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STUDIES OF THE HEAVIEST ELEMENTS AT DUBNA

ЛАБОРАТОРИЯ ЯДЕРНЫХ РЕАНЦИЙ

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Introduction

The discovery and studies of elements 102 through 105 at Dubna were extensively reviewed in the past 7^{1-57} , in particular, at the 3rd Geneva Conference on the Peaceful Uses of Atomic Energy in 1971 $7^{6,77}$.

During the two past years, the efforts of the JINR Laboratory of Nuclear Reactions in the field of transuranic elements were concentrated on further studies of kurchatovium (element 104) and nielsbohrium (element 105), as well as on the investigation of the possible region of superheavy elements with the unique cyclotron accelerated germanium and xenon ion beams, which were first made available at Dubna. The purpose of this lecture is to outline primarily some experimental results that may be of major interest to chemists. The contributions of JINR authors to the speculations concerning the new stability island, the future heavy ion accelerators and other transuranium topics have been discussed in other summaries and reviews $\frac{18-10}{2}$.

Kurchatovium (Ku)

In the 1966 experiments 117 our group succeeded in identifying chemically element 104 as ekahafnium. We were able to separate few atoms of the element obtained as a sum-atomic-number product by bombarding a 242 Pu target with 22 Ne ions, from the much larger number of actinide nuclei produced through various nuclear interactions. The transmuted atoms recoiling from the target were stopped in the stream of nitrogen gas and some gaseous chlorinating agents were introduced downstream to obtain molecules of chlorides. The separation method, being essentially frontal chromatography, was based on the much higher volatility of ekahafnium tetrachloride compared with that of actinide chlorides. $KuCl_4$ was eluted from the column and the gas was passed through a detector chamber whose walls were covered with mica sheets to detect fragments from the spontaneous fission events of kurchatovium nuclei: some 16 of these were registered in 1966 and about 60 were in the more extensive work published in 1969 $^{/12/}$.

The more recent work of our group $\frac{13}{13}$, which I shall describe in some detail, was connected with chemical isolation of the particular isotope, kurchatovium-259.

In 1969 Oganessian et al. $\frac{1}{14}$ carefully measured the half-lives of 259 Ku and 260 Ku and reported the values of 4.5 sec and 0.1 sec, respectively. We made an analysis of kurchatovium "decay curves" from our early chemical experiments to show that both isotopes might have been present. The contribution of ²⁵⁹K_u seems to have been predominant as the apparent half-life is much longer than 0.1 sec and as the chemical processing was not fast enough to permit the detection of the 0.1 sec activity at reasonable efficiency. The new experiments were designed to obtain "pure" 259 Ku. The flow rate of the carrier gas was made relatively low in order that the 0.1 sec activity might decay in the volume next to the target (see Fig. 1). TiCl₄ (2mm of Hg) and SOCL, (4.3 mm) vapours served as chlorinating agents. A part of the open tubular glass column (sections I and II in Fig. 1) was maintained at about 400 °C, while over another (III) a negative temperature gradient was superimposed. as is shown in the middle part of Fig. 1. Mica detectors were placed along the axis throughout sections II and III of the column. Section I served merely as a sort of filter. After terminating a run, which usually lasted for about a couple of days, the mica was etched chemically to look for fission fragment tracks. These are shown as circles and dots in the bottom of Fig. 1. Scandium and hafnium



Fig. 1. Top: The apparatus used for chemical isolation of kurchatovium-259. Plot: Temperature regime of the column. Bottom: Distributions of $44 {\rm mS}_c$, 170,171 Hf and fission fragment tracks along the column; dots - $^{259}{\rm Ku}$, circles- $^{256}{\rm Fm}$.

activities which were also produced in the targets served for monitoring the behaviour of non-volatile chlorides of the group ill elements and the volatile chlorides of group IV, respectively. The 44m S_c and 170,171 lif distributions were scanned using a Ge(Li) gamma-ray spectrometer. It is seen from Fig. 1 that there are two distinct groups of fission fragment tracks. One of them (circles) is associated with scandium chloride adsorption band. This is very likely to be due to the decay of 256 Fm . The other group (dots) is observed in section III, within

