SYNTHESIS AND SEARCH FOR HEAVY TRANSURANIUM ELEMENTS

G. N. Flerov

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An example of the important role which is played by D. I. Mendeleev's Periodic Law in the investigation of the structure of a substance is the synthesis and identification of a number of transuranium elements (from neptunium to mendelevium) which was completed in the USA in 1955. This period in the history of discovery of the transuranium elements is associated with the development of nuclear technology. Elements from neptunium to fermium were synthesized by multiple-capture of neutrons in intense neutron fluxes from reactors and nuclear explosions. In such a method of synthesis, the reaction products are found mixed with one another and with the starting substance, and therefore chemical separation of the elements which are of interest to the experimentalists is unavoidable. The success of these investigations in many respects was determined by the similarity in chemical properties which exists between the actinide and lanthanide families. This similarity lies at the basis of the so-called actinide hypothesis, which permitted Dr. Glenn Seaborg and his co-workers to analyze the complex mixture of elements and isotopes synthesized in reactors and explosions. It is no wonder, therefore, that the pioneers in the discovery of the whole series of transuranium elements proved to be specialists in the field of radiation chemistry (Glenn Seaborg, S. Thompson, etc.).

In this period, despite the novelty and complexity of the many aspects of the problem itself and the experimental procedures being used, which frequently were created by the researchers themselves for solving the problems which arose, and despite the enormous quantity of data obtained, there were almost no serious errors. Individual inaccuracies were rapidly detected and eliminated.

However, in the attempts to advance further into the region of heavy nuclei ($Z > 101$), certain objective difficulties were revealed, all more or less concerning the synthesis, identification, and study of the properties of the new elements. The principle ones were the sharp decrease of the lifetime of the elements being synthesized and also the reduction of the probability of their formation in the reactions with increase of atomic number. Figure 1 shows a general representation of the dependence of the half-life on the atomic number of the element in the region from californium to kurchatovium.

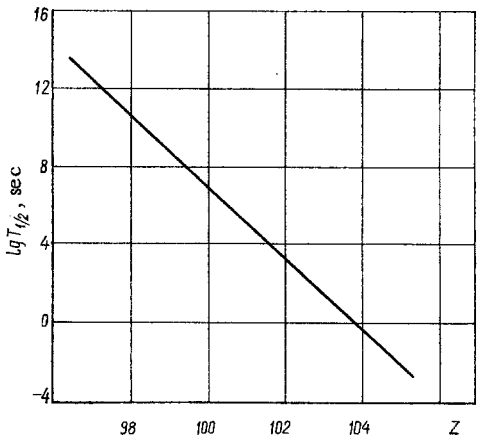

Fig. 1. Reduction of lifetime of elements with increase of Z.

The classical technique for chemical identification based on processes which take place in the liquid phase requires a finite time for separation of the new elements (about 1 min from the instant of synthesis). After this time the relatively short-lived isotopes have decayed almost completely. Consequently, mendelevium proved to be the last element identified by classical methods of chemical separation in the liquid phase.* It was necessary to find new methods for synthesizing the heavy transuranium elements.

* After carrying out in Dubna a systematic study of the properties of 6 isotopes of element 102 [1-7], it was ascertained that the isotope 102^{255} has a half-life of 3 min and can be studied by means of ion-exchange.

