Chemical identification of dubnium as a decay product of element 115 produced in the reaction ^{48}Ca + ^{243}Am

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The recent discovery of the elements 115 and 113 in the reaction ^{48}Ca + ^{243}Am was confirmed by an independent radiochemical experiment based on the identification of the long-lived decay product dubnium.

In experiments[1–6] performed at the Flerov Laboratory of Nuclear Reactions, JINR, 17 isotopes of new chemical elements with the atomic numbers 112–118 have been synthesised. The complete fusion reactions resulting from accelerated ^{48}Ca ions bombarding the targets of ^{238}U, ^{242,244}Pu, ^{243}Am, ^{245,248}Cm and ^{249}Cf were used for the production of the above superheavy elements (SHEs). The nuclei of new elements appeared to mainly undergo α-decays (one or several) until the decay chain ends with a spontaneous fission (SF). The half-lives of the new nuclides range from 0.5 ms to 0.5 min, depending on the proton and neutron numbers of the synthesised nuclei. These results point to a considerable increase in the nuclear stability of SHE isotopes with increasing neutron number. Essentially, they provide the first experimental confirmation of the theoretical predictions of the existence of an 'island of stability' in the domain of hypothetical superheavy elements.

Thin target layers (~0.3 mg cm^{-2}) of highly enriched actinide isotopes were irradiated with ^{48}Ca ion beams of a strictly preset energy. Recoil nuclei knocked out of the target were separated from the ^{48}Ca ions and various reaction products by means of a gas-filled separator, which was tuned to transmit the complete fusion products with an efficiency of about 40%. The decay of nuclei was registered by position-sensitive silicon detectors mounted in the separator focal plane. Correlated decays of single atoms, *i.e.*, chains of sequential α-decays terminated by a spontaneous fission event (α-α-α-…SF), registered by the detector array are interpreted as decay sequences of unknown nuclides. Their identification is based on their radioactive decay properties and the reaction mechanism, in particular, on the characteristic dependence of the yield of neutron-evaporation products on the excitation energy of the compound nucleus. Investigating these dependences requires time-consuming measurements of the production cross-sections of the nuclei of interest at various energies of the ^{48}Ca ion beam.

At the same time, chemical identification of isotopes in the observed decay chains could give us the identity of the atomic numbers of nuclei in the decay chain and provide independent evidence for the discovery of a new element (elements). Such an experiment was proposed[7] for the element 115.

An isotope of the element 115 with the mass number 288 was synthesised[6] in the reaction ^{48}Ca + ^{243}Am → ^{288}115 + 3n. It undergoes five sequential α-decays

$$115 \xrightarrow{\alpha} 113 \xrightarrow{\alpha} 111 \xrightarrow{\alpha} 109 \xrightarrow{\alpha} 107 \xrightarrow{\alpha} 105 \xrightarrow{SF}$$

ending with the spontaneous fission of ^{268}Db (Figure 1).

The total time of the first five α-transitions is about 20 s. The half-life of the spontaneously fissioning final nucleus ^{268}Db was estimated from the three observed events to be $T_{1/2} = 16^{+19}_{-6}$ h. Oganessian *et al.*[6] did not exclude that the experimentally

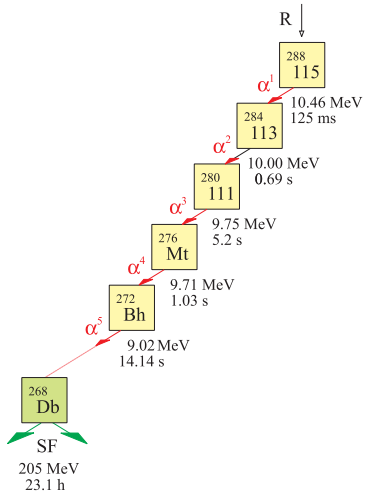

Figure 1 Decay of an isotope of element 115 synthesised in the reaction ^{48}Ca + ^{243}Am using the Dubna Gas Filled Recoil Separator.[6]

observed spontaneous fission of the odd-odd nucleus ^{268}Db could be due to its electron capture followed by a rapid fission of the even-even nucleus ^{268}Rf (T_{SF} ~ 1.5 s). This does not change the conclusion that the experimentally registered long half-life of the spontaneous fission is determined by the decay of ^{268}Db. Due to only a few observed events, it was not also excluded that the isotope ^{268}Db may, in some cases, undergo α-decay followed by the spontaneous fission of ^{264}Lr.