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the hafnium adsorption band and must be due to kurchatovium. It may be noted that less than 0.1% of scandium activity was found in section III of the column. Since only 6 tracks are associated with about 30% of Se in the beginning of the column, this non-volatile spontaneous fission activity can hardly account for a single track within the hafnium band. It was measured directly that hafnium atoms pass the distance from the target to the adsorption band site in some 0.4 sec. This most probably holds for ekahafnium too and the single track between the two groups may be explained as the decay of a 259 Ku atom "in flight", while most of the kurchatovium atoms undergo decay after they have been adsorbed on the column walls or the detector surface. The fact that there are essentially no tracks between two zones provides evidence for both the absence of ²⁶⁰ Ku in the column and no long-distance transportation of the non-volatile activity.

Combination of chemical and transmutation data (see Ref. $\frac{7137}{3}$) provides a conclusive assignment for $\frac{259}{8}$ Ku .

This work has proved once again that spontaneous fission of 259 Ku was observed in the 1966 Dubna chemical experiments. Ghiorso's group at Berkeley have repeatedly thrown doubt on the possibility of a considerable spontaneous fission branching in the decay of this nuclide. This was based on their spontaneous fission systematics rather than on any conclusive experimental data. Recently, Druin et al. 157 at Dubna have measured the SF/a ratio for 259 Ku quantitatively and found this to be about 7%.

Nielsbohrium (Ns)

Shortly after the discovery experiments of Flerov et al. 16 /our group performed chemical identification of 2-sec 261 Ns 17 . The experimental apparatus was similar to that shown in Fig. 1, except that temperature gradient was extended also over section II of the column and the highest temperature (section I) was only 300°C.

So the curve had much less precipitous slope. This allowed us to compare the volatility properties of element 105 chloride with those of various other elements. Vapours SOCL $_{2}$ (7 mm of Hg) and TiCl $_{4}$ (1.3 mm) were used of as chlorinating agents. The total of 18 decay events of nielsbohrium were observed on the mica detectors. In Fig. 2 distributions of some radioelements produced in heavy ion reactions and processed using the described equipment are plotted in an "integral form", i.e., the ordinate is the percentage of the activity deposited on the column before the given abscissa is reached. The nielsbohrium histogram (dashed line) cannot be directly related to the other distributions as the two following effects should be taken into account. First, there might, in principle, be one-two decays in flight (the expected number is 0.5) and so the first lower step of the experimental histogram is possibly shifted towards the higher temperatures compared to its "true" position. No correction was made for this, otherwise it would be necessary to make some arbitrary judgement about the origin of the tracks. Second, the position of bands in frontal chromatography must be a function of the duration of the experiment. For a short-lived activity this time equals

its lifetime, rather than the nominal length of the run. We have clearly observed a dependence of band position on lifetime, producing simultaneously several hafnium isotopes by bombarding a Sm target with Ne ions. The distribution of short-lived hafnium isotopes resulting from a long run could be traced afterwards by measuring their lanthanide descendants (see Fig. 2, bottom). Based on these data, we feel that the nielsbohrium distribution in Fig. 2 should be shifted some 50⁻ towards the lower temperatures if it is to be compared with the chemical behaviour of such activities as 170,171 it and 90 Nb that have half-lives of 10 hours or so. The corrected distribution is shown as the solid histogram in Fig. 2. This provides evidence that nielsbohrium chloride is more volatile than hafnium tetrachloride but perhaps less volatile than niobium pentachloride. This is a sort of behaviour that one would expect for ekatantalum.



Fig. 2. The integral distributions (see explanation in the text) of various activities on the temperature gradient in the experiments with nielsbohrium chloride. The experimental and corrected distributions of fission fragment tracks from N_8 decay are shown as dashed and solid histograms, respectively. Bottom: Distribution of iii isotopes of different half-lives.