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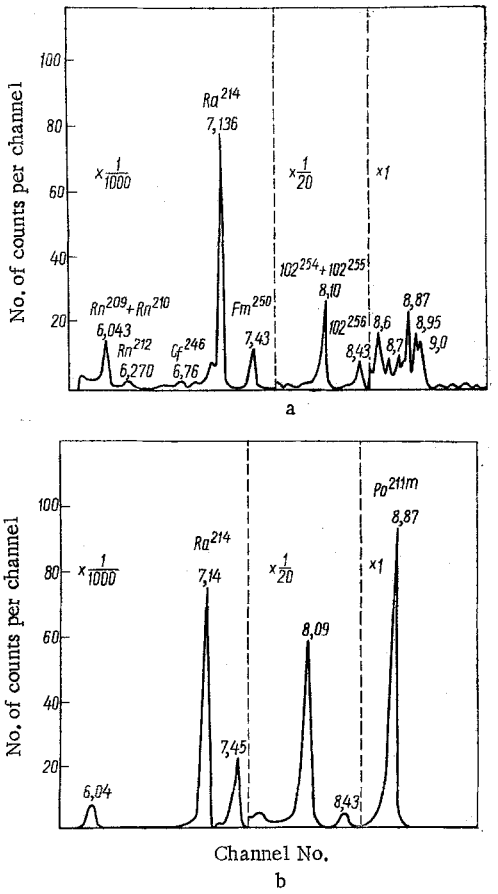

Fig. 2. Spectra of α-particles emitted by the products of the reactions $Cf^{249} + C^{12}$ [28] and $Pb + C^{12}$ [30] (*a* and b respectively).

The second period of research in the region of the transuranium elements is associated with the use for the synthesis of new elements of accelerated heavy ions instead of neutrons. The idea arose that the new nuclei might be formed by fusion of carbon, oxygen, and neon atoms, etc., accelerated to the required energy, with the nuclei of heavy elements (uranium, plutonium, americium, etc.).

In 1954, by the initiative of Academician I. V. Kurchatov in Moscow, the development of a project was commenced for the acceleration of heavy ions for the synthesis of new isotopes and elements and also for studying nuclear reactions. Simultaneously with the planning, work was started on the production and acceleration of multicharged ions in the standard U-150 cyclotron and the first experiments with accelerated ions of carbon, nitrogen, and oxygen. The design of the new accelerator was completed in Dubna by 1960. Reactions with heavy ions, in addition to the possibility of increasing abruptly the charge of the nucleus by several units, permitted significant shortening of the time required for separation and identification of the new nuclei.

The use of the recoil originating by fusion of the heavy ion with the target nucleus, in conjunction with the technique of rapid measurement under low background conditions of α-decay and spontaneous fission, comprise the basis of the physical methods of identification and permit, in principle, reduction of this time to 10^{-8} sec.

The time required for chemical identification of an element was also reduced significantly, due to conversion to chemical separation in the gaseous phase. The recoil nuclei ejected from the target immediately entered an active gaseous medium. As chemical reactions and transportation in work with gases can be accomplished considerably more rapidly than in a liquid medium, by using a special quite complex technique [8-10] nuclear reaction products can be separated and recorded successfully which have lifetimes of the order of tenths of a second.

The recoil method also permits use of mass separators operating in the accelerator beams for identification of the new nuclei, which opens up wide possibilities for studying short-lived nuclei which are remote from regions of stability. In Dubna, N. I. Tarantin et al. operated a mass-separator of a type which permitted the time of separation to be reduced to 10^{-3} sec [11].

Another difficulty – the reduction of reaction yield with increase of atomic number of the nuclei products – is considerably more complicated to overcome. The probability of forming heavy transuranium elements in reactions which proceed via the stage of formation of a compound nucleus with subsequent boil-off of several neutrons, is reduced roughly speaking by a factor of 10 or more with increase of Z by unity. The difficulties are aggravated all the more in that the yield from multiple reactions leading to background products, in this case is practically unreduced. This imposes extremely high demands on the selectivity of the methods of identification (both chemical and physical).

Elements 102, 103, and 104 were synthesized by the acceleration of heavy ions; the first atoms of element 105 were also produced. However, the objective difficulties mentioned above and possibly also certain subjective circumstances led to the fact that the history of the discovery and study of the properties of these elements turned out to be a history of errors and controversies.

