth elements was achieved by gradient elution with α -hydroxyisobutyric acid (α -HIB).

$^{88}\text{Zr}/^{88}\text{Y}$ from α -irradiated SrO

SrO was irradiated with 36 MeV α -particles at the U200. The oxide was dissolved in HCl . 1 mg La was added as carrier and the hydroxides were precipitated by adding ammonia solution. The precipitate was dissolved in conc. HCl and passed through an anion exchange column. Zr is adsorbed under these conditions, whereas La is eluted. Zr can be removed from the column with 4 M HCl . ^{88}Y is formed *via* electron capture decay from ^{88}Zr .

^{192}Ir , ^{191}Pt , $^{199/198}\text{Au}$, $^{197\text{m}}\text{Hg}$, ^{200}Tl and ^{203}Pb from neutron-activated Pt, Hg and Pb

530 mg Pb, 7 g HgCd_2 , and 12 mg Pt were irradiated with thermal and high-energy neutrons at the spallation neutron facility SINQ for one hour. The high average energy of the neutrons feeds a broad spectrum of reaction channels like n, xn (with x up to 12) or $n, xpyn$ reactions and their decay products. The mentioned nuclides were mainly formed in such reactions.

^{192}Ir and $^{199/198}\text{Au}$

The Pt target was dissolved in conc. HNO_3 /conc. HCl . It contained mainly isotopes of Ir, Au, and Pt. The solution was evaporated to dryness, the residue fumed several times with conc. HNO_3 and dissolved in 2 M HNO_3 for final use. The content of less than 100 μg of Pt carrier per sample did not influence the chemical behaviour of the other radio nuclides.

^{191}Pt

From the irradiated HgCd_2 -target, the mercury and cadmium were removed by distillation in an Ar/H_2 stream at 650°C . The residue, containing mainly Pt and Au isotopes, was dissolved in conc. HNO_3 .

 $^{197\text{m}}\text{Hg}$

Carrier-free Hg isotopes were obtained thermochromatographically by heating the irradiated Pb target to 600°C in an Ar/H_2 stream. The released mercury was then adsorbed on a gold foil. 95% of the Hg activity could be removed from the Au foil by washing with conc. HNO_3 .

 ^{200}Tl and ^{203}Pb

The residual Pb target contained mainly Pb and Tl isotopes. It was dissolved in conc. HNO_3 and the solution was evaporated to dryness. After dissolution in 1 M HNO_3 , conc. HCl was added to precipitate the macro amount of lead as PbCl_2 . The solution was again evaporated to dryness, the residue dissolved in 1 M HNO_3 , and this solution passed through a Pb-specific ion exchange column (EICHROM). Pb is the only element which is completely retained on this column. The Tl-containing eluate was used for the experiments.

The PbCl_2 precipitate was dissolved in 1 M HNO_3 and an aliquot of this solution was used to study the chemical behaviour of lead.

Ion exchange separation

The tracers listed above were used to study their elution behaviour on cation and anion exchange resins aimed to obtain a decontamination factor from the lanthanides/actinides as high as possible. Exchange columns (8×40 mm) made from polyethylene were filled with the strongly acidic cation exchanger DOWEX50 $\times$ 8 (150–200 mesh, H^+ -form) or strongly basic anion exchanger DOWEX1 $\times$ 8 (150–200 mesh, required anion-form). Several feeding solutions and eluents like HCl, HNO_3 , HBr, HF and mixed solutions were tested. From these studies, the optimum separation conditions were selected for a final chemical separation scheme.

The measurements of the distribution ratios of the tracer nuclides and the determination of the decontamination factors were carried out by standard γ -spectrometry yielding values up to 10^2 due to the detection limits (depending on overall activity and measuring time).

Higher decontamination factors for lanthanides were determined with the ICP-OES technique, which allows determinations down to the ppb level.

Final chemical separation scheme

The copper chips were dissolved in 10 ml 7 M HNO_3 adding some drops of conc. HCl. The tracer activities and $700\text{ }\mu\text{g}$ of La carrier were added and the hydroxides precipitated with concentrated ammonia solution. Under these conditions, Cu and most of the group 6–14 elements remain in solution as ammonia complex, group 4 and 5 elements as well as La and heavy actinides precipitate. In order to re-

move traces of copper, the procedure was repeated twice by dissolving the hydroxide in 1 M HCl and adding ammonia solution again. The precipitate was dissolved in 0.5 ml 2 M HNO_3 and adsorbed on a DOWEX50 $\times$ 8 column (8×40 mm, 150–200 mesh, H^+ -form). The group 4/5 fraction was eluted with 2 ml 1 M HF. Finally, the solution was evaporated on PE foils ($30\text{ }\mu\text{g}/\text{cm}^2$) using a hot helium stream. The overall separation time from EOB until start of measurements was 2–3 hours.