In the recent years, many data on the physico-chemical properties of bromides of transition metals have been accumulated, which were scarce before. Based on these results, the present author suggested that bromides might be advantageous for studies on transactinide elements. experiments on the thermochromatographic Some behaviour of bromides of 1, Ta, Zr and other elements were performed in Transuranium Research Laboratory at ORNL by Keller, Silva and Zvara, It has been shown that a $Br_2 + BBr_3$ vapour mixture is an efficient brominating agent for the recoil atoms of the mentioned elements at temperatures as low as 200 °C. Under these conditions nickel metal is not attacked by the carrier gas. With the target chamber and target support made of nickel, the brominating mixture could be passed through the chamber Then the experimental apparatus is simpler than that shown in Fig. 1. Moreover, the chemical yield is higher than it was with downstream introduction of reactive spceies, when only some 50% of the thermalized atoms could be swept from the target chamber with inert gas, another 50% being irreversibly adsorbed on the walls of the chamber.

The apparatus used at Dubna is shown diagrammatically in Fig. 3 (top). It served for a quite recent study of nielsbohrium bromide. We bombarded a $800\mu g/cm^2$ americium target $(92\%^{243}A_m + 8\%^{241}A_m)$ on a 10μ m nickel foil with 22 Ne ions. The beam intensity was up to $3x10^{12}$ ions/sec, the particle energy being adjusted from the original 176 MeV to 122 MeV by aluminism degrading foils and the target substrate.

Our cylindrical target chamber was 21 mm long and 22 mm in diameter. It was maintained at a temperature of about 250 °C. The carrier gas was dry helium with bromine (50 mm of H_g) and BB_{r3} (1 mm of H_g) vapours. A thin wall 4 mm i.d. nickel tube served as the column. Mica sheets were placed throughout all the column except for the first 20 cm long section which was designed for separating from the gas stream nonvolatile molecular species and aerosols including the minute amount of sputtered target material. At the column exit we used a small charcoal filter to remove from the gas stream even the most volatile bromides that were not adsorbed in the column.

Over a large part of the column the temperature gradient was strictly constant as is indicated in Fig. 3.

The distributions of some activities traced by gammaspectrometric measurements of the 8 cm long section of the column after a 40 h run are presented in Fig. 3.

For some elements, oxide bromides are of similar volatility as bromides, and, of course, even the highest purity helium contains enough oxygen to react with tracer atoms. But it follows from the work of Leppert et al. $^{/18/}$, who investigated the reaction of a number of oxides with BBr₃ including most of the elements involved in our experiments, that with the excess of BBr₃, bromides are mainly obtained. This is why we discuss our results in terms of the volatility of bromides only. It can be noticed in consulting literature data compiled in Table 1, that the sequence of the absorption bands of known elements follow the order of their volatilities (boiling points).

Compound	Boiling (sublim point, °C	.) Melting point ° C
RbBr	1352	
Sc Bra	930 subl.	960
Zr Br ₄	360 subl.	450
Hf Br,	323 subl.	425
Nb Br5	356	
Ta Br ₅	344	
Ge Br	186	
As Br_3^{*}	221	
Se 2 Br 2	227	
Se Br4	75 dec.	

Table 1

The half-lives of the isotopes of known elements under study were of the 10 h order of magnitude. Again, due to



Fig. 3. The experimental apparatus, the temperature regime of the column and the differential and integral distributions of various activities in the experiments with nielsbohrium bromide. Circles and histograms are for fission fragment tracks from the decay of Ns. Bottom: Distribution of Hf isotopes of different half-lives.

the 2-sec half-life of nielsbohrium, the observed position of its histogram must be corrected for the difference in half-lives if the adsorption behaviour of nielsbohrium is to be compared with that of niobium. We again performed an experiment with some isotopes of hafnium of different half-lives in the given conditions. Thus the position of the nielsbohrium adsorption band could be corrected (solid line histogram in Fig. 3). It will be seen that nielsbohrium bromide seems to be less volatile than niobium pentabromide.

Elements 106 and 107

These elements seem to be within reach through heavy ion-induced reactions in the near future. Bombardments of 246 Cm with 22 Nr ions are in progress at Dubna to look for spontaneous fission of 263 106. This is why we sought for conditions favourable for their fast chemical identification by thermochromatography. The new elements are expected to be congeners of tungsten and rhenium, respectively, and so the study $^{/19}$ / was performed with 175,176 W and 177 Re produced by bombarding an erbium target with 12 C ions and a holmium target with 16 O ions.