The first attempts to synthesize and identify element 102 were made in 1955. In concurrent work by American, British, and Swedish researchers [12], the synthesis was reported of 20 atoms of element 102 with mass number 253 or 251, undergoing α-decay with $T_{1/2} = 10$ min. The authors somewhat hastily conferred the name "nobelium" on this "new element"; however this work was later admitted to be erroneous

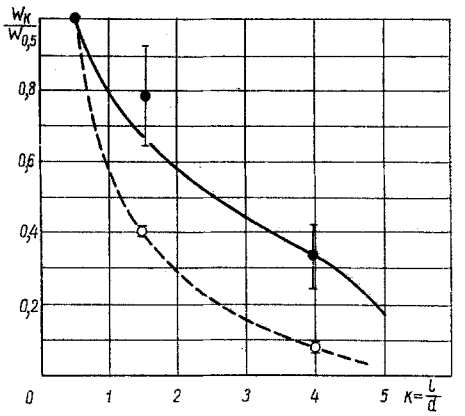

Fig. 3

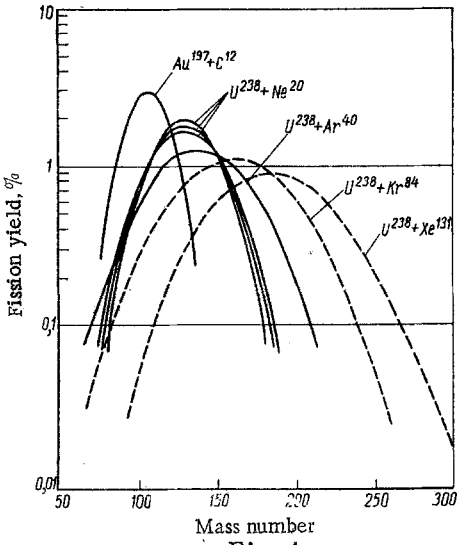

Fig. 4

Fig. 3. Angular distribution of kurchatovium nuclei ($T_{1/2}$ = 0.1 sec), synthesized in the reaction $Pu^{242} + Ne^{22}$ (black circles). For comparison, the angular distribution is given of the spontaneously fissile isomer of americium in the reaction $Pu^{242} + Ne^{22} \rightarrow Am^{242mf}$ (k is the ratio of the collimator depth l to the aperture diameter d).

Fig. 4. Distribution according to fission fragment mass in reactions with heavy ions: ——) experimental results; – – – –) numerical estimates.

[13]. Shortly after, several more papers appeared on element 102 [14-17] but all these were not much better than the first.

Element 102 was identified reliably for the first time in Dubna (isotope 102^{256}) [1]. In later papers [2-7], this conclusion was confirmed and, moreover, a systematic investigation of the properties of six isotopes of element 102 was carried out. As a result, sources of error were cleared up which had been committed in the first unsuccessful experiments [12-17].

The history of the discovery of element 103 was also found to be quite complex. In 1961 in Berkeley (USA), A. Ghiorso et al. announced the discovery of a new element with atomic number 103 (isotope with mass number 257) and they called it "lawrencium" [18, 19]. However, an analysis of the possible errors and backgrounds was not undertaken in this work. Experiments carried out in Dubna [20-24] later showed that the work was erroneous.

In 1965, a group of experimenters in Dubna established unambiguously that in the reaction $Am^{243} + O^{18}$ the isotope 103^{256} is formed [20]. Up to the present time, three isotopes of element 103 with mass numbers 255, 256, and 257 [20-24] have been discovered and studied. Careful searches for an isotope with the characteristics of the American element 103 – lawrencium – did not give positive results [21].

After publication of the experimental data on element 103 obtained in Dubna, A. Ghiorso once again analyzed his results and altered their original interpretation, but without giving any new experimental verification. It was reported [19] that in their first paper [18] it was obviously not the isotope 103^{257} that had been detected but one of the heavier isotopes: 103^{258} or 103^{259}. Detailed analysis shows [23, 24], that such an interpretation is contradictory to the experimental data given by A. Ghiorso et al. in the 1961 paper [18] and should be acknowledged as unacceptable.