Eight identical experiments were performed. Final samples were measured for α -decay and SF up to 960 hours. A ninth experiment was carried out under identical experimental conditions but without chemical separation (direct catch). The catcher surface was cleaned from sputtered ^{243}Am and then contacted with solid-state track detectors in order to determine the overall amount of spontaneously fissioning nuclei.

Measurement

For the registration of α -particles and SF fragments, we used a detection array consisting of four identical chambers, each equipped with two opposite semiconductor detectors with an active area of 6 cm^2 , mounted at a distance of 4 mm. All counting chambers were inserted in a neutron detector array consisting of 72 ^3He counters, which were positioned in three cylindrical layers in order to register prompt neutrons. The detection efficiency for fission fragments was about 90% and for single neutrons 40%. In the course of a 330-hour test run prior to the experiment, no coincident background events were detected.

3. Results and discussion

The sorption and elution behaviour of the tracer nuclides ^{177}Ta , $^{92\text{m}}\text{Nb}$, ^{88}Zr , ^{175}Hf , ^{88}Y , ^{167}Tm and ^{169}Yb on the cation and anion exchangers using several eluents was studied under identical conditions as in the experiment. It turned out that HCl, HBr and HNO_3 media as well as some mixed HCl/HF and $\text{NH}_4\text{Cl}/\text{NH}_4\text{F}$ solutions are not suitable because of high elution volumes or high salt content. Optimum conditions were found using 2 M HNO_3 as feeding solution and 1 M HF as eluent. The results obtained under these con-

Fig. 2. Elution behaviour of the tracer nuclides ^{177}Ta , $^{92\text{m}}\text{Nb}$, ^{88}Zr and ^{175}Hf on the cation exchanger with HNO_3 and HF solution, respectively.

ditions are shown in Fig. 2. Chemical yields of 92% for Ta, 94% for Nb, 87% for Zr and 85% for Hf were obtained, respectively.

No ^{88}Y , ^{167}Tm and ^{169}Yb could be found in the eluate, yielding a decontamination factor of $< 10^2$, a result, which was limited by the detection limits. With ICP-OES a decontamination factor of 5.6×10^5 for added La carrier (1 mg) was measured.

As can be seen from Fig. 2, Ta and, to some extent, also Nb are not completely adsorbed on the cation exchanger from the 2 M HNO_3 feeding solution. They show partial desorption already after 1 ml elution volume, which represents approximately the free column volume. After formation of the anionic fluoride complexes, these two elements are completely eluted. Zr and Hf strongly adsorb from the feeding solution, and are desorbed only after their transformation into fluoride complexes with the elution maximum at 2 ml. Thus, the separation conditions were selected such, that no loss of group 5 elements occurred. This was achieved by dissolving the lanthanum precipitate in < 0.8 ml, corresponding approximately to the free column volume. The fraction between 1 and 3 ml was collected. These experimental conditions were used as final separation scheme for all experimental runs.

To model the chemical behaviour of transactinides with $Z \geq 106$ the separation procedure was applied to ^{192}Ir , ^{191}Pt , $^{199/198}\text{Au}$, $^{197\text{m}}\text{Hg}$, ^{200}Tl , and ^{203}Pb tracers under identical conditions as in the real experiment. The first precipitate of $\text{La}(\text{OH})_3$ carried 36% Pb, 1% Hg, 1% Au, 15% Pt, 14% Ir, and no Tl. After twice dissolving the precipitation in HCl and precipitating again, no γ -lines of the corresponding nuclides could be detected in the lanthanide precipitate containing the group 4/5 elements, which refers to a separation of $> 95\%$.

In the Figs. 3 and 4, the γ -spectra of sample 8, as an example, before and after the chemical separation procedure, are shown. Chemical yields of 52% for Ta, 51% for Hf, 61% for Nb and 50% for Zr, respectively, were obtained from the measured α -lines of ^{90}Nb and $^{86/89}\text{Zr}$, produced in fission reactions, and of the added ^{175}Hf and ^{177}Ta tracers. Taking into account all 8 samples, an average value of $60 \pm 20\%$ was found for the yield of group 4/5 elements.

Ag and Cd isotopes, formed as β -decay products of the compound nuclei from the $^{48}\text{Ca} + \text{Cu}$ reaction as well as the added Y and Tm tracers were separated completely from the group 4/5 elements.

Some other radio nuclides such as $^{47/48}\text{Sc}$ and $^{100/101\text{m}}\text{Rh}$ behave similar to group 4/5 elements on the cation exchanger and are also partially eluted with HF. The separation yields for these two elements were 40 and 97%, respectively. Due to the high β -activity in the final sample, mainly from different isotopes of Zr and Nb, that were produced in the $^{243}\text{Am}(^{48}\text{Ca}, f)$ reaction, the resolution of the α -spectra was unfortunately very poor, which prevented the direct determination of the decontamination factors for actinides.

