

# MODEL EXPERIMENTS ON CHEMICAL PROPERTIES OF SUPER HEAVY ELEMENTS IN AQUEOUS SOLUTIONS

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*Dedicated to Adam Sobiczewski in honour of his 70th birthday*

This paper presents a brief review of model experiments on investigation of chemical properties of transactinide elements, ranging from 104 to 116. The possibilities of isolation of the nuclei of these elements from nuclear reaction products, using the ion-exchange method, are also considered.

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## 1. Introduction

The investigation of the chemical properties of transactinide elements is a fundamental problem of the present-day radiochemistry. The first transactinide element with  $Z = 104$  is a special interest. According to the Periodic Law, its chemical properties should be generally similar to those of the 4<sup>th</sup> group of elements, Ti, Zr and Hf, and very different from the preceding elements of the actinide series with  $Z \leq 103$ .

However, the experimental verification of this definite prediction is associated with great difficulties, first of all, due to the short lifetimes and the extremely small production cross sections of isotopes of element 104, which can be synthesized in the nuclear reaction induced by heavy ions. Nevertheless, as early as in 1966 [1] it was shown that the higher chloride of element 104 has a similar volatility as the hafnium chloride, while both are much more volatile than the compounds of the actinide series elements. Thus, the expected abrupt change in properties in going from  $Z = 103$  to 104 has been confirmed. Such studies of the second hundred elements, carried out using the methods of gas-solid chromatography and thermochromatography

of halides or other volatile compounds, have been extended [2]. They are indispensable in working with nuclides whose lifetimes lie in the range from 0.1 to 10 seconds and which decay via spontaneous fission, for instance,  $^{259}\text{Rf}$ .

Another, traditional experimental approach is the study of the properties of transactinide elements by solution chemistry methods. It rests on the achievements of classical radiochemistry. Here the chromatographic principle is also widely used. But in contrast with gas chromatography processing where the substance can migrate down the column with a high speed, the retention time in an ion exchange or similar column is necessarily much longer. In 1970 Silva *et al.* [3] utilised the then newly discovered  $\alpha$ -active isotope  $^{261}\text{104}$  with a half-life of about 1 min (it remains to be the longest-lived known isotope of the element) in the first attempt to investigate the properties of element 104 by an ion exchange chromatographic technique. They performed several hundred standard batch experiments: after the products of the nuclear reactions had been accumulated during several minutes, they were passed through a cation exchange column in a solution of ammonium  $\alpha$ -hydroxyisobutyrate. Actinide elements were almost irreversibly adsorbed on the resin, whereas Zr and Hf appeared in the first drops of the eluate. Just in these drops 17  $\alpha$ -particles with energies from 8.2 to 8.4 MeV were detected, which corresponded to the known  $\alpha$ -decay properties of  $^{261}\text{Rf}$  and its daughter  $^{257}\text{102}$  ( $T_{1/2} = 20\text{ s}$ ). The authors of Ref. [3] arrived to the conclusion that the behaviour of element 104 was entirely different from trivalent and divalent actinide elements but was similar to Hf and Zr.

Five years later Hulet *et al.*, Ref. [4] investigated the extraction of element 104 with trioctylmethylammonium chloride from concentrated solutions of hydrochloric acid by using the same approach. They detected a total of 6  $\alpha$ -particles with energies from 8.0 to 8.4 MeV in three different fractions of the eluate and concluded that the chloride complex strength of element 104 was notably greater than that of the trivalent actinides and much more like that of Hf.

It should be noted that the overall experimental efficiency achieved in Refs [3] and [4], judging by the known cross section of the synthesis reaction, was very low despite the use of a sophisticated and expensive automated apparatus. A typical experiment included the following operations and processes:

1. periodic accumulation of nuclear reaction products, that recoiled from the target, on a collector using the gas-jet technique;
2. delivery of the collector to the technical apparatus and applying the activity onto a column;
3. forcing the eluent through the column;

4. evaporation of the eluate drops to dryness, the flaming of the residue;
5.  $\alpha$ -spectrometric measurements of the obtained samples by a system of semiconductor detectors.

At all the steps, losses of  $^{261}\text{104}$  could occur as a result of its radioactive decay (during long-time operations) and/or a low chemical yield, or because the measurement geometry was poor ( $\leq 2\pi$ ). To minimize total losses, the operations should be compatible to the half-life of the nuclide under study and other losses should be kept at minimum. As is seen from the first experiments [3,4] this is the difficult requirement to meet. At the same time, in order to make a more detailed study of chemical properties of element 104, one needs quantitative investigations with much better counting statistics.