However, direct production of ^{268}Db *via* the transfer of 25 nucleons (10 protons and 15 neutrons) to the ^{243}Am target from ^{48}Ca at an energy close to the Coulomb barrier is extremely unlikely.[8] Therefore, the relatively long lifetime of ^{268}Db and its characteristic decay mode (SF) allowed us to perform, for the first time, an experiment aimed at the chemical identification of Db (Z = 105) as the descendant product from the decay of the element 115, which is produced *via* the ^{48}Ca + ^{243}Am reaction with a cross section of only about 3 pb (3×10^{-36} cm^2).

According to the atomic configuration in the ground state ([Rn]5d^{14}6$p^3$7s^2), Db should belong to the Group V of the Periodic Table as a heavier homologue of Nb and Ta. By studying 34-second ^{262}Db,[9] it was established that Db, like Nb and Ta, is well adsorbed on glass from concentrated HNO_3. In the process of extraction by Aliquat 336 from chloride solutions,[10] its behaviour was closer to that of Nb and differed from Ta and Pa (a pseudo-homologue), while in extractions from fluoride solutions it behaved analogously to Nb and Ta and differed from Pa. In general, one observes the theoretically

predicted[11,12] inversion of the chemical properties within a group of homologues with the transition from 5*d* to 6*d* elements. As was expected, the chemical properties of Db are closer not to Ta but to Nb. Investigations of volatile bromides and chlorides of Db also confirm this conclusion.[13]

For chemical identification, the element should be separated according to its group properties. For this purpose, we developed a method of sorption extraction for the Group V elements as anionic fluoride complexes. Bearing in mind that the $Z = 105$ isotope of interest undergoes SF, we paid special attention to separating the Group V elements from the actinides and, most importantly, from spontaneously fissioning isotopes of californium, ^{252}Cf ($T_{1/2}$ = 2.65 y, SF – 3.1%) and ^{254}Cf ($T_{1/2}$ = 60.5 d, SF – 99.7%). The separation from the actinides including Lr simultaneously answers the question if ^{268}Db undergoes an additional decay followed by spontaneous fission of ^{264}Lr.

The experiment was performed at the FLNR (JINR) U-400 cyclotron in June, 2004. The principal scheme of the set-up for target irradiation is shown in Figure 2.

The 32 cm^2 rotating target consisted of the enriched isotope ^{243}Am (99.9%) in the oxide form. The target material was deposited onto 1.5 μm Ti foils to a thickness of 1.2 mg cm^2 of ^{243}Am. The target was bombarded by ^{48}Ca ions with an energy corresponding to 247 MeV in the middle of the target with an average intensity of 5×10^{12} ion s^{-1}. A 10 mm diameter collimator was used to limit the irradiated area. The recoiling reaction products on leaving the target passed through a second 12 mm collimator positioned 10 mm from the target and were stopped in a copper catcher block. The 50 mm diameter catcher was positioned on the beam axis, 100 mm downstream from the target. The efficiency of collecting the products of fusion of ^{243}Am and ^{48}Ca nuclei in such a geometry (capture angle of ±12.5°) was close to 100%. The range of recoils in the copper catcher did not exceed 3–4 μm. Eight similar experimental runs with duration between 20 and 45 h were performed.

For each run, after the end of irradiation, the catcher block was transported to the radiochemical laboratory. The surface was thoroughly cleaned of any aerosol particles carrying ^{243}Am, and then a 7- to 10-μm layer (120–180 mg of Cu) was cut from the surface using a micro-lathe. The copper chips were dissolved in 10 ml of concentrated HNO_3. The final nitric acid solution was a complex system containing a large amount of copper (catcher material), products of reactions of ^{48}Ca with Cu, implanted into the catcher ^{243}Am fission fragments, and products of multinucleon transfer reactions (from the ^{48}Ca nucleus to the ^{243}Am target nucleus), including the long-lived spontaneously fissioning isotopes ^{252}Cf and ^{254}Cf. For the spectrometric control of the procedure of isolation of the Group V elements and actinides, we added aliquots of nitrate solutions containing ^{92m}Nb ($T_{1/2}$ = = 10.15 d), ^{177}Ta ($T_{1/2}$ = 56.6 h), ^{169}Yb ($T_{1/2}$ = 32 d) and ^{167}Tm ($T_{1/2}$ = 9.25 d) to the solution.