The history of the filling of the "empty spaces" in Mendeleev's Table confirms that in all cases the authors of the papers, the results of which were open to doubt, carried out additional experiments and showed either their validity or rejected them as erroneous statements. In the given case, the group in Berkeley, unfortunately, did not proceed along this route.

A group of chemists in Dubna studied the behavior of the chlorides of element 103 using the isotope 103^{256} as the example and they showed that it belonged to the actinide family [25]. The results of the Dubna

experiments on isotope 103^{256} were confirmed completely in Berkeley [26]. A striking example of an investigation carried out at the limits of sensitivity and rapidity is the discovery of element 104 and the study of its physical and chemical properties.

In 1964, in the Nuclear Reactions Laboratory of the Joint Institute for Nuclear Research, by the irradiation of Pu^{242} with accelerated Ne^{22} ions, nuclei were synthesized which undergo spontaneous fission with a half-life equal to 0.2 to 0.4 sec approximately. Spontaneous fission of the new emitter was detected in the decay background of spontaneously fissile isomers and other activities which are formed in significant quantities in the reaction Pu + Ne and therefore, in the first paper the half-life could not be measured accurately. The difficulties which the experimenters encountered are illustrated by the following figures: with a maximum flux intensity of accelerated neon ions (tens of microamperes of ion current) the observed yield amounted approximately to one atom per hour. However, despite these difficulties analysis of the shape of the excitation function for the detected emitter and of the results of control experiments permitted the authors of the paper to draw the conclusion, that the isotope which had been synthesized is one of the isotopes of element 104 and, in all probability, its mass number is 260 [27].

In order to establish the fact of synthesis of a new element, chemical identification is most important. Consequently, in Dubna further experiments were conducted on the synthesis of element 104 using high-speed separation in the gaseous phase [8, 9]. As a result of an independent chemical method the fact was established, that by the irradiation of Pu^{242} with Ne^{22} ions, isotopes of element 104 are formed which undergo spontaneous fission with a half-life of a few tenths of a second. It should be noted however that in the chemical experiments the isotopes could not be separated. This implies that, together with the short-lived isotopes of kurchatovium in the experiments, it is possible that longer lived isotopes were also present, the contribution from which gave the observed decay curve.

In 1967 the chemical identification experiments were repeated, using improved techniques [10]. The results confirmed the conclusion drawn in the first paper concerning the membership of element 104 to Group 4 of the periodic system of elements.

The authors of the paper in which element 104 was identified by means of physical and chemical methods, suggested that it should be called "kurchatovium" (Ku) in honor of the distinguished Soviet physicist Igor Vasil'evich Kurchatov.

However, the history of the discovery of element 104 was not concluded with this. Five years after the first Dubna experiments, A. Ghiorso and his colleagues reported the synthesis of three α-radioactive isotopes of element 104 – kurchatovium — with mass numbers 257, 259, and 261 and half-lives of 4.5, 3, and 60 sec, respectively [28, 29]. In these experiments, a target of Cf^{249} was irradiated with accelerated C^{12} and C^{13} ions. In order to identify and determine the characteristics of the isotopes, the α-spectra of the reaction products were analyzed. The conclusion which can be drawn about the properties of the isotopes of element 104, based on the results of these experiments, mainly confirm the results of the first work on element 104 carried out in Dubna [27]. However, A. Ghiorso et al. strove to find and, in their opinion, found considerable contradictions and attempted to cast doubt on the results obtained in Dubna.

It should be noted that the primary α-spectra in experiments on the synthesis of heavy transuranium elements almost always occur distorted to some or other degree by the α-activities which are formed by the irradiation of unavoidable impurities of lead in the target and with characteristics close to those expected for element 104 [16, 30]. Nevertheless, A. Ghiorso et al. did not in general discuss this problem, although in spectra derived by them there are clearly lines which have their origin in the lead impurities (Fig. 2).