We found that with SOCl₂ vapours (25 mm of Hg) fin nitrogen gas and with the temperature on the gradient part of our column ranging from 450° to 80° C, tungsten is deposited at about 150°C (Fig. 4). This volatility behaviour is very specific in the sense that both kurchatovium and nielsbohrium do not vield so volatile compounds. We believe that tungsten oxide chlorides are involved here. The transportation time for tungsten atoms from the target to the adsorption band was measured directly and found to be a fraction of a second. Thus, if the properties of element 106 chlorides are actually close to those of tungsten, one might succeed with the outlined thermochromatographic technique. Chlorides and oxide chlorides of rhenium are similar to the corresponding tungsten species as to high volatility. With this in mind, we looked for other possible working media. We found

that with wet air as a carrier gas and with 450° to 100° C on the column, rhenium is deposited at 300°C or so. Hf,Nb,Ta and W d'd not give any voistile species under these conditions. We believe that $HReO_4$ is responsible for this unique behaviour of rhenium atoms. Here again we have good prospects for achieving fast chemical identification of elements 107 provided that this is a good homologue of rhenium.



Fig. 4. The distribution of tungsten activity in the thermochromatographic processing. The hatched histogram was obtained with nitrogen gas purified from oxygen, the solid line histogram with 0.1% of O_2 in the carrier gas. Solid curve: temperature distribution.

Superheavy Elements

It is commonly believed that the most prospective method for synthesis of superheavy elements wou!d be nuclear reactions with very heavy ions. Flerov $\frac{20}{30}$ suggested that the compound-nucleus-fission like processes in, e.g., uranium plus uranium interactions might yield fragments with an extremely broad range of atomic and mass numbers and so they might serve as the source of nuclides that could not be produced by any other methods. Subsequently, Oganessian and Karamian $\frac{21}{7}$ made some estimates based on extensive studies of mass and charge distributions in fission induced by "common" heavy ions (from carbon through argon), to show that even xenon projectiles on uranium target might hopefully provide a reasonable yield of fragments close to $\frac{298}{114}$, which is the centre of the predicted superheavy region.

Complete fusion reactions always give only very neutron-deficient isotopcs, whereas the maximum stability of superheavy nuclides against spontaneous fission lies by chance near the β -stability valley. Nevertheless, Swiatecki ^{/22/} suggested that these reactions might be operative for production of superheavy elements. He proposes to synthesize some nuclides a few // units beyond ll4 ("overshoot"). These must very rapidly decay by alpha disintegration chains ^{/23/} to yield isotopes in the region of Z - 114 with better neutron to proton number ratio. Their half-lives are expected to be in detectable limits and spontaneous fission is to be their main decay mode. The bombardment of thorium with germanium ions to produce Z 122 and/or 120 was proposed as the most prospective target/projectile combination.

In the outlined experiments, the beams of germanium, xenon and maybe, uranium ions with energies of > 6 MeV/nucl. and reasonable intensities are needed. Most of the existing heavy ion machines are capable of accelerating ions not heavier than oxygen, with the exception of the Dubna U-300 cyclotron, which provides even zinc ions. Moreover, in 1971 Shelayev et al. 24 /succeeded in accelerating xenon ions. They combined the

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U-300 and U-200 to form a tandem system. The ${}^{136}\chi_e{}^{+9}$ ions are accelerated in the larger machine to an energy of about 120 MeV. This beam is fed through a 60 m beam-pipe into the smaller cyclotron, where the ions are stripped when passing through a 40 μ g/cm² carbon foil. The ${}^{136}\chi_e{}^{+36}$ ions are accelerated to the final energy of about 850 MeV. The bombardments are perfoimed using a circulating beam. At present, the beam intensity is about 3x10¹⁰ particles/sec. Similarly, ${}^{74.76}$ Ge⁵⁺ ions are fed from U-300 to

Similarly, $^{74.76}$ Ge⁵⁺ ions are fed from U-300 to U-200 and Ge^{+17,18,19} ions can be accelerated to an energy of about 600 MeV.

Following are some results of the recent Dubna experiments $^{26-28/}$ on the production of superheavy elements and the interaction problems of extremely heavy projectiles.