Isolation of the Group V elements from the nitric acid solution was accomplished using the following steps:[14]

(*a*) Separation of the reaction products from the macro component (copper) *via* their quantitative co-precipitation with lanthanum hydroxide at pH 7 in ammonium media; the copper remained in solution as an ammonium complex. The precipitation step was repeated in order to prepare thin spectrometric sources with a minimum content of ballast material at the final stage. The final precipitate was then dissolved in 2 M HNO_3.

(*b*) Separation of the Group V elements from lanthanum and actinides *via* sorption from nitric acid on Dowex 50×8 cation-exchange resin and subsequent elution of the Group V elements as fluoride anionic complexes with 2 ml of 1 M HF. The eluent was then evaporated to a volume of 0.1 ml.

(*c*) Preparation of thin sources (working samples) for measurements by depositing the solution onto a polyethylene foil (0.4 μm thick, 15 mm in diameter) using a capillary, with subsequent drying in a stream of hot helium.

In control experiments with non-irradiated copper catchers and added into the solution radionuclides ^{92m}Nb, ^{177}Ta, ^{167}Tm and ^{169}Yb it was shown that the present technique allows the extraction of Group V elements with an efficiency of 90% and suppressing the lanthanides by a factor of ~10^5. From spectrometric measurements with irradiated sources, ^{92m}Nb and ^{177}Ta were isolated with efficiencies of 85±5% and 75±5%, respectively, with the suppression of actinides by a factor of ≥ 8×10^3 (this value is estimated from the detection limits of ^{169}Yb from the working samples).

The chemical procedure took 2 to 3 h, starting from the end of irradiation until the beginning of detector measurements. With this routine of work, we could count on the effective registration of atoms with the half-life $T_{1/2} \geq 10$ h.

For the detection of α-particles and spontaneous fission fragments, we used a detection module with four identical chambers, each with two semiconductor detectors. The detectors had a surface area of 6 cm^2 and were mounted facing each other with a 4 mm spacing in between. The test sample was placed between the two detectors. All the chambers were positioned inside a neutron detector registering spontaneous fission neutrons. The neutron detector had 72 ^{3}He counters positioned in a polyethylene moderator in three concentric rings at different distances from the chamber with samples. The detector array was calibrated with ^{248}Cm and ^{252}Cf sources. The efficiency of detecting fission fragments in the semiconductor detectors was about 90% and neutrons were detected with an average efficiency of about 40%. In the course of the 330 h test run performed before the experiment no background SF events were detected.

During eight runs of irradiating the ^{243}Am target with ^{48}Ca ions (with a total beam dose of 3.4×10^{18}), we detected 15 spontaneous fission events. The measurements were carried out for 957 h. The 15 SF events appeared in a 174 h interval following the start of the measurements. No SF events were detected in the subsequent 783 h. The conditions of irradiation (duration of each irradiation and ^{48}Ca beam dose) and the results of measurements for every sample (energy of spontaneous fission fragments, corrected for energy loss in the source and backing layers, neutron multiplicity registered by ^{3}He counters for every single event of spontaneous fission, and the time of the event registration from the start of measurements) are given in Table 1. The half-life of 32^{+11}_{-7} h determined from the time distribution of SF events agrees with the half-life obtained in the physical experiment within statistical errors (Table 2).