## 2. Rutherfordium — Element 104

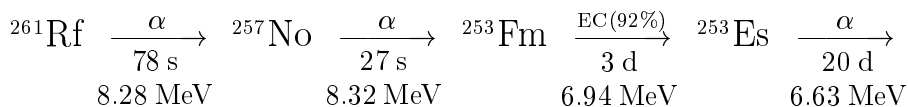
A basically new approach was used to the experiments on the solution chemistry of rutherfordium which resulted in much higher efficiency, *i.e.* the yield of detectable atoms. This was achieved due to the use of some novel techniques [5].

Any experiment virtually consists of three successive stages:

1. separation of the Rf atoms from the target material and isolation from the mixture of the radionuclides produced in the bombardment; as a rule, certain degree of separation from actinides is required because their isotopes interfere with the detection of Rf isotopes (radiochemical “decontamination” may take place at the second stage too);
2. actual investigation of the element properties when conditions are created to obtain several separate “chemical fractions” and the element gets in one or another fraction depending on the relative probability of the chemical reaction involved;
3. evaluation of the results of the previous stage by determining the quantity of atoms of the nuclide under study in different fractions.

The essence of our approach was, as follows:

At the first stage Rf is separated from actinide elements, which are inevitable produced in the same bombardment with much higher cross sections, to a high enough degree. Thus, at the last stage, there is no necessity to measure the short-lived Rf isotope itself and to carry out the low efficiency operations of quick preparation of samples and radioactivity measurements. Instead, some long-lived nuclide in the decay series of the Rf isotope can be registered [6]. For  $^{261}\text{Rf}$ , the decay chain is:



Then the time available for making a sample for measurements is now a day or even more, in accordance with the half-lives of  $^{253}\text{Fm}$  and  $^{253}\text{Es}$ . In this case, nothing prevents the possibility of thorough radiochemical purification and of achieving a high chemical yield of the nuclide to be measured and samples can be prepared for counting in nearly  $4\pi$  geometry.

The first two stages (1 and 2) are realised as a truly continuous process [5], which makes it possible to exclude losses due to decay during accumulation of the activity. Obviously, the equipment required is simpler and can be automated more easily. The atoms recoiling from the target are adsorbed on the surface of particulates of an aerosol and with it through a capillary they are transferred to the chemical apparatus at a distance of several meters within a few seconds. There the particulates are absorbed directly in the solution, which serves for the separation of elements.

In order to examine some possibilities of rapid continuous isolation of  $^{261}\text{Rf}$  from the products of bombardment of the  $^{248}\text{Cm}$  target on a set of ion-exchange columns, the model experiments with Hf and Zr radionuclides as the homologues of Rf were performed by Szegłowski *et al.* [5]. It has been shown for the first time ever that in HF solution Rf forms stable anionic complexes which are sorbed on an anion-exchange resin, *i.e.* the element behaves similar to Zr and Hf, members of the 4<sup>th</sup> group of the transition metals. The distribution coefficients and decontamination factors of the elements under study, which are functions of the composition of the solution, the nature of the solvent, the concentration of complexing agent and other factors were determined. The method opened up the possibility of obtaining quantitative data on the properties of Rf in solution.

Using this method, continuous isolation of rutherfordium from the nuclear reaction products was elaborated. Experiments were performed at the JINR heavy ion cyclotron (U400). Target  $^{248}\text{Cm}$  was bombarded with  $^{18}\text{O}$  with an energy of 97–102 MeV [5] at a beam intensity of up to  $4 \times 10^{12}$  pps during 24 hours. The nuclear reaction products recoiled from the target were transported by the aerosol jet (NaCl in argon) through a teflon tube to the unit consists of three ion-exchange columns connected in series. The transplutonium elements (TPE) and Ln were isolated by sorption on the first cation exchange column (5) with the subsequent concentration of Rf on an anion exchange one (6). Then the Rf descendants were sorbed on another cation exchange column (7). The identification of  $^{261}\text{Rf}$  has been performed by registering its descendants:  $^{253}\text{Fm}$  and  $^{253}\text{Es}$  after their disruption, by a

small volume of 6M  $\text{HNO}_3$  from the last cation exchange column, Dowex-50x8, see 7 in Fig. 1. A schematic diagram of the experimental equipment is presented in Fig. 1.