In the eight final samples, 15 spontaneous fission events were detected within 174 hours at a maximum measuring time of 960 h. A decay analysis of the 15 events points to a single component with a half-life of 32^{+11}_{-7} hours.

Using the corrected values for the fission fragment energies [14] a value of about 230 MeV for the total kinetic en-

Fig. 3. γ -spectrum of an aliquot of the dissolved Cu chips (7 M HNO_3 solution).

Fig. 4. γ -spectrum of the separated sample 8 (2 ml 1 M HF solution).

ergy (TKE), calculated from the 12 measured coincidences, was obtained. The neutron multiplicity yielded 4.2 [12] per fission. A cross section for the production of the long-lived SF nuclide from the $^{48}\text{Ca} + ^{243}\text{Am}$ reaction of $4.2^{+1.6}_{-1.2}$ pb could be determined [12].

The observed overall SF rate in the direct catch experiment was 158 events in 72 days at a beam dose of 2.9×10^{17} . With the obtained separation factor from lanthanides (and actinides) of 5.6×10^5 , actinide isotopes could yield in 174 hours not more than 2×10^{-3} spontaneous fission events.

According to the conditions of the chosen chemical separation procedure, the detected SF nuclide cannot be assigned to any heavy actinide. Furthermore, because of the measured half-life of 32 h, only interferences from actinides with half-lives between 20 and 40 h would have to be considered. Under these conditions, there are only four possible candidates: ^{246}Cf (35.7 h), ^{252}Fm (25.4 h), ^{255}Fm (20.1 h) and ^{245}Bk (1.8 d), respectively. All these isotopes have SF branches of less than 5%. Moreover, if these actinide nuclides would have passed the column, also longer-lived actinides such as $^{252/254}\text{Cf}$ would have been observed. This is obviously not the case, since after 174 h, no SF events were observed during a residual measuring time of 786 h.

Therefore, the observed long-lived nuclide has to be a transactinide with chemical properties corresponding to that of the group 4 and 5 elements. It forms a hydroxide in ammonia solution co-precipitating with lanthanum hydroxide and forms strong anionic fluoride complexes in HF solutions.

Since our experiment was not sensitive to α -decay chains from the produced 115 nuclides, the applied chemical separation should be specific to Rf and Db and not to any other element with $Z \geq 106$. In the following, we assume that the transactinide elements as well as the lighter homologues are oxidized to the highest oxidation state during the dissolution of the Cu-chips with concentrated HNO_3 .

Seaborgium

Literature data show that carrier-free tungsten isotopes form anionic compounds in ammonia solution which are not co-precipitated with $\text{La}(\text{OH})_3$ [17]. Anionic monomeric species of W and Mo are formed already at pH 3.7 [18], and from calculations, the same behavior with only slight differences is expected for Sg [19]. Bohrium

Due to the frequent nuclear medical applications, the aqueous chemistry of carrier-free $^{99\text{m}}\text{Tc}$ and ^{188}Re is the subject of many detailed studies [20]. The liquid chemistry of these two elements is dominated by the pertechnetate/perrhenate species over a wide range of pH [18]. Perhenate and pertechnetate do not precipitate as hydroxides.

Hassium

Gas phase experiments with element 108 (Hs) showed that HsO_4 is volatile already at temperatures of about -40°C [21]. Therefore, Hs is expected to be released already during the dissolution of the copper chips in concentrated nitric acid, especially during the evaporation of the final sample.

Elements Mt-114

Carrier-free ^{192}Ir , ^{191}Pt , $^{199/198}\text{Au}$, $^{197\text{m}}\text{Hg}$, ^{200}Tl as well as low-carrier ^{203}Pb were shown to remain in the solution after the precipitation step. Additionally, in the real experiment, the lighter homologues of the elements Mt, Ds and 111 – Ag, Cd and Rh – were nearly completely separated (see Figs. 3 and 4).

4. Conclusions

The long-lived spontaneously fissioning nuclide, which was produced in the $^{243}\text{Am}(^{48}\text{Ca}, xn)^{291-x}115$ reaction and chemically separated as described, can be assigned to an isotope of Db or Rf, because heavier elements up to element 114 as well as trivalent actinides are well separated. Therefore, this radiochemical experiment can be considered as a confirmation of the recent discovery of the elements 115 and its daughter, element 113, in the $^{243}\text{Am}(^{48}\text{Ca}, xn)^{291-x}115$ reaction based on the published decay chain [1].

Nevertheless, the accurate determination of the atomic number of the spontaneous fissioning isotope of Db or Rf after EC decay of Db [14] requires additional investigations. The long half-life of 32 h eventually allows more complex separation procedures followed by the measurement of several fractions.

This experiment opens up perspectives to study the chemical behaviour of transactinides under novel conditions. Thanks to the observed long half-life, more detailed investigations of chemical properties can now be envisaged. An example is the study of the kinetic behaviour, which was not possible in the past due to the short half-life of the known isotopes.

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