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Pioneering experiments on the chemical properties of element 113

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First experimental results of a chemical investigation of element 113 independently confirm the synthesis of the new elements 115 and 113 in the nuclear fusion reaction of ^{48}Ca + ^{243}Am and indicate a chemical behaviour resembling a species with a high volatility, a weak interaction with inert surfaces and an enhanced reactivity towards gold surfaces.

The discovery of new superheavy elements with the atomic numbers 113–118 including the observation of new neutron-rich isotopes of elements 104–112 represent one of the scientific highlights in the past decade. The priority experiments were carried out at the Joint Institute for Nuclear Research (JINR) in Dubna (Russian Federation) at the accelerator facility U-400 of the G. N. Flerov Laboratory of Nuclear Reactions (FLNR).[1,2] The synthesis was performed through complete nuclear fusion reactions of the doubly magic nucleus ^{48}Ca with neutron-rich actinide nuclei (^{238}U, ^{237}Np, ^{242,244}Pu, ^{243}Am, ^{249}Cm, ^{249}Bk, ^{249}Cf). More recently, the observed synthesis of elements 112–117 was confirmed at Helmholtz Centre for Heavy Ion Research (Darmstadt, Germany)[3,4] and Lawrence Berkeley National Laboratory (LBNL) (Berkeley, USA).[5] In 2012, based on an IUPAC decision, elements 114 and 116 were given the names Flerovium (Fl) (to honor the FLNR and its founder Academician G. N. Flerov) and Livermorium (Lv) [to honor Lawrence Livermore National Laboratory (LLNL) (USA), scientists from which actively participated in the research in Dubna].

Relatively long lifetimes observed for some of the newly discovered nuclides allowed one to perform pioneering experiments[6–8] to explore the chemical properties of elements 112 (Cn) and 114 (Fl), which showed that both elements are highly volatile at ambient temperature and adsorb on gold surfaces at negative (Celsius) temperatures. Statistical analysis of published data[7] made it possible to estimate the standard adsorption enthalpy of element 112 on gold surfaces as $-\Delta H_{\mathrm{ads}} = 52^{+4}_{-3}$ kJ mol^{-1}. It led to the conclusion that the trends of properties established by D. I. Mendeleev's periodic table are preserved within Group 12 elements and Cn is a heavier homologue of mercury.

The aim of this work was to perform the first experiments to reveal the chemical properties of element 113. In accordance with the periodic table, element 113 is a heavier homologue of Tl in Group 13 having most probably the electronic ground state configuration [Rn]$5f^{14}6d^{10}7s^27p^1$. First theoretical predictions of the properties of element 113 were made in the 1970s by Keller *et al.*,[9] generally related to attempts to find superheavy elements in nature. The synthesis of the relatively long-lived isotopes ^{284,285}113 performed recently in Dubna have substantially stimulated interest in the theoretical estimations of important chemical properties for element 113.[10–13]

Nowadays, investigations of the adsorption behaviour on gold surfaces based on fast gas chromatography methods are most frequently used to explore the chemical properties of superheavy elements on a one-atom-at-a-time basis with lifetimes lasting for seconds. These methods are based on the thermalization of the recoiling products of heavy-ion beam interactions with target atoms in inert gas atmospheres, followed by the transportation of volatile species by a gas jet through a thin capillary into a detector module with the subsequent adsorption of test elements on the surface of Si-semiconductor detectors covered by a thin gold layer and the detection of their unique decay. The method implies that this element is highly volatile and not interacting with any inert surface of the transport capillary (usually, Teflon). The deposition temperature determined in the experiment is a key parameter that characterizes the nature of the chemical element as reactive or inert. This is a pioneering method to chemically characterize elements 112 and 114.[6–8]