Analysis of the spectra and of the half-lives for the individual lines reveal in the paper by Ghiorso et al. [28] a number of discrepancies and, obviously, simple errors and casts doubt on the accuracy of the determination of the characteristics of the α-active isotopes of element 104. It is quite obvious that the authors of this paper [28] should repeat the experiments under "purer" conditions. For the present, it remains to conclude that in the attempts to synthesize α-active isotopes of element 104, the group in Berkeley were unable to avoid the traditional errors.

Recently, in Dubna, the procedure used in the first experiments on the physical identification of element 104 was improved and used for the synthesis and identification of element 105. The neutron background in the vicinity of the detectors was reduced considerably as a result of transition to experiments in the extracted beam of the cyclotron (the experiments on element 104 in 1964 were carried out in the internal beam).

Moreover, a considerable separation of recoil nuclei ejected from the target was achieved, due to singularities in the angular distributions of the products of the various reactions [31, 32]. The characteristics of nuclear reactions with heavy ions are a very sharp maximum in the angular distribution of the recoil nuclei for reactions which proceed via a compound nucleus stage (element 105 must be formed in a reaction of this type). In other reactions, the angular spread of the final nuclei is considerably greater. By the introduction of suitable collimation, nuclei of element 105 can be separated from a considerable portion of the other reaction products.

This procedure was tried in a new series of experiments on the synthesis of the spontaneously fissile isotope of element 104, detected earlier in the $Pu^{242} + Ne^{22}$ reaction. By changing the degree of collimation the experimenters studied the total angular distribution of the nuclei of this isotope and showed that it actually corresponds to a reaction which proceeds via the stage of compound nucleus formation (Fig. 3). Based on careful analysis of the results obtained, the conclusion was drawn that the emitter being studied is formed in a reaction of total fusion of Pu^{242} and Ne^{22} nuclei with subsequent boil-off of several neutrons and, consequently, it belongs to kurchatovium [32]. Naturally, the collimation method did not make it possible to define more precisely the mass number of the isotope, but it permitted significantly to reduce the background of extraneous fission products and to refine the half-life of kurchatovium, which was found to be 0.1 ± 0.05 sec.

All the above-mentioned gives a basis for finally concluding that the synthesis of element 104 and the observation of its decay was first established in 1964 in Dubna [27] and confirmed by an independent gas-chromatography method in 1966 and 1967 [8-10], and also by experiments for studying the angular distributions of the reaction products in 1969.

Thus, the Nuclear Reactions Laboratory of the Joint Institute for Nuclear Research in Dubna, in which teams of scientists from the Socialist countries are carrying out investigations on the transuranium elements, counts among its assets the discovery of the three heavy elements 102, 103, and 104. Preliminary data on element 105, obtained in 1968, are being checked at present by the collimation method.

Now a third period in the history of the transuranium elements is starting – the synthesis and search for superheavy elements with atomic numbers $Z = 110$-126. In this case, a peculiar psychological barrier must be overcome by the physicists, which is complicated by the effect of a catastrophic reduction of the half-lives of the new elements to be synthesized and in their formation reaction cross sections. Moreover, until recently we were unable to accelerate ions sufficiently heavy to enable us to pass on to a study of the new region.* However, there is a quite real hope of success in synthesizing and searching for superheavy elements. The question concerns the increased stability of nuclei with closed nucleon shells (a nucleus with "magic" numbers of protons or neutrons).

In the transuranium region there is already one similar example: the so-called subshell with the number of neutrons in the nucleus $N = 152$. Subshells originate in deformed nuclei as a result of the evacuation of energy levels in the vicinity of the Fermi boundary and they provide nuclei with additional stability. The subshell $N = 152$ was discovered by systematic study of the properties of elements 96-102. It is precisely because of this effect that the lifetime of certain isotopes of element 102 has proved to be of the order of a minute.

Recently, the question of the existence of further magic numbers of protons and neutrons is receiving greater attention, especially in connection with the new region of relative stability in the vicinity of $Z = 110$-126 and $N = 184$, predicted by many authors [34-36]. On the basis of numerous calculations the conclusion can be drawn that the existence of superheavy nuclei is possible and these are sufficiently stable for experimental study.