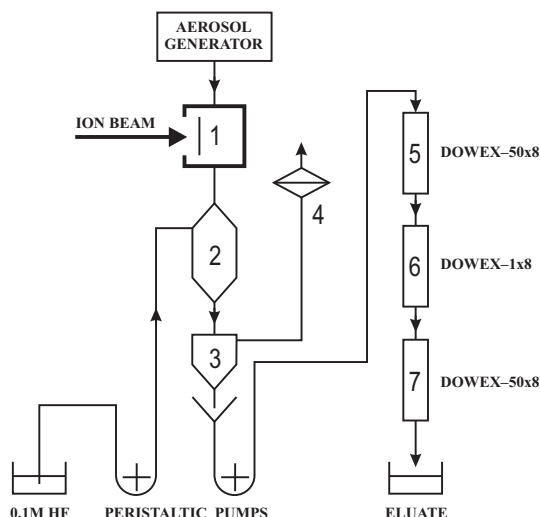


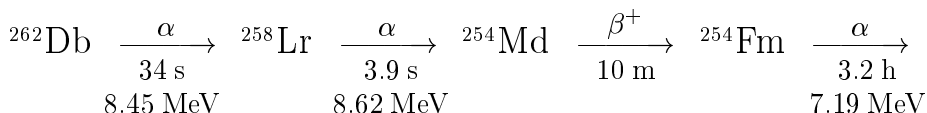
Fig. 1. Scheme of the separation apparatus: 1 — target chamber, 2 — absorber, 3 — degasser, 4 — filter, 5,6,7 — ion exchange columns.

Extraction of Rf, Zr, Hf, Th, Pu (IV) and Nb (V) by tributylphosphate (TPB) in benzene from aqueous solution of HCl were studied by Czerwiński and co-authors [7]. The results obtained gave the evidence for a deviation of Rf chemistry from that of other elements of the 4<sup>th</sup> group. These unexpected results for extraction from chloride solutions indicate that at times Rf behaves more like tetravalent Pu than Zr or Hf. In addition, extraction of Rf increases with the growth of a hydrogen ion concentration, while Zr and Hf exhibit no such effect. These results indicate that the Rf forms neutral salt complexes more readily than Zr and Hf. The extracted complex for Rf appears to be different from those of Zr and Hf. These differences, along with the differences between Db and its the 5<sup>th</sup> group homologues, indicate an unexpected trend in the behavior of transactinide elements and show that the properties exhibited by lighter homologues can not be simply extrapolated to predict the chemical properties for transactinide elements.

### 3. Dubnium — Element 105

Studies of the halide complexes of dubnium, Db, in aqueous solutions were performed on 34 s  $^{262}\text{Db}$  produced in the  $^{249}\text{Bk} (^{18}\text{O}, 5n)$  reaction by Gregorich *et al.* [8].  $^{262}\text{Db}$  was uniquely identified by measuring energy and

time distribution of the alfa particles and fission fragments emitted in its decay. Time-correlation pairs of  $\alpha$  particles from the decay of  $^{262}\text{Db}$  and its daughter,  $4.3\text{ s }^{258}\text{Lr}$ , were also measured. Both alpha-particle energies and decay times can then be used as identification criteria. The decay chain for  $^{262}\text{Db}$  nuclide is presented below:

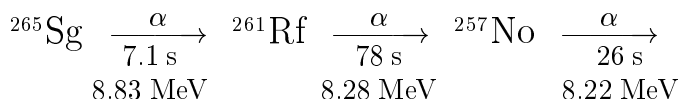


The studies of chemistry of elements 105 (Db) were performed by Zvara *et al.* [9,10] and by Gregorich *et al.* [11]. Zvara *et al.* [9] used gas thermochromatography on the relatively volatile bromide and chloride compounds. They used the  $^{243}\text{Am}$  ( $^{22}\text{Ne}$ ,  $4\text{--}5n$ ) reaction to produce  $2\text{ s }^{261\text{--}260}\text{Db}$  which they detected by spontaneous fission (SF) decay, using track detectors along the thermochromatographic column. The observed position of fission tracks was similar to that of hafnium and was interpreted by Zvara *et al.* as being the expected position of eka-tantalum. Db was found to adhere to glass surfaces upon fuming with concentrated nitric acid, a property very characteristic of the 5<sup>th</sup> group of elements, niobium and tantalum, and the pseudo-member of that group, protactinium. Tantalum extracts under a broader range of conditions than does niobium. Assuming the elements of group 5 to continue this trend, dubnium should also form extractable species and be quantitatively extracted under the same conditions as tantalum is extracted. However, it was found that dubnium did not form extractable anionic fluoride complexes [11]. This non-tantalum-like-behaviour was discussed in Ref. [11]. The tendency to hydrolize or to form high coordination number fluoride complexes in dubnium may be much stronger than in tantalum, leading to non-extractable species. It should be noted that the extraction conditions [11] were such that niobium was not extracted into the MIBK (methyl isobutyl ketone) phase.

#### 4. Seaborgium — Element 106

Sg (106) was discovered in 1974 in the reaction  $^{249}\text{Cf}$  ( $^{18}\text{O}$ ,  $4n$ ) by Ghiorso *et al.* [12], who found the half-life,  $T_{1/2}$ , to be  $0.9 \pm 0.2\text{ s}$  and the production cross-section,  $\sigma$ , to be  $0.3\text{ nb}$  for the alpha decay branch at a state bombarding energy of  $94\text{ MeV}$ . Later on, Druin *et al.* [13] reported  $0.63\text{ s}$  and  $0.6\text{ nb}$ , respectively, for the spontaneous fission (SF) branch; their bombarding energy was  $95 \pm 1.5\text{ MeV}$ . Some lighter isotopes of Sg were synthesized by the bombardments of Pb with Cr projectiles:  $^{258}\text{Sg}$  with  $T_{1/2} = 2.9\text{ ms}$  (mostly SF);  $^{259}\text{Sg}$  with  $T_{1/2} = 0.5\text{ s}$  (mostly  $\alpha$ -active);  $^{260}\text{Sg}$  with  $T_{1/2} = 4\text{ ms}$

( $\alpha/\text{SF} = 50 : 50$ ); and  $^{261}\text{Sg}$  with  $T_{1/2} = 0.25$  s (mostly  $\alpha$ -active), see Refs [6] and [12–17]. Isotopes  $^{262}\text{Sg}$  and  $^{264}\text{Sg}$  are not known yet. Oganiesian *et al.* reported in Ref. [17] that,  $\alpha$ -active  $^{265}\text{Sg}$  and  $^{266}\text{Sg}$  with  $T_{1/2}$  of about 3 s and 15 s, respectively, have been discovered by bombarding, again an actinoid target,  $^{248}\text{Cm}$ , with  $^{22}\text{Ne}$ . Unfortunately, their  $\sigma$  values are several times smaller than that for  $^{263}\text{Sg}$ . The decay chain for nuclide  $^{265}\text{Sg}$  is:

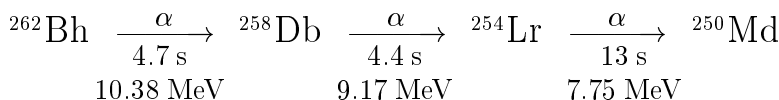


In the atoms of the heaviest elements one finds extremely dense clouds of electrons moving with relativistic velocities. This is why the studies of the chemical properties of the elements are subject of interest. This problem is reported in details in Ref. [18].

For the first time, chemical separation of element 106, Sg, was performed in aqueous solutions by Shädel *et al.* [19]. The isotopes  $^{265}\text{Sg}$  and  $^{266}\text{Sg}$  were produced in the  $^{248}\text{Cm} + ^{22}\text{Ne}$  reaction at a beam energy of 121 MeV. The reaction products were continuously transported by means of a KCl aerosol jet to the computer-controlled liquid chromatography system [19]. Sg was detected by measuring correlated  $\alpha$ -decays of the daughter isotopes  $^{261}\text{Rf}$  78 s and  $^{257}\text{No}$  26 s. For the isotope  $^{266}\text{Sg}$ , there was evidence for spontaneous fission branch. The chemical results show that the most stable oxidation state of Sg in aqueous solution is +6, and that like its homologues Mo and W, Sg forms neutral or anionic oxo or oxohalide compounds under suitable conditions [19]. In these experiments, Sg exhibited properties very characteristic of group 6, and did not show U like properties.