Similarly to the cases of Cn and Fl, the chemical identification of element 113 is based on the assumption that it is a typical member of Group 13 revealing properties in accordance with its specific treatment as a member of the 7th period including relativistic DFT simulation of its interactions with gold clusters of 4 to 58 atoms represented the adsorption site of element 113 on the stable gold (111) surface ($-\Delta H_{\mathrm{ads}}$ = 106±10 kJ mol^{-1}).[10,11] The relativistic calculations made by Pershina *et al.*[12] predict that element 113 is weakly adsorbed on inert surfaces due to the strong contraction and stabilization of the outer $7p_{1/2}$ orbital. The estimated value of $-\Delta H_{\mathrm{ads}}$ on Teflon is 14 kJ mol^{-1} or 16 kJ mol^{-1} on polyethylene. At the same time, element 113 is predicted to strongly adsorb on gold surfaces in a He/H$_2$ atmosphere ($-\Delta H_{\mathrm{ads}}$ = = 158.6 kJ mol^{-1}).[12] Element 113 is expected to be deposited at about 400–450 °C on gold surfaces. In the presence of oxygen and water traces, 113(OH) becomes the most favourable species, which is predicted to show a much weaker adsorption on the gold surface. In that case, the expected adsorption enthalpy is $-\Delta H_{\mathrm{ads}}$ = 113 kJ mol^{-1}.[13] Both predictions transform to an expected deposition upon first surface encounter with gold held at room temperature. As Si-detectors intrinsically cannot be heated significantly above room temperature, only a lower limit of ΔH_{ads} can be determined.

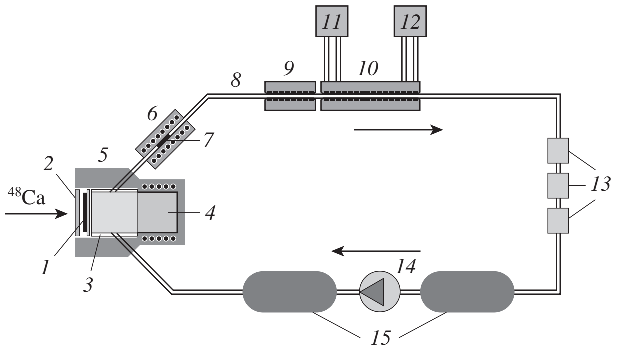

Figure 1 Schematic diagram of the experimental setup for studying the chemical properties of element 113: (*1*) ^{243}Am (1.5 mg cm^{-2}) + ^{nat}Nd (15 μg cm^{-2}) target on the backing of Ti (2 μm); (*2*) vacuum window (4 μm Ti foil); (*3*) cylindrical quartz insertion; (*4*) beam-stop with water cooling; (*5*) target chamber; (*6*) oven; (*7*) quartz filter; (*8*) transport capillary; (*9*) isothermal detector of 16 pairs of Au(Si) detectors at ambient temperature; (*10*) cryodetector of 32 pairs of Au(Si) detectors; warm end at +20 °C and cold end at –50 °C; (*11*) water thermostat; (*12*) cryothermostat; (*13*) gas purification system; (*14*) pump; and (*15*) buffer volumes.

Recently, five longer-lived isotopes of element 113 (mass numbers of 282–286) have been discovered. The ^{282}113 isotope was synthesized in hot fusion between ^{48}Ca ions and ^{237}Np target material, others were detected as daughters and grand-daughters in the decay chains of corresponding 115 and 117 isotopes, which were produced in reactions of ^{48}Ca with ^{243}Am or ^{249}Bk targets, respectively.[14]

The current experiment was performed using the U-400 cyclotron at FLNR (JINR). The nuclear fusion reaction of ^{48}Ca with ^{243}Am was used in the experiment leading to the formation of ^{284}113 and ^{285}113 nuclides. The lifetimes of these isotopes are ~1.0 and ~6.0 s, respectively. The target material ^{243}Am (99.52%) was produced at Research Institute of Atomic Reactors (RIAR) (Dimitrovgrad, Russian Federation). The target used for irradiation was prepared by the molecular plating of ^{243}Am with an admixture of ^{nat}Nd oxide (15 μg cm^{-2} of Nd) on Ti backing foil (2 μm, 4 cm^2) at FLNR. The final target thickness of ^{243}Am was 1.5 mg cm^{-2}.