In 1967 P. Fowler, studying the elementary composition of cosmic rays, discovered tracks in the photoemulsions which could have been left by nuclei with $Z = 103$-110 [37]. Further investigations will be necessary for a definite conclusion about the magnitude of the charge of these nuclei, on the assumption that they are superheavy nuclei, but already the posing of the problem about the existence in nature of nuclei heavier than uranium has radically changed the situation in the region of investigation of the far-transuranium elements. Besides synthesis, a search for superheavy elements in natural materials has been undertaken.

* J. Wheeler, even in 1955, had the boldness to predict the existence of nuclear-stable systems of nucleons with mass up to 600, based on the overall representation of the properties of nuclei [33].

The results of the investigations carried out in Dubna in this direction give the basis for assuming that in the earth's core there exist long-lived spontaneously fissile emitters. It was first established that in dielectric media placed in contact with lead, and also in certain samples of lead glasses, fission fragment tracks were observed and which cannot be attributed to the spontaneous fission of lead [38]. The authors of this paper have assumed that the observed effect is caused by the spontaneous fission of a heavy chemical element, possibly similar to lead or another element accompanying lead. If the half-life of the emitter is greater than 10^8 years, then in order to cause the observed effect its content in the samples must amount to at least 10^{-12} to 10^{-13} g/g. According to modern representations of the structure of Mendeleev's Table, in the region of the far-transuranides the atomic number of eka-lead is 114.

These results have recently been verified in Dubna by a new independent method [39]. Fragments from spontaneous fission were recorded with large proportional counters having an extremely low natural background (one count per 30 days). This technique, in contrast to the dielectric detector procedures used previously, permit a search for spontaneous fission to be undertaken in almost any minerals and compounds. For this purpose, the samples to be investigated were ground to powder and without preliminary chemical processing they were placed in the counter. The results of the measurements carried out with two different samples of lead glasses coincide with the data obtained for these glasses by the method of counting the tracks left by the fragments from spontaneous fission in the glass [38]. In these experiments, particular attention was paid to the problem of background. Sources of background in the glass detectors, as well as in the counters, may be due to uranium impurities in the lead or to the fission of lead by the action of cosmic rays. Activation analysis for the uranium content and a special experiment with lead obtained by electromagnetic separation (isotope 208) showed that the overall background contribution did not exceed 10-15% of the number of events observed.

The investigation of a large number of samples containing bismuth, mercury, and tungsten showed that in these cases the observed effect is tens and hundreds of times less and does not fall outside the background limits [40].

Naturally, in such a situation, an attempt must be made to find a mineral in the earth's core which is enriched in the emitter being studied. We have already obtained preliminary results in this direction. In certain experiments with samples of lead minerals, a positive effect has been observed which is comparable with the results on the lead glasses.

P. Price et al. (USA) attempted to detect the tracks of fission fragments from superheavy analogs of lead and gold in cardistonite and auriferous sand which were several hundreds of millions of years old. However, the results obtained were negative [41]. This circumstance was treated by the authors as disproof of the results of the Dubna experiments in the search for superheavy elements. In our opinion this conclusion is premature, as it cannot be excluded that in the samples of P. Price, processes might have occurred which would lead to the disappearance of the tracks from spontaneous fission fragments.

An extremely important measurement will be the measurement of the number ν (the number of neutrons from a fission event) of this emitter. It is possible that it will prove to be considerably greater than for all the known nuclei which undergo spontaneous fission. According to theoretical predictions, the value of $\nu = 10$ cannot be excluded for element 114 [42]. However, in view of the low magnitude of the effect being observed, this is an extremely complex problem. Even more stringent requirements will be imposed on the background conditions than in the experiments with the proportional counters.