## 5. Bohrium — Element 107 and Hassium — Element 108

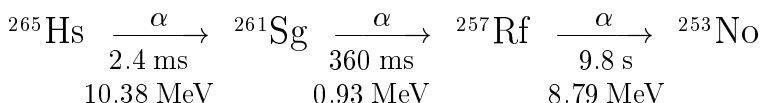
Element 107, Bh, was synthesized by fusion of  $^{209}\text{Bi}$  and  $^{54}\text{Cr}$  performed by Münzenberg *et al.* [20]. The reaction is:  $^{209}\text{Bi} (^{54}\text{Cr}, n)^{262}\text{Bh}$ . A previously unknown  $\alpha$ -emitter of 4.7 ms half-life and 10.38 MeV  $\alpha$ -energy could be connected to the decay chain shown below:



Another isotope of element 107 (Bh) with mass number 261 was produced under the same conditions as previous one. The reaction was:  $^{209}\text{Bi} (^{54}\text{Cr}, 2n)^{261}\text{Bh}$ . The nuclide has half-life time of 0.9 ms [21] and a complex  $\alpha$ -spectrum. Oganiesian *et al.* [22] claim to have detected this isotope as early as 1976. A search for neutron-rich isotopes of element 107 such as  $^{266}\text{Bh}$ ,

which might have half-life of the order of minutes [22], was carried out during the bombardment of  $^{254}\text{Es}$  with  $^{16}\text{O}$ , without success [23]. Discovering such isotopes, which were theoretically predicted, would enable using aqueous solution chemistry for investigation of their chemical properties.

Münzenberg *et. al.* [24,25] detected the isotope of element 108 (Hs) with mass number of 265. The reaction for synthesis was:  $^{208}\text{Pb} (^{58}\text{Fe}, n)^{265}\text{Hs}$ . Alpha-decay chain of the isotope is presented below:

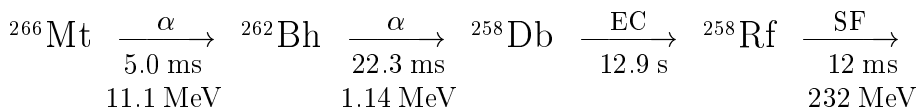


The same group observed a second isotope,  $^{264}\text{Hs}$ , with half-life 0.08 ms which was produced by fusion of  $^{207}\text{Pb}$  with  $^{58}\text{Fe}$  [25,26].

The possibilities of studying the chemical properties of Bh and Hs in aqueous solutions according to model experiments with their homologues Re and Os were investigated. With this goal, the behaviour of Re and Os on cation-and-anion-exchangers in the sulfuric acid media was studied in details [27]. An developed earlier [5] and effective ion-exchange method for continuous isolation of ultra trace amounts of short-lived transactinide elements in the process of their synthesis with simultaneous chemical studies of these elements in solution containing various complexing agents was used for these studies.

## 6. Meitnerium — Element 109 and Elements 110, 111, 112

In 1982 Münzenberg *et al.* [28] succeeded in detecting one single decay chain, when bombarding  $^{209}\text{Bi}$  with  $^{58}\text{Fe}$ . It connected this  $\alpha$ -emitter with a decay chain already known from the synthesis of bohrium. The synthesis was performed via reaction:  $^{209}\text{Bi} (^{58}\text{Fe}, n)^{266}\text{Mt}$ . This decay chain quoted from Ref. [28] is:

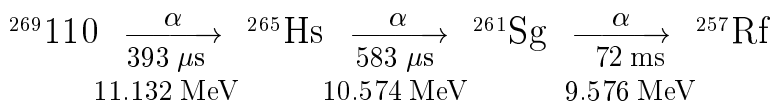


In 1988 the same group found a second atom of meitnerium which was correlated with the decay chain reported in Ref. [29]. This discovery was corroborated by Oganessian *et al.* [6], who radiochemically detected a long-lived end product of  $\alpha$ -decay chain,  $^{246}\text{Cf}$ .