Figure 1 shows a schematic diagram of the experimental setup. A stationary target was installed on a supporting copper grid (about 80% transmission) with a ^{243}Am oxide layer facing the beam and irradiated by a ^{48}Ca beam that passed through a 4 μm Ti vacuum window. The average intensity of ^{48}Ca particles was 3×10^{12} s^{-1} at the beam energy of 242 MeV in the middle of the target. The nuclear reaction products recoil from the irradiated target and were stopped in the inert carrier gas mixture (70% He + + 30% Ar, 110 kPa) flushing through the target chamber (volume, ~21 cm^3; $D_{\text{in}} = 2.1$ cm; $L = 6$ cm). Aerosol particles produced by beam sputtering were stopped on a quartz wool filter heated to 600 °C, which was installed directly after the chamber. Then, volatile species were further transported through a thin ($D_{\text{in}} = 2$ mm) Teflon capillary 4 m in length (total volume, 12 cm^3) into the detection system. The carrier-gas flow rate was 1.5 dm^3 min^{-1}; thus, the maximum transportation time of the reaction products from the target chamber to the detectors, assuming a complete gas exchange in the target chamber and transport capillary, was about 1.5 s. After passing the detector, the gas mixture was pumped through filters of high purification from oxygen and water traces and was loped back into the target chamber. The Teflon capillary was inserted into a hermetic metallic tube, and a large part of it (from the quartz wool filter to the detector) was heated to 70 °C.

Two detection modules in series were used in order to detect the events. The first one comprises 32 detectors mounted on opposite sides in a channel and facing each other, thus forming a chromatography channel with 16 sandwich-like detector pairs which form a rectangular stationary chromatographic surface.[15] Their surfaces were dusted with a thin layer of gold. All these detectors were kept at ambient temperature. The second module was made as a cryodetector comprising 32 of the above pairs of detectors also covered with a thin layer of gold. The temperature along the detector array changed from 20 °C (the first pair of the detectors) down to –50 °C (the last pair of the detectors). The active surface area of each detector in both modules was 1.12 cm^2 (14×8 mm). The gap between detectors was 2 mm. In the described geometry, the efficiency of detection of alpha particles and coincident spontaneous fission fragments by each pair of detectors was 86%. The energy resolution for alpha particles in the carrier gas was about 200 keV (FWHM).

The deposition yield of ^{185}Hg formed in a fusion reaction of ^{48}Ca with ^{nat}Nd was measured simultaneously in order to determine and ensure the level of target integrity and the status of detectors surface. During irradiation for 324 h, the distribution of ^{185}Hg was