The total kinetic energy of the fission fragments ($\overline{\text{TKE}}$), determined as the sum of amplitudes of the time-coincident signals from both detectors and corrected for energy loss in the source and backing layers was about 235 MeV. This result also agrees with the data from the physical experiment ($\overline{\text{TKE}}$ ~225 MeV). The average neutron multiplicity per fission, determined from the coincidences of signals of different repetition factors from ^{3}He counters, was ν ~4.2 (Figure 3). Both parameters, *i.e.*, the high $\overline{\text{TKE}}$ value and the high neutron multiplicity, give evidence for the fission of a rather heavy nuclide. For comparison, in the spontaneous fission of ^{248}Cm, $\overline{\text{TKE}}$ = = 181 MeV and ν = 3.14 and in ^{252}Cf, $\overline{\text{TKE}}$ = 185 MeV and ν = 3.75.

The ninth experiment was carried out under the same conditions, with the same beam energy as in the previous eight runs, but without chemical separation of the ^{243}Am + ^{48}Ca reaction products. The goal of this experiment was to determine the

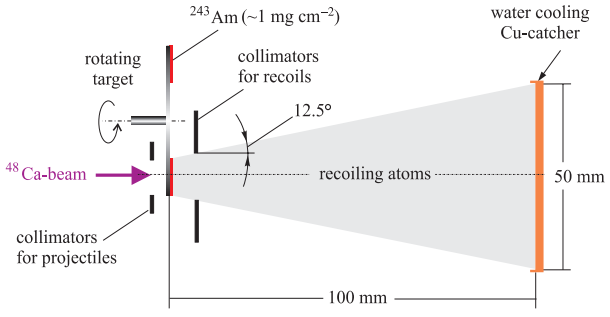

Figure 2 Scheme of the experiment.

Table 1 Conditions and results of the experiment.

No.	Time of irradiation/h	^{48}Ca ion beam dose	Energies of fission fragments E_1/E_2 (MeV)	Neutron multiplicity registered in every event of spontaneous fission	Time of registration/h
1	20	2.5×10^{17}	120/126	2	20
2	22	3.7×10^{17}	–/86	1	74
3	22	3.4×10^{17}	131/124	1	15
			116/122	2	72
4	22	2.9×10^{17}	104/120	1	22
			97/125	1	29
			100/128	1	51
5	38	6.7×10^{17}	117/118	2	6
			108/107	3	9
			110/104	0	15
			–/76	2	68
6	23	3.9×10^{17}	120/114	2	39
7	22	3.6×10^{17}	–	–	–
8	45	7.4×10^{17}	119/110	2	5
			118/105	2	93
			65/58	3	174

background of spontaneously fissioning nuclei (mainly Cf isotopes) implanted into the catcher. After the end of irradiation for 20 h (the ^{48}Ca ion beam dose was 2.9×10^{17}), the catcher surface was cleaned of ^{243}Am-carrying aerosol particles and put in contact with a PETP solid-state track detector. During the course of a 72 day exposure, the detectors were changed at certain time intervals and subjected to a physico-chemical treatment in order to develop latent tracks of SF fragments.

A total of 158 tracks were counted, the counting rate of about 2 events per day was not virtually changed throughout the procedure. With a separation factor of Group V elements from actinides of $\geq 8\times10^3$, actinide isotopes, including ^{264}Lr, could yield in 174 h no more than 0.02 spontaneous fission events.

Note that, in our experiment, the Group V and IV elements are separated together. That is why the method used can be applied to the isotopes of the element 105, as well as to those of the element 104, the final descendant products of decay from parent nuclei with $Z = 115$ and 114. However, the observation in our experiment of spontaneous fission of Rf isotopes, as final descendant products in decay chains of the 114 element isotopes, is excluded because of the negligibly low cross section of the reaction ^{243}Am(^{48}Ca,pxn)$^{290-x}$114 as compared with that of ^{243}Am(^{48}Ca,xn)$^{291-x}$115, and due to short life times of the nuclides (a few seconds) in the decay chains of the element 114 isotopes[1–5] (see 'Chart of the Nuclides' on the back cover of the issue). That is why all the events of spontaneous fission detected in the reaction ^{243}Am + ^{48}Ca should only be attributed to the decay chain of the element 115.