A number of bombardments have been performed to check the "overshoot" proposal. The experimental device used to look for spontaneous fission of the bombardment products is shown in Fig. 5. The shortest detectable



Fig. 5. Schematic view of the device for detecting shortlived spontaneous fission activities.

half-life was about 10^{-3} sec. Unfortunately, no spontaneous fission events which could be attributed to superheavy elements have been observed. The sensitivity of the technique in terms of cross sections was about 10^{-34} cm² for half-lives in the range from 10^{-3} sec to 1 day. More details concerning the conditions of the experiments are presented in Table 2.

Table 2

	rubic b	
Target + particle	Particle energy on the target, MeV, lab.system	Upper limit of cross section for superheavy ele- ments, cm ²
²³² Th + ⁷⁴ Ge	380 - 440 400 - 450 420 - 480 0 - 620 *	2x10 -34 1x10 -34 2x10 -34 3x10 -35
^{23 2} Th + ⁷⁶ Ce	$\begin{array}{r} 410 - 470 \\ 470 - 530 \\ 520 - 570 \\ 540 - 590 \\ \end{array}$	2x10 -34 5x10 -35 7x10 -35 7x10 -35 7x10 -35
²³⁸ U + ⁷⁶ Ge	0 - 600 * 440 - 500 510 - 570	4x10 -33 1x10 -34 1x10 -33

* Thick target; off-line measurements by contacting the target with track detectors to look for longer halflives.

The following data may help in explaining the lack of success. One might suppose that the fusion of the very heavy projectiles with the targets or the compound systems formed, possesses some novel features compared to the interaction of not so heavy ions. Based on some recent experiments $\frac{127}{1}$, this does not seem to be the case. The fission product yields from the bombardment of $\frac{238}{10}$ with $\frac{40}{10}$ A ions and of $\frac{181}{10}$ Ta with $\frac{84}{10}$ Kr ions were carefully measured. In the two combinations comp-

lete fusion must yield similar nuclei. Figure 6 shows that for the primary fission fragments, the mass and isotopic distributions observed in the bombardment with K_r ions coincide within the experimental errors with the results obtained with the more "common" target/ projectile combination. This means that the gross fission properties of the compound system do not change drastically with the projectile/target mass ratio. By integrating the mass yield distribution, the fission cross section for Th + Kr was found to be 350 mb for 550 MeV ions/27/



Fig. 6. a - The mass distribution of the primary fission fragments in the reaction 2380 ± 40 A (open circles) and 1875 ± 40 A (dots).



Fig. 6. b. The isotopic distribution of fission fragments (rare earth region) in the above reactions.

For the "overshoot" method it is essential that the excitation energy of the compound nucleus be low. For 232 Th + 74 Ge the lowest possible value (at the particle energy corresponding to the interaction barrier) was originally predicted to be some 15-20 MeV. The essential assumption involved was that the effective nuclear radius parameter r_0 is the same as for much lighter projectiles. Unfortunately the experiments /28/ did not confirm this assumption. Figure 7 shows that in the bombardment of Th+Ge different interaction channels are characterized by considerably different barriers. The reaction of transfer of several nucleons, that were traced by measuring the yields of some isotopes of U, Pa and Th,become observable at the energy corresponding to the value (+ 1.4 fm). Meanwhile, for fission normal L. fragment formation which was traced by the radiochemical isolation of gold activities, the interaction barrier



Fig. 7. Cross sections for fission and transfer reactions as a function of the particle energy in the bombardment of $^{232}{\rm Th}$ with $^{74}{\rm Ge}$ ions.

is some 30 MeV higher ($r_0 - 1.3$ fm). This implies that the lowest possible excitation energy of the compound nucleus must be 50 MeV or so. Thus the number of neutrons that should evaporate to remove the excitation energy is larger than it was expected. It is well known that the probability for the compound nucleus to avoid prompt fission decreases exponentially with the number of neutrons to be evaporated. So if the superheavy elements do exist, then the reason why they were not observed in these experiments may be the low mass number obtained and/or the too high fission probability of the compound nucleus. From this point of view, fission of extremely heavy compound systems might be a more plausible way of the production of superheavy elements. In this case one can expect that fragments will be formed in a broad range of atomic and mass numbers so that most of the superheavy region may be covered. Also, fragments of a particular mass will be created with very different excitations, including, hopefully, the low ones. The large width of these distributions, on the other hand, prevents high cross sections. It is likely that complete fusion with the formation of spherical system is not the necessary condition for these broad distributions and incomplete fusion might yield similar results.