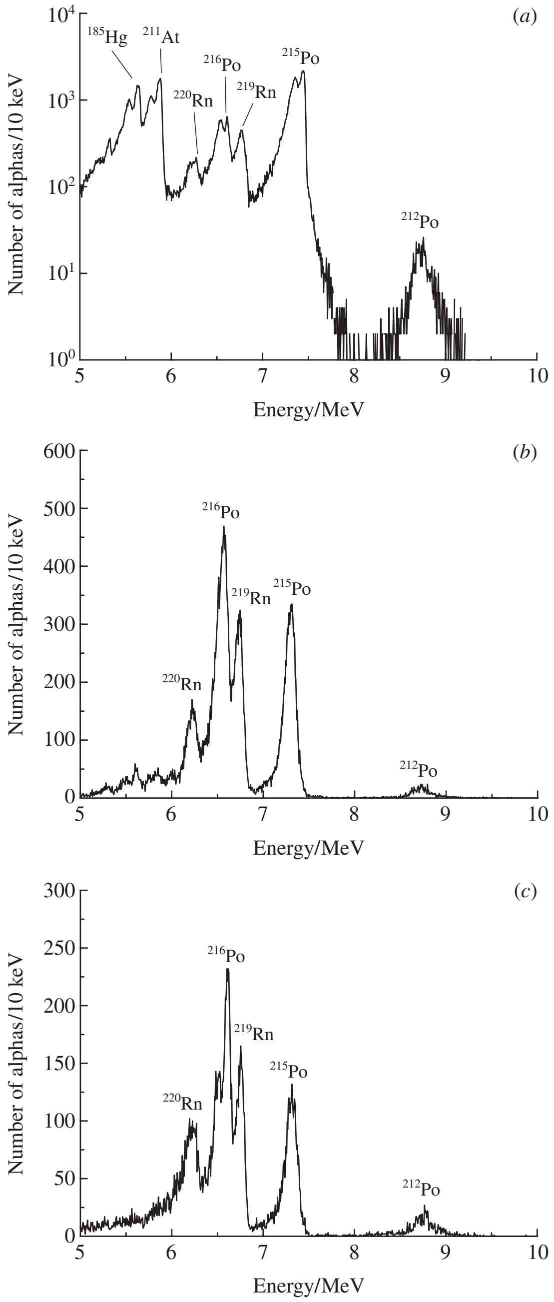

Figure 2 Integral alpha spectra from all detectors of (*a*) isothermal detector (20 °C), (*b*) warm end of the cryodetector (20 °C) and (*c*) cold end of the cryodetector (–50 °C); measurement time, 37.5 h; integral particle flow, 4.2×10^{17}.

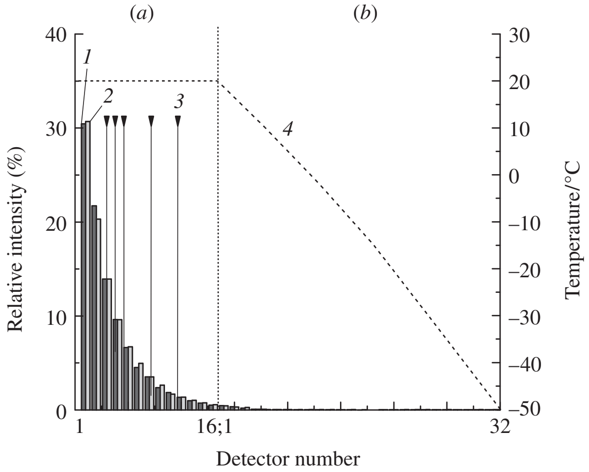

Figure 3 Distribution of (*1*) ^{185}Hg and (*2*) ^{211}At in the detector modules together with (*3*) the position of the observed decay chains attributed to ^{284}113; dashed line (*4*) represents the temperature gradient from +20 to –50 °C at (*a*) isothermal and (*b*) cryomodules of the detector.

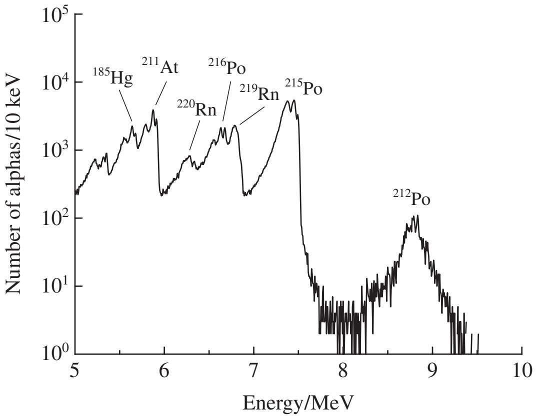

Figure 4 Typical alpha sum spectrum recorded as a decay chain of the element 113.

not disturbed and there was no significant evidence for the target to be destroyed. The production levels of At and Rn isotopes, which were synthesized as a result of multinucleon transfer reactions between ^{48}Ca and ^{243}Am, also confirmed the target integrity. The overall irradiation time for the first two targets was 710 h. The integral flux of ^{48}Ca ions on both targets was 8×10^{18}. Only the radionuclides of volatile and relatively inert elements reached the detector modules, as it was expected for the gas jet transportation of the products of the reaction ^{48}Ca + ^{243}Am. Typical alpha spectra were measured by an isothermal detector at ambient temperature [Figure 2(*a*)] and by a cryodetector at +20 °C [Figure 2(*b*)] and –50 °C [Figure 2(*c*)]. The energy spectra show the measured energy of alpha particles. The energy loss varies in the range of 50 to 400 keV when traversing through gas and Au on the opposite detector, depending on the angle of emittance of alpha particles.