The element 110 was synthesized for the first time at SHIP, GSI, Darmstadt. Two isotopes with mass number  $A = 269$  and  $A = 271$  were produced in reactions:  $^{208}\text{Pb}(^{62}\text{Ni}, n)^{269}110$  and  $^{208}\text{Pb}(^{64}\text{Ni}, n)^{271}110$ . Both isotopes

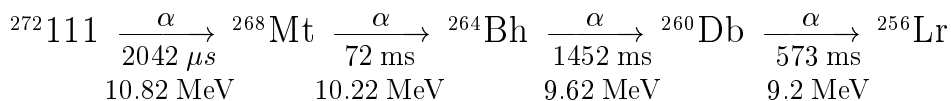


decay by  $\alpha$ -emission with half-lives given in Ref. [30]. The decay chain assigned to  $^{269}\text{110}$  is presented below [30]:

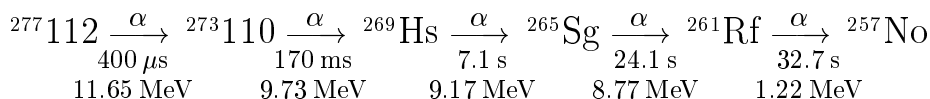


Recently, the new isotope  $^{270}\text{110}$  has been obtained by S.Hoffmann *et al.* [31], using reaction:  $^{207}\text{Pb}(^{64}\text{Ni},n)^{270}\text{110}$ .

In December 1994, Hofmann *et al.* [32] produced in GSI, Darmstadt three nuclei of the isotope  $^{272}\text{111}$ . These three nuclei were observed in bombardment of  $^{209}\text{Bi}$  targets with  $^{64}\text{Ni}$  projectiles of 318 MeV and 320 MeV. The cross sections were 1.7 pb and 3.5 pb, respectively. The nuclei decayed by  $\alpha$ -emission into the new and so far heaviest isotopes of the elements 109 and 107 with mass number 268 and 264, respectively. The  $\alpha$ -decay chains were followed down towards the known nuclei  $^{260}\text{Db}$  and  $^{256}\text{Lr}$ , as follows [32]:



The next superheavy element 112 was produced and identified in GSI, Darmstadt by Hofmann *et al.* [33, 34]. The decay chains of the isotope  $^{277}\text{112}$  were observed while bombarding  $^{208}\text{Pb}$  targets with  $^{70}\text{Zn}$  projectiles of 344 MeV kinetic energy. The isotope decays by emission of alpha particles. Two  $\alpha$ -decay chains were observed. One of them quoted from Ref. [33] is presented below:



Chemical identification of newly discovered isotopes is very important, because for the first time decay chains of new nuclei do not end by isotopes discovered earlier. Moreover, chemical identification of the proton number of the new nuclei is very important, because all the members of the above decay chains were unknown. Since Hg is expected to be a homologue of element 112, the short-lived mercury isotopes were a subject of our interest [35]. These isotopes emit  $\gamma$ -rays which allow convenient monitoring of chemical isolation of Hg from the products of the nuclear reaction. We examined the sorption of short-lived isotopes of Hg on metallic silver from diluted sulphuric acid solutions. The outline of experimental equipment is presented in Fig. 1. The first column, filled with hydrated antimony pentoxide (HAP),

retained radionuclides which interfere with  $\gamma$ -measurements of mercury. It is noteworthy to point out, that on this column iridium, which might be a homologue of element 109 (Mt), was also retained. In the second column, filled with granulated glass coated by metallic silver, the short-lived isotopes of mercury were selectively sorbed. In the experiment the second column was replaced by another one filled with Dowex-1, and isolation with identification by gamma spectrometry of above mentioned mercury isotopes (among them  $^{181}\text{Hg}$  with half-life of 3.6 s !) were carried out. The obtained results, see Ref. [35], have shown that the applied method makes possible rapid isolation of mercury and its daughter isotopes. This method is expected to be useful for studies of homologues of the other newly discovered superheavy elements, such as: 109, 110, 111. The  $\gamma$ -spectrum of the Ag-column measured in the on-line regime, while the 0.05M sulphuric acid solution containing mercury isotopes and their descendants was passing through the column, is shown in Fig. 2 [35]. From Fig. 2 one can see that all lines in spectrum belong only to the mercury isotopes and its descendants.