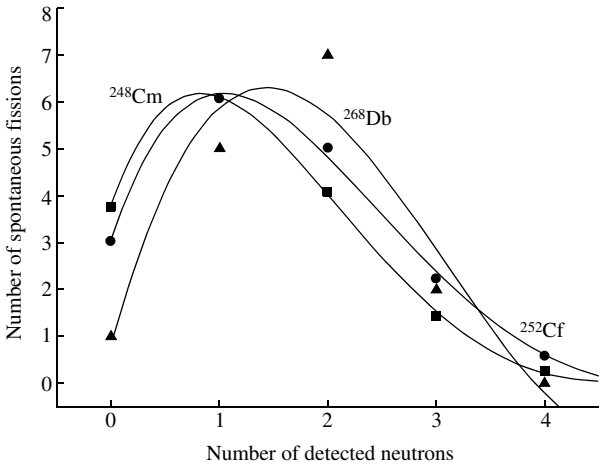

Figure 3 The neutron multiplicity registered by ^{3}He-counters per act of spontaneous fission (without taking into account the efficiency of the detector): (▲) samples 1–8; (●) distribution of thin ^{252}Cf sources measured in the experiment; (■) the same for the ^{248}Cm thin source. The lines are drawn to guide the eye; the arrows point at the distribution maxima. The data for ^{252}Cf and ^{248}Cm are in the scale 1:10000.

Table 2 shows data of the two experiments aimed at the determination of properties of the isotope of the element 105, which closes the chain of sequential α-decays of the element 115 produced in the reaction ^{243}Am + ^{48}Ca.

From the yield of spontaneously fissioning nuclei, one can determine the cross section of the parent nucleus, the element 115, as an evaporation product from the reaction ^{243}Am + ^{48}Ca. According to our data, this formation cross section is about 4 pb (4×10^{-36} cm^2). This agrees with the value ($\sigma_{3n} \sim 3$ pb) measured in the experiments with the gas-filled separator.

Note that due to the high efficiency of the chemical separation of the reaction products and the possibility of employing relatively thick target layers, the yield of the isotopes of superheavy elements produced with the present experimental technique is about a factor of five higher than that obtained with kinematic separators.

For the first time, the chemical identification of Db as a final descendant product of the decay of element 115 produced in the reaction ^{243}Am + ^{48}Ca was performed.

The data obtained in this work confirm that the spontaneous fission observed in experiments[6] is due to the decay of the element with the atomic number $Z = 105$.

The properties of the isotope ^{268}Db produced in the decay chain of the element 115 using the gas-filled recoil separator agree by all measured parameters with the data of the present chemical experiment on the determination of its atomic number.

The decay chain of the element 115 synthesised in the reaction ^{243}Am + ^{48}Ca consists of five sequential α-transitions resulting in the formation of the long-lived nucleus of the element 105 leading to spontaneous fission. A hypothesis of the possible α-decay of the Db nucleus has been totally rejected by the conditions of chemical separation of the reaction products in this experiment.

Thus, the data from the present experiment are the independent evidence for the synthesis of the element 115, as well as the element 113, in the reaction ^{243}Am + ^{48}Ca.

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Table 2 Results of physical and chemical experiments.

	Physical experiment	Chemical experiment
Separation method	Kinematic separator	Radiochemical separator
Separation efficiency	35%	80%
Detection method	Decay chains of nuclei with $Z = 115$	SF of nuclei with $Z = 105$
Energy of the ^{48}Ca ion beam at the center of the target layer	246 MeV	247 MeV
Total ^{48}Ca beam dose	4.5×10^{18}	3.4×10^{18}
Thickness of the ^{243}Am target	0.36 mg cm^{-2}	1.2 mg cm^{-2}
Number of detected spontaneous fission events	3	15
Formation cross section for the nuclei with $Z = 115$	$2.7^{+4.8}_{-1.6}$ pb	$4.2^{+1.6}_{-1.2}$ pb
Half-life	16^{+19}_{-6} h	32^{+11}_{-7} h
Total kinetic energy of spontaneous fission fragments	~225 MeV	~235 MeV
Average neutron multiplicity per fission act	–	4.2
Identification method of SF-decaying nuclei in the reaction ^{48}Ca + ^{243}Am	Method of excitation functions (Z = 115)	Isolation of Group V elements (Z = 105)

separations and to A. A. Voinov, I. V. Shirokovsky, O. N. Malyshev, V. A. Gorshkov and S. P. Tretyakova for their assistance with the irradiations and long-time measurements.

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