Alpha decays recorded by the detectors belonged generally to ^{185}Hg, ^{211}At and ^{219,220}Rn radionuclides and their daughters, which are ^{212,215,216}Po. The almost complete adsorption of ^{185}Hg and ^{211}At on the isothermal detector module surfaces at 20 °C [Figure 3(*a*)] was observed in the experiment, while only trace amounts were detected in the cryodetector [Figure 3(*b*)]. ^{185}Hg and ^{211}At distribution on the detector modules was similar to that observed in studies on the chemical properties of Cn and Fl.[6–8]

Five pairs of correlated fission fragments were recorded in the experiment at the detector pairs 3, 6, 7, 10 and 13. Figure 4 shows a typical sum spectrum of a pair of the detectors.

As noted previously, two relatively long-lived isotopes of element 113 are formed in a reaction between ^{48}Ca and ^{243}Am. The atomic masses of these isotopes are 284 and 285 and the average lifetimes are ~1.0 and ~6.0 s, respectively. The half-life decay periods of the final products of chains decaying in spontaneous fission mode are 27 h (^{268}Db) and ~22 s (^{281}Rg), respectively. Experimental data obtained using the Dubna Gas-Filled Recoil Separator (DGFRS) FLNR[14,16,17] were applied to a search for the correlated chains of alpha decays of isotopes ^{284,285}113. No correlated chains of events corresponding to the isotope ^{285}113 were found. However, five correlated chains of events of the isotope ^{284}113 were identified (Figure 5). The search was applied for expanded time intervals ($0 \leqslant t \leqslant 5t_{1/2}$). The following ranges were selected for alpha decay energies: ^{284}113 – 9750 keV $\leqslant E_\alpha \leqslant$ 10250 keV, ^{280}Rg and ^{276}Mt – 9000 keV $\leqslant E_\alpha \leqslant$ 10000 keV, ^{272}Bh – 8750 keV $\leqslant E_\alpha \leqslant$ 9250 keV.

The random probability of coincidental observation of the given decay chains was performed applying the Poisson statistics.[18] Thus, probabilities for stochastic coincidences are 7.6×10^{-3}, 6.6×10^{-5}, 2.0×10^{-4}, 6.7×10^{-10} and 5.7×10^{-6} for decay chains [Figure 5(*b*)–(*f*)], respectively. The direct synthesis of ^{284}113 in the multinucleon transfer reaction of ^{243}Am with ^{48}Ca ions (18 protons and 23 neutrons) is virtually impossible. Therefore, we conclude that the isotopes of 113–Rg–Mt–Bh–Db elements detected in the experiment are the daughters of primarily produced short-lived nuclide ^{288}115, as observed previously [Figure 5(*a*)].[14,16,17]

With consideration for all transport and detection efficiencies, one would expect to detect 10 decay chains related to ^{284}113 and two decay chains of ^{285}113 under the experimental conditions. However, only five chains of ^{284}113 and no chains of nuclide ^{285}113 have been detected. This observation combined with the fact that isotope ^{284}113 which has a short half-life ($t_{1/2}$ ~ 1 s) was detected proves that transport time was not the reason for