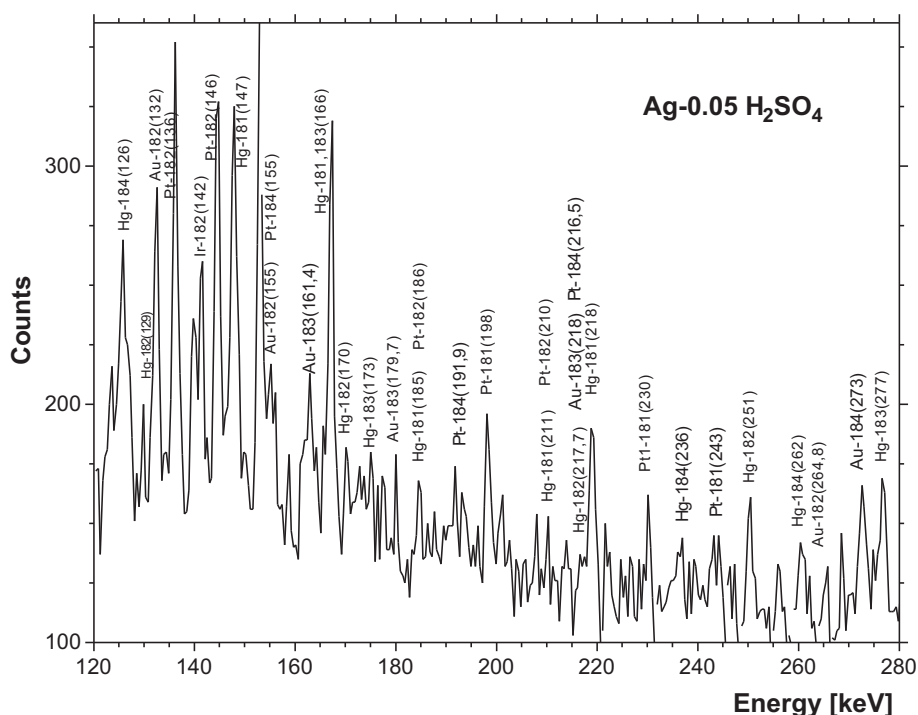
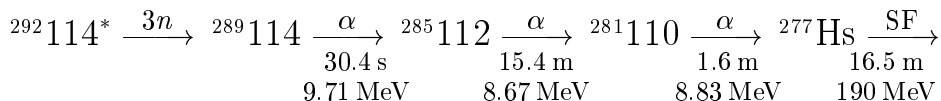


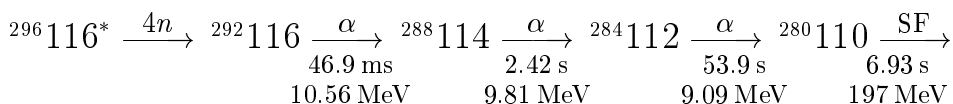
Fig. 2. The  $\gamma$ -spectrum of the Ag-column measured in the on-line regime.

## 7. Element — 114 and Element 116

At the end of 1998, the experiment, which consisted in bombardment by intense beams of  $^{48}\text{Ca}$  of the  $^{244}\text{Pu}$  target and was aimed at searching a new isotope of element 114, was performed by Oganessian *et al.* at JINR in Dubna [36]. A decay chain, consisting of three sequential  $\alpha$ -decays and spontaneous fission was observed in this experiment [37]. The decay chain quoted from Ref. [37] is shown below:



The first observation of decay event of nuclide  $^{292}_{116}$  in the experiment on the synthesis of  $Z = 116$  nuclei in the reaction  $^{248}\text{Cm} + ^{48}\text{Ca}$  was reported by Oganessian *et al.* [38]. The observed decay sequence quoted from Ref. [38] is:



Since nuclei of super-heavy elements are  $\alpha$ -emitters and they emit  $\alpha$ -particles with similar energy, and the cross-sections of their production are very low, an exact attributing of detected particles to the proper nuclide might have encountered difficulties. That is why, using the chemical methods for high efficient separation of one element from another is so important.

So far, there were no reports on experiments using the chemical method for studies elements 114 and 116. There are two basic requirements for use of chemical methods as convenient tools of investigations: a proper half-life time and an enough high yield of a studied isotope. Solution chemistry requires isotopes to have half-life times of the order of several seconds, whereas the gas chemistry makes it possible to work with isotopes having much shorter ones. It is noteworthy, that theory predicts for super-heavy elements so-called 'stability island'. Their half-life times might be as long as several minutes and more. This fact would allow to use chemical methods for studies of chemical properties of these isotopes and, what is important, chemists might corroborate their assumption as to chemical behaviour similarity between them and their homologues.

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