Experiments have been carried out recently in Dubna to measure the value of ν for a naturally spontaneously fissile emitter. A neutron detector was devised for this which was based on proportional counters filled with He^3. The main difficulty of the experiment is due to the neutron background created by cosmic rays. Despite the heavy-duty concrete shielding in which the equipment was installed, the background due to the penetrating μ-mesons from cosmic rays was several times higher than the expected neutron count. Experiments are currently being carried out in mines, with samples of lead ores and glasses containing a new natural spontaneously fissile emitter as impurity, in order to reduce the cosmic ray background.

The experimental progress in the region of superheavy transuranium elements (synthesis in accelerators) obviously is not expected to be an easy problem. It will be necessary to develop new methods of recording and identifying the superheavy nuclei; it will be necessary to accelerate new heavier particles (the heaviest of the particles used at present for synthesis is argon). However, argon and calcium are already making experiments possible for synthesizing certain isotopes of elment 114 in reactions proceeding

via the stage of compound nucleus formation with subsequent boil-off of several neutrons. It is true that the products in this case will be neutron-deficient nuclei, considerably remote from the supposed neutron shell N = 184. The reaction Pu + Zn → element 124 appears to be more promising as it permits an approximation to this shell, yet remaining at the same time – according to the number of neutrons – within the limits of the predicted region of relative stability [43].

Fission may turn out to be a promising method of synthesis [35, 44]. As a result of the fission process of nuclei, products are formed which are distributed over a wide range of charge and mass. Systematic investigation of the charge and mass distribution of fission fragments (nuclei of elements from gold to uranium have been irradiated with various ions up to argon) carried out over recent years in Dubna have shown that dispersion of the fission fragments according to mass increases rapidly with increase of the parameter Z^2/A of the compound nucleus, and the maximum of the distribution is shifted to the side of high Z-number [45] (Fig. 4). For example, in the reaction U^{238} (Ar^{40}, f) a significant yield of polonium and astatine nuclei has been detected [46]. If beams of accelerated xenon ions are available, all the known isotopes of the transuranium elements up to Z = 104 to 105 can be synthesized and, obviously, can be advanced somewhat beyond.

As a result of the fission of superheavy compound nuclei, isotopes are formed which are considerably more enriched in neutrons than in reactions with the formation of a compound nucleus. Given sufficiently heavy accelerated ions, for example such as xenon and uranium, we can be confident of producing isotopes of elements 114-126 as fission fragments, with the required number of neutrons ($N \approx 184$). However, the acceleration of such heavy particles is associated with, as yet, great although nonfundamental technological difficulties. Several projects are being worked out along this direction [47-49].

New possibilities have appeared recently in the search for superheavy transuranium elements in cosmic rays. Dielectric detectors have been designed which are capable of giving excellent resolution with respect to charge for heavy nuclei at relativistic velocities. In addition, the newest technology of cosmic flights will open up wider prospects for these investigations than the sounding balloon of P. Fowler.

The detection in nature already of one superheavy element shows that there exists a whole group of shorter-lived nuclei nearby, which can be synthesized and studied in the laboratory. The alluring outlook will then appear: to synthesize these short-lived nuclei using the natural superheavy element as targets. The next step, therefore, in studying the superheavy elements will be made by means of chemical methods. Although the chemical properties of the emitter being observed are still inadequately ascertained, we have confidence that in the not too distant future success will be achieved in this direction. If we are learning confidently to separate the emitter from the minerals and if we can obtain a sufficient number of atoms, then in principle we can prepare a target and irradiate it with various particles. In this we are confident that bombardment of the new element will permit us to synthesize elements which lie adjacent to it, in a similar way as the transuranides were discovered by the bombardment of uranium.

Thus, the achievements in the realms of physics and chemistry of the transuranium elements which have occurred during the last decades have been made possible by knowledge of the Periodic Law, thanks to the enormous amount of data on the relations between the properties of the different elements contained in it. In its turn, investigations in the region of the transuranium elements not only will deepen our knowledge of the nuclear properties of matter, but will also extend our concepts on the periodic system of elements and extend its boundaries.

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