Figure 8 shows some experimental data obtained in



Fig. 8. The isotopic and mass distributions of nuclei produced in the bombardment of 181 Ta with 136 Xe ions.

the bombardment of a tantalum target with xenon ions. The curves are close to the normal distribution function, which indicates the statistical character of the fission of the compound system involved. These distributions are close to what was expected from the extrapolation of fission yields with lighter projectiles $\frac{21}{2}$.

The isotopic composition was measured $^{28/}$ for the fission produced gold isotopes as a function of the projectile mass, when tantalum targets were bombarded. It can be seen from Fig. 9 that the yield of neutron-rich nuclides increases with the mass of the heavy ion. This trend is prospective from the point of view of the production of the superheavy elements.



Fig. 9. The mass distribution of A_{μ} isotopes in the bombardment of 181 T_B with various heavy ions.

It will be noticed in Fig. 8 that the yields for the fission products with A > 220 are much lower than what one would expect from the extrapolat.on of the lower mass part of the curve. This is very likely to be a consequence of the "cascade fission", i.e., the excited heavy fragments in turn undergo fission rather than the emission of prompt neutrons. This happens due to the high fissionability beyond A 220. Nevertheless, in the bombardment of 238 li with 136Xe ions the yield of the neutronrich and highly fissionable 254 Cf was still measurable $\frac{128}{1000}$, which seems to stem from the above mentioned broad distribution of fragments over excitation energy. Fragments with low excitation survive fission. This again gives hope to detect superheavy fission products. The higher the fission barrier of a nucleus, the higher both its yield and half-life; this is why the experimental search was directed towards rather long-lived activities (T_{1/2} I day).

As a result of long-term bombardments of thick uranium targets with $^{1.36}$ Ve lons, scarce spontaneous fission events from some activity with the half-life of a few months were observed. None of the known emitters possesses this sort of lifetime. The research program outlined for the future includes' measuring the number of prompt neutrons and kinetic energy of fission fragments, identification of mass number using a high-efficiency mass-separator and the chemical identification of the atomic number.

We believe that for chemical separations and studies of superheavy elements one may make use of thermochromatography. In particular, processing elements themselves will be possible as they must be rather volatile. Figure 10 from the paper of Eichler ²⁹ shows for scme elements the standard enthalpy of formation of the monoatomic gas, which equals the enthalpy of sublimation of monoatomic species. The values for superheavy elements were obtained by straightforward extrapolation. In the experimental part of his study Eichler bombarded a uranium oxide target with 660 MeV protons to produce radioisotopes of a large number of elements. He heated



Fig. 10. Standard enthalpy of gaseous monoatomic elements and the extrapolated values for the hypothetical superheavy elements. Numbers of the periods of the Mendeleev system and the atomic numbers of the elements are indicated in the bottom.

the target to 1000°C in a stream of hydrogen gas to find that in about three hours all elements with All>40 kcal/mole were quantitatively volatilized from the target. The yield of indium with $\Delta H = 60$ kcal/mole was still high. There was an excellent separation from actinide and other high melting transition elements. Separation factors of >10⁴ were observed. The products volatilized from the uranium oxide were condensed at the "injection port" of a 5 mm i.d. quartz thermochromatographic column filled with quartz support (150-200 μ m). Hydrogen at the flow rate of 20 ml/min was used as a carrier gas in 60 min experiments. A thermochromatogram of the elements of the sixth period of the Mendeleev Table is displayed in Fig. 11.



Fig. 11. Thermochromatographic separation of carrier free elements of the sixth period.

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The technique described has some very important advantages when one is going to work with new elements. First, compared with wet chemistry, there is, in the practical sense, no reagent introduced that might dilute the target material and separated fractions, and cause difficulties in the subsequent treatment. Second, due to the unique properties of hydrogen one can easily avoid losses of anything except for the hydrogen itself, using very simple chemical and/or cryogenic traps. Finally, it is easy to get samples suitable for alpha and spontaneous fission counting (cf. experiments on kurchatovium and nielsbohrium chemistry described above).

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