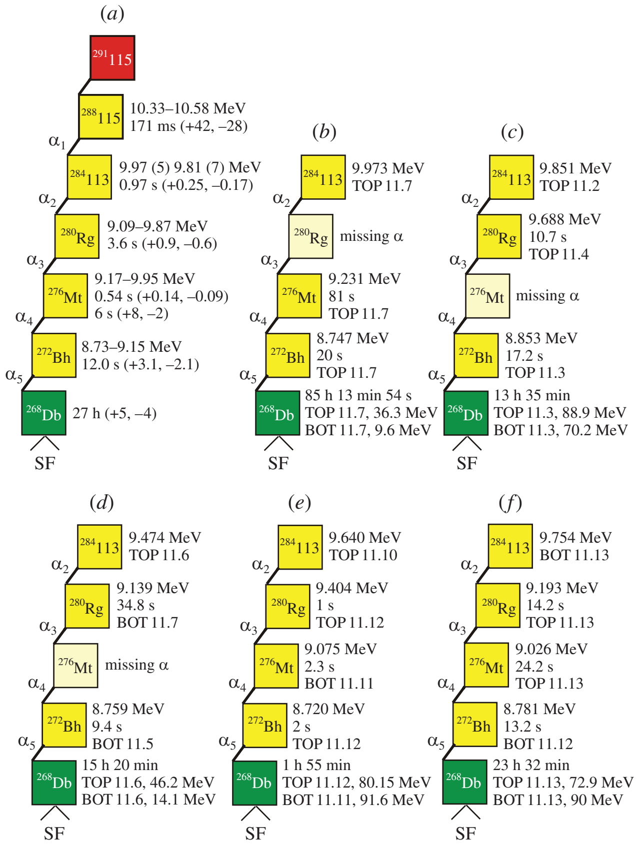

Figure 5 (*a*) Average lifetimes and decay energies acquired at DGFRS and (*b*)–(*f*) decay chains attributed to ^{284}113, measured in the experiment.

the low efficiency observed in the experiment. One of the most probable reasons might be that the stopping of products recoiling from the target into the beam plasma heated carrier gas is due to the density loss in the carrier gas diminished. Consequently, a part of the products was lost due to implantation into the target chamber walls.[19] Additional experiments are necessary to verify this hypothesis.

Figure 3 shows the distribution of the decay observations of $^{284}113$ and ^{185}Hg in the gas chromatography detector. Element 113 should be relatively volatile in accordance with theoretical predictions, *i.e.*, exhibit a weak adsorption on inert surfaces and reveal a strong adsorption interaction on gold surface similar to its lighter homologue thallium in Group 13.[10–13] However, thallium was shown to react in contact with quartz immediately to TlOH.[20] This reaction also cannot be excluded to occur for element 113. Therefore, we conclude that the element 113 species observed in our experiment is generally confirming theoretically predicted properties listed for atomic element 113. It is a volatile (gas-jet transportation at 70 °C) and does not strongly adsorb on surfaces like Teflon. Its final deposition occurred on the surfaces of detectors covered with gold and held at ambient temperature. The observed deposition pattern of the five events does not allow for the absolute quantification of an adsorption enthalpy on gold surface. However, the observation of its deposition at room temperature rather than in the temperature gradient makes it possible to estimate a lower limit adsorption enthalpy of the observed element 113 species on gold as $-\Delta H_{\mathrm{ads}} > 60$ kJ mol^{-1}.

Thus, we experimentally detected five decay chains characteristic of $^{284}113$, which is the alpha-decay daughter of $^{288}115$, using the irradiation of ^{243}Am targets with a ^{48}Ca beam, product thermalization and the transportation of reaction products by a gas jet through a quartz filter and a Teflon capillary to the silicon detectors (gold covered surfaces).

The chemical species of element 113 observed here for the first time was found volatile; it weakly interacts with Teflon surfaces but reacts much more strongly with gold surfaces. Its adsorption behaviour is comparable to the adsorption properties of mercury and astatine in our experiment. Based on an analogous behaviour to the next lighter homologue thallium, we tentatively attribute the observed species to either atomic element 113 or its hydroxide 113(OH).

In general, the experimental data independently confirmed the synthesis of element 115 in the nuclear fusion reaction $^{48}Ca + {}^{243}Am$.

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